

KURNS

Progress

Report

2025



Institute for Integrated Radiation and Nuclear Science,
Kyoto University

KURNS Progress Report 2025

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Preface for the KURNS Progress Report 2025

It is our pleasure to announce that the KURNS Progress Report 2025 has been published. This report contains all the accomplishments of research and related activities at the Institute for Integrated Radiation and Nuclear Science, Kyoto University (KURNS) during the fiscal year 2025 (hereafter called FY2025). Many research subjects have been enrolled in FY2025, which cover various fields of nuclear science and technology, materials science, radiation life science and radiation medical science. It means that our institute continues to play a distinctive role as a Joint Usage/Research Center, promoting an extensive range of studies from fundamental to applied research with research reactors and accelerators.

In FY 2025, KUR operated for 1077 hours and KUCA was 0 hours. Note that here KUCA has zero operating hours due to efforts to move to low enriched uranium fuel. KUR concluded its operational use on April 23, 2026. KUR has always been at the center of the institute, serving not only as its symbol but also as a source of cohesion. Marking this milestone, the institute is now embarking on a new chapter. In addition to KUR and KUCA, we have conducted joint research at hot laboratories, various accelerator facilities, and so on. In total, we accepted 3698 man-day researchers and students for using research facilities and for attending scientific meetings. We will continue to promote joint research by making full use of the institute's diverse and extensive range of unique facilities and specialized equipment.

We strive for safe and stable operations for nationwide use, making it our primary mission to provide scientists with the opportunity to conduct research and education. We are happy to dedicate our support to enable users to conduct significant interdisciplinary research at KURNS.

Kumatori, June 2, 2026

Ken Kurosaki Director,

KURNS

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**I. ANNUAL SUMMARY OF EXPERIMENTAL
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I-1. PROJECT RESEARCHES

Project 1

Development of Neutron Imaging Application

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OBJECTIVES and ALLOTTED RESEARCH SUBJECT: Neutron imaging provides valuable information which cannot be obtained from an optical or X-ray imaging. The purpose of this project is to develop the imaging method itself and the experimental environment for expanding the application area of neutron imaging. The allotted research subjects are as follows:

ARS-1: Measurements of Boiling Two-Phase Flow Dynamics in a Heated Tube (D. Ito et al.)

ARS-2: Void Fraction of Silicon-oil Flow with Microbubbles in Horizontal Tube (H. Asano et al.)

ARS-3: Adaptation of Neutron Radiography for the Evaluation of Two-phase Flow (H. Umekawa et al.)

ARS-4: Observation of Condensation on a Single Flat Tube (R. Matsumoto et al.)

ARS-5: An integrated neutron radiography-based observation of spalling behavior in concrete containing ground granulated blast furnace slag (M. Kanematsu et al.)

ARS-6: Visualization of the Reactant Solution Distribution in a Mixer of a Super-critical Hydrothermal Flow-Type Reactor Using Gd³⁺ Ion Solution as a Tracer (S. Takami et al.)

ARS-7: Visualization of Root Water Uptake in Organic Substrates through Three-Dimensional Root Reconstruction from Cylindrical Panoramic Imaging (U. Matsushima et al.)

ARS-8: In-situ Observation of Lithium Migration in Solid Electrolyte by Using Neutron Radiography (S. Takai et al.)

ARS-9: Measurement of coolant distribution in a flat laminate vapor chamber utilizing neutron radiography (K. Mizuta et al.) (not performed)

MAIN RESULTS AND THE CONTENTS OF THIS REPORT:

ARS-1 performed visualization of boiling two-phase flow by high-speed imaging. A stainless-steel tube with an inner diameter of 7mm was electrically heated and the boiling two-phase flow was observed in the tube by high-speed neutron imaging at 1,000 fps.

ARS-2 applied to neutron imaging to void fraction measurements of silicone oil/gas two-phase flow to investigate the effect of void distribution on the heat transfer characteristics. From measurement results, at flow rate of 1.0 l/min, the bubbles appeared to flow more in the upper region due to buoyancy, but at 2.0 l/min, they were observed to be homogeneously dispersed.

ARS-4 evaluates the condensation on a single flat tube. In this experiment, dropwise condensation was observed by neutron imaging, and as the droplets grew and coalesced, increasing in size, their downward motion due to gravity was observed.

ARS-5 investigates the integrated neutron radiography-based observation of spalling behavior in concrete containing ground granulated blast furnace slag.

ARS-6 visualized the reactant solution distribution in a mixer of a supercritical hydrothermal flow-type reactor using Gd³⁺ Ion solution as a tracer.

ARS-7 combined a three-dimensional (3D) root surface model reconstructed from cylindrical panoramic photographs with neutron D₂O absorption images, enabling non-destructive correlation of root position with water uptake in organic substrates.

ARS-8 applied the electric field to the model cell to observe the lithium migration in the solid electrolyte through neutron imaging.

ARS-9 intended to investigate the coolant distributions in a flat vapor chamber by using neutron radiography, however, the experiments were not performed in this year.

Measurements of Boiling Two-Phase Flow Dynamics in a Heated Tube

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INTRODUCTION: Neutron radiography (NRG) is a powerful technique for visualizing multi-phase flow phenomena. It has been widely applied to thermal-hydraulic studies, including boiling two-phase flows and void fraction measurements. In addition to steady boiling flows, NRG is especially useful for investigating highly transient phenomena such as reflooding during emergency cooling processes. Reflooding is a key process in nuclear reactor safety, where cooling water re-enters a heated channel and quenches the overheated surface. The complex interaction between liquid penetration, vapor generation, and quenching front propagation remains not fully understood due to limited visualization techniques in opaque structures. The objective of this study is to visualize and quantify the reflooding process in a heated tube using high-speed neutron imaging.

EXPERIMENTS: Neutron imaging experiments were conducted at the B-4 facility of Kyoto University Research Reactor (KUR). A high-speed imaging system consisting of an optical image intensifier coupled with a high-speed camera was used to capture neutron transmission images at 1000 fps. A stainless-steel tube with an inner diameter of 7 mm was used as the test section. The tube was electrically heated by Joule heating using a DC power supply. After reaching a high-temperature condition, cooling water was injected into the test section to initiate the reflooding process.

RESULTS: The instantaneous neutron transmission images during the reflooding process are shown in Fig. 1. At the onset of water injection, a sharp decrease in void fraction is observed near the inlet, indicating the penetration of the liquid front. The liquid front propagates upward along the tube, accompanied by complex vapor-liquid interactions. Behind the quenching front, intermittent vapor generation and condensation behavior are observed. The high-speed NRG successfully captured these rapid transient behaviors, demonstrating its capability to visualize reflooding phenomena inside opaque metallic channels. These results indicate that high-speed neutron imaging is an effective tool for investigating the detailed mechanisms of reflooding, including liquid front dynamics and transient vapor structures.

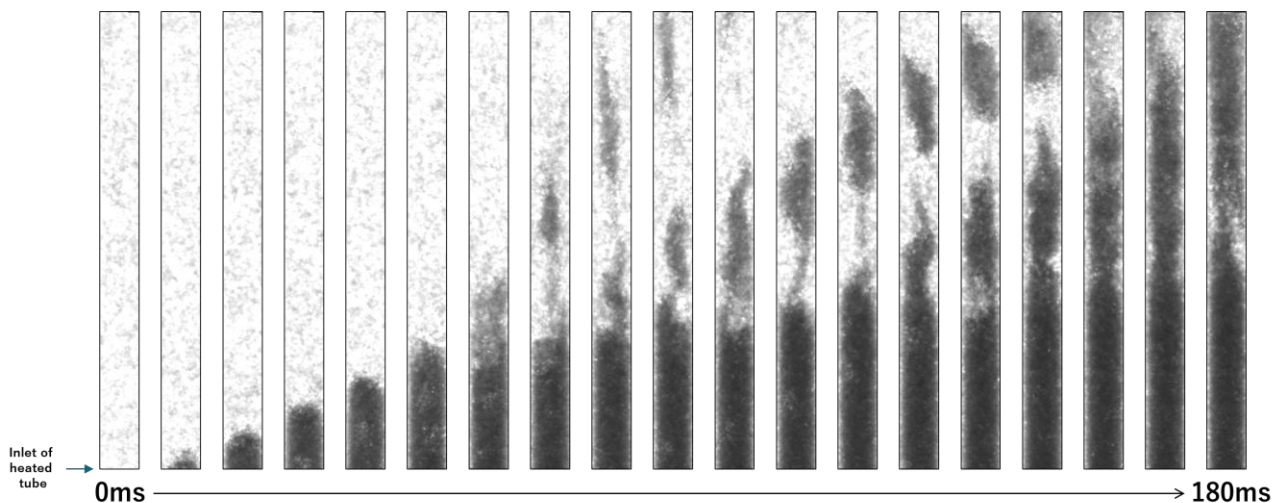


Fig.1. Instantaneous neutron transmission images during the reflooding process.

Void Fraction of Silicon-oil Flow with Microbubbles in Horizontal Tube

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INTRODUCTION: A method to reduce frictional resistance by adding microbubbles to the lubricating oil in automotive cooling systems is under investigation. In our previous research, we measured the heat transfer coefficient and pressure loss in horizontal pipe flow and found that no significant effect on pressure loss was observed in the laminar flow region. However, the results showed that adding microbubbles at a gas volumetric flow rate ratio above a certain threshold improved the heat transfer coefficient. The measurement results of the channel cross-sectional average void fraction by using an electrostatic capacitance sensor showed that, at low gas volumetric flow rate ratio, the void fraction was lower than the gas volumetric flow rate ratio; however, at high gas volumetric flow rate ratios where an increase in heat transfer coefficient was observed, the void fraction became higher than the gas volumetric flow rate ratio. This is considered to be because bubbles flowed not only along the center of the tube but also along the tube wall, where the liquid velocity is lower. Therefore, to clarify the distribution of microbubbles in the channel, visualization experiments using neutron radiography were conducted.

EXPERIMENTS: Silicone oil was used at 36°C. Dry air from an air cylinder was injected upstream of the test section, and microbubbles were generated by the strong shear forces produced by rotational stirring. The bubble diameter measured in preliminary experiments was approximately 90 μm , based on the Sauter mean diameter. A stainless-steel tube with an inner diameter of 3 mm and a wall thickness of 0.5 mm was used to visualize the flow within the horizontal tube. The temperature of the silicone oil was set to 36°C to match the dynamic viscosity coefficient (8.07 mm²/s) of the lubricating oil at 80°C in the actual equipment.

RESULTS: Brightness distribution perpendicular to the tube axis is shown in Fig. 1. The right side of the horizontal axis represents the top of the pipe. These are average values over a 260-pixel (4.42 mm) section along the axis. No changes in the brightness distribution were observed at a gas volumetric flow rate ratio of 10% or less for 1.0 l/min, or at 12% or less for 2.0 l/min, respectively. The reason is that silicone oil has a high gas absorption capacity, and the pressure in the measuring section was higher at the higher flow rate. At a flow rate of 1.0 l/min, the bubbles appeared to flow more in the upper region due to buoyancy, but at 2.0 l/min, they were observed to be homogeneously dispersed. However, in this experiment, the presence or absence of bubbles near the tube wall could not be confirmed.

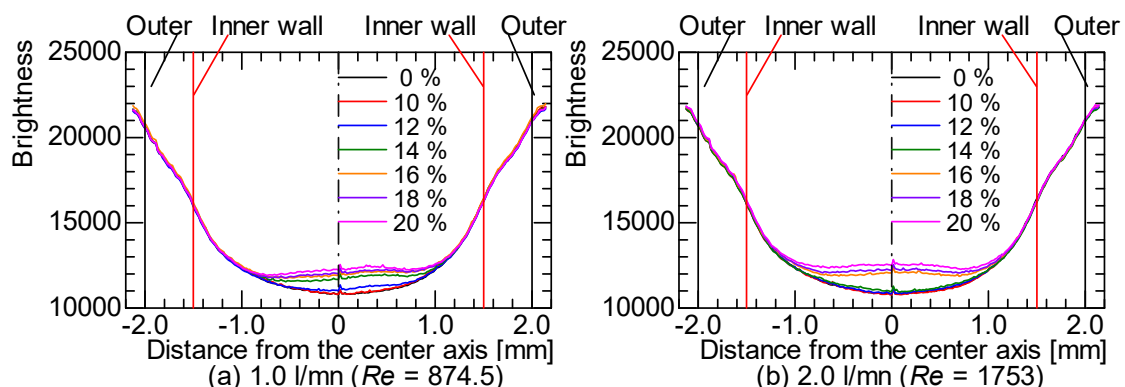


Fig. 1 Brightness distribution perpendicular to the tube axis.

Adaptation of Neutron Radiography for the Evaluation of Two-phase Flow

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INTRODUCTION: Rapid cooling of high-temperature and high-heat-flux materials is a key issue in several industrial fields, such as nuclear power plants, electronic devices and metal processing. In these applications, a detailed understanding of film boiling is required. For effective rapid quenching, continuous supply of cooling liquid, and the continuous removal of generated vapor should be achieved. In this study, to clarify the fundamental characteristics of these phenomena, the quenching of bottom-closed heated pipe was visualized using thermal neutron radiography.

EXPERIMENTS: In this investigation, the three kinds of bottom-closed tubes with inner diameters of 4mm, 6mm and 8mm were heated to a predetermined temperature (800 deg.C), after which water was injected from the top of the tube. During the quenching process, wall temperatures were recorded, and internal flow behavior was dynamically visualized using neutron radiography.

RESULTS: Figure 1 shows the visualization results during the quenching process. These figures correspond to the sequential images around the quenching time indicated by thermocouple (TC1). In these figures, especially at the bottom re-gion of 6mm and 8mm tubes, inverted annular flow regime can be observed clearly. Figure 2 is time-strip image whole quenching process. In this figure, quench front line can be evaluated, and these lines show good agreement with the estimation by temperature trend. Moreover, occurrence of flooding can be observed near the bottom region of 6mm and 8mm tubes.

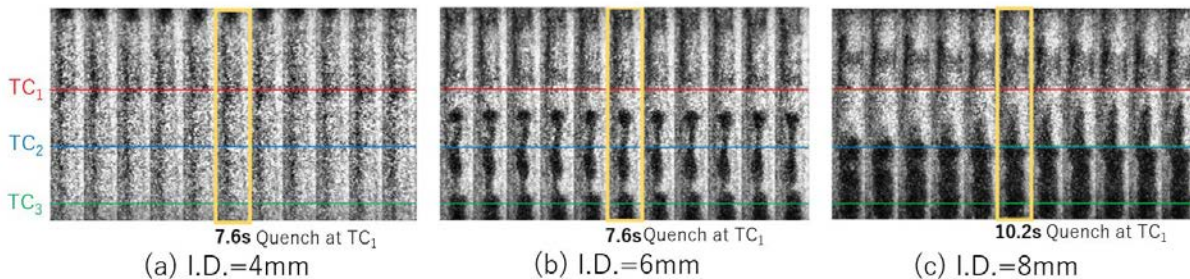


Fig.1 Sequential Images of Quenching Phenomena around TC1 (1000fps).

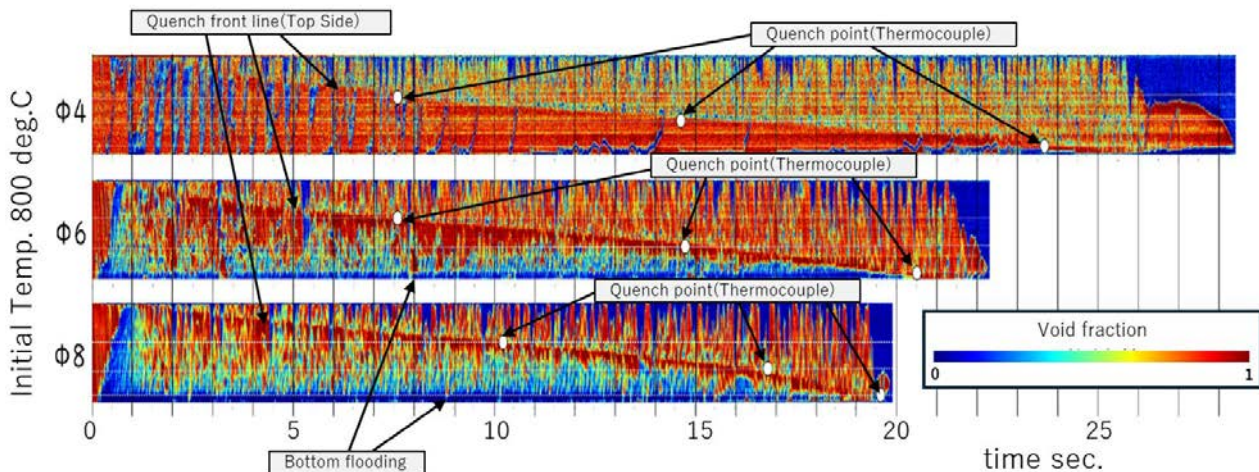


Fig.2 Time-strip Image of Quenching Phenomena.

Observation of Condensation on a Single Flat Tube

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INTRODUCTION: The development of heat exchangers employing aluminum flat tubes for outdoor units of heat-pump air conditioners has been progressing. Although aluminum has a lower thermal conductivity than copper, which has traditionally been used as a heat-exchanger material, its light weight and ease of forming allow for the miniaturization of refrigerant flow channels. Reducing the channel diameter increases the heat-transfer surface area and decreases the required refrigerant charge.

Because aluminum is hydrophilic, condensation occurs when exchanging heat with ambient air, leading to filmwise condensation in which a continuous liquid film forms on the surface. When filmwise condensation develops on the heat-exchanger surface, the liquid film introduces additional thermal resistance and reduces the heat-transfer coefficient. Moreover, partial blockage of the air-flow path by the condensate disturbs the air stream and increases pressure loss, ultimately degrading the heat-exchange performance. In general, heat exchangers employ fins to secure sufficient heat-transfer area; however, the complex fin geometry makes it difficult to analyze condensation behavior as a simple phenomenon.

Therefore, in this study, condensation experiments are conducted on a single flat tube as a fundamental investigation, with the objective of measuring the condensation distribution and liquid-film thickness.

EXPERIMENTS AND RESULTS: A single flat tube with a width of 18.6 mm and a thickness of 2 mm was vertically installed, and a refrigerant at 0 °C was circulated inside the flat tube to cool its surface temperature. An experiment was then performed with humidified ambient air to form a condensate film on the tube surface. The experiment was conducted for approximately 40 minutes, during which the behavior of the liquid film was observed.

The condensate water distribution image after 40 minutes obtained by neutron radiography is shown in Fig. 1. Because the amount of moisture supplied to the air was small, filmwise condensation did not occur. Instead, dropwise condensation was observed, and as the droplets grew and coalesced, increasing in size, their downward motion due to gravity was observed. This periodic coalescence resulted in intermittent downward flow within the liquid film, as confirmed during the observations.

The behavior of condensate can be observed using neutron radiography.

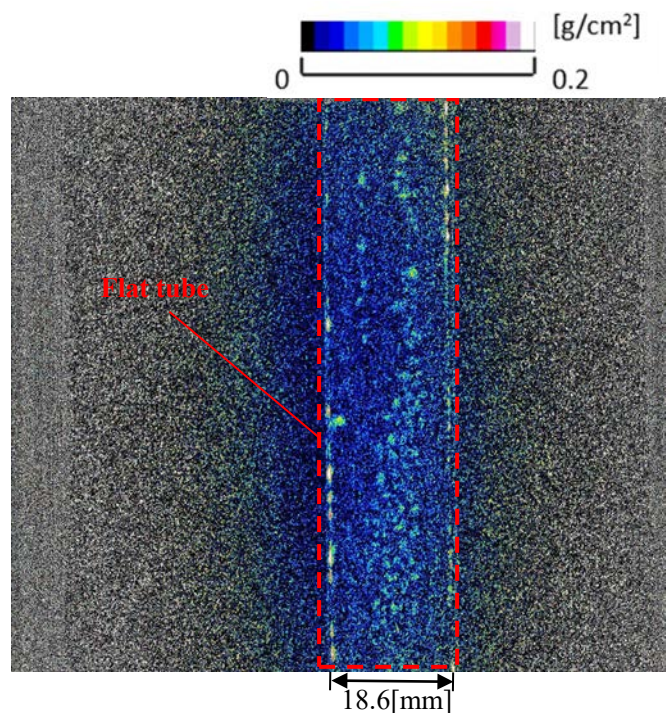


Fig.1 Condensate water distribution on a flat tube.

An integrated neutron radiography-based observation of spalling behavior in concrete containing ground granulated blast furnace slag

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INTRODUCTION: Understanding moisture behavior in concrete during fire exposure is essential for elucidating the spalling mechanism [1]. In this study, heating experiments were conducted on concrete containing ground granulated blast furnace slag using neutron radiography for integrated observation. The influence of the admixture on spalling was investigated based on temperature, pressure, and moisture conditions within the concrete.

EXPERIMENTS: Concrete specimens (70×100×30 mm) with a water-to-binder ratio (W/B) of 22% are prepared. Two mix conditions were considered: one using ordinary Portland cement (N) and the other in which 40% of the binder was replaced with ground granulated blast furnace slag (BB). Temperatures and internal pressures were measured at the positions shown in Fig.1. The specimens were heated from the bottom surface using a quartz heater. Neutron radiography was performed at B-4 port of KUR.

RESULTS: Fig. 2 and Fig. 3 show the results of the heating experiment. Fig. 2 presents the temporal change in relative moisture content obtained by neutron radiography, while Fig. 3 shows the temporal changes in temperature and pressure in the BB specimen. The relative moisture content shown in Fig.2, expressed per unit paste, was determined using separately prepared calibration specimens, following Ref. [2]. As shown in Fig. 2, a difference in drying rate, which is considered to influence spalling behavior, was observed between N and BB. In addition, the moisture distribution could be tracked over time from the relative moisture content images. As shown in Fig. 3, in the BB specimen, pressure increased at a position 5 mm from the heated surface, whereas no significant increase was observed at 10 mm. Further detailed measurements and analyses are required to elucidate the fundamental mechanisms of spalling.

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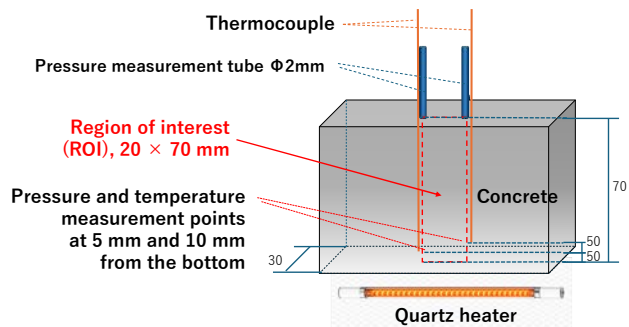


Fig. 1 Outline of concrete specimen

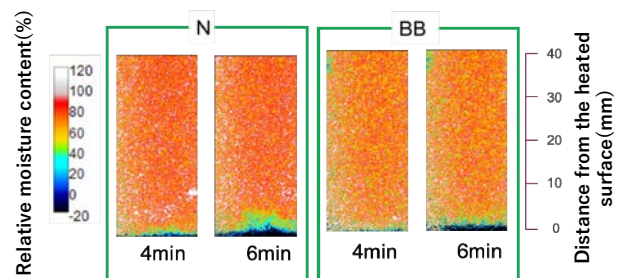


Fig. 2 change in relative moisture content

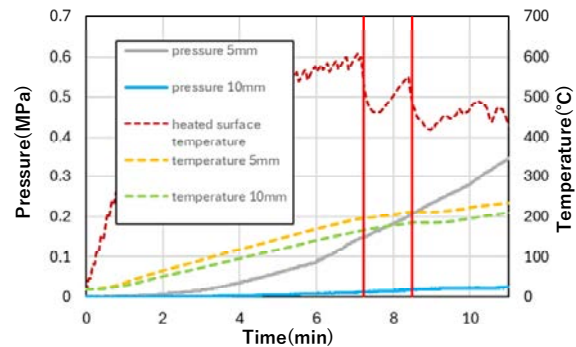


Fig. 3 Relationship between pressure and temperature in concrete under heating (BB)

Visualization of the Reactant Solution Distribution in a Mixer of a Supercritical Hydrothermal Flow-Type Reactor Using Gd^{3+} Ion Solution as a Tracer

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INTRODUCTION: Supercritical hydrothermal synthesis is a method for producing metal oxide nanoparticles in supercritical water. In flow-type reactors, the aqueous metal ion solution is instantaneously heated by mixing with a stream of heated water in a mixer. Previous studies have shown the density distribution of water in the mixer, which reflects the temperature distribution there. In this study, we attempted to visualize the distribution of the reactant solution by using Gd^{3+} ions as a tracer in neutron radiography.

EXPERIMENTS: The measurements were carried out at the B4 port of the Kyoto University Reactor (KUR). The experimental setup was similar to that used in previous studies.¹⁻⁴ In this study, a 0.01 mol/L Gd^{3+} ion solution was delivered using a high-pressure pump and mixed with supercritical water in a mixer. Neutron radiography images were continuously acquired with an exposure time of 30 s. A neutron radiography image of the mixer filled with air was also acquired and subtracted from the neutron radiography images obtained during operation.

RESULTS: Fig. 1 shows neutron radiography images of a mixer in a flow-type supercritical hydrothermal reactor. The Gd^{3+} ion solution reached the mixer 6 min after the start of $\text{Gd}(\text{NO}_3)_3$ supply. The images indicated that the darker region, corresponding to the presence of Gd ions, was located in the lower part of the side tube. The image at 6 and 10 min further showed that the dark region was present on the right side of the vertical tube. These behaviors were consistent with the water density distribution reported in previous studies. Therefore, the density profile mainly resulted from mixing between supercritical water and room-temperature water, rather than from heat conduction through the inner wall of the mixer. These images also revealed the progression of plugging in the mixer during hydrothermal synthesis.

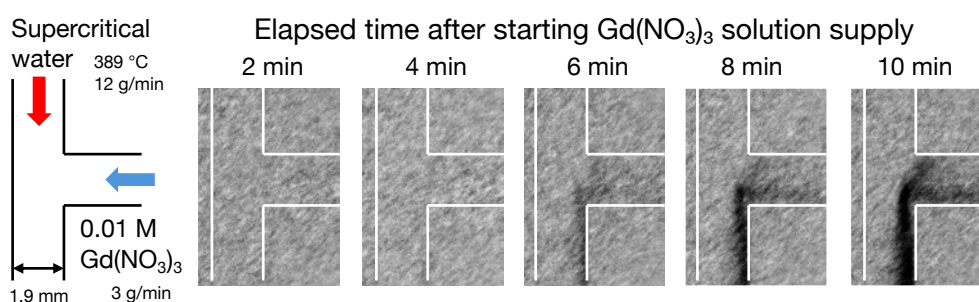


Fig. 1. Neutron radiography images of a mixer in supercritical hydrothermal reactor during Gd^{3+} solution supply.

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Visualization of Root Water Uptake in Organic Substrates through Three-Dimensional Root Reconstruction from Cylindrical Panoramic Imaging

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INTRODUCTION: Non-destructive observation of root systems in organic substrates is essential for understanding plant water uptake. Neutron imaging visualizes water distribution with high hydrogen sensitivity, and a deuterium oxide (D₂O) tracer enables selective imaging of root-mediated water uptake. However, the low contrast between roots and organic substrates hinders accurate root localization in neutron images. To address this limitation, we combine a three-dimensional (3D) root surface model reconstructed from cylindrical panoramic photographs with neutron D₂O absorption images, enabling non-destructive correlation of root position with water uptake in organic substrates.

EXPERIMENTS: Komatsuna (*Brassica rapa* var. *perviridis* ‘Wakami’) was grown in quartz glass tubes (22 mm i.d., 120 mm high) filled with a rice-hull/soil substrate (3:1 v/v; $n = 10$). Each tube was rotated on a motorized stage and photographed using a digital camera. Panoramic unrolled images were synthesized through slit-scan compositing, and root regions were extracted using a Frangi vesselness filter, then back-projected onto a cylinder to generate a 3D root surface model. Virtual transmission images were subsequently produced from this model by parallel projection. Neutron imaging was conducted at the E2 beamline of the Kyoto University Research Reactor, and a relative transmittance map was computed from the ratio of images acquired before and after D₂O supply. A geometric correction that maximized the overlap between root regions and D₂O-absorbing regions was then applied.

RESULTS: Panoramic unrolling of the rotated glass tubes (Fig. 1a, b) enabled registration of detected root regions onto the cylindrical surface (Fig. 1c), from which virtual transmission images were generated (Fig. 1d). Overlaying the neutron D₂O absorption map (Fig. 1e) revealed a systematic vertical shift of $+88 \pm 12$ px and a scale factor of 1.15 ± 0.05 between the two modalities; geometric correction subsequently aligned the root regions (green) with the D₂O-absorbing regions (white) effectively (Fig. 1f). However, grid-based correlation yielded only a weak positive correlation coefficient ($r \approx 0.1$, $n = 10$), constrained by the low signal-to-noise ratio of thin-root water uptake signals within the thick substrate and by the inherent limitation of 2D projection capturing only the cylindrical surface roots.

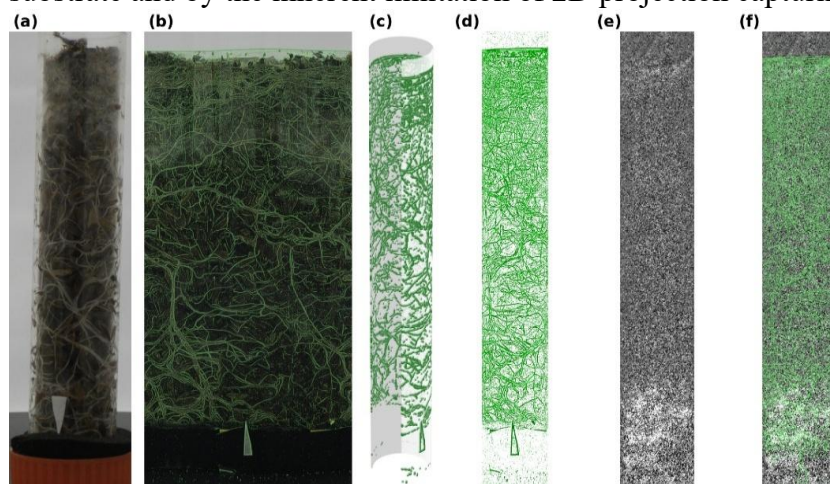


Fig. 1. (a) Quartz glass-tube pot used for Komatsuna cultivation. (b) Cylindrical panoramic unrolled image with root regions detected by a Frangi filter overlaid in green. (c) Three-dimensional reconstruction of the root system on the cylindrical surface (cut model). (d) Virtual transmission image generated from the reconstructed root model. (e) Neutron relative transmittance map showing D₂O absorption. (f) Overlay of the virtual transmission image (green) and the neutron D₂O absorption map (white) after geometric correction.

In-situ Observation of Lithium Migration in Solid Electrolyte by Using Neutron Radiography

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INTRODUCTION: In recent years, research on All-Solid-State Batteries (ASSBs) has been intensively pursued from various perspectives. Lithium ions migrate from the anode to the cathode during discharge, the visualization of which is one of the key challenges for the investigation of ASSBs. We have carried out the lithium diffusion coefficient measurement through the neutron radiography (NR) technique by exploiting the large differences in neutron attenuation coefficients between ⁶Li and ⁷Li [1-3].

In the present study, we applied the electric field to the model cell to observe the lithium migration in the solid electrolyte through the neutron imaging.

EXPERIMENTS: ⁷Li-LAGP (⁷Li_{1.5}Al_{0.5}Ge_{1.5}(PO₄)₃) solid electrolyte and ⁶Li-LMO (⁶LiMn₂O₄) electrode material were prepared by the conventional solid-state reaction method using lithium isotopes. A test cell of Cu / ⁶Li-LMO / PEO / ⁷Li-LAGP / PEO / ⁶Li-LMP / Al was fabricated and conducted a current at 200 - 250°C. The mounted amount of cathode and anode materials are 3 and 6 mg, respectively. Before, after, and during the electrolysis, NR images were measured by CCD camera at the B4 guide tube in IIRNS, Kyoto University.

RESULTS: Fig. 1 represents the (a) NR image of the test cell before the electrolysis, (b) divided image of the cells (after electrolysis by before), and numerical plot of the divided image. Anode and cathode correspond to the right and left side of the image in (b) and (c). Through electrolysis, a part of ⁶Li of the anode is introduced into the electrolyte, decreasing the gray value at the anode with increasing it at the electrolyte in the vicinity of the interface. On the other hand, when ⁷Li is introduced into cathode, gray value decreases at the cathode interface. The trend of the variation in radiography image can explain the direction of lithium migration in the solid electrolyte.

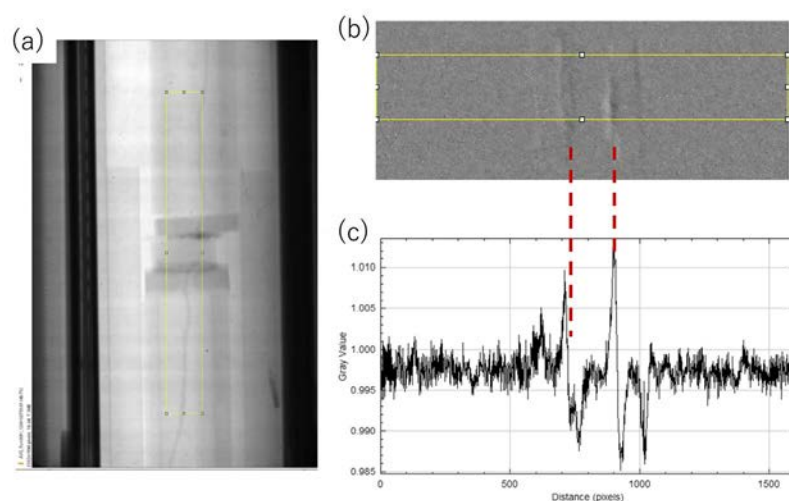


Fig. 1 (a) Neutron radiography image of the test cell heated up to 250°C. (b) Divided image of the cell after the electrolysis by the pristine one. (c) numerical plot of the divided image. Right side: anode and left side: cathode.

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Applications of Neutron Imaging to Cultural Heritage and Paleontology

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INTRODUCTION: Non-destructive imaging techniques play a crucial role in the study of cultural heritage objects and paleontological specimens. Among these, X-ray CT is widely used due to its accessibility and high spatial resolution. However, X-ray imaging is primarily sensitive to electron density and often provides limited contrast for materials with similar atomic numbers. Neutron imaging, in contrast, is sensitive to nuclear interactions and exhibits high contrast for light elements such as hydrogen, even in the presence of dense materials. This complementary sensitivity makes neutron imaging particularly valuable for investigating fossil structures embedded in mineral matrices. In this study, neutron CT and X-ray CT were applied to fossilized bivalves. The objective is to compare the imaging characteristics of both techniques and evaluate the effectiveness of neutron imaging for paleontological analysis.

EXPERIMENTS: Neutron CT measurements were carried out at the B-4 port of the Kyoto University Research Reactor (KUR). A series of neutron transmission images were acquired while rotating the specimen, and tomographic reconstruction was performed to obtain three-dimensional internal structures. The sample used in this study is a fossilized bivalve collected in Fukui Prefecture. The fossil is embedded in a sedimentary matrix, making internal observation challenging by conventional methods. For comparison, X-ray CT measurements were also conducted using a general-purpose laboratory X-ray CT system. The reconstructed volumes of both neutron and X-ray CT datasets were analyzed to evaluate differences in contrast, visibility of internal structures, and the detectability of features such as shell morphology and internal fillings.

RESULTS: The reconstructed cross-sectional images obtained from neutron CT and X-ray CT are shown in Fig. 1. X-ray CT clearly visualizes the overall morphology of the fossil; however, the contrast between the fossil shell and the inclusions is limited due to their similar X-ray attenuation coefficients. In contrast, neutron CT provides enhanced contrast between the fossil and the inclusions. This enables improved identification of internal structures that are difficult to distinguish in X-ray CT images. Furthermore, neutron CT reveals fine structural features within the fossil, including variations in shell density and possible internal voids or infillings. The complementary nature of neutron and X-ray imaging is evident: X-ray CT is effective for capturing high-resolution morphological information, while neutron CT provides unique sensitivity to compositional differences, especially involving light elements. The combined use of neutron and X-ray CT provides a more comprehensive understanding of paleontological specimens, highlighting the potential of neutron imaging in cultural heritage and fossil studies.

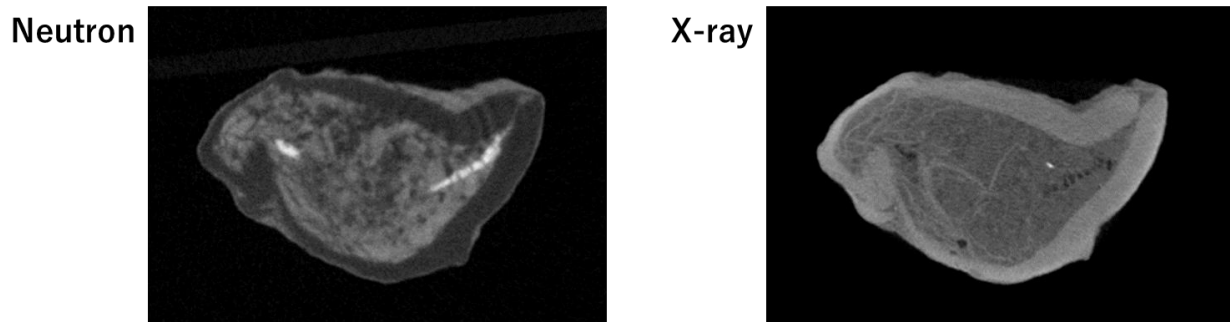


Fig.1. Reconstructed cross-sectional images obtained from neutron CT and X-ray CT

I-1. PROJECT RESEARCHES

Project 2

Estimation for 3D Distribution of Biological and Chemical Doses for BNCT

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BACKGROUNDS AND PURPOSES: For the further advancement of BNCT, it is important to improve the estimation for biological effect due to neutrons. At Heavy Water Neutron Irradiation Facility of Kyoto University Reactor (KUR-HWNIF), the fundamental studies for BNCT in the fields of medicine, biology, chemistry and pharmacology, etc., have been performed. Through these studies, the difficulty in the estimation for neutron biological effects is being acutely realized. In the BNCT irradiation field, it is difficult to monochromatically generate neutrons with a specific energy, and neutrons with the various energy ranges such as thermal, epi-thermal and fast neutrons, and also gamma rays are mixed. Furthermore, the neutron energies and the types and energies of secondary charged particles due to the nuclear reactions in living body are different. The biological effect estimation for BNCT is being studied by multiple groups in the world, but the consistent estimations have not been possible due to the difficulty of estimation for the biological effects, particularly regarding neutrons. The final goal of this project research is to develop a consistent estimation method of the biological effects for BNCT. In the third year of this research project, 2025, the developments of the estimation method for the 3D dose distribution, focused on the biological and chemical effects were continued. For the biological dose estimation, the estimation methods using three-dimensional co-culture, which can model the structure of living tissues and organs, were mainly studied. For the chemical dose estimation, the estimation methods using chemical dosimeters such as gel dosimeters were studied.

RESEARCH SUBJECTS: The collaboration and allotted research subjects (ARS) were organized as follows;

ARS-1 (R7P2-1): “Establishment of beam-quality estimation method in BNCT irradiation field using dual phantom technique (IX)”, Y. Sakurai, et al..

ARS-3 (R7P2-3): “Development of a BNCT evaluation system using a three-dimensional model”, K. Igawa, et al..

ARS-4 (R7P2-4): “Verification of BNCT effect on hematological cancer cells and dosimetry for expansion of BNCT case”, S. Yoshihashi, et al..

ARS-5 (R7P2-5): “Development and evaluation of 3D gel dosimeter for the measurement of dose distribution in BNCT”, S. Hayashi, et al..

ARS-6 (R7P2-6): “Establishment of three-dimensional dose distribution estimation method for BNCT using radiochromic gel dosimeter (III)”, Y. Sakurai, et al..

ARS-7 (R7P2-7): “Evaluation of boron dose and dosimetric performance of NC-RFG for BNCT”, J. Shimbo, et al..

ARS-9 (R7P2-9): “Development of an epithermal neutron spectrometer for boron neutron capture therapy”, Y. Ota, et al..

ARS-10 (R7P2-10): “Measurement of wavelength-dependent luminescence of BaFBr:Eu for beam quality assurance of BNCT”, K. Tanaka, et al..

ARS-12 (R7P2-12): “Experiments for boron dose measurement with glass dosimeter in BNCT”, Y. Morizane, et al..

For ARS-2 (R7P2-2) and ARS-11 (R7P2-11), the experiments continued until April 2026, and these subjects were not completed by the end of fiscal year 2025. ARS-8 (R7P2-8) could not be performed due to the scheduling and preparation issues. So, the reports of these subjects do not appear.

Establishment of Beam-quality Estimation Method in BNCT Irradiation Field using Dual Phantom Technique (IX)

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INTRODUCTION: Before the start of treatment with BNCT, the relative biological effectiveness (RBE) for the fast neutrons incident to the irradiation field must be estimated. Measurements of RBE are typically performed by biological experiments with a phantom. Although the dose deposition due to secondary gamma rays is dominant, the relative contributions of thermal and fast neutrons are virtually equivalent in a water and/or acrylic phantom. Uniform contributions to the dose deposited from thermal and fast neutrons are based in part on relatively inaccurate dose information for fast neutrons. The aim of this study is to establish the accurate beam-quality estimation method mainly for fast neutrons by using two phantoms made of different materials [1]. In 2025, verification experiments for the dual phantom technique were continued using Heavy Water Neutron Irradiation Facility installed in Kyoto University Reactor (KUR-HWNIF) [2].

MATERIALS AND METHODS: One of the dual solid phantoms was made of polyethylene with natural lithium fluoride for 30 weight percent (LiF-polyethylene phantom), and the other phantom was made of polyethylene with 95%-enriched lithium-6 fluoride for 30 weight percent (⁶LiF-polyethylene phantom). Cells were divided in two groups. One was treated group using p-boronophenylalanine (BPA, containing B-10), and the other was non-treated group. In addition to the conventional cell suspension, the cell in collagen gel was also prepared. These cell samples were placed at the depths of 0 cm, 2 cm, 5 cm and 8 cm in the phantoms. Figure 1 shows one of the experimental geometries. The depth dose distributions for the thermal neutron, fast neutron and gamma-ray components were determined based on the simulation calculation results normalized referring to the measured values. The epi-thermal neutron irradiation mode was used for the phantom experiments.



Fig. 1. One of the experimental geometries.

RESULTS: It was confirmed that the survival fraction became smaller as the thermal neutron flux became larger in the LiF-polyethylene phantom. The survival fraction for the non-treated cell was higher than that in BPA-treated cell. In the ⁶LiF-polyethylene phantom, the survival fraction showed no difference between non-treated and BPA-treated cells. These confirm the results from the previous years, with the same trends observed for the cells in suspension and gel. Incidentally, the differences were observed in the slopes of the dose-survival curves between these cell sample types.

ACKNOWLEDGMENT: This work was supported by JSPS KAKENHI Grant Number JP 24K03082.

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Development of a BNCT Evaluation System Using a Three-Dimensional Model

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INTRODUCTION: Accurate assessment of neutron-induced biological effects is essential for optimizing boron neutron capture therapy (BNCT). Conventional two-dimensional (2D) culture systems fail to reproduce the structural and microenvironmental complexity of living tissues, including cell–cell and cell–extracellular matrix interactions. Recent advances in three-dimensional (3D) models have demonstrated that radiation responses are strongly influenced by tissue architecture, suggesting limitations in 2D-based evaluations [1].

This study aims to develop a BNCT evaluation system using physiologically relevant 3D culture models by comparing neutron-induced responses between 2D and 3D systems, thereby improving the accuracy of radiosensitivity and RBE assessment.

EXPERIMENTS: Human cancer cell lines, endothelial cells, and patient-derived fibroblasts were cultured in both 2D and 3D collagen-based systems. The samples were exposed to neutron irradiation at the Kyoto University Research Reactor (KUR). Dosimetry was verified using gold foil and thermoluminescent detectors. Following irradiation, cells were incubated for 72 hours. Cell viability was assessed by trypan blue exclusion following enzymatic dissociation (trypsin for 2D, collagenase for 3D), with automated cell counting used to quantify total and viable cells. In 3D models, histological analysis and image-based quantification were performed. Statistical analysis evaluated the effects of dose, cell type, and culture dimensionality.

RESULTS: Neutron irradiation induced a dose-dependent decrease in cell viability in both 2D and 3D cultures, however, the magnitude of this effect differed significantly between the two systems. In 2D cultures, survival decreased markedly with increasing doses across all cell types, with endothelial and osteosarcoma cells exhibiting particularly high radiosensitivity. In contrast, 3D models showed an attenuated response, characterized by increased radioresistance, especially in stromal cells, and reduced variability among cell lines. Direct comparison between 2D and 3D cultures revealed consistently higher survival in 3D systems across all dose levels (0–25.8 Gy), underscoring the critical influence of tissue architecture on neutron response. Notably, patient-derived cells such as normal fibroblasts and cancer-associated fibroblasts demonstrated stable resistance irrespective of culture conditions. Histological analysis of the 3D models revealed dose-dependent matrix contraction and reduced tissue thickness, accompanied by a gradual decline in cell number. Methodological validation showed strong agreement between histology-based analysis and live-cell counting, confirming the robustness of 3D quantification.

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Verification of BNCT Effect on Hematological Cancer Cells and Dosimetry for Expansion of BNCT Case

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INTRODUCTION: The number of newly diagnosed patients with major hematologic cancers (malignant lymphoma, multiple myeloma, and leukemia) in Japan was 58,547 in 2019 [1], and 5,853 hematopoietic stem cell transplants were performed in FY2022 as the last treatment option for these refractory hematologic cancer patients [2]. While autologous transplantation is a desirable medical technique for patients because it does not require long-term administration of immunosuppressive drugs and prevents immune reactions (GVHD) caused by HLA incompatibility, the grafts contain tumor cells, which must be removed and treated with chemotherapy and radiotherapy, which are pre-treatment procedures. We are now conducting basic research to remove tumor cells in grafts by Ex Vivo BNCT. In this study, the response of PVA-GTA-I gel dosimeter to thermal neutrons, fast neutrons, and gamma-ray was evaluated to use the gel dosimeters for dose assessment in cell bags.

EXPERIMENTS: PVA-GTA-I gel is prepared by adding PVA as the main component, KI as the reactant, Fructose as the reducing agent, GTA as the gelling agent, and GDL as the gelation promoter by adding GTA. In this study, 100 mM of B-(OH)₃ was added to PVA-GTA-I gel to make it sensitive to thermal neutron beams. The prepared gels were sealed in a 2D-case with a thickness of 4 mm. A sample was prepared consisting of 10 stacked 2D-cases. The sample was placed inside a water phantom, and half of the sample was covered with a ⁶LiF Teflon plate which absorbs thermal neutrons. The ⁶LiF Teflon plate enabled a difference in the ¹⁰B(n, α)⁷Li reaction rate, allowing for a comparison of the thermal neutron response. Neutron irradiation was performed using the KUR heavy water irradiation facility, and the irradiation time was 60 minutes. The color reaction of each 2D case was measured using scanner, and the absorbance was evaluated from the pixel values.

RESULTS: Fig. 1 shows a scam image of the 2D case after neutron irradiation. The sample on the side not shielded by the ⁶LiF Teflon plate exhibited stronger color reaction. In other words, it was demonstrated that the PVA-GTA-I gel to which B-(OH)₃ had been added exhibits high sensitivity to thermal neutrons.

The total dose was defined as the sum of the boron dose, hydrogen dose and gamma dose, and was calculated using the following method. The response to the boron dose was determined using the difference in sensitivity resulting from the presence or absence of the ⁶LiF Teflon plate. Furthermore, the response to gamma dose was calculated by combining gamma-ray measurements using BeO-TL, simulation results and the results of gamma-ray calibration experiment using ⁶⁰Co source. Fig. 2 shows the results of the evaluation of the response of each dose component.



Fig. 1 Response of the irradiated sample (left: overall view, right: first layer)

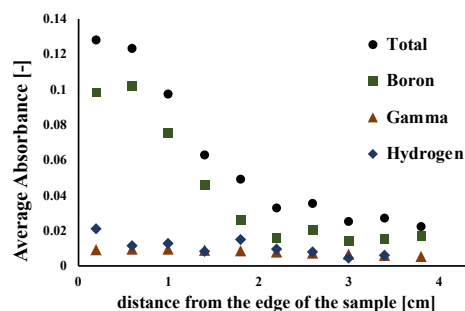


Fig. 2 Absorbance response distributions

Development and evaluation of 3D gel dosimeter for the measurement of dose distribution in BNCT

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INTRODUCTION: Three-dimensional (3D) gel dosimeters have been developed to facilitate the volumetric verification of complex conformal dose distributions in radiotherapy [1]. These dosimeters consist of radiosensitive chemical species dispersed within an aqueous gel matrix to preserve the spatial information of the absorbed dose. The 3D dose distribution is reconstructed from the amount of reaction products, typically measured via imaging modalities such as MRI or optical computed tomography (OCT). We previously reported a PVA-GTA-I radiochromic gel dosimeter that utilizes red color development resulting from the formation of a polyvinyl alcohol (PVA)-iodine complex [2, 3]. In a recent study, as a preliminary step toward 3D evaluation using optical CT, we assessed the pseudo-depth dose distribution of a reactor-based neutron beam by arranging small gel dosimeters containing ¹⁰B and ⁶Li in a block-like configuration [4]. In the present work, for comparison with these prior results, we fabricated similar small-scale dosimeters sensitized with gadolinium, which possesses a significantly larger thermal neutron absorption cross-section, and evaluated their performance.

EXPERIMENTS: The PVA-GTA-I gel dosimeter comprises partially saponified PVA, potassium iodide (KI), glutaraldehyde, fructose, glucono- δ -lactone, and distilled water. To enhance thermal neutron sensitivity, gadolinium chloride (GdCl₃, 50 mM), containing natural abundances of ¹⁵⁷Gd (15.7%), was added to the gel formulation. The resulting solution was dispensed into PMMA cuvettes (4.5 mL, 1 cm path length). Neutron irradiations were conducted at the Heavy Water Neutron Irradiation Facility (HWNIF) of the Kyoto University Research Reactor (KURR, 1 MW) in air at room temperature. Three distinct beam modes (thermal neutron-rich, fast neutron-rich, and mixed) were generated using a heavy water spectrum shifter and cadmium thermal-neutron filters.

RESULTS: Figure 1 illustrates the results obtained in the mixed mode as a representative case, where position numbers correspond to depth. In all experimental sets, the color intensity attenuated from the surface (left) to the deeper positions (right). Samples containing gadolinium exhibited localized increases in coloration, a trend qualitatively similar to that observed with ¹⁰B and ⁶Li. However, the change in absorbance (proportional to the absorbed dose) for the gadolinium-doped samples was comparable to those of ¹⁰B and ⁶Li, despite the vastly large thermal neutron capture cross-section of ¹⁵⁷Gd (240,000 barns) relative to ¹⁰B (3,850 barns) and ⁶Li (870 barns). This discrepancy suggests that gadolinium ions significantly inhibit the oxidation of iodide ions. Consequently, these results indicate that gadolinium is unsuitable as a thermal neutron sensitizer for PVA-iodine-based gel dosimeters.

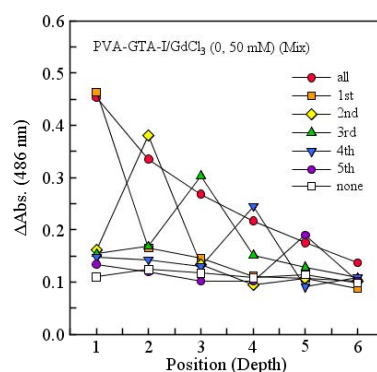


Fig. 1. The change of absorbances in depth (corresponding to relative depth dose distribution) in mixed mode, and the irradiated PVA-GTA-I radiochromic gel dosimeters.

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Establishment of Three-dimensional Dose Distribution Estimation Method for BNCT using Radiochromic Gel Dosimeter (III)

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INTRODUCTION: Development in accelerator-based irradiation systems for BNCT is underway. BNCT using newly developed accelerator systems is being implemented at multiple facilities around the world. However, there are still issues that need to be improved for further advancement of BNCT. The advancement of dose estimation is one of these, and the estimation of biological dose is particularly important. As part of the advancement of biological dose estimation for BNCT, we focus on chemical effects, which are the precursor to biological effects. We are studying on the estimation method using chemical dosimeters, especially gel dosimeters. Gel dosimeters are made of materials similar to living tissue and can be formed into any shape. Using a phantom made from gel dosimeter that models human body, three-dimensional dose distribution estimation becomes possible. Among the various types of gel dosimeters, we are especially focusing on “radiochromic gel dosimeters” that can be read out using optical CT, etc.. The purpose of this study is to establish three-dimensional dose distribution estimation method for BNCT using multiple types of radiochromic gel dosimeters. In 2025, the characterization for micellar gel dosimeters was continued [1].

MATERIALS AND METHODS: Leucocrystal violet (LCV) was selected as dye of micelle gel dosimeters, because it is extremely clear before irradiation, based on our previous study [2]. The micelle gel dosimeters were prepared based on 1 mM of LCV in the 96% water and 4% gelatin according to the method of Babic et al. [3]. For dose rate independency and temporal coloration reduction, the type and concentration of surfactant and sensitizer were changed. In addition, boric acid or urea was added for the estimation of boron or nitrogen dose components, referring to Gambarini et al. [4]. Furthermore, a dosimeter in which heavy water was replaced with light water was also prepared to estimate hydrogen dose component. The amount of additives was based on our previous study. The characterization experiments were mainly performed using Heavy Water Neutron Irradiation Facility of Kyoto University Reactor (KUR-HWNIF) [5].



Fig. 1. An example of color change before and after the irradiation using the LCV micelle gel dosimeters.

RESULTS: From the experiment results, it was confirmed that the dose response of each gel dosimeter depended on the linear energy transfers (LETs) of the particles produced in each dosimeter. Using these results, the factors for dose-component discrimination method were determined and verified for different energy spectra, and the validity was confirmed.

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Evaluation of Boron Dose and Dosimetric Performance of NC-RFG for BNCT

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INTRODUCTION: To establish a dose evaluation method for boron neutron capture therapy (BNCT), we investigated the applicability of a novel nanoclay-based radiofluorescent gel dosimeter (NC-RFG) with LET-independent characteristics (LET: linear energy transfer) using the Heavy Water Neutron Irradiation Facility (HWNIF) at the Kyoto University Research Reactor (KUR).

EXPERIMENTS: Experiments were conducted to evaluate the sensitivity change due to boron addition (using the Irradiation Rail Device on December 10) and to investigate the applicability to two-dimensional dose measurement inside acrylic phantom (using the Large Sample Carrier on February 4). NaBH₄ was added to the NC-RFG, and the ¹⁰B concentration was varied from 0 to 10 or 22 ppm. After irradiation in the mixed neutron irradiation mode at KUR-HWNIF, sensitivity was evaluated using a fluorescence spectrophotometer (RF-6000, Shimadzu), and two-dimensional distributions were analyzed using a fluorescence image scanner (GELSCAN3, iMeasure).

RESULTS: Sensitivity increased linearly with boron concentration from 0 to 10 ppm (Fig. 1), while no increase was observed under X-ray irradiation, indicating that the response originates from ¹⁰B(n,α)⁷Li nuclear reaction. Using an X-ray calibration curve obtained at RIKEN, the boron dose coefficient was estimated to be $7.56 \times 10^{-14} \text{ Gy} \cdot \text{cm}^2 \cdot \text{n}^{-1} \cdot \text{ppm}^{-1}$, which agrees well with the theoretical value of 7.43×10^{-14} (Ref. 2). This demonstrates, for the first time, direct experimental quantification of boron dose and its validity. The depth-dose distribution measured with NC-RFG showed a similar attenuation trend to that obtained by gold wire measurements (Fig. 2). The NC-RFG (22 ppm ¹⁰B) data were corrected using the aerated data of the same sample, assuming that the response under aerated conditions represents the gamma-ray component in deoxygenated conditions, based on LET-dependent response differences due to dissolved oxygen. Although the correction is complex, NC-RFG reflects thermal neutron distribution and is effective for continuous evaluation of mixed radiation fields. Since this study demonstrated, for the first time, the feasibility of experimentally evaluating the boron dose coefficient, future work will focus on a more detailed assessment and extension to nitrogen dose coefficient evaluation. Furthermore, more advanced methods for separating mixed radiation components will be investigated to enhance the overall accuracy of dose evaluation.

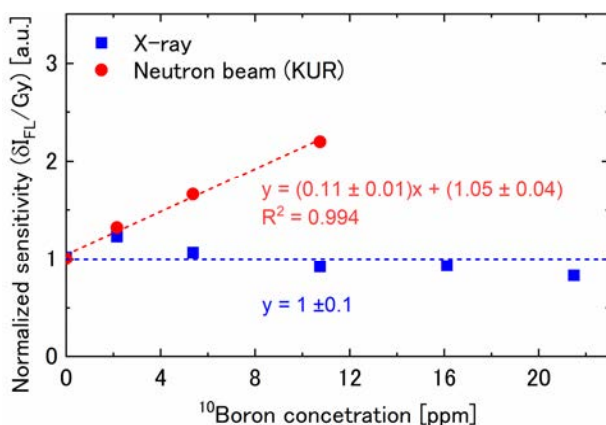


Fig. 1 Normalized sensitivity of the NC-RFG, using the value at 0 ppm boron concentration as the reference.

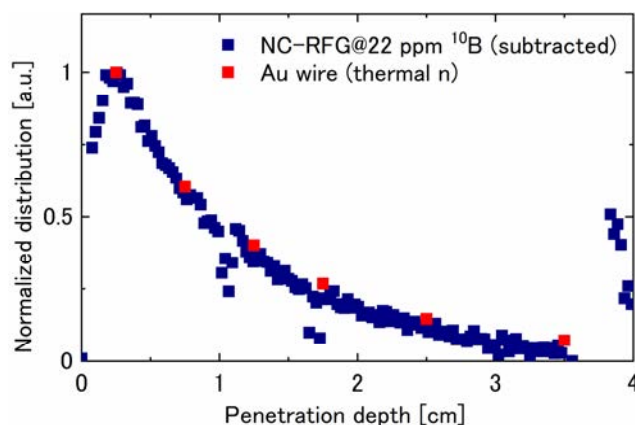


Fig. 2 Depth profiles of NC-RFG and Au wire.

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Development of an epithermal neutron spectrometer for boron neutron capture therapy

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INTRODUCTION: The neutron spectrum is essential for accurate dose evaluation in boron neutron capture therapy (BNCT), where the epithermal neutron component is dominant. Multi-sphere neutron spectrometers are widely used to measure the epithermal neutron spectrum. The spectrum depends on initial spectrum because of its flat energy response ranging from 1 eV to 10 keV. To improve reliability of the results, we developed an epithermal spectrometer with a threshold-like energy response. The performance was validated at Kyoto University Institute for Integrated Radiation and Nuclear Science (KURNS).

EXPERIMENTS: The spectrometer is composed of a 0.1-mm thick copper plate sandwiched between 5-mm thick boron carbide (B_4C) neutron absorbers and acrylic plates. The B_4C neutron absorber is a mixture of 80% B_4C and 20% acrylic resin by weight. The neutron energy spectrum is measured via the copper plate activity produced by the neutrons passing through the acrylic and B_4C layers. The components of the spectrometer were stacked at the beam exit window. The copper plate was placed perpendicular to the surface of the beam exit window. The spectrometer with a 300×300 mm surface covers the 120-mm diameter aperture. All neutrons are incident on the spectrometer from the reactor through the collimator. Following a 50-minute irradiation of the neutron beam, the copper plate was exposed to an imaging plate after a 10-minute cooling time to measure the activity depth profile, which depends on the incident neutron energy. The response matrix was derived from the depth profile simulated by Particle and Heavy Ion Transport code System ver.3.34 for 16 incident neutron energy groups from 0.01 eV to 16 MeV. Using the measured depth profile and the response matrix, the neutron energy spectrum of the beam was derived with an iterative unfolding algorithm. As an initial guess spectrum, the multi-foil activation method was utilized for the unfolding procedure [1].

RESULTS: Figure 1 shows the neutron energy spectrum obtained using our spectrometer, compared with the KURNS reference spectrum. The experimental result reproduced the reference spectrum below 100 eV, while it shows an underestimation above 100 eV. This underestimation may be attributed to neutron detection at the different resonance peaks of the two natural isotopes of copper. The radioactive nuclides with different half-lives were produced on the copper plate. The time-dependence of the activity should be considered in the response matrix. Now we are improving the response matrix by accounting for the elapsed time.

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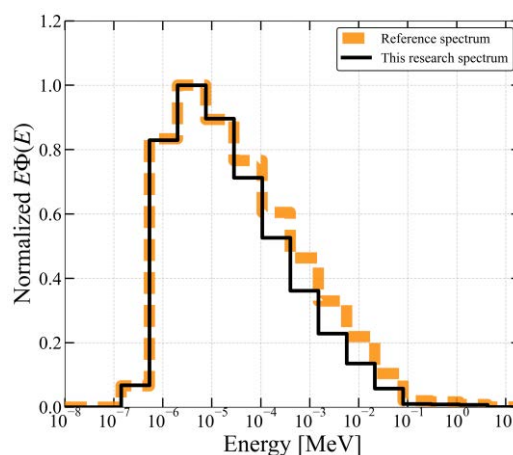


Fig. 1. Neutron energy spectrum obtained using the epithermal neutron spectrometer (black solid line), compared with the KURNS reference spectrum (orange dashed line).

Measurement of wavelength-dependent luminescence of BaFBr:Eu for beam quality assurance of BNCT

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INTRODUCTION: Beam fluence spatial distribution is required to be measured for quality assurance in the irradiation field, in boron neutron capture therapy. The imaging plate has been tested for usage in this purpose[1]. In imaging plate, the light for excitation and luminescence are investigated with respect to a single constant range of wavelength. However, BaFBr:Eu, *i.e.*, the luminescence material for imaging plate, has multiple peaks in its excitation spectrum[2]. Each peak corresponds to a excited state of electron and ion BaFBr:Eu. The efficacy in excitation to each level is expected to depend on the beam quality of the irradiated radiation, which deposits energy on the luminescence material. In this case, the dependence of the quantity of the luminescence on the wavelength of the excitation light, or possibly luminescence, will specify the beam quality. Also, the absorbed dose can be estimated from the quantity of the luminescence. This study investigated the use of the luminescence for measurement of both quantity and quality of the radiation.

EXPERIMENTS: In this study, BaFBr:Eu was fabricated and utilized as a sample, in case the blue film distort the influence of the beam quality of the dependence of the luminescence quantity of wavelength. This is based on the background that BaFBr:Eu in the imaging plate (Fujifilm Corporation, BAS-TR) is embed in blue-colored film. The samples were irradiated with gamma rays from ⁶⁰Co, and neutrons and gamma rays at the standard mixed neutron irradiation mode and standard thermal neutron irradiation mode of Kyoto University Reactor Heavy Water Neutron Irradiation Facility at 1 MW of the power [3]. In the latter, the samples were driven with Irradiation Rail Device. The fluorescence spectrofluorometer (Hitachi corporation, F-2700) was used to measure the dependence of the fluorescence quantity on the wavelength.

RESULTS: An example of the spectrum is shown in Fig. 1 for fabricated BaFBr:Eu. The ratio of the luminescence quantity by excitation at 630 nm to that at 650 nm is 1.13 for mix mode and 1.26 for thermal mode, 1.52 for Co. These aspects suggest that the dependence of the quantity of the luminescence on the wavelength is a promising indicator of the beam quality. The attempt to measure the beam quantity by using the luminescence dependence on wavelength is undergoing for usage of BAS-TR and fabricated BaFBr:Eu.

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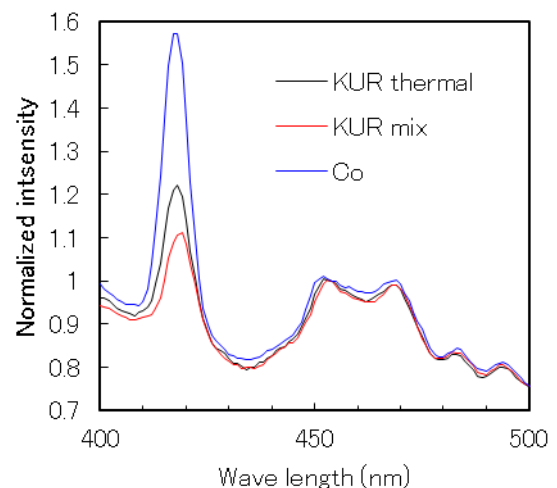


Fig. 1. Luminescence spectrum of fabricated BaFBr:Eu

Experiments for boron dose measurement with glass dosimeter in BNCT

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INTRODUCTION: The resulting reaction produces alpha particles and ${}^7\text{Li}$, which deposit energy within a range of approximately one cell diameter. This makes BNCT particularly promising for treating deep-seated tumors as a low-invasive cancer therapy, which are otherwise difficult to manage with conventional therapy [1]. Accurate evaluation of the individual dose is essential for the safe and effective planning of BNCT treatment. The total dose delivered to tissue consists of contributions from four components: the boron dose from the ${}^{10}\text{B}(\text{n},\alpha){}^7\text{Li}$ reaction, the nitrogen dose, the hydrogen dose and the gamma dose. Of these, the boron dose is the main therapeutic component, yet it is challenging to measure accurately in clinical conditions. In this study, glass dosimeters were employed in combination with a ${}^6\text{Li}$ -based sensitivity approach to evaluate the boron dose component in a phantom. This dosimetry approach is expected to provide a practical accessible method of evaluating individual boron doses, which will ultimately contribute to optimizing patient-specific irradiation planning for deep-seated treatment in BNCT.

EXPERIMENTS: Glass dosimeters were encased in holders that were fabricated using a 3D printer. Two samples were prepared for each type; Dosimeters coated with about 2.0 g of ${}^6\text{Li}_2\text{CO}_3$ powder, and uncoated dosimeters. The prepared samples were irradiated within a phantom under two distinct conditions, thermal neutron and epithermal neutron. In a separate experiment, boric acid was homogeneously dissolved in the phantom water (1% ${}^{10}\text{B}$ concentration) to simulate a clinically relevant boron distribution and measured dose distribution between pure water and boric water.

RESULTS: As shown in Fig.1, A sufficient dose difference was observed between the ${}^6\text{Li}_2\text{CO}_3$ powder-coated dosimeters and the uncoated control dosimeters when exposed to thermal and epithermal neutrons. This demonstrates that the differential dosimetry approach using ${}^6\text{Li}_2\text{CO}_3$ powder can detect the thermal neutron-induced dose component with adequate sensitivity under these conditions. In contrast, no significant dose difference was detected when boric acid was dissolved homogeneously in the phantom water. This suggests that the method is less sensitive when boron is distributed in a dilute aqueous medium, probably because the local concentration of the neutron-reactive material near the dosimeter is insufficient.

Further investigation should be consider to establish the practical applicability of this approach and determine the minimum detectable concentration of ${}^6\text{Li}$ or ${}^{10}\text{B}$. In addition, we are also considering to develop the conversion factor that combines experimental measurements with Monte Carlo simulations. Flux-to-dose conversion factors will enable the quantitative evaluation of boron doses in future experiments.

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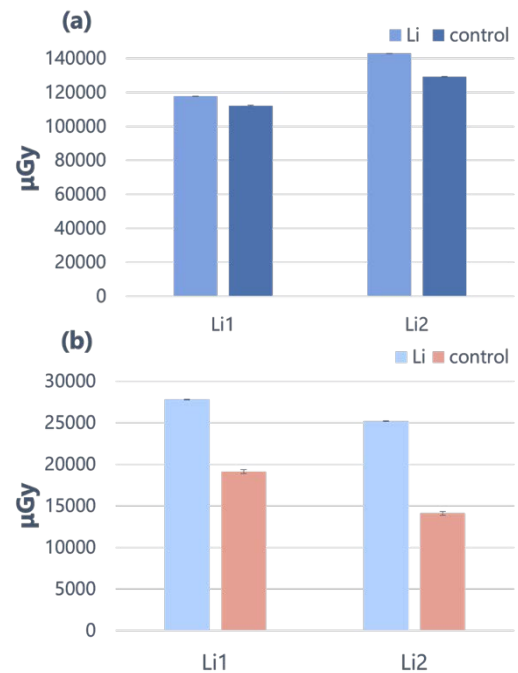


Fig.1. Dose results for glass dosimeters containing ${}^6\text{Li}_2\text{CO}_3$ powder and the control group, irradiated by (a) thermal neutrons and (b) epithermal neutrons.

I-1. PROJECT RESEARCHES

Project 3

Advancement of radiation detectors aimed at application in accelerator BNCT

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BACKGROUNDS AND PURPOSE: With the initiation of insurance-covered treatments using accelerator-based BNCT systems at medical institutions, the number of BNCT cases is expected to increase significantly. As part of the quality assurance (QA) and quality control (QC) processes for clinical treatments, it is necessary to measure the thermal neutron flux and gamma-ray dose prior to irradiation. Currently, conventional methods developed for reactor-based BNCT, such as the gold foil activation technique and thermoluminescent dosimeters (TLDs), are still in use. However, these methods are complex, and the continued availability of TLDs is uncertain. Consequently, there is a strong demand from medical institutions for simplified, yet highly accurate, measurement techniques.

Furthermore, there is an increasing need for the development of advanced radiation detectors applicable to BNCT, including those capable of measuring epithermal and fast neutron fluxes, prompt gamma emissions, and neutron energy spectra. In response to these demands, this project aims to enhance radiation detector technologies for practical application in accelerator-based BNCT systems.

RESEARCH SUBJECTS:

R7P3-1: Study on the Measurement of Neutron Fluence and Gamma-ray Distributions Using a Combination of Thermoluminescent Plate and Converter (K. Shinsho et al.)

R7P3-2: Improvement To Increase Accuracy of Absolute Fast Neutron Flux Intensity Monitor for BNCT (I. Murata et al.)

R7P3-4: Study of Optically Stimulated Luminescent Dosimeter in BNCT Irradiation Field (N. Matsubayashi et al.)

R7P3-5: Development of Scintillator for Thermal Neutron Detector in BNCT (N. Matsubayashi et al.)

R7P3-6: Development and optimization of a Bonner sphere spectrometer for intense neutrons in BNCT (A. Masuda et al.)

R7P3-8: Establishment of Characterization Estimation Method in BNCT Irradiation Field using Bonner Sphere and Ionization Chamber (IX) (Y. Sakurai et al.)

R7P3-9: Development of Real-time Boron-concentration Estimation Method using Gamma-ray Telescope System for BNCT (IV) (Y. Sakurai et al.)

R7P3-11: Basic research on methods for evaluating the radiation quality of neutron irradiation field (N. Hu et al.)

R7P3-12: Development of a new SOF detector probe balancing radiation resistance and flexibility (M. Ishikawa et al.)

R7P3-13: Neutron energy spectrum evaluation based on 2D thermal neutron flux distribution measurement with SOF detector (M. Ishikawa et al.)

R7P3-14: Evaluation of response characteristics of semiconductor detectors in neutron irradiation field (N. Hu et al.)

R7P3-15: Characteristics of Etch Pit Shapes Induced by Recoil Protons on Solid-State Nuclear Track Detectors in Epithermal Neutron Irradiation Field for BNCT (T. Takata et al.)

R7P3-16: 4H-SiC Neutron Image Sensor for Boron Neutron Capture Therapy (V. T. Ha et al.)

R7P3-17: Study on the Optimal Size of a LiCAF Scintillator for a Neutron Ambient Dose Detector in BNCT (L. Zhao et al.)

R7P3-18: Measurement of Thermal Neutron-Induced Soft Error Rates in Semiconductor Memories (H. Tanaka et al.)

R7P3-19: Development of a Real-Time Monitoring System to Enhance Treatment Reliability in Boron Neutron Capture Therapy and Application to Nuclear Decommissioning (S. Kurosawa et al.)

Research subjects R7P3-1-5,7, 11,14,15,16 are research and development on methods to measure thermal and fast neutrons and γ -rays. R7P3-9, 19 propose methods to obtain boron distributions in real-time. R7P6, 8, 13 propose new methods for measuring neutron spectra. R7P3-12, and 17 have succeeded in obtaining real-time measurements of thermal neutrons in the irradiation field of BNCT. All of them are expected to be applied to accelerator BNCT.

Study on the Measurement of Neutron Fluence and Gamma-ray Distributions Using a Combination of Thermoluminescent Plate and Converter

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INTRODUCTION: Boron Neutron Capture Therapy (BNCT) is one of the radiation therapies that uses neutrons and ^{10}B drugs, which accumulate in tumors. BNCT is expected to be a next-generation cancer therapy that can improve patients' quality of life (QOL), as it enables selective irradiation of cancer cells at the molecular level. However, dosimetry techniques in mixed neutron–gamma fields have not yet been established. Therefore, in this study, we focused on the measurement of neutrons and gamma rays using a two-dimensional thermoluminescence dosimeter (2D-TLD). We have previously reported that the thermoluminescence (TL) of a Cr-doped Al_2O_3 ceramic plate sandwiched between Cd plates can selectively measure the thermal neutron fluence in a BNCT irradiation field without being affected by mixed γ -rays [1,2], and that the TL characteristics of a high thermal conductivity type BeO ceramic plate (Na-undoped) can selectively measure the γ -ray fluence without being affected by neutrons [2,3,4]. In this study, we investigated a new method for selective fast neutron measurement using a novel TL plate combined with a converter based on the elastic scattering reaction between fast neutrons and hydrogen atoms.

EXPERIMENTS: A 3 mm thick high density polyethylene (HDPE) converter was placed. A high-density polyethylene (HDPE) plate with a thickness of 3 mm was used as a neutron converter to generate recoil protons via elastic scattering between fast neutrons and hydrogen atoms. BeO ceramic plates (Na-undoped) were placed on both the front and rear surfaces of the HDPE to detect the spatial distribution of deposited dose induced by recoil protons. Neutron transport and energy deposition were evaluated using a Monte Carlo simulation code. The absorbed dose in the front and rear BeO plates was calculated as a function of neutron energy. For comparison, the same configuration without the HDPE converter was also simulated to confirm the contribution of recoil protons.

RESULTS: As shown in Fig. X, the absorbed dose in the BeO plates exhibits a clear dependence on neutron energy. When the HDPE converter is introduced, a significant difference in absorbed dose between the front and rear BeO plates is observed, particularly in the fast neutron energy region (>0.1 MeV). This difference originates from recoil protons generated in the HDPE, which preferentially deposit energy toward the forward direction. In contrast, when the converter is not used, the absorbed dose in the front and rear BeO plates shows almost identical values over the entire energy range, indicating that the observed asymmetry is attributed to recoil protons. These results demonstrate that the proposed configuration enables selective detection of fast neutrons based on the difference in absorbed dose between the front and rear detectors.

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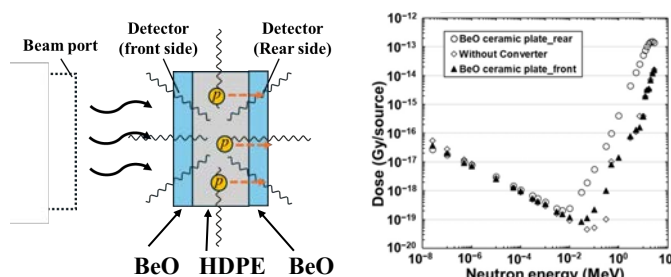


Fig. X. Schematic configuration of the proposed detector and calculated neutron energy response.

Improvement To Increase Accuracy of Absolute Fast Neutron Flux Intensity Monitor for BNCT

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INTRODUCTION: BNCT is a promising cancer therapy which kills only tumor cells selectively. The neutron field of neutron sources for BNCT includes not only thermal and epi-thermal neutrons but also fast neutrons that are harmful to the human body. Therefore, we have to measure the absolute integral flux intensity of fast neutrons (10 keV ~ 1 MeV) to evaluate their exposure dose. Now we are developing a monitor to precisely measure it and repeatedly improving the monitor [1]. In the previous research, the experimental value was overestimated by about 226 % compared to the calculated value [2], which is attributed in part to the non-constant sensitivity of the monitor. Therefore, this work aims to modify the shielding structure to achieve constant sensitivity for more accurate measurements.

EXPERIMENTS: The monitor consists of a 6.2 cm cubic polyethylene (PE) moderator with a 1.0 cm thick ¹⁰B shield on the irradiation surface and a Cd-covered GaN foil at the center. To achieve constant sensitivity to fast neutrons, we propose a design that places multiple types of absorbers in front of the ¹⁰B wall. In this study, preliminary experiments were conducted at KUR to verify the simulation accuracy and the linearity of combined absorbers. Four configurations were tested: (1) no absorber, (2) Fe, (3) Nb, and (4) a combination of Fe and Nb (half-area each). Irradiations were performed for 15 min in 5 MW mode for configurations (1), (2) and (3), and 90 min in 1 MW mode for configuration (4) operation, respectively.

RESULTS: Irradiations were successfully performed for all the four configurations, and activation of the GaN foils was observed. Currently, the experimental data are being processed to deduce the absolute fast neutron flux intensity. Specifically, we are calculating correction factors to normalize the experimental values for comparison with the MCNP5 simulation results. A preliminary analysis indicates that the relative change in flux intensity for each absorber (Fe and Nb) follows the predicted result. The linearity of the combined absorbers (Fe+Nb) is also being evaluated by comparing the superimposed result of individual absorber results with the actual measurements.

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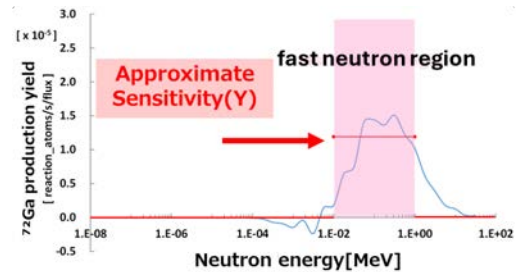


Fig. 1 Sensitivity of the fast neutron monitor in previous research.

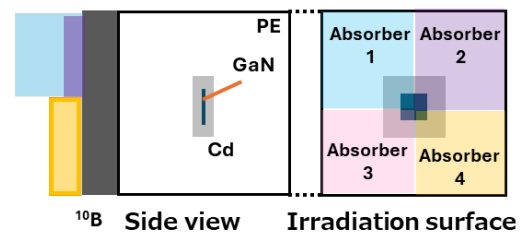


Fig. 2 Design of the monitor for sensitivity optimization featuring multi-type absorbers

Study of Optically Stimulated Luminescent Dosimeter in BNCT Irradiation Field

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INTRODUCTION: The BNCT irradiation field consists of neutrons with a wide energy range and undesired gamma-rays, which are mainly caused by nuclear reactions with surrounding structures. For quality control/quality assurance (QA/QC), the undesired gamma-ray dose in a phantom must be measured [1]. To evaluate gamma-ray dose in the BNCT irradiation field, a thermoluminescent dosimeter (TLD) composed of beryllium oxide powder enclosed in a quartz glass capsule, which is less sensitive to thermal neutrons, has been used. However, as the TLD was out of production, a new gamma-ray dosimeter is required for QA/QC of BNCT [2]. In this study, we selected optically stimulated luminescent dosimeter (OSLD), which is made of $\text{Al}_2\text{O}_3\text{:C}$. The OSLD has been used in other radiation therapies, has almost no fading effect and can be measured repeatedly. However, it is difficult to completely eliminate the thermal neutron sensitivity of OSLD. In this study, we investigated whether it is possible to measure the thermal neutron flux via the activation method using OSLD (^{27}Al (n, γ) ^{28}Al).

EXPERIMENTS: The irradiation tests of the BNCT irradiation field were performed at Heavy Water Neutron Irradiation Facility (HWNIF) at Kyoto University Research Reactor (KUR) [3]. A cadmium shutter, which shields only thermal neutrons, was installed to control the thermal neutron flux with each openness. To investigate the activation of the OSLD, radioactivity was measured immediately after the irradiation test using a high-purity Ge detector. Neutron reaction rate of the OSLD was evaluated by the count rates, the detection efficiency of the Ge detector, and the average cross section. We compared it with thermal neutron flux measured by the gold activation method. The irradiation tests were carried out by changing the openness to 0, 100, 200, 300, and 600 mm.

RESULTS: Fig. 1 shows the ratio of the reaction rate to the thermal neutron flux. The results of the linear fitting shown in Fig. 2 indicate $R^2 = 0.98$, confirming that the relationship is approximately linear. In conclusion, the OSLD was capable of measuring thermal neutron response via the activation method, and it was suggested that it could self-correct its sensitivity.

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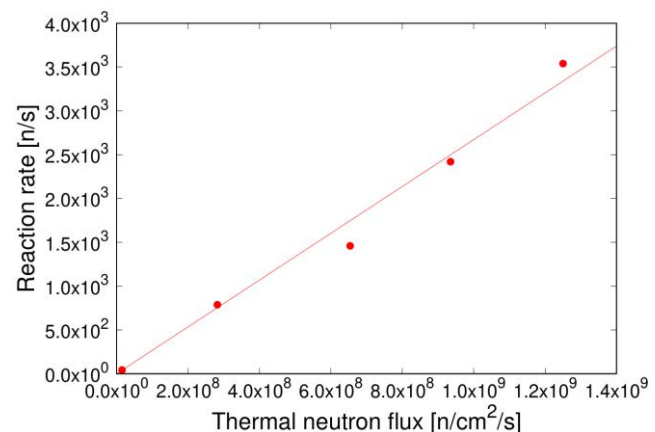


Fig. 1. Relationship between thermal neutron flux and ^{27}Al neutron reaction rate.

Development of Scintillator for Thermal Neutron Detector in BNCT

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INTRODUCTION: In recent years, accelerator-based BNCTs have been developed worldwide, and clinical cases are increased. The activation method has been used for measurement of thermal neutron flux but cannot measure in real-time. The development of thermal neutron detectors as neutron monitors is needed. The neutron monitor using Eu:LiCaAlF₆ (LiCAF) scintillator with quartz fiber was developed [1]. It is necessary for the use of the scintillator as neutron monitor to determine the relationship between the count rates obtained by the detector and thermal neutron flux. Considering the increase in the clinical cases, we must calibrate the detector in primary national standard field. However, the neutron intensity of the standard field is much lower than that of the BNCT irradiation field. In this study, we applied pulse shape discrimination (PSD) to the neutron detection in BNCT irradiation field (Fig. 1). The neutron monitor uses a light guide to prevent the effects of the photodetector, but the waveform becomes less sharp, making it difficult to use the PSD. In this study, we investigated the length of the light guide that is unaffected by a photomultiplier tube (PMT).

EXPERIMENTS: The irradiation tests of each detector were performed at Kyoto University Research Reactor-Heavy Water Neutron Irradiation Facility (KUR-HWNIF) [2]. The Ce doped LiCAF scintillator, which has a short decay time, was directly attached to the PMT. Considering the use in BNCT, a 30 cm square polyethylene block was placed at the collimator, and measurements were taken with detectors positioned 0, 5, and 10 cm above the block surface (15, 20, and 25 cm from the beam center). We measured the pulse height distribution by multichannel analyzer to evaluate the effect of the PMT.

RESULTS: As shown in Fig. 2, at distances of 20 and 25 cm from the beam center, the neutron events can be distinguished in the high channel region. The count rates summarized from 1000 to 2000 ch were 59.6, 1123, and 963 cps at 15, 20, and 25 cm, respectively. Based on these results, it was confirmed that the light guide of at least 20 length is necessary to prevent the effect of the PMT.

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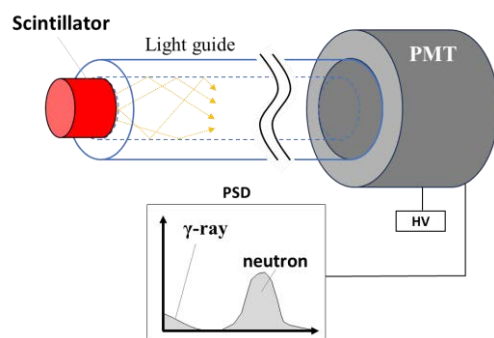


Fig. 1. Schematic layout of neutron detector

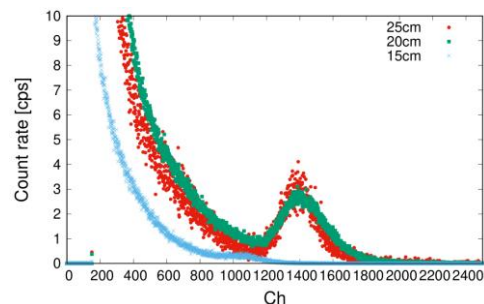


Fig. 2. Pulse height distribution at distances of 15, 20, and 25 cm from the beam center.

Development and optimization of a Bonner sphere spectrometer for intense neutrons in BNCT

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INTRODUCTION: Neutron spectral fluence measurement is required in boron neutron capture therapy (BNCT). A Bonner sphere spectrometer (BSS) has been developed that incorporates a small lithium-glass scintillator to enable the measurement of intense neutrons up to $10^9 \text{ cm}^{-2} \text{ s}^{-1}$ in this study. The BSS was modified to include variations in moderator thickness that are optimal for measurements focusing on epithermal neutrons, and demonstration measurements of BNCT epithermal neutrons were conducted in the Kyoto University Research Reactor (KUR).

EXPERIMENTS: The Bonner sphere detectors with the small lithium-glass scintillator coupled with an optical fiber and a photomultiplier (PMT, Hamamatsu R9880U-21) were set in the heavy water irradiation facility of the KUR [1]. Output signals from the PMT were processed using a preamplifier (ORTEC 113) and a signal processing and acquisition system (Amptek PX5). Measurements were performed sequentially with varying sizes of moderators,

RESULTS: Fig. 1 shows the dependence of the measured count rates of the Bonner sphere detector on the sphere diameter and compares it with the count rates predicted by Monte Carlo simulations. The two measurement series were performed completely independently in different years. The experimental results and calculated values were normalized by the result of the 3" detector of the 2nd measurement. The trends of the measurement results generally agreed with the simulation, but the count rates for detectors smaller than 2.5" were higher than the calculated results. This is likely due to the significant influence of the low-energy scattered neutrons in the irradiation room on the smaller detectors, and inaccuracies in the modeling of the detector structure in the simulations. They will be investigated mainly through further simulations, and correction factors should be introduced if necessary.

The BSS response matrix will be precisely determined through measurements at the reference neutron fields of the National Institute of Advanced Industrial Science and Technology (AIST) and Monte Carlo simulations. Using these results, the measurement results of the KUR experiment will be unfolded, and the neutron spectral fluence will be derived.

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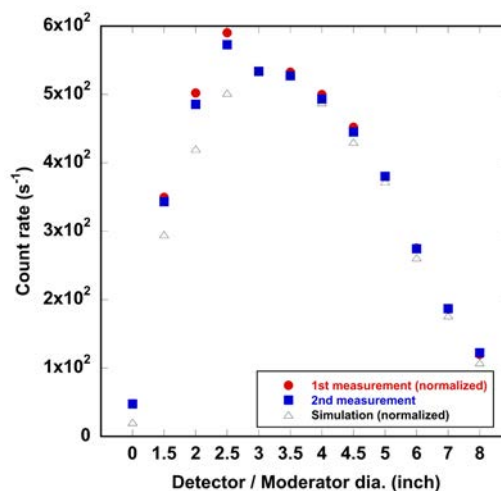


Fig. 1. Results of the Bonner sphere measurements.

Establishment of Characterization Estimation Method in BNCT Irradiation Field using Bonner Sphere and Ionization Chamber (IX)

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INTRODUCTION: Development in accelerator-based irradiation systems for BNCT is underway. BNCT using newly developed accelerator systems is being implemented at multiple facilities around the world. Considering this situation, it is important that the estimations for dose quantity and quality are performed consistently among several irradiation fields, and that the equivalency of BNCT is guaranteed, within and across BNCT systems. Then, we are establishing QA/QC system for BNCT. As part of the QA/QC system, we are developing estimation method for neutron energy spectrum using Bonner-sphere technique [1]. In our spectrometer, liquid such as pure water and/or boric acid solution is used as the moderator. A multi-layer case with multiple moderator layers is prepared. The moderator and its thickness are changeable without entering the irradiation room, by the remote supply and drainage of liquid moderator in the several layers. For the detector, activation foils are remotely changed, or online measurement is performed using SOF detector, etc. As a new type of spectrometer, we are developing the Cylindrical Hemisphere Accurate Remote Multilayer Spectrometer (CHARMS) [2]. The experimental verification for the effectiveness of a prototype of CHARMS was continued in 2025 as well [3].

MATERIALS AND METHODS: A LiCaAlF_6 scintillation neutron detector is positioned at the center of CHARMS, and it is surrounded by three layers of liquid moderators. With a remote operation of liquid moderator supply and drainage system from outside the irradiation room, we can realize a fully remote-operating neutron spectrometer. The performance of CHARMS in measuring the neutron energy spectrum was evaluated at Heavy Water Neutron Irradiation Facility of Kyoto University Reactor (KUR-HWNIF) [4].

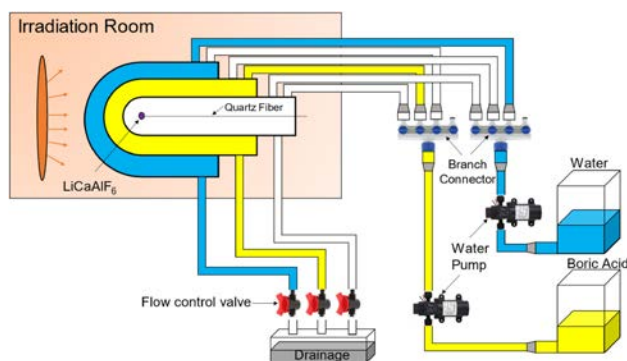


Fig. 1. Outline of a prototype CHARMS.

RESULTS: The neutron energy spectrum of the BNCT irradiation at KUR-HWNIF was measured using CHARMS without the need to enter the irradiation room. The evaluated neutron energy spectrum closely matched the results of simulation calculations. The entire measurement process took approximately one hour by using CHAMRS. It was confirmed that CHARMS can provide a reliable neutron energy spectrum with a short measurement time compared to conventional methods. It is considered that CHARMS is a promising neutron spectrometer for use in BNCT irradiation field in the future.

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Development of Real-time Boron-concentration Estimation Method using Gamma-ray Telescope System for BNCT (IV)

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INTRODUCTION: It is important to decide the boron concentrations for tumor and normal parts in the dose estimation for BNCT. To improve the dose estimation in BNCT, a method for estimating the spatial distribution of boron concentration online in real time is expected. The information about the boron concentration distribution can be obtained using the prompt gamma-ray analysis (PGA) for the prompt gamma rays from boron-10 (B-10). The improved gamma-ray telescope system is settled at Heavy Water Neutron Irradiation Facility of Kyoto University Reactor (KUR-HWNIF) [1-3]. This system consists of an HPGe semiconductor detector and a collimation system including two lead collimators. The gamma rays through these collimators can be detected, and the telescope view-field can be changed by moving the two collimators independently. The experimental verification for the ability to distinguish between tumor and normal parts was continued in 2025 as well [4].

MATERIALS AND METHODS: The phantom experiment was performed using the epi-thermal neutron irradiation mode at KUR-HWNIF. The phantom size was 20 cm × 20 cm × 20 cm. Within the phantom, a 5-cm diameter acrylic hollow sphere was placed as the tumor. Both the phantom and tumor sphere were filled with different concentrations of boric acid water. For example, the tumor spheres with the B-10 concentration of 70, 100 and 200 ppm were put into a phantom with B-10 concentration of 20 ppm. The irradiation field was set to 12 cm in diameter. The tumor sphere was fixed at the center of the telescope view-field. The 1st and 2nd telescope collimators were set at the bottom of the telescope.

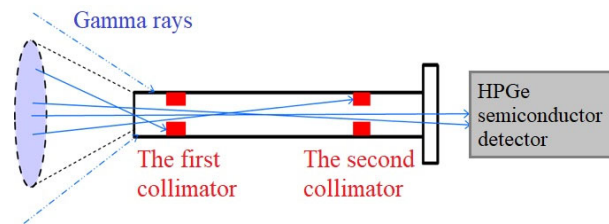


Fig. 1. Outline of the collimation system.

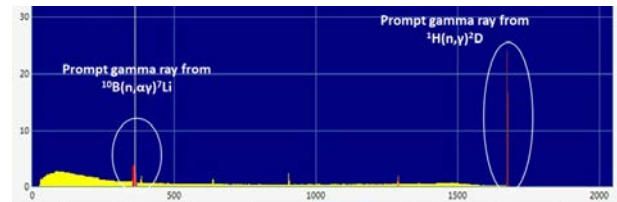


Fig. 2. An example of the gamma-ray energy spectrum by the telescope system.

RESULTS: The count rates for 478-keV prompt gamma rays with the different irradiation conditions were obtained from the experimental results. The experimental count rates were almost 30 cps (s^{-1}) for the 70-ppm tumor sphere, 33 cps (s^{-1}) for the 100-ppm tumor sphere, and 46 cps (s^{-1}) for the 200-ppm tumor sphere. Therefore, because the time required to reach 1000 counts was just over 30 seconds, the time course of B-10 concentration can be estimated almost in real-time. From the results of the experimental verification, the effectiveness and usefulness of the improved gamma-ray telescope system were confirmed.

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Basic research on methods for evaluating the radiation quality of neutron irradiation field

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INTRODUCTION: In a typical BNCT irradiation field, multiple types of radiation are present, making accurate measurement challenging. A proportional gas counter is commonly used to assess the radiation quality in mixed fields, such as neutron–gamma environments. To evaluate the biological effects of a neutron beam, a tissue-equivalent proportional counter (TEPC) is employed. In this method, the gas density is reduced to simulate a micrometer-scale tissue volume.

EXPERIMENTS: The experiment was conducted using the epithermal neutron irradiation mode at KUR operating at 1 MW. The TEPC was positioned free-in-air at the center of the irradiation field, and data were collected over a period of one hour. The acquired raw data were converted into a lineal energy spectrum (y-distribution) and compared with results obtained from Monte Carlo simulations using the Particle and Heavy Ion Transport code System (PHITS). Following validation of the PHITS lineal energy distribution, Monte Carlo simulation was performed for different neutron sources (KUR thermal mode, cyclotron-based epithermal neutron source (CBENS), and CICS-1 accelerator system) and the relative biological effectiveness (RBE) was calculated.

RESULTS: As shown in Fig. 1, the RBE values for the different neutron sources vary, which is consistent with previously published report [2]. This result indicates the PHITS Monte Carlo simulation may be used to estimate biological effectiveness of neutron sources.

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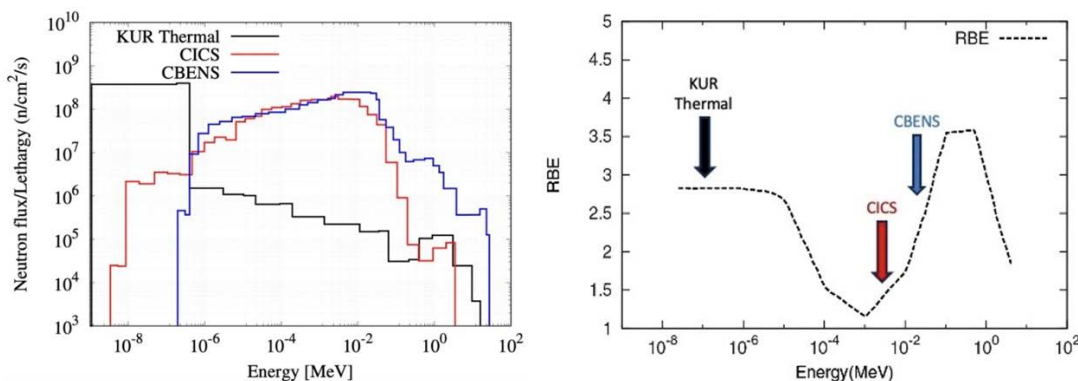


Fig. 1. Left) Neutron energy spectra per lethargy of KUR Thermal (black), CICS (red), and CBENS (blue). Right) RBE versus Neutron Energy with KUR, CBENS, and CICS spectra. The arrows indicate the peak neutron flux for each neutron spectra.

Development of a new SOF detector probe balancing radiation resistance and flexibility

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INTRODUCTION: Our laboratory has developed a Scintillator with Optical Fiber (SOF) detector, which features a plastic scintillator attached to the tip of an optical fiber, for the purpose of real-time thermal neutron fluence measurement during Boron Neutron Capture Therapy (BNCT) irradiation. Previous collaborative experiments confirmed the SOF detector's long-term stability and wide dynamic range, maintaining linearity from 10^4 to 10^{10} n/cm²/s, but also revealed that the primary cause of signal degradation was the deterioration of the plastic optical fiber. While quartz optical fibers showed almost no signal degradation in subsequent trials, their bending performance was significantly poor, leading to concerns regarding probe breakage during practical use. Consequently, this research aims to develop a new probe that incorporates both the flexibility of plastic optical fibers and the radiation resistance of quartz optical fibers.

EXPERIMENTS: In the 2025 fiscal year, degradation characteristic tests were performed using a high-intensity thermal neutron irradiation field at the Slant irradiation hole of the Kyoto University Research Reactor (KUR). The SOF detector methodology estimates pure thermal neutron flux by calculating the difference between signals from a probe containing a ⁶LiF neutron sensitizer mixed into the plastic scintillator and one without the sensitizer. For these experiments, Saint-Gobain BC620 was utilized as the reflector, and four distinct quartz fiber probe configurations were fabricated to evaluate the radiation hardness of individual components, including variations with and without the neutron sensitizer and the reflector coating. Irradiation was conducted at a reactor power of 5 MW for approximately five hours, with the measurement position set 181 cm from the lower end of the Slant hole to minimize counting losses. The results indicated a signal decrease of approximately 2% only in the probe containing ⁶LiF without a reflector coating, whereas the standard SOF probe configuration—including both ⁶LiF and a reflector—showed no significant signal degradation. In contrast, plastic fiber probes measured simultaneously experienced extremely high signal volumes and significant counting losses, which prevented the collection of effective data. The stability observed in the standard quartz probe configuration confirms its potential for high-dose clinical applications where plastic fibers typically fail. However, the estimated thermal neutron flux from the measurement position at 181 cm exceeded 1×10^{10} n/cm²/s, which is remarkably higher than the previously reported flux of 2×10^8 n/cm²/s at a position closer to the source (70 cm from the bottom). This discrepancy strongly suggests that the detector output at this location is likely dominated by non-neutron signals, potentially including high-energy gamma rays or Cerenkov radiation generated within the optical fiber itself. Future development will focus on isolating these background components to ensure that the increased radiation resistance of the fiber translates into more accurate neutron dosimetry in high-intensity irradiation environments.

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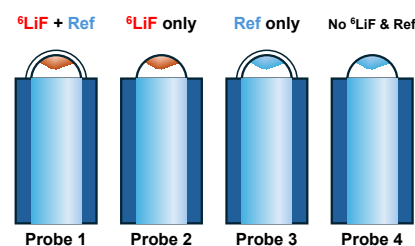


Fig.1 SOF detector probes categorized by individual components.

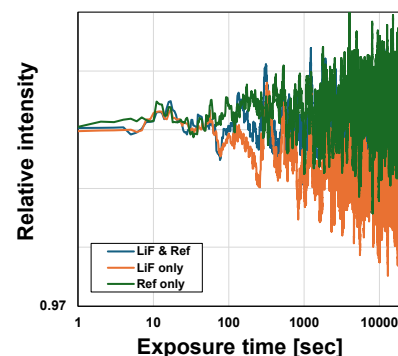


Fig.2 Accelerated degradation test of SOF detector components.

Neutron energy spectrum evaluation based on 2D thermal neutron flux distribution measurement with SOF detector

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INTRODUCTION: We have been developing a SOF (Scintillator with Optical Fiber Detector) detector—a plastic scintillator attached to the tip of an optical fiber—for real-time thermal neutron fluence monitoring during boron neutron capture therapy (BNCT) irradiation. Previous experiments at KUR have confirmed the wide dynamic range of the SOF detector (linearity from 10^4 to 10^{10} n/cm²/s)^[1]. In accelerator-based BNCT, neutrons are generated through nuclear reactions at the target; however, long-term operation may cause target degradation and potentially alter the generated neutron energy spectrum. Changes in beam diameter or irradiation position may also affect the spectrum. Therefore, as a quality assurance measure for the accelerator BNCT irradiation field, this study investigates a method for estimating the neutron energy spectrum at the beam port surface from two-dimensional (2D) neutron flux distribution data measured with the SOF detector in a water phantom.

EXPERIMENTS: Two-dimensional thermal neutron flux measurements in a water phantom were performed using the SOF detector in epithermal neutron mode (CO-0000-F) (Fig. 1). A 20 cm × 20 cm × 20 cm acrylic water phantom (PMMA wall thickness: 3 mm) was used. Measurements were carried out at 1 cm intervals up to a depth of 12 cm along the beam axis and within ±7 cm in the lateral direction. The reactor output was 1 MW, and the measurement time was 10 seconds per point, resulting in a total measurement time of approximately 50 minutes. Dedicated software for 2D scanning measurements was used, and the data were monitored in real time during irradiation. The measured 2D distribution was compared with Monte Carlo simulations performed using PHITS. The neutron energy spectrum of the beam was taken from that reported by Prateepkaew^[2]. Figure 2 shows that the 2D measurements obtained with the SOF detector are in relatively good agreement with the PHITS calculations. ML-EM^[3]-based neutron energy spectrum estimation was also performed using the measurement data, however, the estimated spectrum did not agree well with the reported spectrum^[4] especially at thermal-neutron energy region (Fig.3).

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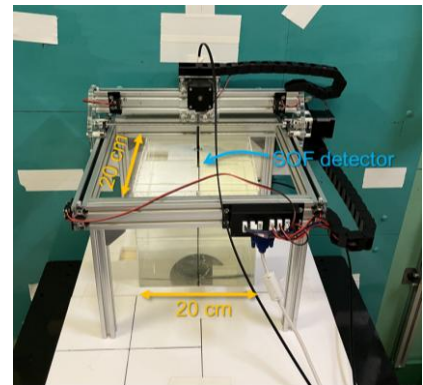


Fig.1 Geometry for 2D neutron flux distribution using scanning device.

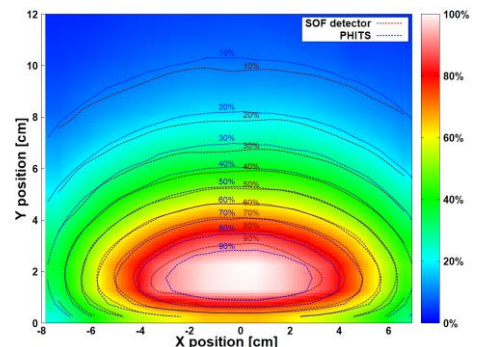


Fig.2 Comparison of measured and calculated relative distributions in the epithermal irradiation mode (CO-0000-F).

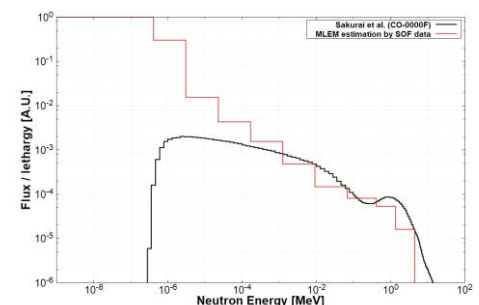


Fig.3 ML-EM-based neutron energy spectrum estimation using measurement data.

Evaluation of response characteristics of semiconductor detectors in neutron irradiation field

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INTRODUCTION: The Centre for Medical Radiation Physics, University of Wollongong, Australia, has developed a silicon microdosimeter to measure directly the microdosimetric spectrum. The detector has an array of micro-sized sensitive volumes, which can measure the energy deposition at the cellular-level. The use of this detector in a mixed neutron/gamma ray radiation field is of high interest, particularly for evaluating the dose deposited to a volume mimicking a single cell.

EXPERIMENTS: The measurements were performed using the heavy water irradiation facility (epithermal irradiation mode) of the Kyoto University Research Reactor under 1MW. The silicon microdosimeter fitted with an enriched boron-10 converter was placed inside a water phantom placed on the couch top (Fig 1.).

RESULTS: As shown in Fig. 2, the energy deposition distribution and the dose lineal energy distribution ($yd(y)$) was obtained and the results closely matched Monte Carlo simulation results. The results indicate the silicon microdosimeter are able to detect single-event alpha particles from the boron neutron capture reaction.

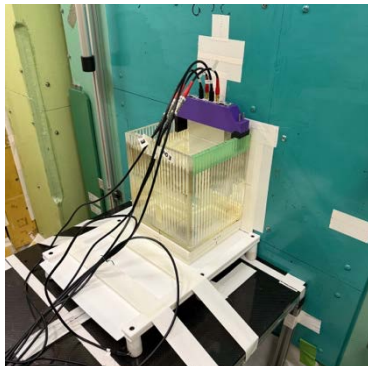


Fig. 1. Image of the experimental set up using the heavy water irradiation facility.

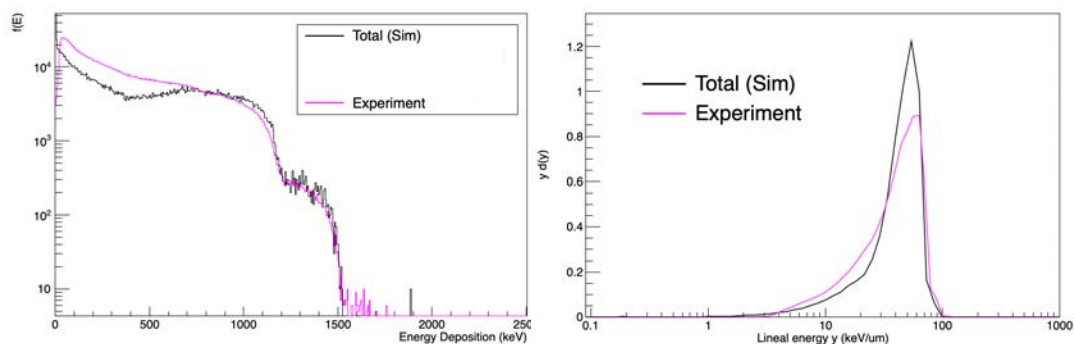


Fig. 2. Left) Energy deposition distribution, Right) dose lineal energy distribution ($yd(y)$), obtained using the silicon microdosimeter with the boron-10 converter.

Characteristics of Etch Pit Shapes Induced by Recoil Protons on Solid-State Nuclear Track Detectors in Epithermal Neutron Irradiation Field for BNCT

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INTRODUCTION: In the dosimetry of boron neutron capture therapy (BNCT), it is essential to separately measure doses from thermal neutrons, fast neutrons, and gamma rays. Within this mixed field of neutrons and gamma rays, the paired ionization chamber method is typically used for fast neutron dosimetry. This method inherently requires the compensation of gamma-ray doses, which introduces uncertainty in the calculated fast neutron dose. Additionally, the radiation field can be disturbed by the gas cavity, which must be considered. This study explores the use of a solid-state nuclear track detector (SSNTD) as an alternative method, as it is not sensitive to gamma rays and causes less field disturbance than ionization chambers. It is established that the shape of pits formed on the SSNTD through chemical etching is influenced by the LET of incoming particles [1]. We report an analysis of the pit shapes created by recoil protons in the epithermal neutron irradiation field for BNCT.

EXPERIMENTS: Commercially available SSNTDs (Baryotrack, Nagase Landauer, Ltd.) were irradiated at Heavy Water Neutron Irradiation Facility in KUR [2]. As a recoil proton converter, a polyester film with a thickness of 200 μm was attached on the detector surface. The detector was placed in free air at the collimator outlet and irradiated for 30 minutes during 1 MW operation. Post-irradiation, the detectors were etched with 6M NaOH solution for 7 hours at 70°C. The surface profiles of the etched detectors were measured by using a 3D confocal laser microscope (LEXT4000, Evident Corporation) with 100 $\times$ objective lens [3]. The measurement was performed with the support of the Kyoto University Nanotechnology Hub under “Advanced Research Infrastructure for Materials and Nanotechnology Project” sponsored by the Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan.

RESULTS: By analyzing the surface profile, parameters of the pit shapes were obtained. Figure 1 illustrates the distributions of the measured pit diameters and depths. Pits with a diameter of 2 μm and a depth of 0.4 μm were observed most frequently, with larger diameters and greater depths occurring less often. These features imply that the distribution of pit shapes reflects the incident proton energy spectrum. In future work, we will examine the feasibility of estimating the recoil proton spectrum by analyzing pit shape parameters.

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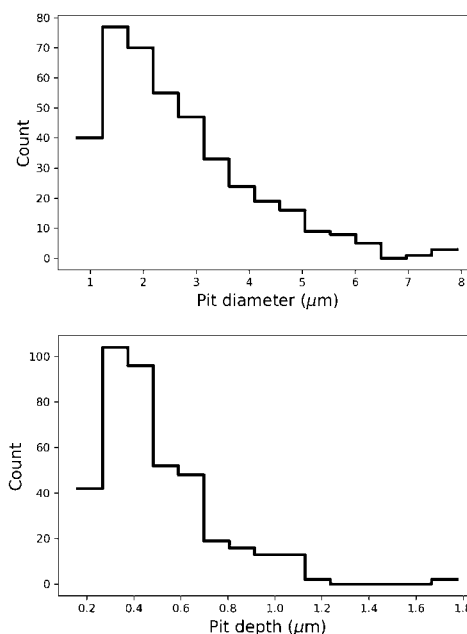


Fig. 1 Frequency distribution of the diameter and depth of etch pits.

4H-SiC Neutron Image Sensor for Boron Neutron Capture Therapy

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INTRODUCTION: Boron neutron capture therapy (BNCT) has been attracting attention as an advanced treatment method on cancer because this therapy has a great advantage on a minimally invasive and selective treatment. In the BNCT, to make this therapy more accurate one, it is better to measure the neutron beam's position and profile. On the other hand, silicon carbide (SiC) semiconductor image sensors have been developed as radiation hardened electronics [1,2], and we are suggesting a neutron CMOS image sensor by combining the SiC CMOS image sensor and neutron conversion layer for the real time imaging [3]. In this year, we fabricated the neutron CMOS image sensors and tested the device operations under neutron irradiation.

EXPERIMENTS: In this study, neutron sensors were designed based on 4H-SiC substrate with three transistor-active pixel sensors (3T-APS) structure which amplifies the signal in each pixel. A neutron conversion layer with ¹⁰B was formed inside the aluminum layer on the top of the sensor by ion implantation. By changing the thickness of aluminum layer under the neutron converter with ¹⁰B, it is possible to control the energy entering the detection area and thereby change the number of electron-hole pairs generated during the ionization process. On the other hand, a circuit simulation model of the neutron sensor was also developed.

RESULTS: The SiC neutron sensors for BNCT were designed and fabricated based on 3T-APS structure and investigated for their performance by using LT spice simulation. Simulation results show the neutron detection ability of SiC neutron sensors based on 3T-APS structure and a parasitic n-p-n structure in the detection area. These results laid the foundation for fabricating SiC neutron sensors for BNCT and testing thermal neutron irradiation.

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Study on the Optimal Size of a LiCAF Scintillator for a Neutron Ambient Dose Detector in BNCT

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INTRODUCTION: Accurate evaluation of neutron leakage dose is essential for radiation protection in Boron Neutron Capture Therapy (BNCT). A moderated spherical neutron detector was designed for the direct measurement of the newly proposed operational quantity, the neutron ambient dose, H^* ^[1]. The detector assembly consists of two polyethylene layers, a gadolinium layer, and a tungsten layer, with a relatively large diameter of 27 cm. A thermal neutron detector Eu-doped LiCaAlF₆ (LiCAF) scintillator was placed at the center. Due to the thick moderator structure, a relatively large LiCAF scintillator was required to improve detection efficiency while maintaining gamma-ray event suppression^[2]. In this research, the characteristics of gamma ray discrimination were confirmed.

EXPERIMENTS: Neutron–gamma discrimination performance was evaluated at the E3 thermal neutron beam port of the Kyoto University Research Reactor under irradiation conditions both with and without a cadmium cover. The cadmium cover was used to suppress thermal neutrons because cadmium has a very large cross section for thermal neutrons and produces gamma rays via the ¹¹³Cd(n,γ) reaction, thereby enabling gamma-ray-only irradiation of the LiCAF scintillator.

Three relatively large LiCAF crystals were prepared: (i) a 2 mm cube, (ii) a 2 mm diameter × 1 mm thick disk, and (iii) a 2 mm diameter × 0.5 mm thick disk. Each crystal was coupled to a quartz glass light guide and connected to a photomultiplier tube (PMT) for irradiation measurements.

RESULTS: Among the samples, the 2 mm diameter × 0.5 mm thick disk (iii) exhibited the best gamma-ray suppression, with a gamma-ray contribution of 0.4% within $\pm 3\sigma$ of the neutron peak, as shown in Fig. 1. This scintillator was therefore selected for the designed neutron ambient dose detector.

The detector was subsequently fabricated and tested in a BNCT irradiation field, demonstrating good performance in H^* measurement with high detection efficiency.

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[2] K. Watanabe et al., Nucl. Instrum. Methods Phys. Res. A 802, 1 (2015).

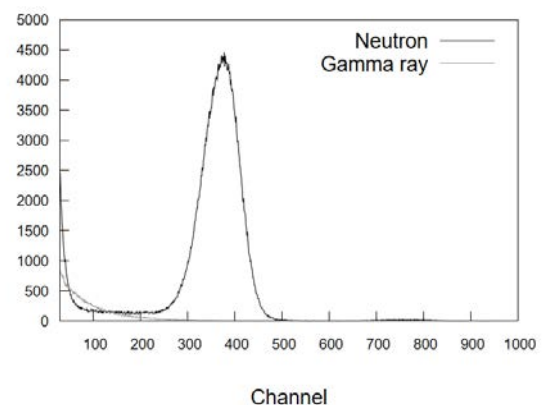


Fig. 1. Pulse height distribution of sample (iii): a 2 mm diameter × 0.5 mm thick disk.

Measurement of Thermal Neutron-Induced Soft Error Rates in Semiconductor Memories

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INTRODUCTION: Radiation-induced soft errors are one of the concerns threatening the reliability of semiconductor devices. Assessing the soft error risk is critically important especially in highly reliable applications, such as automotive and data center [1, 2]. In the terrestrial environment, thermal neutrons are one of the sources triggering the soft errors [3]. Susceptibility to the thermal neutrons depends on the amount and location of ^{10}B atoms introduced during manufacturing as a contaminant. However, such ^{10}B information is generally unavailable, making it difficult to predict the impact of thermal neutrons. In this context, irradiation testing is essential to assess the reliability risk due to thermal neutrons. This work investigates the thermal neutron-induced soft errors in static random-access memories (SRAMs) with two different process technologies. Soft error rates (SERs), which are the occurrence rates of soft errors, are measured by thermal neutron irradiation testing.

EXPERIMENTS: Thermal neutron irradiation was performed using Heavy Water Neutron Irradiation Facility (HWNIF) at KUR [4]. Irradiation modes used were “OO-0000F” and “CO-0000F.” Fig. 1 shows the energy spectra of these modes together with the terrestrial one [5]. Test samples were two SRAM chips with different technologies, which were labeled as Tech. A and Tech. B. The SERs were statistically calculated according to the JEDEC standard [6]. Thermal neutron fluxes at the sample positions were measured by the gold activation method.

RESULTS: The thermal neutron susceptibility was examined by comparing the SERs under “OO-0000F” and “CO-0000F” irradiations. Fig. 2 shows the measured SERs for Tech. A and Tech. B. For both the technologies, the SER under “CO-0000F” was significantly lower than that of “OO-0000F.” This clearly demonstrates that these SRAMs are susceptible to thermal neutrons. When focusing on the SER ratio between “OO-0000F” and “CO-0000F”, the ratio is higher for Tech. A than for Tech. B. One of the possible reasons for this is that the ^{10}B atoms are more abundant for Tech. A. Further analysis will be required to understand this difference.

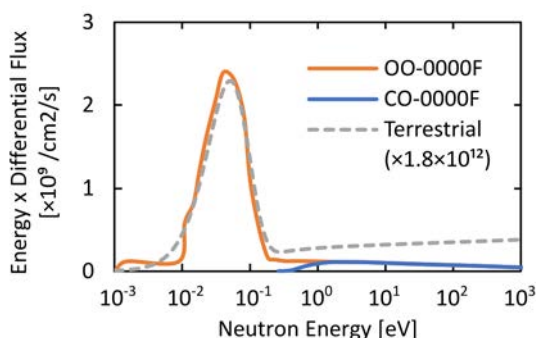


Fig. 1. Neutron energy spectra of KUR HWNIF [4] and terrestrial environment [5]

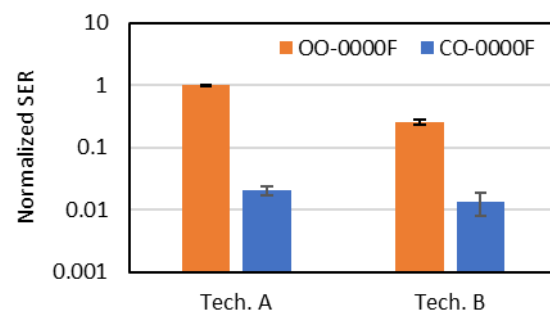


Fig. 2. Measured SERs for Tech. A and Tech. B SRAMs under two irradiation modes.

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Development of a Real-Time Monitoring System to Enhance Treatment Reliability in Boron Neutron Capture Therapy and Application to Nuclear Decommissioning

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INTRODUCTION: Real-time evaluation system of treatment effects in Boron Neutron Capture Therapy (BNCT) is important technique and required to improve treatment reliability. During BNCT, 478-keV gamma rays are emitted from the nuclear reaction between neutrons and ^{10}B , and various detectors have been proposed to image them. However, most methods have collimators [1–3], which act as scattering sources and reduce gamma-ray sensitivity. Although Compton cameras do not require collimators, their large size remains a drawback. In this study, we investigated the potential of an optical fiber-based dosimeter, previously tested with a ^{60}Co source and nuclear reactors at the Kyoto University Research Reactor Institute, and applied at the Fukushima Daiichi Nuclear Power Plant.

EXPERIMENTS: Four detector units were fabricated by coupling optical fibers with a newly developed scintillator based on the zero-dimensional perovskite-type halide crystal $\text{Cs}_3\text{Cu}_2\text{I}_5$. As shown in Fig.1, thermal neutrons were irradiated onto a boric acid sample using the E-3 neutron guide tube, and 478 keV prompt gamma rays were detected. The scintillator exhibits high light output and negligible hygroscopicity, providing advantages under conditions of low light collection efficiency, such as optical fiber readout systems. Image reconstruction was performed using a center-of-gravity algorithm. Considering the high count rates in practical environments, single-photon counting is difficult; therefore, we adopted a current-mode measurement without explicit energy discrimination. The influence of gamma rays other than 478 keV was also investigated.

RESULTS: Imaging of 478 keV gamma rays was successfully demonstrated using the optical fiber-based detector. The E-3 neutron guide tube, with a thermal neutron flux of approximately three orders of magnitude lower than actual treatment conditions, allowed single-photon counting. Imaging results with energy discrimination were compared to those using all spectral events (pseudo current mode), showing no significant difference. Consistent results were also obtained with an actual current-mode setup. Measurements using a Ge detector near the boric acid sample (Fig.1) showed 2.2-MeV and 511-keV gamma rays. However, 478-keV gamma-ray events were dominant. The 2.2-MeV gamma-event is negligible due to the small scintillator size (a few mm). Thermal neutron flux and background sources (e.g., oxygen-induced 511 keV gamma rays and activation) were silent in this experiment. Although evaluation under realistic conditions is required, in this time, we successfully demonstrated the imaging with current mode.

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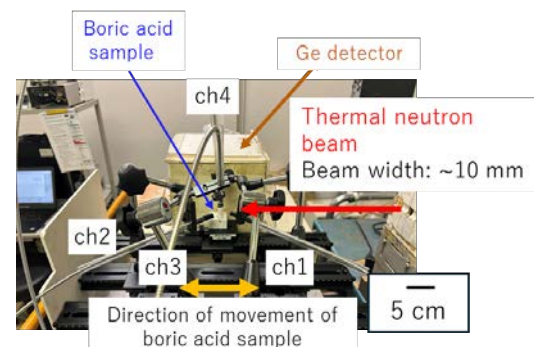


Fig.1. Schematic view of the setup for 478-keV gamma-ray detection with optical fiber-type detectors.

I-1. PROJECT RESEARCHES

Project 4

Project Research on Advances of Mössbauer Spectroscopy in Isotope-Specific Studies

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OBJECTIVES OF RESEARCH PROJECT

The primary objective of this research project is to leverage the unique capability of Mössbauer spectroscopy to extract precise information regarding electronic states, magnetic properties, and lattice dynamics for specific isotopes. The main objectives of this project research on advanced isotope-specific investigation on the frontier of materials science by the methods of Mössbauer spectroscopy. Developments of Mössbauer spectroscopy provides more useful and valuable methods in modern materials science.

MAIN SUBJECTS AND RESULTS OF THIS REPORT

(R7P4-1, K. Kitase) ⁶¹Ni Mössbauer spectroscopy of Fe-Ni Hofmann-type complex

K. Kitase et al. applied ⁶¹Ni Mössbauer spectroscopy to Fe-Ni Hofmann-type complexes, determining Ni isomer shifts and providing rare coordination data for these polymers.

(R7P4-2, Y. Kobayashi) ¹⁹⁷Au Mössbauer Spectroscopy of Au dithiolene complex, Au(40Et)

Y. Kobayashi et al. analyzed a neutral radical gold dithiolene complex, finding the Au state in the Au(IV) region rather than Au(III), consistent with DFT calculations.

(R7P4-3, Y. Kobayashi) Mössbauer Spectroscopy of Au Microparticle-supported Catalysts

The group estimated the Debye temperature of Au particles on TiO₂ and SiO₂ using the recoilless fraction to investigate lattice vibrations in catalytic environments.

(R7P4-6, R. Masuda) Analysis of iron compound nanoparticles in Zeolite

T. Kato et al. identified ferrihydrite nanoparticles in HY-zeolite. At 3 K, these showed super-paramagnetic behavior influenced by synthesis temperature and heat treatment.

(R7P4-8, K. Shinoda) Intensity Tensors of Quadrupole Doublet due to Fe²⁺ Ions of Amphibole

K. Shinoda et al. determined Fe intensity tensors using single-crystal hornblende, facilitating peak separation in complex multi-site minerals by relating γ -ray direction to peak intensities.

(R7P4-9, M. Tabuchi) Understanding of valence and local states for Fe ion in Fe and Ni substituted Li₂MnO₃ during charge and discharge runs

M. Tabuchi et al. monitored Fe oxidation in battery electrodes, finding significant valency shifts to tetravalent or pentavalent states during activation, depending on the initial calcination atmosphere.

(R7P4-10, M. Satish-Kumar) Pressure-temperature-fluid evolution and oxygen fugacity changes from calc-silicate granulites at Rundvågshetta, Lützow-Holm Complex, East Antarctica

This study estimated iron oxidation states in garnet from high-temperature rocks, finding Fe³⁺ (73.20%) strongly partitioned, slightly below traditional EPMA-based predictions.

(R7P4-12, K. Yonezu) Experimental Preliminary Approach on the Precipitation Mechanism of Banded Iron Formation (BIF) Part 4: Precipitation Behavior of Iron-bearing Siliceous Deposit Formed at Menengai Geothermal Power Plant, Kenya

K. Yonezu et al. modeled Banded Iron Formation (BIF) mechanisms using scale from the Menengai geothermal plant, characterizing iron speciation in modern siliceous deposits via ⁵⁷Fe Mössbauer spectroscopy.

(R7P4-14, S. Ishiwata) Spin and charge ordering in perovskite-type oxides Ca_{1-x}Bi_xFeO₃ probed by Mössbauer spectroscopy

The evolution of charge disproportionation (CD) and intermetallic charge transfer (CT) was investigated in high-valence iron oxides. The study explored how Bi-doping and high-pressure synthesis influence the formation of layered charge-ordered structures.

(R7P4-15, Y. Kamihara) Magnetism and Electronic Phase in a H-doped Iron-based Superconductor

Y. Kamihara *et al.* reported the first derivation of the Debye temperature (approx. 400 K) for H-doped SmFeAsO (Sm1111). The study combined SQUID magnetometry with ⁵⁷Fe Mössbauer spectroscopy to evaluate the material's magnetic and electronic phases.

(R7P4-16, T. Ohta) Mössbauer Characterization of a Non-Heme Fe^{III}-OOH Semiquinonate Intermediate

T. Ohta *et al.* performed characterization of an Fe^{III}-OOH semiquinonate intermediate. By fitting the spectra with Lorentzian lines, they obtained precise isomer shift and quadrupole splitting parameters to define the electronic state of the non-heme iron complex.

^{61}Ni Mössbauer spectroscopy of Fe-Ni Hofmann-type complex

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INTRODUCTION: Spin-crossover (SCO) phenomenon is a reversible spin transition between high-spin and low-spin state. Hofmann-type complex is the one of coordination polymer with bridging ligand $[\text{M}(\text{CN})_4]$. In Hofmann-type SCO complex, SCO phenomenon usually occurs in Fe center. ^{57}Fe Mössbauer spectroscopy is useful tool to investigate SCO complex since the isomer shift (IS), quadrupole splitting (QS), and spectrum shape of ^{57}Fe Mössbauer spectroscopy are sensitive to the coordination environments or spin state of Fe sites, so ^{57}Fe Mössbauer spectroscopy is widely used by many researchers to investigate SCO phenomenon of Fe center in many complexes. Hofmann-type complex contains other Mössbauer active nuclei such as Ni, Au. Nevertheless, other type of Mössbauer spectroscopy such as ^{61}Ni Mössbauer spectroscopy is much less researched than ^{57}Fe Mössbauer spectroscopy. Now we synthesized Fe-Ni Hofmann-type complex $\text{Fe}(\text{Ethyl Isonicotinate})_2[\text{Ni}(\text{CN})_4]$ (Fe-Ethyl Iso-Ni) and $\text{Fe}(\text{Allyl Isonicotinate})_2[\text{Ni}(\text{CN})_4]$ (Fe-Allyl Iso-Ni) and researched the properties of this complex by ^{61}Ni Mössbauer spectroscopy.

EXPERIMENTS: The complex was synthesized using Mohr's salt $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$, citric acid, $\text{K}_2[\text{Ni}(\text{CN})_4] \cdot \text{H}_2\text{O}$, and pyridine-type ligand ethyl nicotinate or Allyl Isonicotinate. First, Mohr's salt and ligand were dissolved into water. Then aqueous solution of $\text{K}_2[\text{Ni}(\text{CN})_4]$ was dropped into this solution. The powder sample was obtained as two types of colors, large amount of yellow powder and small amount of orange powder. We can separate yellow powder from orange powder. We confirmed that both types of powder are same formula and isostructural by elemental analysis and powder X-ray diffraction measurement. We charged powder of complexes into a 10 mm diameter holder for ^{61}Ni Mössbauer spectroscopy.

Table 1. Parameter of ^{61}Ni Mössbauer spectroscopy

	$\delta / \text{mm s}^{-1}$	FWHM	Peak area	Ni thickness
$\text{V}_{86}\text{Ni}_{14}$ alloy	0	0.95	0.0416	0.0794
Fe-Ethyl Iso-Ni	-0.06	1.21	0.0208	0.116
Fe-Allyl Iso-Ni	-0.002	1.07	0.0329	0.270

RESULTS: Fig. 1 shows ^{61}Ni Mössbauer spectroscopy of $\text{Ni}_{86}\text{V}_{14}$ alloy, Fe-Ethyl Iso-Ni and Fe-Allyl Iso-Ni. These spectra have singlet peak. The parameter of spectra is shown in table 1. The thickness of the latter two complexes is thicker than the $\text{Ni}_{86}\text{V}_{14}$ alloy sample. Nevertheless, the absorption intensity of the spectrum of both complexes are smaller than $\text{Ni}_{86}\text{V}_{14}$ alloy. This result could be explained as Hofmann-type coordination polymers contains organic long ligands and flexible framework. As result, the recoil fraction in both complexes is smaller than that of $\text{Ni}_{86}\text{V}_{14}$ alloy.

REFERENCE: K. Kitase, T. Kitazawa, S. Kitao, T. Kubota, Y. Kobayashi, M. Seto, J. Radioanal. Nucl. Chem., 335 (2026) <https://doi.org/10.1007/s10967-026-10820-4>

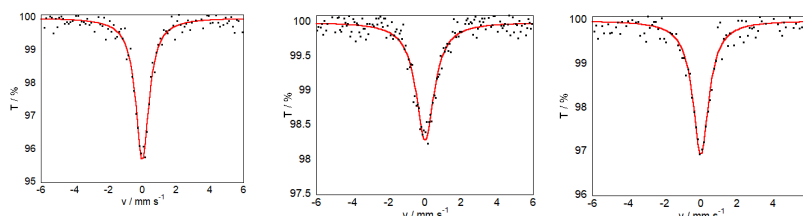


Fig. 1. ^{61}Ni Mössbauer spectroscopy of $\text{Ni}_{86}\text{V}_{14}$ alloy (left), Fe-Ethyl Iso-Ni (center) and Fe-Allyl Iso-Ni (right).

^{197}Au Mössbauer Spectroscopy of Au dithiolene complex, Au(4OEt)Y. Kobayashi¹, S. Yokomori², N. Kojima³, N. Matsushita³ and M. Seto¹¹ Institute for Integrated Radiation and Nuclear Science, Kyoto University² Graduate School of Science and Engineering, Ibaraki University³ Research Center for Smart Molecules, Rikkyo University

INTRODUCTION: Metal dithiolene complexes are a class of redox-active molecules whose frontier orbitals are composed of metal-centered d orbitals and ligand-based π orbitals. In general, oxidation in these complexes is considered to occur predominantly on the ligand framework, with only a slightly minor change in the formal oxidation state of the metal center. In the gold dithiolene complexes as well, one-electron oxidation from a monoanionic Au(III) complex to a neutral radical species has been interpreted as essentially ligand-centered, because no significant change indicative of partial oxidation at the Au site was detected in the ^{197}Au Mössbauer spectra,^[1] despite theoretical predictions of a small contribution of Au d orbitals to the SOMO. Notably, the neutral radical gold dithiolene complex Au(4OEt), which we recently synthesized, exhibits a canted antiferromagnetic transition below approximately 8 K, raising the possibility that Au-derived spin–orbit coupling contributes to the emergence of this magnetic order. In addition, theoretical calculations have suggested the presence of a small but non-negligible spin density on the Au atom, implying that, in this system, the one-electron oxidation may still be predominantly ligand-centered while being accompanied by a detectable change in the electronic state in the vicinity of the Au nucleus. In order to investigate the valence state of the Au in Au(4OEt), we investigated the ^{197}Au Mössbauer spectra of this complex system.

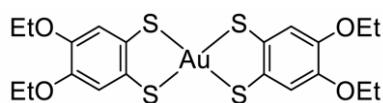


Fig. 1. Molecular structure of Au(4OEt).

EXPERIMENTS: The Mössbauer spectroscopic measurement of the 77.34 keV transition in ^{197}Au was carried out with both source and absorber cooled down to 18 K. A ^{197}Pt source was obtained by the neutron irradiation for 98% enriched ^{196}Pt metal by the nuclear reaction of $^{196}\text{Pt}(n,\gamma)^{197}\text{Pt}$ in the Kyoto University Reactor. The isomer shift of Au foil was referenced as 0 mm/s. The spectra was calibrated by using six lines of α -Fe foil at r.t.

RESULTS: The isomer shift (δ) and the quadrupole splitting (Δ) of Au(4OEt) are $\delta = 4.50$ mm/s, and $\Delta = 2.76$, respectively. On the other hand, δ and Δ of (Ph₄P)[Au(4OEt)] are $\delta = 4.24$ mm/s, and $\Delta = 2.84$, respectively. As shown in Fig. 2, these positions are deviated from the δ - Δ correlation belt of Au(III) state and are situated in the area of the Au(IV) state, which is consistent with our DFT calculation.

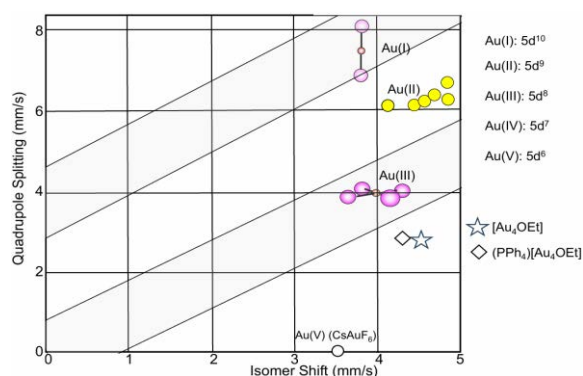


Fig. 2. Quadrupole splitting vs. isomer shift for Au complexes.

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Mössbauer Spectroscopy of Au Microparticle-supported Catalysts

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INTRODUCTION: The recoilless fraction is a parameter that reflects lattice vibrations and provides important knowledge in physics, chemistry, etc. We have succeeded in estimating the Debye temperature not from the absolute amount of spectral absorption area but from its temperature dependence curve and estimated that of some Au compounds [1]. We applied this technique to Mössbauer measurements of Au-supported catalysts and investigated the temperature dependence in more detail.

EXPERIMENTS: Prepared samples were Au particles on a TiO₂ supporter (Au/TiO₂) and Au particles on a SiO₂ supporter (Au/SiO₂). The Au contents were 2~10wt% in Au/TiO₂ and 2wt% in Au/SiO₂. The TiO₂ or SiO₂ powder was put in the HAuCl₄ aqueous solution, and Au(OH)₃ was precipitated on the powder surface by adjusting the pH while stirring. The obtained powder was dried and calcined at 300°C for 4 hours to prepare the sample. Using KUR, the ¹⁹⁷Pt γ -ray sources for ¹⁹⁷Au Mössbauer measurements were prepared by neutron irradiation to 98%-enriched ¹⁹⁶Pt metal foil. The γ -ray source and samples were cooled to the same temperatures using a helium refrigerator.

RESULTS: In the ¹⁹⁷Au Mössbauer spectra of the Au/TiO₂ and Au/SiO₂ samples, all components showed a single peak like metallic Au. It was found that Au was chemically in the metallic Au state in all samples. Figure 1 shows the temperature dependence of the absorption area, normalized by the absorption area at 20K for each measurement temperature. The Debye temperature of each sample was determined by comparing its temperature dependence with that expected for each Debye temperature. The results showed that the Au/TiO₂ 10wt%Au sample, expected to have a large particle size, had a Debye temperature of 159K, similar to that of bulk Au. In contrast, the Au/TiO₂ 2.5wt%Au sample, with smaller particle sizes, had a considerably lower Debye temperature of 128K [2]. The Debye temperature of the Au/SiO₂ 2wt%Au sample was 168K, and no decrease in Debye temperature was observed compared to Au/TiO₂. Lattice vibrations affect molecular adsorption and surface transport, and are therefore thought to be related to catalytic activity.

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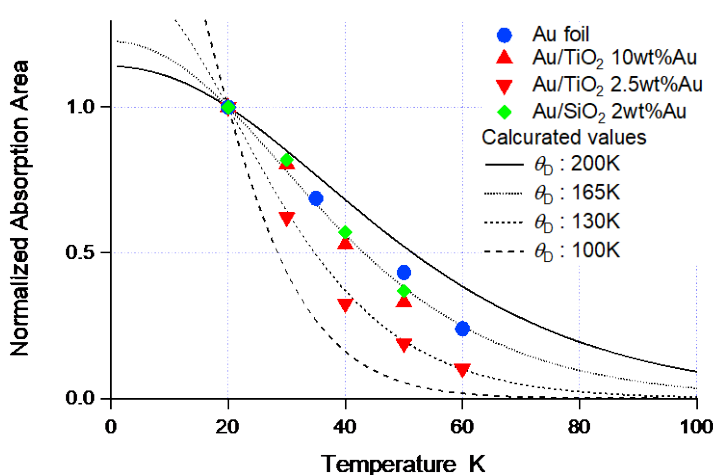


Fig. 1 The temperature dependences of the normalized absorption area obtained from the spectra (markers), together with that calculated using the Debye temperature (lines). The absorption area is normalized to its value at 20 K.

^{197}Au Mössbauer Spectra for Frozen Aqueous Solution of Gold(III) complex

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INTRODUCTION: As shown in a book published in 1976 [1], the distribution curves and phase diagram for the hydrolysis of AuCl_4^- complex was determined using potentiometry. Recently, ligand exchange for AuCl_4^- complex has been directly observed using the XAFS method. We have studied in order to observe this exchange using XAFS and other methods. ^{197}Au Mössbauer spectroscopy is widely known to be sensitive to ligand exchange of gold(III) ions. It is able to distinguish between oxygen and chlorine ligands. However, Mössbauer spectroscopy is considered extremely difficult to measure in aqueous solutions due to recoil phenomena. On the other hand, since ^{197}Au Mössbauer spectroscopy is performed at extremely low temperatures (approximately 20 K), it has been suggested that the aqueous solution may freeze and change its state. We hypothesized that we could achieve measurement by combining these two drawbacks. We conducted research with the aim of obtaining spectra of gold(III) aqueous solutions.

EXPERIMENTS: Aqueous solutions of AuCl_4^- complex were prepared at various concentrations. The pH were kept below 1 or above 13. To prevent uneven distribution of the aqueous solution in the cell and subsequent freezing, each solution was rapidly and completely frozen using liquid nitrogen before being placed in the Mössbauer apparatus.

^{197}Au Mössbauer spectra were measured at Kyoto University Research Institute of Nuclear Science. The ^{197}Pt isotope ($T_{1/2} = 18.3$ h), γ -ray source feeding the 77.3 keV Mössbauer transition of ^{197}Au , was prepared by neutron irradiation of isotopically enriched ^{196}Pt metal at the Kyoto University Reactor. The measurement temperature was around 20 K, and the measurement was performed by the transmission method.

RESULTS: Figure 1 shows ^{197}Au Mössbauer spectra for each AuCl_4^- complex solution frozen. It was found that the sufficient ligand changes of AuCl_4^- complex occurred and that it was preserved even in a frozen state.

REFERENCE:

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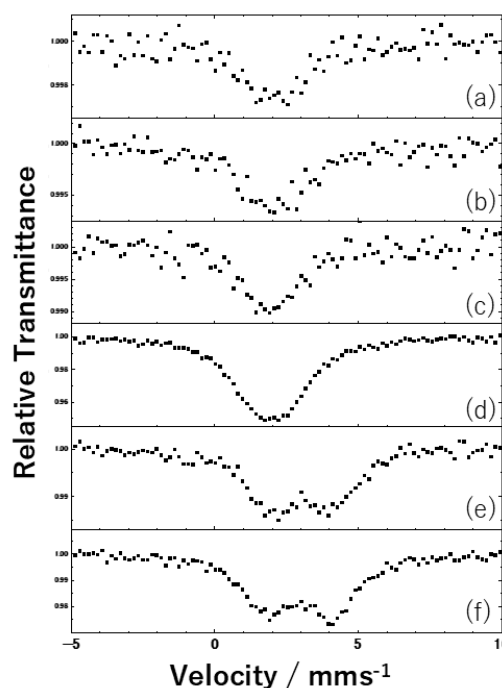


Fig. 1. ^{197}Au Mössbauer spectra for HAuCl_4aq solution frozen. (a) 200 mM, $\text{pH}<1$, (b) 500 mM, $\text{pH}<1$, (c) 1000 mM, $\text{pH}<1$, (d) $\text{HAuCl}_4 \cdot 4\text{H}_2\text{O}$ solid, (e) 250 mM, $\text{pH}>13$, (f) 500 mM, $\text{pH}>13$.

Analysis of iron compound nanoparticles in Zeolite

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INTRODUCTION: Zeolites are a series of porous materials whose general formula is $M_{1/n}(AlO_2)(SiO_2)_x \cdot yH_2O$ (M: an element with valence $n+$). They adsorb various substances within their pores and are used for deodorization and water purification. They are also used as hosts for the formation of nanoparticles. When nanoparticles are synthesized in zeolites, element-selective measurements are extremely effective for analyzing the nanoparticles, because the nanoparticles are indivisible from zeolites. Therefore, Mössbauer spectroscopy has been employed for the analysis of iron-compound nanoparticles in zeolites and elucidated that a variety of compounds are formed depending on the type of zeolites and the preparation methods [1]. Here, we studied the properties of nanoparticles synthesized by a chemical exchange method.

EXPERIMENTS: The sample nanoparticles were prepared using the powders of HY-zeolite $H(AlO_2)(SiO_2)_{7.35}$ and an aqueous solution of ferric chloride tetrahydrate $FeCl_2 \cdot 4H_2O$. The ion-exchange was performed at 50 °C or 70 °C. The zeolite powders including nanoparticles were dried in the air and a part of them were heat-treated in the air at 400 °C. Thus, we have four samples depending on the synthesis process: exchange temperature (50 °C or 70 °C) and heat-treat (none or 400 °C). The Mössbauer spectra of the samples were measured at room temperature in the air or at 3 K in the vacuum.

RESULTS: All the Mössbauer spectra at room temperature showed doublet pattern with the isomer shift of ~ 0.35 mm/s (relative to metal iron) and quadrupole splitting of ~ 0.9 mm/s. On the other hand, the spectra at 3 K were analyzed with one quadrupole doublet and one magnetic sextet. An example of the spectra at 3 K is shown in Fig. 1. These spectra agree that the nanoparticles were ferrihydrite $mFe_2O_3 \cdot nH_2O$ and showed super-paramagnetic behavior partially due to the small particle size even at 3 K [2]. The size of nanoparticles depends on the synthesis process: it would be larger at the temperature of ion-exchange process $T_{\text{exchange}} = 50$ °C or with the heat treatment.

REFERENCES:

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- [2] X. Wang *et al.*, *Environ. Sci.: Nano*, **3** (2016) 190-202.

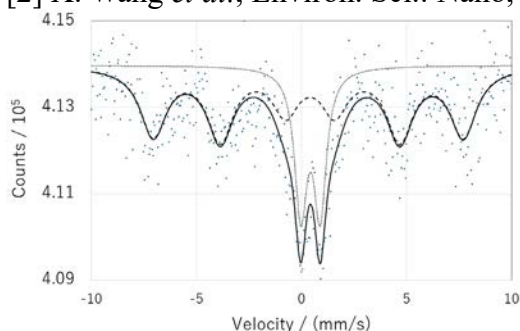


Fig. 1 The Mössbauer spectrum of iron nanoparticles with $T_{\text{exchange}} = 50$ °C without heat treatment. The spectrum was measured at 3 K.

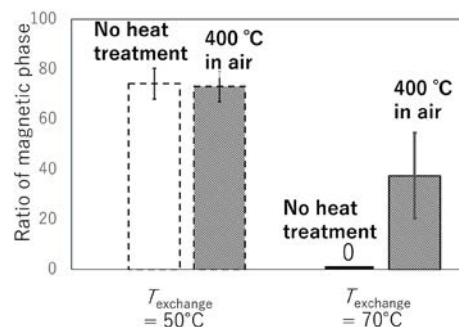


Fig. 2 The Ratio of magnetic components for each samples.

Intensity Tensors of Quadrupole Doublet due to Fe²⁺ Ions of Amphibole

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INTRODUCTION: A peak intensity (I) of quadrupole doublets of Mössbauer spectra is defined as $I_h/(I_h+I_l)$, where I_l and I_h are the lower and higher peak's intensity of quadrupole doublets, respectively [1]. The peak intensity of Mössbauer spectra of single crystal varies depending on the direction of γ -ray relative to crystal axes. The intensity tensor relates the direction of γ -ray to the peak intensity. This indicates that peak intensity can be calculated from intensity tensor and incident γ -ray direction. Amphibole is a multi-sites solid solution mineral that includes four M -sites. Thus, single crystal of amphibole shows complex Mössbauer spectra where multiple quadrupole doublets are overlapping. Peak separation of such a complex Mössbauer spectra is difficult, because it often results in multiple solutions of peak separation. Although intensity tensors due to Fe ions at M -sites is essential properties to select an appropriate fitting result from multiple solutions, intensity tensors due to Fe ions in amphibole are unknown. We reported an intensity tensor of Mössbauer quadrupole doublet due to Fe³⁺ at M site of oxyhornblende [2]. In this study, we report Mössbauer spectra of amphibole including Fe²⁺ and Fe³⁺ ions, its peak separation, and an intensity tensor due to Fe²⁺ and Fe³⁺ of amphibole.

EXPERIMENTS: A single crystal of hornblende from unknown occurrence was used for this study. Two crystallographically oriented thin sections perpendicular to b -axis were prepared by measuring X-ray diffraction methods. Ten Mössbauer spectra of oriented thin sections and one powder spectrum were measured. Mössbauer measurements were carried out in transmission mode on a constant acceleration spectrometer with an Si-PIN semiconductor detector (XR-100CR, AMPTEK Inc.) and multi-channel analyzer of 1024 channels. A 3.7GBq ⁵⁷Co/Rh of 4mm ϕ in diameter was used as γ -ray source. An ⁵⁷Fe-enriched α -iron foil was used as velocity calibrant. Two symmetric spectra were folded, and velocity range was ± 4 mm/s. Mosswin program was used for peak separation.

RESULTS: Fig.1 shows Mössbauer spectrum of amphibole single crystal under γ -ray // b -axis and peak separation by fixing isomer shift, peak width and quadrupole doublet obtained from powder spectrum. Doublet peak assignments of Bancroft [3] were adopted. In this experiment, thin sections normal to the c -axis were not completed because of perfect cleavage normal to the c -axis of amphibole. Thus, directions of incident γ -ray relative to the crystal axes were limited to directions around the b -axis. As the result, spectra normal to the b -axis were not measured, and reliable intensity tensors of four quadrupole doublets were not obtained. To compensate uncompleted direction of incident γ -ray, we must measure Mössbauer spectra normal to the a -axis.

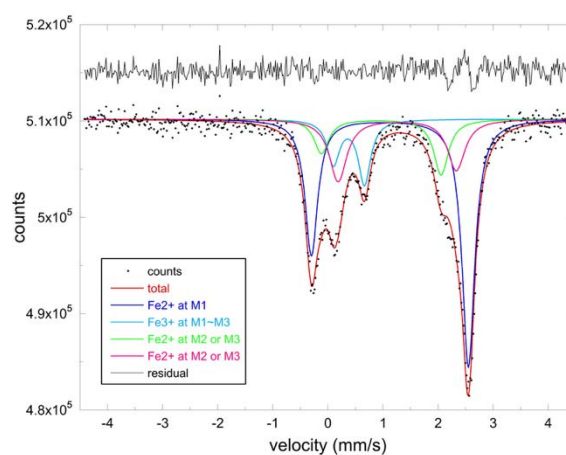


Fig. 1. Mössbauer spectrum of amphibole single crystal under γ -ray parallel to the b -axis and a peak separation of four QS doublets.

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Understanding of valence and local states for Fe ion in Fe and Ni substituted Li_2MnO_3 during charge and discharge runs

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INTRODUCTION: Large scale lithium-ion battery (LIB) is needed for EV and ESS applications. Low-cost positive electrode material is necessary for these applications. Therefore, Fe and Ni substituted Li_2MnO_3 is developed for above object [1, 2]. In this report, two $\text{Li}_{1+x}(\text{Fe}_{0.2}\text{Ni}_{0.1}\text{Mn}_{0.7})_{1-x}\text{O}_2$ ($0 < x < 1/3$, FNM217) samples were selected to check the change in valence state of Fe ion using ^{57}Fe Mössbauer spectroscopy during the electrochemical activation process.

EXPERIMENTS: The sample was prepared by coprecipitation-calcination method [2]. Final calcination atmospheres was selected to air (217A) or N_2 (217N) at 850°C for 10h. After washing with distilled water, the two samples were prepared for characterization. The electrochemical activation process was constructed as follows; The charged capacity increased from 80 mAh/g (1st) by 40 mAh/g step until 200 mAh/g (4th) after discharged to 2.0 V for Li/sample cell. At 5th cycle, the cell charged to 4.8 V and then discharged to 2.0 V to finish activation process. The active material was sealed in Al-laminate sheet for ^{57}Fe Mössbauer measurement. The isomer shift (IS) value was calculated using α -Fe foil. The Fe valency was examined for first charged samples (217A-1c and 217N-1c) as well as above as-prepared ones (217A and 217N).

RESULTS: XRD data showed that all samples were single phase of monoclinic Li_2MnO_3 -type structure. ^{57}Fe Mössbauer data fitted by two symmetric doublets with different isomer shifts (IS) for 217A. The IS values were +0.3561 (19) mm/s for main (89.6%) component and -0.042 (9) mm/s for minor (10.4%) one. Former and latter components can be assigned as tri and tetra-valent Fe ones, respectively [3]. On the other hand, the spectrum for 217N was fitted by a symmetric doublet (IS=+0.3 mm/s), indicating that 217N contains only trivalent Fe. Therefore, Fe valency was reduced by N_2 calcination.

Next, Fe valency for first charged samples were checked. As shown in Fig. 1, three symmetric doublets needed to fit each spectrum. The IS value for A component for 217A-1c was like that for 217N-1c, whereas those values of B and C components were different from each other. From the IS values of A components, they can be assigned as trivalent Fe ion. The IS values for other B and C components of 217A-1c were highly negative ones (-0.3 mm/s), where those of 217N-1c were nearly zero (-0.1 mm/s). The B and C components of 217A-1c and 217N-1c can be assigned to pentavalent [4] and tetravalent [3] Fe states, respectively. The difference may originate from the difference in initial Fe valency for 217A and 217N, as mentioned before.

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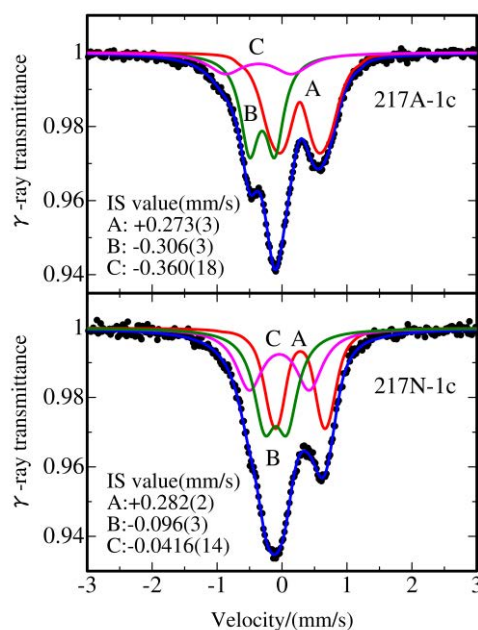


Fig. 1 ^{57}Fe Mössbauer spectra for 217A-1c and 217N-1c samples.

Pressure-temperature-fluid evolution and oxygen fugacity changes from calc-silicate granulites at Rundvågshetta, Lützow-Holm Complex, East Antarctica

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INTRODUCTION: Garnet is a very common mineral in the Earth's crust. It has a peculiar crystal structure and solid solution series which can incorporate Fe^{2+} and Fe^{3+} in varying conditions of its formation. In this study we attempted to estimate the $\text{Fe}^{2+}/\text{Fe}^{3+}$ ratio of grossular andradite garnet from a natural sample collected from Rundvågshetta in East Antarctica using messbauer spectroscopy. This locality is famous for its occurrence of ultra-high temperature metamorphic rocks, which are supposed to have attained a peak metamorphic temperature condition of $>900^\circ\text{C}$. The sample studies is a garnet bearing calc-silicate rock having a mineral assemblage of scapolite/plagioclase + clinopyroxene + garnet.

EXPERIMENTS: Petrographic observation of the sample revealed 3 types of garnets: i. garnet exhibiting a porphyroblastic texture with a diameter of approximately 2 mm, ii. garnet with an equigran-ular texture with a diameter of approximately 0.3–0.6 mm, and iii. garnet crystallizing in a coronal pattern with a thickness of approximately 0.05–0.2 mm. Of these, type-iii garnet showed the strongest grossular-andradite compositional variations when measured using SEM-EDS. Regardless of the presence or absence of compositional change, we prepared double polished thin slabs of 10 samples to perform local Mössbauer spectral measurements. Measurements were carried out following the previous studies [1,2]

RESULTS: As shown in Fig. 1, Mössbauer spectra of garnet were fitted with two doublets for Fe^{2+} and Fe^{3+} . The garnet grains in scapolite-rich domain contain on average 73.20 % Fe^{3+} , which is approximately 6 % lower than that predicted from the charge-balance calculation of Droop (1987) based on EPMA results. Clinopyroxene grains in scapolite-rich domain show only one doublet of Fe^{2+} , indicating all iron exist as ferrous iron. In the garnet-absent domain, clinopyroxene spectra were fit with two doublets for Fe^{2+} and Fe^{3+} , and, Fe^{3+} content is limited to 5.83 %. Although the clinopyroxene in scapolite-rich domain may contain few percent of Fe^{3+} within an analytical uncertainty, these results suggest that Fe^{3+} is strongly partitioned into garnet, and the esseneite content of clinopyroxene is only a few percent.

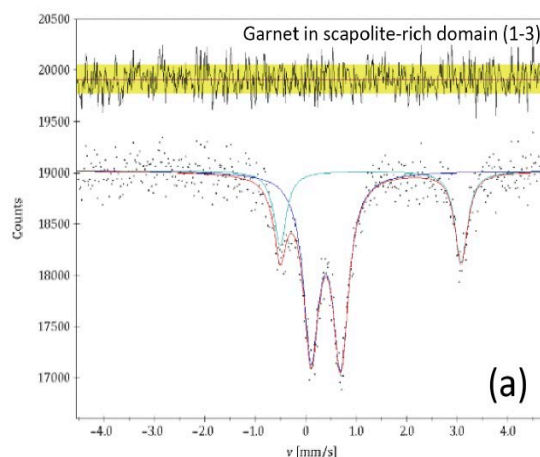


Fig. 1 Representative Mossbauer spectra of garnet

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Experimental Preliminary Approach on the Precipitation Mechanism of Banded Iron Formation (BIF) Part 4: Precipitation Behavior of Iron-bearing Siliceous Deposit Formed at Menengai Geothermal Power Plant, Kenya

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INTRODUCTION: Banded Iron Formation (BIF) is chemically precipitated sedimentary rock at Precambrian age. Current iron resource widely used in our industries largely depends on BIF. Commonly the hydro-thermal water, anoxic water was mixed with oxic seawater (e.g., [1]). However, the formation mechanism of BIF was not yet clarified. In order to approach the formation mechanism, we have done ambient temperature batch experiments, elevated temperature batch experiments and the experiments using natural system (e.g., [2]). Therefore, this study aims to more understand the formation mechanism of BIF and characterize iron-bearing siliceous deposits in terms of iron speciation from Menengai geothermal power plant in Kenya.

EXPERIMENTS: Several scale samples were collected from multiple locations of the Menengai geothermal power plant, including separators, atmospheric silencers, reinjection wells, brine lines and cooling towers. The samples were analyzed by XRD, XRF, petrograph and NMR (²⁹Si and ²⁷Al) prior to Mössbauer analysis.

RESULTS: As shown in Fig. 1, ⁵⁷Fe Mössbauer spectrum of iron bearing siliceous deposits collected from Menengai geothermal power plant suggested that Fe has 3 different species, Fe³⁺, magnetite and amorphous phases. The ratios of those 3 species are 33.7 %, 22.7 % and 43.5 %, respectively. Still we need to combine the results from ²⁷Al and ²⁹Si NMR for identification of possible iron bearing silicate. The results analyzed included the sample formed under both anoxic and oxic conditions. Most of oxic samples do not contain Fe²⁺, while the samples collected from separator or silencer is likely to have Fe²⁺ by the relatively reduced environment. The information is helpful to understand the formation mechanism of siliceous deposits and further of BIF. In addition, the knowledge can be expanded to the mitigation method for the silica scale formation in geothermal industry.

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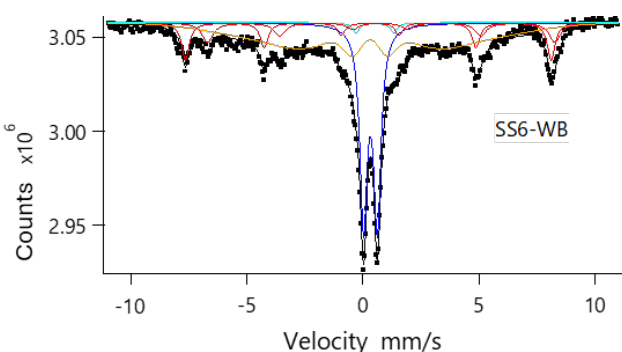


Fig. 1. An example of ⁵⁷Fe Mössbauer spectrum of iron bearing siliceous deposits collected from Menengai geothermal power plant.

Spin and charge ordering in perovskite-type oxides $\text{Ca}_{1-x}\text{Bi}_x\text{FeO}_3$ probed by Mössbauer spectroscopy

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INTRODUCTION: Perovskite-type iron oxides with anomalously high valence have attracted wide attention due to the versatile magnetic properties. The orthorhombic perovskite CaFeO_3 with Fe^{4+} exhibits a G-type spiral spin ordering and charge disproportionation (CD) described as $2\text{Fe}^{4+} \rightarrow \text{Fe}^{3+} + \text{Fe}^{5+}$ [1]. Recent research revealed that $\text{Ca}_{0.5}\text{Bi}_{0.5}\text{FeO}_3$ with $\text{Fe}^{3.5+}$ exhibits a CD with a $\text{Fe}^{3+}/\text{Fe}^{4.5+}$ layered charge ordered (CO) structure and intermetallic charge transfer (CT) described as $3\text{Bi}^{3+} + 4\text{Fe}^{4.5+} \rightarrow 3\text{Bi}^{5+} + 4\text{Fe}^{3+}$ [2,3]. The CD and localized electrons in CaFeO_3 facilitates the emergence of CO phases upon doping. Additionally, the hybridization in Bi-O bonds potentially affects the formation of CO phases. In this study, we focused on CaFeO_3 , which exhibits more localized electrons and $\text{Ca}_{1-x}\text{Bi}_x\text{FeO}_3$ was synthesized systematically, in order to explore the evolution of CO phases and CT.

EXPERIMENTS: Polycrystalline samples of $\text{Ca}_{1-x}\text{Bi}_x\text{FeO}_3$ were synthesized by annealing the nominal $\text{Ca}_{1-x}\text{Bi}_x\text{FeO}_{3-\delta}$ samples with oxidizing agent (KClO_4) at 1000 °C and 8 GPa for 1h in a cubic-anvil-type high-pressure apparatus. The ^{57}Fe Mössbauer spectra were measured with a ^{57}Co source in Rh. The velocity was calibrated with $\alpha\text{-Fe}$.

RESULTS: As shown in Figure 1(a) and (b), the Mössbauer spectra for $\text{Ca}_{0.8}\text{Bi}_{0.2}\text{FeO}_3$ and $\text{Ca}_{0.67}\text{Bi}_{0.33}\text{FeO}_3$ at 300 K exhibits clear singlets, indicating few oxygen vacancies. Below the phase transition temperature, both samples show two sextets corresponding to Fe^{3+} and Fe^{5+} , indicating onset of CD, $2\text{Fe}^{4+} \rightarrow \text{Fe}^{3+} + \text{Fe}^{5+}$. The relative areas of Fe^{3+} and Fe^{5+} are consisted with the nominal Fe valence at room temperature, revealing that no CT occurs during phase transition. Interestingly, unlike $\text{Ca}_{0.8}\text{Bi}_{0.2}\text{FeO}_3$, the $\text{Ca}_{0.67}\text{Bi}_{0.33}\text{FeO}_3$ sample shows an intermediate metastable phase at 190 K, where the Fe^{3+} sites are spin-ordered while the Fe^{5+} sites remain spin-disordered. The discrepancy suggests that the Bi doping level plays a significant role in stabilizing phases with novel spin and charge ordering. By combing the Mössbauer spectra with the structural, magnetic and electronic characterizations, we established a comprehensive phase diagram for $\text{Ca}_{1-x}\text{Bi}_x\text{FeO}_3$. This demonstrates that the hybridization of Bi-O bonds facilitates versatile phases with extraordinary magnetic and order ordering.

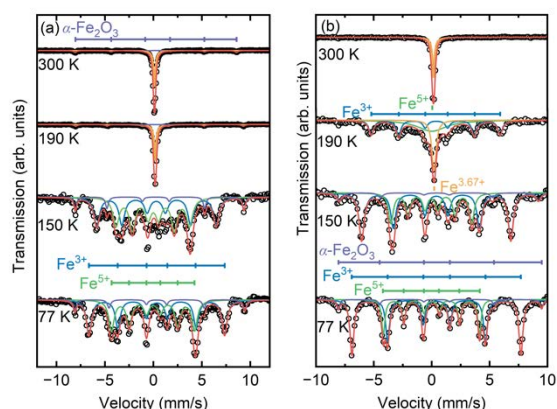


Fig. 1. The Mössbauer spectra for (a) $\text{Ca}_{0.8}\text{Bi}_{0.2}\text{FeO}_3$ and (b) $\text{Ca}_{0.67}\text{Bi}_{0.33}\text{FeO}_3$ at various temperatures.

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Research on Magnetism and Electronic Phase in a H-doped Iron-based Superconductor IV

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INTRODUCTION: The research in 2022-2024. The magnetic moment of a polycrystalline H-doped SmFeAsO (Sm1111) [1-6] was measured using SQUID magnetometer (Quantum Design MPMS). The magnetic critical-current-density (J_c) of the Sm1111 is 22.3 kA/cm². The lattice constants and volume as a function of H-doped Sm1111 samples. The F contents are determined for the Sm1111 sample using Volume Vegard's rule. Paramagnetic ⁵⁷Fe Mössbauer spectra at $T = 4.2, 10, 20, 50, 77, 300$ K were demonstrated for H-doped Sm1111 with ~10 at.% H contents.

EXPERIMENTS: The phase purity, lattice constants, and crystal structure of the sintered powders were examined by powder x-ray diffraction (XRD) Rigaku Rint2500 using Cu K alpha radiation from a rotating anode. The magnetic moment (M) of polycrystalline H-doped Sm1111 was measured using a SQUID magnetometer (Quantum Design MPMS) at several temperatures (T) and magnetic fields (H). The ⁵⁷Fe Mössbauer spectroscopy was performed using 14.4 keV Gamma-rays from a ⁵⁷Co source. The Mössbauer spectra of H-doped Sm1111 were analyzed using the code Moss Winn [7].

RESULTS: Figure 1 exhibits Isomer shift as a function of temperature for the Sm1111 polycrystalline sample. The obtained isomer shift (δ) values, which were obtained at each T , were used along with the theoretical formula to refine the Isomer shift- T plot. We derived and refined the Debye temperature ~400 K for the H-doped Sm1111. This value is first report for H-doped Sm1111.[8]

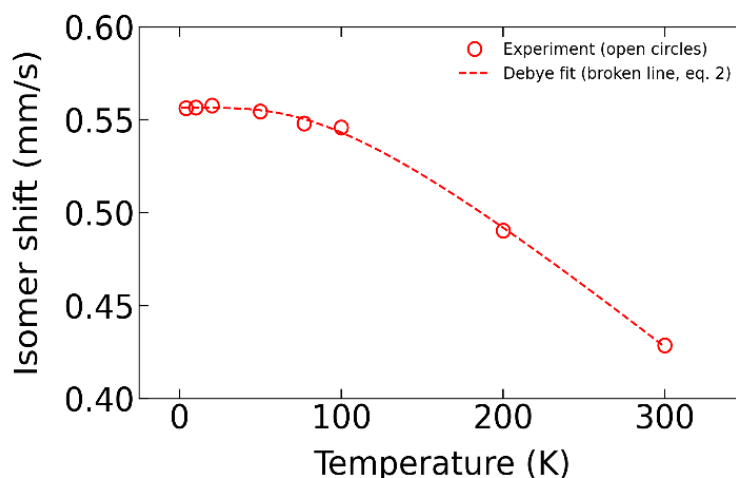


Fig. 1. Isomer shift as a function temperature for the H-doped Sm1111.

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Mössbauer Spectroscopic Characterization of a Non-Heme Fe^{III}–OOH Semiquinonate Intermediate

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INTRODUCTION: Studies on transient organic radicals in catalysis attracts significant interests due to the functional importance of such species in the active site of metalloproteins. In this study, an Fe^{III}–OOH semiquinonate intermediate, generated via reaction of ferric hydroquinone complex with potassium superoxide under cryogenic conditions, was characterized by Mössbauer spectroscopy.

EXPERIMENTS: Conventional transmission ⁵⁷Fe Mössbauer spectra were recorded using ⁵⁷Co in a Rh matrix as a γ -ray source at 4.2 K. The Doppler velocity and isomer shift (δ_{Fe}) for ⁵⁷Fe Mössbauer spectra were calibrated using the sextet of an α -Fe foil. The recorded spectra were fitted using Lorentzian lines to obtain Mössbauer parameters, i.e., δ_{Fe} and quadrupole splitting (ΔE_{Q}).

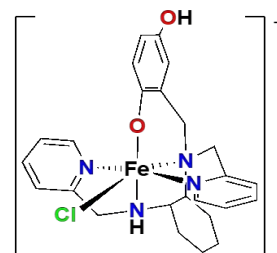


Fig. 1. Fe_HqBPCA (**1**)

RESULTS: The characterization of complex **1**, **Int-1**, and **Int-2** was performed using Mössbauer spectroscopy. The zero-field Mössbauer spectra of ⁵⁷Fe-enriched samples were measured at 4.2 K. The Mössbauer spectrum of complex **1** exhibits two distinct species with a quadrupole doublet characterized by an isomer shift δ_{Fe} of 0.51 mm s⁻¹ and a quadrupole splitting ΔE_{q} of 1.24 mm s⁻¹ and a quadrupole doublet characterized by an isomer shift δ_{Fe} of 1.38 mm s⁻¹ and a quadrupole splitting ΔE_{q} of 3.37 mm s⁻¹. The former parameters are characteristic of a ferric species with $S = 5/2$, whereas the later are typical of a ferrous species with $S = 2$. These observations suggest that complex **1** may undergo valence tautomerism between ferric-hydroquinone and ferrous-semiquinonate forms under measurement conditions. The major component of **Int-1** demonstrates Mössbauer quadrupole doublet characterized by an isomer shift δ_{Fe} of 0.50 mm s⁻¹ and a quadrupole splitting ΔE_{q} of 0.78 mm s⁻¹, parameters that are characteristic of ferric species with $S = 5/2$. These observations indicate that the Fe^{III} oxidation state of **1** remains unchanged upon reaction with superoxide. It is therefore plausible that antiparallel coupling of semiquinonate radical with the Fe^{III} center ($S = 5/2$) yields a total spin state of $S = 2$, rendering **Int-1** EPR silent under X-band excitation. The Mössbauer spectrum of **Int-2** reveals involvement of Fe^{III} center ($S = 5/2$), as evidenced by an isomer shift δ_{Fe} of 0.47 mm s⁻¹ and a quadrupole splitting ΔE_{q} of 0.72 mm s⁻¹.

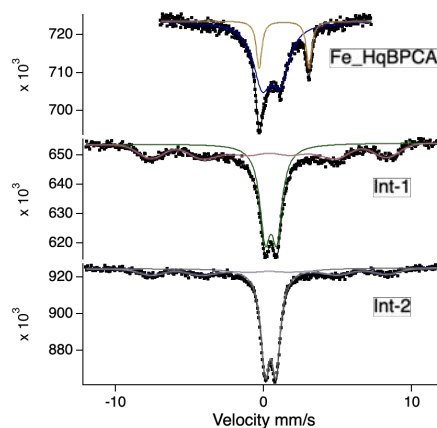


Fig. 2. Mössbauer spectra of **1**, **Int-1**, and **Int-2**.

I-1. PROJECT RESEARCHES

Project 5

An Operando Spectroscopic Methodology for Elucidating the Transient Intramolecular Dynamics of Aromatic Polymers under Ionisation

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PROGRAM OVERVIEW:

Aromatic ring polymers demonstrate significant potential for radiation detection without fluorescent dopants; nonetheless, fundamental mechanisms governing such luminescence remain largely unmapped. Observations indicate that exposing these materials to low-intensity charged particles elicits unconventional optical responses that defy explanation via traditional fluorescence frameworks. It is postulated that these signals originate from a transient metamorphosis of the polymer's intrinsic properties during irradiation—a phenomenon that terminates abruptly upon removal of the source. To transcend inherent constraints of "post-irradiation" analysis, an operando methodology has been pioneered. This platform facilitates simultaneous, real-time tracking of interatomic bonding, structural geometry, and electronic configurations. By capturing these "active" molecular states, this enquiry endeavours to pinpoint critical thresholds where optical characteristics transition, thereby unveiling the intricate reorganisation of energy levels occurring during ionisation.

IMPLEMENTATION:

To advance this enquiry, a versatile experimental framework has been engineered to integrate polymer samples with radiation sources within diverse analytical suites. Navigating the stringent spatial limitations of Raman, infrared, and ultraviolet-visible spectrophotometers necessitated a radical reappraisal of source design. Whilst the ultimate objective remains the fabrication of tailored sources via 3D printing, technical hazards during the prototypical phase were mitigated by refining a deposition method based upon 2D printing techniques. By employing functional media upon paper substrates, a significant breakthrough was achieved in the manufacture of prototype emitters. Initial trials focused on Polyethylene Terephthalate to probe its molecular behaviour in situ via operando measurement. These experiments yielded results synchronised with the irradiation cycle: whilst X-ray diffraction confirmed that the underlying atomic framework remained intact, a profound suppression of Rayleigh scattering was witnessed alongside a sharp amplification of Raman signals. Such a distinctive correlation cannot be attributed to internal luminescence alone, suggesting a temporary shift in the electronic landscape. Having amassed a substantial corpus of spectral data, current efforts are occupied with defining the "singular points" of this transition. This knowledge is instrumental in establishing a new paradigm for designing superior radiation-sensitive materials based upon the transient dynamics of polymers under ionisation.

ACKNOWLEDGEMENTS:

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Elucidation of the Dynamic Structures of Polymers under Ionisation

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INTRODUCTION:

We aim to elucidate the nature of anomalous optical signals observed in synthetic molecules upon exposure to charged particles—signals that are difficult to attribute to internal luminescence, such as fluorescence. It is highly probable that these signals arise from a 'switching phenomenon', whereby exposure to weak charged particles induces a temporal modification of the molecule's intrinsic optical properties. Given that this phenomenon vanishes immediately upon the cessation of irradiation, acquiring detailed observations has historically proven elusive. However, it is increasingly evident that the electronic states within the molecule undergo a dramatic transition between a steady state and an ionised state in response to the initiation and termination of irradiation. By employing operando measurements to track the real-time dynamics of bond strengths, constituent elemental arrangements, and electron distributions under irradiation, this research seeks to establish the fundamental physical mechanism underlying this hitherto unknown switching phenomenon.

IMPLEMENTATION:

Focusing on the enigmatic optical signals exhibited by undoped fluorescent polymers under environmental levels of charged particles, we formulated a working hypothesis: ionising radiation, resonating with the dynamic structure of the base adjacent to the aromatic ring, temporarily delocalises the intramolecular electron distribution. This induces a novel molecular mode that modifies inherent optical properties. To verify this hypothesis, we conducted pilot experiments for the operando measurement of molecular states under ionising radiation, integrating polyethylene terephthalate (PET) samples with a Strontium-90 source within various photometric instruments. These observations yielded critical data approaching the mechanism's core. Specifically, while X-ray diffraction confirmed the static structure remained stable during irradiation, the optical response exhibited profound alterations. We successfully captured mysterious, correlated events unexplained by conventional theories: a substantial attenuation of Rayleigh scattering for visible light, accompanied by a simultaneous, significant increase in Raman scattering intensity. Crucially, these anomalous responses vanished instantly upon removal of the radiation source, with all spectra returning perfectly to their original stable states. The acquisition of these reversible and highly correlated data sets provides robust evidence of the project's progression. Future work will focus on identifying critical points at which the optical system undergoes dramatic transitions relative to dose, while further scrutinising transition processes of bond strengths and electron distributions to fully reveal the mechanisms of ionisation-induced dynamic structural transformation.

ACKNOWLEDGEMENTS:

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Advancements in the Development of an Operando Ionization Measurement System

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INTRODUCTION:

Undoped fluorescent polymers containing aromatic rings have emerged as a significant focal point in radiation measurement technology. Conventionally, their luminescence behavior has been interpreted through ladder model based on electronic transitions within the molecular structure. However, in the detection of environmental-level charged particles, pulsed fluorescence emissions—distinctly inconsistent with existing theoretical predictions—have been repeatedly observed specifically in the presence of charged particles. To transcend these theoretical frameworks and elucidate this phenomenon as a physical reality, "operando measurement" has become an indispensable methodology, enabling the dynamic tracking of intramolecular electron distribution and bond strength variations associated with the activation and deactivation of radiation.

IMPLEMENTATION:

In this study, we established a robust foundation for operando measurements by developing original sample and radiation-source support stands compatible with a wide array of spectrometers, designed to capture the "live" molecular electronic states within polymers during irradiation. Using this system, we have recently completed a comprehensive spectral analysis under irradiation for a broad range of aromatic polymers, including PET. Following the acquisition of an extensive dataset documenting changes in various spectra in the presence and absence of charged particles, we are now concentrating our efforts on the most critical objective: identifying the "singular points" where optical properties switch. The successful identification of these singular points will reveal, for the first time, the dynamic process of orbital energy reorganization under ionization. These research achievements have served as a novel bridge between science and society through their dissemination to approximately 2,000 high school students. During this initiative, 460 students who deeply internalized the technical essence of the research re-synthesized the information into hand-drawn illustrations in their own sketchbooks. Subsequently, at the Expo 2025 Osaka, Kansai, Japan, these students took the stage with their sketchbooks to enthusiastically introduce our findings to the visitors. This seamless flow of knowledge—from researchers to students and onward to the general public—garnered significant media attention, standing as a testament to the profound social impact of this project.

ACKNOWLEDGEMENTS:

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Progress in the Development of a Radioactive Source Unit for an Operando Ionization Measurement System

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INTRODUCTION:

In the field of safety management for radiation facilities and nuclear power plants, polymer materials containing aromatic rings have attracted significant attention as functional elements for radiation detection. While their fundamental operational principle is based on the ionizing effects of radiation, the transient, microscale reaction processes occurring at the onset of irradiation remain insufficiently understood. We aim to develop an operando measurement system designed to monitor these microscale physical and chemical transitions within polymer matrices in real-time. A critical technical challenge in realizing this system is the in-house fabrication of radioactive sources with customizable geometries, ensuring compatibility with the highly restricted spatial envelopes of various analytical instruments.

IMPLEMENTATION:

To achieve a precise analysis of intramolecular electronic states under environmental-level low-dose irradiation, it is imperative to simultaneously integrate both the polymer sample and the radiation source within the highly restricted sample chambers of X-ray diffractometers and various spectrometers. Commercially available radioactive sources, which are characterized by standardized dimensions, present significant challenges for such integration across diverse analytical platforms. While our ultimate vision involves the utilization of 3D printing technology for customized source molding, we pivoted toward establishing foundational techniques using a more accessible 2D printing approach to mitigate substantial capital and technical risks during the prototyping phase.

Through this strategic approach, we successfully developed a precision deposition technique using potassium solutions as functional ink on paper substrates, representing a major milestone in the fabrication of prototype beta-particle sources. The cutting-edge insights gained from this project extend far beyond the laboratory. We conducted specialized technical lectures for approximately 2,000 high school students, providing them with a foundational understanding of this innovative measurement technology. Among them, 460 students who demonstrated a profound mastery of the technical essence took a leading role at Expo 2025 Osaka, Kansai, Japan. These students shared and interpreted the research findings for an audience of approximately 10,000 visitors, including both the general public and subject-matter experts. This initiative, bridging the gap between frontier science and society, contributes significantly to the cultivation of the next generation of scientists and the enhancement of public literacy regarding radiation science.

ACKNOWLEDGEMENTS:

This work was supported by JSPS Grant-in-Aid for Challenging Research Exploratory Grant Number 24K21749.

I-1. PROJECT RESEARCHES

Project 6

Advanced Applications of Materials Irradiation and Characterization Techniques Using High Energy Particles

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OBJECTIVE:

We have developed and maintained various irradiation facilities (KUR SSS for neutrons and KURNS-LINAC targets for electrons) and characterization facilities (positron annihilation spectroscopy and thermal desorption spectroscopy systems) in Institute for Integrated Radiation and Nuclear Science. For stimulating joint-use research projects in this area, this project aims to further advance the development and the improvements of facilities and to study novel irradiation effects.

RESEARCH SUBJECTS:

R7P6-1 Study to improve transport and measurement performance of slow positron beamline

The current performance of the positron beam was examined to confirm the availability of typical analytical techniques after a problem with the positron source occurred.

R7P6-2 Micro-vickers hardness in neutron-irradiated Fe-Cr alloys: Part 2

The progress of phase separation in Fe-Cr binary alloys was evaluated by positron annihilation spectroscopy and correlated with hardness for samples neutron-irradiated at 473K and 573 K.

R7P6-3 Positron and Gamma ray excited light-emissions from ZnO

The radiation-induced luminescence spectra of a ZnO crystal substrate were investigated, specifically comparing emission characteristics when excited by positrons and gamma rays.

R7P6-4 Hydrogen trapping behavior in vacancy type defects in B2-type Fe-Al alloys

Positron annihilation spectroscopy was used with elastic recoil detection analysis to investigate hydrogen-vacancy interactions in the Fe-Al alloys irradiated at KURNS-LINAC.

R7P6-5 Structural changes in Mn-DLC films due to Mn elution

Changes in vacancy-type defects within the DLC film accompanying Mn dissolution were investigated using positron annihilation spectroscopy (PAS) with a slow positron beam.

R7P6-6 Evaluation of density in diamond-like carbon coatings using positron annihilation spectroscopy

DLC coatings were systematically characterized using positron annihilation spectroscopy, with a focus on the relationships among deposition conditions, defect structure, and film density.

R7P6-7 Evaluation of irradiation damage and the effects of heat treatment on Au-Si ion-irradiated SiO₂/Si substrates using positron annihilation spectroscopy

To evaluate the Au-Si ion irradiated damage of SiO₂/Si surfaces before and after annealing, positron annihilation spectroscopy was performed.

R7P6-8 Effects of pulse irradiation by charged particles on damage structures in metals

The effects of pulse irradiation on the defect evolution in metals were evaluated for pulse-ion-irradiated samples and numerical simulations were performed for comparison.

R7P6-9 Development of slow positron beamline using electron linear accelerator

A slow positron beam generated by a 30 MeV electron beam was introduced to a sample chamber; the annihilation gamma-rays were successfully detected by a scintillation and Ge detectors.

Study to improve transport and measurement performance of a slow positron beamline

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INTRODUCTION: Positron annihilation spectroscopy is an important analytical method to detect vacancy-type defects and vacant spaces of materials. Energy-variable mono-energetic positron beams (slow positron beams) are essential to perform depth-dependent positron annihilation spectroscopy of surface layers such as ion-implanted layers or thin films. As moderation efficiencies to obtain slow positron beams are typically low ($<10^{-4}$), intense positron sources are required for practical use. A positron source using pair-creation by gamma-rays from a nuclear reactor have been developed by using Kyoto University research Reactor (KUR) to obtain a slow positron beam for materials analysis. In October 2025, a problem with the positron source occurred and the positron intensity decreased to $>50\%$ of those before the problem. Owing to high radiation levels of the activated source, it cannot be repaired during the yearly operation of KUR. Thus, we investigated the state of the positron beam and examined the availability of analytical techniques without repairing the source.

EXPERIMENTS: Energy distributions of the positron beam from the source were evaluated by changing the chopper bias voltage of a positron pulsing system[1]. Count rates of a Ge detector were used as a measure of the positron intensity. Doppler broadening annihilation radiation (DBAR) and positron annihilation lifetime spectroscopy (PALS) were examined for the positron beam without any extraction voltages. For scintillation detectors used in PALS measurements, three types of scintillation BaF₂ crystals (small $\phi 20 \times 20$, Medium: $\phi 25 \times 25$, Large: $\phi 50 \times 40$ mm) were compared[2]. A Kapton sample was measured at an acceleration energy of 2 keV as a reference material.

RESULTS: We found that electrical connection for the source electrodes (converter, moderator and lens) was inactive. Fig. 1 shows the chopper voltage dependences of positron count rates. Without extraction voltages, positron energies around 10 eV were observed. It can be ascribed to charging-up effects of the electrodes. Fig. 2 compares the count rates for three-types of BaF₂ crystals. Previously used small BaF₂ gave ~ 14 cps but the introduction of the medium BaF₂ increased to ~ 23 cps without serious degradation of the spectra. Appropriate lifetimes cannot be obtained with the large BaF₂ as shown in the previous study[2]. Thus, the medium size BaF₂ was chosen for PALS measurements.

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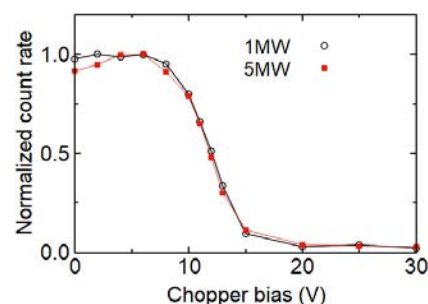


Fig. 1. Normalized count rates as a function of chopper bias voltage.

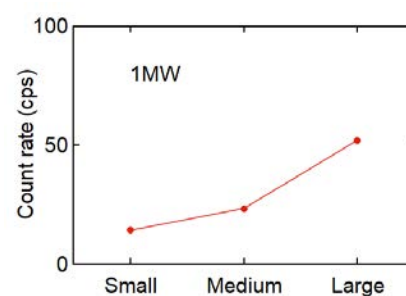


Fig. 2 Count rates of lifetime spectroscopy for different BaF₂ sizes.

Micro-Vickers Hardness in Neutron-irradiated Fe-Cr Alloys: Part 2

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INTRODUCTION: Ferritic stainless and heat resistant steels used as nuclear reactor peripheral materials have high Cr content [1]. In these materials, ductility and toughness remarkably decrease, and hardness and tensile strength increase by aging from 593 to 813 K. This phenomenon is caused by the formation of Fe-rich and Cr-rich phase, and is called 475°C embrittlement [2]. These changes in mechanical properties are an important issue in terms of evaluation of aged deterioration when it was used as a reactor structural material. Positron annihilation spectroscopy (PAS) is very powerful tool to obtain the information of vacancy-type defects (even single vacancies) and precipitates. In Fe-Cr alloys used in this study, positron affinity of Fe is lower than that of Cr [3]. Therefore, we can detect the formation of Fe-rich phase in phase separation of Fe-Cr alloys using PAS. The purpose of this study is to detect the progress of phase separation using PAS, and to obtain the correlation between the hardness and phase separation in Fe-Cr binary alloys irradiated with neutrons at 473K and 573 K.

EXPERIMENTS: Fe-x wt.%Cr (x = 0, 9, 15, 30, 45, 50, 70, 85, 91, and 100) binary alloys were used in this study. The weight of high purity Fe (99.99%) and Cr (99.99%) were measured, and samples were melted by the arc melting. For neutron irradiation, samples with diameters of 3 mm and thickness of 0.25 mm were cut using the wire electric discharge machine. Fe-xCr (x = 30, 45, 50, 70, 85, 91, and 100) were annealed at 1273K for 1h, and Fe-xCr (x = 0, 9, 15) were annealed at 1073K for 1h in a vacuum ($< 4 \times 10^{-4}$ Pa), and then water-quenching was performed for the suppression of phase separation. The neutron irradiation was carried out at the Material Controlled Irradiation Facility (SSS) of Kyoto University Reactor (KUR) [4]. The doses were 0.44×10^{-3} , 0.5×10^{-3} , and 2.1×10^{-3} dpa, at 473 K (0.5×10^{-3} dpa) and 573 K (0.44×10^{-3} and 2.1×10^{-3} dpa). Vickers hardness was measured at room temperature using an HMV-T2 (Shimadzu) with a load of 0.9807 mN (Hv0.1) and a dwell time of 15 s.

RESULTS: The hardness increased after neutron irradiation in all samples. In Fe-xCr (x = 0, 9, and 15), the hardness is highest after irradiation at 473 K. In Fe-xCr (x = 30, 45, 50, 85, and 91), the hardness of samples irradiated at 473 K for 47 h is almost the same as that of samples irradiated at 573 K for 42 h. In Fe-70Cr irradiated at 473 K, it is expected that irradiation-induced defects, especially those present in the Cr-rich phase, have a significant influence on the hardness. In Fe-xCr (x = 15, 30, 45, 50, and 70) irradiated at 573 K for 200 h, the hardness is lower than that after irradiation at 573 K for 42 h. It is expected that long irradiation times lead to significant progress of phase separation, and that the wavelength of Fe/Cr compositional fluctuations may increase. Consequently, dislocations may migrate more easily through the two phases. This possibility should be investigated using thermally aged Fe-Cr alloys.

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Positron and Gamma ray excited light-emissions from ZnO

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INTRODUCTION: Zinc oxide (ZnO) is a widely studied wide-bandgap semiconductor because of its excellent optical properties and diverse applications in optoelectronics, gas sensors, and scintillator technologies [1]. ZnO is known to exhibit visible defect-related luminescence, such as a broad green band emission. The specific characteristics of this visible emission are highly dependent on the type, concentration, and distribution of native point defects—such as oxygen vacancies or zinc interstitials—present within the crystal lattice. Photoluminescence (PL) spectroscopy, using laser or UV light as an excitation source, has been the standard technique for characterizing these defects. While PL is generally surface-sensitive due to the limited absorption depth of light, different types of radiation such as positron and gamma ray can probe various depths and volumes within the crystal. This brief report investigates the radiation-induced luminescence spectra of a ZnO crystal substrate, specifically comparing emission characteristics when excited by positrons and gamma rays.

EXPERIMENTS: Sample used for the present study is ZnO bulk single crystal substrate. The ZnO substrate was irradiated at room temperature with gamma-rays of 1.17 and 1.33 MeV from a cobalt-60 source and positron source of Institute for Integrated Radiation and Nuclear Science, Kyoto University. The photo-emission measurements were performed by using a charge coupled device (CCD) equipped spectrometers.

RESULTS: The provided spectral data illustrates the luminescence intensity of a ZnO single crystal as a function of wavelength under two distinct excitation sources. The bottom panel displays the luminescence spectrum induced by gamma-ray excitation [2]. This spectrum exhibits a characteristic, broad emission band extending from approximately 400 nm to over 800 nm, with a pronounced peak maximum centered around 550 nm. This broad green-yellow emission is typical for bulk ZnO crystals and is generally attributed to deep-level intrinsic defects dispersed throughout the crystal lattice. In contrast, the top panel shows the luminescence spectrum obtained under positron excitation. While a broad emission band in the green region (500–600 nm) is still evident, the spectral shape and quality differ significantly from the gamma-ray excited spectrum. One possible reason for these observed spectral differences is considered to be the difference in the penetration depths of the respective radiation types. Gamma rays are highly penetrating electromagnetic radiation. They deposit their energy deep within and throughout the bulk of the ZnO crystal. Consequently, the gamma-ray excited spectrum represents the averaged defect luminescent properties of the entire bulk volume. In contrast, positrons interact strongly with matter and possess a significantly shallower penetration depth. When a positron interacts with the sample, it implants and thermalizes primarily within the near-surface region of the crystal. Therefore, the positron-excited spectrum acts as a highly localized probe, reflecting the specific defect structure near the surface.

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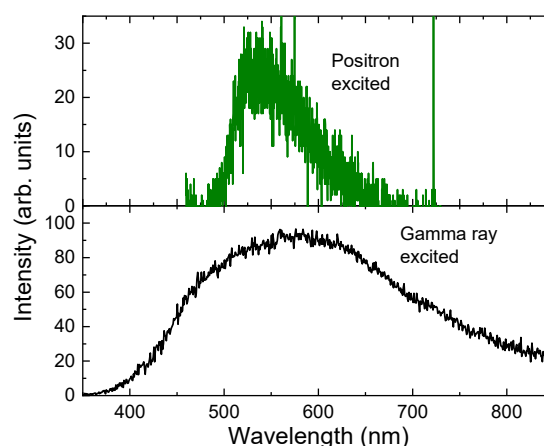


Fig. 1. Radiation-induced luminescence spectra of a ZnO single crystal substrate. The top and bottom panels display the emission spectra under positron and gamma-ray excitation, respectively.

Hydrogen trapping behavior in vacancy type defects in B2-type Fe-Al alloys

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INTRODUCTION: Fe-Al alloy is used as high strength at high temperature material because of its good properties such as specific strength to weight ratio, oxidation resistance. First-principles calculation indicates that not only one hydrogen atom but multiple hydrogen atoms can be trapped in a single vacancy in B2 ordered Fe-Al alloys. However, the interaction between vacancy and hydrogen atoms in this Fe-Al alloy is not cleared yet. We have been investigated this interaction in Fe-Al intermetallic alloy. Previous experiments have reported the changes in the positron annihilation S-parameter distribution and a decrease in positron lifetime, suggesting the possibility of hydrogen trapping at vacancies near the surface of this alloys [1]. To verify whether the changes observed by positron annihilation measurements were due to the trapping of hydrogen into vacancies, elastic recoil detection analysis (ERDA) was performed on the same sample.

EXPERIMENTS: Fe50at.%Al alloy sample was prepared by arc melting method. Sliced samples with the thickness of 0.5 mm were annealed at 1273 K for 20 h and cool down to 973 K slowly and then quenched. These specimens were irradiated with 8 MeV electron up to the fluence of 4×10^{18} /cm² by LINAC at KURRI. After irradiation, hydrogen was injected for the sample at 0.1 mA/cm² in a NH₄SCN solution bath added 0.001 mol/L H₂SO₄ by electro chemical method for 8, 16 and 80 hours. They were examined by using slow positron beam at KUR and ERDA measurement.

RESULTS: Figure 1 shows the hydrogen distribution obtained by ERDA measurement in a sample injected with hydrogen after electron beam irradiation. For comparison, the figure also shows the distribution of S-parameters, which represent the depth distribution related to vacancies measured with slow positrons, corrected for positive and negative inversion, as a solid line of the same color. Since the depth resolution of ERDA is at least 10 nm and the surface position is determined using another sample, the surface here does not necessarily precisely coincide with that for positron analysis. As the hydrogen injection time increases, the peak of ERDA gradually shifts to deeper level, and overall intensity increases. This indicates that hydrogen is penetrating and diffusing in the depth direction. Also, the trend of the hydrogen distribution of S-parameter and ERDA for each charging time are similar. That is, as the charging time increases, the peaks shift to deeper regions and are located at almost the same depth. This suggests that the S-parameter distribution reflects the distribution of hydrogen, and consequently indicates the presence of hydrogen within the vacancies.

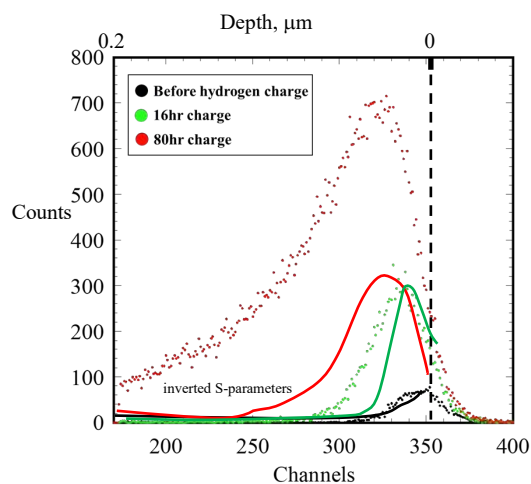


Fig. 1. The depth profile of hydrogen in Fe-Al alloy injected with H after electron irradiation by ERDA. For comparison, the solid line represents the positive/negative inverted S-parameter distribution.

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Structural Changes in Mn-DLC Films due to Mn Elution

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INTRODUCTION: Research on metal-storage DLC films capable of storing bioactive metal elements within metallic implants and supplying them in trace amounts to the body is gaining attention. Understanding the metal release process requires knowledge of the structure of the deposited metal-containing DLC films and how this structure changes due to metal release. Particularly, vacancy-type defects within DLC films containing metals are thought to influence both the amount of metallic elements the DLC film can contain and the amount of metallic elements that are slowly released. In this study, focusing on DLC films containing Mn, which has bone-promoting effects and antioxidant properties, changes in vacancy-type defects within the DLC film accompanying Mn dissolution were observed using positron annihilation spectroscopy (PAS) with a slow positron beam.

EXPERIMENTS: The Mn-DLC film samples were deposited on Si wafers using Ar sputtering with a C/Mn mixed target. The film thickness was 400-500 nm. The Mn-DLC films were immersed in distilled water, removed after 12 h, 24 h, and 48 h, dried, and stored. PAS measurements were performed using a slow positron beam system installed at the B-1 hole of KUR. Positrons generated in the reactor were thermalized, and accelerated up to 30 keV just before the irradiation chamber, and irradiated onto the sample. The γ rays generated by annihilation were observed using a Ge detector.

RESULTS: X-ray photoelectron spectroscopy (XPS) measurements showed the surface Mn composition decreased from 6.6% before immersion to 2.1% after 48 h. Pulsed 2 keV positrons were injected into the Mn-DLC film, and the positron annihilation lifetime was determined by observing the decay of the gamma ray intensity. The positron annihilation lifetime (PALS) increased with immersion time, indicating that the hole-type defects in the Mn-DLC film grew larger. On the other hand, the S-parameters decreased upon immersion. This is considered to be due to changes in the chemical environment around vacancy-type defects.

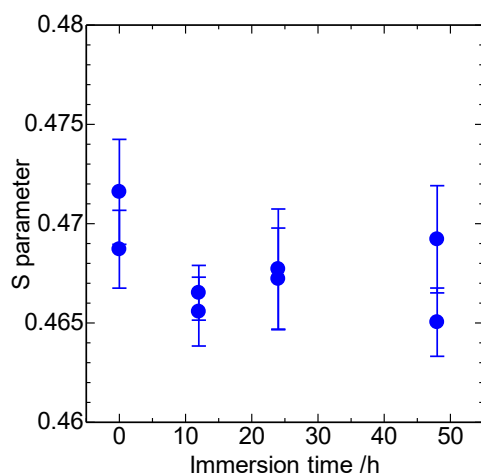


Fig. 1. Dependence of S-parameter of Mn-DLC film on immersion time.

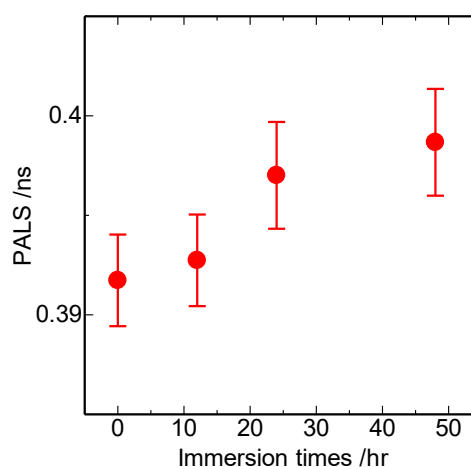


Fig. 2. Dependence of PALS of Mn-DLC film on immersion time.

Evaluation of Density in diamond-like carbon coatings using positron annihilation spectroscopy

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INTRODUCTION: Enhancing the efficiency and durability of mechanical systems has driven increasing interest in carbon-based materials with reduced friction and wear. Among these, diamond-like carbon (DLC) coatings have attracted significant attention due to their favorable tribological behavior combined with high hardness and chemical stability [1]. The properties of DLC are strongly dependent on their atomic structure, which can vary from graphite-like carbon (GLC) to polymer-like carbon (PLC) depending on deposition conditions and fabrication techniques [2]. Such structural variations are governed by factors including hydrogen content and the sp^2/sp^3 ratio, which directly influence the packing density of the carbon network. In addition to the bonding structure, the presence of nanoscale defects and free volume plays a critical role in determining the overall density and associated mechanical properties of DLC coatings [3]. These defects, often associated with open-volume regions, can alter the local atomic arrangement and consequently affect coating performance. Therefore, a precise evaluation of density in relation to defect structure is essential for understanding and optimizing DLC materials. Positron annihilation spectroscopy (PAS) is a highly sensitive technique for probing such open-volume defects and free volume at the atomic scale. In this study, the density of DLC coatings deposited at varying bias voltages is systematically characterized using PAS, with a focus on elucidating the relationships among deposition conditions, defect structure, and film density.

EXPERIMENTS: The DLC coatings were synthesized using a plasma-based ion implantation and deposition (PBII&D) on Si (100) substrates. To control the microstructural characteristics and resulting coating density, negative bias voltages ranging from -1 to -10 kV were adjusted during deposition. The duration time was set to achieve a coating thickness of 1 μm .

RESULTS: Figure 1 presents the variation in coating density and positron lifetime as a function of applied bias voltage. As the bias voltage increased, the density of the DLC coatings decreased, indicating a reduction in atomic packing. Correspondingly, the positron lifetime decreased with increasing density, suggesting a decrease in open-volume defects within the carbon network. This inverse relationship confirms that positron lifetime is sensitive to density-related defect structures in the coatings. The results highlight the strong correlation between deposition conditions, density, and defect characteristics.

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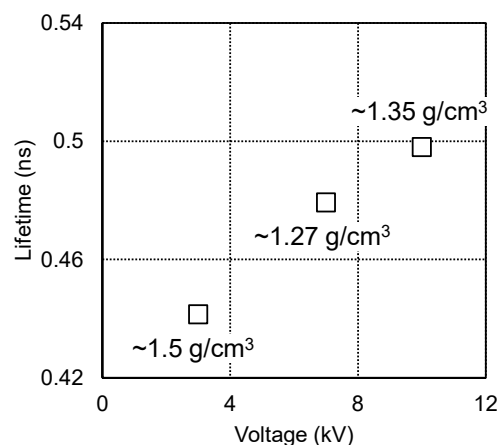


Figure 1. Coating density and positron lifetime as a function of bias voltage.

Evaluation of Irradiation Damage and the Effects of Heat Treatment on Au-Si Ion-Irradiated SiO₂/Si Substrates using Positron Annihilation Spectroscopy

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INTRODUCTION: To evaluate the Au-Si ion irradiated damage of SiO₂/Si surfaces before and after annealing, which might be important to control the formation of Au nanoparticles inside the SiO₂ layer by Au-Si ion irradiation and successive annealing, we have performed the positron annihilation spectroscopy in the present study.

EXPERIMENTS: The ions emitted from the AuSi alloyed metal ion source were irradiated on the substrates using the acceleration voltage of 7 kV at an fluence of $6 \times 10^{15} \text{ cm}^{-2}$. Then the S-parameters obtained from the Doppler broadening for the energy of the γ -ray by the positron-electron annihilation were measured before and after annealing at 1000°C for 45 or 150 min. under atmosphere.

RESULTS: Figure 1 shows the S-parameters obtained for the ion irradiated SiO₂/Si surfaces. For the initial SiO₂/Si surfaces, the S-parameters shows the small peak between the positron energy of 0-4 keV with the peak position around 2 keV, corresponding to the existence of SiO₂ layer on the Si substrate. After the ion irradiation, the S-parameters were significantly decreased in the SiO₂ layer. The reason of the decrease is not known at present, however, almost the same phenomena was reported for the B-ion implanted SiO₂ layer [1]. When the sample was annealed for 45 min., the decrease of the S-parameters was slightly recovered. After the annealing of 150 min., the S-parameters for the SiO₂ layer were almost the same values with the un-irradiated SiO₂ layer, showing the full recover of the irradiated damage in the SiO₂ layer by the annealing. However, the S-parameters for the positron energy below the 4 keV, which correspond to those for Si substrate, were lower than those of the un-irradiated and 45 min.-annealed SiO₂ layers. The results might be indicating the diffusion of the ion-induced defects into the Si substrate from the SiO₂ layer through the SiO₂/Si interface. The relation between the formation of Au nanoparticles inside the SiO₂ layer and such defects is a task for the future.

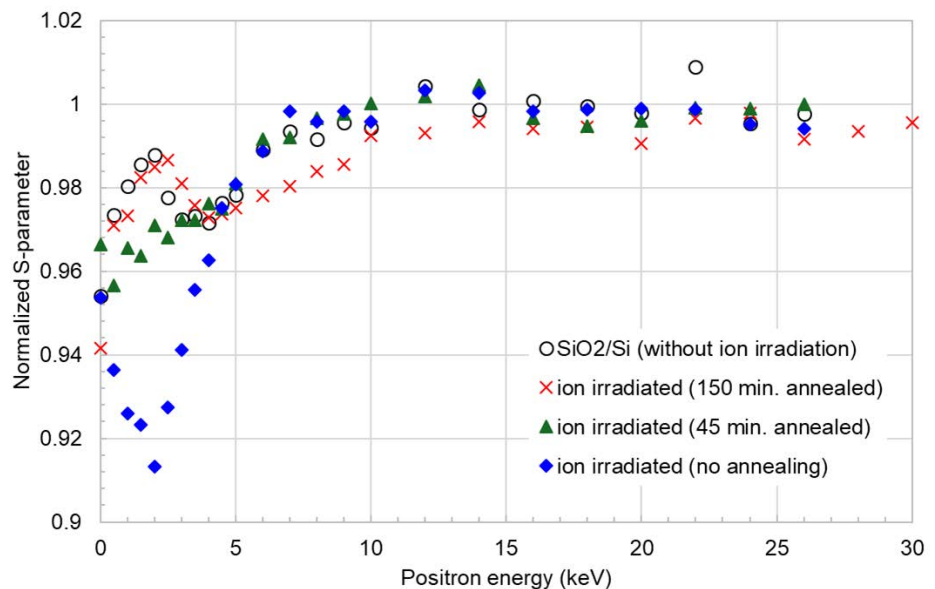


Fig. 1. S-parameters of SiO₂/Si surfaces with and without Au-Si ion irradiation before and after annealing.

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Effects of Pulse Irradiation by Charged Particles on Damage Structures in metals

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INTRODUCTION: Irradiation experiments are crucial for the development of nuclear materials. High-energy charged particles, such as ions and electrons produced in accelerators, are commonly employed in these experiments. The time structures of ion and electron beams from accelerators that use radio frequency or beam scanners are characterized by periodic/discontinuous intensities. The purpose of this study is to clarify the effects of pulse irradiation on the damage defect evolution in metals, by using ion irradiation and simulation.

EXPERIMENTS: Self-ion irradiation of Ni with the accelerating energy of 2 MeV was performed by a tandem accelerator in the Research Center of Ion Beam Technology, Hosei University. The irradiation temperature, total damage, and duty ratio are 573 K, 0.1 dpa and 0.1, respectively. The damage rate of 1 kHz pulse beam, 1 Hz pulse beam and continuous beam are 5.6×10^{-6} , 5.7×10^{-6} and 7.1×10^{-6} dpa/s, respectively. The accumulation of defects after ion irradiation was investigated by the S parameter measurements with positron annihilation spectroscopy in the slow positron beam-line at the Kyoto University Research Reactor at room temperature.

RESULTS: Reaction kinetics were employed to obtain the reaction rates among point defects and their defect clusters. Fig. 1 shows the accumulation of vacancies after 0.1 dpa self-ion irradiated Ni at 573 K with three irradiation conditions: 1 kHz (pulse duration/period = 10^{-4} s/ 10^{-3} s), 1 Hz (0.1s/1s), and continuous beams. Some differences in vacancy accumulation are evident. But the difference of 1 kHz and 1 Hz are 1 % and 5 % of the continuous beam, respectively. The penetration length of the positron varied by changing the accelerating voltage to obtain information at the irradiated area. The S parameter depends on the total number of vacant sites in Ni, including vacancies and irradiation-induced clusters. To avoid the effect of the surface, we chose the area from 300 to 800 nm for the S parameter. As shown in Fig. 2, there exist differences between unirradiated and irradiated specimens. For both simulation and experiment, the effect of 1 Hz irradiation is the highest. But the difference between 1 kHz and 1 Hz irradiation is small in S-parameters compared to the simulation. They are within 0.6 % of the continuous beam. One of the reasons for the difference is that only the residual vacancy concentration obtained by the simulation is used for the comparison. The effects of interstitial type dislocation loops are not introduced. Loops are edge dislocations with a Burgers vector of $1/3[111]$. There exists a dilatational field on one side of the dislocation lines. The detailed comparison with the experiment will require accounting for the effects of loops.

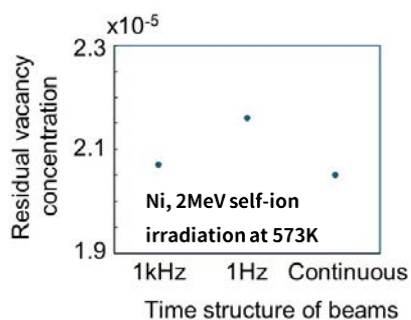


Fig. 1. Simulation of the accumulation of residual vacancies.

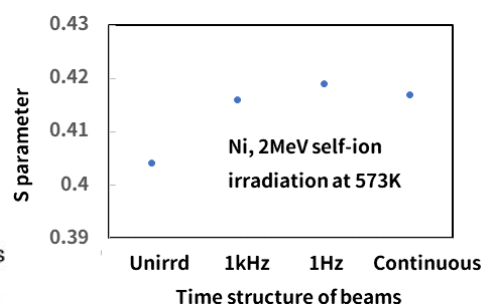


Fig. 2. S-parameter of positron annihilation spectroscopy.

Development of slow positron beamline using electron linear accelerator

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INTRODUCTION: Slow positron beams (energy-variable monoenergetic positron beams) have been widely used to investigate vacancy-type defects in thin films or near-surface layers in ion irradiated materials. Intense slow positron beams can be generated through pair-creation reactions by large-scale experimental facilities such as nuclear reactors and accelerators. In terms of accelerator-based positron sources, so far, radiofrequency-driven electron linear accelerators (LINAC) have been mainly used. In principle, LINAC-generated positron beams are not continuous (i.e., pulse beams), while reactor-based positron beams are continuous. The pulse beams can be potentially applied for unique applications such as pump-probe type measurements [1]. This study aims at such unique applications using KURNS-LINAC.

EXPERIMENTS: A 30 MeV electron beam was used to generate slow positrons by the KURNS-LINAC. A linear storage section was installed to control time structures of the slow positron beam. An original pulse width of 5 μ s can be stretched or delayed by controlling the waveforms applied the entrance and exit electrodes in the linear storage section. The beamline was extended to a sample chamber at a measurement room next to an accelerator target room. The annihilation gamma-rays at a sample holder were detected by a scintillation detector for positron lifetime spectroscopy and a Ge detector for Doppler broadening measurements, respectively.[2]

RESULTS: The narrow pulse width of original positrons cannot be used for the Ge detector, as it saturates the detector operation. Thus, the linear storage section is required for the Doppler broadening measurements. Fig. 1(a) shows the gamma-ray spectrum obtained from a reference sample (Kapton). A 511 keV peak originating from positron annihilation were observed. In addition, three intense peaks can be observed in the range of 1000 – 1500 keV. These peaks are attributed the background gamma-rays from Co-60 and K-40. Such signals increase unwanted background levels around the 511 keV peak. Thus, a gate signal synchronizing with positron release from the linear storage section was applied to a multichannel analyzer used to measure gamma-ray spectra. Fig. 1(b) shows the spectrum with the gate signal. The background signals were successfully reduced. Our experimental results showed that positron pulse widths can be effectively controlled by operating the linear storage section for accelerator-driven original positron pulses.

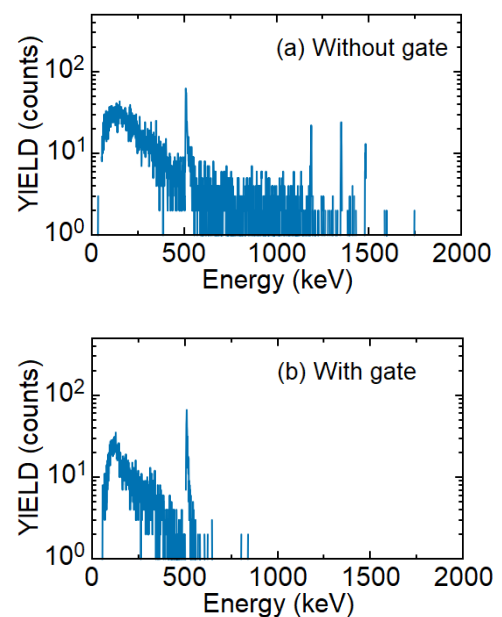


Fig. 1 Gamma-ray spectra obtained with and without gate signals.

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I-1. PROJECT RESEARCHES

Project 7

Chemical and electronic properties of Actinide compounds and their applications

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INTRODUCTION: Actinides exhibit characteristic chemical and electronic properties originating from 5f electrons, and their chemistry is important for nuclear fuel-cycle technology, separation science, radiochemistry, and medical-nuclide production. This project integrates actinide separation chemistry, data-driven extractant design, decay-product separation, and actinyl-ion coordination chemistry by using the hot-laboratory infrastructure of KURNS. In the present period, three submitted studies were summarized: machine-learning-assisted evaluation of extractants for minor actinide separation, Ra-228 recovery from Th resources for Ac-225 production, and bifunctional monoamide-diamide resins for selective adsorption of actinyl ions.

EXPERIMENTS: URhSi and URhGe single crystals were prepared by arc melting and examined by single-crystal X-ray diffraction. The OECD/NEA IDEaL database was linked to regression workflows with clustering of DGA, BTP, CMPO and related extractants. La extraction by D₂EHFA/n-dodecane was measured with ADA buffers as a model for Ac chemistry. Precise ¹⁰¹Ru-NQR measurements of URu₂Si₂ were continued under uniaxial [100] stress. For Ac-225 production, 6.0 g ThO₂ was processed using PVPP in 8 M HNO₃ to recover Ra-228 and recycle Th. Silica-supported DMAA-TMMA resin was synthesized and tested for U(VI), Ce(III) and Th(IV) adsorption in nitric acid.

RESULTS: URhSi and URhGe were confirmed to have TiNiSi-type structures, but Rh and Si/Ge sites are interchanged and the Rh-Si network in URhSi is strongly deformed, implying different 5f ground states and interlayer coupling. Class-specific machine-learning regressions gave higher accuracy than whole-dataset models, showing that extractant class and descriptors carry chemical significance. In D₂EHFA extraction, high distribution ratios were accompanied by pH decrease due to proton release, and ADA did not sufficiently suppress the pH shift. Ru-NQR under 0.23 GPa [100] stress showed no linewidth anomaly around the 18 K hidden-order transition, indicating no magnetic moment appears under this condition. PVPP adsorbed Th with a capacity of 0.140 g-Th/g-PVPP and recovered 96.6% of Ra-228 from a Th-Ra mixed solution; residual Th was reconverted to porous ThO₂. DMAA-TMMA and DMAA-TPMA resins enhanced U(VI) adsorption over a monoamide resin, with higher U(VI) than Ce(III) uptake and high Th(IV) affinity for DMAA-TMMA.

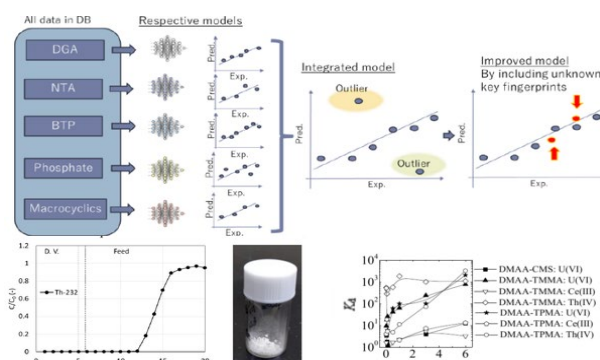


Fig. 1. Representative results: machine-learning workflow for MA extractants (top), PVPP-based Th/Ra separation and reconverted ThO₂ (bottom left), and adsorption behavior of monoamide-diamide resins toward U(VI), Ce(III), and Th(IV) in nitric acid (bottom right).

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Crystal Structure and Characterization of Actinide Intermetallic Compounds

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INTRODUCTION: Strongly correlated electrons in actinide compounds are the origin of various phenomena such as heavy fermion formation and exotic superconductivity coexisting with magnetic orderings. These phenomena are based on specific ground states of 5f electrons located in the crystal lattice. It is therefore critically important to determine atomic arrangements around the uranium site of interest. In this study, we focus on intermetallic uranium compounds showing ferromagnetic superconductivity. URhSi and URhGe are known to crystallize in the orthorhombic TiNiSi-type structure. Despite the very similar ferromagnetic properties, only the latter shows superconductivity.[1,2] In this study we reinvestigate the crystal structure of these compounds to precisely characterize the local atomic arrangements.

EXPERIMENTS: The samples were synthesized by arc-melting starting materials under an argon gas atmosphere. Small single crystals suitable for single-crystal X-ray diffraction were extracted from the arc-melted ingot. X-ray diffraction was measured using monochromated Mo K α radiation with an imaging plate detector.

RESULTS: We confirmed that URhSi and URhGe crystallize in the TiNiSi-type structure. Overall structural characteristics can be understood as follows: TiNiSi-type structure is derived from the ordered hexagonal AlB₂-type (known as ZrBeSi-type) structure. Here, uranium occupies the triangular site, while Rh and Si(Ge) form a honeycomb-like network. Indeed, these characteristics are well preserved in URhSi and URhGe. However, surprisingly, Rh and Si(Ge) sites are swapped with each other. Furthermore, Rh-Si network is strongly deformed in URhSi. As a result, the layered structure is no longer maintained, and interlayer coupling becomes significant. Note that all the existing experimental study on the crystal structure of these compounds consistently reported similar structural parameters for these compounds. The present results strongly suggest that the 5f ground state under the crystal field in URhSi and URhGe is different from one another. Moreover, the interlayer coupling in URhSi suggests modification on the conduction band. The ferromagnetism of these compounds might therefore have different origins. Further investigations on the evolution of the ferromagnetic interaction in the URh(Si,Ge) alloy are in progress.

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Development and Validation of a Machine Learning Scheme for Actinide Separation Chemistry

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INTRODUCTION: Minor actinides in spent nuclear fuel pose major heat and toxicity challenges despite their small quantities, motivating efforts to separate them from rare earth elements to reduce repository burden. While selective extractants are crucial, their compatibility with organic solvents strongly affects performance, making solvent choice equally important. This study combines systematic extraction experiments with chemoinformatics and machine learning to accelerate solvent and extractant screening, focusing especially on fluorinated solvents. Molecular features are mapped into Hansen solubility space to predict solubility and extraction behavior. To address the greater complexity of extractant design, the work builds data-driven models using curated databases and clustering to compensate for sparse data. Rather than inventing new ligands, the strategy optimizes side chains of established extractant frameworks such as DGA, BTP, and CMPO as shown in Fig. 1. Results indicate that medium-length straight alkyl chains favor fluorinated solvents and that tuning with co-components can further enhance performance. Overall, the study advances toward a unified predictive model for americium extraction by integrating chemical insight with machine learning.

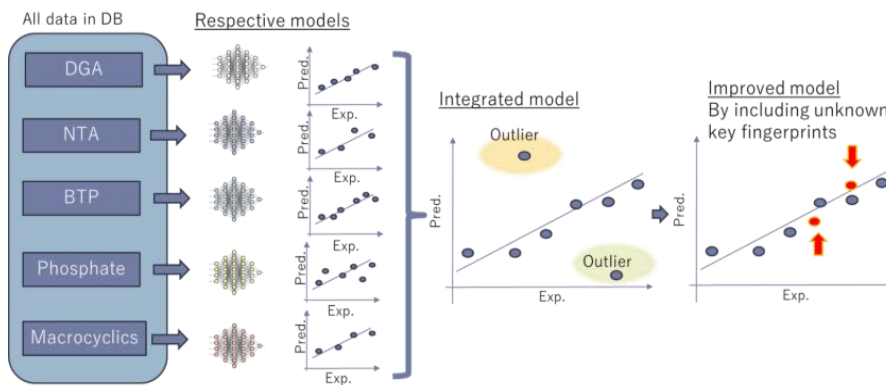


Fig. 1. Approach to identify the model to find a candidate MA extractant

RESULTS: The OECD/NEA IDEaL database was connected to machine learning workflows and performed a variety of regression analyses on extractant performance (Fig. 2). By clustering extractants into several groups and conducting regressions separately for each group while varying kernel types, we found that group-specific models achieve higher accuracy than models trained on the entire dataset. This indicates that extractant class and descriptor choice strongly influence regression performance and may reflect underlying chemical significance. Moreover, even extractants that are poorly fitted by regression—or appear as outliers—can provide valuable insights, suggesting opportunities to discover new extractants and identify key molecular features beyond conventional model predictions.

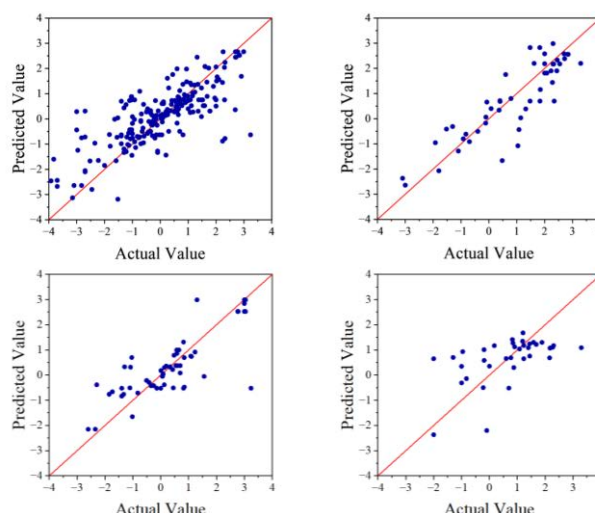


Fig.2 Result of regression with respective clusters: (1) all, (2)DGA, (3)BTP, and (4)CMPO

Solvent extraction of ^{228}Ac by D_2EHPA under pH range.

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INTRODUCTION: Actinium is the earliest element in the actinide series, which has 29 isotopes excluding metastable states, and is considered similar to lanthanum in chemical properties. In terms of oxidation state, actinium has only trivalent state like the elements after americium in the actinide series. Most isotopes of actinium are produced by nuclear reactions and have short half-lives of a few seconds to a few minutes. ^{225}Ac , one radioisotope of actinium, is used in targeted alpha therapy and has been reported to be highly effective to the cancer therapy, especially for prostate cancer [1]. ^{228}Ac , one of the thorium series, reaches radiolytic equilibrium with the parent nuclide ^{228}Ra in about 30 hours. Therefore, once separated, the same amount of ^{228}Ac can be collected again [2]. ^{228}Ac emits photons over a wide energy range and its emission rate is very high. With these properties, ^{228}Ac is expected to facilitate basic research on actinium, such as chelate chemistry, which is also required for labeling. Previously, the extraction behavior of ^{228}Ac and La by D_2EHPA in the pH range is evaluated using shaking time, extractant concentration dependence, and pH as indicators for study basic coordination chemistry of actinium.

In this study, the ADA buffer concentration dependence has been investigated using La for the suitable extraction condition investigation.

EXPERIMENTS: The buffer ADA is used as the aqueous phase, and n-dodecane (nDD), one of hydrocarbonate solvents with properties such as nonflammability, chemical stability and low viscosity, were used as the organic phase. Initially, La was dissolved in the aqueous phase. Under the various ADA concentration condition, La in the aqueous phase was extracted into the organic phase by complexation with D_2EHPA . The distribution ratio was used as an index to evaluate how much La in the aqueous phase was extracted into the organic phase.

RESULTS: In regions where the D_2EHPA concentration is high—that is, where the partition coefficient is large—the pH decreases (Fig. 1). This is thought to be due to the effect of proton release by D_2EHPA . Proton release resulting from complexation between D_2EHPA and La causes the pH to decrease. The problem is that the pH decrease cannot be suppressed due to the buffer effect of ADA.

This suggests that ADA is hardly dissociated into protons, meaning that its buffer function is not being fully exerted.

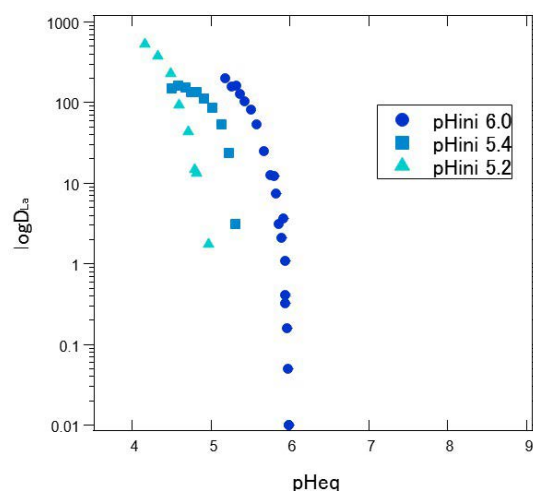


Fig. 1. pH Dependence of the Distribution Ratio D_{La} at Equilibrium

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Study of Heavy Fermion Superconductor URu₂Si₂ NQR/NMR under uni-axial stress

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INTRODUCTION: Understanding of "Hidden ordering" in URu₂Si₂ is fairly progressed recently [1], whereas the definitive answer is still missing. This issue is very exciting since the hidden ordering is supposed to be new spontaneous symmetry breaking. Considering the recent experimental

results, a few possible space groups of hidden order symmetry have been selected previously [2,3]. If the hidden order symmetry is once determined, the corresponding multipolar order parameter is spontaneously determined.

EXPERIMENTS: In this study, the local symmetry of Ru site is determined in the hidden order state without uni-axial stress and with uni-axial stress along the [100] and [110] directions [4], by means of precise Ru-NQR measurements. These measurements again support the 4-fold symmetry at Ru site in the hidden order state without uni-axial stress. Furthermore, certain cases can be excluded from the previously proposed possible ones. In this year, the Ru-NQR measurements under uni-axial stress [100] direction are continued.

RESULTS: Fig. 1 shows the T-dependence of ¹⁰¹Ru NQR linewidth W_{1Q} at zero field under uni-axial stress σ along [100] direction of 0.23GPa. As ¹⁰¹Ru is $I=5/2$ nuclei, there are two NQR peaks are observed *i.e.* ν_{1Q} ($I = \pm 3/2 \leftrightarrow I = \pm 1/2$) and ν_{2Q} ($I = 5/2 \leftrightarrow I = \pm 3/2$). In the present case, the linewidth for ν_{1Q} results are presented. The T-dependence of W_{1Q} shows no anomaly at the hidden ordering around 18K in the case under [100] stress. This indicates that no magnetic moment appears at the hidden order phase transition under [100] stress.

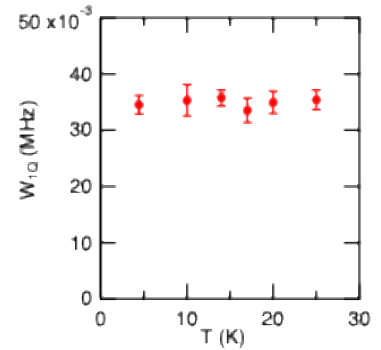


Fig. 1. T-dependence of ¹⁰¹Ru NQR linewidth W_{1Q} at zero field under uni-axial stress $\sigma[100]=0.23$ GPa.

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Fundamental Study on Extraction/Separation of Actinides and Their Decay Products for Medical Nuclide Production

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INTRODUCTION: Targeted alpha therapy using Ac-225 has been shown to be effective in treating cancer, and research using Ac-225 is currently being actively conducted. To improve the production capacity of Ac-225, a method has been proposed that utilizes naturally abundant Th-232 to generate Ra-229 via the (n,γ) reaction of Ra-228 using thermal neutrons, and then produces Ac-225 through the decay of Ra-229. However, the amount of Ra-228, a decay product of Th-232, present in natural ThO₂ is extremely limited. Therefore, it is necessary to efficiently separate this trace amount of Ra-228 from the large quantities of Th-232. In previous studies, a method was developed to recover the non-adsorbed Ra-228 by adsorbing Th-232 using polyvinylpyrrolidone (PVPP) [1]. In this study, the effectiveness of Ra-228 separation using PVPP was evaluated by conducting a series of processes from ThO₂ conversion to Ra-228 separation. Furthermore, a fundamental study on reconversion of the used Th into a porous oxide form to facilitate the subsequent re-separation of Ra-228 after equilibrium was performed to achieve more efficient recovery of Ra-228 from Th resources.

EXPERIMENTS: The primary conditions for this series of processes, such as reaction temperature and concentration of nitric acid, are the same as those in previous experiments [1-3]. However, due to the increased amount of ThO₂, certain conditions, such as the amount of adsorbent, have been modified accordingly. In this study, 6.0 g of ThO₂ was treated to evaluate Ra-228 separation using PVPP. Since previous Th adsorption experiments on PVPP were performed using trace amounts of Th-232, a breakthrough experiment of Th on PVPP was conducted to determine the adsorption capacity. The feed solution consisted of 20 mL of 8 M HNO₃ solution in which 2.0 g of thorium nitrate was dissolved. The bed height of PVPP was 93 mm (1.8 g) and the flow rate was set to 0.25 mL/min. After the separation of Ra-228, Th in solution was recovered as thorium oxalate using 0.05 M oxalic acid and then reconverted to a porous oxide form by heating at 630°C for 2 h.

RESULTS: Fig. 1 shows the breakthrough curve of Th-232 on PVPP. The Th adsorption capacity on PVPP under the present conditions was determined to be 0.140 g-Th/g-PVPP. Based on this result, the column separation conditions were optimized, resulting in a recovery ratio of 96.6% for Ra-228 from a Th-Ra mixed solution using PVPP. As described above, the effectiveness of Ra-228 separation using PVPP in the target fabrication process was demonstrated. Moreover, the conversion of Th remaining after the separation of Ra-228 into a porous oxide form was successfully achieved as shown in Fig. 2.

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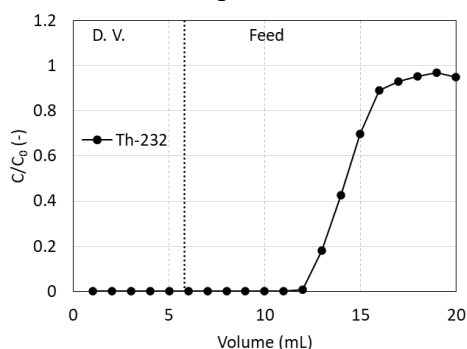


Fig. 1. Breakthrough curve of Th-232 on PVPP.



Fig. 2. ThO₂ (reconverted).

Effect of Chain Length of Bifunctional Monoamide-Diamide Resin on Adsorptivity to Actinyl Ions

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INTRODUCTION: Development of highly selective compounds for hexavalent actinide species (actinyl ions, An(VI), AnO_2^{2+}) has been important. We have been focusing on monoamide compounds which coordinate with An(IV) and (VI) species in nitric acid media. It is known that diamide compounds are used for separating not only An(IV) and (VI) species but also An(III) and lanthanide(III) (Ln(III)) ones. To increase the possibility of coordination of carbonyl oxygen atoms with an AnO_2^{2+} ion from the spatial viewpoint, bifunctional resins consisting of both monoamide and diamide compounds have recently been synthesized. As a result, adsorptivity to U(VI) was found to become improved compared with that of resins consisting of exclusive monoamide structure as expected[1]. In this study, effect of the chain length of diamide structure in the bifunctional resin on the adsorptivity was investigated.

EXPERIMENTS: In the previous study, *N,N*-dimethylacrylamide (DMAA) and *N,N,N',N'*-tetrapentylmalonamide (TPMA) were used as monoamide and diamide, respectively. In this study, *N,N,N',N'*-tetramethylmalonamide (TMMA) were used instead of TPMA (Fig. 1). The bifunctional resin was synthesized similarly to the previous study using two steps, where chlorine atoms of polychloromethylstyrene (CMS) were substituted by TMMA [1], and the final silica-supported resin (Silica DMAA-TMMA) was obtained. Adsorptivity of the resin was examined by a batch method using U(VI), Ce(III), and Th(IV) up to 6 mol/dm³ (= M) HNO_3 at room temperature.

RESULTS: Dependence of K_d values of each resin for U(VI), Ce(III), and Th(IV) (representative of An(IV)) on HNO_3 concentration is shown in Fig. 2. The values of DMAA-TMMA and DMAA-TPMA for U(VI) are basically identical, increasing with increasing concentration of HNO_3 . The adsorptivity of both resins are significantly improved compared with the monoamide resin (DMAA-CMC). Besides, the K_d values for U(VI) are higher than for Ce(III), suggesting that, as expected, bifunctional monoamide-diamide resins may improve adsorptivity to An(VI) by suppressing the adsorption for Ln(III). On the other hand, it was also clarified that DMAA-TMMA have higher selectivity for Th(IV).

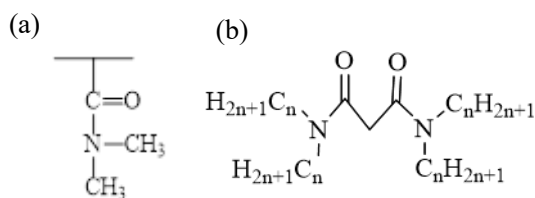


Fig. 1. Chemical structures of (a) DMAA and (b) diamides (TMMA (n=1), TPMA (n=5)).

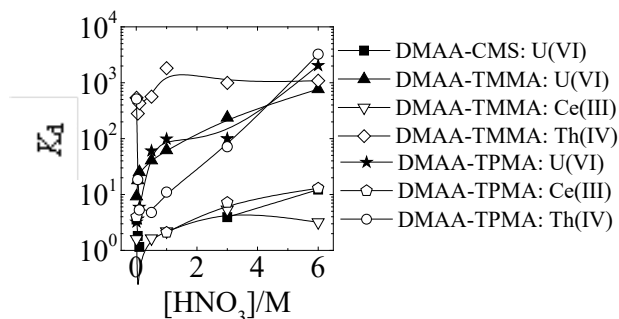


Fig. 2. Adsorptivity of monoamide-diamide resins to U(VI), Ce(III), and Th(IV) in HNO_3 .

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I-1. PROJECT RESEARCHES

Project 8

Production of medical RI by reactor irradiation

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INTRODUCTION: The domestic production of medical radioisotopes (RIs) is increasingly important for cancer radiotheranostics because Japan still relies on limited overseas supplies of therapeutic radionuclides. This project integrates reactor irradiation, radiochemical separation and radio-pharmaceutical development for ¹⁷⁷Lu, ¹⁶¹Tb and ²²⁵Ac, with consideration of KUR-based studies and future JRR-3 production. In this period, work focused on real-time ¹⁷⁷Lu/Yb separation, scale-up no-carrier-added ¹⁷⁷Lu production, preliminary ¹⁶¹Tb production from natural Gd, JRR-3 Lu-177 production trials, ¹⁷⁷Lu⁻ and ²²⁵Ac-labeled PSMA agents, LAT1-targeted metal RI probes, and feasibility testing of an alternative ²²⁵Ac route via Ra-228(n,γ) irradiation.

EXPERIMENTS:

Step	Key conditions	Purpose
¹⁷⁷ Lu/Yb separation	Extraction chromatography with on-line gamma monitoring using CZT or Ge detectors.	Optimize rapid n.c.a. ¹⁷⁷ Lu separation.
Scale-up ¹⁷⁷ Lu production	Long-term irradiation of enriched ¹⁷⁶ Yb at KUR and LN2-resin column separation.	Define automated separation requirements.
JRR-3 Lu-177 production	Direct [¹⁷⁶ Lu]Lu ₂ O ₃ and indirect [¹⁷⁶ Yb]Yb ₂ O ₃ targets sealed in quartz ampoules and irradiated in HR-1 at JRR-3.	Evaluate JRR-3 capability and issues for test-scale ¹⁷⁷ Lu supply.
¹⁶¹ Tb production	Neutron irradiation of natural Gd followed by Gd/Tb separation using LN2 resin.	Assess domestic ¹⁶¹ Tb feasibility.
Radiopharmaceuticals	¹⁷⁷ Lu-PSMA-617 and ²²⁵ Ac-PSMA-617 synthesis and mouse bio-distribution.	Evaluate preclinical RI usability.
Probe and ²²⁵ Ac studies	LAT1-targeted chelator compounds and Ra-228-supported MgO irradiation targets.	Expand agents and ²²⁵ Ac routes.

RESULTS AND DISCUSSIONS: On-line γ-ray monitoring distinguished Yb-related peaks from ¹⁷⁷Lu during extraction chromatography. In scale-up testing, long-term irradiation yielded ¹⁷⁷Lu with a specific activity of ca. 1.0 x 10⁷ Bq/mg, about three times higher than the previous hydro-irradiation condition; most Yb was removed with 2.25 M HNO₃ and ¹⁷⁷Lu was eluted with 4 M HNO₃ after ca. 660 min. At JRR-3, irradiation of 1 mg Lu in HR-1 for 14 d was estimated to produce ca. 400 GBq of ¹⁷⁷Lu, while irradiation of 1 mg Yb produced ca. 500 MBq; although specific activity and target re-irradiation remain issues, the quantity and quality were sufficient for labeling and non-clinical tests. Natural Gd irradiation and LN2-resin separation gave distinct Gd/Tb elution profiles, and Ge-detector analysis confirmed separation of ¹⁵³Gd, ¹⁵⁹Gd and ¹⁶¹Tb, indicating that ¹⁶¹Tb suitable for radiopharmaceutical production and animal pharmacokinetic studies can be prepared without enriched ¹⁶⁰Gd. ¹⁷⁷Lu-PSMA-617 was obtained in nearly quantitative yield, whereas ²²⁵Ac-PSMA-617 was obtained in 64% yield; mouse biodistribution showed high renal uptake with additional liver and bone accumulation, requiring further optimization of ²²⁵Ac/daughter-nuclide behavior. LAT1-targeted tyrosine-based chelator compounds were synthesized. Ra-228-supported MgO targets were irradiated safely at KUR, and further purification and long-term α-spectrometry are planned to verify Th-229 production.

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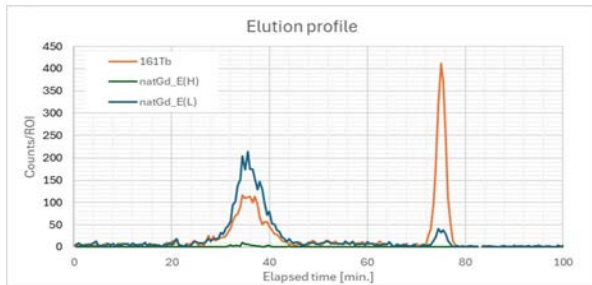


Figure 1. Elution profile of extraction chromatography with an 11 mmφ × 240 mmL column packed with LN2 resin measured with a CZT detector.

Development of Radiotheranostic Agents Utilizing a Variety of Radioisotopes

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INTRODUCTION: The domestic demand for therapeutic radioisotopes (RIs) in nuclear medicine is increasing; however, the supply remains largely dependent on unstable imports. This highlights the need to promote domestic production of medical RIs and to establish a comprehensive framework for nuclear medicine diagnosis and therapy. In this study, ¹⁷⁷Lu produced at the Kyoto University Reactor (KUR) was used to synthesize established radiopharmaceuticals, including ¹⁷⁷Lu-PSMA-617. Cytotoxicity and therapeutic efficacy were evaluated in tumor-bearing mouse models, and the quality of the produced ¹⁷⁷Lu was assessed through comparison with previously reported data. In addition, ²²⁵Ac obtained from a ²²⁹Th/²²⁵Ra–²²⁵Ac generator was used to synthesize ²²⁵Ac-PSMA-617. These radiopharmaceuticals were similarly evaluated for cytotoxicity and therapeutic efficacy in tumor-bearing mouse models, and the quality of the produced ²²⁵Ac was assessed by comparison with the literature.

EXPERIMENTS: Radiolabeling of PSMA-617 with ¹⁷⁷Lu and ²²⁵Ac: To 10 µL of the PSMA-617 precursor (2 mg/mL in 0.1 M acetate buffer), 10 µL of L-ascorbic acid (100 mg/mL), 140 µL of 0.1 M acetate buffer (pH 5.8), and either ¹⁷⁷LuCl₃ (9.15 MBq, 10 µL) or ²²⁵AcCl₃ (0.25 MBq, 20 µL) were added to obtain a final volume of 180 µL. The reaction mixture was then incubated in an aluminum block heater at 90 °C for 30 min for ¹⁷⁷Lu labeling, or at 95 °C for 15 min for ²²⁵Ac labeling. Biodistribution study in Slc:ddY mice: Each mouse was intravenously injected via the tail vein with either ²²⁵Ac-PSMA-617 (62.5 kBq/100 µL) or ¹⁷⁷Lu-PSMA-617 (37 kBq/100 µL). At 1 h post-injection, the mice were euthanized, and major organs were harvested and weighed. The radioactivity in each organ was then measured using a γ-counter, and the percentage of injected dose per gram of tissue (%ID/g) was calculated.

RESULTS: ¹⁷⁷Lu-PSMA-617 was obtained in nearly quantitative radiochemical yield. In contrast, the radiochemical yield of ²²⁵Ac-PSMA-617 was 64%. In this study, only a limited amount of ²²⁵Ac was available, which precluded sufficient optimization of the labeling conditions. Further evaluation, including assessment of reproducibility, remains to be addressed in future work.

In biodistribution studies using Slc:ddY mice, ²²⁵Ac-PSMA-617 showed high uptake in the kidneys, with additional accumulation in the liver and bone. Comparison with ¹⁷⁷Lu-PSMA-617 revealed no major differences in overall organ distribution; however, higher uptake of ²²⁵Ac-PSMA-617 was observed in the liver and bone. Previous reports have also shown that Ac³⁺ tends to accumulate in the liver and bone. For ²²⁵Ac, nuclear recoil associated with radioactive decay is a key challenge. During α-decay, a high-energy α particle (approximately 5–8 MeV, ⁴He nucleus) is emitted, resulting in recoil of the daughter nuclide in the opposite direction. The energy involved greatly exceeds chemical bond energies, potentially leading to disruption of the chelator complex and release of daughter nuclides. These may redistribute locally or migrate via blood or lymphatic circulation, contributing to off-target radiation exposure. In particular, as ²²⁵Ac undergoes multiple α-decay steps, control of daughter nuclide behavior is critical for therapeutic applications, making in vivo stability a key design consideration. In this study, limited reproducibility in animal experiments prevents definitive attribution of the observed accumulation to free ²²⁵Ac or its daughter nuclides. Further investigation is required. Nevertheless, comparison with previously reported data suggests that the ¹⁷⁷Lu and ²²⁵Ac used in this study are of sufficient quality for preclinical applications.

Preliminary Study on the Production of ^{161}Tb Using $^{\text{nat}}\text{Gd}$ and Radiochemical Separation at the Kyoto University Research Reactor

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INTRODUCTION: Terbium-161, ^{161}Tb (half-life: 6.95 days) is a β^- -emitting radionuclide that is produced indirectly during reactor irradiation via the $^{160}\text{Gd}(n,\gamma)^{161}\text{Gd}$ reaction, followed by the β^- decay of ^{161}Gd (half-life: 3.66 minutes). The half-life and decay characteristics of ^{161}Tb are similar to those of therapeutic radionuclide ^{177}Lu ; however, ^{161}Tb emits a significantly higher number of internal conversion electrons and Auger electrons compared to ^{177}Lu , suggesting superior therapeutic efficacy from a theoretical standpoint. Its effectiveness has been demonstrated in animal studies and clinical trials conducted overseas [1]. In Japan, however, ^{161}Tb remains difficult to obtain, and there are no reported cases of its production. In this study, fundamental investigations on reactor-based production were conducted using natural gadolinium containing 21.86% ^{160}Gd , focusing on production characteristics and chemical separation behavior.

EXPERIMENTAL: The gadolinium (Gd) samples used for irradiation were prepared from an ICP-MS elemental standard with natural isotopic abundance. A solution containing an amount equivalent to 1.0 mg of Gd was transferred into a quartz tube, concentrated to dryness by heating, and then vacuum-sealed. The quartz tube was loaded into a hydraulic rabbit system of the research reactor at the Kyoto University Institute for Integrated Radiation and Nuclear Science and irradiated for 47 hours at a thermal power of 1 MW. The irradiated Gd target was dissolved in 6 M HNO_3 four days after irradiation and adjusted to 0.25 M HNO_3 . For the chemical separation of Gd and Tb, the extraction chromatography method developed by Źółtowska et al. [2] was used. Column separation was carried out using LN2 resin (Eichrom). A jacketed glass column (11 mm i.d. $\times$ 240 mm length, Kiriya Glass) was used for the separation. The eluate from the column was monitored online using a CZT detector (RadAngel, Kromek).

For the separation of Gd and ^{161}Tb , regions of interest (ROI) were set at 97.4 and 103.2 keV for ^{153}Gd , and 363.54 keV for ^{159}Gd used as a tracer for Gd, and at 74.6 keV for ^{161}Tb . Fractions of Gd and Tb were collected based on elution curves expressed as count rates per unit time for each ROI. Gd was first eluted with 0.2 M HNO_3 , and after confirming its elution, ^{161}Tb was subsequently eluted with 0.45 M HNO_3 . The collected fractions were further trapped on separately prepared DGA cartridges, and qualitative analysis of the Gd and Tb fractions was performed using a Ge detector (ORTEC).

RESULTS: The elution curves of Gd and Tb obtained using the CZT detector showed distinct profiles for each element (Fig. 1). However, it was found that Compton scattering components originating from ^{153}Gd contributed to the ROI of ^{161}Tb . Therefore, careful attention is required when determining the timing of fraction collection. At the same time, this issue can be avoided by confirming the γ -ray spectra measured simultaneously, thereby preventing misidentification. Qualitative analysis of the separated Gd and Tb fractions using a Ge detector confirmed that ^{153}Gd ($T_{1/2} = 240.4$ d), produced via the $^{154}\text{Gd}(n,2n)$ reaction, ^{159}Gd , produced via the $^{158}\text{Gd}(n,\gamma)$ reaction, and ^{161}Tb were successfully separated in the irradiated samples. Since the half-life of ^{159}Gd is 18.5 hours, its presence can be easily confirmed even one week after irradiation. Furthermore, its relatively high γ -ray energy (363 keV) makes it easy to detect, facilitating the identification of the Gd fraction.

CONCLUSION: In this study, we evaluated the production and radiochemical separation of ^{161}Tb . Our results indicate that it is possible to produce and purify ^{161}Tb of sufficient quality for radiopharmaceutical production and pharmacokinetic studies in animal models without the need for isotopically enriched ^{160}Gd , suggesting the feasibility of using natural Gd as a target material.

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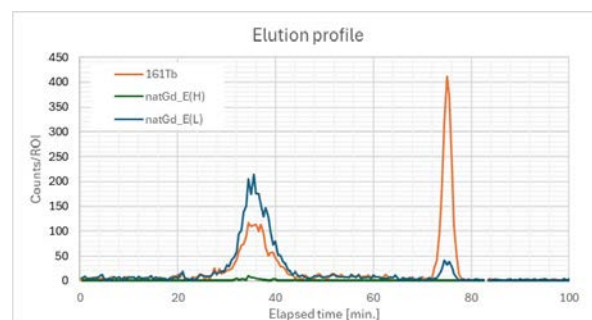


Figure 1. Elution profile of extraction chromatography with an 11 mm ϕ $\times$ 240 mmL column packed with LN2 resin measured with a CZT detector.

On-line monitoring of $^{177}\text{Lu}/\text{Yb}$ separation by extraction chromatography

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INTRODUCTION: ^{177}Lu (half-life: 6.64 d), produced indirectly by neutron irradiation of ^{176}Yb , is a β^- emitter and one of the applicable nuclides for targeted radioisotope therapy (TRT). To date, ^{177}Lu -DOTA-TATE for the treatment of somatostatin-overexpressing tumors, and ^{177}Lu -PSMA-617 for the treatment of castration-resistant prostate cancer, have been investigated for TRT use.

In extraction chromatography, bis-2-ethylhexylphosphonic acid and 2-ethylhexylphosphonic acid are well known as substrate adsorbed extractants for ^{177}Lu separation, and recently some extractants have explored. In these studies, typically the concentrations of Yb and Lu in the eluate are determined by radiometric or elemental concentration measurements after collecting a certain volume of solution that has passed through the column. However, some technique that allows for faster analysis is desirable for the best condition survey.

In this study, we developed a real-time monitoring technique for ^{177}Lu and radioactive Yb using the on-line γ -ray detector (CZT detector or Ge semiconductor detector) in the separation of ^{177}Lu from neutron irradiated Yb targets by extraction chromatography.

EXPERIMENTS: The experiments were conducted using the on-line monitoring separation system previously reported in detail [1].

RESULTS: Fig. 1(a) shows an overview of the Lu/Yb separation experiment using enriched ^{176}Yb : the first horizontal axis is the elapsed time, the second is the γ -ray energy, and the vertical axis is the logarithm of the intensity (count rate). This figure shows that the peak energies and their intensities in the γ -ray spectra change with time. To examine in detail, the type of nuclide elution taking place at each stage,

the γ -ray energy is evaluated in relation to the elapsed time (Fig. 1(b)). Focusing on the γ -ray energy range above 100 keV, high intensities were detected at around 110, 131, 177, 198, 282, 308, and 396 keV for times between 300 and 400 min. These γ -ray peaks are attributed to ^{169}Yb and ^{175}Yb . In the region around 510 min, high intensities were detected at 110 keV as well as 208 keV. Especially at the latter γ -ray energy, the intensity was confirmed only in this region. Since these can be attributed to ^{177}Lu (113 keV and 208 keV), it can be assumed that most of the Yb is leached out by passing 1.5 M HNO_3 , and the remaining resin residue, composed mainly of Lu, is leached out by 4 M HNO_3 after 500 min.

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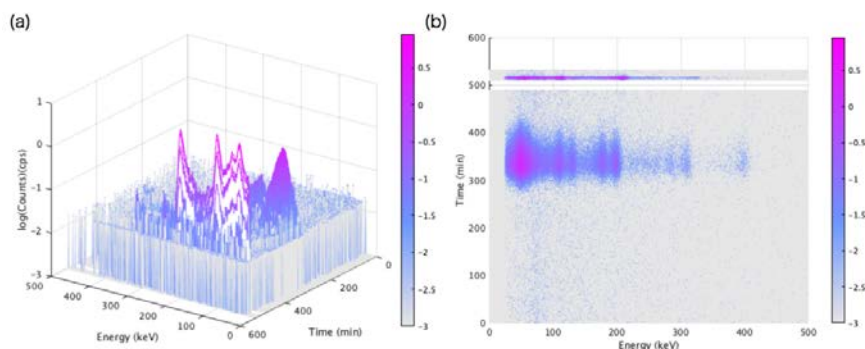


Fig. 1. Overview of the Lu/Yb separation experiment using enriched ^{176}Yb (a) and γ -ray energies relation to time (b).

Fundamental Research on the Production of Lu-177

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INTRODUCTION: Radioisotopes are widely utilized in Japan as diagnostic and therapeutic agents. However, Japan relies on imports for key nuclides such as Mo-99, which are produced in nuclear reactors overseas. This reliance raises concern that international circumstances could disrupt domestic supply and impede drug development. Therefore, JAEA has undertaken radioisotope production trials using the JRR-3 reactor, which resumed operation at the end of February 2021. Among these efforts, particular focuses are made on production trials for Lu-177, as detailed below.

EXPERIMENTS: For the Lu-177 production test at JRR-3, [176Lu] Lu₂O₃ (enriched to 64.3%) for the direct method and [176Yb] Yb₂O₃ (enriched to 99.9%) for the indirect method was used as the irradiation target.

Solutions of the target materials (1 M HNO₃) were weighed and dispensed into quartz ampoules to have an irradiation target element of approximately 0.1 mg. The solutions were then evaporated to dryness and sealed to prepare the irradiation samples. These samples were placed in irradiation capsules, pressure welded and irradiated in HR-1, which is neutron irradiation facility in heavy water tank of JRR-3.

RESULTS: The result shows that about 400 GBq of Lu-177 can be produced by irradiating 1 mg of Lu in HR-1 for 14 days. However, since the specific activity is lower than that of Lu-177 distributed in Japan it is difficult to immediately use the current product as a pharmaceutical raw material, but it was confirmed that JRR-3 can produce radioisotopes of sufficient quantity and quality for testing such as labeling tests and non-clinical studies. Similarly, the result shows that approximately 500 MBq of Lu-177 was produced by irradiating 1 mg of Yb for 14 days, suggesting that irradiation of a few grams of Yb could generally meet the domestic demand. Due to the high cost of the enriched target, re-irradiation of the recovered targets is necessary. We are also planning research and development to improve a column chromatographic separation process for the target radioisotope.

Conclusions: The trial irradiations for Lu-177 have confirmed the producing capability of JRR-3. Several issues were identified including specific activity and the need for re-irradiation. Moving forward, we plan to investigate these issues. Although KURNS facilities were not used in FY 2025, we'll advance the development of production and separation processes for broad range of radioisotopes in cooperation with KURNS.

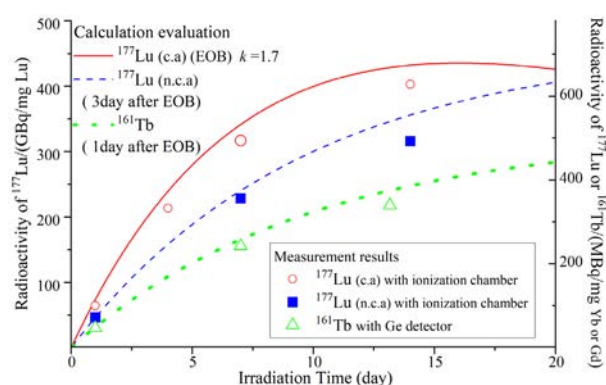


Fig 1 Evaluation of Lu-177 production volume

Development of metal radioisotope-labeled agents for cancer radiotheranostics

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INTRODUCTION: In recent years, the concept of theranostics—aimed at the efficient integration of diagnosis and therapy—has been increasingly advocated. In the field of nuclear medicine, radiotheranostics has attracted considerable attention as an approach that enables the development of both diagnostic and therapeutic radiopharmaceuticals based on an identical molecular scaffold, simply by altering the labeled radioisotope. This strategy is expected to promote highly efficient diagnosis and treatment. In particular, nuclear medicine therapies for cancer are anticipated to yield significant therapeutic efficacy, thereby creating a strong demand for the development of novel cancer-targeted radiotheranostic agents. The objective of this study is to develop novel low-molecular-weight metal radioisotope-labeled molecular probes targeting cancer for radiotheranostic applications, with a focus on targeting L-type amino acid transporter 1 (LAT1).

DRUG DESIGN CONCEPT: For the advancement of radiotheranostics, the use of metal radioisotopes represents a highly effective strategy, as it enables the development of both diagnostic and therapeutic agents based on an identical molecular scaffold simply by altering the radioisotope used for labeling. However, the incorporation of metal radioisotopes generally requires the introduction of relatively bulky chelating moieties into the probe structure, which may compromise the biological activity of the parent scaffold, particularly in the case of low-molecular-weight probes. Since the natural substrates of LAT1 are amino acids, it is challenging to incorporate bulky chelating structures while retaining substrate-like biological activity toward LAT1. Therefore, in this study, rather than designing probes as LAT1 substrates, we aimed to develop them as inhibitors with binding affinity to LAT1. Based on this strategy, novel metal radioisotope-labeled probes were rationally and precisely designed.

EXPERIMENTS: We have previously succeeded in the development of an ¹⁸F-labeled probe exhibiting high selectivity for LAT1, a transporter highly expressed in cancer cells among the LAT family [1-3]. Based on this prior knowledge, two types of control compounds were designed and synthesized using a tyrosine-based scaffold, with the introduction of a chelating moiety.

RESULTS: Two types of control compounds were successfully synthesized through multistep synthetic routes. Further design and synthesis of additional compounds are currently in progress, with subsequent studies planned for metal radioisotope labeling and evaluation of their biological efficacy.

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Investigation of suitable $^{177}\text{Lu}/\text{Yb}$ separation condition in extraction chromatography

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INTRODUCTION: Addressing the growing demand for ^{177}Lu in nuclear medicine, high-efficiency separation of ^{177}Lu and Yb has become a critical issue. However, due to the similarities in the ionic radii, oxidation states, and chemical behavior of Lu and Yb, their separation remains a significant challenge [1-3].

Our objective of this irradiation is to identify requirements for an automated column separation system that enables the scale-up of non-carrier-added ^{177}Lu production through long-term irradiation in KUR, by compiling challenges encountered in the separation of ^{177}Lu obtained from KUR-hydro (R7P8-4) irradiated Yb targets by extraction chromatography.

EXPERIMENTS: The irradiation conditions consisted of long-term irradiation (47 hours at 1 MW followed by 6 hours at 5 MW in a continuous cycle). The experimental apparatus used in this study consists mainly of a pump, a column with a water jacket and a CZT detector (GR1, Kromek). The solution is pumped up and introduced into the 30 cm column, which is filled with extraction resin (LN2, Eichrom, 50–100 μm). The column temperature is kept constant by a thermostatic water circulation system. The solution that has passed through the column is introduced into a lead shield with a γ -ray detector installed. Then, the solution is collected in a sample vial as it passes through a bundled flow path in the ring shape for γ -ray measurement.

RESULTS: As shown in Table 1, the specific activity of ^{177}Lu was $(1.042 \pm 0.01) \times 10^7$ Bq/mg, and the quantity generated was three times larger than that of a hydro (1MW24h) irradiation one.

Table1 Irradiated enriched ^{176}Yb sample data				
Exp. date	Yb_2O_3 (mg)	^{177}Lu (Bq/mg)	^{169}Yb (Bq/mg)	^{175}Yb (Bq/mg)
2026/1/22	1.071	$(1.042 \pm 0.01) \times 10^7$	n.d.	$(1.392 \pm 0.04) \times 10^6$

During the $^{177}\text{Lu}/\text{Yb}$ separation experiment, the peak energies and their intensities in the γ -ray spectra change with time. High intensities were detected at around 110, 131, 177, 198, 282, 308, and 396 keV for times around 600 min. These γ -ray peaks are attributed to ^{169}Yb (110, 131, 177, 198, and 308 keV) and ^{175}Yb (114, 282, and 396 keV), respectively. In the region around 660 min, high intensities were detected at 110 keV as well as 208 keV. Especially at the latter γ -ray energy, the intensity was confirmed only in this region. Since these can be attributed to ^{177}Lu (113 keV and 208 keV), it can be assumed that most of the Yb is leached out by passing 2.25 M HNO_3 , and the remaining resin residue, composed mainly of Lu, is leached out by 4 M HNO_3 after 660 min.

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Study on Ac-225 Production via the (n, γ) reaction of Th-decay Product Ra-228

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INTRODUCTION: Regarding the production of Ac-225, an α -emitting radionuclide used in targeted alpha therapy, the currently supplied Ac-225 is produced by separating it from Th-229 generators, which are derived from U-233. However, since U-233 is a fissile nuclide and its handling is strictly regulated, it is not suitable for the large-scale production of Ac-225. To meet the increasing demand for Ac-225 in recent years, a new production method has been proposed: irradiation of Ra-228, a daughter nuclide of naturally abundant Th-232, with thermal neutrons to produce Ra-229 via the (n, γ) reaction, followed by the production of Ac-225 through the decay of Ra-229 [1]. In this study, to demonstrate the feasibility of this method, an irradiation target consisting of Ra-228 supported on MgO was fabricated, and thermal neutron irradiation was carried out at the Kyoto University Research Reactor (KUR).

EXPERIMENTS: The MgO targets used for irradiation at KUR were fabricated by chemically treating a solution containing Ra-228. First, to prevent contamination of the irradiation target with Th-232 and the subsequent production of U-233, polyvinylpolypyrrolidone (PVPP) was used to remove trace amounts of Th present in the Ra solution. The solution was then adjusted to a nitric acid-methanol mixture, and Na in the solution was removed using a strongly basic anion exchange resin. After the removal of Th and Na, sodium carbonate and magnesium nitrate solutions were added, and RaCO₃ and MgCO₃ were co-precipitation. The recovered precipitates were heated at 400°C for 0.5 h to convert them into oxides. Fig. 1 shows the MgO target containing Ra-228.

Thermal neutron irradiation at KUR was conducted over five weeks from December 2, 2025, to February 5, 2026, with a total irradiation time of 30 h at 5 MW and 233 h at 1 MW. The radioactivity of Th-229 produced by this irradiation is estimated to be 1.26×10^{-3} Bq.



Target Appearance	White powder
Target composition	MgO + RaO
Weight of the target	0.088 g
Radioactivity of Ra-228	3.98 kBq

Fig. 1. Specifications for Ra-228-containing MgO target for thermal neutrons irradiation.

RESULTS: The irradiation proceeded without incident, and no deformation of the target or damage to the quartz glass tube was observed. After dissolving the target in 8 M nitric acid, Th was recovered using PVPP, and α -spectrometry was performed. The results showed that the Th-229 peak (4.846 MeV) is obscured by adjacent peaks of Th-228 (5.340 and 5.423 MeV), which is produced by the decay of Ra-228. Therefore, as an alternative method to verify the production of Th-229, detection of the At-217 peak (7.069 MeV), which is well separated from the adjacent Po-212 peak (8.785 MeV), was considered instead of direct detection of the Th-229 peak. Based on these results, further purification of the sample and long-term measurements are planned to evaluate the production of Th-229 via thermal neutron irradiation of Ra-228.

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I-2. COLLABORATION RESEARCHES

- 1. Neutron Scattering***
- 2. Nuclear Physics and Nuclear Data***
- 3. Reactor Physics and Reactor Engineering***
- 4. Material Science and Radiation Effects***
- 5. Geochemistry and Environmental Science***
- 6. Life Science and Medical Science***
- 7. Neutron Capture Therapy***
- 8. Neutron Radiography and Radiation Application***
- 9. TRU and Nuclear Chemistry***
- 10. Health Physics and Waste Management***
- 11. Others***

Small-angle X-ray scattering analysis of stabilized di-nucleosomes

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INTRODUCTION: In eukaryotic cells, genomic DNA is packaged into chromatin in the nucleus. The nucleosome is the repeating unit of chromatin, composed of 145-147 base pairs of DNA wrapped around a histone octamer containing two molecules each of histone proteins H2A, H2B, H3, and H4. Chromatin structure changes dynamically during various nuclear processes, such as gene transcription, DNA repair, and replication. These structural changes are influenced by histone modifications and chromatin-associated factors. Because the arrangement of neighboring nucleosomes is an important determinant of chromatin conformation, di-nucleosomes provide a simple model system for structural analysis. We analyzed di-nucleosomes by small-angle X-ray scattering (SAXS). To develop methods for structural analysis of chromatin conformation in solution using a di-nucleosome system, we examined a sample stabilization strategy to improve homogeneity for SAXS measurements.

EXPERIMENTS: Di-nucleosomes were assembled by in vitro reconstitution. Recombinant histones were combined with a DNA fragment under high-salt conditions, followed by a gradual decrease in salt concentration to promote nucleosome formation. The assembled di-nucleosomes were subsequently isolated by polyacrylamide gel electrophoresis [1]. Di-nucleosomes were gently fixed in a continuous sucrose gradient containing glutaraldehyde (GraFix) [2]. The samples were then dialyzed against SAXS buffer and concentrated prior to small-angle X-ray scattering (SAXS) measurements. SAXS intensity of the corresponding buffer was measured for background subtraction using the same procedure as for the samples. The scattering data were collected using a laboratory-based SAXS instrument equipped with a high-brilliance point-focused Cu K α source. To evaluate sample quality, including aggregation and dissociation, sedimentation velocity experiments were performed by analytical ultracentrifugation (AUC) [3]. In addition, the oligomeric state of the samples was analyzed by mass photometry.

RESULTS: At a concentration of 0.8 mg/mL, the SAXS sample showed substantial polydispersity. The AUC profile indicated the presence of 2- and 3-mer associated species, aggregated particles, and degraded components in addition to the di-nucleosome fraction, with degraded species accounting for approximately 50% of the total. Mass photometry yielded results consistent with the AUC analysis, despite the dilution required for the measurement. Based on these results, we used mass photometry to examine the GraFix fractions, dialysis and sample concentration conditions, and identified conditions that reduced sample heterogeneity. These analyses will be further pursued to establish sample preparation conditions for more homogeneous SAXS measurements of di-nucleosomes in solution, providing a basis for future structural studies of chromatin assemblies with additional components.

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Development of a full circumference ellipsoidal neutron supermirror

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INTRODUCTION: A new research reactor construction project, of which Kyoto University is one of three core institutions, is underway at the Monju site in Tsuruga City, Fukui Prefecture. The reactor's primary purpose is to produce results for academic and industrial applications using neutron beams and contribute to various fields, especially the local community. Effectively using the focusing mirror is one of essential to competing with leading institutes in the fields of neutron beam science and technology. We have developed a fabrication method for aspherical focusing supermirrors with a metal substrate [1]. The metal substrate is robust and ductile, and it can produce a steeply curved surface with high shape accuracy. It can also withstand high radiation and temperatures, even near the neutron target and moderator. We have achieved a smooth surface for high- m supermirror coating. Here, m is the maximum critical angle of the mirror in units of the critical angle of natural nickel. In this report, we present our progress on high- m neutron-focusing supermirrors on a full circumference ellipsoidal metal substrate.

EXPERIMENTS: We fabricated ellipsoidal metallic substrates with electroless nickel-phosphorus plating using ultra-high-precision cutting technology, correction processing, and mechanical precision polishing. Initial precise manufacturing was conducted on a CNC machine in the KURNS workshop for developing neutron optical devices. The ultra-precise manufacturing, polishing, and cleaning of the substrates were performed at RIKEN. Supermirror coating was performed using an ion beam sputtering machine (KUR-IBS) at KURNS. $m=6$ NiC/Ti supermirrors, of which number of maximum effective number of layer 12180, were deposited on a metal substrate with the semi-major and semi-minor axes of the supermirror are 1,250 mm and 65.4 mm, respectively. The full-circumference ellipsoidal mirror consists of 54 substrates, which were assembled at RIKEN to form one ellipsoidal focusing mirror. We also evaluated the neutron reflectivity of supermirrors deposited on a silicon (Si) substrate that were deposited simultaneously with the metal substrates at the CN-3 port of the KURLS reactor and the C3-1-2(MINE) port of the JRR-3 reactor.

RESULTS: As shown in Fig.1, a full circumference ellipsoidal $m=6$ supermirror with a length of 0.9 m was completed and installed to J-PARC MLF BL06(VIN ROSE). Although these are still preliminary results, we obtained a focusing spot as designed.

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[2] M. Hino, *et al.*, Nucl. Instr. and Meth., **797**, 265 (2015)



Fig.1 Photograph of a full circumference, ellipsoidal neutron supermirror made of 54 mirror pieces.

Characterization of cold-neutron flux of the TRIUMF Ultracold Advanced Neutron Source

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INTRODUCTION: The TRIUMF Ultracold Advanced Neutron (TUCAN) collaboration has been developing an accelerator-driven superthermal ultracold neutron (UCN) source at TRIUMF [1]. The method adopted here utilizes inelastic scattering of neutrons in superfluid helium, which exhibits a strong energy dependence around 1 meV. Characterization of the cold neutron spectrum incident on the superthermal converter is therefore a critical aspect of this source. To carry out this characterization effectively, we developed a neutron activation analysis method using cadmium and borosilicate glass as filters, sensitive to thermal and cold neutron energies, respectively. This method was tested at KUR-CN3 beamline and then used at TRIUMF to characterize the neutron spectra of the TUCAN source with different operational conditions.

EXPERIMENT In each data-taking run, a gold foil (typically 20 mm×20 mm× 50 μm) was placed in a holder, as shown in Fig. 1, with different filters positioned in front of it. The proton beamline typically operated for 5 minutes. The neutron flux was subsequently evaluated by counting the characteristic gamma rays at 412 keV emitted following neutron capture on ¹⁹⁷Au.

RESULTS: The results of the activation measurements for different moderator conditions of the TUCAN source are shown in Fig 2. The two different moderator conditions measured were 1) with room temperature heavy water (D₂O) moderator, and 2) with D₂O and liquid deuterium (LD₂) moderator. The horizontal axis is the thickness of the borosilicate glass plates used as filters. The observed difference in slopes between the two conditions indicates a change in the neutron temperature between these configurations. A more detailed analysis is currently underway.

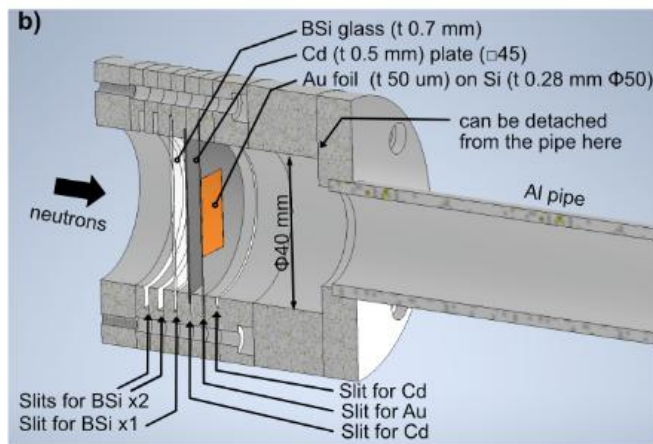


Fig. 1. Holder used to install gold foils and filters in the cold neutron monitoring port of the UCN source.

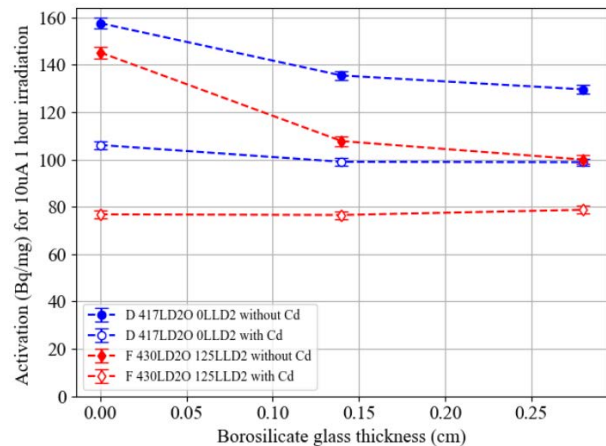


Fig. 2. Results of the activation measurement with different moderator conditions of the UCN source

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Development of absorption grating devices for CN-3 beamline at KURNS

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INTRODUCTION: Neutron imaging techniques are powerful, versatile, nondestructive analytical tools used in many research fields. Since its proposal in 2006[1], neutron Talbot-Lau interferometry (nTLI) has attracted considerable attention; it uses three gratings: a source grating (G0), a beam splitter grating (G1), and an analyzer grating (G2). G0 and G2 are absorption gratings, while G1 is a phase grating. Absorption gratings are important devices, and gadolinium (Gd) is used in their fabrication due to its high absorption cross section. This study presents a novel fabrication method for absorption gratings that uses an optimized Gd-based multilayer for ultra-high precision cutting.

EXPERIMENTS:

The Gd-based multilayer consists of gadolinium (Gd) and titanium (Ti). The G0 and G2 gratings were created by microcutting the multilayer on a substrate. These gratings were evaluated using an nTLI installed in the CN-3 port of the Kyoto University Reactor (KUR) [2]. For the nTLI at the CN-3 port, which detects thermal neutrons, the required effective Gd thickness is greater than 20 μm . To achieve this thickness, the Gd film was coated using an ion beam sputtering instrument (KUR-IBS) at KURNS, as shown in Fig. 1.

We fabricated a very flat aluminum substrate with a thickness of 20 mm for ultra-high precision cutting prior to deposition. We used the Gd/Ti bilayer to fabricate the G0 and G2 gratings, which had 2,000 and 400 layers, respectively. The deposition times for the Gd and Ti layers were five and 30 seconds, respectively. After sputtering, we shaped the Gd/Ti multilayer using an ultra-high-precision cutting machine (NPIC-M200, Nagase Integrex Co., Ltd.) at RIKEN. The G0 and G2 grating pitches were 125 μm and 8.5 μm , respectively, within 50 mm squares.

RESULTS: Fig.2 shows the measured visibility of the newly fabricated G0 and G2 gratings at the nTLI at the CN-3 port. The average visibility of the pixels in the central area (marked by the yellow rectangle) was 0.28. Based on neutron transmission efficiency, we estimated the total thicknesses of the G0 and G2 films to be 46 μm and 8.5 μm , respectively. Considering the lesser thickness of the G2 grating for the neutron wavelength of the CN-3 beamline, the average visibility is reasonable.

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Fig.1 Typical measured visibility of the nTLI installed to the CN-3 beamline.

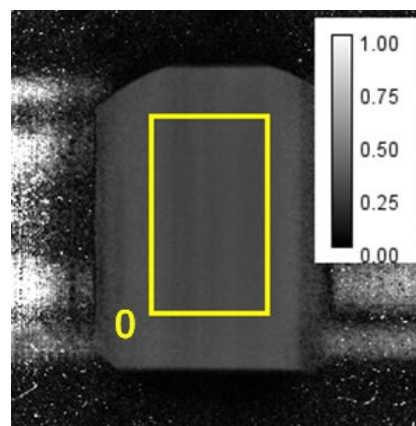


Fig.2 Measured visibility maps obtained with the newly fabricated G0 and G2.

Structural Analysis of Viscosity Index Improver Molecules Using Small-Angle X-ray Scattering

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INTRODUCTION: Lubricating oils are necessary for friction reduction and high wear durability of sliding surfaces in machine components, and the development of the best oils is highly demanded by the industry. Viscosity index improver (VII) is a type of additive used to mitigate the decrease in viscosity of lubricating oil due to temperature rise. Classical textbooks explain that VII molecules work by changing their equivalent radius in the base oil according to the oil temperature. However, there are few papers investigating the equivalent radius of VII molecules by small-angle X-ray scattering (SAXS) and/or small-angle neutron scattering (SANS) [1], and there is still room for discussion about the behaviour and working mechanism of VII molecules in oil. This study aimed to investigate the radius of gyration of a VII polymer in base oil at different temperatures by SAXS, and the behaviour of the VII polymer was discussed.

EXPERIMENTS: To investigate the radius of gyration of VII polymer, a SAXS instrument (NANOPIX, Rigaku) with a Cu-K α X-ray source (MicroMAX-007, Rigaku) and a semiconductor 2D detector (HyPix-6000, Rigaku) were used. The 1.2 mm-thick aluminum cell with optical windows made of 20 μ m heat resistant engineering plastic film (Superio-UT, Mitsubishi Chemical) was used for the measurement. The cell temperature was successively increased to 25, 40, 60, 80 and 100°C, and the final measurement was carried out again at 25°C after cooling to check the degeneration of the VII molecule. Comb-shaped poly(methyl acrylate) (PMA) type VII was prepared as a model one used in the study. Squalane was used as a model base oil, and the concentrations of PMA in squalane were 0.5 mass%.

RESULTS: The SAXS profiles from squalane with comb-PMA type VII at each temperature are shown in Fig. 1. The profiles change as the temperature rises. Fig. 2 shows the Guinier plots, and the radii of gyration (R_g) and the former scattering intensities ($I(0)$). It was found that the comb-shaped VII molecule exhibited a gradual increase in R_g as the temperature rises. While this behaviour is similar to that of VIIs described in classical textbooks, it differs from that of another type of comb-shaped VII investigated previously. In the future study, we plan to separately discuss the role and behaviour of the main and side chains of comb-shaped VII molecules.

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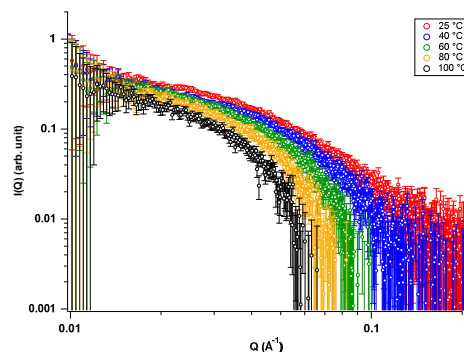


Fig. 1 SAXS profiles at each temperature.

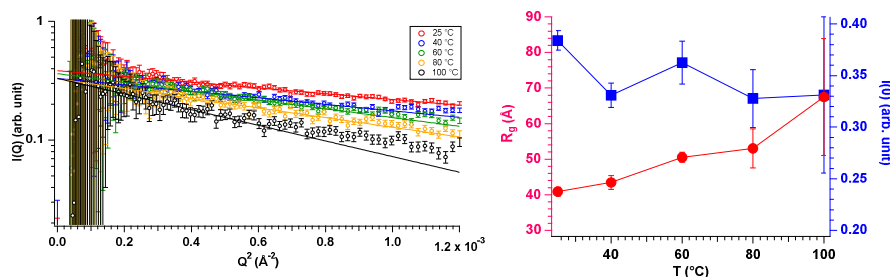


Fig. 2 Guinier plots (left) and the estimated R_g (right) at each temperature.

Measurements with neutron interferometer and development of the half-mirror with suppressing distortion

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INTRODUCTION: Neutron interferometry is a powerful technique for studying fundamental physics. Numerous interesting experiments [1] have been performed since the first successful test of a single-crystal neutron interferometer [2]. However, the single-crystal interferometer is inherently not able to deal with a neutron that has a wavelength longer than twice its lattice constant. In order to investigate problems of fundamental physics, including tests of quantum measurement theories and searches for non-Newtonian effects of gravitation, the interferometry of cold neutrons is extremely important, since the sensitivity of interferometer for small interaction increases with the neutron wavelength. A large scale of interferometer also has the advantage to increase the sensitivity to small interactions. One of the solutions is an interferometer using neutron multilayer mirrors [3,4]. We can easily control parameters such as Bragg angle, reflectivity, and Bragg peak width by selecting the deposited material and tuning the bilayer thickness and the number of layers. We have demonstrated a multilayer interferometer for pulsed cold neutrons at the beamline 05 NOP in J-PARC MLF [5]. In the case of pulsed neutrons, the phase of interference fringes depends on neutron wavelength which is resolved by time-of-flight. Recently we tried to measure the neutron scattering lengths of ³He, ⁴He, and Xe gas with the interferometer. The gas cell which can be installed into the narrow gap of the interferometer was developed. These measurements provide important information for research in the physics of many-body systems of nuclei.

EXPERIMENTS AND RESULTS: In order to enlarge the number of neutrons, We are continuing to develop the neutron supermirrors which can reflect wide bandwidth of wavelength for the interferometer by using Ion Beam Sputtering facility in KURNS. In this study, we further reduced the number of layers compared to previous designs, resulting in a material with low reflectivity but extremely low deformation, that causes contrast loss of the interferogram. Figure 1 shows the reflectivity of the half mirrors with wide bandwidth, measured at MINE2 in JRR3. We will apply the mirrors to the interferometer.

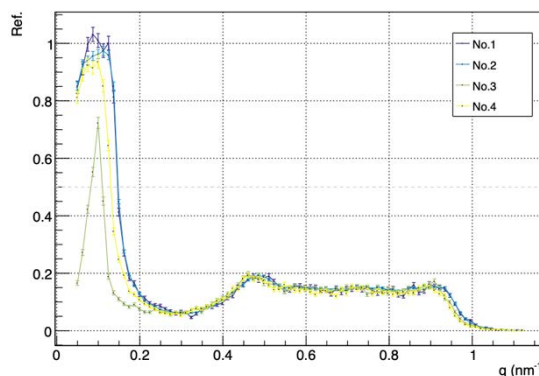


Fig. 1. Reflectivity of the half mirror with wide band of neutron wavelength measured at MINE2 in JRR3.

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Measurement of Natural Uranium Samples Using Fast Fission Neutron Coincidence Counting

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INTRODUCTION: We have been developing a Fast Fission neutron Coincidence Counting (FFCC) method applicable to nuclear materials containing neutron absorbers, such as fuel debris at the Fukushima Daiichi Nuclear Power Plant and concealed nuclear materials in the field of nuclear security [1]. In the FFCC method, samples are irradiated with fast neutrons, which are less affected by neutron absorbers, and the amount of nuclear material is estimated by measuring neutrons generated by fast neutron-induced fission. The method has previously been demonstrated experimentally for samples on the order of several tens of grams. In this study, measurements were conducted to investigate the response of the FFCC method for kilogram-scale uranium samples.

EXPERIMENTS: Figure 1 shows the experimental setup. A natural uranium sample was placed at the center, with four EJ-301 liquid scintillators (5-inch diameter and 5-inch length) arranged on both sides and a D–D neutron generator positioned behind the sample. A polyethylene shield was placed between the neutron generator and the scintillators to prevent direct neutron incidence. The sample was irradiated with neutrons, and the emitted fission neutrons were detected by the scintillators. Measurements were performed with a neutron intensity on the order of 10^5 n/s for uranium masses of 0 kg (blank), 1 kg, 5 kg, 10 kg, and 20 kg.

RESULTS: In the FFCC method, the time difference between neutron detection events for each detector pair is calculated [1]. Figure 2 shows a histogram of the time-difference event for a 10 kg uranium sample (time-difference spectrum). Fission neutrons produce a peak near 0 ns, while neutrons scattered between detectors (crosstalk) produce peaks shifted from 0 ns. As a result, three peaks appear in the time-difference spectrum. These results confirm that coincidence events originating from fission neutrons were successfully detected. In addition, measurements for other samples showed variations in the fission neutron count depending on the uranium mass. These results suggest that the FFCC method is applicable to uranium samples on the kilogram scale.

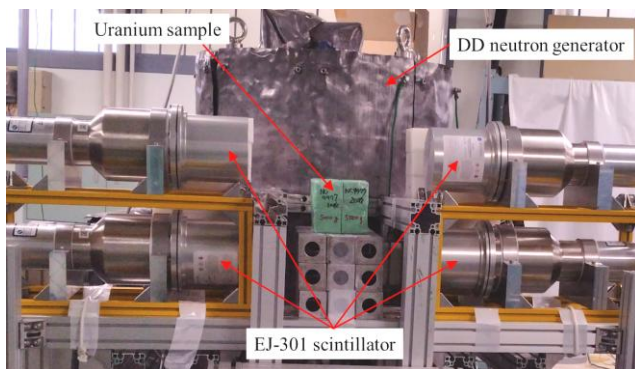


Fig. 1. Experimental setup.

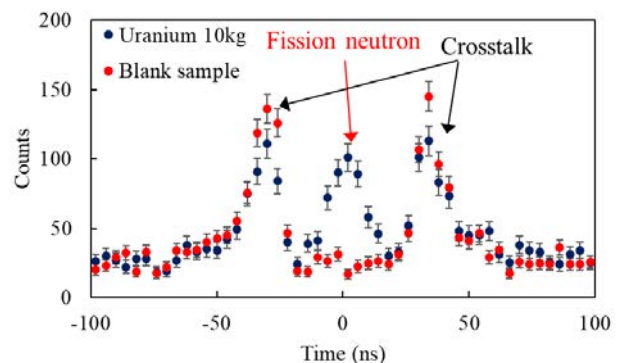


Fig. 2. Time-difference spectrum.

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Neutron resonance analysis technique in neutron time-of-flight for highly enriched uranium

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INTRODUCTION: The ISCN of the JAEA is developing a combined neutron resonance analysis (NRA) system aims to identify and quantify fissile material in an unknown sample. Neutron resonance transmission analysis (NRTA) and neutron resonance capture analysis (NRCA) are used together with the proposed neutron resonance fission neutron analysis (NRFNA) [1,2,3]. Using a pulsed neutron beam with the neutron time-of-flight (TOF) technique, prompt capture gamma rays and fast fission neutrons from fissile nuclides are measured at the sample location with plastic scintillators able to perform n/γ pulse shape discrimination (PSD). In this paper, we report a demonstration experiment for highly enriched uranium samples performed at KURNS-LINAC.

EXPERIMENTS: The KURNS-LINAC operated with an acceleration energy of ~30 MeV. Pulsed neutrons were produced with a repetition rate of 50 Hz and a pulse width of 2 μs. Enriched uranium (²³⁵U: 93%) foils were used with thickness up to 6 mm. Between the beam exit and the sample, a borated polyethylene and lead collimator with an aperture of 2.0 cm was used. At the sample position, an assembly of ten hexagonal PSD plastic scintillation detectors (Eljen, EJ-276D) were used for NRFNA and NRCA with a 3.0 cm lead shielding. Between the sample and the NRTA detector (⁶Li-glass, GS20), a second collimator with an aperture of 1.0 cm was placed. The setup used is shown in Fig. 1. The output signals of the detectors were sent to the CAEN digitizer V1730D (14bit, 500MSample/s); the data were recorded in list mode with 1h autosave.



Fig. 1. Experimental setup.

RESULTS: Obtained data are being analyzed by an advanced PSD technique [4]. Fig. 2 shows the TOF spectra of 6 mm thick enriched uranium foils. To be noted, the GS20 spectrum is shifted in time, due to the further position of the detector from the sample. As in the case of shielded samples [5], the characteristics peaks from ²³⁵U(n,f) reaction are observed at all energies in the neutron spectrum; a linear mass correlation can be obtained after normalization for beam, intensity variation. In the gamma-ray spectrum, the 6.67 and the 20.87 resonance peaks from ²³⁸U(n,γ) reaction are visible and can be used to evaluate sample self-shielding effects. This experiment confirmed the possibility not only to identify fissile nuclides, but, more importantly, to quantify them.

ACKNOWLEDGEMENTS: This research was implemented under the subsidy for “promotion of strengthening nuclear security and the like” of the MEXT (the Ministry of Education, Culture, Sports, Science, and Technology of the Japanese government).

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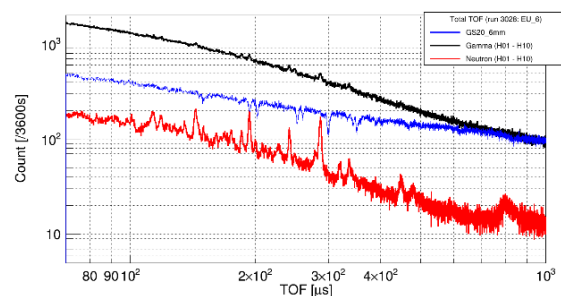


Fig. 2. TOF spectra of 6 mm thick enriched uranium: neutron (red), gamma (black), GS20 (blue).

Basic Study on Improving Experimental Accuracy in the Fast Energy Range of Neutron Cross Section Measurement Using KURNS-LINAC

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INTRODUCTION:

We have been conducting neutron cross section measurements for nuclear fuel materials and minor actinids by using KURNS-LINAC [1]. Those experimental studies have been performed with time-of-flight method (TOF method). Because KURNS-LINAC uses a photo-neutron source, measurements of neutron cross section above approximately 1 keV in the TOF method exhibited large uncertainties due to the influence of bremsstrahlung X-rays generated during photo-neutron production called “gamma-flash”. In particular, those uncertainties have a significant impact on the resonance analysis performed using the measurement data. Therefore, in this study, we focused on an iron filtering system to reduce gamma-flash and improve the accuracy of measurement data at energies of 1 keV or higher. The iron filtering system utilizes neutron cross-section windows in natural iron at 20, 73, and 82 keV to extract quasi-monochromatic neutron flux and had been installed the B-1 beam hole in KUR. In particular, the use of iron filter beam at JPARC MLF/ANNRI has yielded significant results in the study of neutron cross section measurement in the fast energy range [2]. In this study, an iron filtering system has been installed and characteristics of filtered neutron beam were measured.

EXPERIMENT:

The iron filtering system has been installed on end of 12m neutron flight path. The iron filter has 2 inch length. The filtered neutron beam was measured by TOF method. A neutron detector was employed Li-glass detector. In the experiments, a measurement of the iron filtered beam and the neutron beam with resonance filters (In, Ag, Mn, Co) were performed.

RESULTS:

Figure 1 shows obtained TOF spectra. A red line is the iron filtered beam and a blue line is the neutron beam with the resonance filters. Here, the TOF spectrum obtained using the resonance filter was used as a reference to determine the energy of the TOF spectrum obtained with the iron filtering system. Furthermore, the dips caused by saturation resonance (black arrow) are considered to represent the background level. As the results, the quasi-monochromatic neutron flux in the energies of 73 keV and 20 keV were obtained.

In those experiments, we successfully extracted quasi-monochromatic neutron beams with energies of 73 keV and 20 keV using the iron filtering system in the KURNS-LINAC pulsed neutron source.

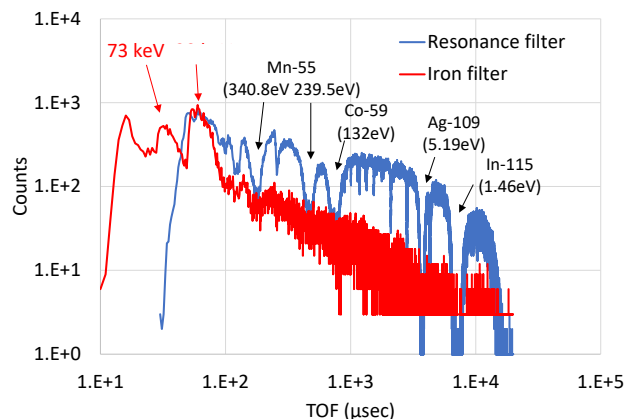


Fig.1. Measured TOF spectrum

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Search for Tetraneutron with an NAA-like Approach for Magnesium

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INTRODUCTION: Compared to typical nuclei composed of both protons and neutrons, the tetraneutron is composed of only four neutrons. Motivated by a recent observation of a possible tetraneutron bound state [1], we have been searching for its experimental signature at KUR. A tetraneutron bound state, if it exists, would travel a sufficiently long distance, and have a chance to induce a ($^4\text{n},\text{n}$) reaction with a nucleus (^AZ), leading to the production of ^{A+3}Z . This product can be identified by detecting γ rays from the β -decay of ^{A+3}Z with a germanium detector. This approach resembles neutron activation analysis (NAA), in which a nuclide (^AZ) in a sample is identified and quantified by detecting γ rays from ^{A+1}Z produced via neutron capture.

Recently, theoretical calculations of tetraneutron-induced reaction cross sections have been made possible using an optical and statistical Hauser-Feshbach model code, CoH3 [3]. The calculations reveal that the $^{26}\text{Mg}(^4\text{n},2\text{n})^{28}\text{Mg}$ cross section is exceptionally large, around 10 barns, compared to typical ($^4\text{n},\text{n}$) cross sections.

EXPERIMENTS: We conducted a tetraneutron search experiment using two samples, ^{26}Mg -enriched ^{26}MgO power and high-purity magnesium pellets. These samples were irradiated at the hydraulic conveyor (Hyd) for ten and one hours, respectively, at the KUR thermal power of 1 MW. As an example, the γ -ray spectrum for the ^{26}MgO sample is shown in Fig. 1. Although many photopeaks from impurities were identified, photopeaks from ^{28}Mg were not observed. Based on this result, we will derive an upper limit on ^4n emission rate in uranium fission.

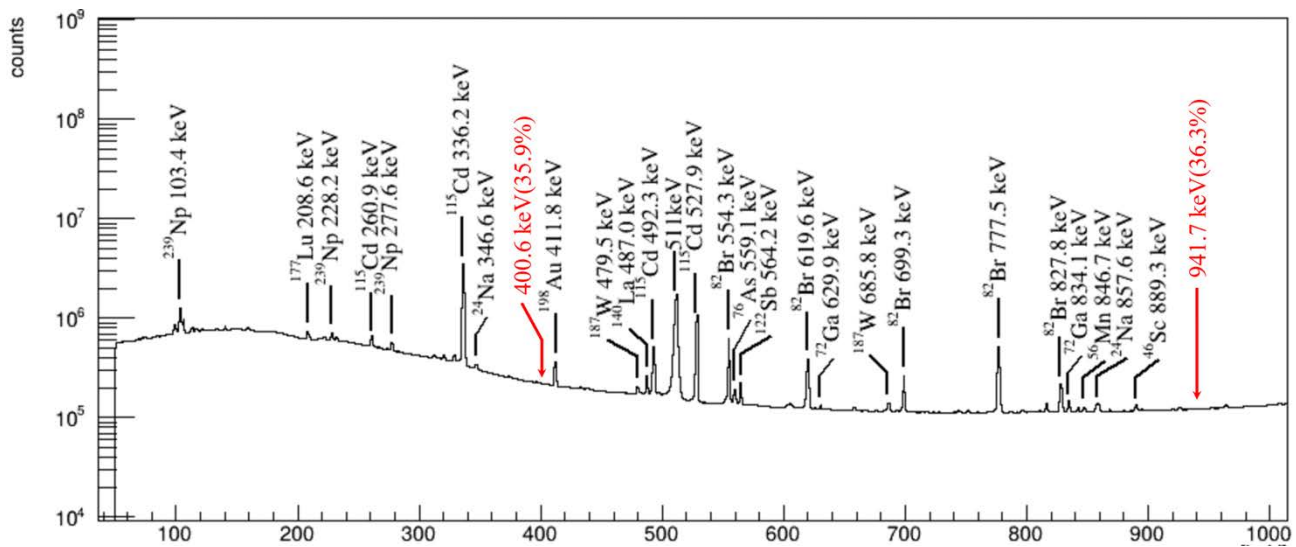


Fig. 1 The γ ray spectrum for the ^{26}MgO sample.

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Characteristics of thimble-type ionization chamber for an intense epi-thermal neutron beam

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INTRODUCTION: The need to ensure traceability to neutron standards at AIST for hospitals performing boron neutron capture therapy (BNCT) has been discussed. For this purpose, we are developing a real-time detector that can measure high-intensity neutrons used in BNCT. It is required that the detector can measure neutrons in appropriate measurement time at standard neutron fields whose fluxes are 4 to 5 orders of magnitude lower than those at BNCT as well. Then, we are developing an ionization chamber for measuring high-intensity epi-thermal neutrons with fluence rate of $10^9 \text{ cm}^{-2}\text{s}^{-1}$ or higher in the BNCT neutron field. As part of the characterization of these chambers, we measured their voltage characteristics.

EXPERIMENTS: The ionization chambers are thimble-type and consist of an aluminum chamber and an aluminum needle. One chamber is filled with ^3He (0.1 atm.) and Kr (0.9 atm.) gases, while another has ^6LiF deposited on its inside walls. Tests on these ionization chambers were conducted using a pulsed white neutron source based on an electron linac (KURNS-LINAC) and at the KUR heavy water facility. The relative energy responses were measured using the time-of-flight method with a pulsed white neutron source. The voltage characteristics were evaluated at KUR, where a high-intensity neutron flux is available.

RESULTS: Fig. 1 shows the results of applied voltage vs output current for ionization chambers with ^3He gas and ^6LiF foil. These results were obtained by applying a voltage to the outer electrode and extracting current from the central pin. It can be seen that the two types of ionization chambers exhibit the same trend for the same voltage. Previous measurements were conducted in the range of 100 V to 1000 V, and the results were found to be constant. A clear transition from the recombination region to the ionization region was observed. It was found that the device can be used as an ionization chamber at voltages generally 50 V or higher. Moving forward, we will evaluate the effects of dark current and gamma rays.

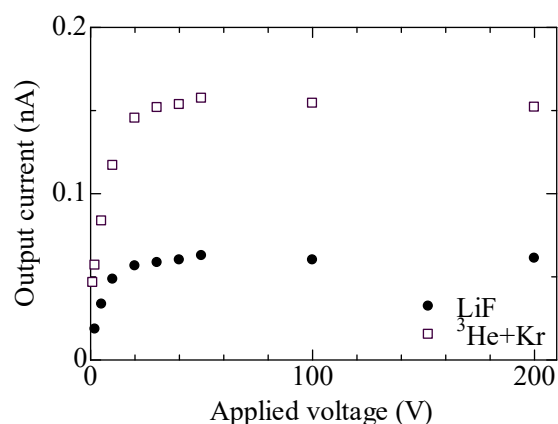


Fig. 1. Results of output current vs applied voltage for ionization chambers with ^3He gas and ^6LiF foil.

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Neutron Capture Cross-Section Measurements at TC-Pn in KUR for Tungsten among Nuclides in Decommissioning

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INTRODUCTION: Regarding the nuclides of concern at clearance levels [1] in decommissioning, thermal-neutron capture cross-sections have been measured by an activation method using the thermal column pneumatic tube (TC-Pn) of KUR [2-7]. The present work selected ¹⁸⁶W nuclide and measured the thermal-neutron capture cross-section for production of ¹⁸⁷W with a clearance level of 10 Bq/g [1].

EXPERIMENTS: Tungsten (W) samples were prepared as 99.95% pure, 5-μm-thick metal foils. Neutron irradiation for one hours using TC-Pn at 1-MW operation was performed three times as follows: I) The neutron flux was measured using Au and Co monitors to determine the ¹⁸⁶W cross-section. II) The measurement of ¹⁸⁶W was repeated using a simple comparison method with Au. III) To confirm the accuracy of the obtained ¹⁸⁶W cross-section, the W sample was irradiated as a neutron flux monitor together with Au and Co monitors. After neutron irradiation, the γ-rays emitted from each sample were measured using a high-purity Ge detector installed at the Hot Laboratory in KUR. The sample was placed at a distance of 110 mm from the front surface of the Ge detector. The γ-ray peak efficiencies of the Ge detector were measured beforehand with a mixed γ-ray source and a γ-ray reference source of ¹⁵²Eu. Gamma-rays emitted from ¹⁸⁷W were measured in sufficient yields. Six γ-rays emitted from ¹⁸⁷W were observed at γ-ray energies of 479, 551, 618, 625, 685 and 772-keV.

ANALYSIS AND RESULTS: The reaction rates of the flux monitors and W samples were derived from the γ-ray yields. Based on the *Westcott's* convention[8], the thermal-neutron capture cross-section of ¹⁸⁶W was derived using the neutron flux results and the reaction rates of ¹⁸⁶W. The result of cross-sections of ¹⁸⁶W were found to be 43.5 ± 1.2 barns for the irradiation I, which was 14% larger than the evaluated value of 38.1 ± 0.5 barns [9]. In irradiation II, a value of 43.1 ± 1.3 barns was obtained, confirming the reproducibility of the result from the irradiation I. Furthermore, when the neutron flux was measured using W as a monitor in irradiation III, the results were obtained as shown in **Figure 1**. The ¹⁸⁶W cross-section obtained in this study gives a neutron flux which is consistent with the neutron fluxes obtained with the Au and Co monitors.

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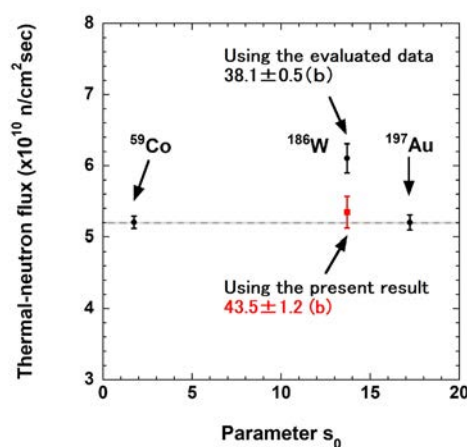


Figure 1 Thermal-neutron flux measured using the ¹⁸⁶W thermal-neutron capture cross section obtained in this study.

Progress in measurements of the response of a GAGG Scintillator to monoenergetic electron irradiation at KURNS-LUNAC

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INTRODUCTION: GAGG (Ce:Gd₃(Al, Ga)₅O₁₂) scintillators[1] have attracted considerable attention for radiation detection because of their high light yield, good energy resolution, and non-deliquescent nature. Owing to these properties, we are investigating the potential of GAGG scintillators for high-precision β -ray measurements aimed at elucidating the decay modes of nuclei in the termination region of the r-process.

EXPERIMENT: In this study, low-intensity electron beams with energies of 4–8 MeV, provided by KURNS-LINAC, were directly irradiated onto the GAGG scintillator in order to investigate its response to monoenergetic electrons.

RESULTS: The spectrum obtained with 4 MeV electron irradiation is shown in Fig. 1 (blue line). To suppress the γ -ray background in the experimental room, a plastic scintillator was placed in front of the GAGG scintillator. By requiring coincidence between the two detectors, electrons incident on the GAGG scintillator were identified. The spectrum obtained in coincidence with the plastic scintillator is shown in magenta in Fig. 1. By combining the experimental data with Monte Carlo simulations based on the Geant4 toolkit[2], we extracted the single-electron component while taking the contribution of each spectral component into account. Figure 2 shows the extracted single-electron spectra for incident energies of 4–8 MeV. Further analysis will be carried out to evaluate the detector response function in detail.

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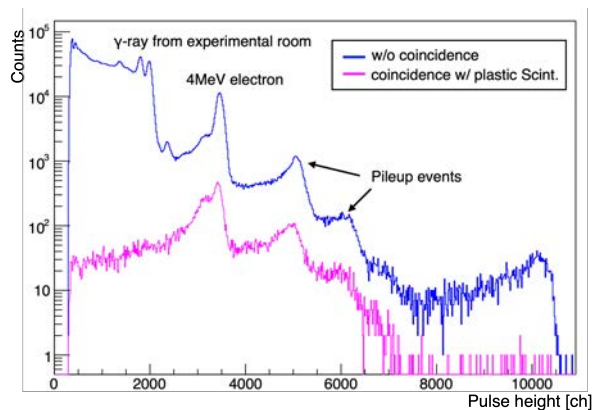


Fig. 1. Singles spectrum (blue) and coincidence spectrum with the plastic scintillator for 4 MeV electron irradiation.

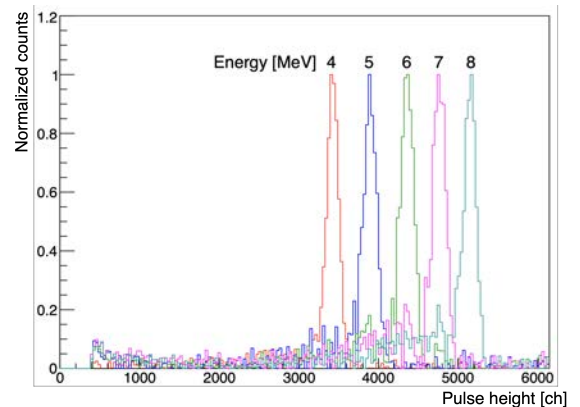


Fig. 2. Singles-electron spectra for incident energies of 4-8MeV, with normalized intensities.

Measurements of neutron capture cross sections of ^{78}Se at KURNS-LINAC

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INTRODUCTION: The disposal of long-lived radioactive waste generated from nuclear power plants remains a critical issue. Nuclear transmutation of long-lived fission products (LLFPs) via neutron capture reactions is expected to reduce the environmental burden associated with geological disposal [1]. Selenium-79, which has a half-life of 3.27×10^5 years [2], is one such LLFP targeted for transmutation. In transmutation systems without isotopic separation, stable Se isotopes ($^{74, 76, 77, 78, 80, 82}\text{Se}$) coexist with ^{79}Se ; therefore, neutron capture cross section data for the Se stable isotopes are also required, as they influence the overall transmutation efficiency of the system. In this study, measurements of neutron capture cross section for ^{78}Se , one of the stable isotopes of selenium, were performed at the Kyoto University Institute for Integrated Radiation and Nuclear Science - Linear Accelerator (KURNS-LINAC).

EXPERIMENTS and RESULTS: We measured neutron capture cross sections of ^{78}Se using the time-of-flight (TOF) method at the KURNS-LINAC. Electron beams with an energy of 37 MeV, an average beam current of 10 μA , a pulse width of 100 ns, and a repetition rate of 100 Hz were bombarded onto a water-cooled tantalum target. Neutrons were produced by photo-nuclear reactions induced by bremsstrahlung X-rays and were moderated by a light water moderator (17 cm diameter, 30 cm height). Moderated neutrons were guided to the experimental area at 135 degrees relative to the electron beam direction. An enriched ^{78}Se sample (20 mm diameter x 1 mm thickness, 99.6% enrichment, net weight 2.0 g) was placed 12.1 m from the neutron source. Neutron capture gamma-rays emitted from the ^{78}Se sample were detected using an array of 12 BGO detectors (2-inch diameter $\times$ 2-inch length). Detector signals were digitized using a CAEN V1730S module to record two-dimensional data of TOF and Pulse Height (PH). Figure 1 shows the measured TOF spectra of the ^{78}Se sample and the blank (without sample), respectively. A 383 eV resonance peak of ^{78}Se was observed at 44.5 μs . Furthermore, an enriched ^{10}B sample was used to determine the incident neutron energy spectrum by counting 478 keV gamma-rays via $^{10}\text{B}(n, \alpha \gamma)^7\text{Li}$ reactions. Neutron capture yields were subsequently derived by subtracting both sample-dependent and sample-independent backgrounds.

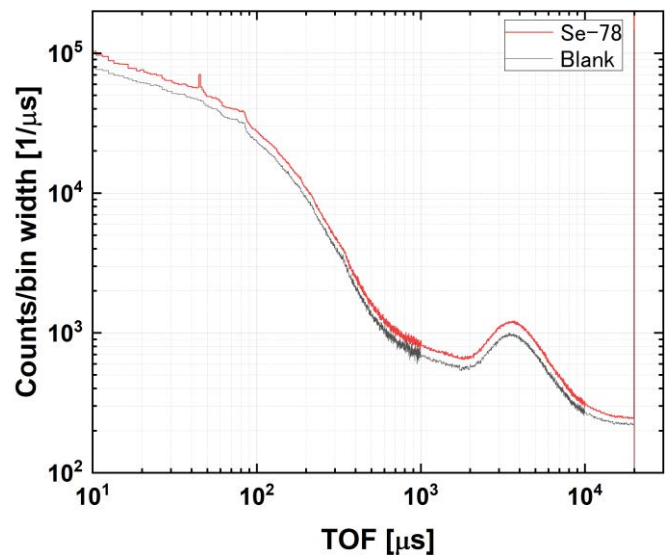


Fig. 1 TOF spectra of ^{78}Se (red) and Blank (black). The x-axis is TOF. The y-axis is counts per bin width.

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Sub-nanosecond Lifetime Measurements of Nuclear Excited States Using LaBr₃(Ce) Detectors

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INTRODUCTION: Unstable isotopes with β -decay half-lives of several days or longer are widely used in medical and scientific applications; however, for some nuclides, nuclear data have not been sufficiently validated. Sub-nanosecond lifetimes of excited states often show large uncertainties or inconsistencies among reported values. In this study, lifetimes of selected excited states were remeasured using β - γ and γ - γ delayed coincidence techniques with LaBr₃(Ce) detectors, which offer both high time and energy resolution. The target nuclides include ¹⁷⁷Hf, ¹⁴¹Pr, ⁷⁵As, and ¹⁰³Rh, aiming at improving the reliability of nuclear data.

EXPERIMENTS: The parent nuclides ¹⁷⁷Lu ($T_{1/2} = 6.6$ d), ⁷⁵Se ($T_{1/2} = 119.8$ d), ¹⁴¹Ce ($T_{1/2} = 32.5$ d), and ¹⁰³Ru ($T_{1/2} = 39.2$ d) were produced via (n, γ) reactions using the Kyoto University reactor (1 MW, neutron flux: 1.6×10^{13} n cm⁻² s⁻¹). Source activities during measurements were below 5 kBq. Measurements were conducted using two plastic scintillators (detectors 1 and 2: 50 mm $\times$ 20 mm and 30 mm $\times$ 30 mm $\times$ 1 mm) and two LaBr₃(Ce) detectors A and B (38 mm $\times$ 8 mm) as described in Table 1. The time resolution was 1–2 ns for 100 keV γ rays.

RESULTS: For the 113 keV level of ¹⁷⁷Hf, lifetimes of 0.550(5) ns were obtained, consistent with the evaluated value. For the 321 keV level, 0.780(7) ns was obtained, supporting the recent result of 0.792(17) ns [2] rather than the evaluated value [1] (Fig. 1). The 145 keV level of ¹⁴¹Pr was measured for the first time as 1.85(1) ns, in agreement with evaluation [3]. For ⁷⁵As (199 keV), 0.888(20) ns was obtained from γ -ray coincidence, consistent with previous results [4]. For ¹⁰³Rh (93 keV), the first LaBr₃-based measurement yielded 1.15(7) ns, also consistent with evaluation [5]. These results are preliminary but demonstrate improved precision using LaBr₃(Ce) detectors. Remeasurement with modern techniques is important for refining nuclear data.

Table 1. Target nuclides, excited levels, detector combinations, and evaluated values.

Nuclide	Level (keV)	Detector combination	Evaluated value (ns)
¹⁷⁷ Hf	113	plastic (1, 2) + LaBr ₃ (A)	0.541(14) [1]
	321	plastic (2) + LaBr ₃ (A)	0.665(16) [1]
¹⁴¹ Pr	145	plastic (2) + LaBr ₃ (A)	1.85(3) [3]
⁷⁵ As	199	LaBr ₃ (A, B)	0.885(30) [4]
¹⁰³ Rh	93	LaBr ₃ (A, B)	1.11(3) [5]

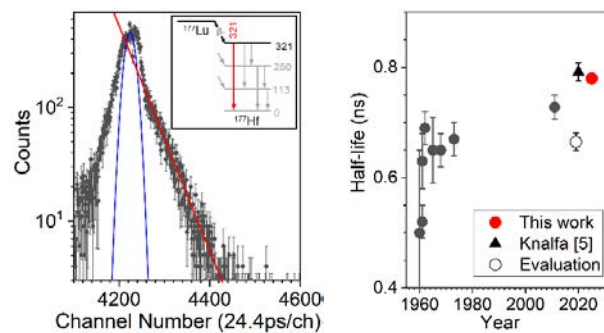


Fig. 1 Delayed coincidence time spectrum for the 321 keV level of ¹⁷⁷Hf (left) and comparison of the measured lifetime with previous and evaluated values (right).

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Study on Improving the Accuracy of Fission Cross-section Measurement

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INTRODUCTION: In the development of advanced nuclear reactors, improving the accuracy of fission cross sections for minor actinides (MAs) is one of the critical issues. However, the accuracy of fission cross-section data of MAs is currently insufficient to fully meet engineering requirements. That is because the fission cross-section measurement with a fission chamber has large uncertainties due to strong alpha decay background and the limitation of target mass imposed by the fission chamber. To overcome these difficulties, we aim to apply another technique that detects prompt neutrons emitted by fission reaction with a recoil proton type neutron detector for measurement of fission cross sections. We constructed a full solid-angle detector system and conducted performance tests on it at KURNS-LINAC TOF beam line.

EXPERIMENT: The linac was operated with a pulse width of 1 μ s, a repetition rate of 50 Hz, a peak current of about 0.5 A, and an electron energy of about 30 MeV. We used a flight path in the direction of 135 degree with respect to the linac electron beam. A high enriched uranium foil (U_3O_8 -Al alloy, 93% U-235) used as a sample was set in the neutron beam line at a distance of 11.25 m from the neutron target. The sample was surrounded with five recoil proton type neutron detectors (EJ-276 plastic scintillator) and five BGO detectors. The signals from the detectors were digitized using a CAEN V1730D module. In order to distinguish between gamma and neutron signals, the Neutron/Gamma pulse shape discrimination (PSD) method was applied [1]. On the other hand, the 478-keV gamma rays from a ^{10}B sample were also measured with BGO detectors for determination of neutron flux at the sample position.

RESULTS: Typical two-dimensional histogram of pulse height and PSD for the high enriched uranium sample measurement is shown in Fig. 1. The PSD value was defined as a ratio of the differences in charge integration between short and long gates. Neutron-induced events could be separately observed in the PSD region from 0.35 to 0.45. By gating on the neutron-induced PSD region, we have obtained the TOF spectrum corresponding to the fission events of U-235. In this study, we implemented an advanced data-driven PSD method, enhancing the analysis of neutron and gamma signals for fission cross-section measurements.

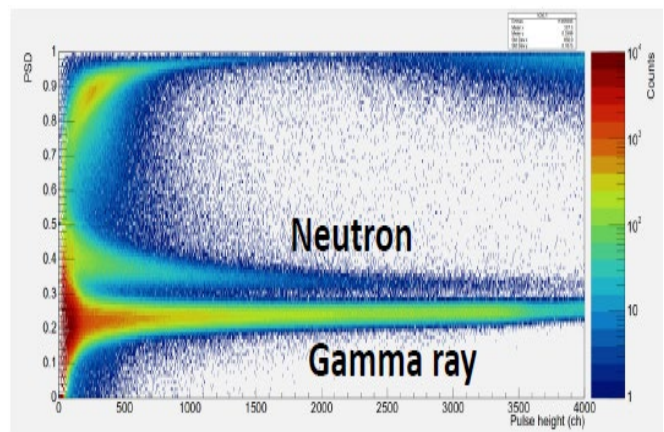


Fig. 1 2D histogram of pulse height and PSD

Acknowledgment

This work was supported by MEXT Innovation Nuclear Research and Development Program Grant Number JPMXD0224020564.

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Reactor noise measurement using ex-core detector system

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INTRODUCTION:

Reactor noise measurements targeting KUR were conducted from FY2014 to FY2021 using fission chambers for startup monitoring system as in-core detectors and He-3 detectors as ex-core detectors. However, because thermal neutrons were the primary measurement target, sufficient results could not be obtained due to various challenges, including the distance between the reactor core and the detectors [1, 2]. In this study, epithermal neutrons were selected as the measurement target, and the analysis methodology was also improved. Reactor noise measurements of KUR were therefore carried out again. The detectors used were BF₃ neutron detectors equipped with Cd covers, and measurements were performed at the CN-1 and B-3 experimental holes. The results of this study contribute to the advancement of nuclear characteristics analysis for high-power research reactors.

EXPERIMENT:

The detector system used in this study, referred to as “ERYNGII” [3], is designed to efficiently measure epithermal neutrons. In this system, thermal neutrons are shielded by a cadmium (Cd) plate, while epithermal neutrons are moderated by polyethylene, enabling effective detection of epithermal neutron flux. This detector system was installed at the front of CN-1 and B-3 experimental holes, and time-series data of the epithermal neutron flux were acquired. The measurements were conducted during nighttime on Wednesdays, when the concentration of Xe-135 in the fuel had reached saturation and control rod operations due to automatic operation of KUR were minimal.

RESULTS:

Figure 1 shows measured time-sequence data. Analysis of the time-sequence data were carried out using the variance-to-mean method in an attempt to derive the prompt neutron decay constant (β_{eff}/l). As shown in Table 1, it is found that the variance-to-mean analysis could not be successfully performed for any of the measurement datasets obtained from CN-1 and B-3. The calculated values were obtained using MVP3 [4] with JENDL-5 [5], and account for the average burnup. Further analyses using the Rossi- α method and the autocorrelation function method are planned for future work.

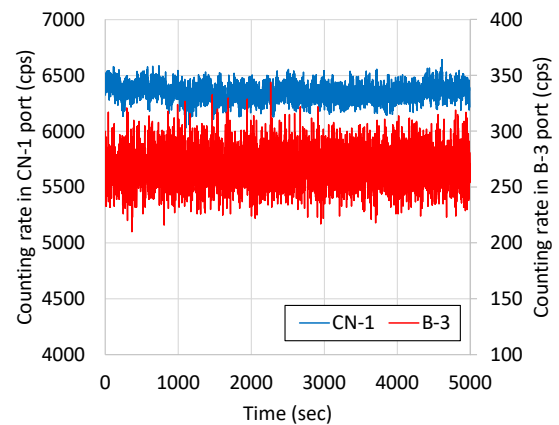


Figure 1 Measured time-sequence data

Table 1 Obtained prompt neutron decay constant

CN-1	B-3	Calc.
48.3 (/s)	Non	114.3 (/s)

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Characterization of Spent Nuclear Fuel Discharged from Kyoto University Reactor

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INTRODUCTION: Nuclear properties of a spent fuel are determined by its isotopic compositions. They are often evaluated based on the burn-up of the fuel. The passive γ ray measurement (PG) has been considered effective to confirm the burn-up, as well as the radiation dose and the radiation toxicity, etc. In this work, the PG was tested for a spent fuel element discharged from Kyoto university reactor (KUR).

EXPERIMENTS: Spent fuel element FL-14 discharged from KUR 4.8 years ago was stored vertically in a rack equipped on the bottom of the spent fuel pool. The burn-up of FL-14 is 24 GWd/t. The air environment above the pool is shielded from γ ray exposure from FL-14 by layer of light water with 5.2 m in thickness. For PG with a HPGe placed above the pool, a pipe was immersed in the water as shown in Fig. 1. The lower end of the pipe was plugged so that inside the pipe was filled with air. γ rays only pass through the pipe can be counted. The inner diameter of the pipe was 28 mm to keep the radiation dose at the top end of the pipe to be around 2 μ Sv/h.

RESULTS: In Fig. 2, measured spectra are shown. The room back spectra was measured with the HPGe placing 1 m apart from top of the pipe. No fission product (FP) γ ray was measured, proving the shielding capability of the light water layer. Above the pipe, γ rays from FPs, ^{137m}Ba , ^{134}Cs , $^{152,154}\text{Eu}$, ^{106}Rh and ^{144}Pr were measured as well as those from ^{60}Co . The total counting rate of γ rays was 2.7 kcps. One of the difficulties to determine the burn-up of FL-14 by the activity ratio of $^{134}\text{Cs}/^{137}\text{Cs}$ is less peak to base ratio of 605 keV γ ray from ^{134}Cs . That might be due to detection of scattered components of 662 keV γ rays from ^{137m}Ba . In the next measurement, we will try to store fuel elements horizontally to reduce the component. Since the cooling time after the discharge was within a several years, we can easily measure γ rays from $^{106}\text{Ru}(T_{1/2}=1.02\text{ y}) \rightarrow ^{106}\text{Rh}(T_{1/2}=30\text{ s})$ and $^{144}\text{Ce}(T_{1/2}=285\text{ d}) \rightarrow ^{144}\text{Pr}(T_{1/2}=7.2\text{ m})$. ^{106}Rh is known for its radiation toxicity so reprocessing of a spent commercial fuel assembly is prohibited before cooling time of 15 y. Quantification of the toxicity by measuring the γ ray would be useful to allow the cooling time reasonably shorter.

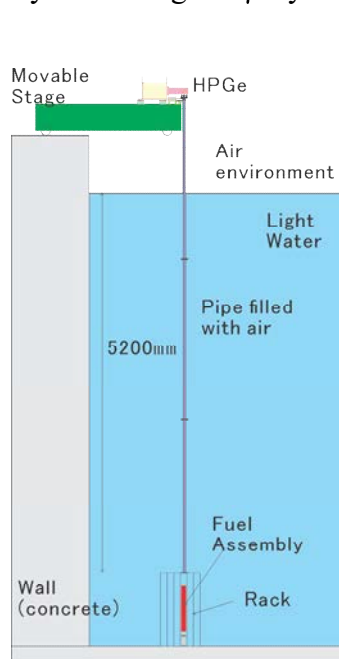


Fig. 1 Spent fuel pool and measurement geometry.

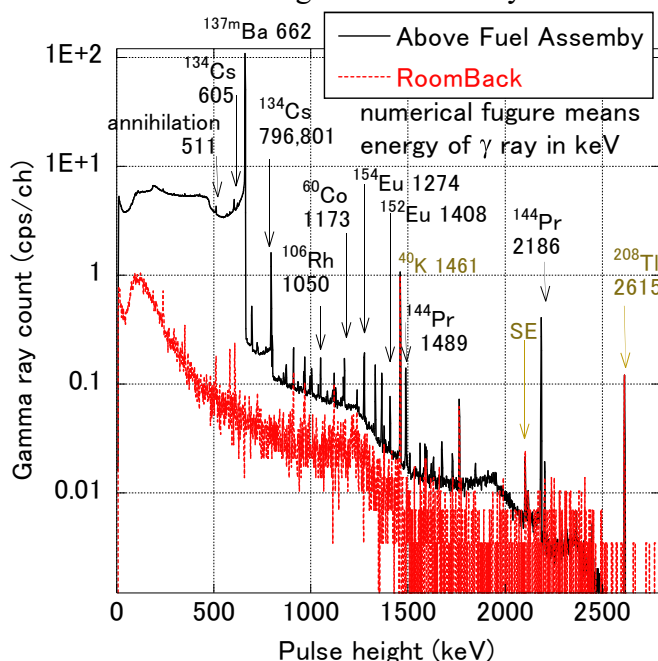


Fig. 2 Measured γ ray spectrum.

Neutron flux measurement in sub-pile room for evaluating KUR facility activation

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INTRODUCTION: For the decommissioning of the KUR, it is important to evaluate the amount of radio activation materials such as the experimental equipment, the biological shield and so on. In general, the evaluation of the activation dose is mainly based on numerical calculations. In numerical calculations, the neutron spectrum and absolute neutron flux are important input information. However, the absolute values often differ by several 10% or more between the calculated and measured values. With that background, the research project has been conducting neutron flux measurements on the surface of biological shields and constructing a numerical calculational model [1]. Furthermore, neutron flux measurements required for radiation evaluations related to decommissioning have not been conducted at nearly all reactors, including research reactors. With this background in mind, this study has commenced neutron flux measurements on the surface of the KUR biological shielding and within the fuel storage facilities in the KUR reactor core tank using the foil activation method or neutron detectors, starting in fiscal year 2022. In the 2025FY, a neutron flux measurement was conducted in KUR sub-pile room.

EXPERIMENT: Neutron flux measurement in the sub-pile room were using by Au foils. One Au foil sample was 10 mm in diameter and 0.05mm thickness (approximately 75 mg). The foils were placed at 4 locations around the primary cooling water outlet piping in the sub-pile room shown in Fig.1. Neutron irradiation was conducted from Oct. 29 to 30 in 2025. KUR has been operated with 5MW in the period. After the irradiation, a gamma ray (411 keV) emitted from Au-198 were measured using a Ge detector.

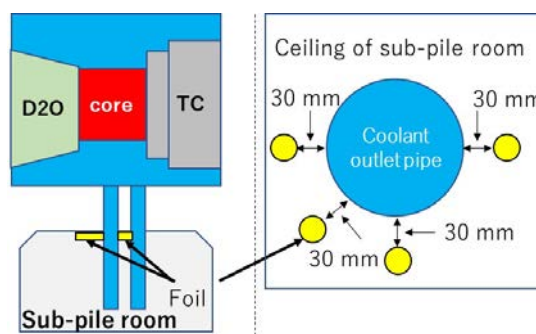


Fig.1 Location of Au foils setting

RESULTS: The averaged counting rate was 0.028 (cps) by the Ge detector. Where the all samples were measured simultaneously. The neutron flux was evaluated based on the measurement under the following assumptions:

- 1) The neutron flux was assumed to be thermal neutron flux, and self-shielding due to resonance absorption was neglected.
- 2) Attenuation of the neutron flux within the sample was neglected.
- 3) Perturbation of the neutron flux due to the placement of the sample was neglected.
- 4) The neutron temperature was assumed to be room temperature and the neutron capture cross section of Au-197 was 98.69 (b).

As the results, the thermal neutron flux was 1.94×10^4 ($n/cm^2/sec$) and the calculated thermal neutron flux in the sub-pile room was 3.0×10^4 ($n/cm^2/sec$) using PHITS2 [2] with JENDL-5 [3]. The experimental and calculated values agreed by an order of magnitude. In future work, we plan to perform a neutron flux evaluation without relaxing the above assumptions.

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Radiation Effects on the Piezoelectric Response of PZT under 30 MeV Electron Irradiation

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INTRODUCTION: In this study, we investigate the potential application of piezoelectric elements for radiation measurement. The electromechanical coupling coefficient k — calculated from the resonant and antiresonant frequencies — is used as a key performance metric for these elements. Previous work reported changes in k when PZT elements were directly irradiated with high-energy Xe ion beams [1–3]. To clarify how k in PZT depends on radiation type, we are conducting irradiation experiments with a high-energy electron beam at KURNS-LINAC [4]. Because the k value of PZT tends to decrease above a certain temperature, continuous measurements under 20 MeV electron irradiation were performed on a single PZT element while minimizing thermal effects in fiscal years 2023–2024. Those results showed a monotonic decrease in k proportional to cumulative electron dose, with the reduced k persisting after irradiation ceased [5].

EXPERIMENTS: In the 2025 experiments, the electron energy was increased from 20 MeV to 30 MeV to investigate the effect of energy variation on the k value of PZT elements. Two campaigns were carried out (July and December 2025), each lasting three days, during which a 30 MeV electron beam irradiated PZT piezoelectric elements. Beam current was adjusted to keep the element surface temperature within a range where thermal effects could be neglected. The PZT elements were disk-shaped (radius 9 mm, thickness 1 mm), identical in specification to those used previously. Surface temperature was monitored continuously with thermocouples during irradiation. Each irradiation lasted approximately 15 minutes; frequency–impedance measurements were performed only after the element surface temperature had returned close to room temperature. Measurements were taken six times per day to obtain sufficient data points.

RESULTS: Irradiation of a single PZT element with a 30 MeV electron beam over several days produced a monotonic decrease in k with cumulative dose. The reduced k persisted during irradiation interruptions. Comparison with the 20 MeV data indicates a different rate of k decrease for the same cumulative dose. Future work will include additional experiments varying electron energy to examine these differences in detail.

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Characterization of Ni-Si nanoparticles precipitated in Cu-Ni-Si alloy using Small-Angle X-ray Scattering

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INTRODUCTION: Cu-Ni-Si alloy is renowned precipitation-strengthened Cu alloys and is widely used as electrodes in electronic devices. Recent progress of miniaturization in the electronic devices requires further strengthening and higher conductivity for the Cu-Ni-Si alloy. In this alloy, Ni-Si nanoparticles are precipitated in Cu matrix and improve the mechanical strength. In contrast, solute Ni and Si reduce the electric conductivity. Therefore, the precise control of precipitation by aging heat treatment is an important issue. Small-angle X-ray scattering (SAXS) can quantitatively characterize the shape and amount of such nanoscale precipitates in alloys and provide useful knowledge for precipitation mechanism [1,2]. Therefore, the SAXS measurements of the Ni-Si precipitates in Cu-Ni-Si alloy was conducted in this study.

EXPERIMENTS: The Cu-Ni-Si alloy was first solution-treated and then aged at the temperature between 623 and 773 K. The SAXS measurements were performed using the SAXS instrument (NANO-Viewer, Rigaku) with Mo K α radiation. Scattering patterns were measured using a photon-counting-type two-dimensional detector (PILATUS 100k, Dectris) using a Si converter with the thickness of 1000 μm . The X-ray path from the X-ray entrance slit to the window just before the detector was evacuated to reduce the background scattering from air. The SAXS measurements were performed using two sample-to-detector distances of 0.4 and 1.7 m to cover wider q range, where q is the magnitude of the scattering vector. The scattering patterns were reduced using our original program worked in Igor Pro (WaveMetrics).

RESULTS: Fig. 1 shows the SAXS profiles of the Cu-Ni-Si alloys before and after aging heat treatment. The scattering intensity of the aged sample increases and has a shoulder compared to that of the sample before aging. This contribution is the scattering of the Ni-Si nanoparticles. Because of the recent improvement of the SAXS instrument, the q minimum of the profiles was extended from 0.14 nm^{-1} to about 0.07 nm^{-1} . The present results clearly show that the improvement is beneficial to cover whole the q range of the scattering of the Ni-Si nanoparticles. We will proceed the analysis of size and amount of the Ni-Si nanoparticles.

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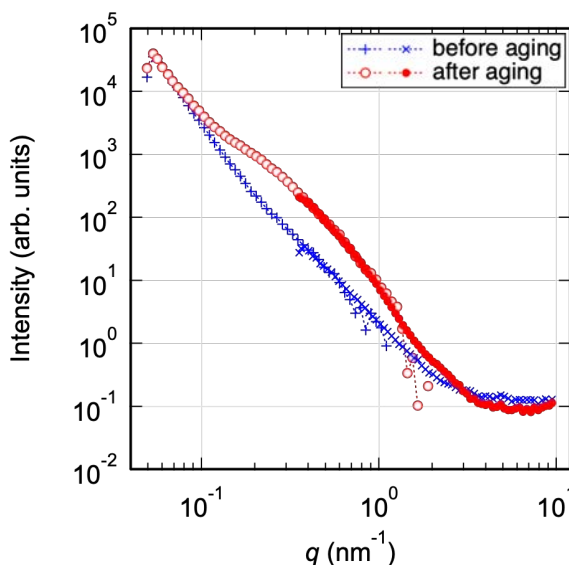


Fig. 1. SAXS profiles of Cu-Ni-Si alloys. Crosses and circles indicate the alloys before and after aging heat treatment, respectively.

Electrochemical reduction of Fe-containing spinel electrodes for lithium-ion secondary batteries

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INTRODUCTION: In recent years, lithium-ion secondary batteries expand to a wide range of applications, from portable electric appliances to automotive batteries. Spinel-type $\text{Li}_4\text{Ti}_5\text{O}_{12}$ has been practically used as a strain-free insertion negative electrode material. This material exhibits three-dimensional Li-ion diffusion through vacant sites in its structure, suppressing the formation of SEI (solid electrolyte interface) films on the electrode that increase electrical resistance. Therefore, we have focused on a similar structure and examined LiFeTiO_4 (LFTO), which operates at approximately 2 V against Li metal. Previous research has shown that LFTO has an inverse spinel-type structure with an ion-distribution of $[\text{Li}_{0.5}\text{Fe}^{3+}_{0.5}]^{8a}[\text{Li}_{0.25}\text{Fe}^{3+}_{0.25}\text{Ti}^{4+}_{0.5}]_2^{16d}\text{O}_4$ [1]. Furthermore, a significant decrease in discharge capacity has been observed from the first to the second cycle in liquid-type Li-ion secondary batteries using LFTO. However, the reason remains unclear. Therefore, LFTO was prepared under different sintering conditions, and the relationship between the crystal structure of LFTO and its electrochemical properties was investigated from the changes in crystal structure under each charge-discharge state.

EXPERIMENTS: The sample was prepared by conventional solid-state reaction. As starting materials, Li_2CO_3 , Fe_2O_3 and TiO_2 (anatase) were used and mixed by ball-milling for 30 min at 400 rpm and followed with sintering at 1000 K for 12 h and 1273 K for 6 h in air. The obtained samples were characterized by powder X-ray diffraction and electrochemical testing. As anode and electrolyte, the lithium metal and 1 M LiPF_6 in EC-DMC (3:7) were used respectively.

RESULTS: Mössbauer spectroscopy was performed to determine the oxidation state of Fe in LFTO (1273K for 6h) (Fig. 1). Oxidation states and coordination numbers were obtained from isomer shifts (IS) and quadrupole splitting (QS) by fitting for three different Fe components. The results showed that approximately 70% were trivalent in the octahedrons, and the remainder were divalent, occupying roughly equal proportions in the tetrahedrons and octahedra. The presence of some Fe^{2+} suggests the formation of oxygen vacancies. It is also possible that all Fe^{3+} in the tetrahedrons were reduced to Fe^{2+} during sintering and some moved from the tetrahedron to the octahedron sites.

The initial discharge capacity was 68% of the theoretical capacity, which corresponds to the amount of all Fe^{3+} reduced to Fe^{2+} by discharge. On the other hand, the LFTO at 1073 K for 12 h showed an initial discharge capacity was 58% of the theoretical capacity. Rietveld analysis showed the Fe occupancy in the tetrahedron of the former was 0.542(2), while that of the latter was 0.520(2), showing a slight difference in the Fe occupancy in the tetrahedron, and it was found that the high sintering temperature resulted in a 2% higher occupancy compared to the low one. Therefore, it was revealed that while all Fe^{3+} contributes to discharge, slight differences in Fe occupancy within the tetrahedron result in different electrochemical properties.

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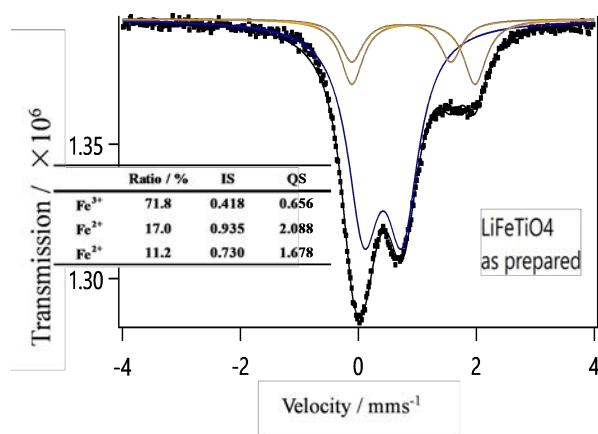


Fig. 1 Mössbauer spectrum of LiFeTiO_4 at 300 K prepared by sintering at 1273 K for 6 h.

Effects of lead addition on tritium retention behavior in advanced fusion materials

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INTRODUCTION: Tritium will be produced by (n, α) reaction with lithium (Li) in the fusion reactor because the amount of tritium in nature is limited. Tritium breeder requires good chemical stability and ease of tritium recovery. Lithium silicate (Li_4SiO_4) has good chemical stability and good crushing load. Advanced tritium breeder materials should have both of higher tritium production rate and recovery efficiency. Lead (Pb) is a neutron multiplier and has lower affinity with tritium that contributes to the improvement of the tritium breeding ratio. The addition of Pb into Li ceramics is expected to have a potential advantage for the establishment of efficient tritium recovery system.

EXPERIMENTS: LSO-x wt.%Pb, was prepared at Wuhan University of Technology. Those samples were irradiated by neutron at Kyoto University Research Reactor (KUR) with the neutron fluence of $8.0 \times 10^{15} \text{ n cm}^{-2}$ and $8.0 \times 10^{16} \text{ n cm}^{-2}$ (1MW 4h and 5MW 1h). After the neutron irradiation, tritium release behavior was evaluated by Tritium-thermal desorption spectroscopy (T-TDS) system by heating the samples separately from R.T. to 1113 K with the heating rates of 10, 20, and 30 K min^{-1} . In T-TDS, HT was oxidized to HTO using copper oxide, and all tritium species released by heating, including HT and HTO, were captured in a water bubbler. Liquid scintillation counter (LSC) was used to measure the total T amount trapped by the water bubbler at Shizuoka University.

RESULTS: Fig. 1 show the HTO and HT desorption rates of pure LSO at heating rate of 10 K/min, 20 K/min and 30 K/min. Fig. 2 show the HTO and HT desorption rate of LSO-10 wt.%Pb, heating rate are 10 K/min, 20 K/min and 30 K/min. The desorption peak of HTO consisted of a single peak, whereas three peaks were observed for HT at around 400 K, 600 K, and 800 K. These differences were considered to be caused by variations in the trapping sites. Also, it was observed that as the heating rate increases, the peak temperature was shifted toward the high-temperature side. When compared with the desorption peaks of pure Li_4SiO_4 ceramics, the present desorption peak was markedly shifted to a lower temperature, which lead to conclude that lead addition improve the efficient tritium reconvery at lower temperature.

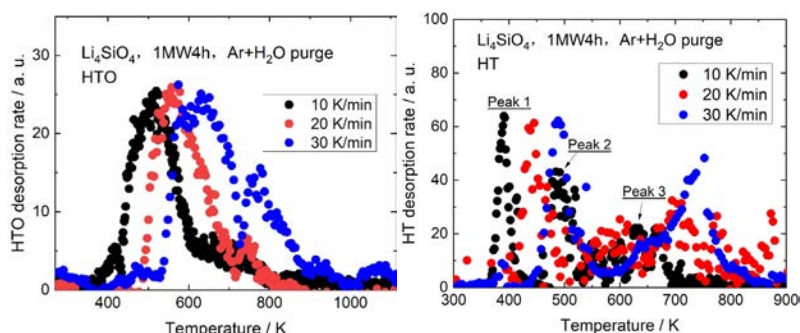


Fig.1 T-TDS spectra of Li_4SiO_4 (Left: HTO, Right: HT)

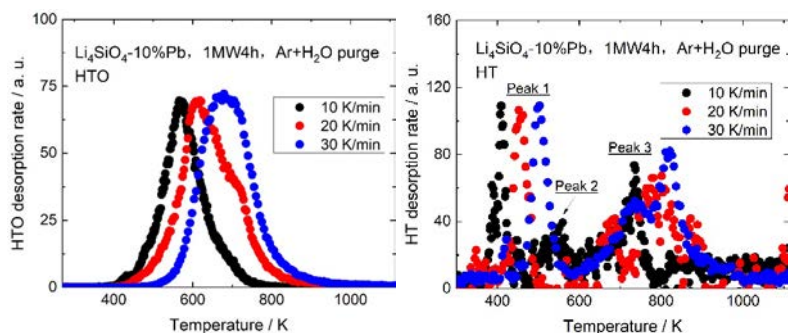


Fig.2 T-TDS spectra of Li_4SiO_4 -10%Pb (Left: HTO, Right: HT)

Point defect and impurity effect on mechanical property and irradiation damage evolution in $\text{Cr}_{0.8}\text{FeMn}_x\text{Ni}_y$ concentrated solid solution alloy

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INTRODUCTION: It has been reported that some concentrated solid solution alloys (CSAs) and high entropy alloys (HEAs) have unique properties such as a high radiation resistance and corrosion resistance at elevated temperatures compared with conventional nuclear component materials [1-2]. This attractive property could make CSAs and HEAs candidates for high temperature nuclear reactor components. In this study, we focused on microstructure and mechanical property in FCC-type CSAs and an HEA in order to better understand the effect of point defect and impurity on mechanical property and irradiation damage evolution.

EXPERIMENTS: The materials used in this study are FCC-type single phase $\text{Cr}_{0.8}\text{FeNi}_x\text{Mn}_y$ alloys, prepared by arc-melting or induction furnace in high-purity argon atmosphere, followed by solution annealing at 1160 °C for 24 hrs. For the estimation of the stacking fault energy (SFE), samples were cold-rolled down to 0.25 mm in thick, then punched out for tensile test. The in-situ irradiation experiment was performed using the multi-beam High Voltage Electron Microscope at Hokkaido University. All the TEM samples were electron-irradiated at 500 °C up to 1 dpa.

RESULTS: The estimated SFE values from 5%-deformed $\text{Cr}_{0.8}\text{FeNi}_x\text{Mn}_y$ alloys seemed to increase as a function of Ni and Mn concentration. In addition, the tensile test revealed that the yield strength, the elongation, and even hardening rate were increased with increasing Ni and Mn concentration. Furthermore, $\text{Cr}_{0.8}\text{FeNi}_x\text{Mn}_y$ alloys including high C and N indicated a higher yield strength and a longer elongation compared to the high purity $\text{Cr}_{0.8}\text{FeNi}_x\text{Mn}_y$ alloys. Electron irradiation experiment at 400°C showed the formation of black dots and frank loops (FLs), but no cavities in $\text{Cr}_{0.8}\text{FeNi}_x\text{Mn}_y$ alloys (Fig. 1). The decrease in FL number density with increasing Ni and Mn concentration was observed, but no obvious rela-

tionship with loop size. Furthermore, $\text{Cr}_{0.8}\text{FeNi}_x\text{Mn}_y$ alloys including high C and N tended to have a higher FL number density and smaller size.

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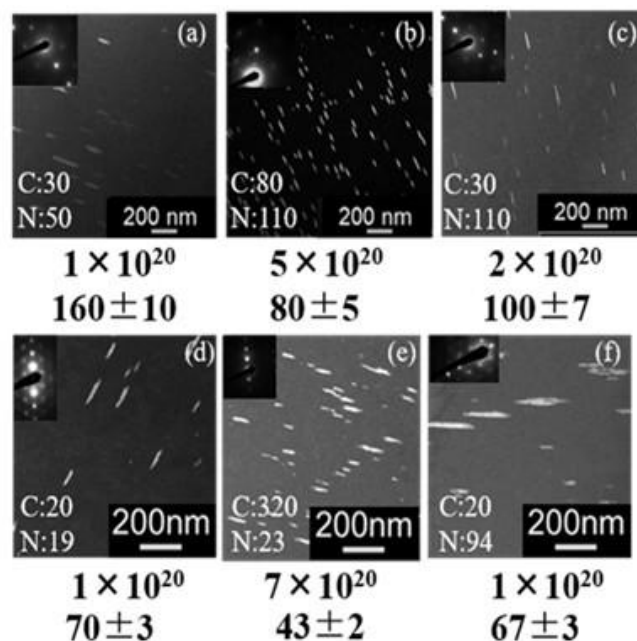


Fig.1 Dark field relrod image of Cantor alloys (a-c) and $\text{Cr}_{0.8}\text{FeMnNi}$ alloys (d-f) electron-irradiated at 400 °C to 0.2 dpa. Impurity concentration (wppm) is shown in the lower left corner. Loop number density (m⁻³) is shown in the first line at the bottom outside the image, and mean loop diameter (nm) is shown in the second line.

Controlled Interfacial Polymerization Polyamide/Polysulfone Membrane Probed by Slow Positron Beam

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INTRODUCTION: Polyamide/Polysulfone nanofiltration membranes have been widely used in water treatments due to their high efficiency, low energy consumption [1]. The surface charge, pore size and its distribution in the polyamide layer play an important role in membrane selectivity. A more uniform polyamide layer might be prepared by controlling the interfacial polymerization reaction rate[2]. In this study, TMC/hexane solution was slowly added to the piperazine (PIP) solution through a controlled injection. And the obtained composite membranes have been investigated via positron annihilation techniques coupled with a positron beam.

EXPERIMENTS: 10 ml PIP aqueous solution was dropped onto the PES substrate. Then 5 ml pure hexane was added. After 1 min, TMC/hexane solution was added to the pure hexane with an injection pump at a constant speed. The total releasing and reaction time of TMC solution was 2 min, 3 min, 4 min and 5 min, respectively. The prepared samples were named as IP-2, IP-3, IP-4 and IP-5, respectively. Positron measurements were performed with the position beam system at the Institute for Integrated Radiation and Nuclear Science, Kyoto University.

RESULTS: As shown in Fig. 1, the *S-E* curves of the four membranes reveal the differences in their layer structures, despite exhibiting a similar shape. The difference in *S* parameters in the low incident energies suggests structural differences within the polyamide layers. Moreover, the results of the o-Ps lifetimes and the molecular weights of polyamide by GPC are presented in Table 1. It is interesting to find that the decrease in the reaction rate leads to a smaller free-volume hole size (shorter o-Ps lifetime) and lower Mw/Mn, suggesting that longer injection time should lead to a narrower distribution of molecular weight. Since the injection rate was the sole variable in the preparation process, the reaction rate would be responsible for the different structures of the polyamide layers. These findings suggest that controlling monomer release rates could promote more uniform and complete crosslinking reactions.

Table 1 The o-Ps lifetime and molecular weight of polyamide

	o-Ps life-time [ns]	o-Ps intensity[%]	Mn	Mw	Mw/Mn
IP-2	1.60	23.5	12945	3292	79.7
IP-3	1.54	28.7	12705	4117	75.5
IP-4	1.51	30.0	12024	1930	52.0
IP-5	1.27	26.4	10060	7892	56.0

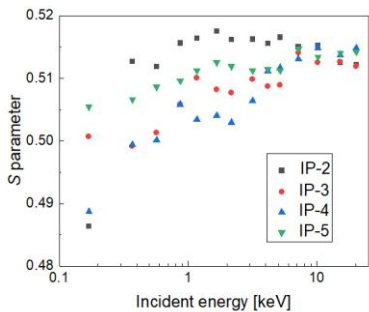


Fig. 1 *S-E* plots of the samples

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Complex Structure of Ions Coordinated with Hydrophilic Polymer 26. "Diffusion-induced Orientation" Observed in PA6 and Amphiphilicity in Iodine-doped Polymers Considering Dynamic Behavior of Polyiodides.

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INTRODUCTION: At first, interacted structuring between polyamide 6 (PA6, NylonTM-6) and iodine (polyiodides, I_n^{m-} , "PolyIod" as below) had been considered in discussion of structural transition of PA6 crystal (α -phase) or conversion of hydrogen bonding in PA6 crystallites.[1] However, while preparation of the samples is not so difficult and WAXD patterns are also observed explicitly, structure analysis and quantitative reproduction have not been concluded even in half century after the first report.

One of characteristic and unexpected behaviors observed in "iodine-doping" for PA6 is "diffusion-induced orientation" which is considerably unique as modification of orientation in mesoscopic scale achieved (almost) simultaneously with rapid ionic diffusion and coordination in PA6 crystallite; "diffusion-induced orientation" is almost simultaneously observed both in SAXS range of X-ray diffraction and in WAXD for crystallite structure.[2]

And, additionally, iodine-coordinated ("iodine-doped") structures are activated and/or modified by environment (ionic diffusion responding with humidity, modified abnormal intercalation,, etc.) not only during initial diffusion of "PolyIod" into PA6 matrix but also after "iodine-doping"; easy ionic exchange activated in iodine-doped PA6 or other matrices is applied for hybrid nano-composite without synthesis nor melting nor casting for matrices.

RESULTS AND DISCUSSION: The author regards that lots of unconscious factors complicate results and that interaction invisible as chemical structure of "PolyIod" molecules plays intrinsic roles on all behavior through nano and mesoscopic scales. For example, utilizing activated ionic diffusion deeply through "iodine-doped" PA6 matrix and ionic exchange *a posteriori* ("secondary doping" of inorganic salts as nano filler or "as shaped" hybrid composite).

Arguing PA6, "diffusion-induced orientation" observed on "(1st) iodine doping" to PA6 matrix may be a singular case depending on relation between lattice constants (symmetry) provided by PA6 α -crystallite and "PolyIod".[3] However, rapid diffusion and modified orientation suggest self organization provided by "iodine bonding" and dynamics (coupling and decoupling) between "PolyIod"'s under anisotropic coordination layout.

These results suggest *dynamic interaction and structures* for both (guest) "PolyIod" and (host) polymer matrices.[4] While direct experimental observation of suggested models may not be easy, such hypothesized views can reasonably explain unexpected experimental issues previously reported.

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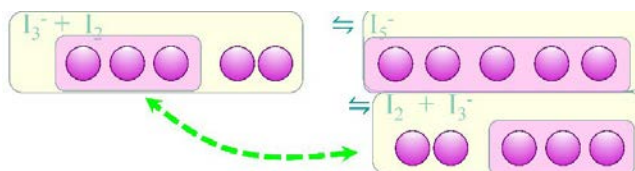


Fig. 1. example of dynamic coupling and decoupling (linear) models between "PolyIod" to explain amphiphilicity and "diffusion-induced orientation"; linear or L-shaped self coordination between "PolyIod" is induced by "iodine bonds" as halogen bonding or "σ-hole".

Correlation between the spin-relaxation of $^{111}\text{Cd}(\leftarrow^{111}\text{In})$ in glycerin solution and its viscosity

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INTRODUCTION: Quasi-droplets, extremely tiny water droplets as small as 1/10,000th the thickness of a human hair, show various functions differently from normal water [1], but the exact mechanism behind their functions or even the basic properties, such as internal pressure, pH, and viscosity, are still unknown because of their smallness. Radioactive nuclei can be a good probe for such extremely small substances [2]. Steffen demonstrated the applicability of radioactive nuclei to the measurement of viscosity [3]. Still, their result is unreliable due to insufficient time resolution and limited knowledge of the ion status in the solvent. This time, we studied the correlation between the viscosity and the spin relaxation of $^{111}\text{Cd}(\leftarrow^{111}\text{In})$ using time differential perturbed angular correlation (TDPAC) at higher time resolution and under controlled conditions, intending the application to the studies of quasi-droplets.

EXPERIMENTS: Typical four-counter TDPAC measurements were performed for the 171-245 keV cascade in $^{111}\text{Cd}(\leftarrow^{111}\text{In})$ in the samples of glycerin aqueous solution of viscosity η within the range of 0.5~4000 mPa·s. The viscosity of each sample was controlled by changing its temperature and density. The pH of each sample was maintained below 3 by adding dilute HCl_3 to eliminate the possible interference to the spin relaxation pattern caused by the multiple formation of ligand at the intermediate pH [4].

RESULTS AND DISCUSSION: Observed $G_{22}(t)$ spectra consist of two components, a fast relaxation caused by the after-effect and a slow relaxation after 60 ns. The slow component was dominant at lower viscosities up to several tens mPa·s, while the fast component was dominant at higher viscosities. A simple exponential function was applied to the slow component of each $G_{22}(t)$ to evaluate the decay constant of spin relaxation, λ . Even though the obtained values of λ were ten times smaller than those reported by Steffen, they were consistent with the models of lower η proposed by Abragam [5] and higher η by Marshall [6]. The correlation between λ (sec^{-1}) and η (mPa·sec) can be expressed in a simple function (Fig.1):

$$\lambda = \frac{2.4 \times 10^5 \eta}{1 + 1.3 \times 10^{-3} \eta^2}$$

With the present result, we expect that the viscosity of quasi-droplets can be determined by the TDPAC method once a method of introducing ^{111}In to the quasi-droplets is developed.

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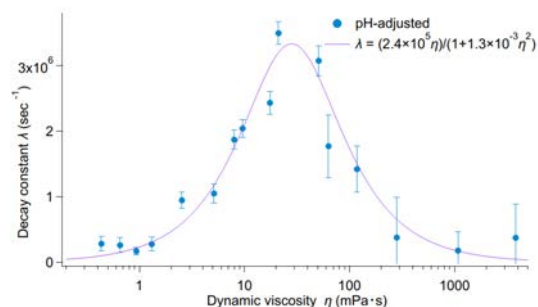


Fig. 1. Correlation between the decay constant of spin relaxation and the viscosity of the solution.

Deformation of Local Structures in Hafnium Metal Induced by Hydrogen Absorption

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INTRODUCTION: Hafnium (Hf) metal is a well-known material as a control rod in a nuclear reactor due to the large neutron-capture cross sections of some of the isotopes. Because the control rods are exposed in high-temperature and high-pressure cooling water for a long period, however, hydrogen atoms arising from the coolant are absorbed into the lattice of the metal, which causes the formation of lattice defects leading to the embrittlement of the material such as a cracking of a cladding tube for the control rods. In order to prevent nuclear accident, it is important to understand the characteristics of the hydrogen-induced lattice defects as well as the thermal behavior of hydrogen impurities themselves in Hf. In the present work, we have tried to observe the effect of hydrogen absorption on the local structures using two different nuclear spectroscopic techniques: perturbed angular correlation (PAC) spectroscopy with the $^{181}\text{Hf}(\rightarrow^{181}\text{Ta})$ probe and positron annihilation lifetime spectroscopy (PALS). Here, we report preliminary results on the effect of hydrogen absorption into the lattice of Hf matrix.

EXPERIMENTS: A Hf metal plate ($5 \times 5 \times 0.5 \text{ mm}^3$) of a purity of about 96.4% with 3.6% Zr impurity was irradiated with thermal neutrons in the research reactor of Kyoto University (KUR) to produce the $^{181}\text{Hf}(\rightarrow^{181}\text{Ta})$ probe, and TDPAC measurements were performed by detecting the 133-482 keV cascade γ rays with the intermediate level of $I = 5/2$ having a half life of 10.8 ns. Apart from the PAC experiment, PALS measurements were performed for a pristine and hydrogen-absorbed Hf plates at room temperature using a positron source of ^{22}Na . The PALS spectra were analyzed with a fitting program PALSfit [1]. For the absorption of hydrogen atoms into Hf lattice, we adopted an electrolytic reaction of water in 0.5 M H_2SO_4 aqueous solution by applying a constant current of 1.5 A cm^{-2} for 90 min to a Hf plate as the cathode. The detail of the electrolysis is described in Ref. [2].

RESULTS: PALS measurements disclosed the effect of hydrogen absorption on the local structure in the Hf lattice, demonstrating enlargement of vacancy clusters, which signifies the change in the local structure of the lattice. As shown in Fig. 1, PAC measurements reveal that the probe occupies only a single site in the pristine Hf metal, but another component was observed in the PAC spectrum for the hydrogen-absorbed Hf, showing local deformation by hydrogen insertion. Both the results of spectroscopies are consistent. For further discussion on the hydrogen effect, detailed analyses are underway.

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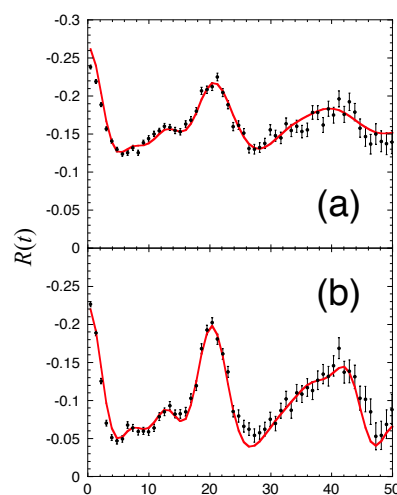


Fig. 1. PAC spectra of the $^{181}\text{Hf}(\rightarrow^{181}\text{Ta})$ probe (a) in a pristine and (b) in a hydrogen-absorbed Hf plate.

Study on ^{99m}Tc Separation/Concentration Technology from ^{99}Mo by (n, γ) Method (4)

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INTRODUCTION: Previous studies have demonstrated the successful recovery of recoil “hot atoms” from irradiated β - MoO_3 targets, indicating that the extraction efficiency of ^{99}Mo increases continuously over time [1]. A key objective of the present study is to evaluate the recyclability of the target material following the aqueous extraction process. To investigate this, β - MoO_3 powder from previous experiments which had been previously immersed in water for extraction and subsequently dried, was re-irradiated to assess its ^{99}Mo production capacity and structural stability. Furthermore, this research expands the scope of investigation to evaluate the extraction efficiencies of alternative molybdenum oxides, such as Mo_4O_{11} .

EXPERIMENTS: The hydrated sample was prepared by immersing β - MoO_3 in water at 80°C for 12 hours. The dehydrated counterpart was subsequently obtained by calcining the hydrated material in a furnace at 280°C for 2 hours. For comparison, Mo_4O_{11} was synthesized from MoO_2 under an argon atmosphere at 600°C for a duration of three days.

All samples were irradiated at the Kyoto University Reactor (KUR) with a thermal neutron flux of $3 \times 10^{13} \text{ n cm}^{-2} \text{ s}^{-1}$ for 20 minutes; these parameters were selected to remain consistent with established irradiation conditions from previous studies [1]. Following irradiation, the powders were dispersed in distilled water and stirred for 5.5 hours at room temperature to facilitate the aqueous extraction of the recoil atoms. The final extraction yield of ^{99}Mo was determined by quantifying the induced radioactivity via gamma-ray spectrometry using a high-purity germanium (HPGe) detector.

RESULTS: The ^{99}Mo extraction efficiencies for the various molybdenum oxide samples are illustrated in Fig. 1. Among the tested materials, the dehydrated β - MoO_3 exhibited the highest extraction rate. In contrast, the hydrated sample showed a significantly lower extraction efficiency compared to the pristine β - MoO_3 phase. This suggests that the presence of lattice-bound water may serve as a barrier, inhibiting the migration of ^{99}Mo recoil atoms from the crystalline bulk to the grain surfaces. Measurements of commercial MoO_2 yielded low extraction values, consistent with previously reported data [2]. Moreover, the Mo_4O_{11} synthesized from the commercial MoO_2 precursor demonstrated negligible ^{99}Mo recovery. These results provide further evidence that the specific crystalline structure and oxidation state of molybdenum oxides are decisive factors in governing the mobility and subsequent aqueous extraction of hot atoms.

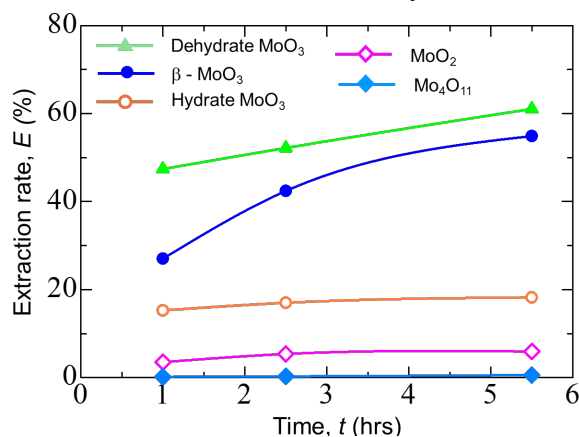


Fig.1 Extraction rate of ^{99}Mo compounds

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Investigation of Cd Occupation Sites in $\text{SrTi}_{1-x}\text{Cd}_x\text{O}_3$ by TDPAC Measurements

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INTRODUCTION: SrTiO_3 is one of the most promising candidates for photocatalytic materials. Trivalent cation doping at the Ti^{4+} site is known to be an effective approach for improving photocatalytic activity. Our previous study demonstrated that the addition of Cd in $\text{SrTi}_{1-x}\text{Cd}_x\text{O}_3$ enhances photocatalytic activity, which reaches a maximum at a Cd concentration of 4% and declines at higher concentrations. In addition, XRD measurements on $\text{SrTi}_{1-x}\text{Cd}_x\text{O}_3$ revealed a systematic increase in the lattice parameter with increasing Cd content, suggesting that Cd occupies the Ti site in the perovskite lattice. For the practical use of $\text{SrCd}_x\text{Ti}_{1-x}\text{O}_3$, it is necessary to obtain microscopic information on impurity sites. Therefore, for the study of the local fields at Cd sites, the TDPAC measurements were performed for $^{111}\text{Cd}(\leftarrow^{111\text{m}}\text{Cd})$ in $\text{SrTi}_{1-x}\text{Cd}_x\text{O}_3$ ($x=0.01, 0.02, 0.03, 0.04, 0.05$).

EXPERIMENTS: Stoichiometric amount of SrCO_3 , CdCO_3 , and TiO_2 powders was mixed in a mortar. The powders were pressed into disks. For TDPAC measurements, about 3 mg of CdO enriched with ^{110}Cd was irradiated with thermal neutrons at Kyoto University Research Reactor, and radioactive $^{111\text{m}}\text{Cd}$ was generated by the $^{110}\text{Cd}(n, \gamma)^{111\text{m}}\text{Cd}$ reaction. The neutron-irradiated CdO powder was dissolved in 6M HCl and added in droplets onto the pre-sintered $\text{SrTi}_{1-x}\text{Cd}_x\text{O}_3$ disk. The disk was sintered in air at 1373 K for 90 min. TDPAC measurements were carried out for the 151-245 keV cascade γ rays of $^{111}\text{Cd}(\leftarrow^{111\text{m}}\text{Cd})$ probe with the intermediate state of $I = 5/2$ having a half-life of 85.0 ns.

RESULTS: Figure 1 shows one of the TDPAC spectra of the $^{111}\text{Cd}(\leftarrow^{111\text{m}}\text{Cd})$ probe in $\text{SrTi}_{1-x}\text{Cd}_x\text{O}_3$ ($x=0.02$) at room temperature. The TDPAC spectrum was successfully reproduced by a two-component fit. From the fitting parameters, the first component exhibited a quadrupole frequency of $\omega_Q = 23.2$ Mrad/s, a fractional intensity of $f = 83\%$, and a Gaussian frequency distribution of $\delta = 77\%$, while the second component showed a quadrupole frequency of $\omega_Q = 49.3$ Mrad/s, a fractional intensity of 12%, and Gaussian distribution of $\delta = 0\%$. For the first component, the large distribution suggests that these Cd atoms reside at disordered lattice sites. In contrast, the second component exhibits no frequency distribution, indicating that Cd occupies a well-defined lattice site. The quadrupole frequency of this component is close to the value of 50.6 Mrad/s previously reported for ^{111}In at the Ti site in SrTiO_3 [1], suggesting that this component may originate from Cd substituting for Ti in the SrTiO_3 lattice. These results suggest that Cd partially occupies the Ti site in SrTiO_3 and may contribute to the enhanced photocatalytic activity.

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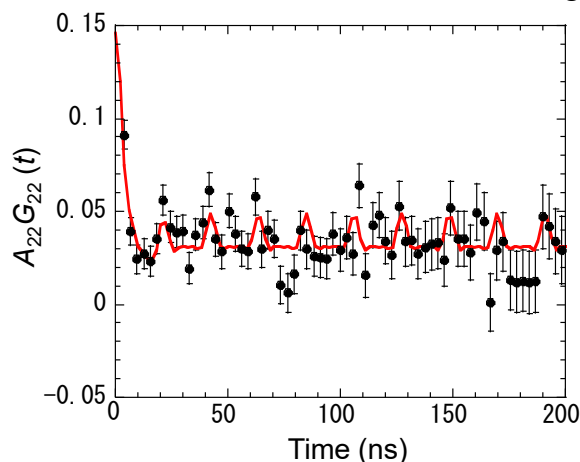


Fig. 1. TDPAC spectrum of $^{111}\text{Cd}(\leftarrow^{111\text{m}}\text{Cd})$ in $\text{SrCd}_{0.02}\text{Ti}_{0.98}\text{O}_3$ at room temperature.

Observation of Waveform of Wien Bridge Oscillator under Irradiation of Bremsstrahlung x-ray Produced by Pulsed Electron Beam

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INTRODUCTION: Field emitter arrays (FEAs) fabricated by contemporary thin-film and micro-fabrication technologies are expected to be used in harsh environments such as radiation fields, and several electronic devices were developed so far for this purpose [1-4]. It is important to confirm stable operation of the electronic circuits in radiation fields. Use of pulsed electron beam to produce pulsed x-ray is a good method to observe the response of the electronic circuit due to radiation. In this report, results of preliminary experiments to confirm the operation of an oscillator under radiation field are described.

EXPERIMENTS: In prior to experiments with FEAs, preliminary experiments were performed with a solid-state electronic circuit instead of vacuum devices. Two junction-type field effect transistors were used. Wien bridge oscillator of which oscillation frequency was approximately 100 kHz was prepared. The oscillator was installed in a capsule made of stainless steel. A battery was used to operate the oscillator. The sample was irradiated by Bremsstrahlung x-ray produced in platinum target under irradiation of 6 MeV pulsed electron beam with a pulse width of 4 μ s and repetition frequency of 1 to 100 Hz. The output of the oscillator was monitored with an oscilloscope via 15 m-long coaxial cable. To reduce the effect of the capacity of the cable, a large resistor was inserted, which reduced the amplitude of the observed signal.

RESULTS: Figure 1 shows a typical wave form of the oscillator. The x-ray was irradiated between -4 and 0 μ s. When the x-ray was irradiated on the sample, the amplitude of the oscillation signal was once reduced, and then recovered. However, such behavior was observed even when the oscillator was moved to the location apart from the target. When the cable that supplies the electric power from the battery was shortened, the reduction of the amplitude was not observed, as shown in Fig. 2. This response was found to be attributed not to the effect of x-ray on the circuit, but rather to that on the battery. From these results, it was suggested that the amplitude of the oscillator was affected by the electric signal which was generated by the accelerator power supply. Further investigation with an improved shielding will be necessary.

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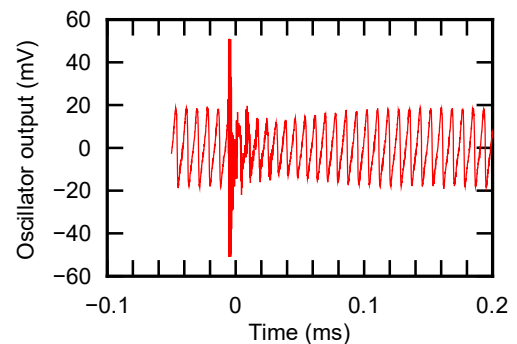


Fig. 1. Wave form of the oscillator under x-ray irradiation.

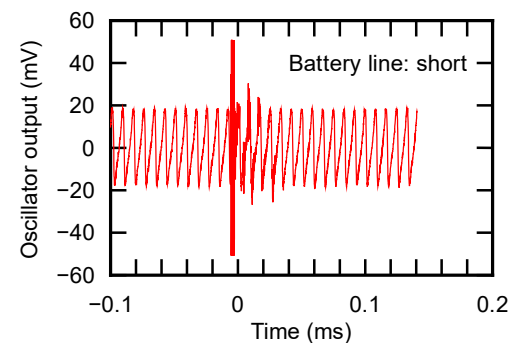


Fig. 2. Wave form of the oscillator with a shortened battery line.

Evaluation of neutron shielding properties and radioactivation of wood and wood composites.

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INTRODUCTION: Neutron shielding is typically achieved using hydrogen-rich materials such as water, polyethylene, and concrete, while boron is commonly used as a neutron absorber due to its large absorption cross-section. Because wood is mainly composed of carbon, oxygen, and hydrogen, wood-based materials can exhibit good neutron shielding performance. In this study, we developed wooden shielding material and evaluated the variability in shielding performance and gamma-ray dose during irradiation at the Heavy Water Neutron Irradiation Facility (HWNIF) in KUR.

EXPERIMENTS: Table 1 shows the materials prepared for neutron shielding test, which have a cross section of 90 × 90 mm and a thickness of 60 mm. Three Au foils were placed in front of and behind the specimen at the upper, middle, and lower positions along the specimen height. These specimens were irradiated with mixed-mode neutron radiation in the HWNIF for 10 min. Gamma rays from the activated Au foils were measured using a high-purity germanium detector, and shielding rates were obtained. Furthermore, the gamma-ray dose behind the specimen during irradiation was measured using a thermoluminescence dosimeter.

RESULTS: Fig. 1 shows the neutron shielding rates measured at different positions. Uniform shielding performance was observed in WS, a mixture of wood and boron, and its shielding rate was comparable to that of PE. This result indicates that WS can provide stable neutron shielding performance comparable to that of conventional shielding materials. Table 2 compares the gamma-ray dose emitted from the specimens. The gamma-ray dose emitted from SG was lower than that from PE. In contrast, WS showed the highest dose due to the 0.478 MeV gamma rays produced via the $^{10}\text{B}(n,\alpha)^7\text{Li}$ reaction. These results suggest that although WS demonstrates favorable neutron shielding capability, additional considerations are required regarding the gamma-ray dose generated during neutron irradiation. Future work will focus on optimization material design by considering the trade-off between shielding performance and radioactivation.

Table 1 List of materials used in this study.

Symbol	Material
PE	Polyethylene
SG	Air-dried sugi wood
WS	Wooden shielding material containing boron

Table 2 Gamma-ray dose from the irradiated specimens.

Material	Dose (Gy)
PE	0.108
SG	0.0849
WS	0.181

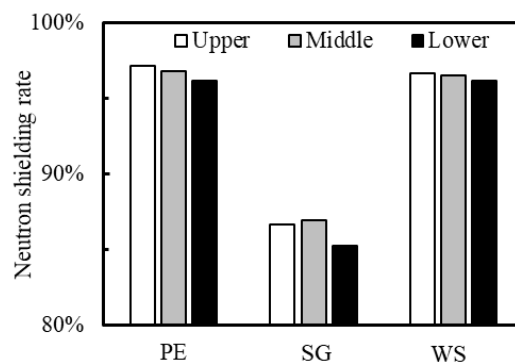


Fig. 1 Neutron shielding rates at given positions along the specimen height (Upper, Middle, Lower).

Slow Positron Probe Analysis of Fe-Cr Alloy Thin Films Subjected to Severe He Ion Irradiation

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INTRODUCTION: If nuclear fusion reactions can be controlled on Earth, an essentially limitless source of clean energy could be obtained. Currently, the design and construction of experimental and prototype reactors are underway, but numerous challenges remain. In tokamak-type reactors, the "blanket" responsible for energy conversion, is subjected to severe neutron irradiation, which alters various material properties during operation. Focusing on Fe-Cr alloys as candidate materials, their magnetism is directly linked to the electromagnetic forces applied to the reactor structure during plasma disruptions. Therefore, understanding the effects of irradiation on magnetism is essential; however, research has been limited due to experimental difficulties. Neutron irradiation experiments are costly and time-consuming, and the activation of samples requires hot laboratories. Alternatively, ion irradiation offers advantages such as shorter duration and non-activation, but the damage is confined to the surface region, complicating magnetic evaluation. To overcome this challenge, a method using ion irradiation of high-quality alloy thin films has been proposed, and research has progressed. To clarify the effects of nuclear transmutation helium, Fe-Cr alloys were subjected to severe helium irradiation, and their structure and magnetism were investigated, leading to the discovery of high-density cavity formation and magnetic hardening phenomena [1]. The latter was not observed in pure iron [2], suggesting that irradiation-induced changes in the atomic distribution of chromium are key. In this study, to gain further insight into irradiation damage structures, Fe-Cr alloy thin film samples were irradiated with helium ions, and defect evaluation was conducted using a low-energy positron beam.

EXPERIMENTS: Using ultra-high vacuum electron beam evaporation, 300 nm thick Fe-20%Cr films were grown on MgO(001) substrates. Helium ions with an energy of 50 keV were irradiated at room temperature using the microwave ion source at the Wakasa Wan Energy Research Center (WERC). The irradiation dose was set to 1.7×10^{17} ions/cm². Small pieces were cut out from the irradiated samples by FIB processing for cross-sectional TEM observation. Furthermore, Doppler broadening measurements were performed at energies up to 30 keV using the low-energy positron beamline, and the S and W parameters were evaluated.

RESULTS: The fabricated thin film was confirmed to be epitaxially grown with the orientation relationship BCC-Fe-Cr(001)[110]//MgO(001)[100]. In the positron annihilation experiment, the S parameter showed a significant increase in the energy range of 5 to 7 keV due to He ion irradiation. Cross-sectional TEM observations confirmed the formation of a high density of cavities with sizes of a few nanometers, and the increase in the S parameter is considered to reflect these nanoscale structural changes.

ACKNOWLEDGEMENT:

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Study on HPLC Elution Behavior of Heavy Lanthanide Metallofullerene

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INTRODUCTION: Lanthanide (Ln) metallofullerene (EMF): Ln@C₈₂ is a clathrate compound encapsulating metal atom in fullerene C₈₂ and known that two or three electrons are transferred to C₈₂ cage from the encapsulated Ln atom [1]. In the previous work, we succeeded to obtain the high-performance liquid chromatography (HPLC) elution behavior on a pyrenyl stationary phase for Ln@C₈₂ of La, Ce, Pr, Nd, Gd, Tb, Dy, Ho, and Er with a toluene as an eluent by the thermal neutron activation method [2]. On the other hand, the results obtained by analyzing long half-life nuclides such as Ce and Tb had problems in accuracy compared to other short half-life nuclides. In this study, we attempted to obtain data again for the long half-life nuclides Tb-160 and Ho-166, and at the same time attempted to improve accuracy by conducting HPLC development and analysis.

EXPERIMENTS: Ln@C₈₂s of Tb, Dy, and Ho were prepared by previously reported [2]. These purified samples were injected into a Buckyprep column using toluene as an eluent. The eluent from the column was fractionated every 20 seconds at room temperature (RT) and 40°C, and every 1 minute at 10°C. These fractions were evaporated to dryness, and re-dissolved in CS₂. These CS₂ solutions were dropped and dried on a filter paper with a diameter of 12 mm, and then sealed in a polyethylene bag. These samples were irradiated with thermal neutrons in the KUR of the Institute for Integrated Radiation and Nuclear Science, Kyoto university. After the irradiation, the γ rays emitted from the samples were measured by a Ge detector.

RESULTS: The figure shows HPLC chromatograms of Tb and Ho fullerenes developed on a pyrenyl stationary phase at 40°C, RT, and 10°C. The HPLC retention time of Ln@C₈₂ fullerene at each temperature were found to be 45.75 ± 0.02 min. at 40 °C, 55.55 ± 0.02 min. at RT, and 61.22 ± 0.03 min. at 10 °C for Tb@C₈₂, and 45.25 ± 0.02 min. at 40 °C, 54.99 ± 0.03 min. at RT, and 61.88 ± 0.03 min. at 10 °C for Ho@C₈₂, respectively. Based on the obtained retention time, the adsorption/desorption enthalpy of Tb@C₈₂ and Ho@C₈₂ to a pyrenyl stationary phase using toluene as an effluent was evaluated from van't Hoff plot and was found to be 8.07 kJ/mol and 8.08 kJ/mol, respectively. The adsorption/desorption enthalpy of Tb@C₈₂ estimated in the previous experiment was about 8.7 kJ/mol, but it is thought that there was a large error in the corrected value because data could not be acquired at the same time as Ho@C₈₂, which has a short half-life.

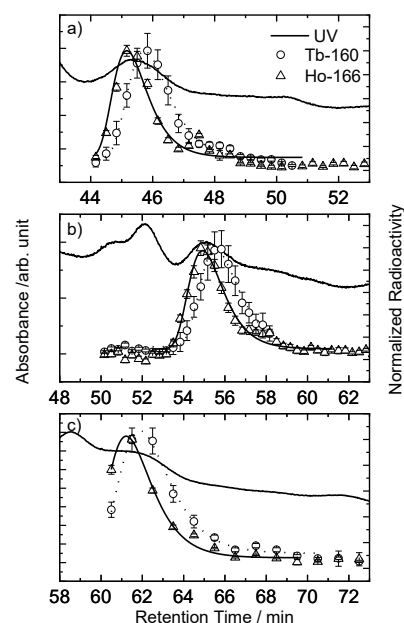


Fig. HPLC chromatograms of Tb and Ho fullerenes developed at a) 40 °C, b) RT, and c) 10 °C.

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Change in momentum distribution of electrons in Mg₂Sn induced by Sb doping

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INTRODUCTION: Mg₂Sn is known as a Kankyo semiconductor which constituents naturally abundant and environmentally friendly elements. This material has been studied as a candidate for a thermoelectric substance in middle-temperature range (600-900 K) [1]. Sb doping induced the increase in n-type conductivity and thermal conductivity. These parameters have a trade-off relationship in the formula of the figure of merit ZT , so it is difficult to improve the ZT through Sb doping. Another phenomenon due to Sb doping attracts attention as an effective method for improving the ZT , because the Sb doping induces the formation of a nano-size region with slightly different lattice constant from Mg₂Sn, which causes the decrease in thermal conductivity due to phonon scattering [2]. The origin of such nano-size region remains unclear at present. In the present study, we have performed positron coincidence Doppler broadening of undoped and 1% Sb doped Mg₂Sn, which provide us the information on the local structure of vacancy-type defects introduced by Sb doping.

EXPERIMENTS: The polycrystals of undoped and 1% Sb doped Mg₂Sn were used as samples. The CDB experiment was performed at the low-energy positron beamline in KUR. The incident energy of the positron beam was 25 keV. The measurements of CDB curves were carried out until the total count of gamma-rays, generating by pair annihilation of positrons with electrons, reaches to 2×10^5 counts. The CDB curves of Mg, Sb, and Sn plates were also measured to use references. The ratio curves of undoped and 1% Sb doped Mg₂Sn, Sb, and Sn relative to Mg were obtained to compare the similarity in CDB curves between these samples, which enables the site identification of vacancy-type defects in undoped and 1% Sb doped Mg₂Sn.

RESULTS: The ratio curves of undoped and 1% Sb doped Mg₂Sn, Sb, and Sn relative to Mg are shown in Fig. 1. The ratio curves of undoped and 1% Sb doped Mg₂Sn are almost the same in the momentum below $7 \times 10^{-3} mc$. In higher momentum region, the ratio curves are different: The ratio curve of the undoped Mg₂Sn is larger than that of the 1% Sb doped Mg₂Sn. In this region, since the contribution of Sn core electrons is larger than that of Sb core electrons, Sb atoms distribute in high concentration around vacancy-type defects in 1% Sb doped Mg₂Sn. From positron annihilation lifetime spectroscopy experiments, it was turned out that Mg vacancies are introduced by Sb doping. Based on the results obtained, it is likely that the defect complexes of Sb atoms and Mg vacancies are formed in the nano-size region.

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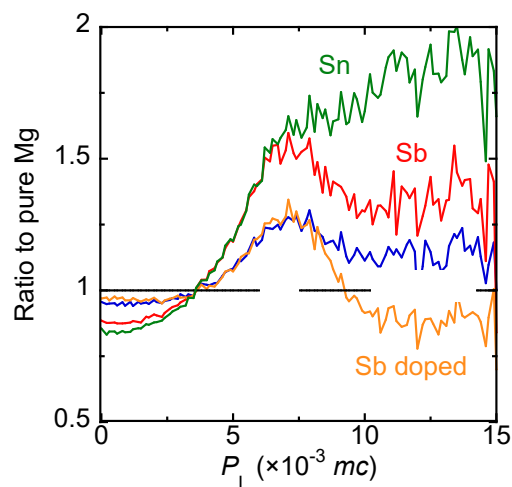


Fig.1 Ratio curves of undoped and 1% Sb doped Mg₂Sn relative to that of Mg. The ratio curves of Sb and Sb were also shown for references.

Strength properties and microstructure/orientation dependence of irradiation defect formation behavior in tungsten materials

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INTRODUCTION: Tungsten (W), which exhibits excellent thermal properties and low sputtering yield, is a promising candidate for plasma-facing materials in the divertor in fusion reactors. The microstructure and the distribution of pre-existing defects in W strongly depend on the manufacturing process, leading to significant variations in structure-sensitive properties such as mechanical strength and irradiation-induced defect formation behavior. In this study, specimens have been prepared from pure rolled W (ITER divertor-grade, stress-relieved) by cutting along different orientations to vary the microstructure and the alignment of pre-existing defects. These specimens have been subjected to electron irradiation experiments to investigate defect formation behavior.

EXPERIMENTS: Specimens will be extracted from rolled pure tungsten (ITER divertor grade, stress-relieved) along different crystallographic orientations. These specimens will be irradiated with an electron beam using the LINAC at the Kyoto University Institute for Integrated Radiation and Nuclear Science. Before and after irradiation, positron annihilation spectroscopy will be employed to identify pre-existing and irradiation-induced defects. Electron backscatter diffraction (EBSD) has been used to analyze the microstructure and crystallographic orientations.

RESULTS: Figure 1 shows the planes with respect to the rolling direction. Figure 2 presents the IPF map (ND) obtained by EBSD. On the TD plane, which is perpendicular to the rolling plane and parallel to the rolling direction, it can be observed that the grains are elongated along the rolling direction. Figure 3 shows the positron lifetime measurement results for each plane. The results differ depending on the plane. In particular, for the TD plane, the lifetime of τ_2 is longer, while its intensity (I_2) is lower compared to those of the other planes. The positron lifetimes for defect-free W and a monovacancy are 110 ps and 180 ps, respectively. The τ_1 values for the ND and RD planes are close to that of defect-free W. These results indicate that, on the TD plane, vacancies exist in the form of vacancy clusters, although their concentration is low. It is considered that vacancy clusters are more likely to grow during the elongation process of grains on the TD plane. Thus, it was found that rolled tungsten exhibits differences even in its microscopic structure as measured by positron lifetime spectroscopy. In future work, electron irradiation will be performed on these specimens to clarify the defect formation processes.

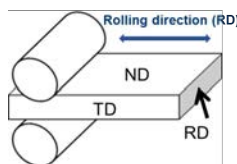


Fig. 1 Orientation of rolling

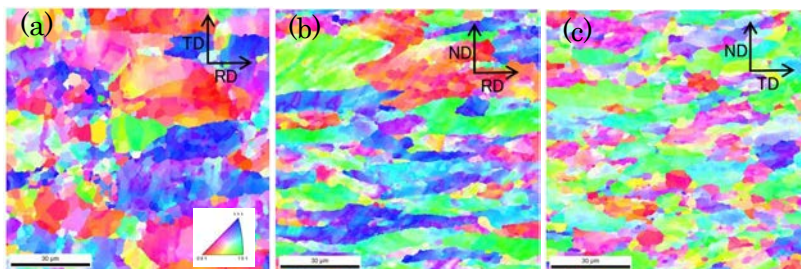


Fig. 2. IPF-map(ND) (a):ND, (b):TD, (C)RD

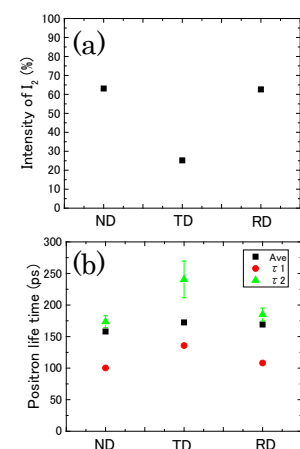


Fig. 3. Intensity of I_2 (a) and positron life time(b).

Research on freezing of materials under high pressure by Bessel-beam driven micro-explosions in transparent materials

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INTRODUCTION: It is possible to create extreme pressure and temperature conditions in the tabletop laboratory experiments with 100-fs ultra-short laser pulses focused inside a transparent material, with intensity in the focal spot significantly above the threshold for optical breakdown [1]. Tight focusing of a laser pulse with a few micro-Joule energy deep inside a crystal can deposit an energy density up to several MJ/cm³ in a sub-micron volume. Pressure created by the plasma expansion is high enough to generate new material phases, which is referred to as micro-explosion [2, 3]. Here we report an experiment relative to a micro-explosion with a layered target made of Al₂O₃ and highly ordered pyrolytic graphite (HOPG). Hence, we demonstrated X-ray diffraction (XRD) analysis of the material under high pressure and high temperature phase.

EXPERIMENTS:

(1) Micro-explosions experiment with femtosecond laser

The beam of a 250 fs amplified Ti:Sapphire laser with a central wavelength of 800 nm was focused on the surface of HOPG. The energy and spot size were 200 nJ and 1.2 μ m, respectively. After laser processing, the structures of void on the surface of HOPG were observed with Scanning Electron Microscope (SEM).

(2) XRD analysis with by Rigaku Ultima IV at KURNS

XRD analysis was demonstrated with the material with void formed inside by micro-explosions to observe high pressure phase of (Diamond and/or Lonsdaleite). X-ray energy was 8.04 keV (Cu-K α).

RESULTS:

Results of observation of void structure with SEM shows the length and diameter were 10 μ m and 350 nm, respectively. The volume of the void is $\sim$ 2 femtolitres.

Figure 1 shows the results of XRD. Signal of 2 θ diffraction with angles of 44.5 and 64.45 degrees. These observations indicate the signals derived from carbon high pressure phase. Further analyses are underway to better understand the data.

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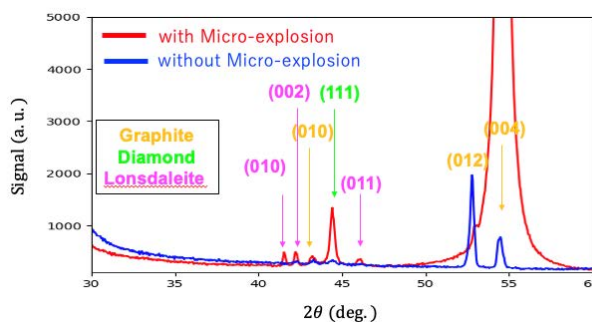


Fig. 1. XRD result

Elucidation of the Dynamics of Rigid Helical Polymers in Hydrocarbon Solvents by Small-Angle X-Ray Scattering and Molecular Dynamics Simulations

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INTRODUCTION: Poly(quinoxaline-2,3-diyl)s, which are denoted as PQXs, constitute a class of rigid helical polymers that exhibit solvent-dependent switching of helical chirality. Previous studies have shown that PQXs bearing chiral alkoxymethyl side chains adopt different helical conformations in response to changes in the solvent environment. In particular, a 100-mer PQX bearing (*R*)-2-octyloxymethyl side chains adopts a right-handed helical structure in tetrahydrofuran and an *M*-helical structure in a mixed solvent composed of 1,1,2-trichloroethane and tetrahydrofuran.¹ Small-angle neutron scattering (SANS)² and quasielastic neutron scattering measurements (QENS),³ combined with molecular dynamics (MD) simulations, have indicated that the spatial arrangement and mobility of the side chains are closely related to the inversion of helical handedness.

Solvent-induced helix inversion has also been observed for PQXs in simple saturated hydrocarbon solvents. A PQX bearing (*S*)-3-octyloxymethyl side chains exhibits an *M*-helical structure in *n*-octane and a *P*-helical structure in cyclooctane. This behavior is remarkable because the inversion is induced by a subtle change between alkane solvents. However, the molecular mechanism by which hydrocarbon solvents control the helical structure remains insufficiently clarified. In the present study, we investigated the structure and dynamics of a 100-mer PQX bearing (*S*)-3-octyloxymethyl side chains (**3oct**) in hydrocarbon solvents by combining SANS experiments with molecular modeling.

EXPERIMENTS AND RESULTS: Initially, the structures of the repeating monomeric units, which comprise two conformational isomers designated as the δ -monomer and λ -monomer, were optimized using the Forcite optimizer in BIOVIA Materials Studio 2024 with the COMPASS III force field at the ultra-fine level. Based on these optimized monomers, right-handed and left-handed 100-mer helical structures, namely *P*- δ -**3oct**, *M*- δ -**3oct**, *P*- λ -**3oct**, and *M*- λ -**3oct**, were constructed and further optimized. For these optimized structural models, small-angle neutron scattering spectra in *n*-octane-*d*₁₈ were simulated using software developed by Prof. Sugiyama. The simulated scattering profiles were then compared with the experimental SANS data (Fig. 1). The present structural models did not provide satisfactory agreement with the measured profiles.

This discrepancy indicates that further refinement of the molecular structures is required in order to reproduce the experimentally observed conformations in solution. Future work will involve a comprehensive conformational search and molecular dynamics simulations to identify structural models that can more accurately describe the scattering behavior of the (*S*)-3-octyloxymethyl-substituted PQX in hydrocarbon solvents.

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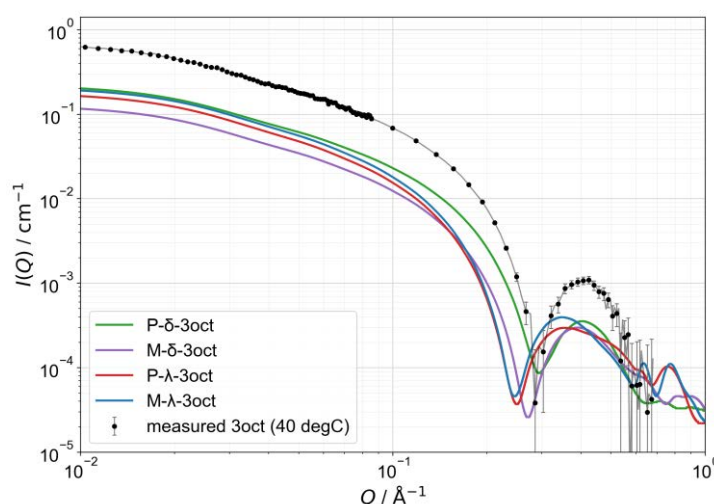


Fig. 1. Log-log SANS profiles of measured **3oct** at 40 °C and four simulated conformations in *n*-octane-*d*₁₈.

Positron annihilation study of electron irradiation-induced vacancy defects in CoCrFeNi medium-entropy alloy

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INTRODUCTION: Tsai *et al.* [1] reported sluggish diffusion in the CoCrFeMnNi high-entropy alloy (HEA), although its mechanism remains unclear. Because the HEA and its subsystems form substitutional solid solutions, the diffusion is expected to occur mainly via a vacancy mechanism, making vacancy formation and migration energies essential to understanding sluggish diffusion. In this study, we estimate the vacancy migration energy in the CoCrFeNi medium-entropy alloy by analyzing vacancy migration and annihilation during post-irradiation annealing.

EXPERIMENTS: An arc-melted ingot of the CoCrFeNi alloy was homogenized at 1373 K for 24 h under an argon atmosphere and subsequently cut into plates with dimensions of 10 mm × 10 mm × 0.5 mm. The plates were mechanically polished and sealed in silica tubes. Solution heat treatment was then performed at 1373 K for 1 h, followed by water quenching. X-ray diffraction analysis confirmed that all samples consisted of a single face-centered cubic (fcc) phase. The samples were irradiated in water with 8 MeV electrons to a fluence of approximately $1 \times 10^{22} \text{ e}^- \text{ m}^{-2}$ at temperatures below 358 K, using the electron linear accelerator at the Institute for Integrated Radiation and Nuclear Science, Kyoto University. Isochronal annealing was subsequently conducted between 373 and 673 K in 25 K increments, with each annealing step lasting 1 h. Positron lifetime measurements were carried out at 297–299 K using a fast–fast timing coincidence system with a time resolution (FWHM) of 180–183 ps.

RESULTS: Before electron irradiation, the positron lifetime spectrum of the solution-treated alloy was characterized by a single lifetime component of 108 ps, indicating that positron annihilation occurs predominantly in the defect-free bulk. After irradiation, the mean positron lifetime increased to 133 ps. Analysis of the irradiated sample shows that a substantial fraction of positrons are trapped and annihilate in monovacancies introduced by electron irradiation, as evidenced by the trapped-positron lifetime component of 180 ps. Assuming a specific trapping rate for monovacancies of $\mu_v = 10^{15} \text{ s}^{-1}$, the vacancy concentration in the irradiated alloy is estimated to be on the order of several atomic parts per million. The evolution of vacancy concentration during isochronal annealing was evaluated using two- or three-component analyses of the positron lifetime spectra. In the temperature range where both dislocations and monovacancies are expected to coexist, the spectra were analyzed by assuming a positron lifetime of 150 ps for dislocations. The vacancy concentration decreases with increasing annealing temperature because vacancies introduced by irradiation gradually disappear as they become mobile during isochronal annealing. The decrease in vacancy concentration was further examined using the isothermal annealing model proposed by Dryzek *et al.* [2]. When a single, temperature-independent vacancy migration energy is assumed, noticeable discrepancies arise between the theoretical fitting and the experimental data. Positron lifetime measurements at higher temperatures reveal the presence of dislocation loop–related components, suggesting that vacancy migration is strongly affected by dislocations. To account for this effect, two vacancy migration energies, H_m^L and H_m^H , were introduced and optimized to reproduce the experimental behavior. The value obtained for H_m^L in the low-temperature region is 0.92 eV, which is nearly identical to the reported migration energy of 0.93 eV for vacancies in CoCrFeMnNi [3].

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Influence of Hydrogen Isotopes on Growth of Vacancy Clusters in Tungsten-Based Materials

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INTRODUCTION: Tungsten (W), a plasma-facing material in fusion reactors, is exposed to hydrogen isotopes (deuterium and tritium) and fusion reaction products such as helium and 14 MeV neutrons. Vacancies and vacancy clusters created by neutron irradiation act as strong traps for hydrogen isotopes, leading to increased tritium retention in W [1]. Therefore, understanding vacancy formation and clustering is essential for accurate evaluation of tritium inventory in the vacuum vessel of fusion reactors. Since displacement damage develops under simultaneous exposure to hydrogen isotope plasma, hydrogen isotopes may influence the evolution of vacancy clusters. The objective of this study is to clarify the effects of hydrogen isotopes on vacancy and vacancy-cluster evolution, with the aim of developing a kinetic model for clustering and annihilation processes. To achieve this, W samples containing vacancy clusters were prepared by electron-beam irradiation, followed by annealing with and without hydrogen exposure. Changes in vacancy-cluster size distributions were investigated using positron annihilation spectroscopy (PAS).

EXPERIMENTS: Disk-shaped W samples were irradiated with 8 MeV electrons using KURNS-LINAC to 10^{-3} displacement per atom (dpa). A portion of irradiated samples was exposed to hydrogen (H) atoms at 200 °C and then heated under H₂ gas atmosphere (0.1 MPa) at 400 and 500 °C for 20–40 h. The remaining samples, without H exposure, were annealed under otherwise identical conditions. Positron lifetime was measured using a ²²Na source [1].

RESULTS AND DISCUSSION: The positron lifetime spectra were well fitted by the sum of two exponential decay components: a short lifetime of approximately 150 ps and a long lifetime of approximately 350 ps. The former corresponds to the annihilation in the defect-free W lattice, whereas the latter is attributed to annihilation at vacancy clusters consisting of roughly 10 vacancies [2]. Annealing at 400 °C resulted in no significant change in positron lifetime for either sample. After annealing at 500 °C for 40 h, the H-free sample exhibited a clear increase in the long-lifetime component to approximately 400 ps, which is attributed to Ostwald ripening of vacancy clusters. However, no significant change was observed in the H-exposed sample. This difference indicates that the presence of H suppressed the growth of vacancy clusters. It is plausible that H atoms inhibited decomposition of small vacancy clusters and subsequent growth into larger clusters.

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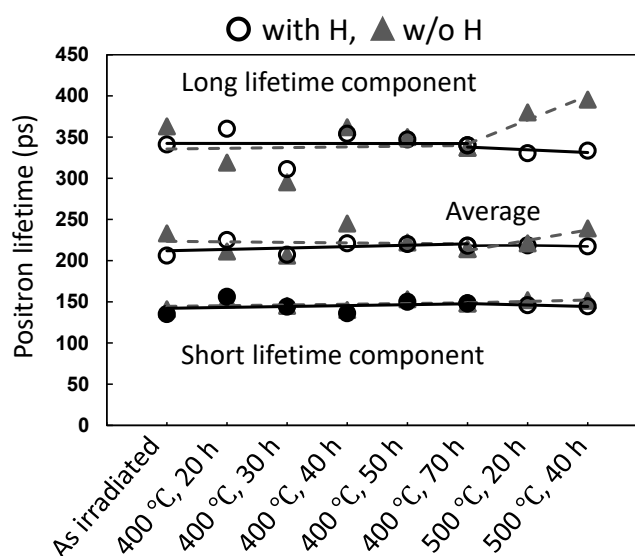


Fig. 1 Change in positron lifetime in electron-irradiated W after heating with and w/o H.

Chemical information contained in X-rays emitted during radioactive decay and its applications

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INTRODUCTION: As radioactive isotopes (RI) emit radiation from individual atomic nuclei, they offer extremely high detection sensitivity, making it possible to detect even minute quantities. Furthermore, activation analysis, which utilises neutrons from nuclear reactors, is widely recognised as a non-destructive analytical method capable of achieving sensitivity at the ppb level for many elements and quantifying multiple elements simultaneously. However, both RI tracer methods and activation analysis are quantitative techniques and cannot identify the chemical state.

We investigate the chemical information contained in the X-rays emitted during radioactive decay. When a RI decays, beta and gamma rays are emitted alongside the nuclear decay, and X-rays are emitted during electronic transitions. Here, we focus on X-rays associated with outer-shell electrons, which are influenced by the chemical state. Whilst we have previously acquired fundamental data using tracers produced by an accelerator, the aim of this study is to obtain data on samples that have been neutron-activated.

EXPERIMENTS: Samples consisting of powders of metals and compounds such as copper, manganese, iron and chromium were irradiated at KUR using Tc-Pn and Pn2. The post-irradiation samples were spread as thinly as possible between adhesive tapes and sealed to form measurement sources. Measurements were performed in the X-ray and γ -ray regions using a silicon drift detector and a high-purity germanium semiconductor detector. The output from these detectors was sent to a digital MCA, and measurements were carried out in list mode.

RESULTS: Although the results are currently being analysed, the spectra obtained show not only X-rays associated with radioactive decay but also X-ray signals generated when the emitted radiation excites the constituent materials of the radiation source and other components. It is necessary to carefully analyse whether the variations in the spectra observed for different radiation sources are due to changes in the chemical state or to changes in the surrounding materials.

ACKNOWLEDGMENT:

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Magnetic Measurements of Irons in new Si-based Ferrite-like Functional Materials using In-flight Melting Materials Method by Mössbauer Spectroscopy

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INTRODUCTION: Glass is used in many applications in our every-day lives and has exciting new applications related to the energy. Soda-lime glass is made of mainly silica (SiO₂) and additive many other materials, such as magnesium, sodium, calcium, aluminum, iron, sulfur, and so on. Our reference glass composition, in percent by weight (wt%), was 72.25 SiO₂, 1.75 Al₂O₃, 4.00 MgO, 8.00 CaO, 14.00 Na₂O as basic components, and 0.015-5 iron in terms of Fe₂O₃ as coloring components. Iron contaminants of less than 0.0005-0.01 wt% from raw materials, and iron injection up to 1.5-5 wt%. We added compatible CaCO₃ and Na₂CO₃ as CaO and Na₂O, respectively. The iron oxides in a glass composition are thought to be present in forms of Fe³⁺ and Fe²⁺. The control parameters for irons are two: (1) the total iron mass weight percent in terms of Fe₂O₃, and (2) the ratio of Fe²⁺ to total iron ions (Fe²⁺/Σ_nFe). The Fe³⁺ component adds a light yellow tint to the glass and absorption in the ultraviolet and visible band, while the Fe²⁺ component adds a blue tint to the glass and absorption in the near-infrared band (1 μm). The transmission from ultraviolet to infrared in glass cannot be explained by simple Fe²⁺ and Fe³⁺ structures. Then, to reveal the exact local structures of irons (Fe²⁺ and Fe³⁺) is necessary. Many scientists proposed many new theories and local structures [1-2], but they did not resolve it completely.

We have prepared reference glass samples with using the Mössbauer isotope ⁵⁷Fe (natural abundance is about 2.119 %) for measurements. The chemical composition of iron oxide in the ⁵⁷Fe enrichment glass was from 0.015 to 5 wt%, and the Fe²⁺/Σ_nFe was from 0 to 0.6. We have investigated these samples by nuclear resonant inelastic scattering and XAFS methods at synchrotron radiation to reveal the local atomic structure around and neighboring iron in sub-nanometer region [3].

We also have prepared new amorphous-Si-based functional glass and semi-melted crystal-like materials with iron, whose chemical composition was the same with the reference glass, for new scientific purposes[4-8], by in-flight melting material method[4-5]. We have also succeeded to make Ferrite-like materials by fast melting and fast cooling cycles[6-7]. We have thought that the high temperature in melting process and their chemical components were similar to those of fly-ash, although the assumed iron-oxides in the Yonden fly-ash was ~1 to ~10(-30) wt%.

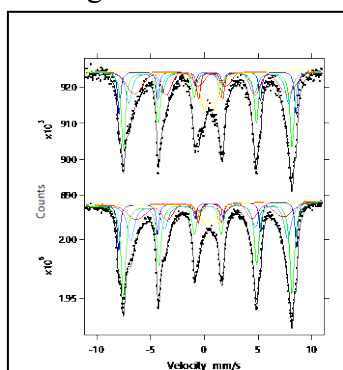


Fig. 1. Mössbauer spectra of Yonden fly-ash collecting with/without (lower/upper panel) the magnetic stirrer bar in slurry.

EXPERIMENTS: We observed two-type black-colored components in Yonden fly-ash gathering with and without the magnetic stirrer bar in a water slurry, because it is difficult to gather black-colored ones directly from the dried fly-ash. The measurements were performed using conventional Mössbauer spectrometer at 273K. The specimens for Mössbauer measurements were tuned to 10 mm-phi pellet.

RESULTS: There was no typical difference between the two-type samples collecting with and without magnet as shown in Fig. 1. Most black-colored components in Yonden fly-ash were thought to be Hematite, Maghemite + Magnetite. Iron oxides in the Yonden fly-ash are integrated with Si (and O), and they are considered to exist rarely as separate particles, which are easy to collect with magnetic tools.

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Development of New-type Diamond Detectors for Neutron

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INTRODUCTION: The diamond is 5.47 eV wide-gap semiconductor with high radiation resistance and thermal conductivity. Diamond detector has energy resolution. Then we developed new-type single/multiple crystal diamond detectors for gamma-ray/x-ray or alpha particles/neutron detection. We proposed these detectors for astrophysics, medical applications and nuclear reactor operations. We planned to arrange these diamond devices with special short wavelength and ultra short pulsed (femtosecond to picosecond range) laser machines. [1-4] First, we developed normal-type single crystal diamond detectors for x/gamma rays and alpha-particles. We first used the much high quality CVD diamond plate, which was homo-epitaxial grown using a microwave plasma chemical vapor deposition (MPCVD). We also used the HPHT diamond plate for comparison. Then we developed simple transmission-type detectors with double-sided illumination capability as shown in Fig. 1. We succeeded to develop new-type single crystal double-sided illumination diamond detectors for only alpha particles detection with normal diamond plate. Then, we used this diamond detector for neutron detection by converting to alpha-particles with Boron (^{10}B) material on the thin electrode of the detector. We also especially planned the large area-size multiple/polycrystalline diamond detectors for single-use, which are required for decommissioning works at Fukushima. Then we proposed new plasma/thermal filament method to make CVD multiple diamond with original electrodes for new-type diamond detectors with simple production. [5-6]

EXPERIMENTS: This time, we used single/multiple diamond crystal by plasma CVD method and HPHT method. The size of the diamond plate was from 2x2 mm to 10x10 mm. We made the same Ti(30nm)/Pt(30nm)/Au(100nm) electrodes on both sides of the diamond plate. At one side, we made guard-ring on one electrode. We coated thin boron (^{10}B) materials (BN powders) on a thin electrode (Ti(30nm)/Pt(30nm)/Au(100nm)) without guard-ring to convert neutron to alpha particles. We used this detector with low noise amplifier (CP-580H etc) as shown Fig.2. We observed thermal neutron in the Heavy Water Neutron Irradiation Facility of Kyoto University Reactor (KUR).

RESULTS: We succeeded to detect neutron signals continuously as shown in Fig. 2 (see pulse shape in the Oscilloscope window). However, within a few minutes, we also observed cyclic current increasing and decreasing without additional pulsed signals. We did not find causes at all. The base metal plate and substrate materials of our diamond detector were not adequate for neutron experiments. These materials have become radioactive as shown in Fig. 3. The prompt gamma rays emitted from ^{198}Au are particularly noticeable

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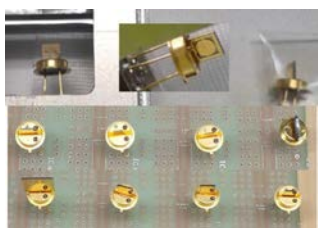


Fig.1 Diamond detectors.

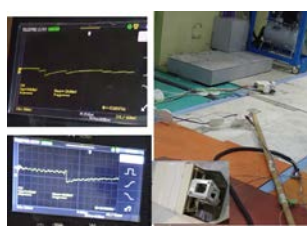


Fig.2. Signals and setup

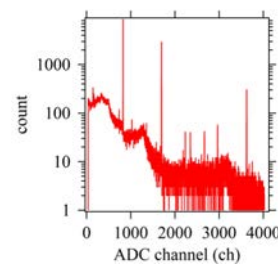


Fig.3. Activation of the case

Effect of zirconium on the tritium release behavior from neutron-irradiated LiAlO_2

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INTRODUCTION: Tritium (T) production using a high-temperature gas-cooled reactor (HTGR) has been proposed to arrange initial T inventory for nuclear fusion reactors [1]. By loading Li-rods in place of Burnable-Poison-rods, T can be produced without significant modifications to the original structural design of HTGRs. One concern is the permeation loss of T at high temperature conditions. In the recent design, the Li rods consist of an alumina container installing LiAlO_2 and Zr pebbles. Zr pebbles play the role of absorbing tritium released from LiAlO_2 and reducing the tritium partial pressure inside the alumina container. To evaluate the tritium confinement performance of the Li rod structure, it is necessary to understand the tritium behavior in an environment where LiAlO_2 and Zr coexist. In this study, neutron-irradiated LiAlO_2 was heated in the presence of Zr, and the behavior of the released tritium was observed.

EXPERIMENTS: The powdered LiAlO_2 was packed into a quartz tube in vacuum and irradiated neutrons with a flux of $2.75 \times 10^{13} \text{ n}/(\text{cm}^2 \cdot \text{s})$ for 3 minutes at KUR. After neutron irradiation, the samples were transported to Kyushu University for tritium release experiments. The schematic diagram of the experimental apparatus is shown in Fig.1. The neutron irradiated LiAlO_2 with broken quartz tube was packed with Zr pebbles in a quartz reaction tube. The LiAlO_2 with Zr pebbles was heated by an electric furnace to 900 °C in Ar gas flow. The tritiated water vapor (HTO) and gaseous tritium (HT) were separately collected in water bubblers. The bubblers were replaced every 5 minutes and tritium concentration was measured by a liquid scintillation counter.

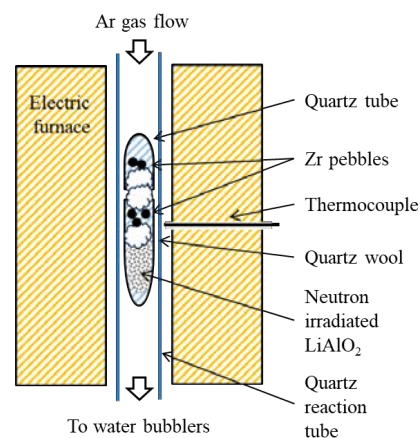


Fig.1. Experimental apparatus.

RESULTS: The release rate of HT and HTO was shown in Fig.1. HTO was released with the onset of heating, and the release rate peaked around 250 °C and then gradually decreased. HT was released from around 500 °C and the release rate peaked at around 750 °C and then sharply decreased. In the absence of Zr pebbles, the majority of tritium released from LiAlO_2 was observed to be HTO in previous works. The detection of HT suggests that the HTO released from LiAlO_2 was reduced to HT through interaction with Zr. The rapid drop in HT release observed above 750 °C is considered to be caused by the accelerated HT absorption into Zr.

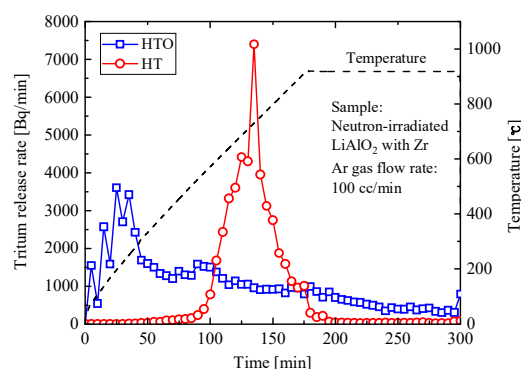


Fig.2. Tritium release from neutron irradiated LiAlO_2 in the presence of Zr pebbles.

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Neutron Irradiation Tests for ITER Diagnostic Systems in JADA

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INTRODUCTION: ITER is the world's largest fusion experiment reactor and is under construction in southern France [1]. Components of diagnostic systems installed in the ITER tokamak are exposed to high neutron radiation. Therefore, it is important to investigate the effect of neutron irradiation on these components. JADA (ITER project Japan Domestic Agency) have been conducted neutron irradiation tests on components of ITER diagnostic systems in KUR from 2018. In fiscal 2025, neutron irradiation tests were conducted on optical elements used in DIM (Divertor Impurity Monitor) [2]. The effects of neutron irradiation on spectral transmittance and reflectance were measured for optical elements used in a spectrometer placed on a PCSS (Port cell Support Structure) and for optical fibers placed on the PCSS, and their applicability was evaluated. The spectrometers, which use the optical elements, are installed in PCSS at three ports: UP01, EP01, and LP02. The expected fluence was calculated using the ITER radiation map, and then the maximum fluence is 5×10^{13} n/cm² at EP01. This evaluation focused on verifying whether the optical elements could withstand this value. The irradiated specimens are one substrate, one dichroic mirror, one dielectric multilayer mirror, and four bandpass filters as optical elements used in the spectrometer, as well as two types of optical fiber strands.

EXPERIMENTS: The neutron irradiation experiment was conducted using Slant Exposure at KUR. The maximum neutron flux during 1 MW operation was 1×10^{12} n/cm²/s. The substrate used was Ohara's SK-1300, made of SiO₂, with dimensions of $\Phi 25.4$ mm x 2 mm. The filters and mirrors were prepared by depositing thin films of a combination of HfO₂, Ta₂O₅, Al₂O₃, MgF₂, and ZrO₂. The optical fiber specimens consist of SiO₂ core, F-doped SiO₂ cladding and polyimide jacket. One type of specimens has an additional carbon layer between cladding and jacket.

RESULTS: The optical transmittance of the substrate in the 200-800 nm range was approximately 90%, and no significant changes were observed before and after irradiation at 5×10^{13} n/cm². Figure 1 shows an example of the transmittance spectra of the 372 nm bandpass filter specimen neutron irradiated with neutron. For irradiation up to 5×10^{13} n/cm², neither a significant decrease in transmittance nor a peak shift is observed. Figure 2 shows an example of the change in transmission of optical fiber by neutron irradiation, as ratio of value before irradiation. No significant decrease in transmittance is observed for irradiation up to 5×10^{13} n/cm².

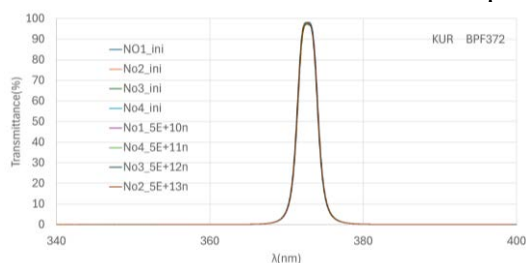


Fig. 1 Transmission spectrum of 372nm bandpass filter specimen irradiated with neutrons

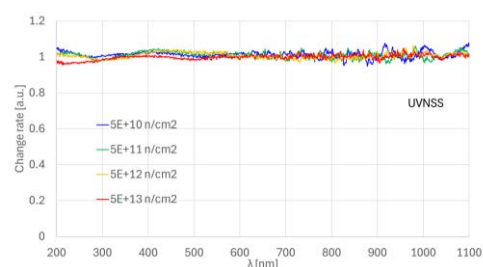


Fig. 2 Transmission of optical fiber by neutron irradiation

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Lithium Vacancy Concentrations Measurements in Positive Electrodes of Charged and Discharged-Lithium Ion Batteries Using Positron Annihilation Spectroscopy

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INTRODUCTION: An oxide-based solid-state rechargeable lithium ion (Li^+) battery is one of the most remarkable next generation devices. To realize the product, it is essential that we have information on the Li^+ ion transfer resistance at electrode/solid electrolyte interfaces and grain boundaries in the solid-state Li^+ ion batteries. In particular, the Li deficient region formed around the interface during the charging and discharging, which is indicative of space charge layer, locally provides less Li^+ ion conduction and then leads to the interfacial Li^+ ion transfer resistance, since the Li^+ ionic conductivity in the solid electrolyte significantly depends on the number of Li^+ ion mobile carriers. Thus, the Li distribution around interface in static or operated solid-state Li^+ ion batteries should be clarified well [1].

EXPERIMENTS: Some Li^+ ion rechargeable batteries consisting of the LiCoO_2 positive electrode and the Li negative electrode with a dimension of $30 \text{ mm} \times 35 \text{ mm} \times 105 \text{ }\mu\text{m}$, including the LiPF_6 electrolyte, were built up by JFE Techno-Research Corporation. Six types of Li_xCoO_2 samples with different x values were prepared for as-received (no bias, $x = 0.99$), break-in by SoC 0% (0% charge, $x = 0.99$), charged by SoC 50% (50% charge, $x = 0.73$) and SoC 100% (100% charge, $x = 0.49$), and charged by SoC 100% and subsequently discharged by SoC 50% (50% discharge, $x = 0.74$) and SoC 0% (100% discharge, $x = 0.99$). Next, positron annihilation spectroscopy (PAS), which was useful technique that enables investigating the behavior of voids in the materials, was employed using energy-variable monoenergetic positron beams (slow positron beams) of KUR-ISOL (B1 port) from the fission reactor with 5 MW, installed at Institute for Integrated Radiation and Nuclear Science, Kyoto University.

RESULTS: Figure 1 shows Li vacancy concentrations in no bias, charged (SoC 0, 50, and 100%), and charged-discharged (SoC 50 and 0%) Li_xCoO_2 ($0.49 \leq x \leq 0.99$) as a function of the depth from the topmost surface. The depth distributions of the Li vacancy increased and decreased, respectively, at each depth by charging and discharging. They are in good agreement with the amounts of Li^+ ion transfer and Li concentrations, respectively, obtained from electrochemical measurements and ERD technique. In particular, it was found that the amounts of the Li vacancies increased with the gradient in the near surface of Li_xCoO_2 .

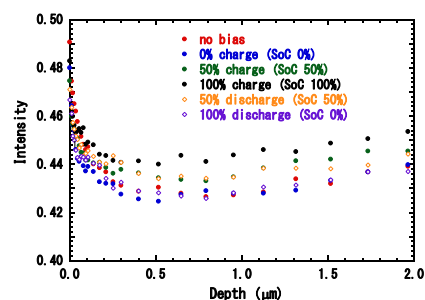


Fig. 1. Li vacancy concentrations in Li_xCoO_2 samples ($x = 0.49 - 0.99$), charged and discharged with 40 mA for each time.

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Acoustic velocities of silicate glass under ultra-high pressure

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INTRODUCTION: It is widely accepted most of the terrestrial planets may have experienced one or several phases of magma ocean (MO) stage¹, consequently a portion of silicate melt could still be present in the terrestrial deep mantle as remnants of a primordial MO. For the anomalous velocity structure observed in the ultra-low-velocity-zones² atop core-mantle-boundary (CMB) is believed to be due to the presence of such melt. Furthermore, recent seismic evidence indicates the presence of a largely molten silicate layer above the core of Mars³ indicating that such basal silicate layers may be widespread phenomena among terrestrial planets. Their long-term survival in deep terrestrial planetary interiors is subject to their gravitational stability. However, scarce data on the melt in the relatively low-pressure range force to use silicate glasses as a structural analogue to melt. In this respect we synthesized silicate glasses by laser levitation technique and performed high pressure sound velocity measurements in a diamond anvil cell.

EXPERIMENTS: The starting mixture of the oxides for silicate glass was heated with CO₂ laser on a stage where a gas flow through the nozzle to provide sufficient buoyancy to levitate the molten silicate bead. The sample was melted once at 1900 ± 5 °C to achieve complete fusion. Quenching of the melted silicate leading to the production of a spherical bead. Mössbauer spectra (Fig. 1) were collected on the glass bead at Kyoto University. High pressure sound velocity measurements were carried out at ETH Zurich using Brillouin spectroscopic technique

RESULTS: Chemical homogeneity of the glass (pyrolite) was confirmed from the microprobe analysis and Raman spectra. Deconvolution of the room temperature ⁵⁷Fe Mössbauer spectrum yielded two paramagnetic doublets with respective isomer shift (IS) and quadrupole splitting (QS) of $\sim 1.04(5)$ and $1.95(5)$ mm/s attributed to Fe²⁺ and $0.31(5)$ and $1.36(5)$ mm/s attributed to Fe³⁺, consistent with the literature. The area covered by the ferric iron was taken for the calculation of the Fe³⁺/ΣFe ratio, which equals to $\sim 0.61(5)$. The Pressure evolution of the Brillouin spectra is shown in the Fig. 2. A systematic increase in the sound velocity was observed in this study.

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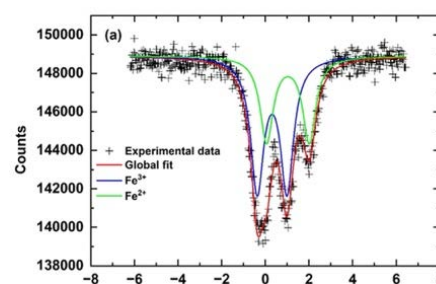


Fig.1: Mössbauer spectrum of the pyrolite-glass with Fe³⁺/ΣFe = 0.61(5).

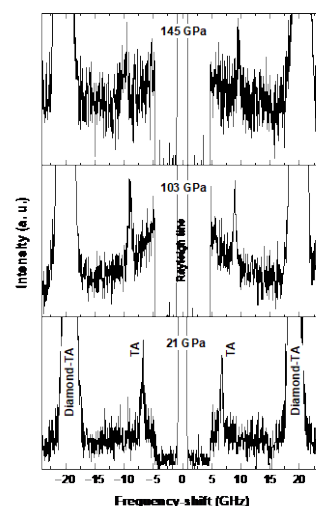


Fig. 2: Representative room temperature Brillouin spectra of pyrolite glass at 21, 103 and 145 GPa. TA represents transverse acoustic mode

Volcanic and Tectonic History of Philippine Sea Plate (South of Japan) Revealed by $^{40}\text{Ar}/^{39}\text{Ar}$ Dating Technique

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INTRODUCTION: The West Philippine Basin (WPB) occupies the western half of the modern Philippine Sea Plate. However, its origin, spreading history and tectonic relationship to the adjacent Izu-Bonin-Mariana arc remains poorly understood (e.g., Ishizuka et al., 2011). In particular, the disposition of the WPB spreading in relation to the nascent Izu-Bonin-Mariana arc is critical in reconstructions of Philippine Sea plate tectonics which, in turn, have ramifications for the subduction initiation of the Pacific Plate.

EXPERIMENTS: Ages of 25 volcanic rock samples were determined using the $^{40}\text{Ar}/^{39}\text{Ar}$ dating facility at the Geological Survey of Japan/AIST. Details of the procedures are reported in Ishizuka et al. (2018). 5-15 mg of phenocryst-free groundmass, crushed and sieved to 180 – 250 μm in size, was analyzed using a stepwise heating procedure. The samples were treated in 6N HCl for 30 minutes at 95°C with stirring to remove any alteration products present in groundmass and interstitial spaces. The samples were further treated with 6N HNO₃ for 30 minutes at 95°C with stirring. After this treatment, samples were examined under a microscope. Sample irradiation was done at the Kyoto University Reactor (KUR). At the KUR, the neutron irradiation was performed for 10 h at the hydro-irradiation port under 1 MW operation, where thermal and fast neutron fluxes are 1.6×10^{13} and 7.8×10^{12} n/cm² s, respectively. Argon isotopes were measured in a peak-jumping mode on an IsotopX NGX noble gas mass spectrometer fitted with a Hamamatsu Photonics R4146 secondary electron multiplier.

RESULTS: We investigated the intersection of a spreading center with an island arc within the WPB, i.e. an arc-ridge-arc triple junction, using a combination of submersible sampling and shipboard geophysics. We use Ar-Ar dating, elemental concentrations and Sr-Nd-Pb radiogenic isotope ratios to constrain the structure, age and origin of magmatism as spreading interacted with the arc.

Between 33-31 Ma the tip of the WPB spreading center was in the back-arc west of the Kyushu-Palau Ridge (KPR) arc. Spreading volcanism had mantle-like Ce/Pb and Th/Ce indicating a lack of slab flux. Radiogenic isotopes form a trajectory towards an EM-2-like mantle component.

The final stage of spreading/rifting-related activity occurred between 31-28.5 Ma when the spreading axis rotated from NW-SE to E-W and propagated eastwards into the arc, and directly intersect the KPR. This caused rifting and overlapping MORB-like magmatism on the frontal line of the KPR arc. During the final stage magmatism La/Sm and Nb/Zr increased while Pb and Sr isotopes became progressively more radiogenic, demonstrating a progressive decrease in the degree of partial melting and preferential tapping of the EM-2 component. This change in melt generation and composition appears a common process at the end of spreading.

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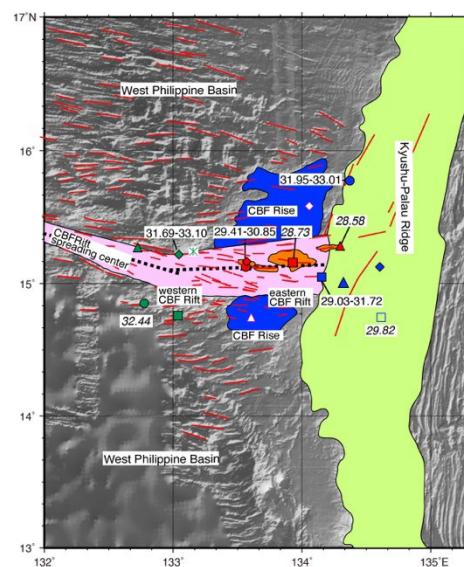


Fig. 1. Geological interpretation of the CBF Rift-KPR intersection area with $^{40}\text{Ar}/^{39}\text{Ar}$ age data (shown in Ma) on the recovered basalt at each station (Ishizuka et al., 2026). The data is based on results of this study and a previous study (ages shown in Italics are from Ishizuka et al.,

Evaluation of water solubility in Mg_2SiO_4 olivine under high pressure

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INTRODUCTION: Water was delivered to the early Earth by hydrated planetesimals. During the subsequent magma ocean stage of the Earth, water was partitioned among major reservoirs; some was incorporated into iron in the core and into silicate minerals in the mantle [1, 2]. Understanding the link between early Earth evolution and its water budget requires evaluating hydrogen storage in the silicate mineral structures. Olivine (Mg_2SiO_4), the dominant mineral in the upper mantle (<400 km depth), still lacks reliable estimates of its water content. This is due to (i) limited hydrogen incorporation into its crystal structure, (ii) possible submicron hydrous inclusions, and (iii) the need for microscale analytical techniques to determine water content while avoiding such inclusions. In this study, we develop a method to evaluate hydrogen incorporation in olivine synthesized under high-pressure and high-temperature conditions using integrated microanalytical techniques.

EXPERIMENTS: In the synthesis experiments, powders of $\text{Mg}(\text{OH})_2$, MgO , and SiO_2 were mixed as starting materials, sealed in an Au–Pd capsule, and placed in a cell assembly. The samples were held at 11.4 GPa and 1250 °C for 30 minutes using a multi-anvil apparatus to synthesize hydrous olivine. The recovered samples were analyzed using secondary ion mass spectrometry (SIMS) to precisely determine the water content as OH groups incorporated into the olivine structure. In addition, a portion of each sample was prepared as an ultrathin section (~100 nm thick) using a focused ion beam (FIB) system and examined by transmission electron microscopy (TEM) for microstructural observation. Furthermore, the same sample was analyzed by the micro-focused X-ray diffractometer (μXRD ; Rigaku SmartLab 9kW) at KURNS, Kyoto Univ.

RESULTS: Olivine occurs as grains smaller than ~150 μm , coexisting with clinoenstatite (MgSiO_3) and quench crystals derived from the melt. The H_2O content of hydrous olivine, determined by SIMS, is 3900 ± 500 ppm. Preliminary μXRD measurements obtained from regions ~100 μm in diameter confirm that the unit cell of hydrous olivine is slightly expanded compared to that of anhydrous olivine (Fig. 1). Transmission electron micrographs and electron diffraction patterns from the olivine grain with the highest water content (4800 ppm) show no evidence of hydrous defects or inclusions, even at the nanoscale (Fig. 2). These results demonstrate that up to ~4000 ppm H_2O can be homogeneously incorporated directly into the olivine structure as hydroxyl groups. They also raise the possibility that previously reported water contents in olivine (8500–8900 ppm), based solely on FTIR measurements [3] for samples synthesized under similar pressure–temperature conditions, may have been overestimated, although more systematic investigation is required.

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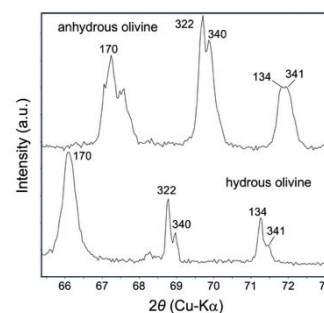


Fig. 1. XRD patterns of anhydrous and hydrous olivine.

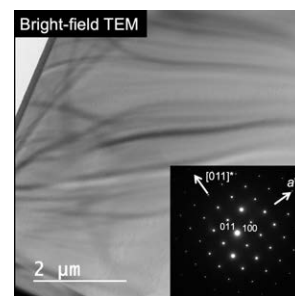


Fig. 2. TEM image and electron diffraction pattern of a hydrous olivine crystal.

Size Distribution and Long-term Concentration Change of Constituent Elements in the Atmospheric Aerosols Observed at Sakai, Osaka

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The atmospheric aerosols which are particles suspended in the atmosphere with wide range of particle size, 10nm ~100 μ m, affect our health and the climate. Element constituents in the atmospheric aerosols have the different concentrations on the particle sizes. By the difference of concentration in elements on the particle size, the effect of particles on our health and the climate change by its size distribution. To estimate source contribution from the natural sources such as the soil and sea spray, artificial sources such as fuel burning, emission from industries and the effect of atmospheric aerosol to our health, we have observed the atmospheric aerosols in different size ranges (9 size ranges)at Sakai, Osaka since 1995. Usually the size distribution of elements have two peaks, referring to V and Sb in Fig.1, that are found in 0.2 ~1.0 μ m as fine particle element and found in 2~5 μ m as coarse particle element. So, the difference of the effect from the coarse particle element and fine particle element are needed to recognize. In this report we show the result of size distribution of analyzed elements and mean concentration and mean size for the elements in the samples analyzed by neutron activate analysis.

The aerosols samples have been collected on polyethylene sheet using Andersen sampler with 9 particle size ranges (<0.43,0.43~0.65,0.65~1.1,1.1~2.1,2.1~3.3,3.3~4.7,4.7~7.0,7.0~11,>11 μ m) on one week sampling time. The elements in some of the samples were analyzed by neutron activation analysis using Kyoto University nuclear reactor. We got the size distribution of 23 elements (Al,Ba,Br,Ca,Ce,Cl,Cr,Cs,Fe,K,La,Mn,Mo,Na,Sb,Sc,Sm,Th,Ti,V,Zn),and got the mean concentration and mean size (Fig.1).

From this results, we have classified the size distribution into three type of size distribution, (1) distributed mainly in the coarse particle, mainly contributed from natural sources such as soil and sea salt:Al, Ba,Ca, Ce,Cr, Fe,Na,Sc,Sm,Th,Ti (2) distributed mainly in the fine particles contributed mainly from the source on burning and the secondary products from gaseous matters:As, Br, Cs ,V(3) distributed in both coarse and fine particles, K, Mo,Sb, Zn Mo.

Also we got the long-term trend(1995-2024) of element concentration in fine and coarse particles. Result of relative (ratio to mean concentration) increase rate for the element in the coarse and fine particles are shown on Fig.2. From this trend , almost of the elements in the fine particles have the decrease trend . This decrease trend might be caused by the fuel and emission control of the industry , incinerator and vehicle by the government . Cl, Sb,V have the clear decrease trend(more than 4% decrease per year).

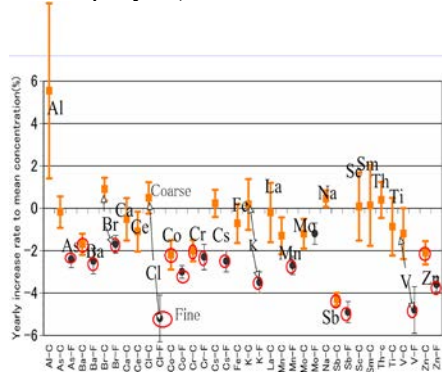


Fig.2 Relative (ratio to mean concentration) increase rate of element in the coarse and fine particles

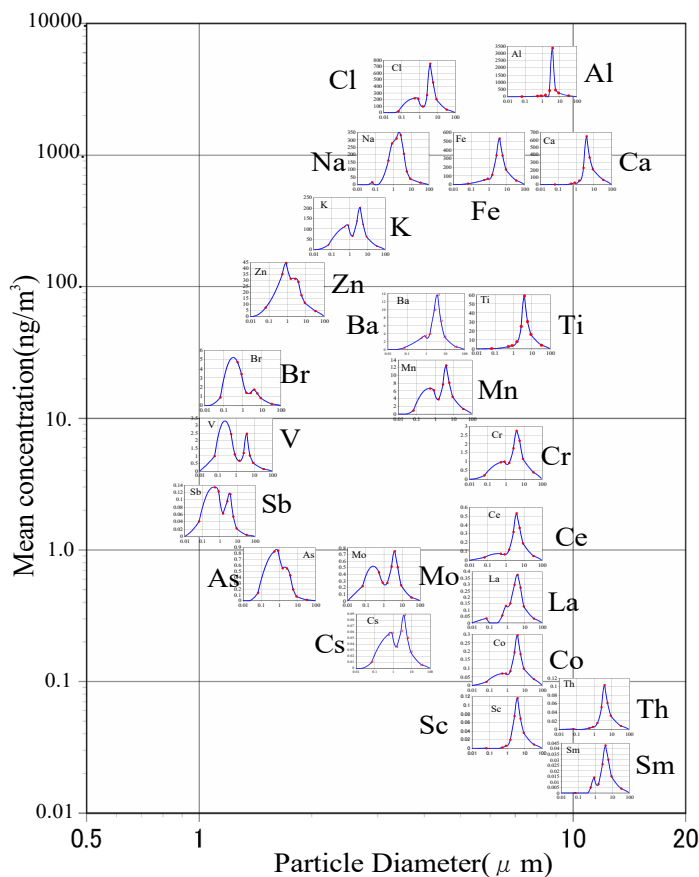


Fig.1 Size distributions of element in the atmospheric aerosols observed at Sakai,Osaka. Each size distribution is located at the position made by mean concentration and mean size for each element.

Laboratory-based analysis of synthetic high-density minerals of $\leq 100\ \mu\text{m}$ crystal sizes using microfocused X-ray beam I

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INTRODUCTION: Planet Earth is also known as the Water Planet. Throughout its 4.6 billion years of evolution history, liquid water covered a major part of Earth's surface and played a crucial role. On the other hand, by recent planetary exploration studies in the inner solar system, it was confirmed that solid materials occurring around Earth had contained a large amount of hydrogen equivalent to orders of magnitude more water than that currently exists on Earth. In other words, the Water Planet now seems not to be particularly rich in water. Thus, it is important to pay more attention to hydrogen within minerals occurring deep inside Earth, which were proposed to store an even larger amount of water than that on Earth.

It has been gradually established that the majority of the Earth's hydrogen indeed exists as structural water within crystal lattices of high-density silicate minerals in Earth's interior [1,2]. This concept mostly stemmed from results of high-pressure and temperature synthesis experiments on a wide variety of high-density minerals, which were often $\leq 100\ \mu\text{m}$ in their crystal sizes. After detailed structure analysis using intense X-ray and neutron beams, which were only available at large-scale experimental facilities, the concept has been continuously reinforced. On the other hand, laboratory-based X-ray diffraction analysis also suggested that hydrogen concentration could be evaluated independently and quantitatively from accurate structure parameters of minerals once these parameters were successfully determined [3]. Thus, in this study, we aim to determine the structure parameters of these synthetic mineral crystals at such small sizes using a micro-focused X-ray diffraction scheme that is available at KURNS.

EXPERIMENTS and RESULTS: In order to evaluate how accurately the lattice parameters can be determined using the micro-focused beam of $100\ \mu\text{m}$, a systematic analysis of already well-characterized standard samples with varying crystal sizes is needed first. For the first year of the project, therefore, we evaluated the lattice parameter of pure aluminum powder with different grain sizes. Figure 1 shows representative results obtained using $\text{CuK}\alpha$ X-ray focusing optics of Rigaku SmartLab 9 kW, which provide implications for how the effects of grain size and diffraction angle are quantitatively corrected. It was proven that there is no effect of the grain size on 2θ and an angle-independent effect on it. These results were essential for obtaining precise lattice parameters for the actual mineral samples [4].

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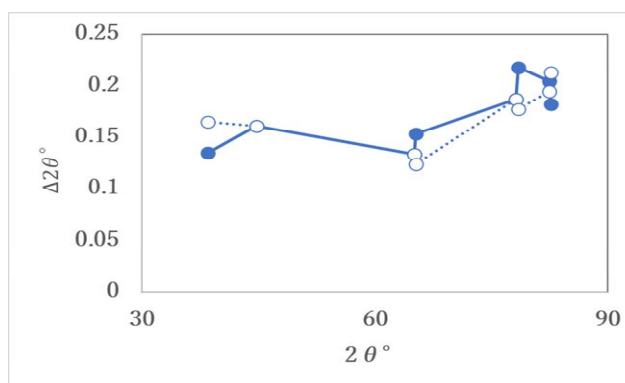


Fig. 1. Systematic diffraction angle error ($\Delta 2\theta$) as a function of 2θ , which was evaluated from micro-focused X-ray diffraction patterns of pure aluminum powder sample of $45\ \mu\text{m}$ (open circle) and of $3\ \mu\text{m}$ (solid circles).

Noble gases and halogens in serpentinite-related metasomatic rocks constrain time-dependent changes in the origin of aqueous fluids in the shallow mantle wedge

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INTRODUCTION: In the ^{40}Ar - ^{39}Ar and I-Xe dating methods, radiogenic daughter isotopes (^{40}Ar and ^{129}Xe) and parent elements (potassium and iodine) are simultaneously determined by converting ^{39}K and ^{127}I to ^{39}Ar and ^{128}Xe , respectively, via neutron-induced nuclear reactions in a nuclear reactor, and by noble-gas mass spectrometry. As halogens (fluorine, chlorine, bromine, and iodine) are also converted to corresponding noble gas isotopes by neutron irradiation and detection limits for noble gas isotopes by mass spectrometry are pretty low (down to ten thousand atoms), they can also be analyzed with high sensitivity by using the same method [1].

Fluids released as plates subduct play an essential role in generating active magma and seismic activity at convergent margins. High-pressure, low-temperature metamorphic belts preserve rocks that record processes along the plate interface in subduction zones and commonly contain ultramafic units derived from the mantle wedge. These ultramafic rocks are generally surrounded by metasomatic zones composed mainly of hydrous minerals. Such metasomatic zones are direct geological records of hydrous fluids moving along the slab-mantle interface and transporting various chemical species. Neither the origin of the fluids nor the pathways followed once they are released by dehydration is well known. We investigated the origins of fluids in several ultramafic bodies and their surrounding metasomatic reaction zones in the Sambagawa metamorphic belt in Japan using noble-gas mass spectrometry on fluid inclusions and crystalline water in hydrous minerals.

EXPERIMENTS: Samples weighing 5-50 mg each and standards for neutron fluence were wrapped in aluminum foil and placed in aluminum capsules with $\phi 10$ mm x 30 mm. The capsules were irradiated with neutrons in KUR. After the irradiation, the samples were sent to the University of Tokyo. The samples were loaded into an ultra-high-vacuum line for noble-gas extraction, purification, and separation. Noble gases were extracted from the samples by heating up to 1800°C, purified with hot Ti-Zr getters, separated into each noble gas with temperature-controlled cold traps, and then their isotope compositions were determined with a noble gas mass spectrometer [1]. The thermal and fast neutron flux was estimated from the production of ^{39}Ar and ^{38}Ar from ^{39}K and ^{37}Cl in the Hb3gr hornblende standard, in which K and Cl contents have been determined.

RESULTS: The noble gas and halogen compositions of the metasomatic rocks revealed two distinct fluid inflow events: (1) serpentinitization of the wedge mantle, in which fluids of altered oceanic crustal basalt origin caused the initial serpentinitization of ultramafic bodies in many localities, indicating that dehydration of altered oceanic crustal basalts continues to be an important source of subduction fluids at depths beneath the wedge mantle, and (2) boundary rock formation (formation of talc, amphibole, and chlorite) in a range of different metasomatic lithologies showing relationships between the type of hydrous mineral and the origin of the fluid, which was heterogeneous from place to place and may have changed with time [2].

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Ar-Ar dating of single mineral grains from heavily-shocked chondrites

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INTRODUCTION: Collisions between celestial bodies in space have occurred repeatedly on various scales from the formation of the solar system to the present day. The temperature and pressure increases caused by collisions vary significantly in both time and space. Quantitatively evaluating these physical parameters allows us to extract information about individual collision events. Timing and physicochemical conditions of collisional events could be recorded in Ar-Ar ages of meteorites due to the high volatility of Ar.

We have developed Ar-Ar dating for samples of sub-milligram mass (e.g., Projects #R3007, R3038, R3127, and R5006). Through previous collaborative research, it became possible to perform Ar-Ar dating with approximately seven temperature extraction steps even on a plagioclase grain of <0.1 mg. During the proposed research period, we aimed to achieve gas extraction and dating with more detailed temperature steps, approximately 10-15 steps, to obtain physical conditions during shock-induced heating.

EXPERIMENTS: In this study, we have isolated plagioclase grains from a heavily-shocked (shock stage 5) L6 chondrite, Northwest Africa (NWA) 7984. Grains of orthoclase [1], wollastonite, and sanidine were used as the standard samples. The meteorite samples and our standard samples were placed in a conical dimple ($\phi 1$, depth ~ 0.5 mm) of a sapphire disk ($\phi 5.5$, 1.5 mm thick), and covered with a sapphire disk ($\phi 5.5$, 0.3 mm thick). Each of the sapphire container was wrapped with pure aluminum foil. These Al-wrapped containers were stacked and sealed in the capsules for the Hydro irradiation. The sample irradiation was conducted in November 2025 under our nominal condition ($1\text{MW} \times 46$ hours + $5\text{MW} \times 6$ hours). The irradiated samples were recovered and transported to Kyushu Univ. in December after the decays of short-lived nuclides.

RESULTS: Our original plan was to conduct Ar isotope analysis in March 2026. However, due to repeated failures of the noble gas mass spectrometer, the analysis scheduled is postponed. The irradiated samples have been preserved, and we plan to conduct noble gas isotope analysis as soon as possible after the repair of the mass spectrometer is completed.

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Absorption of alkali metal ions by white radish sprouts (VII)

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INTRODUCTION: ¹³⁷Cs, released by the Fukushima Daiichi nuclear power plant accident in March 2011, has a long half-life and remains in agricultural soils, causing the harmful rumors. Our previous research has shown the possibility of extracting radioactive cesium through ion exchange decontamination using alkali metal ions [1, 2]. However, the rubidium and cesium ions used in decontamination may affect plant growth. Therefore, we aimed to clarify how alkali metal elements affect the absorption and distribution of inorganic elements by plants by cultivating radish sprouts under different concentration conditions of rubidium nitrate and cesium nitrate and measuring the content of trace elements present in the leaves and stems by activation analysis.

EXPERIMENTS: Aqueous solutions of cesium nitrate and rubidium nitrate at concentrations of 10⁻³ mol/L, 10⁻⁴ mol/L, and 10⁻⁵ mol/L, respectively, were prepared and added to germinated radish sprouts. Four samples of each concentration were cultivated, harvested after 10 days, and separated into leaves and stems. After drying, the trace elements contained in each were quantified by instrumental neutron activation analysis.

The samples in polyethylene capsules were irradiated in Pn-3 for 90 seconds. As comparative standards, the certified NIST Standard Reference Material 1577b Bovine Liver as well as elemental standard for Mg was used. The γ -ray spectroscopic measurements with an HPGe detector were performed repeatedly: the first measurements for 120 – 150 seconds after decay time of 5 - 15 minutes and the second one for 250 - 300 seconds after 60 - 150 minutes.

RESULTS: When comparing the manganese concentrations in the group treated with rubidium nitrate and the group treated with cesium nitrate, the concentration was lower in the group treated with cesium. Since manganese is an element involved in plant photosynthesis, it is thought that the absorption of manganese was inhibited by the addition of cesium, which may have caused the growth disorders.

Next, we will discuss alkali metal concentrations. First, in both the rubidium-added group and the cesium-added group, the potassium concentration decreased as the concentration of the added solution increased. This suggests that the absorption of rubidium and cesium ions is competing with the absorption of potassium ions. On the other hand, the sodium concentration was significantly higher in the cesium-added group compared to the rubidium-added group. Furthermore, no concentration dependence was observed in either group. This suggests that even within the same alkali metal group, the way sodium is absorbed differs between plants treated with cesium and those treated with rubidium.

Considering the decrease in manganese concentration in radish sprouts and the differences in sodium absorption in the cesium-added group, as well as the use of fields after decontamination, it is considered that rubidium is better than cesium for decontaminating radioactive cesium from soil.

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Determination of Abundance of Rare Metal Elements in Seafloor Hydrothermal Ore Deposits by INAA Techniques-12: Determination of Barium in Sulfide-sulfate Ore (2)

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INTRODUCTION: We have studied application of INAA technique to quick search for elemental abundance in mineralized samples collected from seafloor hydrothermal deposits. Barite (BaSO_4 as chemical formula) is one of important gangue minerals in such deposits, and Ba content of sulfide-sulfate ores widely varies from a few hundred ppm to more than 50 wt%. Determination of Ba content of sulfide-sulfate ores using conventional ICP-QMS analysis often provides poor results due to incomplete dissolution of ores during the chemical treatment [1]. We tried determination of Ba content using XRF technique, which allows for rapid analysis through a simple procedure. Here, we report a result of crosscheck analysis between INAA technique and XRF technique.

EXPERIMENTS: Sulfide-sulfate ores collected from an active seafloor hydrothermal field in a sea-mount in Izu-Bonin Arc were provided for the crosscheck analysis. Aliquots for INAA and XRF analysis were split from the same powdered sample. For INAA, the samples of 10-20 mg were irradiated at Pn-3 (thermal neutron flux = 4.68×10^{12} n/cm²/sec) for 30 seconds and the gamma ray activity was measured for 3 minutes after adequate cooling time (2-5 minutes). The peak at 661.66 keV of Ba-137m ($t_{1/2}=2.552$ minutes) was used for quantification. The Ba content in the samples was calculated based on relative comparison of peak areas with a pure barite crystal grain collected from the Kosaka mine. XRF analysis was performed in CSREA, Kobe University, using the energy-dispersive XRF analyzer EA1400 (Hitachi High-Tech). Samples for measurement were prepared according to the loose power method. Determination of 10 elements (Mg, Al, Si, S, K, Ca, Fe, Cu, Zn, As, Pb and Ba) was carried out by switching the voltage applied to the X-ray tube between 15 kV and 50 kV, and by selecting appropriate filters. The Ba content was calculated from the X-ray intensity using the FP method, and a correction was applied for the oxygen content which cannot be detected by XRF.

RESULTS: The results of crosscheck analysis for the Ba content was illustrated in Fig. 1. Contents determined by two analytical techniques generally showed good agreement. However, there was a tendency for the XRF measurements to yield higher values for some samples.

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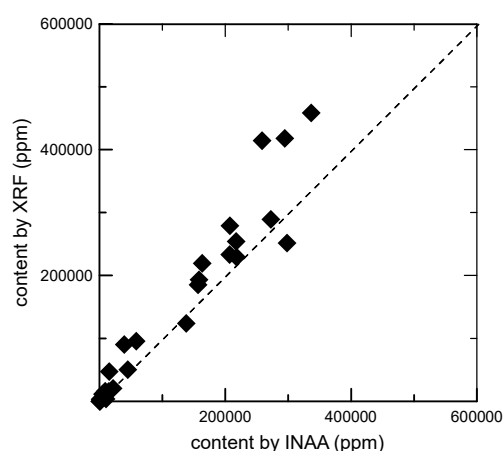


Fig. 1. Result of cross check analyses between INAA and XRF for sulfide-sulfate ores

Fundamental Study on the Migration Behavior of Trace Elements from the Environment into Structural Materials Using INAA

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INTRODUCTION: In Fukushima Daiichi Nuclear Power Plants, various contaminated material had been generated by the severe accidents, especially concrete materials, used as structure materials, experience various conditions such as dipped into contaminated water, stayed under high temperature areas and so on. Therefore, radioactive nuclei, Cs-137 and Cs-134 had been taken in the concrete materials by penetration via solution and/or its melting, and then large amount complicated radioactive wastes were generated [1].

The complexity of contamination into the concrete materials could be related to its micro structure. In this study, the relationship of the penetration behavior and the microstructure was investigated with observation of the micro structure using X-ray and with penetration of Cs into them using INAA.

EXPERIMENTS: Mortar samples (ordinary Portland cements, W/C=0.25 and 0.55, size: 15 mm ×15 mm ×15 mm) were used in the experiments. Acrylic resin was applied to 5 of the 6 surfaces to permit penetration to one surface. The specimens were dipped into 0.01 M CsOH solution for Cs to penetration into themselves for 18 days. Then, dried specimens were grided by a thickness of 0.5 mm 4 times to make powder for INAA of KUR. On the other hand, these specimen transparency images were taken with a X-ray generator (10 keV) and a X-ray image intensifier (Toshiba E5877CS-PIK).

RESULTS: As shown in Fig. 1, in the higher W/C ratio specimen the Cs concentration was higher, which means it is easier for Cs to penetrate into high W/C concrete. Fig.2 shows the transparency images, there are more black spots in W/C=0.55 than W/C=0.25. This black spots are sparse concrete material positions, which might be passes for water containing Cs. From these results, the relationship of the penetration behavior and the microstructure was understood.

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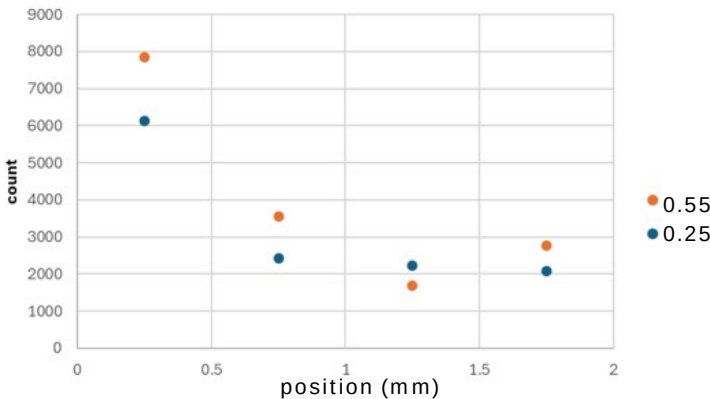


Fig. 1. Cs penetration profiles into concrete specimens
(C/W=0.25 and 0.55)

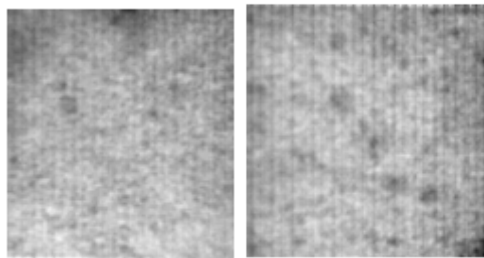


Fig. 2. Transparency images
(C/W=0.25 (left) and 0.55 (right))

Shock deformation and structural evolution of minerals at planetary impact conditions

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INTRODUCTION: Water has played fundamental roles in the evolution of Earth and in the history of organic matter and life. Hydrous asteroids, including C-type asteroids, contain abundant hydrous minerals that store structural water mainly as hydroxyl (OH) groups. Shock-induced dehydration during high-velocity impacts may have reduced the amount of water ultimately delivered to the Earth's oceans and atmosphere. Serpentine, one of the most common hydrous minerals in these asteroids, contains more than 10 wt% structural water. Therefore, understanding the behavior of serpentine under high-velocity impact conditions is essential for evaluating the mechanisms of water transport, retention, and redistribution on the Earth.

In this study, we applied in situ ultrafast X-ray diffraction (XRD) measurements during shock compression to antigorite, a common serpentine mineral, for the first time. Shock pressures up to 106 GPa, corresponding to impact velocities of approximately 8 km/s, were generated to directly observe the structural evolution of serpentine under impact conditions. This year, we mainly focused on analyzing in situ XRD data from shock-compressed antigorite to clarify its structural evolution under high-velocity impact conditions.

EXPERIMENTS: Laser shock-compression experiments coupled with in situ XRD measurements were performed at BL3-EH5 of the SPring-8 Angstrom Compact Free-Electron Laser (SACLA) facility [1]. We prepare a fully dense single-crystal antigorite as the starting material. The target consisted of a polypropylene ablator with a thickness of $50 \pm 1 \mu\text{m}$ and an antigorite layer with a thickness of $50 \pm 1 \mu\text{m}$. The XFEL pulse ($\lambda = 1.13 \text{ \AA}$) was focused to $\sim 10 \mu\text{m}$ vertically and $30 \mu\text{m}$ horizontally at a depth of $25 \mu\text{m}$ within the antigorite sample. The time evolution of antigorite structure under shock compression and subsequent decompression was measured using an XFEL. The recorded two-dimensional diffraction patterns were analyzed using ReciPro software [2]. The arrival time of the shock front at the rear surface of the antigorite sample was determined using a velocity interferometer system for any reflector [1]. The average shock velocity was calculated based on the sample thickness and the measured shock transit time to estimate the shock pressure.

RESULTS: The in situ XRD results showed that shock-compressed antigorite remained crystalline up to 44 GPa, corresponding to an impact velocity of $\sim 4.7 \text{ km/s}$, whereas it transformed into an amorphous state above 66 GPa, corresponding to an impact velocity of $\sim 6.4 \text{ km/s}$. This transformation was completed within a few nanoseconds during the compression stage, and no recrystallization from the amorphous state was observed during subsequent decompression. These observations indicate that shock-induced amorphization is the dominant structural response of antigorite under high-velocity impact conditions. The present results provide important constraints on the collisional history and water-retention processes of hydrous asteroids such as Ryugu and Bennu. In the future of this research, we plan to conduct shock-recovery experiments using a two-stage gas gun to investigate the shock deformation and water behavior of the post-shock samples in detail.

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Development of ^{40}Ar - ^{39}Ar Dating Equipment in KURNS, Kyoto University

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INTRODUCTION: Radiometric dating is an important technique for elucidating the formation and evolution history of terrestrial and planetary materials. The K-Ar and ^{40}Ar - ^{39}Ar methods are invaluable for determining the timing of volcanic eruptions, estimating cooling processes in plutonic rocks, and resolving the timing of heating events on planetesimals and asteroids (e.g., Fernandes, 2024 [1]). The ^{40}Ar - ^{39}Ar dating method with the laser-heating technique is suitable for small samples (e.g., Kelley, 1995 [2] and Hyodo, 2008 [3]). To obtain ^{40}Ar - ^{39}Ar dates on rock and mineral samples from terrestrial and extraterrestrial materials using the facility in KURNS, we have continued developing a ^{40}Ar - ^{39}Ar dating system that includes laser-heating gas extraction and gas purification lines. The system was initially designed by Dr. R. Okazaki (Kyushu University) and has been developed for several years. Previous progress reports have described the system's details and schematic diagram (Nos. 31126, R2079, R3121, R4068, R5122, R6057).

EXPERIMENTS: To extract gas from the small sample, we prepared and adjusted a continuous-wave Nd:YAG laser (LEE Laser model 812TQ), which was installed in 2019 at KURNS. It had a continuous-wave power output of 57 W at the time of installation. However, no laser power output was observed in the latest operation test in March 2025. In FY2025, we again checked the Nd:YAG laser's current condition.

RESULTS: We confirmed that the Nd:YAG laser was malfunctioning. Then we begin considering the replacement of the laser equipment in the KURNS. One possible plan is borrowing a continuous-wave fiber laser (Wuhan Raycus Fiber Laser Technology, RFL-C300) available in KURNS, which has a ~300 W of power output and a 3 mm beam diameter. However, we are concerned that the laser's power output is too high for fusing small rock and mineral samples. Because gradual heating by controlling the laser power from low to high for extracting sample gas stepwisely is desirable for ^{40}Ar - ^{39}Ar dating (e.g. [4], [5]). Another possible plan involves relocating with a diode laser (Spectra Diode Labs., FL20), which has a ~15 W output and a 7.7 mm beam diameter, from Yamagata University. We will continue considering these and other alternatives from several points of view.

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Observation of ^{137}Cs in tree ring of cedar from 2014 to 2025

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INTRODUCTION: Vast forest was markedly contaminated by radioactive plums containing radionuclides especially radiocesium in the wide range of the eastern part of Japan in 2011 [1–8]. As forests have an important role in preventing landslides and maintaining the ecological and hydrological system, the destructive forest should be avoided and an appropriately managed tree-felling should be conducted. In our previous field study [1], most of the radiocesium in the tree rings was directly absorbed by the atmospheric direct uptake via the bark and leaves rather than by roots. We collected cedar samples from 2014 to 2025 and determined radiocesium in annual tree rings of cedar observed at Utsunomiya University Forestry Center.

EXPERIMENTS: The cedar were cut and dried in an oven. They were then packed into U8 containers to create uniform sources for gamma ray measurements. The radioactivity intensity within the samples was measured using a high-purity Ge semiconductor detector (SEIKO EG&G). To quantify the radioactivity intensity of the radionuclides, the peak energy of the gamma rays was used, specifically 662 keV for ^{137}Cs . The detailed measurement methods are described in Ohta et al. [6]

RESULTS: The ^{137}Cs concentration in the annual tree rings of cedar trees did not increase during 2014 - 2025. In cedar trees sampled 3.8 months after the Fukushima accident, the concentration of radioactive cesium in the aboveground sapwood was slightly greater than that in the heartwood. In the Nishiyama area of Nagasaki, where black rain from the atomic bomb is thought to have fallen, the ^{137}Cs concentrations in the annual tree rings of cedar trees were higher in heartwood than in sapwood [8-10]. The annual tree rings of the aboveground parts of the Utsunomiya cedar trees are likely to show increased accumulation of radioactive cesium in the heartwood, similar to the data from the cedar trees observed by Nagasaki [8-10].

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Development of Neutron Detection System for Lunar Water Resource Exploration

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INTRODUCTION: This study aims at the fundamental development of a neutron detection system for a lunar orbiter to investigate the distribution of water resources on the Moon through measurements of lunar-origin neutrons. The presence of water has been suggested in permanently shadowed regions such as the lunar poles and craters. The presence of water on the Moon serves to slow down high-energy neutrons generated by the interaction between galactic cosmic rays and the lunar surface, converting them into thermal neutrons. In other words, observing thermal neutrons originating from the lunar surface using a lunar orbiter can enable the exploration of water resources on the Moon. To enable deployment on a satellite platform, we develop a compact, lightweight neutron detector with a high signal-to-noise ratio, together with a focusing system. The performance of the system is evaluated through ground-based experiments using thermal neutron irradiation.

EXPERIMENTS: As the first step in developing a neutron detection system for satellite deployment, we have developed a neutron detector that combines a plastic scintillator (EJ-270) with a semiconductor photodetector (SiPM). The neutron detector was installed on the sample stage of the CN-3 beamline[1], and the thermal neutrons collimated to 10×1 mm were sequentially irradiated onto multiple locations on the detector surface. The observed signals were identified as neutrons and others using pulse shape separation (PSD) techniques.

RESULTS: Figure 1 shows the signal height, PSD value, and count rate observed when neutrons were irradiated onto the detector. This figure indicates that two types of signals with different PSD values and pulse heights were observed. The intensity of the neutron-induced signal was about 70 cps. These results indicate that the neutron detector we developed is functioning properly with respect to thermal neutrons. While the discrimination capability did not vary significantly depending on the irradiation position, the number of detected neutrons showed a maximum variation of 10% relative to the irradiation position. To investigate the cause of this, we plan to evaluate the irradiation position dependence in greater detail and modify the PSD conditions.

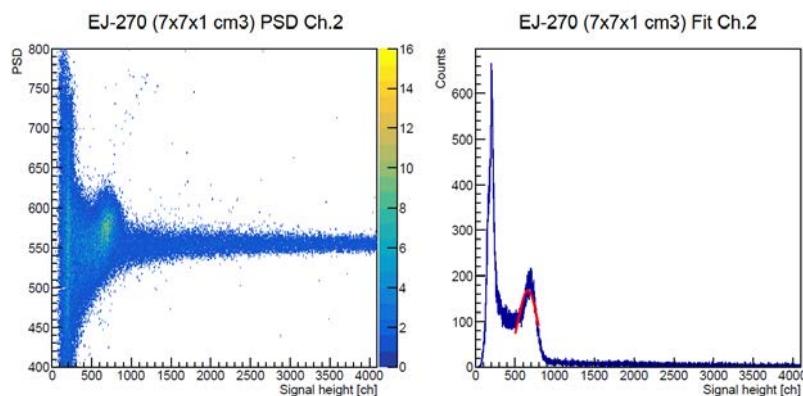


Figure 1. (Left) The relationship between the observed signal height, PSD value, and count, and (right) its projection.

Interaction of molybdate with calcium in saline water

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INTRODUCTION: Molybdenum is a very important element for industry and present in trace amounts in seawater. The recovery of molybdenum in seawater requires a preconcentration step from a large amount of seawater using inexpensive and environmentally friendly reagents. Calcium chloride, which is easily acquired from seawater, was selected in this study. We have investigated the coprecipitation of molybdenum with calcium salts in saline water using radioactive tracers, molybdenum-99.

EXPERIMENTS: Molybdenum-99 was produced using natural isotopic molybdenum through photo nuclear reaction at the KURNS linac [1]. The powder of MoO_3 irradiated above was dissolved with a small amount of 0.1 M NaOH and diluted to prepare a stock solution of molybdenum. A small amount of the stock solution was diluted with 0.5 M NaCl to prepare an initial solution with a molybdenum concentration of 10^{-5} M and then was added with CaCl_2 , NaHCO_3 , and NaOH to induce the precipitation of calcium salts. Molybdenum was recovered by co-precipitating with the calcium salts. Its recovery ratio was calculated based on the total radioactivity and the resulting radioactivity in the aqueous phase.

RESULTS: The molybdenum recovery ratio depended on the concentration of coexisting calcium. The recovery ratio increases significantly when the calcium concentration exceeded 1.5 M in Figure 1. The variation in the data points in the figure might show that factors other than calcium concentration affect the molybdenum recovery ratio. As shown in Figure 2, the molybdenum recovery ratio is significantly affected by pH, particularly when the calcium concentration exceeds 1.5 M. Regardless of the calcium concentration, the recovery ratio was almost constant between pH 6 and pH 11. Conversely, when the pH exceeded 11.8, the recovery ratio decreased.

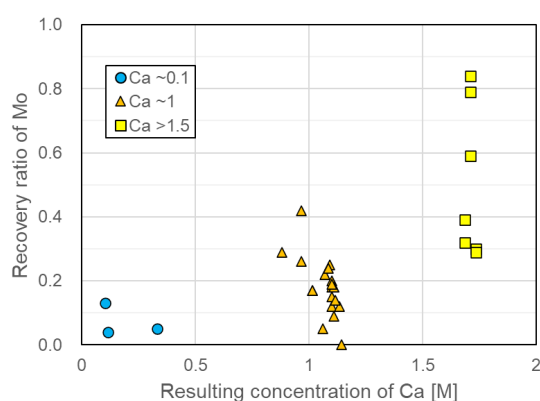


Fig. 1. Recovery ratio of Mo as a function of the resulting concentration of Ca

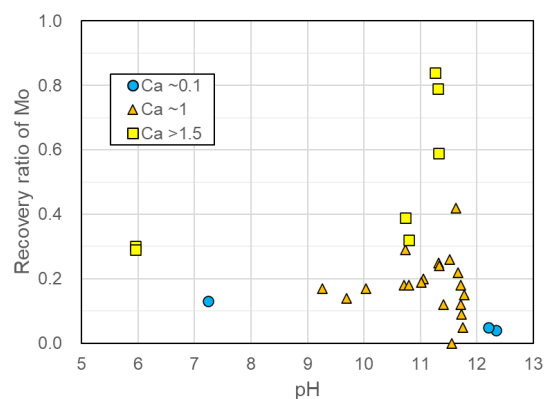


Fig. 2. Recovery ratio of Mo as a function of pH

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Three halogens (Cr, Br and I) in Qiquanhu eucrite

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INTRODUCTION: Eucrites are basaltic achondrites derived from the crust of the asteroid 4 Vesta. These chemical compositions provide important insights into igneous processes and volatile behavior in small planetary bodies. Halogens (Cl, Br and I) are of particular interest because they are highly incompatible and sensitive to secondary processes such as degassing and fluid interaction. However, halogen abundances in Antarctic meteorites are often elevated due to terrestrial weathering, which makes it difficult to extract primary geochemical information. To minimize such effects, Qiquanhu eucrite meteorite was selected in this study. Qiquanhu is a recent fall (847 g) recovered from Tulufan, north-central Xinjiang Uygur Autonomous Region, China, in 2021. In this study, we determined the concentrations of the three halogens (Cl, Br and I) in Qiquanhu eucrite meteorite using RNAA to discuss the degassing history of this eucrite.

EXPERIMENTS: Qiquanhu meteorite was analyzed using RNAA together within the two USGS reference samples (BCR-2 and DTS-2b) as control samples. The analytical procedure of RNAA is described in Ebihara et al. [1]. Samples were weighed into plastic vials and an appropriate amount of each halogen standard solution was dropped on a filter paper and sealed in polyethylene as halogen reference samples. Both of these were irradiated for 20 min at the Kyoto University Research Reactor Institute. Known amounts of three halogens and Mn carriers were added into a Ni crucible with the irradiated reference materials and then were fused in NaOH. The fused cake was digested in water, and a hydroxide precipitate was removed from the supernatant, which contained the halogens. After acidification of the supernatant, iodine was precipitated as PdI₂, and Cl and Br were precipitated as a mixture of AgCl and AgBr precipitates. These precipitates were subjected to the measurement of ¹²⁸I, ³⁸Cl, and ⁸²Br. The chemical yields of the three halogens during radiochemical separation were determined using the reactivation method. To correct the effects of neutron flux gradient and neutron attenuation, samples were sandwiched by a reference standard, which was prepared by dropping a known amount of I standard solution on a filter paper.

RESULTS: The concentrations of Cl, Br, and I in BCR-2 were 102 ± 4 ppm, 0.0875 ± 0.0068 ppm, and 0.012 ± 0.004 ppm, respectively. Those in DTS-2b were 12.3 ± 0.5 ppm, 0.0863 ± 0.0066 ppm, and 0.152 ± 0.010 ppm, respectively. Although our Br data for the two samples were lower than the literature values [1], our Cl and I data were in good agreement with these values. The concentrations of Cl, Br, and I in Qiquanhu were 46.2 ± 1.2 ppm, 0.007 ± 0.002 ppm, and 0.023 ± 0.009 ppm, respectively. The concentration ranges for fall eucrite are 5.7 – 298 ppm for Cl, 0.055 – 0.149 ppm for Br, and 0.010 – 0.048 ppm for I. Although our Cl and I data for Qiquanhu fall within these ranges, its Br value is significantly lower than the literature values [2]. Halogen elements are geochemically classified as incompatible and are generally expected to show positive correlations with other incompatible lithophile elements such as La [2]. However, previous studies [2] observed that halogens in eucrite do not correlate with incompatible lithophile elements, suggesting that their concentrations were affected by degassing during igneous processes. Compared with the literature values for eucrite, Qiquanhu exhibits overall lower halogen concentrations, with a particularly pronounced depletion of Br. This feature suggests that Qiquanhu experienced more extensive degassing than other eucrites.

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Evaluation of Cs and Sr dynamics in forest ecosystems

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INTRODUCTION: The forests which make up about 70% of Fukushima Prefecture remain undecontaminated except for an area of about 20 m from the forest edge. In order to lower the ambient dose rate in the forests and aim for the reconstruction of lifestyles and industries that surround the forests, it is necessary to decontaminate the forests beyond 20 meters from the forest edge in a feasible manner that is appropriate to the local circumstances. The ultimate goal of this study is to evaluate the effect of decontamination on reducing ambient radiation dose within the forest and its negative impact on the forest ecosystem by constructing a model that can quantitatively evaluate the concentration of radioactive cesium in fallen leaves, roots, and soil of trees, as well as the distribution and transfer of nutrients in forest soil. In this fiscal year, we analyzed the distribution and transfer of Cs-137 concentration in vertical soil on forest slopes and in fine roots of trees in the soil as well as nitrogen in the soil.

MONITORING: Litter, organic, and soil layers were collected at approximately 10m grid points (Fig.1) within a 27m x 54m area of a forest slope in Mariyama, Kawauchi Village, Fukushima Prefecture on Sep 3, 2025.

RESULTS AND DISCUSSION: Measurements of Cs-137 concentrations in the litter layer, organic layer, and soil layer revealed that the highest concentration of Cs-137 was in the organic layer, followed by the topsoil layer (0-5 cm). This indicates that the majority of Cs-137 is adsorbed in the organic layer and topsoil layer. As shown in Table 1 and Fig.2 nitrogen concentrations are higher in the surface layer (0-5 cm). Furthermore, the concentration of inorganic nitrogen is much lower than that of total nitrogen, indicating that organic nitrogen is dominant in forest soils, and that a general nitrogen cycle is occurring, similar to other forests studied in this research. Also, from Table 1, it can be seen that in the soil surface layer, the NH₄-N concentration is higher than the NO₃-N concentration at the upper part of the slope (B1, C1), while the opposite is true at the lower part of the slope (B5, C5), showing a difference in inorganic nitrogen depending on the location on the slope. The moisture content at the lower part of the slope is high at approximately 60%, and this result is a phenomenon commonly seen in forest soils under nitrogen restrictions in Japan, suggesting that the impact of disturbance due to the accident is small regarding nitrogen. As the next step, we plan to measure the concentration of Cs-137 in mycorrhizae, and build a model that can evaluate both the effectiveness of decontamination and its impact on forest ecosystems.

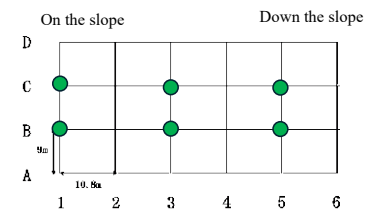


Fig. 1 Sampling points

Table 1 Inorganic nitrogen concentration in soil layers

		NH ₄ -N[mg/kg]	NO ₃ -N[mg/kg]	TN[mg/kg]
B1	Soil layer(0-5cm)	18.41	0.23	4472.81
	Soil layer(5-10cm)	5.18	2.39	1004.47
B5	Soil layer(0-5cm)	5.71	13.28	7133.05
	Soil layer(5-10cm)	7.03	0.65	5738.32
C1	Soil layer(0-5cm)	5.98	3.59	1602.55
	Soil layer(5-10cm)	8.43	1.28	2019.46
C5	Soil layer(0-5cm)	3.60	6.86	4929.02
	Soil layer(5-10cm)	3.12	5.87	4213.44

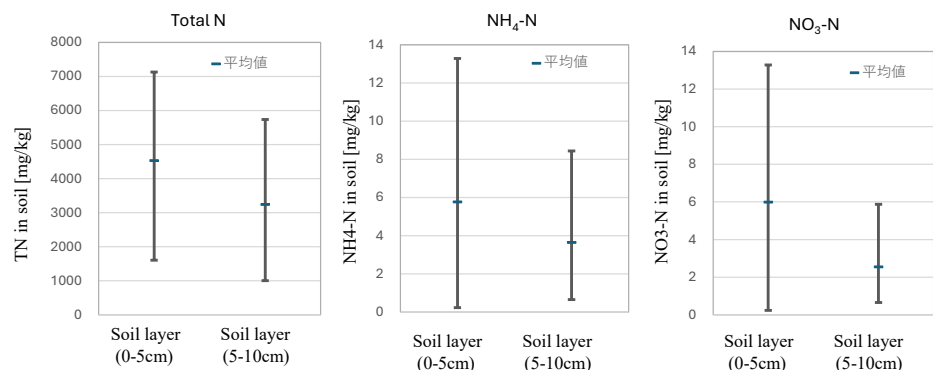


Fig.2 Nitrogen concentration measurements by soil layer

Fission Track Analysis of Fissile Material in Fallout Sample Collected at Maralinga in Australia

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INTRODUCTION: Particles containing fissile materials are formed under conditions of high temperature and high pressure during nuclear disasters or nuclear weapon explosions. These particles can persist in the environment for extended periods. Following the Fukushima Daiichi Nuclear Power Plant accident in 2011, particles containing high concentrations of radiocesium were released and subsequently identified in environmental samples [1, 2]. Some of these particles were found to contain uranium originating from the nuclear fuel. Characterizing these particles provides essential insights for estimating the conditions within damaged reactors. Similarly, particles formed by nuclear explosions carry crucial information for reconstructing the conditions within a fireball. In a previous study, we developed a detection method for fissile substance-containing particles in environmental samples, specifically focusing on those from the Hiroshima atomic bomb [3]. This method combines fission track analysis using a research reactor. The present study aims to detect and analyze fallout particles containing fissile material in samples collected from Maralinga, Australia, one of the major atmospheric nuclear test sites.

EXPERIMENTS: Radioactive fallout particles mixed with sand were collected from the site. Initially, the particles were spread on a sheet of paper and transferred onto a paraffin film by pressing the film against the sample in a stamp-like manner. A fission track detector with dimensions of 20 mm × 20 mm was wrapped in the paraffin film to ensure direct contact between the collected particles and the detector surface. The assembly was irradiated with a thermal neutron fluence of approximately 3×10^{12} n/cm² using the pneumatic irradiation facility (TC-Pn) at the KUR. Following irradiation, the detector was etched in an 8 M NaOH solution at 70°C for one hour. The resulting fission tracks were then observed using an optical microscope.

RESULTS: Figure 1 shows a representative cluster of fission tracks. Similar clusters were observed in samples from Hiroshima and the Daigo Fukuryu Maru (Lucky Dragon No. 5) in our previous analyses. These clusters are consistent with those produced by fine particles containing high concentrations of enriched fissile nuclei derived from nuclear weapons. In this study, the number of tracks in each cluster ranged from several tens to a few hundreds. Furthermore, the ratio of the track density to the thermal neutron fluence for most clusters was comparable to those observed in the Hiroshima soil samples. This indicates that the particles collected at Maralinga contain fissile material at a concentration level similar to the enriched uranium-containing particles from the Hiroshima atomic bomb.

However, based on the observed track distribution, the particle sizes for some clusters are estimated to be from a few to several hundred μm, which is significantly larger than those detected in Hiroshima. Since these larger particles were likely formed through a different process than the smaller ones, further investigations, including chemical analysis and SEM observation, are necessary to elucidate their formation mechanisms.

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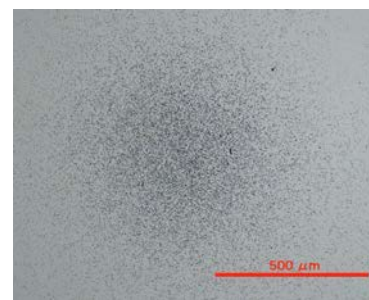


Fig. 1 Example of track-cluster formed by soil particles collected at Marcoo site in Maralinga.

A Study on the Influence of Major Matrix Cations (Ca^{2+} , Na^+ , K^+) on the Analysis of Radiostrontium in Seawater Using a Newly Developed Sr Sorbent

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1. INTRODUCTION: We are developing a method of rapid and safe analysis for radiostrontium in seawater for environmental monitoring. Using a newly developed strontium (Sr) sorbent, "Pureceram MAq® (Nippon Chemical Industries, Ltd.)," composed of barium silicate, it is possible to selectively sorb Sr from seawater.

This study aimed to confirm the interference from major seawater cations in the sorption of Sr, particularly calcium (Ca), which belongs to the same alkaline earth group. This study investigates the sorption behavior of Ca, sodium (Na), and potassium (K) using radioactive tracers.

2. EXPERIMENTS:

2.1 Tracer Preparation

Short-lived radionuclides were produced by neutron activation by 10sec - 30min irradiation: ^{47}Ca (half-life 4.5 d), ^{24}Na (half-life 15 h), and ^{42}K (half-life 12 h). Those irradiated reagents containing these radionuclides were dissolved to prepare tracer solutions.

2.2 Sorption Experiment

Simulated seawater (100 mL adjusted to a 0.1 M HCl) was spiked with the tracers and stirred with Pureceram MAq®. To monitor kinetics, 2 mL aliquots were sampled at 0.5, 1, 2, 3, and 4 hours. The samples were filtered, and the filtrates were measured with a Ge detector. After 4 hours, stirring was stopped, and the mixture was left overnight to reach equilibrium. The supernatant and the Sr sorbent were separated by centrifugation to determine the radioactivity ratio. A parallel experiment used high-Ca seawater (twice of the standard concentration) to assess performance under increased interference.

3. RESULTS AND DISCUSSION: Radioactivity ratios of ^{24}Na , ^{42}K , and ^{47}Ca in the solution remained nearly 100% throughout 4 hours stirring, indicating negligible adsorption by the Sr sorbent. From the overnight samples, the residual rates of ^{24}Na , ^{42}K , and ^{47}Ca in the Sr sorbent were 0.6% or less, it confirms these elements remained in the supernatant.

However, technical limitations were noted for ^{47}Ca tracer. The $^{46}\text{Ca}(\text{n}, \gamma)^{47}\text{Ca}$ reaction yielded low activity due to 0.004% of the natural abundance of ^{46}Ca , and the small neutron cross-section ($\sigma = 0.7$ barn). Therefore, high-precision measurements were not possible. Additionally, the simultaneous production of ^{49}Ca (half-life 8.7 min) requiring a cooling time. Future experiments should allow several days after irradiation for ^{49}Ca decay to improve ^{47}Ca detection accuracy.

4. CONCLUSION: Radioactive tracer experiments effectively track elemental behavior during sorption. The results confirm that Pureceram MAq® have high selectivity for Sr against seawater cations. This method can be effective tool for developing environmental monitoring protocols.

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SAXS/SANS Analysis of *Pinctada fucata* Shell Matrix Protein Nacrein

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INTRODUCTION: Organisms utilize proteins containing intrinsically disordered regions (IDRs) to express specific functions, and understanding their dynamics is essential for elucidating biological phenomena. Small-angle X-ray scattering (SAXS) and small-angle neutron scattering (SANS) directly analyze molecular size and shape in solution, useful for analysis of IDR-containing proteins. This study focuses on nacrein, a shell matrix protein of the pearl oyster (*Pinctada fucata*), which is a model organism for biomineralization. Nacrein interacts with calcium carbonate (CaCO_3), inhibiting random CaCO_3 crystal precipitation and promoting high-purity CaCO_3 deposition. Nacrein is a multi-domain protein consisting of a structured carbonic anhydrase domain and a GXN domain (an IDR with Gly-Xaa-Asn repeats). To understand nacrein's role in shell formation, elucidating the structural characteristics of the GXN domain is essential. Therefore, this study aims to obtain structural information of nacrein in solution through SAXS and SANS measurements.

EXPERIMENTS: Nacrein was extracted and purified from natural pearl oyster shells. Before SAXS measurements, nacrein was purified by size-exclusion chromatography (SEC) to obtain a 1.24 mg/mL sample. Additionally, a solution containing 1.22 mg/mL nacrein and 14 mM CaCO_3 was prepared. SAXS measurements were performed using the Cu-source small-angle X-ray scattering instrument (NANOPIX), installed at KURNS. For SANS, two nacrein samples with solvents of 0% D_2O and 100% D_2O were prepared via SEC, and measurements were conducted at D22 of the Institut Laue-Langevin (ILL).

RESULTS: The overall structure of nacrein in solution was evaluated by SAXS (Fig. 1). Kratky analysis showed typical scattering behavior for proteins containing both a structured domain and an IDR. Furthermore, the pair-distance distribution function $P(r)$ revealed a maximum intramolecular distance D_{max} of 93 Å. This value is significantly larger than expected for the structured carbonic anhydrase domain, predicted from its amino acid composition, indicating a highly extended conformation of the GXN domain in solution. Upon analyzing structural changes during interaction with CaCO_3 , no significant differences were observed in the SAXS profiles with or without CaCO_3 ,

indicating that nacrein interacts with CaCO_3 without undergoing major conformational changes in its overall structure. Subsequent SANS measurements yielded different scattering profiles in 0% D_2O and 100% D_2O (Fig. 2). The radius of gyration R_g obtained by Guinier analysis was 21.5 Å in 0% D_2O and 26.1 Å in 100% D_2O . The difference in R_g arises because the scattering length densities of Gly, Asn, and Asp, which primarily constitute the GXN domain, differ significantly between 0% D_2O and 100% D_2O . Obtaining different spectra by varying the radiation source and solvent D_2O ratio for the same protein has the potential to refine structural ensembles of IDRs obtained from MD simulations. In the future, we aim to yield plausible structural ensembles of nacrein by combining MD simulations with SAXS/SANS.

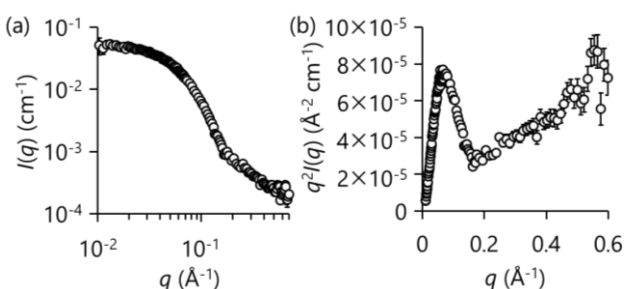


Fig. 1. (a) SAXS profile (b) Kratky plot.

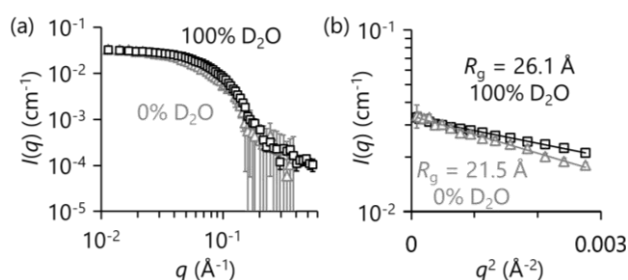


Fig. 2. (a) SANS profiles (b) Guinier plots.

Analysis of the Mint3–FIH-1 interaction that regulates hypoxic stress response.

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INTRODUCTION: Oxygen is essential for many organisms to produce ATP efficiently from nutrients in food. Temporal or local drop of oxygen level induces cells to change the metabolic pathway, which is known as hypoxia stress responses. The responses are mainly triggered by hypoxia inducible factors (HIFs) that activate transcription of a group of genes. Under the normal oxygen conditions, transcriptional activities of HIFs in the cells are inhibited by the factor inhibiting HIF-1 (FIH-1). On the other hand, several cells including tumor cells and macrophages, metabolize nutrients exclusively by nonoxidative glycolytic pathway even under the normal oxygen levels. Munc-18 interacting protein 3 (Mint3) is recently found to activate the hypoxia responses by binding and inhibiting the activity of FIH-1 in these cells [1]. Although the N-terminal 214 residues (Mint3NT) are found to be necessary for the interaction with FIH-1 to inhibit its activity, little is known about inhibitory mechanisms.

EXPERIMENTS: High-expression systems for the N-terminal fragment proteins of Mint3 (Mint3NT) and smaller fragments (Mint3(1–117) and Mint3(101–214)), as well as full-length FIH-1 were constructed by *E. coli* BL21 strains. The proteins were expressed in bacteria grown in LB-broth and ¹⁵N-enriched M9 minimal medium to produce unlabeled and ¹⁵N-labeled proteins, respectively. NMR experiments were performed on a Bruker Avance 600 spectrometer equipped with a triple-resonance probe. A typical ¹H-¹⁵N HSQC experiments were performed at protein concentration of 50 μM. The solvent used were 20 mM sodium phosphate (pH 7.3), and 10% D₂O.

RESULTS: We measured the ¹H-¹⁵N HSQC spectrum of Mint3NT, and found that the dispersion of resonance peaks was very poor particularly along the ¹H-axis, suggesting the absence of strong hydrogen-bonding interactions (Figure 1). Furthermore, the superposition of two HSQC spectra separately recorded for the fragment proteins of Mint3NT (Mint3(1–117) and Mint3(101–214)) reproduced the spectrum of whole protein (Mint3NT), suggesting the absence of significant interaction between the N- and C-terminal regions of Mint3NT.

We also performed a titration experiment of ¹⁵N-labeled Mint3NT by unlabeled FIH-1, and found that the peak intensities for many residues were remarkably decreased in a concentration dependent manner. We analyzed several 3D-triple resonance experiments, and succeeded to unambiguously assign more than 95% of residues. By mapping the change in the peak intensity along the primary structure of the protein, we found that a broad range of residues were significantly affected by the addition of unlabeled FIH-1, suggesting the involvement of these residues in the interaction between Mint3NT and FIH-1.

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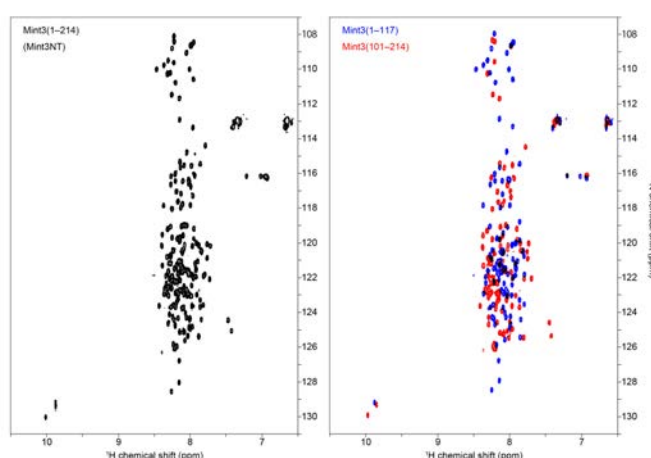


Fig. 1 ¹H-¹⁵N HSQC spectra of Mint3(1–214) (left) and the superposition of fragment proteins (Mint3(1–117) and Mint3(101–214)) (right).

Investigations of Ultra-High Dose Rate Electron Beam Using Alanine

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INTRODUCTION: Alanine dosimeters are organic crystalline amino acids ($C_3H_7NO_2$) that produce a stable free radical upon their irradiation. The concentration of these generated free radicals is proportional to the absorbed dose which can be determined by electron paramagnetic resonance (EPR) spectroscopy. Alanine is water-equivalent (density 1.42 g/cm³, effective atomic number 6.8) and energy-independent (but only above 100 keV) within the therapeutic range (6-25 MeV). However, the response of alanine to ultra-high-dose-rate of electrons has not been reported yet. This experiment aimed to investigate the energy dependence and response of alanine dosimeters to ultra-high-dose-rate electron beams with energies up to 40 MeV from the Electron Linear accelerator facility, Kyoto University.

EXPERIMENTS: Alanine pellets were inserted into a solid water phantom behind a 1 cm buildup phantom and irradiated with electron beams at energies of 9, 20, 30 and 40 MeV, with pulse dose rates up to 10⁶ Gy/s, with pulse duration of 200 ns, and doses ranging from 8, 10, 15, 20, to 30 Gy. The EBT-XD films were placed in front of the alanine pellets during irradiation to verify beam position and validate dose delivery. The energy dependence was evaluated by comparing the relative response of the EPR signal from alanine dosimeters with electron-beam irradiation to that with a Co-60 gamma reference beam (doses 2-50 Gy).

RESULTS: As shown in Fig. 1, alanine dosimeters demonstrated a linear EPR dose response with no significant energy dependence for electron beams over the 9 to 40 MeV energy range. We are currently drafting the results for publication.

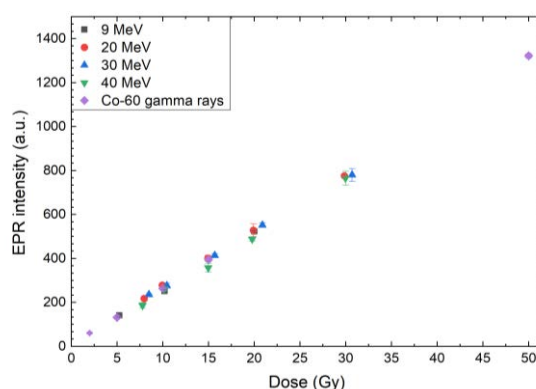


Fig.1. EPR signal amplitude obtained from alanine dosimeter irradiated with 9, 20, 30 and 40 MeV electron beam and Co-60 gamma ray.

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Elucidation of the As(III)-specific aggregation process of the novel identified As(III)-aggregating protein GatB

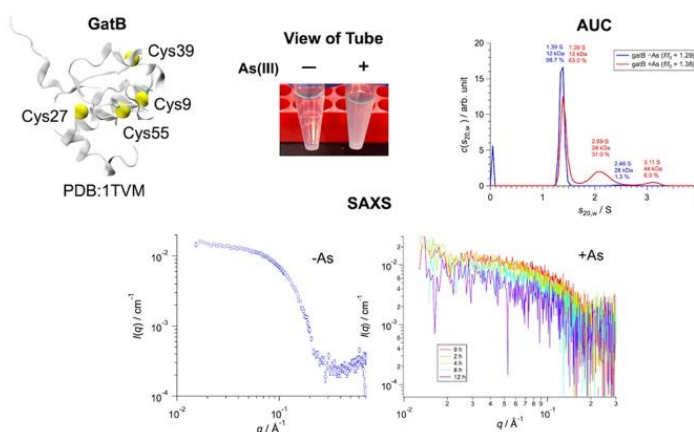
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INTRODUCTION: Trivalent arsenic [As(III)] is highly toxic and can induce protein aggregation by binding to thiol groups [1]. However, the properties of As(III)-aggregating proteins and their aggregation mechanisms remain unclear. We previously showed that As(III)-induced aggregation can function as a protein switch [2]. To systematically identify such proteins, we performed a comprehensive LC-MS/MS analysis of approximately 4,000 proteins in *Escherichia coli*. We identified 23 candidates and confirmed eight as As(III)-aggregating proteins after purification and individual evaluation. Among them, GatB exhibited the strongest aggregation. This study aimed to elucidate the molecular mechanism of the As(III)-specific aggregation process of GatB through physicochemical analyses.

EXPERIMENTS: To identify the As(III)-binding site of GatB, mutants were generated in which the putative reactive cysteine residue (C9) was replaced with serine, threonine, or histidine (C9S, C9T, and C9H). Each mutant and wild-type GatB were purified using Ni-NTA Sepharose, followed by the addition of As(III) to a final concentration of 62.5 ppm and incubation at room temperature for 24 h. Time-dependent changes in molecular assembly size upon As(III) binding were analyzed using DLS (dynamic light scattering), AUC (analytical ultracentrifugation), and SAXS (small-angle X-ray scattering).

RESULTS: Evaluation of As(III)-induced aggregation behavior and As(III) binding of GatB by SDS-PAGE and ICP-MS showed that both the amount of protein and the amount of As(III) in the precipitated fraction increased, suggesting that As(III) was incorporated into the aggregates together with GatB. In the mutants, both the protein amount and the As(III) amount in the precipitated fraction were markedly decreased compared with those in the wild type, indicating that C9 is involved in As(III)-dependent aggregate formation. Moreover, time-course measurements of molecular assembly size by DLS, AUC, and SAXS revealed that GatB aggregates stepwise from monomers to oligomers and eventually to large aggregates upon As(III) binding. Furthermore, MD simulation predicted that As(III) binds to GatB in a bridging manner between C9 and C39, resulting in an increased solvent-exposed surface area of hydrophobic residues. Taken together, these findings suggest that As(III) binding induces hydrophobic interactions, thereby triggering GatB aggregation.



Characterization of transthyretin 49-127 fragment protofibrils by small-angle X-ray scattering combined with analytical ultracentrifugation

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INTRODUCTION: Amyloid fibrils are needle-like protein aggregates associated with various amyloidosis and neurodegenerative diseases. The plasma protein transthyretin (TTR) is a representative amyloid-forming protein that causes TTR amyloidosis. An N-terminally truncated peptide of TTR, designated as TTR49-127, is recognized as a major constituent of TTR-derived amyloid fibrils. The fibril formation process of TTR49-127 is thought to proceed through the formation of precursor protofibrils, which represent a potential target for therapeutic intervention. In this study, we investigated the structure of these protofibrils using a combination of small-angle X-ray scattering (SAXS) and analytical ultracentrifugation (AUC).

EXPERIMENTS: Recombinant TTR49-127 fragment (20 μ M) was dissolved in 20 mM phosphate buffer (pH 7.4) containing 100 mM NaCl. To induce protofibril formation, the samples were incubated statically at 37°C for 3 hours. For comparison, mature amyloid fibrils were prepared by incubating the samples at 37°C for 50 hours with continuous agitation at 1200 rpm. The resulting samples were subsequently subjected to SAXS and AUC measurements to evaluate the size distribution and the average shape or size of the aggregates. SAXS measurements were performed at 25 °C with a 30-minute exposure by a NANOPIX equipped with a HyPix-6000 (Rigaku Corporation). The camera length was 1330 mm. A Cu K- α line (MicroMAX-007HFMR) was used as a beam source, which was further focused and collimated with a confocal multilayer mirror (OptiSAXS). AUC data were obtained at 25 °C and 40000 rpm with a ProteomeLab XL-I analytical ultracentrifuge (Beckman Coulter) and analyzed using SEDFIT based on a sedimentation velocity protocol.

RESULTS: AUC analysis demonstrated the progression of protofibril formation, revealing that over 80% of TTR49-127 in solution existed as protofibrils. The SAXS profile of the protofibrils (Fig. 1A) exhibited a slope close to -1 , indicative of a rod-like morphology, consistent with transmission electron microscopy (TEM) observations [1]. Cross-sectional Guinier analysis estimated the diameter of the protofibrils to be approximately 2.5 nm (Fig. 1B), revealing them to be more than five times thinner than TTR49-127 amyloid fibrils analyzed under the same conditions. Collectively, these results demonstrate that SAXS is a powerful tool for investigating protein aggregates, enabling quantitative characterization of protofibrils.

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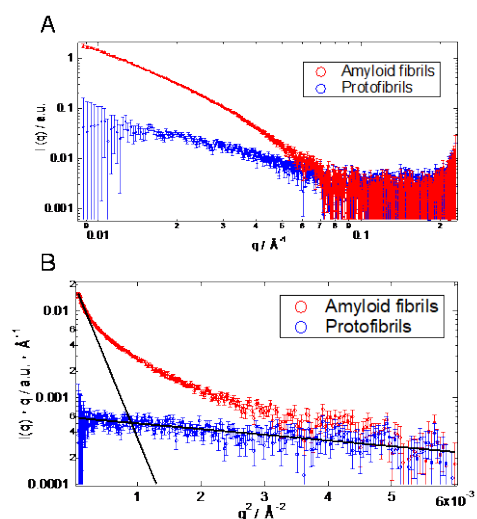


Fig. 1. (A) SAXS profiles and (B) and their cross-sectional Guinier plots of TTR49-127 protofibrils and amyloid fibrils.

Structural and functional characterization of sHsp from psychrophilic archaeon *Methanococcoides burtonii*

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INTRODUCTION: Small heat shock proteins (sHsps) are widespread low-molecular-weight chaperones that prevent protein aggregation under stress [1]. Under non-stress conditions, they form large oligomers; however, upon stress such as heat, they undergo conformational changes that expose hydrophobic surfaces, enabling them to bind denatured proteins. Generally, heat shock causes large sHsp oligomers to dissociate into smaller forms, making it important to analyze their function and structure in this state. However, typical sHsps from mesophiles become active above 37°C, which complicates their study using standard methods. Therefore, we focused on sHsps from the psychrophilic archaeon *Methanococcoides burtonii*, isolated from the approximately 2°C Ace Lake in Antarctica [3]. *M. burtonii* grows between 0 and 28°C, with an optimal growth temperature of 21°C. Its genome encodes two sHsps, Hsp17.3 and Hsp19.8, which differ in size but both contain an α -crystallin domain typical of sHsps. We expressed Hsp17.3 in *E. coli* and performed its characterization.

EXPERIMENTS: The gene for Hsp17.3 was synthesized and cloned into pET23b, then overexpressed in *E. coli* BL21(DE3). The recombinant Hsp17.3 was purified by anion exchange chromatography followed by gel filtration chromatography, and subjected to functional and structural analyses. Its chaperone activity was evaluated based on its ability to suppress aggregation of reduced lysozyme. The oligomeric state was analyzed by sedimentation velocity analytical ultracentrifugation (SV-AUC).

RESULTS: Hsp17.3 was highly expressed in *E. coli* and purified to homogeneity by chromatography. In aggregation suppression assay using reduced lysozyme, Hsp17.3 was able to sufficiently inhibit the aggregation of 30 μ M lysozyme even at 15 μ M concentration. Thus, Hsp17.3 suppressed lysozyme aggregation at approximately half the molar ratio of the target lysozyme. SV-AUC was used to analyze concentration and temperature dependencies in detail. At concentrations of 2 mg/ml or higher, sharp peaks were detected; however, the sedimentation coefficient varied with temperature. As the temperature increased, the apparent molecular weight decreased. At 40°C, the peak intensity diminished, suggesting further dissociation into smaller structures. When the concentration was lowered, the large peak disappeared at 40°C, indicating progression of dissociation. At 0.1 mg/ml, almost complete dissociation into dimers or monomers was observed. Association and dissociation of the Hsp17.3 oligomer are dynamic and highly dependent on temperature and concentration.

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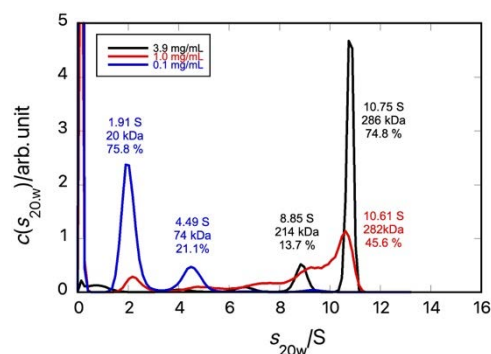


Fig. 1. AUC results of Hsp17.3 at 40°C

Physicochemical characterization of amorphous and nanoparticle structures containing poorly water-soluble compounds prepared by spray-freeze-drying

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INTRODUCTION: Amorphization and particle size reduction are common strategies to improve drug solubility [1]. This study focuses on wet bead milling to produce stable nanocrystals of mefenamic acid (MFA). We investigated how the type and ratio of polymers (polyvinylpyrrolidone; PVP and hydroxypropyl methylcellulose; HPMC) influence the stability and size of the particles, and compared the performance of these nanocrystals with spray-dried solid dispersions.

EXPERIMENTS: MFA suspensions with HPMC or PVP were processed by wet bead milling (UAM-015), while spray-dried samples were prepared for comparison. The physical stability of the samples was monitored for up to 3 months at 40 °C. Powder X-ray diffraction (PXRD) and particle size analysis were used to evaluate changes in crystallinity and particle distribution at specific intervals.

RESULTS: Wet bead milling achieved a time-dependent reduction in the particle size of MFA, with the milling rate significantly influenced by the polymer type and concentration; specifically, higher HPMC content (5:1) slowed the milling rate compared to the 3:10 ratio, while PVP formulations required larger 0.3 mm beads to achieve effective reduction (~300 nm) at a 5:1 ratio. Although all formulations initially showed a decrease in crystallinity, stability studies revealed a clear distinction between the two polymers: HPMC-stabilized nanocrystals remained as free-flowing powders even under high humidity (40 °C/75% RH), whereas PVP-containing samples suffered from moisture-induced aggregation, limiting further analysis to sealed, dry conditions. **Figure 1** shows the PXRD patterns of HPMC-based and PVP-based formulations. According to PXRD analysis, HPMC formulations maintained stable crystallinity regardless of particle size, yet the 3:10 ratio exhibited poor redispersibility and significant sedimentation after two weeks of storage. In contrast, the over-milled PVP 3:10 samples showed an increase in crystallinity after one month. Overall, maintaining samples as suspensions resulted in better particle size stability compared to freeze-dried powders, confirming that the milling behavior and subsequent stability of MFA are highly dependent on the specific polymer-drug composition. These underlying molecular mechanisms will be discussed in detail during the presentation.

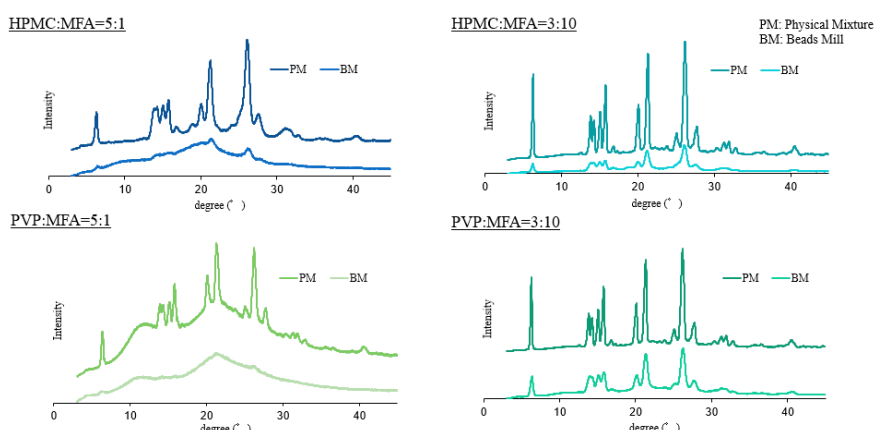


Fig.1 PXRD patterns of HPMC-based and PVP-based formulations.

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Investigation of protein domain motion of PGK through AUC-SAXS measurement and MD simulation

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INTRODUCTION: Biomolecules are natural nanomachines that perform their functions through dynamic motions in solution. Therefore, to understand the molecular mechanisms underlying biomolecular function, it is essential to visualize these motions at atomic resolution. We have succeeded in achieving such visualization by developing the manifold-based gradient method (MBGD), which integrates SAXS data with molecular dynamics (MD) simulations [1]. By applying MBGD to several proteins undergoing domain motions, we have investigated the mechanisms by which protein motions give rise to biological function.

In this year, we focused on phosphoglycerate kinase (PGK) as a representative protein exhibiting such domain motions. We performed AUC-SAXS measurements of PGK and compared the experimentally inferred motions with those observed in MD simulations.

EXPERIMENTS: The PGK sample was loaded onto a size-exclusion chromatography column to obtain a monodisperse protein fraction. After confirming by AUC that the purified PGK was present entirely in the monomeric state, SAXS measurements were performed at a protein concentration of 4 mg/mL at 293 K. The resulting SAXS data were then compared with a 1- μ s MD simulation carried out at 293 K.

RESULTS: During the 1- μ s MD simulation, PGK exhibited an opening-closing domain motion between its domains (Fig. 1A), and the calculated SAXS profile changed depending on this domain motion. A significant difference was observed between the SAXS profile calculated from the MD-derived conformational ensemble (Fig. 1B) and the experimental SAXS profile (Fig. 1C). For example, the radius of gyration estimated from the MD-derived SAXS data was 24.7 Å, whereas that obtained from the experimental SAXS data was 24 Å, indicating that PGK adopts a more compact structure in solution than predicted by the MD simulation.

These results suggest that the inter-domain interactions acting in the MD simulation are weaker than those in the actual solution state. The present SAXS data therefore provide useful experimental information for refining the molecular interactions used in MD simulations.

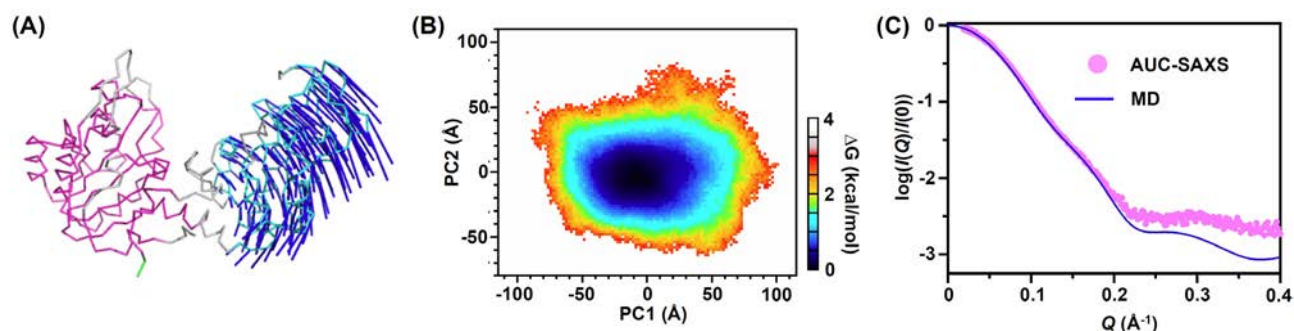


Fig. 1 (A) Domain motion of PGK observed in the 1- μ s MD simulation. (B) MD-derived conformational ensemble projected on the two PC modes. (C) Comparison between experimental (pink) and MD-derived SAXS data.

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Tracking structural growth of transthyretin protofibrils using analytical ultracentrifugation

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INTRODUCTION: Transthyretin (TTR) is a plasma protein carrying small molecules such as thyroxine and retinol. Its amyloid fibril formation is related to systemic amyloidosis known as ATTR, where the fibrils accumulate in organs such as the heart, leading to their dysfunction. Understanding the formation mechanism is thus essential for finding ways to treat the disease. Protofibrils are one of the key species accumulating prior to the amyloid fibril formation. We have recently found that TTR forms protofibrils in an acidic condition [1]. In this study, we tracked structural growth of the protofibrils using analytical ultracentrifugation (AUC).

EXPERIMENTS: TTR at the concentration of 100 μ M in 10 mM HCl and 100 mM NaCl was incubated at 75 °C, and sampled at appropriate time points for AUC experiments. AUC was performed at 25 °C using XL-I (Beckman Coulter, Indiana).

RESULTS: Fig. 1 shows the time dependence of AUC profiles. Immediately after the reaction started (0 h), one peak at $\sim$ 250 kDa was observed, indicating the emergence of aggregates. Several amorphous aggregates were confirmed in the TEM images (Fig. 2, 0 h). These aggregates were identified as oligomers. At 1 h, the AUC profile was largely unchanged from that at 0 h (Fig. 1D, 1 h). In contrast, at 16 h, a broad peak appeared at $\sim$ 1,300 kDa in addition to a peak at $\sim$ 250 kDa (Fig. 1, 16 h). Curvy components were observed in the TEM images (Fig. 2E, 16 h), corresponding to protofibrils. Both the proportion and the molecular weight of the protofibrils increased as a function of time (Fig. 1, 16, 24, 36, and 48 h), indicating protofibril elongation during formation. The elongation was also confirmed by the TEM image (Fig. 2, 48 h).

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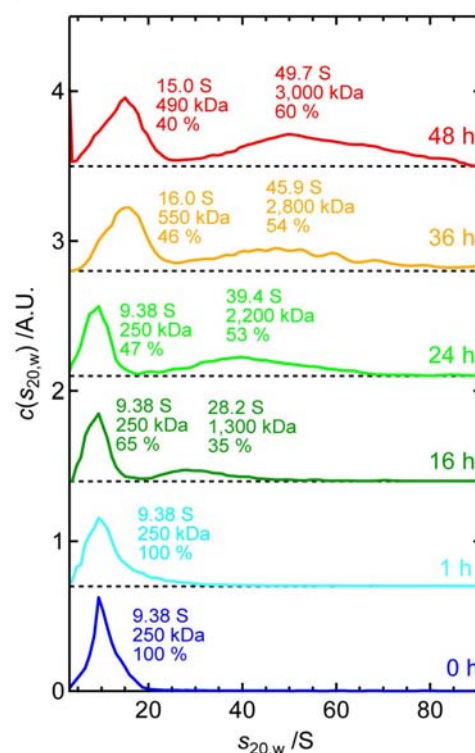


Fig. 1. Time-dependent AUC profiles. The sedimentation coefficient, molecular weight, and population of each component are shown.

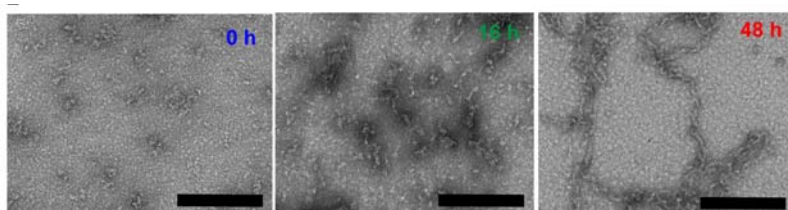


Fig. 2. Negatively-stained TEM images acquired at several time points. The scale bar indicates 200 nm.

2D-Mapping Image of Tumor Model Dried Tissue in the Sub-THz Region

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INTRODUCTION: Spectroscopic analysis in the sub-THz region within the field of tumor pathology is of interest from the perspective of water interactions and is important for understanding hydrogen bonding in the context of MRI.

EXPERIMENTS: The experiment was performed using KURNS-LINAC. The electron beam energy was 39 MeV, and the average beam current was 1.9 μA . The macro-pulse repetition rate was 60 pulses/s. Coherent transition radiation (CTR) from a titanium window was guided to a Fourier transform interferometer in the experimental room via a coherent radiation beamline [1]. A liquid-helium-cooled silicon bolometer was used as the CTR detector. The sample was prepared as a tumor model using cancer drugs at Kwansei-Gakuin University. The sample tissue was sandwiched between two 0.5-mm-thick PVC plates, as shown in Fig. 1.

RESULTS: In a previous study [2], we observed absorption in the tumor area within the spectral region of 11 to 16 cm^{-1} . Figure 2 shows the measurement area for the 2D-mapping image (indicated by the red square). The resulting mapping image is shown in Fig. 3. It was possible to identify the regions of tumor and normal tissues from this image.

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Fig. 1 Experimental setup of the sample.



Fig. 2 Measurement area of 2D-mapping image (square).

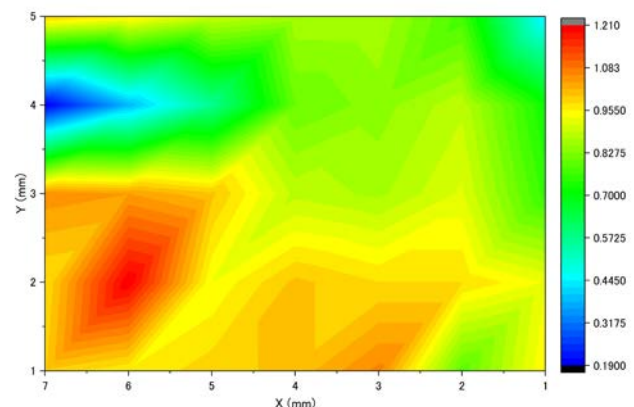


Fig. 3 2D-mapping image of transmittivity in the spectral region from 11 to 16 cm^{-1} .

Smaller oligomers-distributions of hnRNPA2 low-complexity domain by analytical ultracentrifugation measurements

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INTRODUCTION: Proteins with sequence low-complexity (LC domains) have potential to self-associate and play important roles in molecular crowding, called phase separation. Human genetics revealed that patient-derived mutations have impacts on biophysical properties of LC domains, and the F/Y to S mutation in LC domain of hnRNPA2 (A2-LC) has been reported to suppress polymer formation [1]. To monitor a monomeric state and a polymeric state, we compared different mutants of (A2-LC).

EXPERIMENTS: We prepared mCherry-tagged A2-LC (residues 181-341) (mCh:A2-LC) with F291S mutant (A2-LC_F291S), which is a representative mutant known to suppress polymer formation, and a A2-LC mutant (A2-LC_MM) in which multiple F/Y residues were mutated to S (Y257S, Y264S, Y278S, F291S, F309S, and Y319S). All mCherry fusion proteins were expressed in *E. coli* BL21(DE3) cells with 0.5 mM IPTG. Analytical ultracentrifugation (AUC) measurements were performed with a ProteomeLab XL-I (software version 6.2, Beckman Coulter, USA). All samples were prepared at a concentration of 1.0 mg/mL in a solution containing 20 mM Tris-HCl, pH 7.5, 200 mM NaCl, 20 mM β -ME, and 0.1 mM PMSF. The samples were loaded into a cell with a 12 mm optical-pathlength and were measured using Rayleigh interference optics at a rotor speed of 20,000 rpm and temperature of 25 °C. The time evolution of the sedimentation data was analyzed with Lamm formula using SEDFIT software (version 15.01c) [2]. The weight concentration distribution $c(s_{20,w})$ was obtained as a function of the sedimentation coefficient which was normalized to the value at 20 °C in pure water $s_{20,w}$.

RESULTS: As shown in Fig. 1, AUC for mCh:A2-LC, mCh:A2-LC_F291S, and mCh:A2-LC_MM showed the presence of not only monomers but also dimers (and trimers for mCh:A2-LC_F291S). The weight fractions of dimers and trimers for mCh:A2-LC and mCh:A2-LC_F291S were higher than that for mCh:A2-LC_MM, suggesting a stronger tendency for association.

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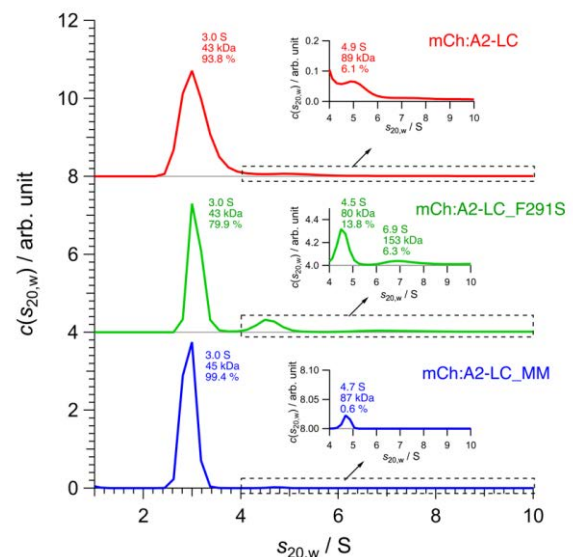


Fig. 1. Smaller oligomers-distributions in mCh:A2-LC samples by AUC measurements. Red, green, and blue lines show the weight concentration distribution $c(s_{20,w})$ for mCh:A2-LC, mCh:A2-LC_F291S, and mCh:A2-LC_MM, respectively. Sedimentation coefficient, molecular mass, and weight fraction for each peak are noted in the figure.

Evaluation of oxidized lens β B2-crystallins involved in age-related cataract

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INTRODUCTION: The transparency of the lens is maintained by the stable long-lived protein interactions, comprising three kinds of crystallin families. Those proteins are very stable, but are decreased their stability with age. A kind of intrinsic damages of lens crystallins, such as heat damage, or extrinsic damages such as UV damage are increased with life time. Several researches suggested the amount of UV damage on lens crystallins could be well described by the amount of modifications of amino acid residues [1]. Oxidation of tryptophan residue (Trp: W) is one of the primary damages for crystallins after heat/UV exposure. Therefore, we made oxidation mimic lens β B2-crystallin and investigated those biochemical effect for oligomerization and stabilities. In order to investigate more details, we used those proteins for SAX experiment to compare β B2-crystallin mimic protein with reported structures.

EXPERIMENTS: Five tryptophan residues in β B2-crystallin were replaced by phenylalanine residues (Phe: F) respectively to simulate the oxidation effect of Trp in β B2-crystallin by site-directed mutagenesis (W59F, W82F, W85F, W151F and W196F). Each sample were purified and used for the SAXS measurements using by NANOPIX (Rigaku, Japan) at 298 K. The detected two-dimensional scattering patterns were then converted to a one-dimensional scattering profile using circular averaging. An ab initio model was built with a dummy atom method, DAMMIF in the ATSAS software package (version 3.1.3) based on the SAXS profile [2]. 7K7U.pdb and 1OKI.pdb were used to generate SAXS profiles for comparing with the experimental results in this study.

RESULTS: SAXS analysis revealed that the β B2-crystallin dimer model in solution resembles the lattice β B1-crystallin dimer (Fig. 1a and 1b), while the tetramer model from resembles its lattice tetramer (data not shown). The W $\rightarrow$ F mutations did not significantly alter the shape of each homo-oligomer; however, the distribution of oligomeric states differed among the mutants. These results suggest that oxidation affects the homo-oligomer assembly of crystallin without dramatically altering its overall structure. Notably, W59F showed an increased proportion of tetramers and decreased thermal stability, indicating that the effects of oxidation are complex rather than uniform. Through these experiments, we were able to obtain insights into the effects of oxidative modification of tryptophan residues—one of the initial focuses of this year's plan—on the homo-oligomeric structure of β B2-crystallin. On the other hand, β B2-crystallin is known to form hetero-oligomers with β A3-crystallin, a partner subunit in the lens [3]. Therefore, in future work, we aim to investigate the hetero-oligomerization ability of the oxidized β B2-crystallin generated in this study with β A3-crystallin, as well as to analyze their structures.

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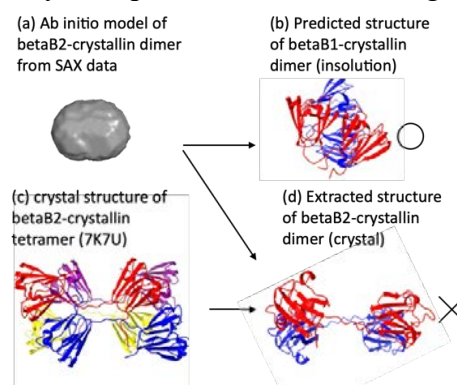


Fig. 1. Superposition of betaB2-crystallin dimer ab initio model and betaB1-crystallin dimer/ extracted structure of betaB2-crystallin dimer.

^{11}C Medical-isotope Production via $^{12}\text{C}(\gamma,n)^{11}\text{C}$ Reaction with Carbon Nanorings

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INTRODUCTION: L- ^{11}C -Methionine is employed as a reagent for PET imaging to diagnose brain tumors [1]. The medical ^{11}C radioisotopes are mostly produced in a cyclotron via the $^{14}\text{N}(p,\alpha)^{11}\text{C}$ reaction by bombarding nitrogen gas with a proton beam [2]. Instead of producing ^{11}C with the cyclotron, we developed a novel method of producing ^{11}C using the bremsstrahlung γ -rays with a carbon nanoring (CNR) to get a reasonable number of the ^{11}C activity.

EXPERIMENTS: Figure 1 shows the experimental scheme for the ^{11}C production via the $^{12}\text{C}(\gamma,n)^{11}\text{C}$ reaction. Bremsstrahlung γ -rays were produced by impinging a 40 MeV electron beam on platinum converter at the electron LINAC facility. The γ -rays were irradiated to CNR sealed in aluminum vessel with non-woven masks as gas inlet/outlet filter. The produced ^{11}C inside the vessel were oxidized to ^{11}CO or $^{11}\text{CO}_2$ in O_2 gas, which was continuously flown during the irradiation. The ^{11}C gas (^{11}CO and $^{11}\text{CO}_2$) were trapped in two 13X molecular sieve columns. 511-keV γ -rays from positron-electron annihilation were detected with CdZnTe detectors. The CNR with a diameter of 0.8 nm ($\text{C}_{36}\text{H}_{24}$) and 1.6 nm ($\text{C}_{72}\text{H}_{48}$) were used as target.

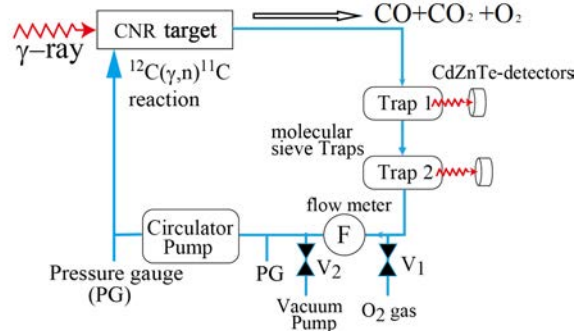


Fig. 1 Experimental scheme to produce ^{11}C and measure the extraction rate of ^{11}C gas.

RESULTS: Table 1 shows the normalized activity of ^{11}C gas for $\text{C}_{36}\text{H}_{24}$ and $\text{C}_{72}\text{H}_{48}$. The normalized activity for $\text{C}_{72}\text{H}_{48}$ was about two times higher than that for $\text{C}_{36}\text{H}_{24}$.

Based on the experimental production rate of ^{11}C gas, we estimate that we can obtain the ^{11}C activity of 117 GBq when we use a $\text{C}_{72}\text{H}_{48}$ target under the conditions with 1) a length of 50 cm, 2) weight of 1.13 kg for 0.155 g/cm^3 , 3) a 40 MeV electron beam intensity of $100 \mu\text{A}$ for 40 minutes bombardment.

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Table 1 Normalized activity of ^{11}C gas for each type of target

Type of target	Normalized activity of ^{11}C gas (kBq/ μA /g/min)
CNR ($\text{C}_{36}\text{H}_{24}$)	14.74 ± 0.09
CNR ($\text{C}_{72}\text{H}_{48}$)	29.11 ± 0.08

Investigation of the Biological Impact of Ultra-High Dose Rate Radiation on Normal and Neoplastic Tissues

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INTRODUCTION: Recent studies suggest that ultra-high dose rate irradiation (≥ 40 Gy/sec), which is over 1,000 times faster than conventional radiotherapy (≈ 0.03 Gy/sec), may reduce side effects while maintaining comparable anti-tumor efficacy [1]. Radiation with different dose rates is known to have different effects on normal tissues [2]. On the other hand, the effect of dose rate on the antitumor effect is minor, and a higher dose rate may improve the therapeutic efficacy ratio of radiation [3]. The purpose of this study is to ascertain whether the dose rate alters the effects of various normal tissues. In addition, experiments were conducted to see if the antitumor effect of radiation could be differentiated by differences in dose rate, which had previously been equivalent, by using drugs that affect the antitumor effect of radiation in combination.

EXPERIMENTS: We aim to clarify the effects of high-dose rate irradiation—which exhibits distinct biological responses compared to conventional X-ray irradiation—on lymphocytes. First, we established in vivo/in vitro irradiation systems for high-dose-rate exposure using electron beams. Next, we administered high-dose-rate irradiation to the head and neck regions of C3H and C57/BL6 mice, confirming that dermatitis in the high-dose-rate group was reduced compared to the conventional dose-rate group, consistent with published reports. Furthermore, lymphocytes isolated from the spleens of C3H mice were irradiated with 0, 2, 4, and 10 Gy using both conventional and high-dose-rate methods. Based on literature indicating the critical role of oxygen concentration in high-dose rate effects, irradiations were conducted under in vitro conditions with varying oxygen levels. Subsequently, using 5% oxygen conditions (validated for high-dose rate effects in mouse skin), additional irradiations of 0, 4, and 10 Gy were performed under both 1% and 5% oxygen conditions.

RESULTS: High-dose-rate irradiation to the head and neck regions of C3H and C57/BL6 mice confirmed that dermatitis was reduced in the high-dose-rate group compared to the conventional dose-rate group, consistent with prior reports. Under 0.1% oxygen conditions, no difference in lymphocyte survival rates was observed between high-dose-rate and X-ray irradiation, and no high-dose rate effect was detected at this oxygen level. Using 5% oxygen conditions (previously validated for high-dose rate effects in mouse skin), 0, 4, and 10 Gy irradiations were repeated under both 1% and 5% oxygen levels. However, under all conditions, no irradiation method-dependent differences in lymphocyte survival rates were observed, and in vitro experiments showed no high-dose rate effect regardless of oxygen concentration.

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Association kinetics of circadian Kai-protein complex in solution

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INTRODUCTION:

Cyanobacterial circadian oscillations arise from complex formation among KaiA, KaiB, and KaiC, coupled to the phosphorylation cycle of KaiC. While the formation of KaiA–KaiC (AC) and KaiB–KaiC (BC) complexes has been characterized, understanding how KaiA associates with the preformed BC complex is essential for clarifying the dynamic regulation of the oscillator.

In particular, once the B_6C_6 is formed, KaiA binding leads to the formation of ABC complexes with variable stoichiometry ($A_nB_6C_6$). In this study, we focus on the kinetics of KaiA association to preformed B_6C_6 complexes using time-resolved small-angle X-ray scattering (SAXS) [1]. The aim is to directly observe how the ABC complex forms and reaches equilibrium immediately after mixing.

EXPERIMENTS:

B_6C_6 was prepared by mixing KaiB and KaiC_{DE} (hyperphosphorylation S431D/T432E mutant) at a molar ratio of $[KaiB] : [KaiC] = 6 : 6$, followed by incubation at 30 °C for 24 hours and purification by size exclusion chromatography. KaiA was added to the preformed B_6C_6 solution at a defined mixing ratio of $[A] : [B_6C_6] = 6 : 1$.

SAXS measurements were performed immediately after mixing KaiA and B_6C_6 at 30 °C using a laboratory SAXS instrument (NANOPIX, Rigaku). Scattering profiles were collected at 2-min intervals immediately after mixing. The scattering intensity was analyzed using Guinier approximation to obtain the forward scattering intensity $I(0)$, which reflects the molecular assembly state.

RESULTS:

Immediately after mixing KaiA with B_6C_6 , the $I(0)$ reached a constant value within the first measurement time point (≤ 2 min) (Fig. 1). The $I(0)$ value agreed well with the calculated value expected for the $A_6B_6C_6$ complex, rather than a mixture of B_6C_6 and $A_{12}B_6C_6$. This indicates that KaiA is not forming a fully saturated $A_{12}B_6C_6$, but instead distributes evenly among B_6C_6 to form $A_nB_6C_6$. Such rapid redistribution implies that KaiA occupancy in ABC complexes is dynamically determined by the mixing ratio rather than fixed stoichiometry. This mechanism enables flexible allocation of KaiA among complexes, which is likely essential for maintaining robust circadian oscillations under fluctuating conditions.

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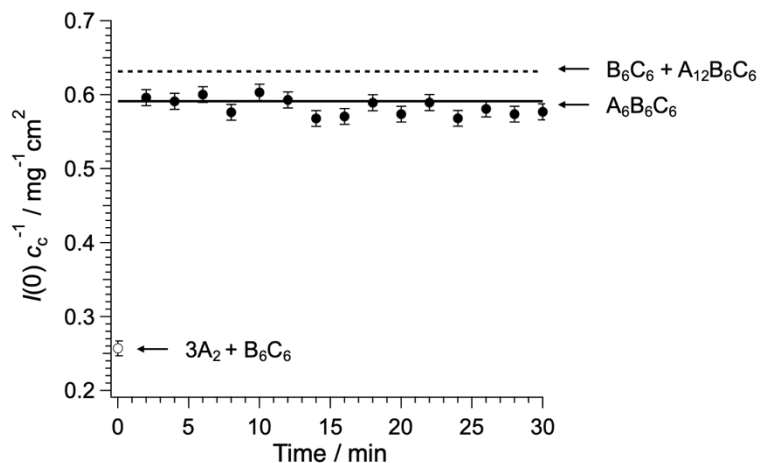


Fig. 1. Closed circles represent the forward scattering intensity normalized by KaiC-concentration ($I(0)c_c^{-1}$) provided by the time-resolved SAXS after mixing KaiA and B_6C_6 . Open circle is the values for the dissociated (A_2 and B_6C_6) state. Solid line corresponds to the calculated $I(0)c_c^{-1}$ -value for $A_6B_6C_6$. Broken black line corresponds to that for the summation of B_6C_6 and $A_{12}B_6C_6$, respectively.

Site-specific deuteration of multi-domain proteins for small angle neutron scattering

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INTRODUCTION: Small-angle X-ray scattering (SAXS) provides structural ensembles of proteins in solution. By comparing these profiles with molecular dynamics (MD) simulations, the dynamics of flexible multi-domain proteins can be analyzed. However, SAXS data interpretation remains ambiguous as it yields only a single curve for the entire molecule. Conversely, small-angle neutron scattering (SANS) with site-specific deuteration can significantly reduce this uncertainty by isolating scattering from specific domains. Despite its potential, preparing such samples remains technically challenging. Specifically, these samples are prepared by ligating protonated and deuterated fragments using ligation enzymes. Using the multi-domain protein Hef—which consists of N-terminal helicase and C-terminal nuclease domains connected by an intrinsically disordered region (IDR) [1,2]—as a model, we previously succeeded in selectively deuterating either only the N- or C-terminal domain using NpuDnaE as a ligation enzyme (Fig 1 a,b). Here, we aimed to prepare a Hef sample where both terminal domains are exclusively deuterated (Fig 1c), leaving only the central IDR protonated to selectively observe its scattering profile.

EXPERIMENTS: Ligation efficiency depends on the enzyme type and steric hindrance at the target site. While NpuDnaE was used for the IDR and C-terminal domain, we screened three enzymes at two positions (six patterns total) to optimize the N-terminal–IDR ligation (Fig 1c). These small-scale tests were performed using non-deuterated proteins to identify the most efficient combination for subsequent large-scale preparation.

RESULTS: Small-scale screening identified a single successful combination using OaAEP. Applying this to site-specific deuteration, we utilized 2 L of deuterated culture for each terminal domain and 2 L of non-deuterated culture for the IDR. This process successfully yielded 1.5 mg of the final site-specifically deuterated Hef protein (Fig 1d).

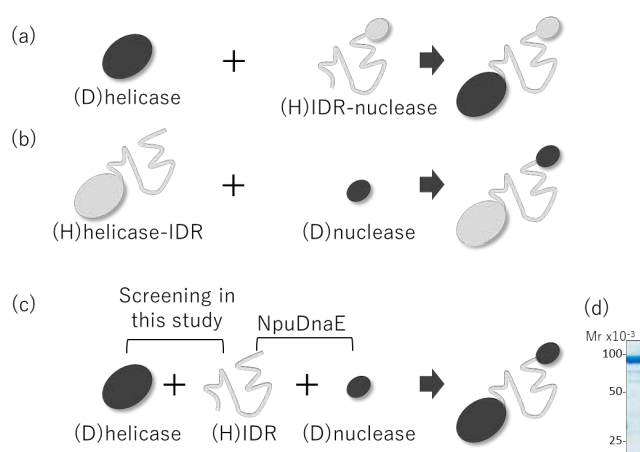


Fig 1. Schematic representation of site-specific deuteration strategies for Hef. (a, b) Preparation of Hef samples with either the N-terminal or C-terminal domain selectively deuterated. (c) Strategy for preparing the site-specifically deuterated Hef where both terminal domains are deuterated while the central IDR remains protonated. The ligation between IDR and nuclease was performed using NpuDnaE, whereas the ligation between helicase and IDR was optimized through screening in this study. (d) SDS-PAGE analysis of the final purified product (c) after ligation and purification.

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Distribution analysis of the chemical modification of the amino acid residues in lens structural proteins during age-related cataract

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INTRODUCTION: Age-related cataracts are a leading cause of blindness worldwide. Several risk factors contributing to cataract development have been identified, among which abnormal aggregation and insolubilization of lens proteins are considered primary mechanisms. Structural alterations induced by covalent post-translational modifications play a critical role in lens protein homeostasis [1]. We have previously demonstrated that such modifications can serve as markers of lens aging. Although mass spectrometry is a powerful tool for quantitative analysis, the spatial distribution of these modifications remains unclear. In this study, we aim to elucidate the distribution of modifications, particularly isomerization and racemization of Asp residues, in lens sections using imaging mass spectrometry (IMS) [2]. To facilitate this analysis, we examined model peptides on a sample plate with a matrix solution to identify peptides based on their molecular mass.

EXPERIMENTS: Model peptides derived from lens proteins were synthesized, purified, and mixed with either CHCA ((4-hydroxyphenyl) acrylic acid) or DHB (2,5-dihydroxybenzoic acid) as matrices for MALDI-TOF MS in 50% acetonitrile. Modification sites (L- α -Asp, L- β -Asp, D- α -Asp, and D- β -Asp) were confirmed by LC-MS/MS at the Kyoto University Institute for Integrated Radiation and Nuclear Science. Samples were mixed with matrixes and loaded onto an imaging plate at three peptide concentrations. Data were acquired in positive ion mode (m/z 100–2500) using a JMS-S3000 (JEOL), and a peak at m/z 1311.70–1312.05 was observed (Fig. 1).

RESULTS: The results suggested that peptide concentrations above 1.0 mg/mL produce detectable images for both matrixes. Notably, DHB enabled more stable detection at lower concentrations compared to CHCA. The observed signal patterns were heterogeneous rather than uniform. Drying of droplets containing the peptide–matrix mixture can generate debris on the plate, non-uniform crystallization which interferes with precise quantitative analysis. The ion distribution images obtained by IMS reflect the signal intensity of ions with a specific mass-to-charge ratio (m/z) at each pixel. By converting these image data into numerical values, the relative abundance of target molecules in the sample was compared. Absolute quantification in IMS is difficult because ion intensity is affected by factors such as matrix crystal state and ionization efficiency. However, among samples prepared and measured under identical conditions, relative comparisons of peptide abundance can be achieved using an index based on pixel count $\times$ mean intensity (Integrated Density). This approach enables quantitative evaluation of peptide abundance in addition to spatial localization.

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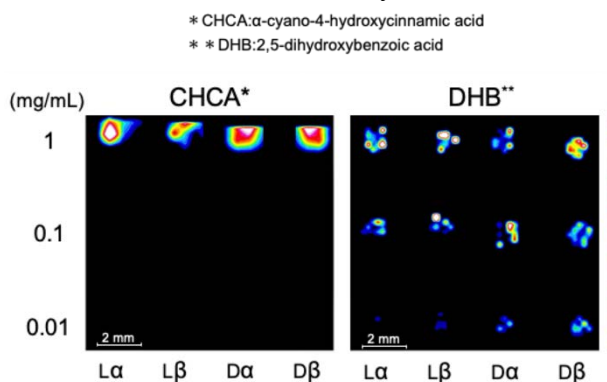


Fig. 1. IMS of lens protein derived peptide containing isomerization/racemization of Asp

Micro–Macro Correlations in the Dynamic Structural Properties of Multidomain Proteins

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INTRODUCTION: Multidomain proteins frequently exhibit intrinsic structural flexibility that underlies their biological functions through allosteric regulation and dynamic intermolecular interactions. In particular, antibodies are known to initiate antigen recognition through the Fab region and subsequently activate effector functions through the Fc region. Increasing evidence suggests that antigen recognition and effector activation are dynamically coupled through allosteric communication pathways embedded within the flexible architecture of antibody molecules. Elucidating such dynamic structure–function relationships is essential for understanding biomolecular function and for establishing principles for functional molecular design. In this study, we investigated the solution structural properties of two distinct multidomain protein systems using small-angle X-ray scattering (SAXS) combined with analytical ultracentrifugation (AUC). One system focuses on the allosteric structural response of human immunoglobulin G (IgG) upon interaction with Fcγ receptor IIIb (FcγRIIIb), while the other addresses the dynamic structural properties of nucleobindin proteins (NUCB1 and NUCB2), which are Golgi-associated proteins involved in glycoprotein processing.

EXPERIMENTS: SAXS and sedimentation velocity AUC measurements were performed for IgG-related samples and NUCB proteins in solution. For the IgG system, single-arm IgG (sIgG) was analyzed to enhance detection of conformational changes associated with FcγRIIIb binding. For the NUCB system, solution properties of NUCB1, NUCB2, and their mixed samples were examined to characterize conformational heterogeneity and possible oligomerization behavior.

RESULTS: For the sIgG/FcγRIIIb system, AUC measurements supported complex formation in solution. Combination of SAXS and AUC analyses enabled extraction of scattering profiles corresponding predominantly to the receptor-bound state while minimizing contributions from free components and aggregates. Structural interpretation of the SAXS data combined with computational modeling is currently in progress. For the NUCB system, AUC analyses suggested no clear formation of stable higher-order oligomers under the examined conditions, while distinct differences in conformational distributions were observed between NUCB1 and NUCB2. SAXS measurements further indicated that NUCB1 exhibited aggregation tendencies, whereas NUCB2 showed solution scattering profiles consistent with relatively expanded conformations. These observations suggest that differences in dynamic conformational properties may contribute to their distinct substrate recognition behaviors. The present study demonstrates the usefulness of SAXS combined with complementary biophysical approaches for characterizing dynamic structural properties of multidomain proteins in solution. Further integration with NMR analyses and computational approaches is expected to provide deeper insights into allosteric regulation and conformational heterogeneity in flexible biomolecular systems.

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Aggregation inhibition development based on p53 aggregates analysis

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INTRODUCTION:

Cancer remains the top cause of death and its incidence rate is also high in Japan. Most anticancer drugs have severe side effects, making treatment a harsh struggle. While molecularly targeted drugs and immune checkpoint inhibitors with significantly reduced side effects have been released in recent years, their applicability is limited to certain types of cancer. In cancer, DNA damage fails to be repaired, causing the bodily systems to function abnormally. Typically, the organism possesses a system that prevents cells from becoming cancerous by repairing DNA damage through the function of tumor suppressor. p53 is one of these factors and is called as the “guardian of the genome” due to its highly effective function. p53 is known to be mutated in about half of all human cancers. Therefore, p53 is a natural anticancer and cancer-preventive drug. In this study, we focused on maintaining the function of p53.

EXPERIMENTS:

p53-DNA binding domain (DBD) was expressed in an E. coli expression system and purified. The p53-DBD solution was treated with a reagent at various concentrations, incubated at 37°C for 1 h, and then analyzed using various techniques, including Blue Native PAGE, transmission electron microscopy (TEM) observation, and nanoparticle tracking analysis.

RESULTS:

We performed nanoparticle tracking analysis on the incubated p53-DBD solution without a reagent, with moderate concentration of a reagent, or with high concentration of a reagent (Fig. 1). As a result, numerous particles derived from p53-DBD aggregates were observed in the p53-DBD incubation samples with either no reagent or a high concentration of a reagent. Interestingly, it was shown that aggregation is significantly suppressed at moderate concentrations of a reagent. We hypothesize that the type of aggregates switches upon the addition of a reagent. TEM observations also confirmed the similar results. Furthermore, BN-PAGE experiments revealed bands in the high-molecular-weight region in samples without or with high concentrations of a reagent. However, at a moderate concentration of a reagent, no bands were observed in the high-molecular-weight region, but bands were observed in the relatively low-molecular-weight region. In the future, we will further analyze the molecular structural characteristics of p53-DBD with a moderate-concentration reagent using AUC, DLS, NMR, or SAXS.

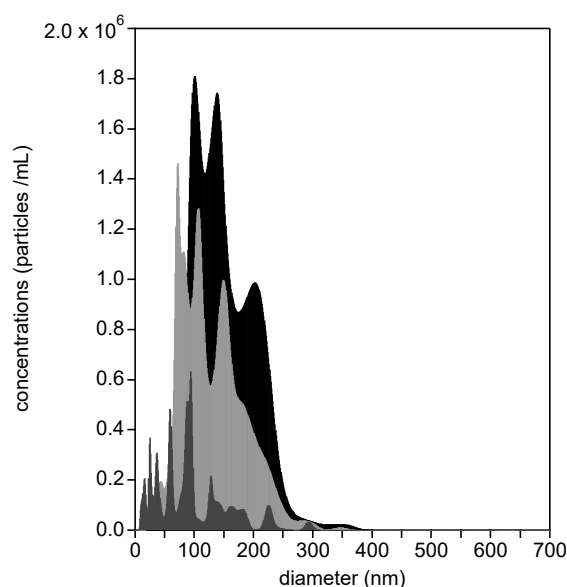


Fig. 1. Nanoparticle tracking analysis of incubated p53-DBD solutions. Black indicates without a reagent, dark gray and light gray indicate moderate and high concentration of a reagent, respectively.

High-resolution in vivo imaging of ^{211}At -labeled radioactive gold nanoparticles

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INTRODUCTION: Drug delivery systems (DDS) that deliver drugs to tumors have attracted attention to enhance treatment efficacy. Gold nanoparticles (AuNPs) are promising drug carriers, and ^{211}At -labeled AuNPs have been actively studied because the alpha particles emitted by ^{211}At provide strong local therapeutic effects[1]. However, the pharmacokinetics of AuNPs remain insufficiently understood. To address this issue, we proposed an “activation imaging” of AuNPs, in which stable ^{197}Au is irradiated with low-energy neutrons and converted into the radioisotope ^{198}Au [2]. The [^{198}Au]AuNPs can then be visualized using the 412-keV gamma rays emitted by ^{198}Au .

EXPERIMENTS: The AuNP precursor, HAuCl_4 , was irradiated with neutrons at the Pn-2 beamline of the Kyoto University Research Reactor (KUR). [^{198}Au]AuNPs were then synthesized, and ^{211}At was labeled onto them (Fig. 1(a)). The ^{211}At -[^{198}Au]AuNPs were administered to tumor-bearing mice intratumorally. Imaging was performed using a high-contrast SPECT (HC-SPECT) detector[3] (Fig. 1(b)), which consists of a $\text{Gd}_3\text{Al}_2\text{Ga}_3\text{O}_{12}$ (GAGG) scintillator array and tungsten collimator, both with a 1 mm pitch. The HC-SPECT employs novel hourglass-shaped collimator, which allows more gamma rays to reach the scintillator, thereby improving image contrast. After imaging, the mice were dissected and the radioactivity of each organ was measured.

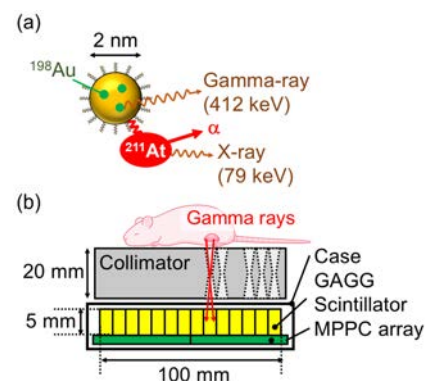


Fig. 1 (a) ^{211}At -[^{198}Au]AuNPs. (b) HC-SPECT.

RESULTS: Fig. 2(a) (left) and (right) show the ^{198}Au and ^{211}At images obtained 12 min after administration, respectively. Image of ^{211}At was obtained by detecting 79-keV X-rays from ^{211}At . For ^{198}Au imaging, maximum-likelihood expectation maximization (MLEM) was applied to improve image contrast. Fig. 2(b) and 2(c) show the ^{198}Au images obtained 3 h and 20 h after administration, respectively. These results indicate that the AuNPs injected into the tumor gradually spread throughout the body. Figure 2(d) shows the radioactivity (%ID/g) of each organ measured 4 days after administration. The tumor showed the highest accumulation, consistent with the imaging results.

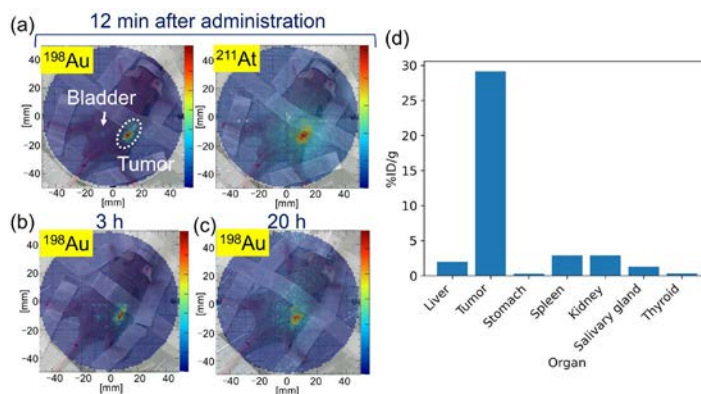


Fig. 2. Images of ^{211}At -[^{198}Au]AuNPs in mice. (a) Distribution of [^{198}Au]AuNPs (left) and ^{211}At (right) 12 min after injection. Distribution of [^{198}Au]AuNPs (b) 3 hours and (c) 20 hours after administration. (d) Radioactivity (^{198}Au) of each organ.

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Screening of enzymes for Asp isomerization in cells constituting the rat lens

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INTRODUCTION: Age-related covalent post-translational modifications play a critical role in protein homeostasis. One of the age-related modifications is isomerization of aspartate residues (Asp). If Asp isomerization is regarded as a new quality index for proteins, then suppression of this isomerization can be considered as maintenance of protein quality at the molecular level. Focusing on this concept, our aims to investigate the enzyme protein L-isoaspartyl (D-aspartyl) methyltransferase (PIMT), which suppresses Asp isomerization *in vivo*^[1]. PIMT recognizes isomerized Asp residues in proteins and converts them back to a succinimide intermediate, which is subsequently hydrolyzed to restore the normal Asp residue. The distribution of PIMT has been partially characterized and is known to be widespread in tissues such as the brain and muscle. However, intriguingly, PIMT has not been identified in the lens tissue, particularly in the rat lens, where its substrate— isomerized Asp— accumulates in large amounts. The reported absence of PIMT in the lens, despite its expected role as a cellular defense mechanism, appears inconsistent with the maintenance of biological homeostasis. To clarify this contradiction, the present study aims to screen for PIMT in the rat lens.

EXPERIMENTS: Rat lenses will be isolated, and PIMT will be screened using RT-PCR and western blotting. Rat kidney and rat lens tissues were homogenized, and total RNA was extracted from each sample using the NucleoSpin RNA Plus kit (Takara). The extracted total RNA was reverse-transcribed using the ReverTra Ace qPCR RT Master Mix (TOYOBO), and the resulting cDNA was analyzed by agarose gel electrophoresis to investigate PIMT gene expression. Both supernatants from homogenates were also subjected to SDS-PAGE. After electrophoresis, proteins were transferred onto a PVDF membrane, and Western blotting was performed using a PCMT1 rabbit polyclonal antibody as the primary antibody and an HRP-conjugated affinipure goat anti-rabbit IgG (H+L) as the secondary antibody.

RESULTS: As shown in Fig. 1 (a), the presence of cDNA was confirmed in both the kidney and the lens, suggesting that PIMT is present in both tissues. In contrast, as shown in Fig. 1 (b), Western blotting confirmed PIMT expression in the kidney, whereas its expression in the lens was not detected. These results indicate that, in the lens, PIMT is present at the DNA level but not expressed at the protein level. This discrepancy may reflect species-specific differences between humans and rats, such as lifespan and exposure to external stresses, suggesting the existence of regulatory mechanisms controlling gene expression. Further investigation of these mechanisms will be the subject of future studies.

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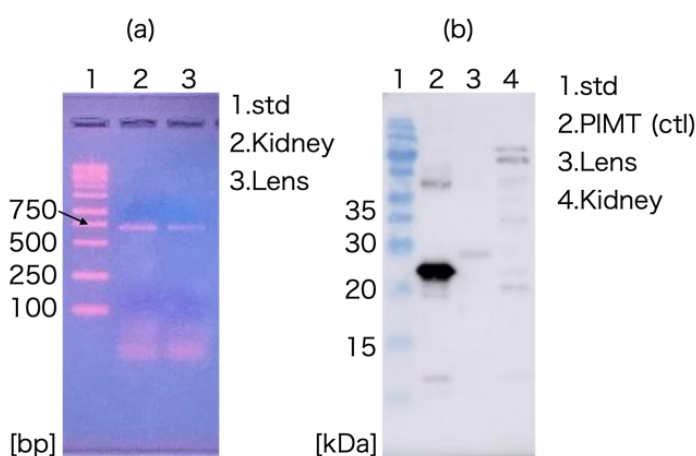


Fig. 1. DNA and Protein expression of PIMT
(a) 3% Agarose gel, (b) Western blotting

Domain-selective Solution Structure Analysis of ER-60 with Segment Deuteration

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INTRODUCTION: ER-60 is an enzyme responsible for oxidative protein folding. ER-60 possesses an **a-b-b'-a'** four-domain structure and regulates substrate folding depending on the redox state of its active site. However, its molecular mechanism remains poorly understood. Because domain configurations dynamically fluctuate in solution, structural analysis at the nanoscale is essential. This study aims to elucidate the domain-level structure and dynamics of ER-60 [1].

In this study, we perform analyses by combining solution scattering methods with molecular dynamics (MD) simulations. Since it is difficult to resolve domain motion using small-angle X-ray scattering (SAXS) alone, we employ domain-selective analysis using small-angle neutron scattering (SANS). Specifically, segmentally deuterated samples are prepared by connecting deuterated and hydrogenated domains via protein ligation [2], enabling selective observation of scattering from specific domains (DS-iCM-SANS) [3].

EXPERIMENTS: The 75% deuterated (*d*) and hydrogenated (*h*) domains were expressed in *E. coli* cultured in M9 medium containing 75% deuterium and LB medium without deuterium, respectively [4]. The deuteration level of *d*(**bb'**) domain was evaluated by MALDI-TOF mass spectrometry (MS). The domains (20 μ M each) were mixed in buffer containing 200 mM Tris-HCl (pH 7.4) and 150 mM NaCl. The ligation enzyme *OaAEP* (0.2 μ M) was then added, and the protein ligation reaction was performed at 20°C for 64 hours. The reaction products were analyzed by SDS-PAGE. The monodispersity of the samples was evaluated by analytical ultracentrifugation (AUC). SAXS measurements were performed at 25 °C using a NANOPIX (Rigaku, Tokyo, Japan). The sample-to-detector distances were 1330 mm and 300 mm, and the *q* range was 0.01-0.80 Å⁻¹.

RESULTS: MALDI-TOF MS spectra showed that *d*(**bb'**) exhibited a distinct mass shift from *h*(**bb'**) (Fig. 1). The deuteration ratio of *d*(**bb'**) was calculated to be 72.5%. This *d*(**bb'**) was used for a ligation reaction to prepare *h*(**a**)-*d*(**bb'**)-*h*(**a'**). The AUC analysis of wild-type and *h*(**a**)-*d*(**bb'**)-*h*(**a'**) ER-60 showed that the samples consisted predominantly of a single species. However, because minor dimeric and trimeric aggregates were present, size-exclusion chromatography-coupled SANS (SEC-SANS) was employed for the SANS measurement to eliminate these aggregates. SEC-SANS experiments have been performed, and data analysis is currently underway.

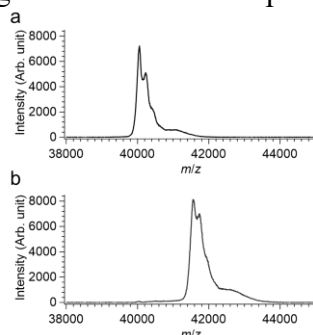


Fig. 1. MALDI-TOF mass spectra of (a) *h*(**bb'**) and (b) *d*(**bb'**). The deuteration ratio of *d*(**bb'**) was 72.5%.

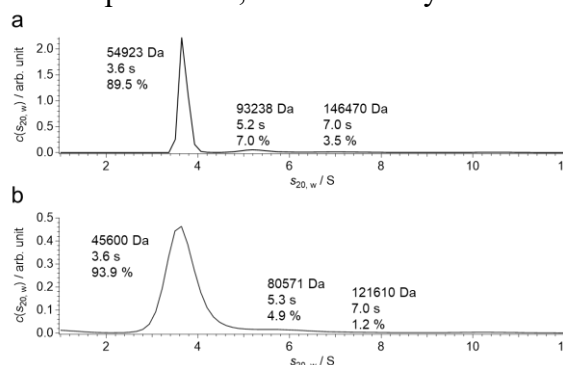


Fig. 2. The AUC profiles of (a) wild-type and (b) *h*(**a**)-*d*(**bb'**)-*h*(**a'**)

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Design, Synthesis, and Evaluation of Hydrophobic Bis(borono) Macrocyclic Polyamines for Boron Neutron Capture Therapy (BNCT)

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INTRODUCTION: Neutron capture therapy using boron-10 (¹⁰B) (BNCT) is one of powerful therapies for local tumor control in the treatment of brain tumor, melanoma, and related diseases. We previously designed and synthesized phenylboronic acid-pendant 9-, 12-, and 15-membered macrocyclic amines such as **10B-1a~3a** and the corresponding Zn²⁺ complexes **10B-1b~3b** (Fig. 1) [1,2], based on high intracellular uptake of cyclen (1,4,7,10-tetraazacyclododecane) derivatives in cancer cells. It was reported that the metal-free **10B-1a~3a** are introduced into cancer cells (A549 and HeLa S3 cells) more efficiently than their Zn²⁺ complexes **10B-1b~3b** with considerably high cancer/normal cells selectivity. Besides, it was found that 12- and 15-membered derivatives **10B-2a~3a** exhibit a higher BNCT effect than **10B-1a**, possibly because **10B-2a~3a** form the corresponding Zn²⁺ complexes **10B-2b & 3b**, which strongly interact with DNA in living cells [3], resulting in the efficient breakdown of DNA upon the neutron irradiation. Dimeric macrocyclic polyamines having one phenylboronic acid unit such as **4~9** and their corresponding Zn²⁺ complexes (Fig. 1), because it was well established that polymeric Zn²⁺ complexes would form more stable complexes with DNA double strand than monomeric Zn²⁺ complexes [4]. However, the intracellular uptake of **4~9** and their Zn²⁺ complexes were lower than monomeric compounds **1~3**, possibly due to their lower hydrophobicity than that of **1~3**.

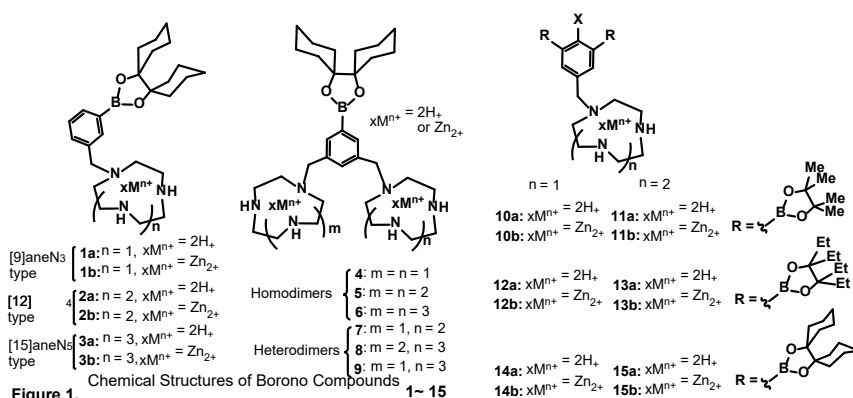
These data prompted us to design and synthesize monomeric polyamines equipped with two boronic ester units **10a~15a** and their corresponding Zn²⁺ complexes **10b~15b** shown in Fig. 1 for higher intracellular uptake and BNCT effect.

EXPERIMENT AND RESULTS:

In 2025, **10a~15a** and their corresponding Zn²⁺ complexes **10b~15b** (Fig. 1) were synthesized. The evaluation of intracellular uptake and BNCT effect of **10a~15a** and **10b~15b** has suggested that more hydrophobic **14a** has highest intracellular uptake and BNCT effect among these compounds [5].

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Characterization of DNA Damage and Mutagenesis Induced by Reactor-Generated Neutron Beams

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INTRODUCTION: Biological effects of radiation are primarily mediated by genomic DNA damage. Consequently, detailed analysis of such damage is fundamental to elucidating the molecular mechanisms underlying these effects. Our previous studies have demonstrated that high linear energy transfer (LET) radiation, such as heavy-ion beams, yields DNA damage profiles distinct from those induced by low-LET radiation, like γ -rays [1, 2]. As another form of high-LET radiation, neutron beams are expected to exhibit specific patterns of DNA damage. Our earlier collaborative research indicated that neutron radiation possesses a higher relative biological effectiveness (RBE) for DNA damage induction and cytotoxicity compared to γ -rays. The biological consequences of DNA damage generally follow two pathways: (i) the direct inhibition of DNA replication leading to cell death, and (ii) the induction of mutations that impair cellular functions. Given that heavy-ion radiation produces a mutation spectrum distinct from that of γ -rays [3], it is hypothesized that neutron radiation also induces characteristic mutations. In this study, we aimed to characterize the mutation spectrum induced by neutron irradiation using a plasmid-based assay.

EXPERIMENTS: pUC19 plasmid DNA was dissolved in TE buffer (10 mM Tris-HCl, pH 7.5; 1 mM EDTA). Samples were prepared with or without 10% (w/v) BSH (¹⁰B-sodium mercaptododecaborate), a boron neutron capture therapy (BNCT) agent. Neutron irradiation was performed at the Kyoto University Reactor (KUR). The total physical dose, including both neutrons and concomitant γ -rays, was set up to 5 Gy. Following irradiation, the DNA was transformed into competent *Escherichia coli* cells to assess mutagenesis. Mutations within the lacZ α gene were screened via the α -complementation assay (blue/white screening), and the mutation spectrum was analyzed by DNA sequencing of the resulting white colonies, according to previously established protocols [3].

RESULTS: Initial screening via the α -complementation assay identified a small number of white colonies (data not shown). However, subsequent DNA sequencing failed to confirm any specific mutations within the lacZ region of these colonies. This result is likely attributable to the limited radiation dose applied in this study. Although neutron radiation exhibits high RBE, the maximum dose was restricted to 5 Gy due to constraints on reactor machine time, which may have been insufficient to induce mutation frequencies above the detection threshold. Notably, even in samples containing BSH—where the ¹⁰B (n, α) ⁷Li reaction was expected to enhance local energy deposition—no confirmed mutations were observed. These findings suggest that within the 0–5 Gy dose range, neutron-induced biological effects might be predominantly mediated by the direct inhibition of DNA replication rather than by the accumulation of point mutations or small deletions detectable by this assay. Further studies utilizing higher doses or more sensitive detection systems are required to further characterize the mutagenic potential of neutron beams.

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Study of Imaging System for Ultra-High Dose Rate Irradiation Field

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INTRODUCTION: In ultra-high dose rate (UHDR) irradiation field, the FLASH effect has been confirmed, which minimizes damage to normal tissue while maintaining unaltered tumor control [1, 2]. The precise mechanism underlying the FLASH effect is unclear. To verify this, it is important to establish a method for evaluating the irradiation field. In this study, we developed the system that measures the two-dimensional distribution of the UHDR irradiation field in real-time by visible light emitted from an acrylic plate using a CMOS camera.

EXPERIMENTS: The schematic layout of the imaging system is shown in Fig. 1. Since luminescence can be observed even in air by electron, the acrylic plate was made 4 cm thick to stop the electron, and blocked light from all directions except that toward the camera. The irradiation tests were performed at the electron linear accelerator in KURNS (KURNS-LINAC). The electron beam energy was set to 8 MeV, and the pulse width was set to 5 μ s. Measurements were taken while varying the distance between the beam port and the acrylic plate from 0 to 600 mm at 100 mm intervals. The distributions were normalized using the results from Radio-chromic films, a common measurement method for UHDR irradiation field, irradiated under the same conditions.

RESULTS: Fig. 2 shows the lateral distribution as a function of distance from the beam port. It was found that the normalized factors are varied by 30% depending on the distance, but the peak intensities roughly corresponded to those of the films. However, the distribution was broader than the films; this is due to the scattering of electrons in the 4 cm thick acrylic. In conclusion, we investigated a method for evaluation of two-dimensional dose distributions using CMOS camera in UHDR irradiation field. Although we were unable to obtain distributions equivalent to the conventional methods, we were able to get the peak position in real-time, suggesting that this method could be used for monitoring the peak positions.

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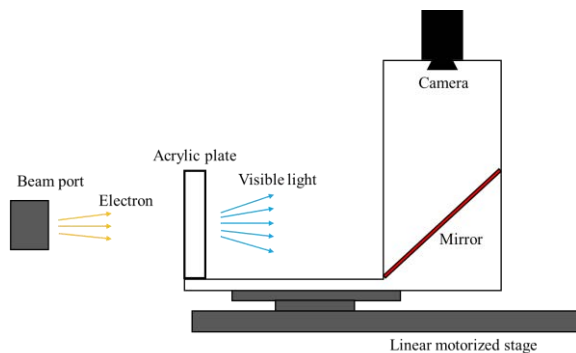


Fig. 1. Schematic layout of imaging system using acrylic plate and CMOS camera.

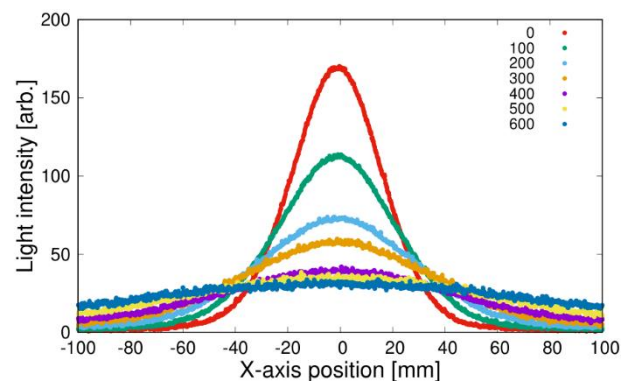


Fig. 2. Lateral distributions measured by CMOS camera while varying the distance from beam port (0~600 mm).

¹⁰B-enriched nitroimidazole derivative with high water solubility as a functional drug for hypoxia-targeting boron-neutron capture therapy

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INTRODUCTION:

Boron neutron capture therapy (BNCT) is a promising radiotherapy based on the nuclear reaction between boron-10 (¹⁰B) and thermal neutrons. The therapeutic efficacy of BNCT depends on the development of boron-containing agents that selectively accumulate in cancer cells. In particular, the development of BNCT agents effective against hypoxic cells is highly desirable, since tumor hypoxia is closely associated with resistance to conventional therapies.

In our previous study, we developed BPA-NI, a nitroimidazole (NI)-containing derivative of ¹⁰B-enriched *p*-boronophenylalanine (BPA), as a hypoxia-targeting BNCT agent, and confirmed its selective accumulation in hypoxic cells together with its cytotoxic effect upon thermal neutron irradiation.¹ On the other hand, improvement of its water solubility remained an important challenge. To address this issue, in the present study we designed a new BNCT agent using a nucleobase capable of forming a complex with BPA, with the aim of improving the aqueous solubility of the BPA-NI-based system while maintaining hypoxia-targeting ability. Since nitroimidazoles are well known as exogenous hypoxia markers owing to their characteristic bioreductive behavior in hypoxic cells, an NI-containing BPA–nucleobase complex (BPA-NI-U) was prepared and evaluated. Herein, we describe its preparation, selective accumulation in hypoxic cells, and cytotoxic effect under thermal neutron irradiation.

EXPERIMENTS:

The BPA-NI-U (0 or 500 μM) was administered to the SAS cells and then the cells were incubated for 20 h. After incubation and wash, the cells were irradiated (neutron, 1 MW) for 45 min at KUR. After incubation, WST 8 was added to the cells, and the cell viability assay was performed using Microplate Reader.

RESULTS:

SAS cells were selected as the target cell line because of their suitability for BNCT studies, and the cytotoxic effect of BPA-NI-U under thermal neutron irradiation was evaluated. After incubation with BPA-NI-U for 20 h under either hypoxic or aerobic conditions, the cells were exposed to neutron irradiation. BPA-NI-U showed little cytotoxicity in aerobic SAS cells, whereas markedly enhanced cytotoxicity was observed in hypoxic SAS cells. By contrast, BPA alone induced cytotoxicity in both aerobic and hypoxic cells without hypoxia selectivity. Taken together, these results indicate that BPA-NI-U preferentially accumulated in hypoxic cells, thereby exerting selective cytotoxic effects upon thermal neutron irradiation.

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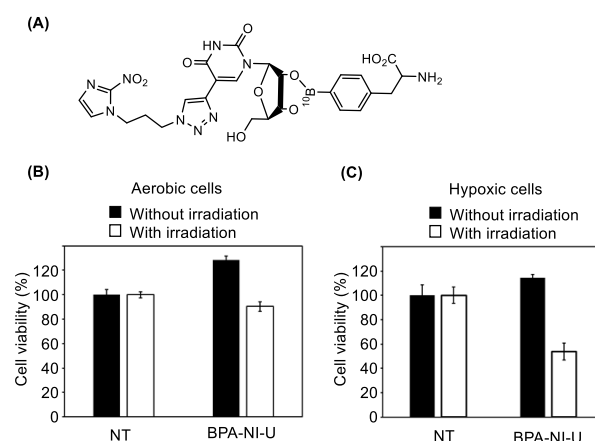


Fig. 1. (A) Chemical structure of BPA-NI-U. ((B) Cytotoxicity of BPA-NI-U against SAS cells upon thermal neutron irradiation under aerobic (B) or hypoxic conditions (C).

Tritium Labeling of Eggshell Membrane, a Natural Composite Biomaterial, via the ${}^6\text{Li}(n,\alpha){}^3\text{H}$ Reaction at KUR Pn-2 for Biodistribution Studies

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INTRODUCTION: Inflammatory bowel disease (IBD) causes intestinal fibrosis and stricture through chronic inflammation, severely impairing patients' quality of life. Intestinal fibrosis is characterized by excessive ECM accumulation and tissue stiffening, with few fundamental therapies available once it progresses. Notably, ECM also governs stem cell niches and epithelial regeneration, suggesting that fibrosis may be controllable as a reversible remodeling process. Eggshell membrane (ESM) is a traditional wound-healing material composed mainly of fibrous proteins. Using ${}^3\text{H}$ -labeled ESM prepared via the ${}^6\text{Li}(n,\alpha){}^3\text{H}$ reaction—a labeling method originally developed at JRR-4 [1] and applied in the present study at KUR—we have investigated its in vivo behavior and biological functions. In a previous study, we reported that oral ESM alleviates lung fibrosis demonstrated that ESM and its major component, lysozyme (LYZ), act as anti-fibrotic agents against pulmonary fibrosis via decorin induction and TGF- β suppression [2]. However, the in vivo kinetics of ESM and its mechanisms of action on the intestinal environment remain largely unclear. In this study, we quantified the biodistribution of ${}^3\text{H}$ -labeled ESM after oral administration and evaluated its effects on intestinal fibrosis and the gut environment, aiming to establish a foundation for novel preventive and therapeutic strategies [3].

EXPERIMENTS: ESM powder was sealed in a quartz tube together with lithium carbonate, and ${}^3\text{H}$ was introduced by neutron irradiation via the ${}^6\text{Li}(n,\alpha){}^3\text{H}$ reaction at KUR Pn-2. The ${}^3\text{H}$ -labeled ESM was mixed with jelly and administered orally to mice, and blood, major organs, and feces were collected at multiple time points ranging from 0.5 to 72 hours after administration. The amount of ${}^3\text{H}$ was measured by liquid scintillation counting, and the tissue distribution was calculated. Statistical analyses were performed using ANOVA followed by appropriate multiple comparison tests.

RESULTS: Following oral administration of ${}^3\text{H}$ -labeled ESM, the radioactivity in blood reached its peak at 5 hours post-administration and gradually declined thereafter. In the gastrointestinal tract, an early peak was observed in the duodenum and small intestine at 2 hours post-administration, followed by widespread distribution to the large intestine, liver, kidney, lung, and skin. The radioactivity in rectal feces reflected the excretion of undigested components and indicated the presence of ESM-derived components retained within the intestinal lumen; however, these components were excreted relatively rapidly.

Conclusion: The KUR Pn-2 irradiation facility proved highly suitable for tritium labeling of protein samples, as it maintains low sample temperatures during neutron irradiation and thereby preserves the integrity of biological materials. However, given that KUR ceased operation in April 2026, we strongly hope that comparable irradiation capabilities will be implemented in the newly planned successor reactor to enable the continuation of such studies.

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Design, Synthesis, and Evaluation of Multiborono Polyamines for Boron Neutron Capture Therapy (BNCT)

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INTRODUCTION: Neutron capture therapy using boron-10 (¹⁰B) (BNCT) is one of powerful therapies for local tumor control in the treatment of brain tumor, melanoma, and related diseases. We previously designed and synthesized phenylboronic acid-pendant 9-, 12-, and 15-membered macrocyclic amines such as ¹⁰B-**1a**~**3a** and the corresponding Zn²⁺ complexes ¹⁰B-**1b**~**3b** (Fig. 1) [1,2], based on high intracellular uptake of cyclen (1,4,7,10-tetraazacyclododecane) derivatives in cancer cells (A549 cells). It was reported that the metal-free ¹⁰B-**1a**~**3a** are introduced into cancer cells (A549 and HeLa S3 cells) more efficiently than their Zn²⁺ complexes ¹⁰B-**1b**~**3b** with considerably high cancer/normal cells selectivity. Besides, it was found that 12- and 15-membered derivatives ¹⁰B-**2a**~**3a** exhibit a higher BNCT effect than ¹⁰B-**1a**, possibly because ¹⁰B-**2a**~**3a** form the corresponding Zn²⁺ complexes ¹⁰B-**2b** & **3b**, which strongly interact with DNA in living cells [3], resulting in the efficient breakdown of DNA upon the neutron irradiation.

Next, we conducted the design and synthesis of monomeric polyamines equipped with two boronic ester units **4a**~**9a** and their corresponding Zn²⁺ complexes **4b**~**9b** (Fig. 1) for higher intracellular uptake of ¹⁰B atoms and more potent BNCT effect.

EXPERIMENTS AND RESULTS:

The synthesis of **4a**~**9a** and their corresponding Zn²⁺ complexes **4b**~**9b** was carried out and their intracellular uptake and BNCT effect were found to be highest among these compounds [4]. In addition, the effect of addition of Zn²⁺ from outside on BNCT effect of these ¹⁰B carriers was evaluated, because it was expected that the Zn²⁺ complexes **4b**~**9b** were supposed to recognize the deprotonate thymidine in DNA in order to place ¹⁰B units at the close position to DNA for more efficient BNCT effect. The results of BNCT experiments have suggested that the effect of Zn²⁺-pyrithione complex on BNCT effect of **4a**~**9a** is not so significant, suggesting that the concentrations of Zn²⁺ in A549 cells are high enough to form their Zn²⁺ complexes in cells.

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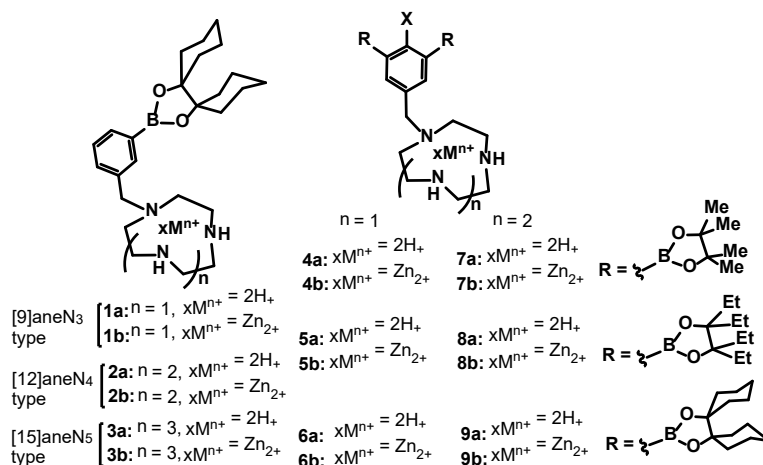


Figure 1. Chemical Structures of Borono Compounds 1 ~ 9

Construction of novel Boron-containing silica nanoparticles and BNCT experiments

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INTRODUCTION: Two type of boron compounds which are boron phenyl alanine (BPA) and borocaptate (BSH) have been mainly used for BNCT. But, these compounds have problems such as BSH having low uptake into cancer cell and BPA having short retention in tumor. Thus, novel boron compounds which can overcome these problems need to be developed for future BNCT.

We used a new type of mesoporous silica-based nanoparticle (MSN) that is modified with polyethylene glycol (PEG) and tetramethylammonium chloride (TMAC) to positively charged on surface. This nanoparticle has a large surface area where a large quantity of boron compounds which include BPA or BSH can be loaded. In recently, we developed novel MSN which has small-size and highly dispersible (sdMSN) into water. The MSN has the ability to flow into bloodstream for long time and result in accumulation into tumor at significantly high concentration. The MSN was also retained for long time in the tumor after intravenously injected to animal body. In this study, we evaluated the biodistribution of new MSN which has positively charged surface that has under 25 nm of diameter.

EXPERIMENTS: We used MSN which was synthesized by sol-gel method using tetraethoxysilane (TEOS) and modified with PEG and TMAC to positively charge on surface. It has the size of less than 25 nm. This MSN was also labeled with Rodamine B dye to trace where it accumulates in mouse body after injection. The MSN was characterized by using TEM, DLS, nitrogen adsorption-desorption analysis and zeta potential. For biodistribution analysis, MSN was intravenously injected to CT26 mouse colon cancer- mouse xenografts at 5 mg/mouse or 2 mg/mouse, and we dissected tumor and organs which include liver, lung and kidney at 24 hours after injection. Tumor and organs were made into thin sections and the fluorescence of MSN was detected with a confocal microscope.

RESULTS: The analysis showed that the size of MSN was less than 25 nm diameter and had homogenous shapes examined by DLS and TEM microscopy. And then, the surface of MSN was positively charged due to modification with PEG and TMAC. The zeta potential of MSN was 38.29 ± 0.77 mV in water. MSN accumulation in the CT26-transplanted mouse was investigated with a fluorescent microscope and confocal microscope after making thin sections. As shown in a figure, MSN was able to be accumulated in the tumor at higher concentration compared with Liver 24 hours after injection. In this study, we clarified that both aging and hydrothermal treatment steps were very important to make sdMSN which can accumulate in the tumor at high concentration.

We are currently attaching boron to MSN. These results suggest that MSN may be an effective boron carrier for BNCT. This MSN may be able to become a new boron reagent for BNCT beyond BPA and BSH if boron compounds are grafted to it.

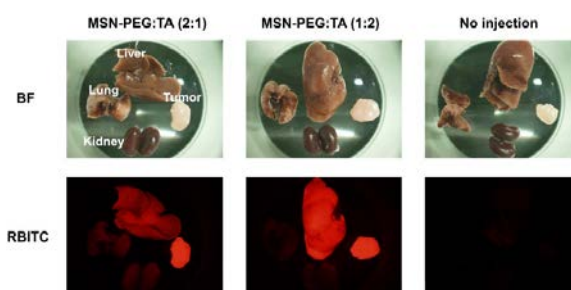


Fig. Biodistribution of MSN in CT26-bearing mouse model.

Synthesis of PEPT1-targeted boron containing dipeptids for pancreatic cancer therapy

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INTRODUCTION: Peptide transporter 1 (PEPT1) has been noticed because it is expressed on various types of cancer cells. Especially, it has been reported that PEPT1 was highly expressed pancreatic cancer PDX model.

We used some dipeptide which contained ¹⁰B that has higher solubility, longer retention ability in the tumor than BPA. These dipeptides are actively taken up into cancer cells by PEPT1 and LAT1 (weakly).

We investigated BNCT efficacy of these ¹⁰B-dipeptides and the abscopal effect on mice which were simultaneously transplanted tumors on right leg and left shoulder when dipeptides are used.

EXPERIMENTS: We used two types of dipeptides including BPA-BPA and His-BPA. Dipeptides were intravenously injected into CT26-transplanted BALB/c mice 2 hours before neutron irradiation. Our aim was to investigate whether the BNCT efficacy of dipeptides can be improved compared with BPA by neutron irradiation at KUR. The thermal neutron was irradiated for 6 minutes at 5MW. After neutron irradiation, tumor volume and body weight measured for 6 weeks after irradiation (up to 42 days after irradiation) to evaluate the BNCT efficacy of dipeptides. For abscopal effect evaluation, BALB/C mouse was simultaneously transplanted CT26 cancer cells on both right leg and left shoulder to make tumor. All mice were injected with dipeptides 1.5 hours before irradiation, and right legs were only irradiated with neutron for 6 minutes at 5MW. For 2-6 weeks after irradiation, tumors volume on both right leg and left shoulder were measured to evaluate abscopal effect induction.

RESULTS:

In previous experiments for tumor accumulation of boron, it has been shown that dipeptides are effective boron carriers. We then investigated BNCT efficacy of these boron compounds. Dipeptides were intravenously injected into CT26-transplanted mice 2 hours before neutron irradiation. These mice were held to 12 mouse holders and placed in front of KUR, and neutron was irradiated. Tumor on dipeptides-injected mice was eradicated, and the tumor regrowth was not observed up to 27 days after BNCT, whereas regrowth was observed on BPA-injected mice. This result indicates that dipeptides have the potential to strongly cure cancer by BNCT.

We have further investigated abscopal effect induction on mice which were simultaneously transplanted CT26 cancer cells on both right leg and left shoulder that were injected with dipeptides before neutron irradiation. The tumor on right leg (irradiated tumor) was eradicated 2 weeks after BNCT, and the distant tumor unirradiated tumor on left shoulder was significantly shrunk between 2-3 weeks post-BNCT compared with mice that were administered BPA.

This work has published to ACS Pharmacology & Translational Science as entitled "L-boronophenylalanine (L-BPA) dipeptide prodrugs enhance boron delivery to tumors, facilitate tumor eradication and induce tumor vaccine effect in mice following neutron irradiation.

We have further been investigating the mechanism of abscopal effect induction by BNCT with dipeptides about the activation of aggressiveness and improved invasiveness of immune cells including CD8+ T-cell, NK cells and macrophages.

Verification of the invitro/in vivo dynamics of Indocyanine green boron-containing compounds

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INTRODUCTION: Based on experience with over 300 cases in specific clinical studies, we have developed an ICG-boron polymer compound by utilizing the characteristic accumulation of indocyanine green (ICG) in brain and spinal cord tumors, as confirmed by the intraoperative ICG fluorescence imaging protocol during surgery.¹ This compound is intended for application in boron neutron capture therapy (BNCT).

EXPERIMENTS: Cultured cells are co-cultured with ICG-boron polymer compounds, which are then taken up by the cells; the cells are subsequently irradiated with neutrons, and the cytotoxic effect is evaluated. Furthermore, since the protocol for this experiment has already been established, by performing irradiation in the heavy water facility for approximately 2 hours per experiment (during 1 MW operation), the cytotoxic effects of multiple types of compounds under various conditions were evaluated. Similarly, using a mouse subcutaneous tumor model, ICG-boron polymer compounds were administered to be taken up by tumor cells, followed by neutron irradiation to evaluate the cytotoxic effect. Balb/C nu/nu mice are planned for use. The experimental protocol has already been established, and evaluations were conducted by irradiating the samples for up to approximately one hour per experiment in the heavy water facility (during 5 MW operation).

RESULTS:

We compared the cytotoxic effects of two ICG compounds, ICG Compound 1 and ICG Compound 2, against L-BPA—a drug currently used in clinical practice—on malignant glioma cells (U87). Irradiation times were set at three intervals: 10 minutes, 30 minutes, and 50 minutes. Furthermore, we confirmed that delaying the excretion of the compounds using the excretion inhibitor V enhanced their cytotoxic effects. Both ICG Compound 1 and ICG Compound 2 demonstrated significantly greater cytotoxic effects than L-BPA. Furthermore, significant cytotoxic effects were observed in the groups treated with ICG Compound 1 and ICG Compound 2 plus L-BPA, as well as in the groups treated with ICG Compound 1 and ICG Compound 2 plus L-BPA and the excretion inhibitor V, across all three irradiation durations (10, 30, and 50 minutes). Next, a subcutaneous tumor model was established in the right hind leg of BALB-c nu/nu 4-week-old mice. Compared to the control group, significant tumor-suppressive effects were observed in the L-BPA monotherapy group and in the groups treated with ICG Compounds 1 and 2. Next, although no significant difference was observed between ICG Compounds 1 and 2 and the L-BPA monotherapy group, they exhibited nearly equivalent tumor-suppressive effects. Compared to the L-BPA monotherapy group, the ICG Compounds 1 and 2 plus L-BPA group demonstrated a significant tumor-suppressive effect.

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Antitumor effect of boron neutron capture therapy for lung tumor in cervical cancer mouse model.

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INTRODUCTION: In Japan, approximately 10,000 women develop cervical cancer and 2,900 women die from the disease annually. The incidence and mortality rates of cervical cancer in Japan are on the rise. Squamous cell carcinoma (SCC) is the most common histological type at 80%, and adenocarcinoma (Adeno) accounts for about 20%. Lung metastases from cervical cancer are often resistant to chemotherapy, making them difficult to treat. Thus, new treatment modalities for cervical cancer are needed. In this study, we evaluated the efficacy and safety of boron neutron capture therapy (BNCT) in a mouse model of cervical cancer lung metastasis.

EXPERIMENTS: BPA (L-isomer) was supplied by Stella Chemifa (Osaka, Japan) and prepared as a fructose complex. Female nude mice (BALB/c Slc-nu/nu), aged 4–6 weeks, were purchased from SLC (Japan). A lung metastasis model was established by injecting cervical cancer cell lines (HeLa and SiHa) into the right lungs, and tumors were allowed to develop over 4–6 weeks.

The mice were divided into two groups: a hot control group (neutron irradiation only) and a BNCT group (intra-peritoneal BPA administration followed by neutron irradiation). In the BNCT group, BPA (250 mg/kg) was administered intraperitoneally 2.5 hours prior to neutron irradiation. After irradiation, body weight was monitored, and survival was evaluated.

RESULTS:

Cervical cancer lung metastasis models were established using HeLa and SiHa cell lines (HeLa: BNCT, n = 6; hot control, n = 5; SiHa: BNCT, n = 6; hot control, n = 6). At 3 weeks after irradiation, all mice in the HeLa model remained alive, whereas in the SiHa model, one mouse in each group had died. They are currently under observation.

Figure 1 shows the changes in body weight in the hot control and BNCT groups in the cervical cancer lung metastasis models (HeLa and SiHa). No significant changes in body weight were observed in either group.

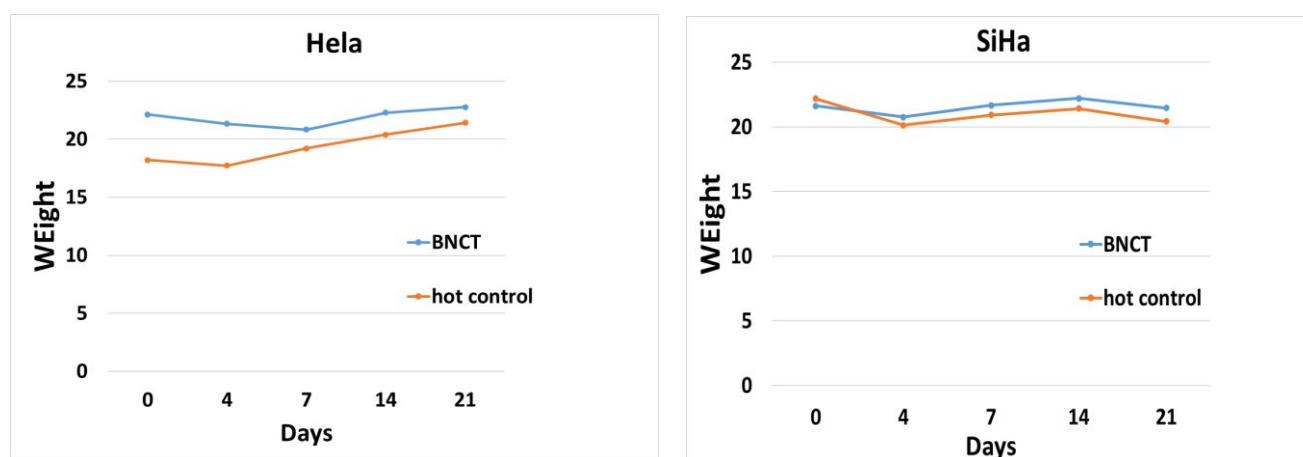


Fig. 1. Body weight in the hot control and BNCT groups in the cervical cancer lung metastasis models (HeLa and SiHa)

Characterization of Early and Late effect of pelvic Boron Neutron Capture Therapy (BNCT) in a mouse model

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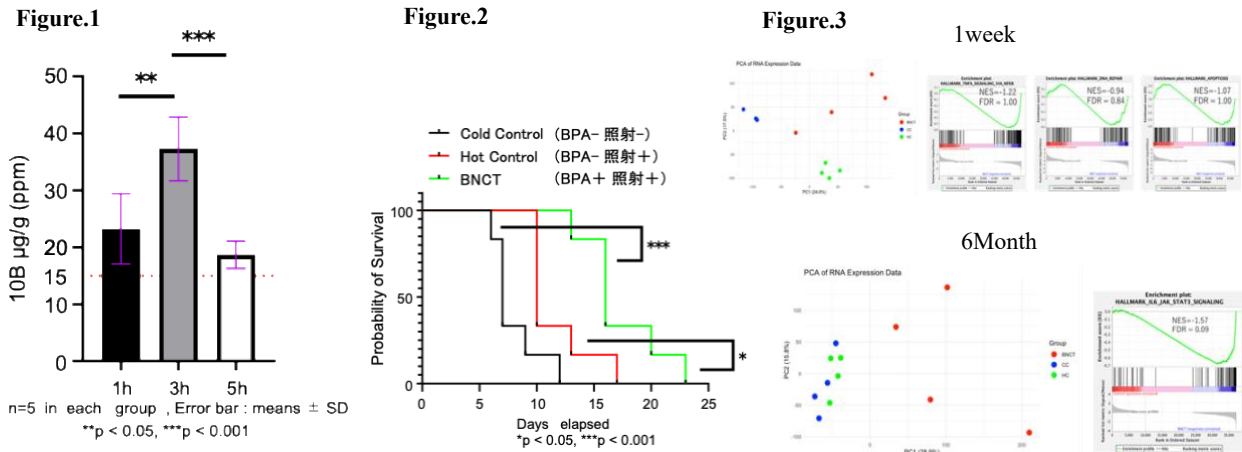
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INTRODUCTION: Previously, we reported the anti-tumor effect of Boron Neutron Capture Therapy (BNCT) for a mouse model of pelvic recurrence of Colorectal cancer (CRC). On the other hand, we could not fully evaluate side effects in terms of immune response or the later complications, because we examined the effectiveness of BNCT in the nude mouse model only a month after treatment. This study investigates early and late effect of pelvic BNCT.

EXPERIMENTS: We used Boronophenylalanine (BPA) as a boron compound. Also, we used seven-week-old female BALB/cCrSlc mouse having physiological environment of immunity. Firstly, we established a mouse model of pelvic recurrence of CRC using Colon-26-Luc cells concentrated to $1.0 \times 10^5/100\mu\text{L}$ in 0.1ml of PBS. The boron concentrations in Colon-26-Luc tumors at 1h, 3h, 5h after 500mg/kg BPA administration intraperitoneally (**Figure.1**). According to this result, we decided to inject BPA intraperitoneally at 3h before irradiation. In BNCT study, animals were divided into three groups; the cold control (no treatment, no neutron irradiation), hot control (neutron irradiation only), and BNCT (intraperitoneal BPA administration and neutron irradiation) groups. Also, we conducted an irradiation experiment using BALB/cCrSlc mice without tumor implantation to investigate the long-term effects on pelvic organs with HE staining and RNA-sequence.

RESULTS: BNCT significantly prolonged survival (median: Cold 7 days, Hot 10, BNCT 16) (**Figure.2**). All toxicity-study mice survived 189 days without severe adverse events except transient weight loss. Histological examination with HE revealed mucosal degeneration in the cecum 7 days after BNCT; however, recovery was observed over time. RNA sequencing indicated a tendency toward local activation of the immune system or a DNA damage response in the early phase after BNCT, whereas findings in the late phase were suggestive of chronic inflammation (**Figure.3**).

Conclusion: BPA administration followed by BNCT was not associated with any lethal adverse events, indicating the feasibility and safety of pelvic irradiation.



Ongoing study: We will continue this study and the results will be published in the future.

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The effect of boron neutron capture therapy (BNCT) to liver metastasis of colorectal cancer

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INTRODUCTION: Management of liver metastases in colorectal cancer is a clinically important issue. However, only 10–15% of patients are eligible for surgery based on the size and number of metastatic lesions. Furthermore, for patients with multifocal, unresectable, or bilateral liver metastases who do not respond to chemotherapy, palliative care is the only option available. In this context, the development and evaluation of new treatment strategies are justified. In this study, we investigated the effectiveness of boron neutron capture therapy (BNCT) to liver metastasis of colorectal cancer using the mouse model.

EXPERIMENTS: We used Boronophenylalanine (BPA) as a boron compound. Also, we used seven-week-old female BALB/cCrSlc mouse having physiological environment of immunity. Firstly, we established a mouse model of liver metastasis of CRC using DLD-1-Luc cells concentrated to $1.0 \times 10^6/100\mu\text{L}$ in 0.1ml of PBS. The boron concentrations in DLD-1-Luc tumors or surrounding organs at 2h, 4h, 6h after 500mg/kg BPA administration intraperitoneally. (**Figure.1**) According to this result, we considered the effects on the pancreas and decided to inject BPA intraperitoneally at 6h before irradiation. In BNCT study, animals were divided into three groups; the cold control (no treatment, no neutron irradiation), hot control (neutron irradiation only), and BNCT (intraperitoneal BPA administration and neutron irradiation) groups.

RESULTS: Luminescence intensity monitored via the In Vivo Imaging System (IVIS) demonstrated that values in the BNCT group were significantly lower than those in the cold control group on day 10 (**Figure.2**), with a non-significant downward trend also observed relative to the hot control group. Upon dissection on day 16, the tumor weight in the BNCT group was significantly reduced compared to the cold control group (**Figure.3**) and showed a decreasing tendency relative to the hot control group, although this did not reach statistical significance. Similarly, while the tumor volume in the BNCT group tended to be lower than in the other two groups, no statistically significant difference was observed (**Figure.3**).

Histopathological analysis showed a reduction in Ki67-positive cells in the BNCT group, suggesting inhibited tumor cell proliferation (**Figure.4**). In contrast, acinar cell necrosis and plasma cell infiltration were observed in the pancreatic parenchyma, indicating potential treatment-induced toxicity in surrounding normal tissue.

Figure1

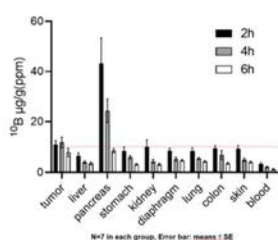


Figure.2

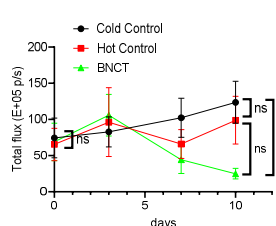


Figure.3

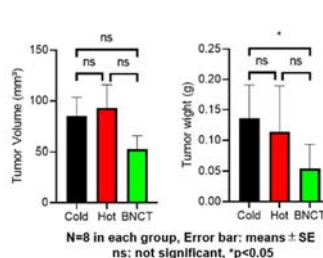
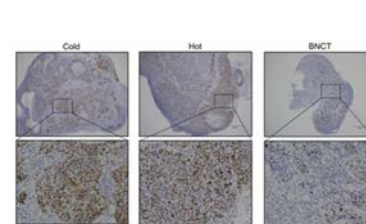


Figure.4



Ongoing study:

Future studies will aim to further elucidate the anti-tumor mechanisms of BNCT and systematically evaluate its impact on adjacent organs. This study will be continued, and the results will be reported in the future.

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Optimization of CSF-Administered BNCT Using High-Concentration ¹⁰BPA Ionic Liquid in a Rat Glioma Model

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INTRODUCTION: Boron neutron capture therapy (BNCT) is a tumor-selective radiotherapy based on nuclear reactions between thermal neutrons and boron (¹⁰B) accumulated in tumor cells. Its therapeutic efficacy critically depends on efficient and selective boron delivery. However, in brain tumors, drug delivery is limited by the blood–brain barrier (BBB), making it difficult to achieve sufficient and homogeneous boron distribution within the tumor. To overcome this limitation, we have previously developed a cerebrospinal fluid (CSF)-based drug delivery approach via cisterna magna administration [1]. In addition, the limited solubility of conventional boronophenylalanine (¹⁰BPA) restricts achievable boron concentrations. In this study, we employed a high-concentration meglumine–¹⁰BPA ionic liquid formulation (Meg-¹⁰BPA IL) and investigated the relationship between administration parameters and BNCT efficacy under reactor irradiation conditions.

EXPERIMENTS: C6 glioma cells were stereotactically implanted into the brains of male Wistar rats (8 weeks old, body weight 180–200 g). The high-concentration boron formulation, Meg-¹⁰BPA IL, was developed by Dr. Makoto Shirakawa at Okayama University and has been patented in Japan [2]. Meg-¹⁰BPA IL was continuously infused into the CSF via the cisterna magna at different boron concentrations and infusion rates (100 or 200 µL/h). Neutron irradiation was performed at the Kyoto University Research Reactor (KUR), a 5 MW light water-moderated reactor, under thermal neutron beam conditions for 20 min, corresponding to an average neutron fluence of 3.7×10^{12} neutrons/cm². During irradiation, the body (excluding the head) was shielded using ⁶LiF materials to reduce unnecessary neutron exposure. Tumor progression was evaluated using T2-weighted MRI before and after BNCT, and tumor volume ratios (post-/pre-treatment) were calculated.

RESULTS: As shown in Figure 1, all CSF-administered BNCT groups showed suppression of tumor growth compared with untreated and neutron-only control groups.

Tumor volume ratios were:

4200 ppm-B, 100 µL/h: 0.94

4200 ppm-B, 200 µL/h: 0.45

8400 ppm-B, 200 µL/h: 1.83

Control groups: >10

The strongest tumor suppression was observed in the 4200 ppm-B, 200 µL/h group. Notably, increasing boron concentration alone did not improve therapeutic efficacy, whereas a higher infusion rate resulted in greater tumor control. These results indicate that infusion rate would be a key determinant of therapeutic outcome in CSF-administered BNCT under KUR irradiation conditions.

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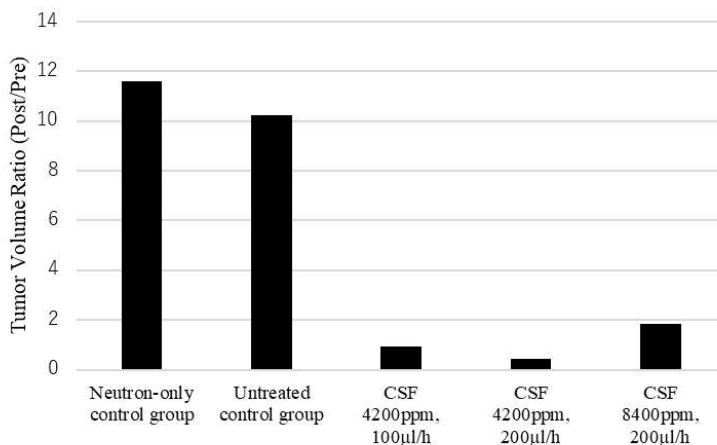


Figure 1. Tumor Volume Ratio (Post/Pre) by Treatment Group

Abscopal Effect Induced by BNCT with Boron-10 Carbide Nanoparticles

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We have been developing nanoparticle (NP) based ^{10}B drugs for BNCT [1]. However, radiotherapy is not systemic but local cancer treatment to limit its efficacy against metastatic cancers. While recent studies suggest that combining BNCT with an immunomodulator (PD-L1 siRNA or anti-PD-1 antibody ($\alpha\text{PD-1}$) [2]) induces abscopal effects to distant tumors, such responses have been rarely observed with single-modality BNCT via intravenous administration [3], or even the other radiotherapy alone. Herein, we report on abscopal effect by BNCT with boron carbide NPs with 50 nm size ($^{10}\text{B}_4\text{C}(50)$).

First of all, we established the mechanochemical process to selectively synthesize $^{10}\text{B}_4\text{C}(50)$ from $^{10}\text{B}(\text{OH})_3$, magnesium and graphite [4]. The $^{10}\text{B}_4\text{C}(50)$ was subsequently coated with hydrophilic polymer, poly(glycerol) (PG), to give $^{10}\text{B}_4\text{C}(50)\text{-PG}$ which is highly dispersed in a physiological environment. The resulting boron-10 drug was administered intravenously to the mice with the CT26 tumors at the leg and back. Only the leg tumor was irradiated by neutron as shown in Figure 1a. As a result, the growth of the primary tumor at the leg was suppressed more efficiently as compared with the control groups of PBS injection, boron drug injection and neutron irradiation as shown in Figure 1b. Meanwhile, the distant tumor at the back was also suppressed in the growth as compared with the other groups as shown in Figure 1b. Notably, the abscopal effect was also observed in 4T1, an immunologically “cold” tumor, as shown in Figure 1d and 1e, although “cold” tumors are typically characterized by an immunosuppressive microenvironment.

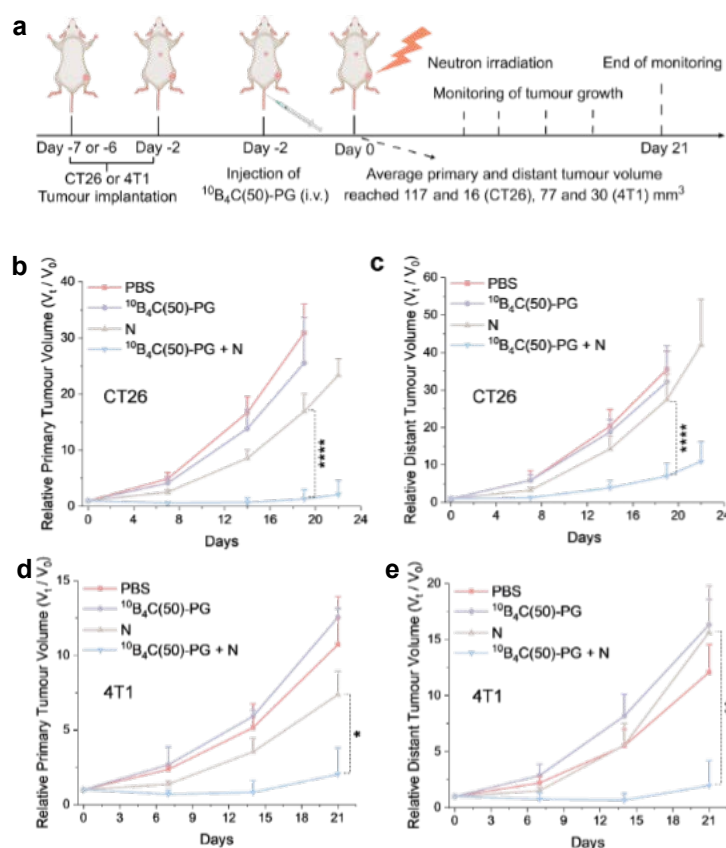


Figure 1. a) Schematic of dual CT26 tumour treatment process with dosage of 5.1 mg [^{10}B]/kg (mouse) and neutron irradiation only at right thigh in *in vivo* BNCT ($n = 6$). b) Relative primary tumour volume at right thigh. c) Relative distant tumour volume at left thigh. d, e) Relative 4T1 primary and distant tumour volume at right thigh and flank, respectively.

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Bifunctional Dodecaborate to Development of Boronated Antibody

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INTRODUCTION: BNCT for cancer is based on the nuclear reaction of ^{10}B with thermal/epithermal neutrons to yield high linear energy transfer α particles (^4He) and recoiling ^7Li nuclei in tumor cells [1]. For a boron delivery agent to be successful in BNCT, the following criteria must be met: i) high tumor targeting selectivity; ii) low systemic toxicity; iii) high water solubility. Mercapto-undecahydro-closo- dodecaborate ($[\text{B}_{12}\text{H}_{11}\text{SH}]^{2-}$, BSH), a class of water-soluble boron cluster compounds with low toxicity, is clinically used as boron carrier for the treatment of brain tumors, although, tumor selectivity and cell membrane permeability of BSH is slightly low. In the course of our developing studies on new boron carrier for BNCT, we have designed and synthesized thiododecaborate ($[\text{B}_{12}\text{H}_{11}\text{S}]^{2-}$) unit-containing tumor seeking compounds such as amino acids, peptides and antibodies [2-4]. Furthermore, we reported the bifunctional type thiododecaborate containing compounds which boron cluster is linked two kinds of organic moiety through S+ sulfanyl groups ($\text{B}_{12}\text{H}_{11}\text{-S+(-R1)-R2}$) [5].

Recently, theranostic drug delivery is strong criteria for effective cancer treatment. In BNCT, development of theranostic type boron carrier is highly noted, because visualization of the boron distribution and determination of tumor/normal ratio by non or minimally invasive examination are very important for the planning of BNCT.

To develop the theranostic type boron carrier for BNCT, we present the design and synthesis of novel bifunctional boron cluster containing compounds which linked fluorescent dye Cy5.5 and alkyl linker (Cy5.5-BSH-Pentyl-COOSu) to conjugate with tumor seeking antibody such as Cetuximab (Erbix, anti EGFR IgG). Furthermore, we report the conjugation of novel bifunctional boron compounds with antibody, and the biological evaluation of boronated antibody as boron carrier for BNCT.

EXPERIMENTS: The solution of Cy5.5-BSH-Pentyl-COOSu in DMSO was added to the solution of Cetuximab in 0.1M NaHPO_4 buffer (pH8.6) and incubated for 24 at 0°C . The mixture was purified with a PD-10 column.

RESULTS: As shown in Fig. 1, Dodecaborated-Cetuximab (Cy5.5-DB-Cetuximab) was delivered a boron atom effectively to EGFR high expressed tumor cell, and enhanced cellular uptake of the DB-Cetuximab by EGF induced effective macropinocytosis induction.

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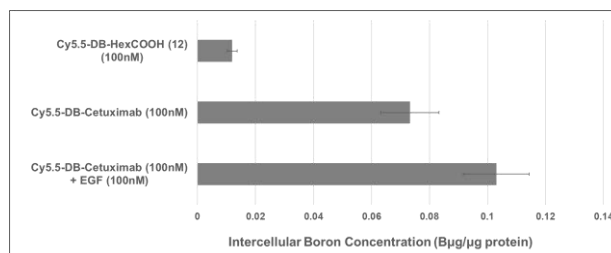


Fig.1. ELISA assay for detecting boron concentration in A431 cell treated with Cy5.5-DB-COOH (compound 12 type, 100nM) or Cy5.5-DB-Cetuximab (100 nM) with or without EGF (100 nM) in cell culture medium containing 10% FBS for 24 h at 37°C . The data are expressed as the mean ($\pm$ SD) of three experiments

Development of Boron Cluster-Loaded Nanoparticles for BNCT

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INTRODUCTION: Boron neutron capture therapy (BNCT) is a less-invasive nuclear therapeutic approach that highly selective to induce cancer apoptosis, without any cytotoxic effects in neighboring cells [1]. BPA and BSH as current clinically approved boron compounds have several limitations related to selectivity and effectivity [2]. To address the issues, a novel self-forming nanoparticle, which is consisted of biodegradable polymers, namely, “Lactosome” with a hydrophobic boron cluster have been developed [3]. In the previous study, the BNCT effects were successfully obtained after exposing neutron irradiation to the AsPC-1 cells (human pancreatic cancer cells) in vitro and in vivo [4]. In this study, we compared in vitro BNCT effect on a boron cluster loaded with different type of Lactosome “type 1” and “type 2” particles.

EXPERIMENTAL PLANS: For in vitro study, AsPC-1 cells are subjected to irradiation at reactor power 1 MW for 10 min or 40 min after 2 hr incubation with boron cluster-loaded Lactosome particles. After the neutron irradiation, the cells are cultured in 12-well plates and incubated under 5% CO₂ at 37 °C for 14 days. Then, colony formation assay is performed after staining.

RESULTS:

In vitro, uptake of B-10 derived from either type 1 and type 2 particles reproducibly was observed but the in vitro neutron-irradiation study with cancer cells was not performed, because it took unexpectedly longer time to prepare the particles.

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Establishment of innovative BNCT treatment method for intractable bladder cancer

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INTRODUCTION: Bladder cancer treatment remains a challenge to every urologist. The current first-line treatment for non-muscle invasive bladder cancer is transurethral resection of bladder tumors followed by intravesical Mycobacterium Bovis Bacillus Calmette-Guérin (BCG) immunotherapy. In case of BCG failure, radical cystectomy is the standard of care in high - risk patients. However, many of them are unfit or they refuse to undergo such an intervention; therefore, other treatment options are required. The usefulness of BNCT to urothelial cancer remains unknown. Here we aimed to investigate whether BNCT suppresses in a previously es-tablished mouse model of orthotopic bladder cancer.

EXPERIMENTS: We established BCG-resistant bladder cancer cell line and examined their molecular and functional characteristics. The response of resistant cells to BNCT was tested in vitro. In vivo experiments were carried out using an orthotopic bladder cancer mouse model to study boron biodistribution after intravesical or systemic administration of boronophenylalanine (BPA). Boron-neutron capture events at the tissue level were detected with CR-39 autoradiography. The therapeutic effect and safety of BNCT were evaluated by bladder weight measurement, serum biochemistry tests and organ histology.

RESULTS: BCG-resistant bladder cancer cells showed clear molecular differences from parental cells, but they remained highly sensitive to BNCT in vitro. In animal experiments, intravesical administration of BPA led to selective accumulation of boron in tumor tissue with very limited systemic exposure, and the optimal treatment time was found to be 1 hour. CR-39 analysis showed that neutron capture reactions occurred mainly in tumor areas. BNCT produced a clear reduction in tumor size compared with systemic delivery (Figure 1). Detailed safety evaluations showed no obvious treatment-related toxicity.

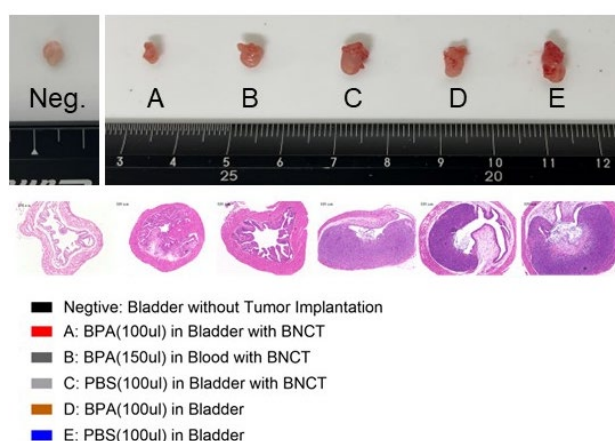


Fig.1

The bladder weight assay of 14 days after Neutron irradiation.

Neutron irradiation for 12 minutes after Boron added for 1 hours

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The Basic Study Aimed at performing the BNCT for Canine Malignant Melanoma

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INTRODUCTION: Malignant melanoma is one of the most major cancers in dogs. Melanomas that occur in the canine oral cavity are highly invasive and difficult to surgically resect. In addition, they are known to be refractory tumors that are difficult to treat with conventional radiation therapy. Therefore, new treatment strategies are needed.

BNCT is a therapeutic method that selectively destroys the tumor while leaving normal tissues almost unharmed by utilizing the nuclear reaction with neutron and boron, which tends to accumulate in the cancer cells. In human malignant melanoma, LAT1, an amino acid transporter of boron compound, is shown to be overexpressed in many malignant tumor cells. In this study, the survival after the neutron irradiation using the canine malignant melanoma cell lines. In addition, the effects of the boron neutron capture reaction (BNCR) on cells were analyzed by examining cell survival rates and double-strand break damage.

EXPERIMENTS: To evaluate the effects of neutrons on canine malignant melanoma cells, canine mandibular lymph node metastatic oral melanoma cells (LMeC), in which LAT1 expression has been confirmed, were used for this study. The effects on cells were evaluated by measuring DNA double-strand breaks via immunofluorescence staining and assessing survival rates via colony formation assays in cells irradiated with neutrons after BPA administration (w/BPA) and cells irradiated with neutrons without BPA administration (w/o BPA).

RESULTS: Fig. 1 shows the results of a comparison of DNA damage between gamma-ray irradiation and BPA treatment. In LMeC cells, BPA-treated cells exhibited greater DNA damage than gamma-ray-irradiated cells. Fig. 2 shows the results of cell viability. The survival rate of cells treated with BPA was significantly lower than that of cells not treated with BPA. Furthermore, at equivalent absorbed doses, w/BPA cells exhibited a higher mortality rate than gamma-irradiated cells. This demonstrates the efficacy of BPA-BNCT against LMeC cells and its superiority over gamma irradiation.

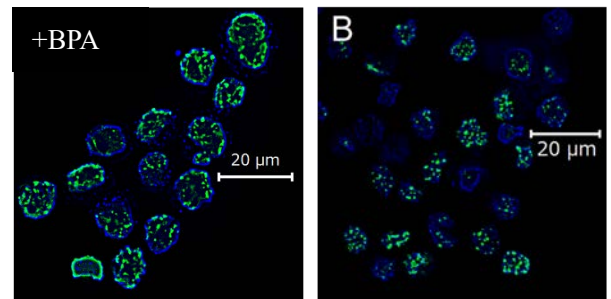


Fig.1 DNA double-strand breaks (showed by green points)

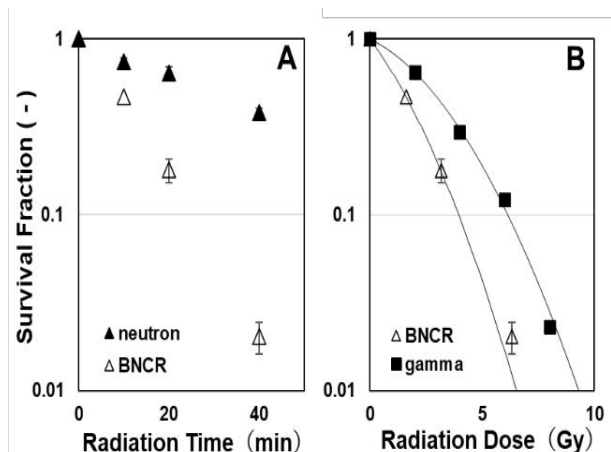


Fig.2 Survival Fraction

Real-time neutron monitoring for BNCT using ^{10}B -integrated HOIP sensors

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INTRODUCTION: Boron neutron capture therapy (BNCT) requires accurate, real-time monitoring of neutron flux under high-flux conditions ($\sim 10^9 \text{ s}^{-1} \text{ cm}^{-2}$). Conventional methods such as ^{197}Au activation lack real-time capability, while scintillator-based detectors suffer from γ -ray interference and limited self-diagnosis. Hybrid organic–inorganic perovskite (HOIP) thin-film devices, originally developed for photovoltaic applications, offer high charge mobility, self-biased operation, and flexible fabrication, making them promising candidates for neutron detection when combined with neutron conversion materials such as ^{10}B . In this study, we develop and evaluate a ^{10}B -integrated HOIP sensor for real-time neutron monitoring in BNCT environments.

EXPERIMENTS: Boron neutron capture therapy (BNCT) requires accurate, real-time monitoring of neutron flux under high-flux conditions ($\sim 10^9 \text{ s}^{-1} \text{ cm}^{-2}$). Conventional methods such as ^{197}Au activation lack real-time capability, while scintillator-based detectors suffer from γ -ray interference and limited self-diagnosis. Hybrid organic–inorganic perovskite (HOIP) thin-film devices, originally developed for photovoltaic applications, offer high charge mobility, self-biased operation, and flexible fabrication, making them promising candidates for neutron detection when combined with neutron conversion materials such as ^{10}B . In this study, we develop and evaluate a ^{10}B -integrated HOIP sensor for real-time neutron monitoring in BNCT environments.

RESULTS: The HOIP sensors demonstrated clear neutron-induced signals in BNCT-relevant high-flux environments. As shown in Fig. 1, the induced current of WC devices increased linearly with neutron flux up to $\sim 10^9 \text{ s}^{-1} \text{ cm}^{-2}$, reaching nA-level signals, whereas WoC devices showed only pA-level responses, confirming that the signal is dominated by neutron conversion events. The γ -ray contribution was estimated using WoC measurements and found to be extremely small ($\sim 0.012\%$), well below clinical tolerance limits. The sensors exhibited stable operation with 1 s temporal resolution, enabling real-time monitoring. Furthermore, no significant degradation was observed up to neutron fluences of $\sim 10^{13} \text{ cm}^{-2}$, indicating sufficient radiation tolerance for BNCT applications. These results demonstrate that ^{10}B -integrated HOIP sensors satisfy key BNCT monitoring requirements, including high sensitivity, linearity, low γ -noise, and real-time capability.

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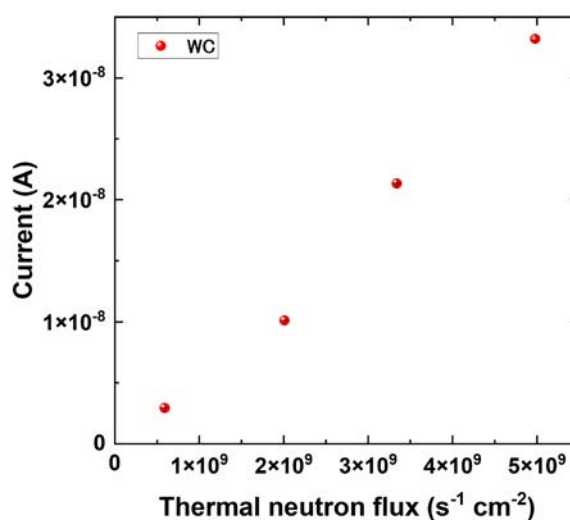


Fig. 1. Neutron flux dependence of radiation-induced current in HOIP sensors with ^{10}B conversion layer

Preliminary Experiment of Neutron Capture Therapy using Intra-peritoneal Administration of ^{10}B -plex encapsulated Water-in-Oil-in-Water Emulsion for Peritoneal Disseminated Tumor Model

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INTRODUCTION: We have been continuously developing boron-containing water-in-oil-in-water (WOW) emulsions for application in the multidisciplinary treatment of primary cancer using boron neutron capture therapy (BNCT). We have experienced the abdominal disseminations of cancer cells; so called cancerous peritonitis in pancreatic cancer, colon cancer, gastric cancer, ovarian cancer, etc. The pathology of cancerous peritonitis is extremely difficult to treat, and if BNCT can improve the condition and bridge the gap to multidisciplinary treatment, the effectiveness of BNCT can be further enhanced. In this study, we performed the preliminary experiments of BNCT with intraperitoneal administration of ^{10}B -plex encapsulated WOW emulsion for peritoneal disseminated colon cancer model, and its survival periods of treated and controlled groups following thermal neutron irradiation was evaluated.

EXPERIMENTS: The internal aqueous phase consisted of the following: 1.2 mL of ^{10}BSH solution (350 mg/mL), 0.12 mL of Lipofectamine 2000, 0.09 mL of sodium hyaluronate (0.25 mL; 10 mg/mL), and 0.09 mL of protamine (0.125 mL; 20 mg/mL). The oil phase consisted of 2 mL of Lipiodol, with surfactants added to each phase. Using our originally developed mixing device, a WOW emulsion was prepared. We prepared mouse colon cancer Colon 26 (5×10^5) model by intra-peritoneally injection. Following the intra-peritoneal injection of 0.2 mL of the emulsion, thermal neutrons at a dose of 3×10^{12} n/cm² were irradiated at Institute for Integrated Radiation and Nuclear Science, Kyoto University. Mouse survival days were measured post-irradiation to evaluate the tumor growth inhibition effect.

RESULTS: The WOW emulsion containing the ^{10}B -plex complex demonstrated longest survival days after thermal neutron irradiation compared with non-irradiated group. As the irradiation was performed two hours after intra-peritoneal injection in this study.

We can expect better results of BNCT, if we will be able to prepare the cancer specific ^{10}B complexes entrapping into WOW emulsion.

Table1. Survival days after BNCT to intra-peritoneal disseminated tumor model.

Day after BNCT	Survival rate after BNCT (%)				
	3	7	11	14	20
BNCT groups					
WOW- ^{10}B oroplex	100	100	100	100	33.3
Neutron only	100	100	100	83	0
Non NCT groups					
WOW- ^{10}B oroplex	100	100	80	40	20
Neutron only	100	100	80	0	0

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Evaluation of the therapeutic efficacy of a novel boron carrier, BBCIP, for BNCT.

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INTRODUCTION: The efficacy of boron neutron capture therapy (BNCT) relies on the selective accumulation of boron within tumor cells [1,2]. While p-boronophenylalanine (BPA) is clinically standard, its dependence on LAT1 transporters necessitates new carriers to overcome tumor heterogeneity. In this study, we evaluated BBCIP, a novel boron carrier targeting the sodium-dependent multivitamin transporter (SMVT) [3]. BBCIP was administered via convection-enhanced delivery (CED) to bypass the blood-brain barrier and maximize local concentration [4,5]. We performed a comparative analysis using SMVT-high (CRL1666) and LAT1-dominant/SMVT-low (F98) brain tumor models to assess how transporter profiles dictate the therapeutic synergy of BBCIP and BPA.

EXPERIMENTS: Pharmacokinetic analysis revealed that BBCIP administered via CED exhibited stable and measurable intratumoral boron kinetics, maintaining therapeutic concentrations in both models. In survival studies, the therapeutic impact varied significantly according to the dominant transporter. In the LAT1-dominant F98 model, BBCIP monotherapy yielded only modest survival prolongation (median survival time [MST]: 29 days vs. 23 days in controls). However, the co-administration of BPA with BBCIP showed a positive trend in extending survival, suggesting a complementary uptake mechanism. Conversely, in the SMVT-high CRL1666 model, BBCIP monotherapy demonstrated robust efficacy, significantly prolonging survival (MST: 30 days vs. 15.5 days in controls). Notably, the addition of BPA in this model provided negligible further benefit, indicating that SMVT-mediated boron delivery alone was sufficient to achieve a therapeutic threshold.

RESULTS: These results demonstrate that the efficacy of BBCIP-based BNCT is strictly dictated by the tumor's transporter landscape. Our findings suggest that in SMVT-enriched tumors, BBCIP can serve as a highly effective primary boron source. In contrast, for tumors with low SMVT but high LAT1 expression, BBCIP may function as a supplemental agent, where combination therapy with BPA addresses the "intratumoral boron gap" left by single-agent protocols. The disparate outcomes between the F98 and CRL1666 models underscore a critical paradigm shift: the necessity of "Precision BNCT." Rather than a universal carrier approach, treatment success relies on tailoring the boron cocktail to the specific molecular and biological signatures of the tumor. This study advocates for the pre-therapeutic screening of transporter expressions to optimize the selection and combination of boron agents, ultimately enhancing the precision and potency of neutron capture therapy.

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Investigation of Potential Adverse Effects of Boron Neutron Capture Therapy on Host Immunity

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INTRODUCTION: Boron Neutron Capture Therapy (BNCT) is a form of radiation therapy for cancer that utilizes neutron capture reactions, wherein boron-10 atoms capture thermal neutrons, subsequently undergoing nuclear fission to produce alpha particles and lithium nuclei [1]. Recent studies have revealed that X-ray irradiation does not suppress anti-tumor immune function but rather activates it. To investigate whether BNCT similarly enhances anti-tumor immunity, we have explored the relationship between BNCT and tumor immunomodulation. The purpose of this study is twofold: (1) to determine whether BNCT induces host immune-activating effects, and (2) to evaluate the composition of infiltrating immune cells within tumors post-BNCT, thereby assessing potential adverse impacts on tumor immunity.

EXPERIMENTS: Tumor cells (SCC7) were subcutaneously implanted into the hind limbs of C3H/He mice. At 12 days post-implantation, neutron irradiation alone or BNCT treatment was administered to the tumor tissue. The BNCT group was injected subcutaneously with 500 mg/kg boronophenylalanine (BPA). One hour post-BPA administration, neutron irradiation was delivered to the tumor site using a 5 MW reactor for 15 minutes. At 12 days post-irradiation, tumor tissues were excised. Single-cell suspensions were prepared using the BD Tumor Dissociation Kit (BD Biosciences, #130-096-730) per manufacturer protocol. Cells were stained with fluorochrome-conjugated antibodies against CD45 (leukocyte common antigen), CD3 (T-cells), CD4 (helper T-cells), CD8 (cytotoxic T-cells), CD69, Eomes, CD62L, Gr1, CD25, Foxp3, Ki67, CD44, CD11b, PD-1, and Fixable stain 620. For intracellular staining, the intracellular fixation and permeabilization buffer set (ThermoFischer) was used according to the manufacturer protocol. All antibodies without Foxp3 were purchased from Biolegend. The antibody for Foxp3 was purchased from Invitrogen. Immune cells in the tumor tissues were measured by Cytex Northern Lights (Cytex) and the data were processed using SpectroFlo software.

RESULTS: After BNCT both CD4+ and CD8+ T cells were not reduced compared to the neutron alone control. Memory subsets of CD8 T cells in the BNCT group were slightly higher compared to the control. In the BNCT group compared to the control group, a higher proportion of cells positive for activation markers represented by CD69 was observed across all T cell subsets. The proportion of regulatory T cells did not differ between the two groups in this experiment.

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Exploration of Methods to Reduce BNCT Adverse Events by Modifying the Normal Tissue Distribution of BPA

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INTRODUCTION: Boron Neutron Capture Therapy (BNCT) is a targeted radiotherapy that utilizes the nuclear capture reaction of thermal neutrons by boron-10, producing high-linear energy transfer (LET) alpha particles and lithium-7 nuclei [1]. The primary boron delivery agent, boronophenylalanine (BPA), is an amino acid analog transported into cells via L-type amino acid transporters LAT1 and LAT2. While cancer cells exhibit elevated LAT1 expression due to metabolic reprogramming, normal tissues also express LAT2, enabling BPA uptake in healthy cells. This unintended accumulation contributes to adverse effects post-BNCT, despite the therapy's theoretical tumor specificity. This study hypothesizes that selective inhibition of LAT1 (tumor-predominant) and LAT2 (normal tissue-expressed) could improve the tumor-to-normal tissue boron concentration ratio. By pre-administering transporter-specific inhibitors prior to BPA infusion, we aim to suppress normal tissue boron uptake while preserving tumor targeting, thereby enhancing BNCT's therapeutic index.

EXPERIMENTS: Tumor Model and Irradiation Protocol: The mice-derived tumor cells (SCC7) were subcutaneously injected into mice lower limb. At a tumor volume of 150–200 mm³, BPA (500 mg/kg) was subcutaneously administered. After BPA administration, a compound inhibiting LAT1 was intraperitoneally injected to the mice. Six and twelve hours after BPA administration respectively, blood was collected by cardiac puncture under anesthesia, followed by euthanasia via cervical dislocation. Thereafter, the liver, stomach, small intestine, large intestine, tongue, skin, brain, tumor, and liver were harvested. To assess BPA biodistribution modulation, anticholinergic (atropine, 2 mg/kg) or cholinergic (pilocarpine, 5 mg/kg) agents were administered intraperitoneally 30 minutes prior to BPA. Tissues were digested in a 3:1 mixture of perchloric acid and hydrogen peroxide, followed by boron quantification via inductively coupled plasma atomic emission spectroscopy (ICP-AES).

RESULTS: In the combination treatment with the LAT1 inhibitor, contrary to our expectations, tumor BPA efflux was also inhibited; however, under the timing of administration used in this experiment, BPA uptake into the tumor likewise appeared to be suppressed. As expected, BPA uptake and washout in normal tissues were not inhibited. In the brain, the T/N ratio was actually reduced when the LAT1 inhibitor was used. This finding suggests that, because LAT1 is expressed at the blood–brain barrier, BPA washout from the brain is also inhibited.

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Construction of a Dose–Response Model for Boron Neutron Capture Therapy: Derivation of Optimal Parameters through Cell and Animal Experiments

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INTRODUCTION: Boron Neutron Capture Therapy (BNCT) is a targeted radiotherapy that utilizes the nuclear capture reaction of thermal neutrons by boron-10, producing high-linear energy transfer (LET) alpha particles and lithium-7 nuclei [1]. Thus, BNCT is a type of particle radiotherapy especially focusing on cellular-level radiotherapy. As in the process of dose calculation in radiotherapy, if the distributions of the boron agent and the neutron beam are known, the dose can be calculated, and the antitumor effect as well as the impact on normal tissues can be estimated from the dose–response curves of both tumor and normal tissues. We have already established a dose–response model for boron neutron capture therapy based on parameters that contribute to the distribution of the boron agent and on the findings accumulated to date [2]. However, our established model includes multiple parameters, and the biological validity of these parameters had not yet been sufficiently examined. The purpose of this study is to derive the appropriate parameters for the established dose–response model of boron neutron capture therapy through cell-based and animal experiments, and to verify its usefulness.

EXPERIMENTS: The aim of this study was to perform BNCT on tumor tissues in C3H and Balb/c mice subcutaneously implanted with tumor cells, and to examine the relationship between boron concentration and antitumor efficacy by applying BNCT at different boron concentrations. Although the irradiation experiments could not be conducted as scheduled because of the experimenter's unexpected condition, we carried out alpha autoradiography using CR-39, which had been planned as one of the experiments to evaluate the response of normal tissues. Alpha autoradiography was conducted using CR-39 solid-state nuclear track detectors. Prior to sample mounting, two crossing fiducial marks were inscribed on the detector surface to provide stable reference points for image registration before and after etching. Tissue sections containing 10B were placed in direct contact with the detector and irradiated with thermal neutrons to induce the $^{10}\text{B}(\text{n},\alpha)^7\text{Li}$ reaction. The emitted charged particles produced latent tracks in the CR-39 detector, which were visualized as etch pits after chemical etching under alkaline conditions. Microscopic images of the specimen were acquired before or independently from etching, using bright-field microscopy and, when necessary, fluorescence microscopy for structural identification such as nuclear localization. After etching, the detector surface was examined under high magnification, and the etched pits were analyzed according to their spatial distribution and morphology. In particular, pit circularity was used as an index to improve the positional accuracy of the estimated boron localization. The pre-etch specimen image and the post-etch pit image were then superimposed using the fiducial marks on the detector, and the resulting composite images were used to determine the microdistribution of boron in the tissue or cells.

RESULTS: We are currently analyzing the alpha autoradiography results of the gastrointestinal tract and tumors following administration of BPA at 500 mg/kg. In the future, we plan to incorporate into the model not only these alpha autoradiography findings but also information on the sizes of the cytoplasm and nuclei in the tissues.

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Cancer Immune Activation and Its Effect on BNCT Using Antagonists of Transcription Factor and Amino Acid Metabolic Enzyme

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INTRODUCTION: In the tumor microenvironment (TME), cancer cells, cancer-associated macrophages (TAM), and myeloid-derived suppressor cells (MDSC) utilize various checkpoints and enzymes to evade the immune system. STAT3 and IDO-1 play a central role as "brakes" in this process. STAT3 is the "command center" of cancer immunosuppression and induces TAM to M2 type. IDO-1 depletes tryptophan, weakening the induction of cytotoxic T cells and enhancing differentiation into regulatory T cells. TAM and MDSC existing in the TME express Dectin-1, a β -1,3-glucan (GC) receptor, in their cell membranes. Therefore, in this study, we utilize to solubilize poorly water-soluble STAT3 and IDO-1 inhibitors with GC and administered them via drug delivery system (DDS) to Dectin-1 expressing cells to induce hot cancer formation, and then investigated the combination effect with low-dose BPA-BNCT.

EXPERIMENTS: Shikonin (SKN) was used as a STAT3 inhibitor, and LY-3381916 (LY) was used as an IDO-1 inhibitor. After mixing *Aureobasidium pullulans* TS-1 strain-produced GC in DMSO, two types of complex nanogels (SKN/GC, LY/GC) were prepared by dialysis. SCC-VII (mouse squamous cell carcinoma) cells were transplanted subcutaneously into the right thigh to create tumor-bearing C3H mice. BPA (125 mg/kg) was administered, followed by BNCT with neutron irradiation (5MW, 14 mins, 5.6 neutrons $\cdot$ cm⁻²) at KUR. SKN/GC, LY/GC, or a mixed cocktail was administered via tail vein 2, 6, 9, and 13 days after irradiation. Thereafter, tumor size and body weight were measured twice a week.

RESULTS: Nanogel aqueous solutions were obtained with SKN at a concentration of 290 μ M (solubilization rate 13%) and LY at 400 μ M (solubilization rate 32%). The particle size and zeta potential of each nanogel were measured using a Zetasizer-Nano ZS. The SKN/GC nanogel had a particle size of 21.3 nm and a zeta potential of -28.5 mV, while the LY/GC nanogel showed 11.3 nm and -20.1 mV.

As shown in Fig.1, the relative increase in tumor volume with neutron irradiation (Figure 1) showed that the group administered both SKN and LY nanogel (G4) had significantly suppressed tumor growth compared to the other monotherapy groups (G2,3) and the no-drug group (G1).

In summary, it has demonstrated that simultaneously inhibiting proteins involved in transcription factor and amino acid metabolism on cancer immunosuppression is effective in controlling the TME to activate cancer immunity followed the enhanced BPA-BNCT.

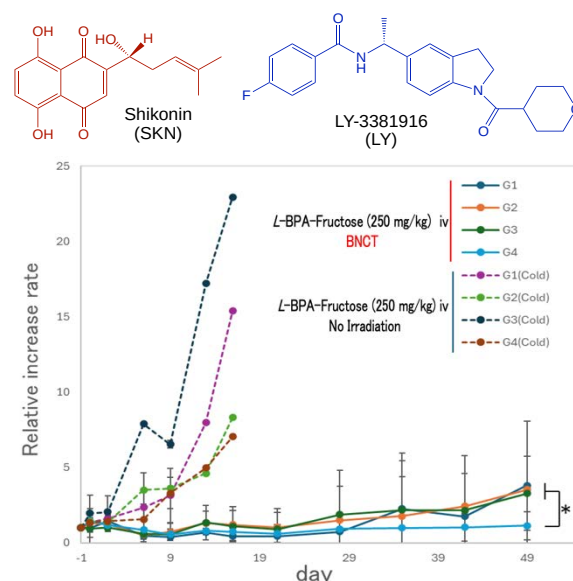


Fig. 1. The combination effect of SKN and/or LY administration on low-dose BPA-BNCT. G1: No Drug, G2: 50 μ M SKN, G3: 50 μ M LY, G4: 50 μ M SKN & 50 μ M LY.

Mechanistic Study of GdNP-PG Nanoparticles-Mediated Gadolinium Neutron Capture Therapy Combined with Anti-PD-1 Immunotherapy

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Gadolinium neutron capture therapy (GdNCT) is a promising binary radiotherapy approach for cancer treatment. It leverages the nuclear reaction between thermal neutrons and ¹⁵⁷Gd, which has an exceptionally high neutron capture cross-section of 254,000 barns. This reaction generates high linear energy transfer (LET) Auger electrons along with low-LET γ -photons, both of which contribute to the destruction of cancer cells.^[1] Immune checkpoint blockade (ICB) immunotherapy is a revolutionary cancer treatment strategy that restores the body's antitumor immune response. It functions through the administration of monoclonal antibodies that block inhibitory receptors on T cells or tumor cells, such as CTLA-4, PD-1, or their ligands (e.g., PD-L1). These checkpoints are typically exploited by cancers to evade immune surveillance. Blocking this interaction with specific monoclonal antibodies restores T-cell-mediated antitumor immunity, leading to durable clinical responses across various tumor types.

Previously, we reported that the combination of GdNP-PG nanoparticle-mediated GdNCT and anti-PD-1 (α PD-1)-mediated ICB immunotherapy effectively suppressed the growth of both primary and distant tumors, showing a significant abscopal effect. To elucidate the mechanisms underlying the therapeutic efficacy of the combination therapy, mice bearing primary and distant CT26 tumors were sacrificed 21 days after neutron irradiation, and the tumors were harvested for immunohistochemical assays. As shown in Figure 1, the populations of CD4⁺ and CD8⁺ T cells in the GdNCT + α PD-1 group were much larger than those in the other groups. The activation and infiltration of CD4⁺ and CD8⁺ T cells in the tumor microenvironment boosted the therapeutic efficacy against primary tumors and induced significant abscopal effects that suppressed the growth of distant tumors. These results suggest that the combination of GdNCT and ICB immunotherapy effectively modulates the tumor immune microenvironment, turning “cold” tumors into “hot” ones.^[2]

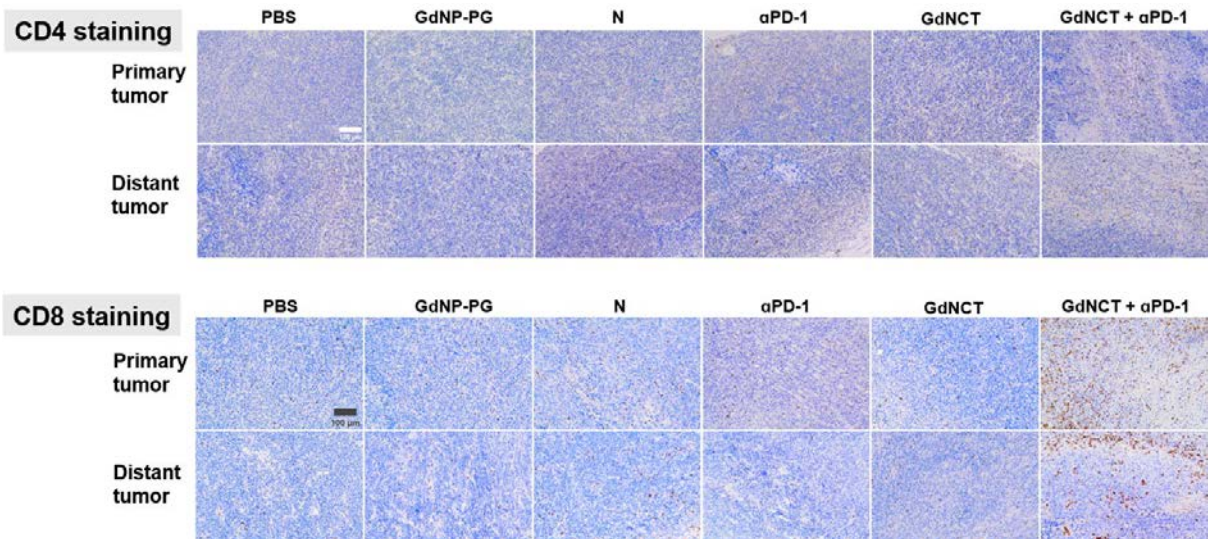


Figure 1. Immunohistochemistry staining of CD4 and CD8 in primary and distant tumors of different groups.

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Clonogenic Survival Assay of U87 MG Cells Exposed to Different Doses of BNCT, Neutrons, and X-rays of the KURNS Progress Report.

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INTRODUCTION: As a binary radiotherapeutic modality based on ^{10}B and neutron irradiation, boron neutron capture therapy (BNCT) involves the selective accumulation of non-radioactive ^{10}B in tumor cells, followed by thermal neutron capture to induce a nuclear reaction that generates high-linear energy transfer (LET) α particles and ^7Li particles. These high-LET particles exhibit strong relative biological effectiveness, causing lethal cellular damage. Owing to their short path length, cytotoxic effects are confined to the cellular scale, thereby minimizing damage to surrounding normal tissues^[1]. Previous radiobiological evaluations of BNCT have predominantly relied on reactor-based neutron sources, whose dose rates and neutron energy spectra differ markedly from those of the accelerator-based systems increasingly used in clinical settings. Consequently, understanding how neutron characteristics influence BNCT radiobiological outcomes represents a critical issue in the field. In this experiment, building upon our prior synthesis of the novel boron agent ANG-LIPO, the present study employs BPA as a reference to systematically investigate the impact of neutron properties on BNCT efficacy and the radiobiological performance of ANG-LIPO.

EXPERIMENTS: Clonogenic survival assay for X-ray and neutron irradiation: Log-phase adherent U87 cells were washed with PBS, trypsinized, centrifuged, and resuspended prior to irradiation. Cells were exposed to X-rays (0, 2, 4, 8, and 12 Gy) or neutrons (0, 0.31, 0.94, 2, and 3.67 Gy), then seeded into six-well plates (800 cells per well) and cultured for 14 days. Colonies were fixed with 4% paraformaldehyde, stained with 0.5% crystal violet, and counted to determine the survival fraction. Clonogenic survival assay for BNCT: Log-phase adherent U87 cells were co-incubated with culture medium containing ANG-LIPO or BPA, with concentrations determined based on a previously established neutron autoradiography method^[2]. Cells were then subjected to neutron irradiation at BNCT doses of 0, 0.67, 1.77, 2.88, and 3.99 Gy. Subsequent procedures were performed as described above.

RESULTS: As shown in Fig. 1, clone formation in U87 MG cells varies under different doses of BNCT, neutrons, and X-rays. Based on the number of colonies, the survival fractions of U87 cells corresponding to each dose were calculated using the formula. The survival fractions for BNCT (ANG-LIPO) were 1.00 ± 0.20 , 0.42 ± 0.03 , 0.04 ± 0.01 , 0.01 ± 0.01 , and 0. For BNCT (BPA), the corresponding values were 1.00 ± 0.09 , 0.48 ± 0.05 , 0.20 ± 0.09 , 0.04 ± 0.01 , and 0.01 ± 0.01 . For neutron, the corresponding values were 1.00 ± 0.06 , 0.84 ± 0.08 , 0.78 ± 0.04 , 0.36 ± 0.02 , and 0.11 ± 0.03 . For X-rays, the corresponding values were 1.00 ± 0.06 , 0.65 ± 0.14 , 0.24 ± 0.04 , 0.02 ± 0.01 , and 0.004 ± 0.004 .

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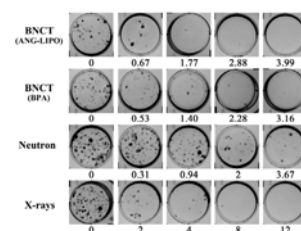


Fig. 1. Clone formation in U87 MG cells following exposure to different doses of BNCT, neutrons, and X-rays

Investigation of BNCT-induced cellular senescence and its modulation by dasatinib in vitro and in vivo

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INTRODUCTION:

Boron neutron capture therapy (BNCT) is a unique radiotherapeutic approach that relies on the nuclear reaction between thermal neutrons and boron-10 selectively accumulated in tumor cells. This reaction generates high-linear energy transfer particles, which can induce severe intracellular damage. While BNCT has mainly been studied as a cell-killing treatment, accumulating evidence from radiation biology suggests that irradiation can also trigger senescence-associated cellular responses. However, such responses after BNCT have not yet been sufficiently characterized. Therefore, we are investigating whether BNCT induces senescence-associated phenotypes in melanoma cells and are continuing to analyze the underlying molecular responses. We are also examining whether dasatinib modulates these BNCT-induced responses in vitro and in vivo.

EXPERIMENTS:

Murine melanoma B16F10 cells were exposed to boronophenylalanine (BPA) prior to neutron irradiation. After irradiation under defined neutron exposure conditions at KUR, the treated cells were subjected to several in vitro assays to evaluate senescence-associated changes, including SA- β -Gal staining, colony formation assay, and protein expression analysis by western blotting. Molecular analyses focused on representative senescence-related signaling molecules. In selected experiments, dasatinib was used to evaluate the pharmacological modulation of BNCT-induced senescence-associated responses.

In addition to the in vitro experiments, in vivo evaluation has also been initiated using a tumor-bearing mouse model to examine the antitumor response to BNCT under boron administration conditions. Tumor growth and treatment-associated responses, including the effects of dasatinib treatment, are currently being analyzed.

RESULTS:

In the in vitro studies, we have obtained preliminary findings suggesting that BNCT induces senescence-associated responses in B16F10 cells. Assessments based on SA- β -Gal staining, clonogenic potential, and expression analysis of senescence-related proteins are currently in progress. In addition, the modulatory effects of dasatinib on these responses are under investigation.

In the in vivo studies, evaluation of therapeutic responses following BNCT has also been conducted, and detailed analyses, including the effects of dasatinib treatment, are ongoing.

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Safety assessment of boron containing nano particle for chest BNCT

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INTRODUCTION: Boronophenylalanine (BPA) has been applied to boron-neutron capture therapy (BNCT) for late stage of head and neck squamous cell carcinoma and the clinical application for other type of cancers are actively expanding. On the other hand, BPA was quickly metabolized and excreted from the body within several hours, and therefore continuously infused during thermal neutron irradiation. To improve the disadvantages of BPA, we have developed 10B containing nano particle, RN-501 [1]. In this study, we investigated short and long term toxicity as pre-clinical safety assessment.

EXPERIMENTS: RN-501 was intravenously injected into eight weeks old BALB/c mice and then the mice were irradiated with thermal neutron which was generated at KUR with 5MW output energy for several time, the duration of which are ranging from 7.5 min to 45 min. The possible risk organs including lung, liver, spleen, esophagus, stomach, small and large intestine were harvested at a week or approximately 6 month after the irradiation. These tissues were fixed with buffered formalin and then embedded into paraffin blocks.

RESULTS: Now, the tissue slices are being processed and stained with hematoxylin and eosin.

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Examination of Improvement of BNCT treatment efficiency by L-phenylalanine deficiency in mice tumor models

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INTRODUCTION: L-Boronophenylalanine (L-BPA), a boronated isotope of L-phenylalanine (Phe), is used as a boron drug and is taken up by the tumor through its enhanced metabolism of amino acids. LAT1 is an exchange transporter, releasing one amino acid molecule into the extracellular space for the uptake of one amino acid molecule into the cell [1]. However, L-BPA is also taken up by normal cells via LAT2 and other pathways [2], and the improvement of the boron concentration ratio (T/N ratio) between tumor and normal tissues has become an issue. In this study, we attempted to improve the therapeutic effect of BNCT by restricting Phe and improving L-BPA up-take.

EXPERIMENTS: 5.0×10^5 human tongue carcinoma-derived cell line SAS and mouse melanoma-derived cell line B16 melanoma were transplanted into the thighs of 6-week-old female nude mice (Balb/cAJcl-nu/nu) and into the shoulders and thighs of inbred mouse (C57BL/6), respectively. Two weeks after transplantation, an immune checkpoint inhibitor (PD-1 inhibitor) was administered subcutaneously to C57BL/6 mice, and both Balb/cAJcl-nu/nu and C57BL/6 mice were switched to a phenylalanine-restricted diet. The following day, a 500 mg/kg solution of L-BPA was administered subcutaneously, and two hours later, the thighs were irradiated with neutrons. After irradiation, the mice were fed a standard diet, and tumor size was measured over a period of several weeks. In addition, in the C57BL/6 mice, a second dose of the PD-1 inhibitor was administered 7 days after the initial dose.

RESULTS: In Balb/cAJcl-nu/nu mice, tumor size decreased following L-BPA administration and neutron irradiation. Furthermore, in the group that received L-BPA administration and neutron irradiation after phenylalanine restriction, tumor size reduction was observed compared to the Phe (+) BNCT group (Figure 1). No significant changes in body weight were observed in these mice.

In C57BL/6 femoral tumors, an improvement in the BNCT effect due to phenylalanine restriction was observed, albeit to a small extent. Furthermore, for shoulder tumors, the combination of a PD-1 inhibitor and BNCT showed a higher tumor suppression effect than the BNCT-alone group. These results are thought to be due to an abscopal effect.

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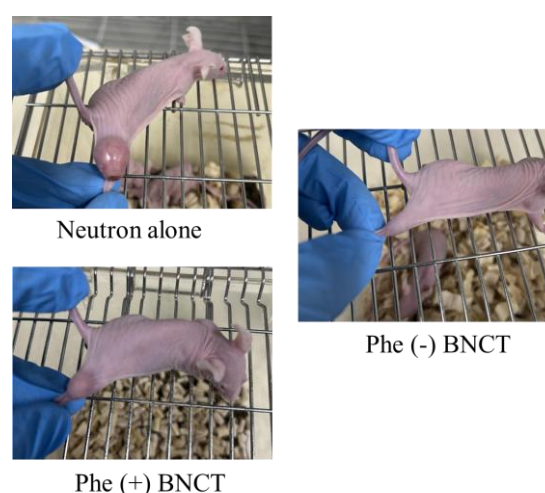


Fig.1 The Effect of Phenylalanine Restriction on BNCT in Balb/cAJcl-nu/nu Mice

Optimization study for non-clinical trials of polymeric BPA

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INTRODUCTION: While 4-borono-L-phenylalanine (L-BPA) serves as the primary therapeutic agent for boron neutron capture therapy (BNCT) by effectively targeting LAT1-expressing tumors [1], its overall efficacy is sometimes hindered by drug efflux. Because LAT1 functions as an antiporter, intracellular L-BPA is exchanged with extracellular amino acids such as tyrosine, leading to compromised retention time within the tumor and eventual therapeutic efficiency [2]. To overcome this limitation, we previously developed a simple technique to conjugate multiple L-BPA molecules with poly(vinyl alcohol) (PVA) via boronate esters. This complex shifts the cellular uptake mechanism to LAT1-mediated endocytosis, prolonging intratumoral retention of L-BPA [3]. Furthermore, we demonstrated that PVA could unleash the potential of the enantiomer of L-BPA, D-BPA, accomplishing enhanced LAT1-selectivity and appreciably prolonged intratumoral retention [4]. In this context, we recently found that the fine-tuning of the chemical structure of an L-BPA-like structure conjugated with PVA critically affects biodistribution. Here, we evaluated BNCT effects of newly developed PVA formulations.

EXPERIMENTS: PVA formulations were intravenously injected into mice bearing subcutaneous CT26 tumors. The tumors were irradiated with thermal neutrons using the Kyoto University Research Reactor (KUR).

RESULTS: All the PVA formulations demonstrated strong BNCT effects. Importantly, a novel PVA formulation achieved an almost complete cure of the tumors (Figure 1). As this formulation exhibits high LAT1-selectivity and a high tumor-to-normal organ (T/N) boron concentration ratio, it may be a promising candidate for practical applications.

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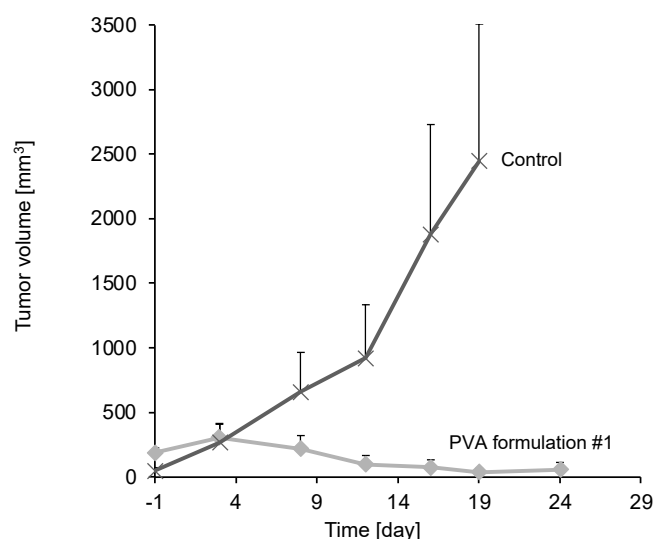


Fig. 1. BNCT effects on subcutaneous CT26 tumors.

Development of localized drug delivery systems

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INTRODUCTION: Currently, boronophenylalanine (BPA) is the most widely used agent in clinical studies. However, to maintain the necessary intratumoral boron concentration for boron neutron capture therapy (BNCT), a high-dose intravenous injection of approximately 500 mg/kg is required. Because BPA distributes to normal tissues to some extent, it cannot always achieve an adequate tumor-to-normal tissue (T/N) boron concentration ratio (2.5 or higher) depending on the cancer type. For such tumors, local administration of a highly concentrated boron agent appears to be a possible approach. Nevertheless, the low water solubility of BPA makes it challenging to prepare high-concentration formulations. Furthermore, because BPA is also recognized by other amino acid transporters such as LAT2 and ATB^{0,+}, there is a concern that it may distribute at high concentrations to adjacent normal tissues. To address these issues, we have been developing a patch-type drug delivery system (DDS) for the minimally invasive local administration of tumor-selective boron agent. In this study, we investigated the feasibility of treating superficial tumors with BNCT using this novel patch-type DDS. Specifically, the patch-type DDS was applied to the tumor site in a mouse subcutaneous tumor model. After a predetermined period, the patch was removed, and neutron irradiation was performed to evaluate the subsequent antitumor effects.

EXPERIMENTS: The patch-type DDS was applied to a subcutaneous CT26 tumor in a BALB/c mouse, and the tumor site was irradiated with thermal neutrons.

RESULTS: As shown in Figure 1, the newly developed DDS significantly inhibited the tumor growth. To further improve the potential, we will optimize the compositions in future study.

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N/A

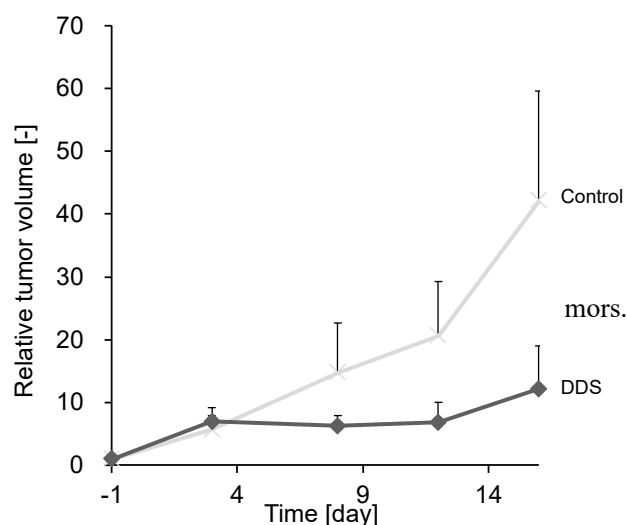


Fig. 1. BNCT effects on subcutaneous CT26 tumors.

Research and Development of New Technology for Boron Neutron Capture Therapy

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INTRODUCTION: Boron Neutron Capture Therapy (BNCT) requires sufficient intracellular accumulation of boron compounds in tumor cells to achieve effective cell killing by neutron irradiation. L-p-boronophenylalanine (L-BPA), one of the most widely used boron carriers, is preferentially taken up by cancer cells via amino acid transporters that are upregulated in tumors [1]. However, intracellular L-BPA levels decline rapidly after uptake, largely because of efflux mediated by specific transporters expressed in cancer cells. In this study, we examined a strategy to enhance BNCT efficacy by inhibiting such transporters and thereby suppressing L-BPA efflux. Using both cultured cells and tumor-bearing mouse models, we evaluated whether blocking L-BPA efflux enhances the antitumor effect of neutron irradiation. Building on the proof-of-concept studies performed in FY2022–2024, the FY2025 study is directed toward clinical translation by expanding the analysis in pancreatic tumor xenograft models and prolonging the interval between L-BPA administration and neutron irradiation.

EXPERIMENTS: In the FY2025 study, we used the human pancreatic cancer cell lines T3M4, Suit-2, and MiaPaCa-2, as well as the mouse triple-negative breast cancer cell line 4T1, all of which express high levels of transporters involved in BPA uptake. For the in vivo irradiation studies, tumor-bearing models were established by subcutaneous implantation of T3M4, Suit-2, and MiaPaCa-2 cells into nude mice. Similarly, 4T1 cells were inoculated subcutaneously into BALB/c mice. L-BPA was administered intravenously via the tail vein at 450 mg/kg. Thereafter, the active stereoisomer of a transporter inhibitor designed to suppress L-BPA efflux was administered at 200 mg/kg three times, at 1, 1.5, and 2.5 hours after L-BPA injection. In the control group, mice received the inhibitor alone without prior L-BPA administration before neutron irradiation. Neutron irradiation was carried out at a reactor output of 5 MW for 30 min. Tumor responses to neutron treatment were monitored and compared between treatment groups.

RESULTS: T3M4, Suit-2, MiaPaCa-2, and 4T1 cells were subcutaneously implanted to establish tumor-bearing models. Following intravenous administration of L-BPA, the animals were divided into three groups. Group 1 received additional injections of a transporter inhibitor at 1, 1.5, and 2.5 h after L-BPA administration. Group 2 received L-BPA alone without subsequent inhibitor treatment. Group 3 received saline instead of L-BPA, followed by inhibitor injections at the same time points as in Group 1. All the mice were subjected to neutron irradiation 6 h after the initial administration of L-BPA or saline. Tumor volumes were monitored for 8 weeks after irradiation. Group 1 showed a marked reduction in tumor volume compared with Group 2, indicating that inhibition of the transporter enhanced the efficacy of BNCT. This enhancement was particularly pronounced in the Suit-2 model. In our previous studies, the potentiating effect of the transporter inhibitor had been demonstrated under conditions in which neutron irradiation was performed 2.5 h after L-BPA administration. In the FY2025 study, enhanced BNCT efficacy by the transporter inhibitor was confirmed when neutron irradiation was carried out 6 h after L-BPA administration. These findings further substantiate the feasibility of this strategy for enhancing the efficacy of L-BPA-based BNCT.

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Development and Preclinical Evaluation of Novel Boron Neutron Capture Therapy (BNCT) Agents

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INTRODUCTION: Epithelial cell adhesion molecule (EpCAM) is a type I transmembrane glycoprotein involved in cell–cell adhesion, proliferation, and signal transduction. Although expressed in normal epithelial tissues, EpCAM is frequently overexpressed in various epithelial malignancies, including colorectal, breast, pancreatic, and ovarian cancers [1]. Its expression level has been associated with tumor aggressiveness, metastatic potential, and prognosis, highlighting EpCAM as a promising target for cancer imaging and therapy [2-4]. We have previously developed a novel anti-EpCAM monoclonal antibody (EpCAM-1) and evaluated an ¹¹¹In-labeled EpCAM-1 antibody in vitro. The radiolabeled antibody demonstrated efficient internalization via EpCAM-mediated uptake, indicating favorable properties for targeted intracellular delivery. Based on these findings, we aimed to exploit the high internalization capacity of EpCAM-1 to develop a boron neutron capture therapy (BNCT) agent by conjugating ¹⁰B to the antibody.

EXPERIMENTS: In vitro evaluation: A colony formation assay was performed using an EpCAM antibody conjugated with ¹⁰B (¹⁰B–EpCAM-1). EpCAM-positive DLD-1 cells were seeded in 12-well plates, and ¹⁰B–EpCAM-1 was added at concentrations of 0, 10, 20, 30, and 50 µg/mL one day prior to neutron irradiation. On the day of irradiation, the cells were collected into Eppendorf tubes and subjected to neutron irradiation. The cells were then cultured at 37°C in a humidified atmosphere with 5% CO₂. Cell damage was assessed at 2 and 4 days post-irradiation using Annexin V/PI staining, and a colony formation assay was performed 10 days after irradiation.

RESULTS: In the colony formation assay, a dose-dependent decrease in cell proliferative capacity and survival fraction was observed in the ¹⁰B–EpCAM-1–treated groups compared with the control group. Furthermore, Annexin V/PI staining revealed that the proportion of damaged cells increased in a dose-dependent manner at both 2 and 4 days after irradiation. The therapeutic efficacy has also been evaluated in tumor-bearing animal models, and the experiments have been completed; data analysis is currently in progress.

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Effective BNCT against BPA-resistant glioblastoma using novel ASCT2-targeted boron carriers

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INTRODUCTION: Boron neutron capture therapy (BNCT) is a tumor-selective radiotherapy that uses the nuclear reaction between ^{10}B and thermal neutrons to generate high linear energy transfer (LET) particles, causing localized cytotoxicity in boron-accumulating cells. 4-Borono-L-phenylalanine (BPA), the most widely used clinical boron carrier, is transported into tumor cells via L-type amino acid transporter 1 (LAT1) [1]. Although BPA-based BNCT shows clinical benefits, its efficacy is limited in tumors with low LAT1 expression due to insufficient boron accumulation. U87MG glioblastoma is a LAT1-low tumor model and is resistant to BPA-based BNCT. To address this, we developed **GluBs**, small-molecule boron carriers targeting alanine-serine-cysteine transporter 2 (ASCT2), which is often upregulated in aggressive cancers and plays a key role in tumor metabolism. In this study, we examined whether ASCT2-targeted boron delivery using **GluB-2**, one of the **GluBs**, improves BNCT efficacy in BPA-refractory glioblastoma [2].

EXPERIMENTS: **GluB-2**, a glutamate-based boron carrier, was designed and synthesized to enable selective uptake via ASCT2. Its biological performance was evaluated in a human glioblastoma U87MG xenograft model. Tumor-bearing mice (female, 5–6 weeks old) were intravenously administered **GluB-2** or BPA at 25 mg [^{10}B]/kg. At 3 h post-injection, when tumor boron accumulation was expected to peak, thermal neutron irradiation was applied to the tumor ($3.8\text{--}4.3 \times 10^{12}$ neutrons/cm²). BNCT efficacy was assessed by monitoring tumor growth, and systemic safety was evaluated by measuring body weight throughout the experimental period.

RESULTS: **GluB-2** achieved tumor boron concentrations of approximately 20 μg [B]/g in U87MG xenograft, reaching the threshold required for effective BNCT. This enhanced tumor selectivity is consistent with ASCT2-mediated uptake in LAT1-low tumor cells. Following neutron irradiation, **GluB-2** treatment resulted in pronounced and sustained suppression of tumor growth compared with both BPA-treated and control groups. In contrast, BPA exhibited only modest antitumor effects.

Importantly, no significant body weight loss or observable systemic toxicity was detected in any treatment group, indicating that **GluB-2** is well tolerated at therapeutically relevant doses. Collectively, these results demonstrate that **GluB-2** enables efficient and selective boron delivery via ASCT2, thereby significantly enhancing BNCT efficacy in glioblastoma models that are less responsive to conventional BPA-based therapy.

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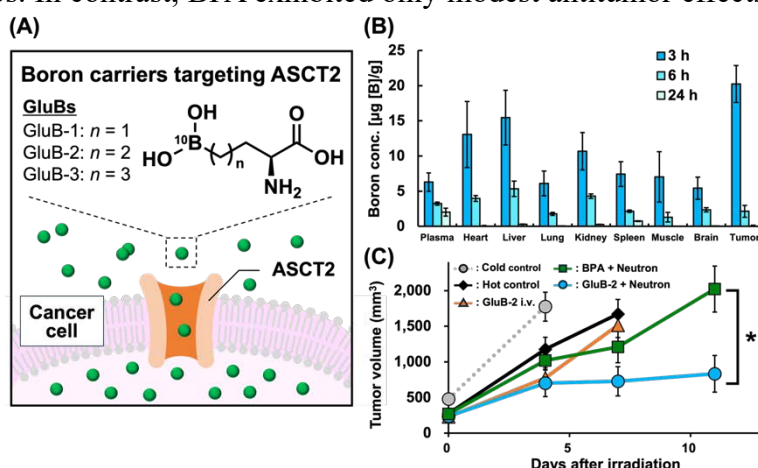


Fig. 1. Novel ASCT2-targeted boron carriers for BPA-refractory cancers. (A) ASCT2-targeted ^{10}B delivery system for BNCT. (B) Boron concentrations in major organs at 3, 6, and 24 h after intravenous (*i.v.*) injection of GluB-2 (25 mg [B]/kg). (C) Tumor volume in mice after BNCT with GluB-2 and BPA (25 mg [^{10}B]/kg *i.v.*). Data are expressed as mean $\pm$ SD ($n = 5\text{--}6$). Significance was determined as $*p < 0.05$ using the two-sided Student's *t*-test.

Boron-Fused π -Conjugated Polymer-Hyaluronic Acid Complex for BNCT

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INTRODUCTION: Development of boron agents with imaging capabilities is required to maximize the therapeutic benefits of boron neutron capture therapy (BNCT), as such systems enable visualization of boron agent accumulation in tumor tissues. Today, ¹⁸F-labeled L-boronophenylalanine (¹⁸F-BPA) is clinically available as a positron emission tomography (PET) tracer to visualize bio-distribution of L-BPA for the prediction of therapeutic efficacy. Although PET imaging can provide images with the spatiotemporally accurate their distribution, PET tracers are costly and their synthesis is restricted to specialized RI facilities. For the perspectives, fluorescence imaging is cost-effective and provides a high signal-to noise ratio, making it a promising alternative to PET.

In this study, we developed a water-dispersible hyaluronic acid (HA) complex of a boron-fused π -conjugated polymer (BCP), which serves as a boron agent. The complex was prepared via a mechanochemical blending strategy using a ball mill^{1,2} and enables subcellular tracking through its intrinsic fluorescence.

RESULTS AND DISCUSSION: We prepared a solid-phase complex of HA and BCP via high-speed vibration milling (HSVM). The resulting mixture was extracted with water and treated with sonicator. The complexation of BCP was confirmed by measuring UV-Vis spectra and ICP-OES, and the boron concentration of the resulting dispersion was determined to be 86 ppm. Judging from the scalability of HA, we can prepare approximately 1000 ppm. Moreover, size distribution of the complex was confirmed by nanoparticle tracking analysis. The mean diameter of the complex was determined to be approximately 100–130 nm; thus, the system is of an appropriate size to achieve tumor accumulation via passive targeting through the enhanced permeability and retention (EPR) effect.

We next demonstrated BNCT activity of current system toward murine squamous carcinoma cell line *in vitro*. Here, we employed L-BPA/sorbitol complex, which is clinically available boron agent, as the control. After exposure to these boron agents for 24 h, the cells were received thermal neutron irradiation. At 24 h-post irradiation, the cell viability was quantified and HA/BCP complex exhibited higher BNCT activity than L-BPA/sorbitol complex. This should be achieved by recognition of HA through CD44 which is overexpressed in cancer cells. Moreover, confocal laser scanning microscopic observation revealed that our system showed strong fluorescence within cells after internalization. For these results, our systems are potentially applicable as a boron agent with imaging property and tumor targeting property.

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L-BPA conjugated tannic acid for efficient boron delivery

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INTRODUCTION: L-boronophenylalanine (L-BPA) is one of the most promising candidates as a boron delivery agent for boron neutron capture therapy (BNCT), owing to its excellent delivery to cancer cells, primarily via the L-type amino acid transporter 1 (LAT1). Despite its advantages, there are still several limitations in delivery including water solubility and duration period within cells. To address these issues, we designed and synthesized L-BPA conjugated tannic acid (TA-BPA) as boron agents for BNCT. Herein, tannic acid efficiently interacts with bioactive proteins via noncovalent interactions, including hydrogen bonding [1], thereby forming nanoassemblies capable of encapsulating proteins. Using the system, we established co-delivery system for boron and immune checkpoint inhibitor as an abscopal effect inducer.

RESULTS AND DISCUSSION: L-BPA was conjugated with tannic acid through boronic acid–cis-diol interactions under basic conditions. Conjugation was confirmed by inductively coupled plasma-optical emission spectroscopy (ICP-OES) and mass spectroscopy (MS), revealing that tannic acid was functionalized with approximately 3-4 BPA molecules. We next conducted complexation of antibody, which is used as the model of immune checkpoint inhibitor, with TA-BPA by mixing these two components. The molecules spontaneously formulated assembly with submicron size, and their core-shell structure were revealed by small angle X-ray scattering measurement (SAXS). Their small size and core-shell structure are advantageous for tumor targeting, primarily via the enhanced permeability and retention (EPR) effect, and for conferring resistance of encapsulated antibodies to proteases. Single-particle analysis using imaging flow cytometry revealed that TA-BPA nanoassemblies encapsulate target proteins with quantitative uniformity across individual particles. Moreover, the nanosystem can release the cargo by responding to ATP molecules, which is abundant in tumor tissues, thereby current system can deliver both boron agent and immune checkpoint inhibitor to tumor tissue and these pharmaceuticals should be delivered to target cells.

We demonstrated BNCT activity toward murine lymphocyte cancer cell line which overexpress model antigen protein, ovalbumin (E.G7-OVA) *in vitro*. Herein, we employed L-BPA-sorbitol complex, which is clinically available boron agent. As a result, our system exhibited higher BNCT activity since our system enhanced cellular uptake efficiencies and prolonged duration period within cells. Finally, we demonstrated therapeutic efficacy in a metastasis mouse model. Notably, our system achieved tumor regression even in satellite tumor regions.

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Attempts to sensitize tumor cells by exploiting the tumor microenvironment

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INTRODUCTION: Boron neutron capture therapy (BNCT) is a radiotherapy that kills tumor cells via the $^{10}\text{B}(\text{n}, \alpha)^7\text{Li}$ reaction [1]. As reported previously, these high-LET particles produce highly complex DNA damages, which are processed by intracellular pathways, including DNA damage repair. Previous researches have been trying to enhance BNCT efficacy through modulation of DNA damage repair. DNA-protein crosslinks (DPCs) are induced by various chemotherapeutic agents and ionizing radiation, and considered to be toxic DNA lesions, as well as DNA double strand breaks (DSBs) since they inhibit crucial cellular processes, such as replication and transcription [2]. In this study, we established Sprtn protease-deficient cells incapable of repairing DPCs, and examined the contribution of this Sprtn protease to cell survival after BPA-BNCT.

EXPERIMENTS: CRISPR/Cas9 system was used to target the 2nd exon of Sprtn protease gene. A 5' and 3' homology arms (1kb) were amplified by PCR from the SCC VII cell genome, and cloned into the targeting vector containing a neo-resistance marker. To generate Sprtn protease-deficient SCCVII cells, the SCC VII cells were transfected with the targeting vector, and six cell clones (#1-6) were obtained. To examine the sensitivity to BPA-BNCT, these cells were exposed to neutron beams (KUR Heavy Water Facility) and then clonogenic cell survival assays were performed. Moreover, SCC VII and Sprtn protease-deficient cells (clone #1) were subcutaneously inoculated into the right hind legs of C3H mice. BPA was subcutaneously administered into nuchal sites in tumor bearing-mice, and after 60 min, mice received neutron irradiation. Tumor volume and body weight was measured every three days.

RESULTS: It has been suggested that mouse Sprtn is essential for normal cell-cycle progression. In the study, six cell clones were obtained. In three clones (#1-3), both alleles contained indels, but one of them did not cause frameshift. In three other clones obtained (#4-6), only one allele carried a frameshifting indel. These results suggest that Sprtn gene is also essential for SCC VII cells consistent with the previous studies. Colony formation assays after BPA-BNCT showed that the survival rate of clone #4-6 were similar to that of parental SCC VII cells (Fig. 1, left). On the other hand, the clone #1-3 were relatively sensitive to BPA-BNCT, although they carried a non-frameshifting indels. Considering that the region encoded by exon-2 is part of the active site of Sprtn protease, even small changes in the amino acid sequence might have an impact on its activity. We also examined the effect of Sprtn deficiency on the BPA-BNCT efficacy using the tumor bearing mice model, and showed that down-regulation of Sprtn slightly suppress tumor growth after neutron-irradiation (Fig. 1, right).

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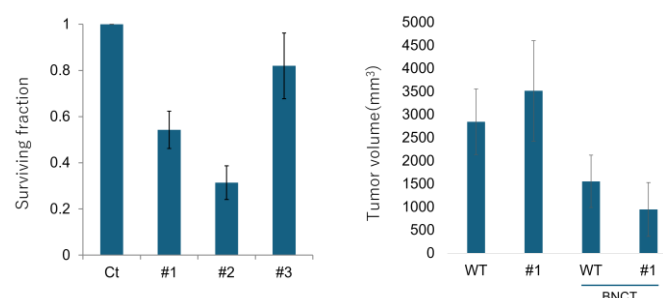


Fig. 1. Relative survival rates of SCCVII and Sprtn protease-deficient cells after BPA-BNCT (left panel). Average tumor volumes of non-treated and BPA-BNCT groups (right panel).

Preliminary Evaluation of the Characteristics and Effectiveness of an In Vitro Concentric Experimental System for Gadolinium Neutron Capture Therapy

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INTRODUCTION: Following our previous studies, we conducted experiments in 2025 to further investigate the cytotoxic mechanisms of gadolinium neutron capture therapy (GdNCT). Among the radiation components generated by the gadolinium neutron capture reaction (GdNCR)—gamma rays, internal conversion electrons, and Auger electrons—electron emissions are considered to have the highest cytotoxic potential. In this study, we established an experimental system designed to exclude the contribution of electron emissions and performed in vitro cellular experiments to evaluate the effects of gamma radiation alone.

MATERIALS AND METHODS: A concentric experimental system was constructed by placing a plastic disk (30 mm in diameter, 1 mm in thickness) at the center of a 10-cm culture dish (KURTR:R5174). The 1-mm-thick plastic was assumed to effectively shield electron emissions, thereby preventing electron-induced cytotoxic effects in the peripheral region while allowing gamma rays to penetrate. U87 cells were cultured in a sub-confluent state in the 10-cm dishes and exposed to neutron irradiation. Gamma-ray dose was measured using thermoluminescent dosimeters (TLDs).

RESULTS: The measured gamma-ray dose reached approximately 3 Gy, exceeding the D_0 value (~ 2 Gy). However, no significant difference in cell survival was observed between the central and peripheral regions.

These findings suggest that the combined cytotoxic effects of thermal/epithermal neutrons and gamma radiation from gadolinium were approximately uniform across the system. Accordingly, the cytotoxic effect of gamma rays and their potential contribution to damage in surrounding tissues appear to be limited. Furthermore, the experimental system effectively excluded the contribution of electron-induced cytotoxicity.

DISCUSSION: In previously reported in vivo studies, GdNCT has demonstrated substantial cytotoxic effects, which are thought to be predominantly attributable to electron emissions. Although the present experimental system did not allow definitive conclusions regarding intracellular uptake of gadolinium micelles, the results strongly suggest that electron emissions generated by GdNCR are the primary contributors to the therapeutic effect. Moreover, the contribution of gamma rays in GdNCT appears to be minimal, consistent with in vivo observations showing only mild radiation-induced damage in surrounding normal tissues. In our prior work, we reported that local thermal neutron irradiation combined with perfusion of a gadolinium-containing solution (1000 ppm) into rat femoral melanoma resulted in tumor shrinkage or complete disappearance without significant damage to adjacent tissues (Narayan, et al, 2012). This finding further supports the notion that electron emissions generated within tumor vasculature are the principal drivers of therapeutic efficacy. Although the contribution of gamma radiation in GdNCT may increase with tumor volume, it is likely limited in tumors of approximately 3 cm in diameter, as assumed in this study. In addition, when intratumoral gadolinium concentration exceeds 500 ppm, neutron self-shielding becomes significant. Therefore, the present experimental system reasonably reflects in vivo irradiation conditions.

CONCLUSIONS: This preliminary study demonstrates that the cytotoxic effect of gamma radiation in GdNCT, as well as its contribution to normal tissue damage, is limited. Our findings indicate that GdNCT is fundamentally an electron-driven therapeutic modality, in which localized energy deposition at the microscale plays a critical role. Future studies using this experimental system will focus on detailed dosimetric evaluation and quantitative analysis of cell survival to further assess the clinical feasibility and optimization of GdNCT.

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In vivo evaluation of a novel boronoagent for LAT1-targeted BNCT

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INTRODUCTION: Currently, the only boron-containing drug used clinically in Boron Neutron Capture Therapy (BNCT) is 4-¹⁰B]borono-L-phenylalanine (BPA), which has limitations such as poor tumor selectivity and low water solubility. The development of novel BNCT agents that overcome these drawbacks is an urgent challenge to improve therapeutic efficacy and expand the range of treatable cancer types. We have previously developed a new boron-containing compound, 5F- α Me-3BPA, through a medicinal chemistry approach involving structural modification of BPA [1,2]. This compound exhibits tumor accumulation comparable to that of BPA, while demonstrating superior tumor selectivity and improved water solubility. Because 5F- α Me-3BPA contains both fluorine and boron atoms within its molecular structure, it has the potential to serve as an ideal theranostic pair. Specifically, labeling with ¹⁸F and ¹⁰B enables its application to both positron emission tomography (PET) and BNCT, respectively, while maintaining the same molecular framework [3]. The objective of this study is to evaluate the in vivo therapeutic efficacy of 5F- α Me-3BPA by conducting neutron irradiation experiments in tumor-bearing mice using its ¹⁰B-labeled form.

EXPERIMENTS: Tumor-bearing mice were administered the ¹⁰B-labeled form of 5F- α Me-3BPA via intraperitoneal injection, followed by neutron irradiation at the Kyoto University Research Reactor (KUR). As controls, a BPA-treated group and a hot control group receiving neutron irradiation alone were included. After irradiation, the animals were returned to the animal facility, and tumor volumes were measured over time. Neutron irradiation experiments at KUR were conducted twice at a reactor power of 5 MW.

RESULTS: In the first of the two neutron irradiation experiments conducted at KUR, tumor growth was markedly slower than expected in the tumor-bearing mice, likely due to technical issues for tumor implantation, which limited the extent of the evaluation. In the second experiment, this issue was mitigated, and a sufficient number of tumor-bearing mice were successfully prepared. Following compound administration and neutron irradiation, a reduction in tumor size was observed in both the 5F- α Me-3BPA-treated group and the BPA-treated group, indicating therapeutic efficacy.

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A Comparative Study of Reactor- and Accelerator-based Boron Neutron Capture Therapy on U87MG Glioblastoma Xenograft Model

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INTRODUCTION: Boron neutron capture therapy (BNCT) is gaining attention as a promising non-invasive cancer treatment. Accelerator-based systems are increasingly favored for their practicality and accessibility, with the assumption that they can provide comparable therapeutic outcomes to reactor-based systems. Therefore, confirming their clinical equivalence is essential for safe and effective implementation. Malignant brain tumors are among the most promising targets for BNCT, and several clinical studies have been conducted. 4-Borono-L-phenylalanine (BPA) is the most widely used boron carrier, accumulating in tumor cells via LAT1, which is highly expressed in glioblastoma and other cancers [1]. In this study, we evaluate the therapeutic equivalence of BNCT between reactor- and accelerator-based systems, using BPA as a reference compound, with the aim of supporting the transition from reactor- to accelerator-based BNCT [2].

EXPERIMENTS: Tumor-bearing mice (female, 5-6 weeks old) were prepared by injecting subcutaneously (*s.c.*) a suspension of human glioblastoma U87MG cells. The tumor-bearing mice were injected *i.v.* with 100 μ L of adjusted BPA at a 25 mg [^{10} B]/kg dose. At 3 h after injection, neutron irradiation was administered to the tumor sites in mice. BNCT efficacy was evaluated by monitoring changes in tumor volume and body weight following irradiation. For KUR reactor-based BNCT, a fluence of $4.0\text{--}4.2 \times 10^{12}$ neutrons/cm² was applied over a 12 min irradiation period. In contrast, for accelerator-based BNCT (HM-20V, Aomori Prefecture Quantum Science Center), a fluence of approximately 1.2×10^{12} neutrons/cm² was applied, with irradiation conducted for 60 min at a power of 100 μ A and 20 MeV.

RESULTS: Tumor growth was significantly suppressed in both reactor-based (Fig. 1A) and accelerator-based (Fig. 1C) BNCT with BPA. In addition, no significant body weight changes were observed in either group, suggesting the absence of notable adverse effects (Fig. 1B and 1D). The results demonstrated comparable BNCT efficacy with BPA between reactor- and accelerator-based systems, underscoring the clinical potential of BNCT. Notably, comparison of the “neutron only” and “no treatment” groups revealed greater tumor growth suppression in the accelerator-based experiment. This difference may be attributed to variations in gamma radiation dose associated with the extended irradiation time required for accelerator-based BNCT.

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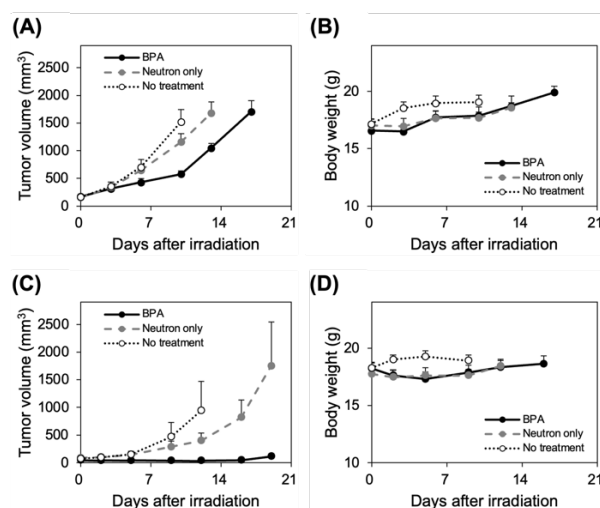


Fig. 1. Anti-tumor effects of reactor- and accelerator-based BNCT with BPA in U87MG xenograft mice model. Tumor volume and body weight changes in mice after BNCT with BPA (25 mg [^{10} B]/kg *i.v.*) at a reactor (A and B) or an accelerator (C and D). Data are expressed as mean $\pm$ SD ($n = 3\text{--}5$).

Tumor-Selective Prodrugs Incorporating Carborane-Containing Amino Acid Payloads Targeting Cancer Amino Acid Metabolism

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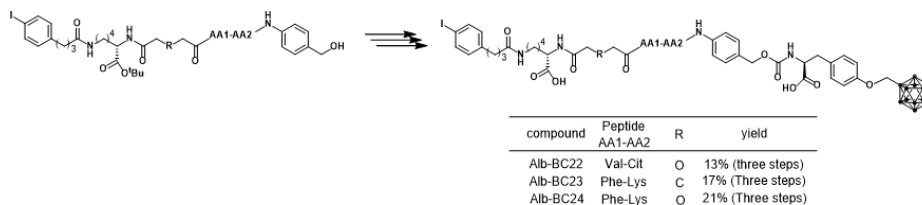
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INTRODUCTION: Amino acid transporters such as LAT1 are highly expressed in cancer cells and support tumor growth by mediating the uptake of amino acids bearing bulky hydrophobic substituents. Exploiting this metabolic feature, we have developed carborane-containing amino acid analogues for tumor-selective boron delivery in BNCT. Among them, BC2 and BCY2 showed high cellular uptake and superior *in vitro* efficacy compared with BPA; however, their poor solubility and suboptimal physicochemical properties have limited *in vivo* evaluation. In this report, we describe the development of albumin-binding prodrug derivatives of BC2 and present their synthesis and preliminary functional evaluation toward *in vivo* application.

EXPERIMENTS & RESULTS: To enable *in vivo* application of BC2, we employed an albumin-binding prodrug strategy incorporating a cathepsin B-cleavable linker. In our previous study, model compounds releasing phenylalanine were synthesized, and their cathepsin B-mediated cleavage was confirmed. Based on these findings, we constructed prodrug derivatives using BC2 as the payload. The prodrugs consist of BC2 conjugated to a carrier moiety comprising an albumin-binding unit and a cathepsin B-responsive linker, enabling tumor-selective release of the boron carrier.

As shown in Scheme 1, a series of albumin-binding prodrug derivatives with varying linker structures and cleavage sequences were synthesized by



Scheme 1. Synthesis of Alb-BC2 Prodrugs

conjugation of BC2 to the carrier moiety, followed by deprotection. The isolated yields of each compound are summarized in Scheme 1.

Alb-BC22 was dissolved in MES buffer and incubated with cathepsin B alone or in the presence of HSA (1 equiv), and the reaction was monitored over time by HPLC. As shown in Fig. 1, Alb-BC22 was efficiently degraded in the presence of cathepsin B, with nearly complete cleavage observed within 24 h, resulting in the release of BC2. In contrast, the compound remained largely stable when HSA was present, indicating that albumin binding suppresses enzymatic cleavage.

A DMSO solution of Alb-BC22 was diluted 10-fold with PBS and mixed with HSA in PBS at a molar ratio of 1:20 (HAS : Alb-BC2). The mixture was incubated at 37 °C for 24 h. The mixture was concentrated using a centrifugal gel filtration cartridge to remove low-molecular-weight species, affording an 8% albumin solution. The boron content was determined by ICP analysis, providing a solution with a boron concentration of 1.13 mg B/mL, which can be used for subsequent *in vivo* studies.

These results suggest that HSA conjugation may enable *in vivo* application of BC2 by improving its solubility. Ongoing studies are focused on evaluating the biodistribution of these conjugates in tumor-bearing mouse models.

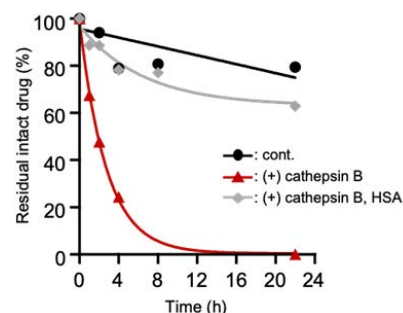


Fig. 1. Cathepsin B-Mediated Degradation of Alb-BC22 with and without HSA

Development of Novel Small-molecule Boron Neutron Capture Therapy Drugs Targeting Tumor-specific Enzymatic Activity

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INTRODUCTION: In boron neutron capture therapy (BNCT), *p*-boronophenylalanine (BPA), the only currently approved BNCT agent, is selectively taken up by tumor cells through LAT1, a biomarker transporter overexpressed in tumor cells. However, BPA faces two key challenges: 1) insufficient accumulation in some cancers due to low LAT1 expression, and 2) gradual leakage from cells over time. Therefore, new BNCT drugs targeting alternative cancer biomarkers with a mechanism for prolonged intracellular retention are needed to expand BNCT indications and improve therapeutic efficacy. In this project, we focused on enzyme activity, which is higher in cancer cells than in normal cells. By last year, we had developed EP-4OCB-FMA, a carborane-containing drug candidate targeting DPP-4, which is upregulated in esophageal cancer, and conducted BNCT experiments in tumor-bearing mice via intratumoral or intravenous injection. This drug is designed for prolonged intracellular retention by generating an aza-quinone methide species upon hydrolysis by DPP-4, which forms a covalent bond with intracellular nucleophiles such as cysteine-containing proteins and glutathione. While this probe performed well with intratumoral injection, systemic administration resulted in insufficient tumor accumulation, presumably due to its short blood half-life due to relatively high enzymatic activity in normal tissues. Therefore, this year we initiated the development of new probes targeting glycosidase, which exhibits enough high activity in cancer cells but low activity in normal cells, expected to offer improved longer blood half-life.

EXPERIMENTS: β -Gal-4OCB-FMP and β -Gal-4OCBOMe-FMP were designed and synthesized based on quinone methide-generating chemistry toward β -D-galactosidase (β -Gal), which is overexpressed in certain types of cancer cells. These agents are hydrolyzed by β -Gal to generate a highly reactive quinone methide intermediate, which rapidly reacts with intracellular nucleophiles such as glutathione, thereby the carborane boron cluster is selectively trapped inside the cancer cells. First, each probe was incubated with H460 and SKOV-3 cells, and boron concentration was evaluated. For the BNCT experiment, with using tumor-bearing mouse models, β -Gal-4OCB-FMP was administered to H460 tumor-bearing mice, and β -Gal-4OCBOMe-FMP was administered to SKOV-3 tumor-bearing mice. Neutron irradiation was performed at 5 mW for 15 minutes, 4 hours after intratumoral (i.t.) injection of each probe. Antitumor efficacy was quantified by measuring tumor size following irradiation.

RESULTS: Boron concentration in treated cancer cells were confirmed to be sufficiently high for BNCT. Based on this finding, BNCT experiments were conducted via intratumoral injection. The results demonstrated that tumor growth was suppressed in a drug- and neutron-irradiation-dependent manner, as shown in Fig. 1. We are therefore currently planning to develop optimized agents with longer blood half-life, and conduct further BNCT experiments via intravenous injection.

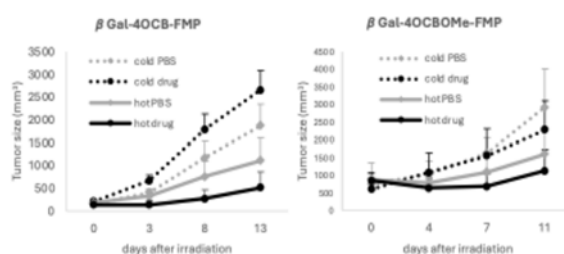


Fig. 1. Antitumor effects of β -Gal-targeted BNCT probes in tumor-bearing mice. Tumor volume was calculated as (longest diameter / 2) $\times$ (shortest diameter)². Data are mean $\pm$ SD (n = 3).

A Study on Improving the Retention of Intratumoral Boron Concentrations Using a Boron-Containing BPA-Embedded Nanofiber Mesh

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INTRODUCTION: Boron neutron capture therapy (BNCT) is a binary cancer treatment modality based on selective accumulation of boron-10 (¹⁰B) in tumors followed by neutron irradiation. The clinically used boron agent, p-boronophenylalanine (BPA), shows favorable tumor selectivity; however, its rapid systemic clearance requires prolonged intravenous infusion, typically over several hours. This limitation complicates clinical workflows and may reduce treatment efficiency. To address this issue, we developed a local drug delivery system using polycaprolactone (PCL)-based nanofiber mesh (NFM) incorporating BPA (BPCL-NFM). PCL is a biodegradable and biocompatible polymer widely used for controlled drug release. This system enables sustained boron release at the tumor site, enhancing tumor retention of ¹⁰B and improving BNCT efficacy while reducing systemic exposure.

EXPERIMENTS: Murine colon carcinoma CT26 cells were cultured in E-MEM supplemented with 10% fetal bovine serum. Neutron irradiation was performed at the Kyoto University Research Reactor (KUR) at 1 MW (5–20 min) for cells and 5 MW (12 min) for animals. BPCL-NFM was fabricated in collaboration with the National Institute for Materials Science and contained BPA up to 3 mg/mL. Morphology and drug release profiles were evaluated. BPA-fructose solution (BPA-f) was prepared as a control. In vitro, intracellular ¹⁰B accumulation was measured by ICP-AES after treatment with BPA-f (10–40 ppm, 1 h) or BPCL-NFM (up to 48 h), and cell survival was assessed by clonogenic assay. In vivo, CT26 tumor-bearing BALB/c mice received BPA-f or BPCL-NFM placed on tumors. Tumor boron concentration was measured over time (3–48 h), and BNCT efficacy was evaluated by tumor growth and body weight.

RESULTS: In vitro, BPCL-NFM showed time-dependent intracellular ¹⁰B accumulation, peaking at 24 h and maintained up to 48 h. The boron level was comparable to that achieved with 25 ppm BPA-f after 1 h exposure, and BNCT-induced cytotoxicity was similar between the two conditions. In vivo, BPA-f showed rapid accumulation with a peak at 3 h, declining to baseline at 24 h. In contrast, BPCL-NFM exhibited gradual accumulation with sustained levels from 15 to 48 h, reflecting controlled release. Therapeutically, BPCL-NFM with neutron irradiation significantly suppressed tumor growth compared with controls. The effect was greater than BPA-f at 125 mg/kg, comparable to 250 mg/kg, but lower than 500 mg/kg, suggesting sustained boron retention contributes to BNCT efficacy.

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Early Cellular Responses to BNCT and Potential Biomarker Identification

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INTRODUCTION:

We previously reported the involvement of HMGB1 and early proteomic alterations following BNCT [1-2]. However, the detailed mechanisms underlying early cellular responses remain unclear. In this study, we aimed to further characterize these responses and identify potential biomarkers to improve BNCT efficacy.

EXPERIMENTS:

Human cancer cell lines, including squamous cell carcinoma lines SAS, HSC3, cervical cancer HeLa cells, and mouse L1210 cells were incubated with BPA for 2 hours in suspension. Cell survival was evaluated by colony formation assay. Culture supernatants and cell lysates were collected at 6 and 24 hours post-irradiation for RNA and protein analyses. For in vivo studies, B16F10 and B16F10mGM melanoma cells were implanted into the hind limbs of C57BL/6 mice. In some groups, macrophages were depleted to assess their contribution. Only the left hind limb tumors were locally irradiated for 60 minutes using a ⁶LiF-containing shield. BPA was administered at 500 mg/kg body weight 30 minutes prior to irradiation. Mice were sacrificed 7 or 14 days after treatment, and blood and tissue samples were collected for analysis.

RESULTS:

The measurement results of thermal neutron fluence and doses for cells (Table 1) and mice (Tables 2 & 3) were indicated. Thermal neutron fluence and radiation doses were successfully measured in both vitro and vivo experiments. BNCT induced significant early cellular responses in the cancer cells. Notably, modulation of *SNHG12* expression altered these responses, suggesting its involvement in BNCT-induced molecular pathways. *SNHG12* may play a regulatory role in these processes. Preliminary analyses also indicated dynamic changes in gene and protein expression following treatment. In vivo responses, including tumor progression and immune-related changes, are currently under investigation.

Further studies will focus on immune mechanisms and in vivo validation will contribute to a better understanding of BNCT and support the identification of biomarkers for improved therapeutic outcomes.

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Table 2. Irradiated doses for local irradiation of mice on Dec.3, 2025 (Cart, irradiation room).

Irradiation time[min]	Position	fluence [cm ²]		[Gy]					
		Thermal neutron [cm ²]	Epi-thermal neutron [Gy]	Thermal neutron [Gy]	Epi-thermal neutron [Gy]	Fast neutron [Gy]	Gamma-ray [Gy]	Physical Dose [Gy]	B-10** (ppm)
10	Center	1.1E+12	1.9E+11	1.4E-01	1.5E-02	1.1E-01	0.2E-02	3.2E-01	7.9E-02
60	Center	6.5E+12	1.2E+12	8.7E-01	9.3E-02	6.4E-01	3.6E-01	2.0E+00	4.8E-01
2	Center	2.1E+11	3.8E+10	2.9E-02	3.0E-03	2.1E-02	6.0E-03	5.9E-02	1.6E-02
4	Center	4.6E+11	8.2E+10	6.1E-02	6.5E-03	4.5E-02	1.6E-02	1.3E-01	3.4E-02
6	Center	6.8E+11	1.2E+11	9.1E-02	9.7E-03	6.7E-02	5.9E-02	2.3E-01	5.1E-02
8	Center	9.1E+11	1.6E+11	1.2E-01	1.3E-02	9.0E-02	5.8E-02	2.8E-01	6.7E-02

Table 1. Irradiated doses of cells on Nov. 19, 2025 (Rail, irradiation room).

Irradiation time[min]	Position	fluence [cm ²]		[Gy]					
		Thermal neutron [cm ²]	Epi-thermal neutron [Gy]	Thermal neutron [Gy]	Epi-thermal neutron [Gy]	Fast neutron [Gy]	Gamma-ray [Gy]	Physical Dose [Gy]	B-10** (ppm)
60	center	3.9E+12	6.9E+11	5.2E-01	5.5E-02	3.8E-01	3.2E-01	1.3E+00	2.9E-01
	Rear side of the shielding panel 1	6.1E+11	1.1E+11	8.1E-02	8.7E-03	6.0E-02	3.3E-01	4.8E-01	4.5E-02
	Rear side of the shielding panel 2	7.1E+11	1.3E+11	9.4E-02	1.0E-02	7.0E-02	3.3E-01	5.0E-01	5.3E-02
60	center	4.0E+12	7.1E+11	5.3E-01	5.7E-02	4.0E-01	1.8E-01	1.2E+00	3.0E-01
	Rear side of the shielding panel 1	6.6E+11	1.2E+11	8.8E-02	9.4E-03	6.5E-02	2.2E-01	3.8E-01	4.9E-02
	Rear side of the shielding panel 2	9.0E+11	1.6E+11	1.2E-01	1.3E-02	8.9E-02	2.2E-01	4.4E-01	6.7E-02

Table 3. Irradiated doses for local irradiation of mice on January 20, 2026 (Cart, irradiation room).

Irradiation time[min]	Position	fluence [cm ²]		[Gy]					
		Thermal neutron [cm ²]	Epi-thermal neutron [Gy]	Thermal neutron [Gy]	Epi-thermal neutron [Gy]	Fast neutron [Gy]	Gamma-ray [Gy]	Physical Dose [Gy]	B-10** (ppm)
60	center	3.9E+12	6.9E+11	5.2E-01	5.5E-02	3.8E-01	3.2E-01	1.3E+00	2.9E-01
	Rear side of the shielding panel 1	6.1E+11	1.1E+11	8.1E-02	8.7E-03	6.0E-02	3.3E-01	4.8E-01	4.5E-02
	Rear side of the shielding panel 2	7.1E+11	1.3E+11	9.4E-02	1.0E-02	7.0E-02	3.3E-01	5.0E-01	5.3E-02
60	center	4.0E+12	7.1E+11	5.3E-01	5.7E-02	4.0E-01	1.8E-01	1.2E+00	3.0E-01
	Rear side of the shielding panel 1	6.6E+11	1.2E+11	8.8E-02	9.4E-03	6.5E-02	2.2E-01	3.8E-01	4.9E-02
	Rear side of the shielding panel 2	9.0E+11	1.6E+11	1.2E-01	1.3E-02	8.9E-02	2.2E-01	4.4E-01	6.7E-02

Neutron Capture Therapy for Cancer Using Gd-Thiacalixarene Complex-Loaded Nanogel Micelles

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INTRODUCTION: We have been studying the application of multifunctional metal complexes, originally derived from analytical reagent research, for biomedicine. Specifically, focusing on the kinetic stability, MRI contrast capability, and the ¹⁵⁷Gd neutron capture ability of the thiacalixarene complex Gd₃TCAS₂ (Fig. 1a), we have previously developed Gd₃TCAS₂-loaded nanoparticles for MRI-guided neutron capture therapy (NCT). However, for optimal biological applications, nano-medicines with a higher loading capacity (LC) and a smaller particle size are desired. Therefore, in this report, we created nanogel micelles (NM) composed of Gd₃TCAS₂ and the block copolymer poly(ethylene glycol)-*b*-poly(L-lysine) (PEG-*b*-PLL) and evaluated their *in vivo* NCT effects. **EXPERIMENTS: Preparation of NMs.** Ln-NM was prepared by mixing an Ln₃TCAS₂ (Ln: Gd or Tb) solution and a PEG-*b*-PLL solution under high-speed stirring. Subsequently, the excess amino groups of the PLL chains were crosslinked using glutaraldehyde. **RESULTS:** Dynamic light scattering (DLS) measurements revealed the particle size of the NM to be 44.9 ± 0.52 nm, which is expected to facilitate passive transport to tumors and provide high blood retention. The ζ potential was -3.9 ± 3.2 mV, suggesting that the particle surface is effectively coated with PEG chains. Furthermore, the NM achieved a high LC of 24.2 ± 4.1 wt%. When mouse colon cancer Colon-26 cells were cultured in the presence of Ln-NM for 24 hours, cells treated with Tb-NM exhibited Tb-derived green luminescence, confirming successful intracellular uptake. For the Gd-NM, the intracellular Gd uptake was measured at 1.18 ± 0.21 nmol/10⁶ cells. Toxicity tests indicated that cell viability began to decrease at a concentration of [Gd] = 100 μ M. When Colon-26 cells loaded with Gd-NM were irradiated with neutrons, their cell viability significantly decreased compared to the control group, successfully demonstrating an *in vitro* NCT effect.

In vivo studies using a Colon-26 tumor-bearing mouse model with tail vein injection of Gd-NM confirmed the accumulation of Gd in the tumor; the ¹⁵⁷Gd concentration in the tumor reached 4.8 ppm 24 hours post-administration. Upon neutron irradiation, the group treated with Gd-NM showed the most suppressed increase in relative tumor volume compared to the control group, the Gd-NM administered without neutron irradiation group, and the PBS with neutron irradiation group, suggesting a strong anti-tumor effect (Fig. 1b). Additionally, no body weight loss was observed in the mice, confirming the biosafety of the nanogel micelles.

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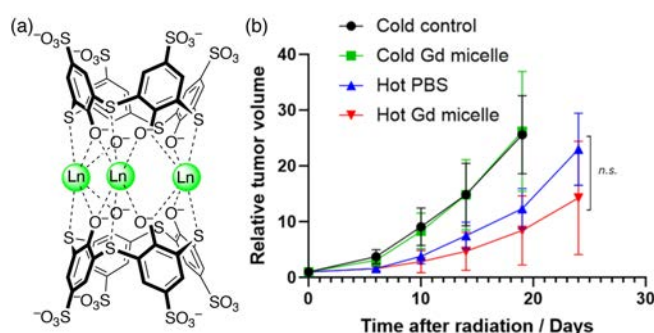


Fig. 1 (a) Ln₃TCAS₂, and (b) changes in relative tumor volume in Colon-26 tumor-bearing mice after neutron irradiation ($n = 6$ for each group).

Drug Discovery of Novel Boron Agents for Tumor Marker-Targeted Precision BNCT

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INTRODUCTION: Drug development is essential for the future advancement of boron neutron capture therapy (BNCT). However, since the regulatory approval of boronophenylalanine (BPA) in 2020, no clinical trials of novel boron agents or studies investigating new combinational strategies for broader indications have been conducted. Although BPA is an excellent boron agent and is preferentially taken up by tumors with high amino acid metabolism, intratumoral heterogeneity is widely recognized across malignant tumors. Therefore, reliance on a single boron agent may limit the future development and broader applicability of BNCT.

The potential value of multi-agent strategies has already been suggested in clinical research on malignant glioma, in which the combination of BPA and borocaptate sodium (BSH), two boron compounds with distinct physicochemical properties, uptake mechanisms, and tumor accumulation profiles, demonstrated greater efficacy than single-agent treatment [1]. In addition, boron agents lacking intrinsic intracellular uptake capability have been reported to achieve improved tumor selectivity and cellular internalization through drug delivery system (DDS)-based approaches, resulting in enhanced therapeutic efficacy. These findings indicate that developing novel boron agents and rational combinational strategies remains both a major challenge and an important research priority in BNCT. Although it is difficult to identify a universal characteristic shared by all malignant tumors, boron agents can be designed according to the biological features of individual cancers and translated from drug discovery into therapeutic application. In particular, tumor-specific molecular features, especially tumor-associated genes characterizing each cancer type, may provide a promising basis for boron agent design. From this perspective, precision BNCT, which integrates gene-guided boron drug discovery with BNCT, holds substantial promise. LAT1, the molecular target responsible for BPA uptake, represents one successful example of this concept. Expanding this strategy to additional tumor markers and molecular targets may further broaden the clinical potential of BNCT.

EXPERIMENTS: Because it is difficult to identify a universal property shared by all malignant tumors, we sought to design boron agents tailored to the biological characteristics of individual cancer types and to advance them from drug discovery to therapeutic application. In this context, tumor-associated genes that characterize each malignancy represent particularly promising targets. The strategy of combining gene-guided boron agent development with BNCT, namely precision BNCT, has substantial future potential [2]. Based on this concept, we conducted a pan-cancer bio-informatics analysis to identify alternative uptake-target genes highly expressed in malignant tumors with low LAT1 expression, since LAT1 is the principal molecular target mediating BPA uptake. Furthermore, we performed drug discovery research focusing on tumor markers secreted by cancer cells, with particular attention to their constituent structures as candidate targets for novel boron agent development.

RESULTS: By focusing on fucose, a structural component of the tumor marker CA19-9, we developed a novel fucose-conjugated boron agent. Uptake analyses in CA19-9-high malignant tumors demonstrated tumor selectivity, and antitumor effects following neutron irradiation were confirmed in both in vitro and in vivo experiments [3].

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Basic Study of Overcoming Immunotherapy Resistance by Boron Neutron Immunotherapy

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INTRODUCTION: Immunotherapy with immune checkpoint inhibitors (ICIs) has become a major component of treatment for advanced cancer. As a therapeutic strategy distinct from conventional cytotoxic chemotherapy, it has demonstrated substantial clinical importance. However, its objective response rate remains limited, generally ranging from only a few percent to approximately 30%, and overcoming low response rates and resistance to immunotherapy is considered a critical challenge for future cancer treatment.

For patients who do not respond to single-agent or dual-agent ICI therapy, combination treatment with radiotherapy has been explored as one potential approach to reactivate antitumor immunity. It has long been recognized that radiotherapy can induce an immune-mediated phenomenon known as the abscopal effect [1]. However, this effect has been extremely rare, and its efficacy has not been demonstrated in prospective clinical studies, with evidence largely limited to case reports.

In the United States, the approval of ipilimumab (Yervoy, anti-CTLA-4) in 2011 marked the beginning of ICI-based immunotherapy, followed by the initiation of clinical trials across various malignancies. In this context, a 2012 report described a patient with advanced melanoma who was re-refractory to immunotherapy and subsequently achieved regression of non-irradiated lesions after the addition of local radiotherapy. This observation raised expectations that the long-recognized but rare abscopal effect of radiotherapy could be more effectively realized when combined with ICI-based immunotherapy [2].

EXPERIMENTS: We used the B16-F10/C57BL/6 model, an advanced melanoma model resistant to both radiotherapy and ICI-based immunotherapy. The antitumor effects at both the neutron-irradiated site and a shielded distant site were evaluated in the following groups: neutron irradiation alone (control group), immunotherapy alone (anti-PD-1 treatment group), BPA-BNCT group, and the combination group of BNCT plus immunotherapy (boron neutron immunotherapy group).

As a result, significant therapeutic effects at the irradiated site were observed in both the BPA-BNCT group and the BNCT plus immunotherapy group. Furthermore, marked tumor suppression at the distant site was observed only in the combination group. This therapeutic effect at the distant site was confirmed to represent an abscopal effect [3].

RESULTS: Using a model resistant to both radiotherapy and immunotherapy, we are conducting mechanistic studies of the antitumor effects of boron neutron immunotherapy, with a particular focus on immune cell dynamics within the tumor microenvironment. We are planning to further develop these findings toward future clinical studies.

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Gamma-Ray Generation from Gadolinium-Containing Inorganic Phosphor Microparticles under Thermal Neutron Irradiation

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INTRODUCTION: Gadolinium-containing rare-earth oxide phosphors with a monodisperse particle size below 1 μm and hollow structures were synthesized through a simple process, and gamma-ray generation from gadolinium under thermal neutron irradiation was investigated. For catalyst- and phosphor-derived materials intended for dosimetric applications, synthesis methods that provide small, uniform particle sizes through a straightforward reaction process are highly desirable. In this study, aiming toward the realization of gadolinium neutron capture therapy (GdNCT) materials, we measured differences in gamma-ray dose as a function of gadolinium doping ratio in spray pyrolysis-synthesized $(\text{Y, Gd})_2\text{O}_3:\text{Eu}^{3+}$ phosphor microparticles.

EXPERIMENTS: Yttrium nitrate n -hydrate, europium (III) acetate pentahydrate, and gadolinium (III) nitrate pentahydrate were used as starting materials and dissolved in distilled water at prescribed concentrations. The prepared aqueous solution was atomized ultrasonically, and the generated droplets were introduced with a carrier gas (air) into a reaction tube placed in an electric furnace maintained at 800°C . Microparticles were obtained by calcination in the high-temperature gas stream. For neutron irradiation, KUR-HWNIF was used as the neutron irradiation field. The gold foil activation method and TLDs (UD-170LS, Panasonic Co.) were used to estimate the gamma-ray doses.

RESULTS: As shown in Fig. 1, the gamma-ray dose increased linearly with increasing gadolinium concentration. On the other hand, commercially available gadolinium reagents generally contain isotopic compositions in which the total fraction of ^{156}Gd , ^{158}Gd , and ^{160}Gd exceeds 65%, whereas ^{157}Gd accounts for only approximately 15%. The present results indicate that, in order to obtain an effective gamma-ray dose, maximizing the neutron capture cross section is essential. Specifically, it is important to synthesize the target material using precursors enriched in ^{157}Gd during the synthesis stage, and furthermore to adopt strategies for highly localized accumulation of the material in the vicinity of malignant tissues.

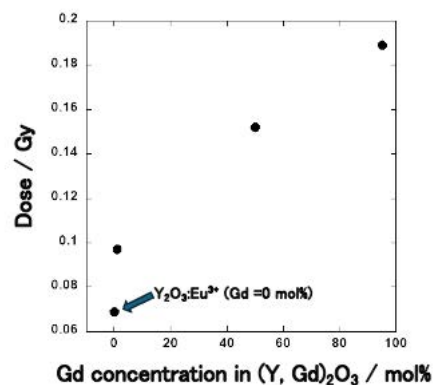


Fig. 1. Dependence of γ -ray dose on gadolinium concentration in $(\text{Y, Gd})_2\text{O}_3:\text{Eu}^{3+}$ (Gd = 1, 50, 95 mol%) and $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$ (Gd = 0 mol%).

Anti-tumor effect of boron containing nano particle for chest BNCT on mouse thoracic tumor model

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INTRODUCTION: Boronophenylalanine (BPA) has been applied to boron-neutron capture therapy (BNCT) for late stage of head and neck squamous cell carcinoma. Although BNCT with BPA is also promising for other types of cancer, the pharmacokinetics need to be improved. Here, we have developed novel 10B containing nano particle, RN-501 [1]. The RN-501 was able to abundantly accumulate into a tumor compared with BPA even by just a single injection, whereas the concentration in normal tissue including lung and blood was lower. It means that RN-501 can be eased a patient's burden at BNCT. In this study, we assessed tumor growth inhibition of BNCT with RN-501 using mouse thoracic tumor model to prove the efficacy of thoracic cancer treatment.

EXPERIMENTS: Mouse colon cancer cell line CT26-Luc, which is luciferase expressing CT26 cells were intra-thoracically injected into BALB/c mice. Then several concentrations of RN-501 or 500 mg/kg of BPA were respectively injected into eight weeks old BALB/c mice intravenously or subcutaneously. The mice were irradiated with 5×10^{12} n/cm² of thermal neutron that was generated from KUR with 5MW output energy for 12 min. Saline, BPA, RN501, and thermal neutron only were respectively used for control. The activity of intra-thoracic tumors was measured for 14 days after the irradiation with In Vivo Imaging System (IVIS). The mouse survival for each treatment was also simultaneously measured.

RESULTS: Now, the results are analyzing and discussed for further study.

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The Efficacy of Boron Neutron Capture Therapy for Local Recurrence and Lymph Node Metastasis in Breast Cancer

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INTRODUCTION: We have previously conducted extensive fundamental research verifying the efficacy of Boron Neutron Capture Therapy (BNCT) across various intractable cancer models [1, 2]. While BNCT offers high tumor selectivity, the development of novel boron delivery agents remains a crucial breakthrough required to overcome the limitations of current clinical compounds. This pharmacological evolution is essential for achieving the precision necessary to treat aggressive or recurrent malignancies [3]. Building upon these findings, the present study evaluates BNCT for refractory breast cancer—the leading cause of cancer-related death among women globally. For elderly patients or those with impaired ADL, invasive interventions are often challenging. Furthermore, local recurrence and supraclavicular lymph node metastasis present critical hurdles, as prior radiotherapy often precludes safe re-irradiation, and vascular invasion can render tumors surgically unresectable. BNCT, which spares adjacent normal tissues like the airway and esophagus, represents a promising alternative for these pre-treated, refractory cases.

EXPERIMENTS: In the in vitro phase, we utilized MCF-7 and paclitaxel-resistant (MCF-7/PTXR) human breast cancer cell lines. Cells were treated with BPA (10 µg B/mL) for 24 hours and subjected to neutron irradiation at 1 MW (0–30 min). Cell viability was quantified via a colony formation assay. The in vivo phase involved female BALB/c nude mice bearing subcutaneous MCF-7 tumors. Mice were randomly assigned into six groups (n = 4 each): (1) untreated control, (2) neutron irradiation alone, and four BNCT groups receiving different boron agents via tail vein: (3) standard BPA, (4) hyaluronic acid-labeled BPA (HA-BPA), (5) PBC-IP, and (6) high-concentration ionic liquid. Neutron irradiation (5 MW, 20 min) was performed 3 hours post-administration, with relative tumor volume monitored for 3 months.

RESULTS: The colony formation assay revealed that BNCT significantly reduced survival fractions in both MCF-7 and drug-resistant MCF-7/PTXR lines in a dose-dependent manner. The comparable sensitivity of the PTXR line indicates that BNCT effectively bypasses conventional chemoresistance mechanisms. In vivo, all BNCT groups exhibited marked tumor growth inhibition. Specifically, HA-BPA demonstrated superior efficacy in reducing tumor volume relative to standard BPA, likely due to enhanced accumulation mediated by the tumor microenvironment. These preliminary results highlight BNCT, supported by novel delivery agents, as a potent, minimally invasive intervention for refractory breast cancer, warranting continued investigation.

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Detection and assessment of off-center nuclear material using the active rotation method

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INTRODUCTION: We have developed a new nuclear material detection method, called the active rotation method, using a neutron rotation machine that rotates a neutron source (Californium-252) at a speed of thousands of rpm. In this method, a water Cherenkov neutron detector (WCND) has been used for a neutron detector. This system is low-cost and transportable. Previous studies have demonstrated the effectiveness of this method, such as in the detection of gamma-ray shielded highly enriched uranium (HEU). However, in previous research, the measurement object was placed at the center position, defined as being aligned on the axis between the center of the rotation device and the neutron detector. In this study, we attempted to detect nuclear material in off-center locations and investigated a method to determine the presence or absence of such displacement.

EXPERIMENTS: The rotation machine can rotate the disk (32 cm in diameter), where a neutron source is installed at its outer periphery, at a rotation speed between 0 and 4000 rpm. A neutron source of Californium-252 whose activity was 1.0 MBq was set in the disk. The size of the WCND is 30x25x30 cm, with four 2-inch PMTs mounted on the ceiling. The WCND was covered with boron-rubber to shield against thermal neutrons. Furthermore, 5-cm-thick lead blocks were placed in front of the WCND to shield gamma-ray generated from water and polyethylene. Between the lead blocks and the rotation machine, polyethylene blocks of 40x25x30 cm were positioned, which contained a 20x20x20 cm internal cavity for placing the measurement object. As for the measurement object, approximately 1 kg of low-enriched uranium (LEU) with an enrichment of about 20 % was used. The rotation speeds were 600 rpm for low speed and 4000 rpm for high speed. In the active neutron method, the presence or absence of nuclear material is determined using an evaluation-value derived from the ratio of integrated counts around the peak at both low and high rotation speeds. Each measurement took 20 minutes, since this method requires both low and high speeds, the total measurement time per case was 40 minutes.

RESULTS: Figure 1 shows an example of the measurement result at 4000 rpm with nuclear material placed at the center position; a distinct peak is observed around 6500 μ s. In this case, the result successfully indicated the presence of nuclear material. While nuclear material could also be detected when displaced by 7.5 cm from the center, the evaluation-value decreased. Since the evaluation-value also changes depending on the mass of nuclear material, this value cannot indicate such misalignment. Therefore, in this study, we investigated a new evaluation approach using Fast Fourier Transform (FFT) analysis. Preliminary analysis found that this approach has potential to demonstrate the off-center distribution of nuclear material.

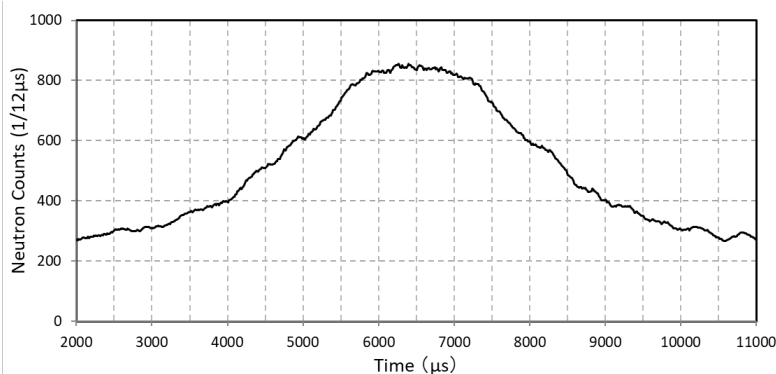


Fig. 1. Neutron count distribution measured at 4000 rpm with nuclear material

***In situ* visualization of the neutron-sensitizing reagent, *p*-borono-L-phenylalanine (BPA), absorbed in green wood cutting of red wine grape by a boron neutron capture reaction.**

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INTRODUCTION: Vegetative propagation is a form of asexual reproduction that produces new individuals from vegetative organs such as roots, stems, or branches. Unlike seed propagation, it ensures the inheritance of superior parental traits without genetic variation, making it indispensable for stable, uniform-quality crop production, particularly in polyploid fruit trees such as strawberries, bananas, and apples. However, crossbreeding remains the primary method of genetic improvement for these fruit trees, in which seed propagation is difficult, posing a challenge to the rapid development of new cultivars capable of overcoming climate change. We have been investigating the application of boron-neutron capture (BNC) reactions for mutation breeding in rice and confirmed that *p*-borono-L-phenylalanine (BPA), a neutron sensitizer, translocates to rice embryos [1, 2]. Subsequently, we successfully obtained various rice mutants exhibiting phenotypes such as dwarfism. These results suggest that BNC-mediated mutagenesis is an effective approach for cultivating seed-propagated crops. However, its applicability to vegetatively propagated plants has not yet been explored. If effective, it could provide a high-efficiency mutation breeding method for such species. Therefore, this study aimed to investigate whether green wood cuttings of the red wine grape Merlot can absorb BPA and whether the sensitizer accumulates in the emerging bud, which is essential for inducing mutations in vegetatively propagated plants.

EXPERIMENTS: *In situ* visualization of BPA in green wood cuttings of red wine grapes> Green wood cuttings of red wine grapes, Merlot (*Vitis vinifera* L. cv. Merlot) were immersed in BPA solution. (500 ppm) for 24 hrs. Slices (10-μm thickness) of an emerging bud of the green wood cuttings were mounted onto a solid-state nuclear tracking detector, CR-39 (20 mm×30 mm), and irradiated with epithermal neutrons for 20 min by applying them to the pneumatic tube in the graphite thermal column (Tc-Pn) of KUR. The irradiated CR-39 plate was etched in 6 M NaOH solution for 60 min at 70°C and was observed under an optical microscope.

RESULTS: Both Fig. 1(A) and 1(B) show close-up images of cross-sections prepared from the same emerging bud of a green wood cutting of red wine grape. Fig. 1(A) and 1(B) show a bright-field image and an α-tracking autoradiograph generated by the BNC reaction, respectively. As shown in Fig. 1(B), many etch pits derived from BPA were imaged throughout the section as small black dots that accumulated in the central part of the emerging bud.

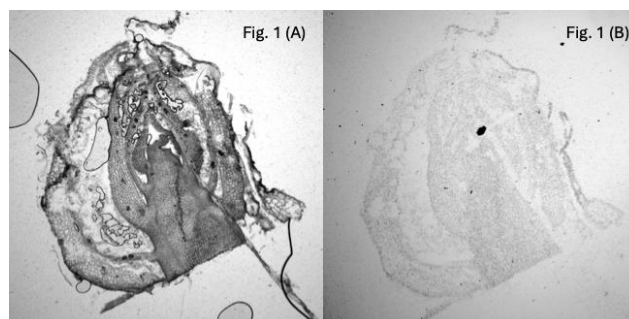


Fig. 1. Detection of BPA absorbed into the green wood cutting of red wine grapes.

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In-situ Porosity Distribution Evaluation of Additively Manufactured Workpiece During Tensile Test Using Neutron Phase Imaging

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INTRODUCTION: Inspection for voids is necessary in metal additive manufacturing (AM) because significant variations in the internal void distribution can occur, potentially affecting quality aspects such as mechanical properties. Previous research has suggested that phase imaging using neutron beams may enable identification of relative differences in void fraction between samples. Therefore, in this report, we investigate the changes in porosity and pore size under tensile loading.

EXPERIMENTS: The experimental setup is shown in Fig.1. Using 1-mm-thick plate-shaped test specimens fabricated by the Powder Bed Fusion (PBF) method, the porosity and pore size under tensile loading were measured using neutron phase imaging. Porosity and pore size were measured under four conditions: no load, elastic deformation, yield point, and maximum load. In-situ observations were conducted while maintaining tensile loads corresponding to each condition, specifically 0, 1500, 3000, and 3500 N.

RESULTS: Figure 2 shows the spatial distribution of porosity measured using neutron phase imaging. As shown in Fig. 2, a trend was observed throughout the evaluation region in which porosity increased with increasing tensile load. In particular, as shown in Fig. 2(c) and (d), it can be seen that porosity increases significantly from the point at which the tensile load reaches the yield point up to the maximum load. Furthermore, the porosity is higher near $X = 2.0$ [mm] and $Y = 2.5$ [mm] than in other regions. Thus, this result confirmed local variations in porosity within the evaluation region.

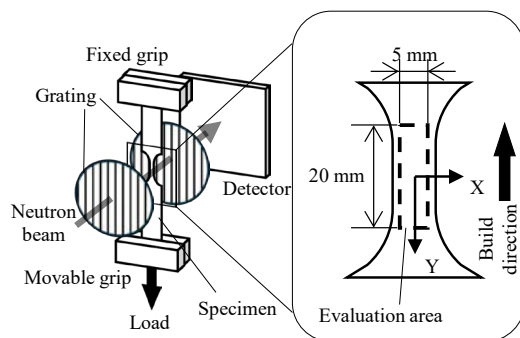


Fig.1 Experimental setup [1]

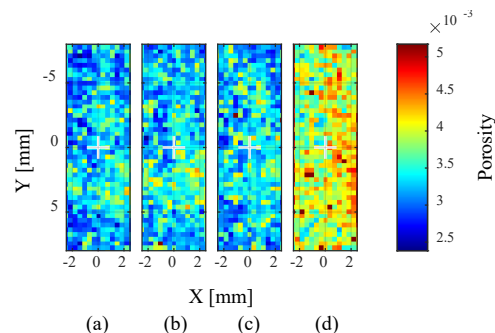


Fig.2 Porosity distribution under loads:
(a) 0 N, (b) 1500 N, (c) 3000 N, (d) 3500 N [1]

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Elucidation of the mechanism of degradation of the tumor suppressor p53, which is dependent on human papillomavirus protein E6.

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INTRODUCTION: It is known that E6AP (E6-associated protein), a kind of ubiquitin ligase, interacts with E6 protein derived from oncogenic human papilloma virus (HPV 16/18) and the enzyme complex induces ubiquitination of p53 resulting p53 degradation by ubiquitin-proteasome system. To understand the mechanism of p53 ubiquitination by E6AP and E6, we use HS-AFM to observe the structural dynamics of the E6AP/E6 complex. In addition to HS-AFM observation, we analyze the structural dynamics of the E6AP/E6/p53 complex using small-angle X-ray scattering (SAXS) methods.

EXPERIMENTS : SAXS and ultracentrifugation analysis of E6AP in FY2024 revealed that purified E6AP was contaminated with partially degraded E6AP, which interfered with the SAXS measurement of full-length E6AP. Therefore, in FY2025, we attempted to improve the method for preparing highly purified full-length E6AP. E6AP had previously been expressed as a recombinant protein in *E. coli* and then purified, but the expression host was changed to animal cells. An extracellular secretion signal was added to the E6AP gene and incorporated into the expression vector pcDNA3.4. This plasmid vector was then transfected into HEK293T cells to induce transient expression of the E6AP protein. E6AP expression and secretion into the culture medium were verified by Western blotting.

RESULTS : Confirmation of E6AP protein expression in the culture medium revealed that the amount of E6AP protein in the medium was extremely small. Therefore, intracellular expression was investigated instead of secretion into the culture medium. The confirmation of E6AP protein expression within cells suggests that secretion into the culture medium via a secretory signal was not very effective. The secretory signal into the culture medium was added to facilitate protein purification, but since intracellular expression is more prevalent, the possibility of purifying the E6AP protein after cell disruption is also considered. However, Western blot analysis revealed that even in animal intracellular expression, a large amount of partially degraded E6AP exists, similar to the expression in *E. coli*. Therefore, in order to purify the full-length E6AP protein, we plan to create a plasmid vector in which a FLAG tag is introduced to the C-terminus of E6AP, and prepare full-length E6AP by a two-step purification using the His-tag at the N-terminus (originally added for purification) and the newly introduced FLAG tag at the C-terminus. Currently, we are working on preparing a plasmid vector in which a FLAG tag is introduced to the C-terminus.

Evaluation of Porosity Distribution in Metal Additive Manufacturing Parts Using Neutron Phase Imaging

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INTRODUCTION: Inspection for voids is necessary in metal additive manufacturing (AM) because significant variations in the internal void distribution can occur, potentially affecting quality aspects such as mechanical properties. However, conventional inspection methods such as polished cross-section observation and X-ray CT cannot adequately inspect voids within the product itself due to limitations such as the destructive nature of the process and poor penetration. Consequently, neutron phase imaging—which is non-destructive and offers high penetration—is attracting attention. To evaluate the effectiveness of neutron phase imaging in three-dimensional distribution evaluation of voids, the correspondence between scattering image and cross-sectional image was investigated.

EXPERIMENTS: The porosity of samples was measured by the neutron phase imaging [1]. The porosity was also measured from the cross-sectional image of samples taken by a digital microscope. Samples were cut, embedded into plastic pellets, and polished for observation. The cross-sectional image was binarized to extract the pore area.

RESULTS: Figure 1 shows the correspondence between scattering image and cross-sectional image. By comparing Figs.1(a) and (b), two groups of voids observed in the cross-sectional image are also observed in the scattering images. The positional correspondence of those voids seems reasonable. In principle, neutron phase imaging should not have the sensitivity to detect such large voids. However, it is estimated that such a correlation can be observed due to scattering caused by microstructures on the surface or edges of large voids. In other words, neutron phase imaging may be capable of detecting voids ranging from the microscale to the millimeter scale.

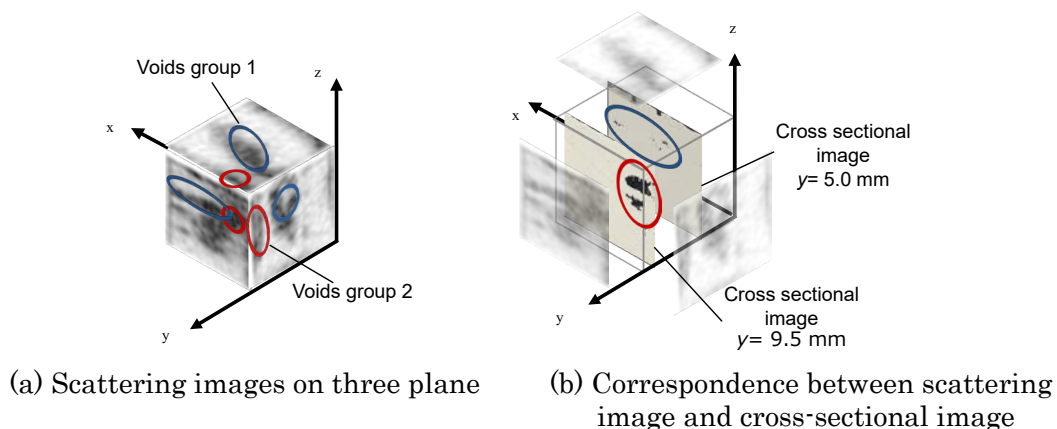


Fig.1 Estimation of three-dimensional correspondence between scattering image and cross-sectional image

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Characterization of Zr(IV)-Ce(IV) coprecipitated hydroxide solid phases

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INTRODUCTION: In safety assessments for the geological disposal of high-level radioactive waste, solubility-limiting solid phase under a relevant groundwater condition is selected for each radionuclide. In the case of radionuclides that behave as tetravalent metal ions, their respective hydroxides are regarded as the solubility-limited solid phases [1]. On the other hand, in actual disposal environments, various elements not only radioactive but also non-radioactive leached from the waste form to coexist in groundwater. Therefore, when tetravalent metal ions precipitate as hydroxides, there is a possibility of forming co-precipitated solid phases with different elements. This study focuses on the state of the coprecipitated solid phase composed of cerium as a lanthanide and zirconium as an analog of a tetravalent actinide. The mother solutions are mixed with given molar ratios, and the coprecipitated hydroxide solid phase is generated under different temperature and pH conditions. The structure of the coprecipitated solid phase is investigated using X-ray diffraction.

EXPERIMENTS: The coprecipitated hydroxide solid phase was prepared by an oversaturation method. Acidic Ce(III) hydrochloride ([Ce] = 0.6 M) solution and acidic Zr(IV) hydrochloride ([Zr] = 0.6 M) solution were first mixed in given molar ratio ([Zr]/[Ce]=10/0, 7/3, 5/5, 3/7, and 0/10). After adjusting the ionic strength (*I*) to *I* = 0.1 with NaClO₄, small aliquots of 8 M NaOH was added to adjust the pH to 8 or 12 to form coprecipitated hydroxide solid phases. The sample solution was then aged at 25 or 70 °C for one month and the pH and Eh were measured. A portion of the coprecipitated solid phase was separated by centrifugal sedimentation, vacuum dried, and the diffraction pattern was measured using a powder X-ray diffractometer (MiniFlex600, RIGAKU).

RESULTS: In the case of [Zr]/[Ce]=10/0, no significant XRD peaks were observed except at pH=12 and 70°C, indicating that the solid phase was amorphous. On the other hand, at pH=12 and 70°C, peaks corresponding to ZrO₂(monoclinic) and ZrO₂(tetragonal) were observed. In the case of [Zr]/[Ce]= 0/10, although Ce(III) was used as an initial solution, but no diffraction peaks of Ce₂O₃ or Ce(OH)₃ were observed and only CeO₂(cubic) was observed. Considering the observed pH and Eh values, it was considered that Ce(III) was oxidized to Ce(IV) during the aging period at pH 8 and 12. In the diffraction patterns of the coprecipitated hydroxide solid phases ([Zr]/[Ce]=7/3, 5/5, 3/7), peaks corresponding to CeO₂(cubic) and ZrO₂(tetragonal) were observed. Both peaks became sharper at higher temperatures after the aging periods, indicating a temperature dependence of crystallization. Furthermore, since there was almost no peak shift in the CeO₂(cubic) peak due to changes in the mixing ratio with Zr, it was concluded that Zr was hardly dissolved in CeO₂(cubic). In this experiment, a Ce(III) hydrochloride was used as the initial solution. As a result, Zr(IV) may not easily enter the Ce(OH)₃ or Ce₂O₃ initially formed by the rapid addition of NaOH, and the Ce(III) solid phase may have subsequently been oxidized to CeO₂(cr).

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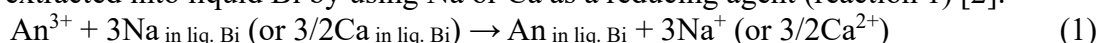
Chemical extraction of Uranium from NaCl-UCl₃ melt

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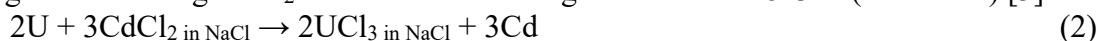
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INTRODUCTION: Chloride salts have been attracting widespread attention as a fuel of molten salt fast reactor [1]. It is necessary to develop a process to recycle the used chloride fuel salt for the effective utilization of the nuclear fuel resources. Therefore, pyrochemical reprocessing process has been proposed for recycling the used salt with NaCl-CaCl₂ base salt, where reductive extraction of actinides (An) from the used salt is one of the main steps: An in the used salt are chemically reduced and extracted into liquid Bi by using Na or Ca as a reducing agent (reaction 1) [2].



In this study, to experimentally investigate the preliminary feasibility for applying the pyrochemical reprocessing process to the used salt with NaCl base salt, reductive extraction test of U from NaCl-UCl₃ salt was performed.

EXPERIMENTS: The reductive extraction test was carried out in a glove box filled with purified Ar gas. The dark brown NaCl (66 mol%)-UCl₃ (34 mol%) salt (m.p. 796 K) had been prepared by chlorinating U metal using CdCl₂ as the chlorination agent in NaCl at 823 K (reaction 2) [3].



The prepared NaCl-UCl₃ (1.642 g) was melted at 821 K in alumina crucible. To proceed with the reductive extraction reaction (reaction 3), Bi-0.4wt%Na alloy and Bi metal were added to the melt in three parts.



The total amounts of the added Bi-0.4wt%Na alloy and Bi metal was 19.379 g and 17.611 g, respectively. According to phase diagram of NaCl-UCl₃ system [4], solid NaCl would be deposited when the concentration of UCl₃ in the melt decreases below around 30 mol% along with the progress of the reductive extraction reaction (reaction 3). To collect the deposited NaCl, MgO tube (6 mm in diameter) was inserted in the melt during the experiment, since NaCl is expected to be deposited preferentially on the surface of MgO of which crystal structure is same as that of NaCl.

RESULTS: After the reductive extraction experiment, MgO tube was removed from the melt. It was found that the surface of the MgO tube was covered by solid deposits together with small amount of brown NaCl-UCl₃ adhered salt (Fig. 1). The solid deposits were considered to be NaCl. The deposits will be analyzed by SEM-EDX in the future. The obtained results might suggest the possibility to construct a simple pyrochemical reprocessing process: Actinides would be recovered together with the NaCl base salt at the reductive extraction step, leaving fission products in the used salt. Then, the recovered actinides could be chlorinated in the recovered NaCl base salt to produce the recycled fuel salt.

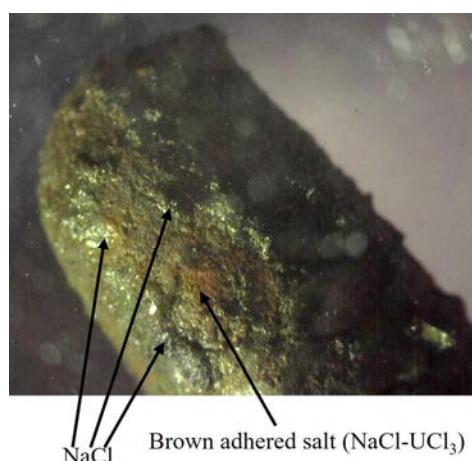


Fig. 1. Photograph of the surface of MgO tube after reductive extraction of U from NaCl-UCl₃ melt at 821 K.

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Coprecipitation of Ca and Sr with europium oxalate for chemical research on element 102, nobelium

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INTRODUCTION: Element 102, nobelium (No), is an actinide element. Nobelium preferentially exists as the +2 oxidation state in aqueous solutions [1] and exhibits similar chemical behavior to that of alkaline-earth metal ions, unlike the other actinides. Furthermore, it was reported that chemical properties of No were similar to those of Sr [2,3]. On the other hand, in our previous research, the coprecipitation behavior of nobelium was observed to be closer to that of Ca[4]; while the No^{2+} ions were almost quantitatively coprecipitated with calcium oxalate (~100%), Sr^{2+} ions showed about 40% co-precipitation yield under the same conditions. It was difficult to compare the differences of relative yields between Ca and No because both Ca and No (co-)precipitated with almost 100% yield. Therefore, other carrier elements are required to compare relative yields of Ca and No. In this study, we aim to compare the behaviors of No with that of Ca and Sr in oxalate coprecipitation systems. To achieve this, coprecipitation experiments of No must be carried out under conditions where the coprecipitation yield of Ca is not quantitative. For this purpose, a different carrier element was used instead of Ca. We selected europium (Eu) as the new carrier element and conducted the coprecipitation experiments of Ca and Sr with europium oxalate towards the future coprecipitation experiment of nobelium.

EXPERIMENTS: ^{85}Sr tracer was produced in the $^{\text{nat}}\text{Rb}(\text{d},\text{xn})^{85}\text{Sr}$ reaction at the RIKEN AVF cyclotron and chemically separated from the target material to ensure its carrier-free state. In addition, ^{47}Ca was produced by thermal neutron irradiation of a $^{\text{nat}}\text{CaO}$ target at 5 MW for 1 h using a pneumatic irradiation system of Kyoto University Research Reactor, and it contained macro $^{\text{nat}}\text{Ca}$ as a carrier. Oxalate (4–200 mM) and europium nitrate (2 mM) solutions were used, and pH was adjusted to 1.5 with nitric acid and sodium hydroxide solution. Then, 5 mL of each solution was mixed at room temperature with 10 μL of a ^{47}Ca or ^{85}Sr tracer solution in 10^{-2} M nitric acid and stirred for 3 min. The precipitates were subsequently collected by suction filtration, and the radioactivity of ^{47}Ca or ^{85}Sr in the precipitates was quantified using a Ge semiconductor detector.

RESULTS: The coprecipitation yield of ^{85}Sr increased monotonically from $10.9 \pm 1.8\%$ to $90.1 \pm 3.1\%$ in the oxalate concentration range of 2–50 mM and did not reach either 0% or 100%. These yields are suitable for direct comparison of the coprecipitation yield of nobelium. In addition, the Sr yields sensitivity to oxalate concentration suggests that the coprecipitation behavior of Sr involves complex formation. In the coprecipitation experiments for Ca, the coprecipitation yield improved with increasing oxalate concentration above 25 mM; however, the yield was lower than that of Sr. This behavior is different from that predicted based on calcium oxalate precipitation. This may suggest that high concentration of Ca ions in the non-carrier-free tracer solution influences the coprecipitation behavior of Ca with europium oxalate.

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Investigation of Group 1 Element Adsorption on $\text{K}_2\text{Zn}_3[\text{Fe}(\text{CN})_6]_2$ Toward the Chemical Characterization of Francium

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INTRODUCTION: Francium (Fr), the element with the highest atomic number among the Group 1 elements, is theoretically predicted to exhibit ionization potentials and electron affinities that deviate from the trends observed in its lighter congeners due to significant relativistic effects [1–3]. However, as Fr has no stable isotopes and is subject to substantial experimental constraints, direct empirical data supporting these theoretical calculations are extremely limited. The objective of this study was to establish an experimental methodology to quantitatively evaluate the chemical properties of Fr and the influence of relativistic effects. Specifically, focusing on the monovalent cation adsorption properties of complex salts composed of ferrocyanide ions and transition metal ions, we developed a method to estimate the standard Gibbs energy (ΔG°) for the adsorption reactions of Rb^+ and Cs^+ onto $\text{K}_2\text{Zn}_3[\text{Fe}(\text{CN})_6]_2$. Furthermore, we evaluated the feasibility of applying this method to experiments involving the short-lived Fr.

EXPERIMENTS: For the experiments using ^{86}Rb , a hydrochloric acid solution of Rb_2CO_3 containing ^{86}Rb produced via neutron irradiation at the Pn-2 facility of the KUR was employed. For ^{137}Cs , a commercially available ^{137}Cs solution was used. First, $\text{K}_2\text{Zn}_3[\text{Fe}(\text{CN})_6]_2$ was synthesized following a previously reported method [4] and immersed in a KCl solution to saturate the interstitial sites with K^+ ions. This solid phase was then mixed and shaken with solutions containing $^{86}\text{Rb}^+$ or $^{137}\text{Cs}^+$, followed by solid-liquid separation via vacuum filtration. The radioactivity of ^{86}Rb or ^{137}Cs in each phase was measured using a Ge detector to calculate the adsorption rate. In this study, we first investigated the time required to reach adsorption equilibrium and subsequently conducted adsorption experiments to calculate ΔG° . Additionally, assuming Fr experiments where handling at tracer levels (ultra-low concentrations) is mandatory, we examined the concentration dependence of ΔG° in the low-concentration region using Cs^+ .

RESULTS: It was confirmed that the adsorption of Rb^+ and Cs^+ reached equilibrium within approximately 3 minutes. This indicates that the current system is sufficiently applicable to experiments targeting Fr isotopes with half-lives ranging from several minutes to several tens of minutes (e.g., ^{223}Fr). The maximum adsorption capacity per unit mass of $\text{K}_2\text{Zn}_3[\text{Fe}(\text{CN})_6]_2$ was determined using a Langmuir plot, and subsequently, the equilibrium constants and standard Gibbs energies were estimated using Kielland plots. As a result, the ΔG° values for Rb^+ and Cs^+ were found to be $-4.51 \pm 0.42 \text{ kJ/mol}$ and $-10.98 \pm 1.15 \text{ kJ/mol}$, respectively, which are consistent with the observed adsorption rate trends.

On the other hand, the ΔG° obtained from the Cs^+ experiments in the low-concentration region was $-2.2 \pm 0.4 \text{ kJ/mol}$, showing a positive deviation compared to the values estimated from high-concentration data. This discrepancy is likely due to the increased relative contribution of adsorption site heterogeneity and physical adsorption, in addition to the chemical ion-exchange reaction, in the low-concentration regime. Moving forward, we plan to optimize the experimental conditions to minimize the contribution of physical adsorption, aiming for a smooth transition to Fr experiments.

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Effective Measures on Safety, Security, Hygiene and Disaster Prevention in Laboratories

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INTRODUCTION: Important aspects of the study can be found in the following keywords, such as safety, security, hygiene and disaster prevention. Nuclear research reactors are one of the representative facilities together with these keywords under their operation. It is effective to investigate the latest status on practical measures on these keywords in various facilities including nuclear research reactors, to compare each other among facilities, and to discuss more optimized ones for our positive safety management. Through this process, it is also essential to investigate the latest international and/or national regulations and the movement of revision of them. In addition, the development of human resources and public literacy on nuclear science and technology is also within the scope of the research. The total discussion contents and their fruits are directly useful for all relating laboratories.

RESEARCH APPROACH: General research approach is as follows; - Measures of safety management during operation or standstill status of the real facilities would be investigated. This information would be used for our research discussion on positive and more optimized safety management. - It would not be a single year research, but maybe two to three years of research for one theme. - Information source of facilities would not be only KUR, KUCA or the other facilities in Kyoto University, but also the Kindai university research nuclear reactor or the facility of National Institute of Fusion Science, etc. This research is active joint-research with these related facilities and positive researchers on safety management. - One of the distinctive features of this research is to involve office staff as cooperators as well as re-searchers and technical staff. In The University of Tokyo, most of the members in Division for Environment, Health and Safety are office staff who know the real situation of safety management in laboratories very well.

Concrete discussion targets in FY of 2025 were determined as (1) 'Optimized safety management of X-rays following revised the national regulation', and (2) 'General understandings of the national guide on the radiation protection of NORM (naturally occurring radioactive materials)'. These were the representative hottest topics in the field of radiation protection and safety.

RESULTS: <X-RAYS SAFETY MANAGEMENT > To understand and apply the Japanese regulation on X-rays safety control revised in October 2025, the contents were in detailed discussed from the viewpoint of real-site control. Future issues to be overcome listed out, and they were presented and shared with experts in an academic meeting. <NORM RP> The latest Japanese activity and movement on NORM problems was discussed based on the new national guide published in November 2025, and future issues to be discussed were listed out. This topic will be one of the thema in 2026.

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Evaluation of Air Dose Rate Distribution Around Monitoring Post Using KURAMA-II

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INTRODUCTION: The Nuclear Regulation Authority has commissioned a nationwide survey of environmental radioactivity levels, including air dose-rate measurements from approximately 80 portable monitoring posts in Fukushima Prefecture. Among these measurements, the air dose rate at the Omaru Community Center (Namie Town) decreased from approximately 4.5 $\mu\text{Gy/h}$ to approximately 1.2 $\mu\text{Gy/h}$ due to decontamination work around the monitoring post from April to May 2025. The data from the monitoring posts represent typical values for their installation sites and do not provide a detailed picture of the air dose rate distribution in the surrounding area. Therefore, we conduct a walking survey around the monitoring posts using the carborne survey system KURAMA-II (Kyoto University Radiation Mapping System) [1] to investigate the distribution of detailed air dose rate following decontamination.

EXPERIMENTS: We conducted a walking survey near the monitoring post installed at the Omaru Community Center, carrying the KURAMA-II on our backs at a height of 1 m above the ground. Using the standard functions of Kyoto University's cloud server, each measurement point is plotted as a dot on a map, with color indicating the air dose rate.

RESULTS: The air dose-rate map shown in Fig. 1 is divided into the following three areas. Area①(blue box): The air dose rate was approximately 1.5 $\mu\text{Sv/h}$, comparable to measurements at the monitoring post. Area②(yellow box): The air dose rate ranged from approximately 1.5 $\mu\text{Sv/h}$ to 5 $\mu\text{Sv/h}$, from levels comparable to those at the monitoring post to several times higher. This is most likely due to the residual contamination resulting from the incomplete decontamination. Area③(red box): The air dose rate ranged from approximately 2.5 $\mu\text{Sv/h}$ to 5 $\mu\text{Sv/h}$, higher than at the monitoring post. Hotspots at 5 $\mu\text{Sv/h}$ or higher were confirmed in Areas② and ③, while no locations with locally high air dose rate (hotspots) were identified in Area①. Furthermore, in Area③, the air dose rate tended to increase as the distance from Area① increased, suggesting that decontamination has not been performed throughout Area③. Based on these results, it is believed that the distribution of air dose rates in areas away from the monitoring posts is due to the presence of locations where decontamination has not been fully carried out or has not been performed at all.

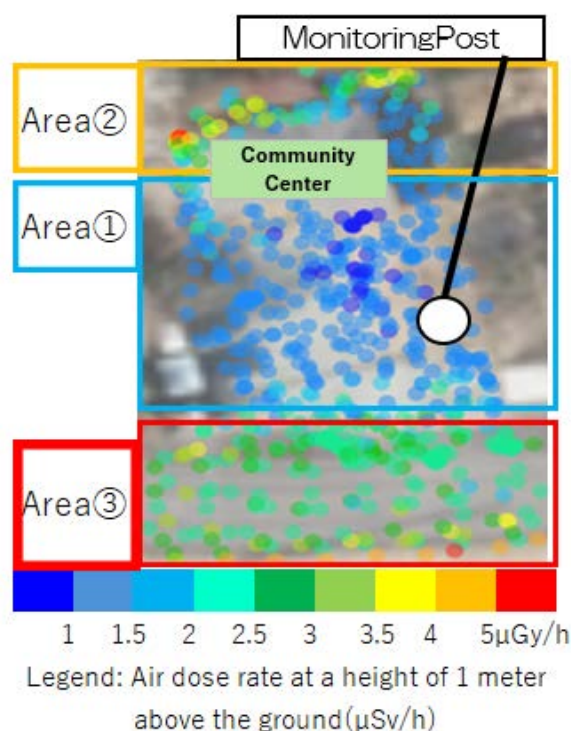


Figure 1. Map of ambient radiation dose rates after decontamination

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Determination of Chlorine and Bromine Concentration in Plastics in Construction Waste by Neutron Activation Analysis

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INTRODUCTION: In the recycling of waste plastic contained in construction waste (construction waste plastic), there are restrictions on chlorine concentration. However, the elemental composition varies from item to item, and this needs to be clarified. In this study, neutron activation analysis (NAA) was performed to analyze chlorine and bromine concentrations in 40 samples, including new plastic products and waste plastic samples provided by intermediate treatment facilities.

EXPERIMENTS: NAA was performed four times for each sample. Samples were irradiated for 3 min with a thermal neutron flux of $2.0\text{--}2.4 \times 10^{13} \text{ cm}^{-2} \cdot \text{sec}^{-1}$ at KURRI. ^{38}Cl ($t^{1/2} = 37.18 \text{ min}$, $E_\gamma = 1642, 2168 \text{ keV}$) and ^{80}Br ($t^{1/2} = 17.68 \text{ min}$, $E_\gamma = 616, 666 \text{ keV}$) were measured by using a Ge semiconductor detector for 300 sec.

RESULTS: Figure 1 shows the typical elemental composition of 40 samples of construction waste plastic obtained through NAA and various measurements. Chlorine and bromine are represented in blue and purple, respectively. Among the 22 building material items, high concentrations of chlorine were detected in 13 items, identifying them as PVC products. In contrast, among the 18 packaging and protection items, high chlorine concentrations were detected in only three items: PVC tape, flame-retardant sheets, and hoses. Since these specific items are easily distinguishable by visual inspection, these results suggest that packaging and protection waste has high recycling potential if these items are properly segregated.

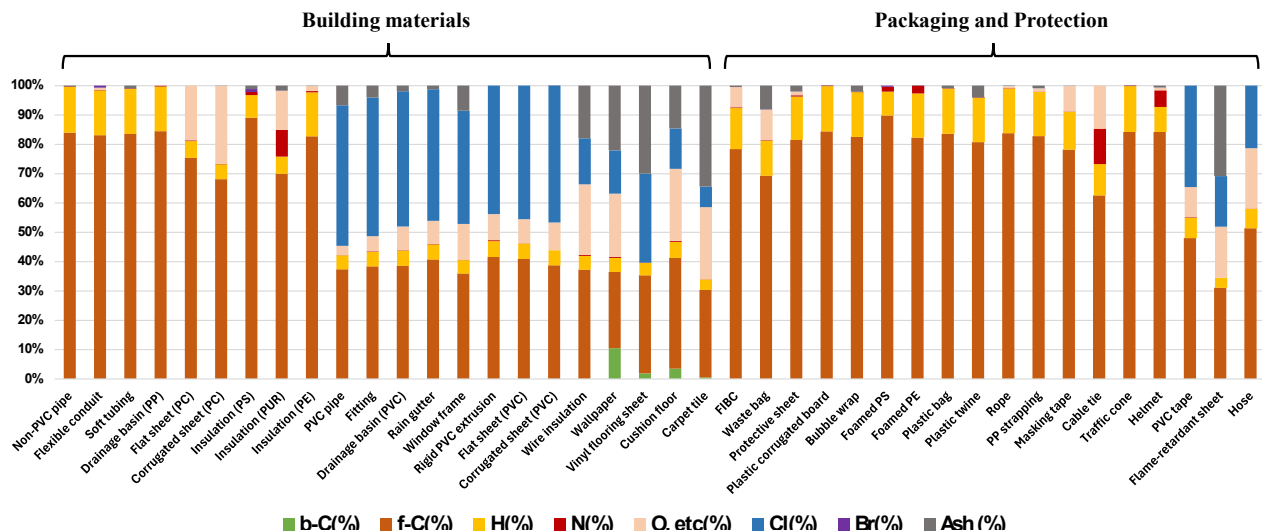


Fig.1. Histograms of chlorine concentrations

Among the building materials, soft PVC products (such as wire insulation, wallpaper, and cushion floor) showed lower chlorine concentrations compared to other PVC products. This is attributed to the high content of additives, such as plasticizers, in addition to the PVC resin itself.

Regarding bromine, concentrations of 0.3–1.0% were detected in only three items: flexible conduits, insulation (PS), and foamed PS. This is considered to be derived from additives used to impart flame retardancy.

Survey of air dose rate distribution around monitoring posts using KURAMA-II

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INTRODUCTION: In Fukushima Prefecture, monitoring posts (MPs) have been installed to monitor the nuclear power plant, and continuous measurements of air dose rates and other parameters are being conducted. Because radioactive nuclides released by the accident at the Fukushima Daiichi Nuclear Power Plant are present in the environment, the air dose rate measured at the MPs may fluctuate not only due to new releases from the plant but also due to environmental changes caused by construction work and other activities around the MPs. Therefore, if there is a significant fluctuation in the air dose rate measured by a particular MP, measuring the air dose rate distribution around the MP is also

considered useful for identifying the cause of the fluctuation. Since comparing the distribution of air dose rates with that prior to the fluctuation is an effective method for identifying the cause, we conducted a walking survey using a backpack-style KURAMA (Kyoto University Radiation Mapping system) -II [1] (Fig. 1) device to determine the distribution of air dose rates around MPs.

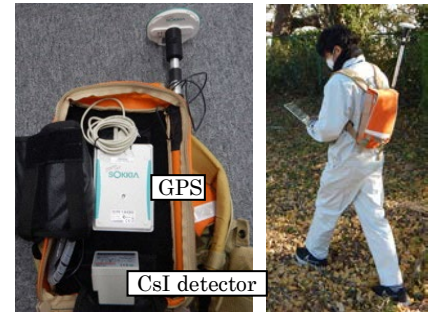


Fig. 1. KURAMA-II in a backpack.

EXPERIMENTS: A walking survey of the air dose rate was conducted by using KURAMA-II with a CsI (TI) scintillation detector (C12137-01, Hamamatsu Photonics) around the MP (an NaI(Tl) detector at 3 m height) at the Kiyohashi Disaster Prevention Community Center and its vicinity in Namie Town on December 11, 2025. The air dose rate along with the GPS data was recorded every second. The measurement route was basically the same as was in the measurement conducted on December 8, 2022. The measurement data were averaged in a 10-meter mesh and visualized to the colored air dose rate maps using GIS software (QGIS 3.44.8).

RESULTS: The air dose rate map is shown in Fig. 2 (a). The air dose rate ranged from 0.05 to 0.19 $\mu\text{Sv/h}$ (the MP air dose rate during the corresponding measurement period was 73 nGy/h). Fig. 2 (b) shows the air dose rate map generated from data collected in 2022 (the MP air dose rate during the corresponding measurement period was 77 nGy/h). Fig. 3 is the

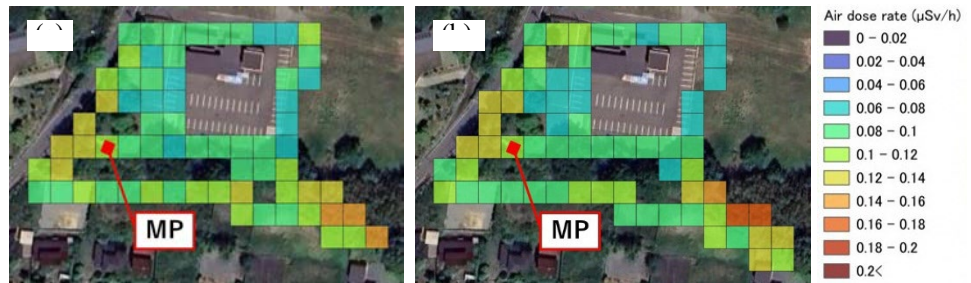


Fig. 2. The air dose rate maps obtained from measurements in (a) 2025 and (b) 2022.

rate of change for each mesh. The rate of change for each mesh generally fell between -25% to +25%. The present result shows that the distribution pattern doesn't change significantly over about 3 years. Given that artificial radionuclides with longer lifetimes persist in the environment in our prefecture and that air dose rates are expected to change over the long term, conducting such surveys regularly remains important.



Fig. 3. The rate of change for each mesh.

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Quantification of Trace Elements in Facility Components for Activation Assessment of the Large Helical Device (LHD)

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INTRODUCTION: Although the activation of particle accelerators has been widely recognized and extensively studied, little attention has been paid to the activation of other radiation-generating facilities. The Large Helical Device (LHD), a fusion experimental device, has previously been operated using deuterium plasma. Neutrons generated during the operation may have induced radioactive nuclides in stainless steel components of the vacuum vessel and in the concrete materials of the experimental hall. To evaluate the activation of the LHD facility, samples were collected from the device and the building materials, and trace elements were quantified by neutron activation analysis (NAA).

EXPERIMENTS: Approximately 100 mg of stainless steel was collected from the outer wall of the device using a drill. The collected material was finely cut and used as an irradiation sample. Similarly, approximately 100 mg of concrete was sampled, crushed into powder, and prepared for irradiation. JSS certified reference materials provided by the Iron and Steel Institute of Japan and GSJ reference materials provided by the Geological Survey of Japan were used as comparative standards. Neutron irradiation was carried out at the Kyoto University Research Reactor (KUR) under the conditions of 1 MW for 10 s and 5 MW for 30 min. Gamma-ray spectroscopy was performed for the irradiated samples using a germanium semiconductor detector.

RESULTS: Figure 1 shows the gamma-ray spectra of the stainless steel and concrete samples irradiated at 1 MW. For the stainless steel sample, ⁵⁶Mn was clearly detected, and several peaks originating from Ni were also observed. Based on these results, the material was estimated to be SUS316 stainless steel. For the concrete sample, peaks corresponding to ²⁴Na and ⁵⁶Mn were identified. The samples irradiated at 5 MW are currently under decay cooling, and quantitative analysis of activation products such as Co and Eu will be conducted in the near future.

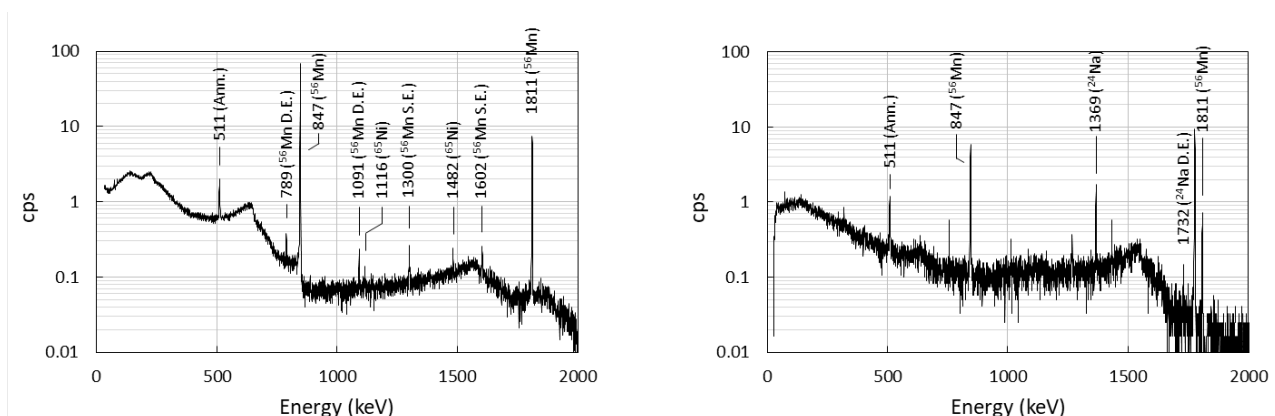


Fig. 1. γ -ray spectra of stainless-steel sample (left) and concrete sample (right) with irradiation of 1 MW - 10 s

Development of a non-destructive assay technique for nuclear material detection and U enrichment measurement by photonuclear reaction induced by bremsstrahlung X-ray

(1) Grand plan

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Development of a non-destructive assay technique for nuclear material detection and U enrichment measurement by photonuclear reaction has been conducted induced by external high-energy photon source. In the present research, focusing on the bremsstrahlung X-ray source technically established and available, the following 4 items are planned to be conducted; (1) development of nuclear material detection method, (2) development of its detector system, (3) demonstration experiment, and (4) nuclear data evaluation, and the research goal is to verify the principles of non-destructive assay technique for nuclear material detection and U enrichment measurement. Montecarlo simulation results of Bremsstrahlung X-ray spectrum and Photo-fission cross-section of ^{235}U , ^{238}U are shown in Fig. 1[1]. With combination of the initial energy of accelerated electron, different response from uranium is expected according to the energy dependence of photo-fission reaction cross-section and uranium isotopes, the correlation of uranium enrichment to the reaction response is expected and the demonstration experiments have just begun in 2025[1-2].

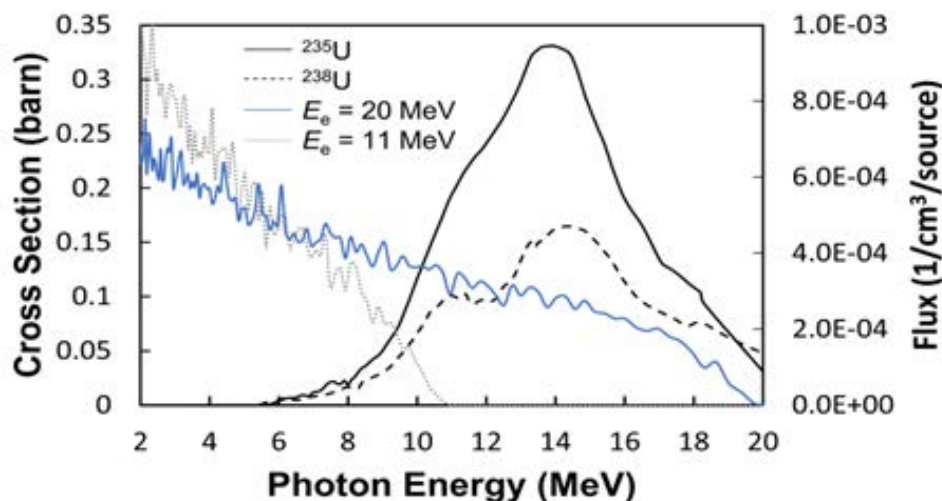


Fig. 1. Photo-fission cross-section of ^{235}U , ^{238}U and Bremsstrahlung X-ray spectrum [1]

REFERENCES:

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Homogeneity of radioactivity in the stainless steel sample produced by nuclear reactor neutron irradiation

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INTRODUCTION: The ongoing decommissioning project at the Tokyo Electric Power Company's Fukushima Daiichi Nuclear Power Station requires the measurement of radioactivity in large quantities of samples, including debris, metal, and sludge [1]. Ensuring the validity and reliability of these radioactivity measurements is an essential requirement for the implementation of this decommissioning project, which is the subject of significant public interest. To ensure validity and reliability, it is effective to verify the validity of the operator's measurements using test samples that contain a uniform concentration of the target nuclide and for which the radioactivity per unit mass has been calibrated. In this study, as a preliminary experiment for the preparation of metallic test specimens, we verified the homogeneity of radionuclides produced by irradiating stainless steel reference materials in a nuclear reactor.

EXPERIMENTS: Japanese Iron and Steel certified reference material JSS 652-17 (stainless steel 316) was used for neutron irradiation sample. The certified values in JSS 652-17 are Mn (0.961 ± 0.004 %), Cr (16.8 ± 0.04 %), and Mo (2.04 ± 0.01 %), respectively. Five samples of approximately 10 mg of JSS 652-17 were weighed using a METTLER TOLEDE XP26V micro balance and heat-sealed in the polyethylene bags. The samples were irradiated for 5 min using KUR PN2 (neutron fluence rate: 5×10^{12} n cm⁻²s⁻¹). After cooling for 1 day, the produced ⁵¹Cr, ⁵⁶Mn, and ⁹⁹Mo in the sample were measured using Ge detector (ORTEC GEM25185).

RESULTS: The decay corrected measured values at the end of the irradiation are shown in Table 1. Analysis of variance of the measured values revealed that only ⁵¹Cr showed statistically significant differences between samples. These results indicate that it is difficult to produce homogeneous samples solely through neutron activation by simple pneumatic irradiation facility.

Table 1 Measured results radioactive nuclides in the JSS 652-27 (stainless steel 316)

Sample No.	Measured No.	⁵¹ Cr cps/mg	⁵⁶ Mn cps/mg	⁹⁹ Mo cps/mg
No. 1	1st	1.20 ± 0.53 %	1405 ± 0.24 %	1.80 ± 0.51 %
	2nd.	1.23 ± 0.42 %	1453 ± 3.7 %	1.99 ± 0.45 %
No.2	1st	1.25 ± 0.73 %	1474 ± 0.37 %	1.90 ± 0.71 %
	2nd.	1.30 ± 0.43 %	1605 ± 4.3 %	2.09 ± 0.43 %
No.3	1st	1.29 ± 0.91 %	1498 ± 0.91 %	1.99 ± 0.92 %
	2nd.	1.30 ± 0.54 %	1636 ± 7.2 %	2.15 ± 0.56 %
No.4	1st	1.28 ± 0.90 %	1495 ± 0.70 %	2.01 ± 0.84 %
	2nd.	1.32 ± 0.65 %	1555 ± 0.64 %	2.09 ± 0.67 %
No.5	1st	1.34 ± 0.92 %	1543 ± 0.65 %	2.08 ± 0.87 %
	2nd.	1.34 ± 0.49 %	1548 ± 0.56 %	2.12 ± 0.45 %
Mean $\pm$ RSD%		1.29 ± 3.5 %	1520 ± 4.6 %	1.29 ± 3.5 %

Each value after “ $\pm$ ” indicates the counting statistics.

REFERENCE:

[1] https://www.dd.ndf.go.jp/files/user/pdf/en/strategic-plan/book/20251225_SP2025eFT.pdf

Study on the Calibration Method for ^{41}Ar Using an Inner-Through Type Ionization Chamber with a Commercial Gas Monitor

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INTRODUCTION: In order to calibrate gas monitors used for measuring radioactive noble gases in nuclear facilities, use of activity reference measurement standard gases shall be required. ^{41}Ar is one of potentially released noble gas from nuclear facilities. And the primary concentration standard for ^{41}Ar , is currently under development at the National Metrology Institute of Japan (NMIJ). In previous study, the response of an inner-through type Ionization Chamber (IC) was calibrated using the activity concentration of ^{41}Ar determined by absolute measurement with the proportional counters [1]. In the preceding fiscal year, the authors measured ^{41}Ar using the IC and evaluated the response ratio of them between P-10 gas (Ar: 90 %, CH₄: 10 %), used in proportional counter measurements, and dry air [2]. Based on this result, we successfully determined ^{41}Ar concentration using the IC when air was employed as the ionization medium. In the present study, with the aim of establishing a calibration system for ^{41}Ar , calibration method using the IC was examined with a commercially available ionization-chamber-type gas monitor.

EXPERIMENTS: Acrylic container (≈ 3 ml) was filled with Pure Argon gas. And ^{41}Ar gas was produced via $^{40}\text{Ar}(n, \gamma)^{41}\text{Ar}$ reaction. Samples were irradiated for 60 s at the bottom of KUR-SLY under operating at 1 MW thermal output. The gamma-ray spectrum of an irradiated ^{40}Ar sample measured using a High-Purity Ge (HPGe) semiconductor detector, the production of ^{41}Ar was confirmed without any impurities. As a test case for calibration, the AlphaGUARD (Bertin Technologies SAS)—an ionization-chamber-based commercial radon monitor—was employed. A gas loop including the IC and the AlphaGUARD was constructed. After sufficient evacuation, ^{41}Ar was injected from the acrylic container into the gas loop, and dry air was subsequently added up to approximately one atmosphere. The gas mixture was then circulated for about 15 minutes using a circulation pump to ensure homogeneity.

RESULTS: On the order of several tens of pA was measured with the IC, which corresponds to an ^{41}Ar concentration of approximately 200 Bq cm⁻³. An increase in the output of the AlphaGUARD was also observed after the injection of ^{41}Ar , indicating the feasibility of calibrating gas monitors using the IC employed in this study. However, because the AlphaGUARD provides output in terms of radon concentration by default, and problems such as the gas tightness, the evaluation of a calibration factor was not achieved. In future work, the system will be improved, and experiments using other gas monitors will be considered, with the aim of developing a robust and reliable calibration method.

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Performance Evaluation of Prompt Response with Cobalt Emitter SPND

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INTRODUCTION: For the safety and autonomous control of next-generation reactors such as Small Modular Reactors (SMRs), in-core neutron flux monitoring with high time resolution is essential. Self-powered neutron detectors (SPNDs) are widely employed for this purpose due to their durability in high-radiation environments. In our previous studies, we evaluated the feasibility of rhodium (Rh) and vanadium (V) emitter SPNDs. While the "delayed-type" emitters of Rh and V provide high sensitivity, they require digital compensation to correct for time delays caused by the half-life of beta decay (e.g., 3.74 min for ⁵²V). In this study, we evaluated a Cobalt (Co) emitter SPND, which is categorized as a "prompt-type" detector [1], to verify its instantaneous response during rapid reactor power changes.

EXPERIMENTS: Measurement was performed at the Kyoto University Reactor (KUR) using the Slant exposure tube (SLANT). The reactor power was varied between 1 and 5 MW to assess the dynamic response of the Co-SPND. The current output was measured using a high-precision digital measurement device [2]. In contrast to Rh and V emitters, which rely on beta decay, the Co emitter generates a signal primarily through prompt γ -ray emission and subsequent internal conversion or photoelectric effects following neutron capture via the ⁵⁹Co (n, γ) ⁶⁰Co reaction.

RESULTS: Figure 1 shows the Co-SPND output current during the reactor thermal power ramp-up. Compared to the V-SPND [3], which requires physical model-based deconvolution due to significant response delays, the raw output of the Co-SPND tracked the reactor power increase near-instantaneously. Although Co-SPNDs accumulate a long-term background signal from ⁶⁰Co ($T_{1/2} = 5.26$ years), it was confirmed that the prompt component dominates the signal during transient operations, providing sufficient accuracy for real-time monitoring without complex deconvolution. The characteristics underscore the suitability of Co-SPNDs for safety protection systems requiring rapid response to power excursions. However, it was observed that the output current began to decrease immediately after the reactor reached the 5 MW plateau. The current eventually dropped to approximately 60% of the expected value based on the initial sensitivity. Further investigations are planned to clarify the underlying physical mechanism of this phenomenon to ensure the reliability and accuracy of Co-SPNDs for steady-state monitoring at rated power.

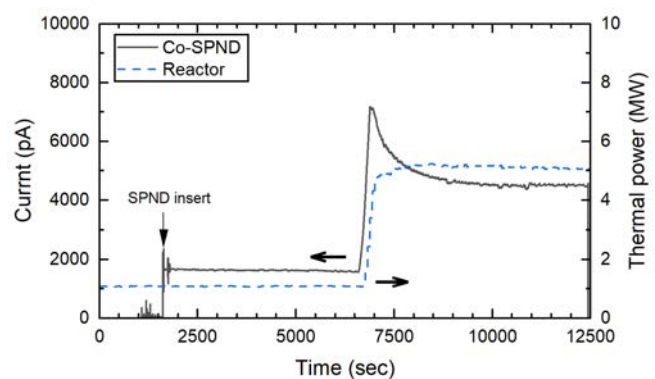


Fig. 1. Time trend of measured signal current to reactor power variation at SLANT.

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- [3] M. Sasano *et al.*, KURNS Progress Report 2024 (2025) 251.

Experimental Study on Fission Chamber Response in High Neutron Flux at KUR Heavy Water Neutron Irradiation Facility

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INTRODUCTION: Accurate monitoring of neutron flux, from reactor startup to full-power operation, is critical for the safety and control of next-generation reactors. Fission chambers (FCs) coated with uranium-235 are primary instruments for this purpose; however, they must transit between the pulse mode and Campbelling modes to cover a wide dynamic range. A seamless transition in the "overlap region" is essential for continuous monitoring, yet it requires rigorous verification under high-flux irradiation to account for pulse pile-up effects. The study was conducted at the Heavy Water Neutron Irradiation Facility of the Kyoto University Reactor (KUR-HWNIF) to evaluate FC performance under high-count-rate conditions.

EXPERIMENTS: Experiments were carried out at KUR-HWNIF, operating at a constant reactor power of 5 MW. The high-power operation is essential for achieving a maximum neutron flux of approximately $1 \times 10^9 \text{ cm}^{-2} \cdot \text{s}^{-1}$ [1], enabling performance evaluation under high-count-rate conditions. To evaluate the detector response across varying intensities, the thermal neutron flux was adjusted by varying the distance from the irradiation port and the thickness of polyethylene (PE) moderators, as summarized in Table 1. The primary objective of experiments was to validate the detector response within the Campbelling mode regime using the signal processing unit [2], specifically targeting the high-output range above $1 \times 10^8 \text{ s}^{-1} \cdot \text{cm}^{-2}$. Additionally, measurements were performed in the overlap region to verify the seamless transition between the pulse and Campbelling modes.

RESULTS: As shown in Figure 1, the experiments at KUR-HWNIF demonstrated linear detector responses across a thermal neutron flux of 4.1×10^6 to $1.5 \times 10^8 \text{ cm}^{-2} \cdot \text{s}^{-1}$. Linearity in pulse mode was confirmed from 2.9×10^5 to $8.2 \times 10^5 \text{ cps}$, while the Campbelling mode (equivalent count rate) covered 3.1×10^5 to $6.6 \times 10^6 \text{ cps}$. An overlap region of more than one order of magnitude was successfully established between the two measurement modes. Furthermore, by applying dead-time corrections to account for pulse pileup, the overlap was expected to extend to approximately two orders of magnitude. The findings validated the system's capability for seamless, wide-range neutron monitoring and demonstrated the high reliability of the FC under high-count-rate conditions.

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Table 1. Irradiation conditions and corresponding thermal neutron flux.

PE thickness [mm]	Distance [mm]
without	0
without	240
40	240
100	240

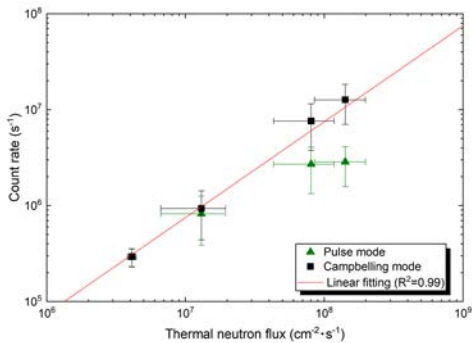


Fig. 1. Linearity of neutron flux to count rate at KUR-HWNIF.

A texture analysis of the excavated early medieval *Haji*-ware of different colour by INAA

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INTRODUCTION: To investigate the reason for the colour difference –white/red– of the early medieval low-temperature-fired pottery (*Haji*-ware), a collection of large number of *Haji*-ware of 13th century, excavated from a rather deep and large deposition in the campus of Kyoto University [1], is examined. It is mainly composed of the classic six types of the early medieval Kyoto *Haji*-ware: white larger bowl, white small bowl, white coaster-shaped plate, reddish medium-sized plate, reddish small plate, and reddish coaster-shaped plate. The four major types, excluding white/reddish coaster-shaped ones, are analyzed this year.

EXPERIMENTS: Conventional INAA was applied to determine the elemental composition of samples of the *Haji*-ware, each of whose main body had been drilled, or scraped, by the alumina [Al₂O₃] drill into fine powder as a sample after removing off the very surface, and then had been enclosed in a polyethylene bag [2]. Every of above-mentioned four types of the *Haji*-ware has seven samples respectively from different pieces. Each of the 28 samples was neutron-irradiated at Pn-3 (1 MW for 90 seconds) for short-lived nuclides, and at Pn-2 (5 MW for 1 hour) for long-lived ones. The gamma-ray spectrometry of the irradiated samples was performed after the irradiation, and the k_0 -standardization method for determination of concentration of elements was performed. For the k_0 method, three standard elements, Au, Lu and Zr, were prepared as a comparator.

RESULTS: With irradiation by Pn-3, concentrations of eleven elements (As, Eu, Ga, K, La, Mg, Mn, Na, Sm, Ti, and V) in almost every 28 sample were determined. The determination for samples irradiated by Pn-2 has not completed so far. From the short-lived nuclides two results can be pointed out here; (i) Na and Mn show clear difference in concentration to divide 28 samples into two groups, which fully correspond with the colour groups (Figs.1&2), and (ii) La cannot be the marker of colour difference, which was for the 14th century *Haji*-ware (Fig. 1). These results may suggest that the colour difference was brought by the texture difference, and that the preparation for clay texture to form the body of ware in 13th century differed from that in 14th century.

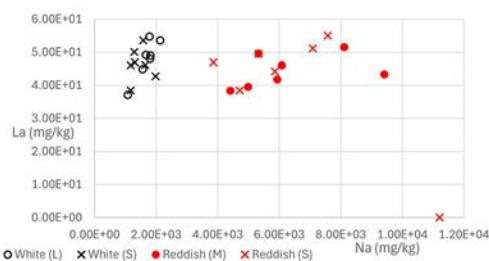


Fig.1 Distribution of 28 samples on the concentrations combination of Na with La.

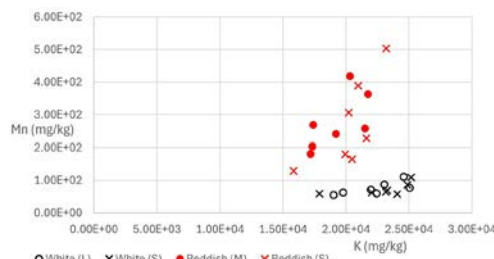


Fig.2 Distribution of 28 samples on the concentrations combination of Mn with K.

REFERENCES:

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Development of Radiation Monitor for Space weather measuring Electrons (RMS-e) and Protons (RMS-p) for the Himawari-10 satellite

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INTRODUCTION: Monitoring of the radiation environment in geostationary orbit around the Earth is very important for space weather forecast. However, the instruments on board the Himawari 8 and 9 meteorological satellites, which are currently operating, have an insufficient energy range and energy resolution to fully monitor the space radiation environment. Therefore, we are developing the Radiation Monitor for Space Weather measuring Electrons (RMS-e) and the Radiation Monitor for Space Weather measuring Protons (RMS-p) instruments for installation on the next Himawari meteorological satellite. The RMS-e instrument is designed to measure the energy and flux of electrons between 50 keV and 6 MeV, while the RMS-p instrument is designed to measure the energy and flux of protons between 10 and 500 MeV, using stacked solid-state detectors (SSDs) made of silicon semiconductors. We evaluated the measurement range for electron energy and the energy resolution of > 2 MeV electrons by irradiating electrons emitted by the Kyoto University Institute for Integrated Radiation and Nuclear Science Linear Accelerator (KURNS-LINAC) onto the engineering model (EM) of the RMS-e instrument. We also evaluated the degree to which electron contamination could contribute to noise in proton observations by irradiating electrons emitted by the KURNS-LINAC to the RMS-p EM.

EXPERIMENTS: RMS-e EM and RMS-p EM were installed in the irradiation room and the monoenergetic electron beam generated by LINAC was injected. The electron energies injected into RMS-e EM were 2.2, 2.6, 2.95, 3.3, 3.9, 4.5, 5.25, 6, and 7 MeV perpendicular to the stacked SSDs. The electron energies injected into RMS-p EM were 2.2, 10, and 30 MeV perpendicular to the stacked SSDs, and perpendicular to the plastic scintillator for eliminating oblique incidence. The outer circumference of the instruments was covered with lead blocks for radiation protection.

RESULTS: Figure 1 shows the energy spectrum obtained using RMS-e EM for all incident electron beams (2.2, 2.6, 2.95, 3.3, 3.9, 4.5, 5.25, 6, and 7 MeV). The energy resolution based on the full width half maximum of the peaks is about 10% for electrons below 5 MeV. Considering the losses caused by electron scattering from the Al shield installed at the entrance and the penetration of high-energy electrons, as well as the small number of detected electrons at very low beam flux, these energy resolutions are sufficient to monitor the high-energy electrons in geostationary orbit around the Earth.

Figure 2 shows the result obtained using RMS-p EM for all incident electron beams (2.2, 10, and 30 MeV). A portion of the >10 MeV electrons become contamination when irradiated onto a stacked SSDs, and >30 MeV electrons become contamination when irradiated onto the scintillator. Considering the prevalence of these electrons in orbit, they have virtually no effect on proton observations.

These results confirm that the performance of these instruments is sufficient for measuring the radiation environment in geostationary orbit.

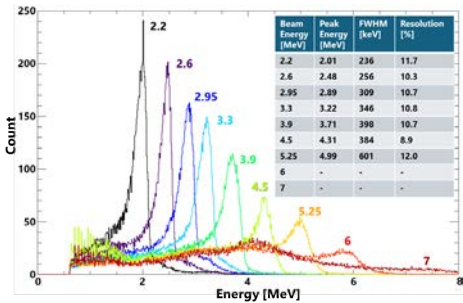


Figure 1. The electron energy spectra measured by RMS-e EM (2.2, 2.6, 2.95, 3.3, 3.9, 4.5, 5.25, 6 and 7 MeV).

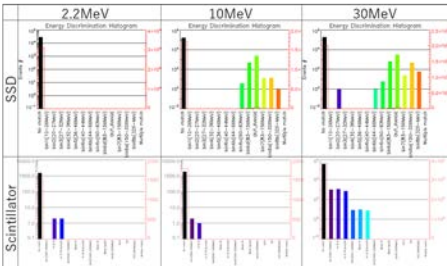


Figure 2. The electron energy discrimination by RMS-p EM (2.2, 10 MeV and 30 MeV).

Observation of Internal Pore Distribution in Additively Manufactured Metal Fabricated by Electron Beam Powder Bed Fusion

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INTRODUCTION: Metal additive manufacturing is widely regarded as a powerful fabrication approach, enabling the fabrication of complex geometries that are difficult to achieve with conventional methods and contributing to weight reduction and functional optimization. However, the process inherently involves rapid melting and solidification and therefore tends to generate internal defects such as microscopic pores. These features can influence the mechanical and functional performance of the final component.

In general, porosity in metal additively manufactured products has been evaluated through destructive techniques, including cross-sectional analysis with optical microscopy. Although such methods provide detailed local information, they are not suitable for evaluating the internal structure of an entire component without damage. To overcome this limitation, we are developing a non-destructive technique based on neutron phase imaging at the CN-3 port of KUR to visualize the porosity distribution within additively manufactured metal structures [1]. By employing a grating interferometer to detect small-angle neutron scattering caused by micropores, it becomes possible to probe the interior of relatively thick samples. This approach also enables observation over a wide area on the order of several tens of square centimeters.

EXPERIMENTS: The samples were approximately 1 cm³ cubes of INCONEL 718 fabricated by electron beam powder bed fusion under different process parameters. Pores with diameters ranging from sub-micrometer to micrometer scales induce small-angle neutron scattering. In this measurement, the “correlation length” [2, 3] of the scattering imaging was set to 0.5 μ m. The total exposure time was 640 s under a reactor thermal power of 5 MW.

RESULTS: Figure 1(a) shows the scattering image obtained by neutron phase imaging. In this image, darker regions correspond to areas where small-angle neutron scattering is more pronounced, indicating the presence of a larger amount of microporosity within the material. Fig. 1(b) plots the visibility reduction averaged over the entire sample against the average relative density evaluated by the Archimedes method. A correlation between the neutron scattering contrast and the bulk density is observed. Further comparison with X-ray CT is planned for future work.

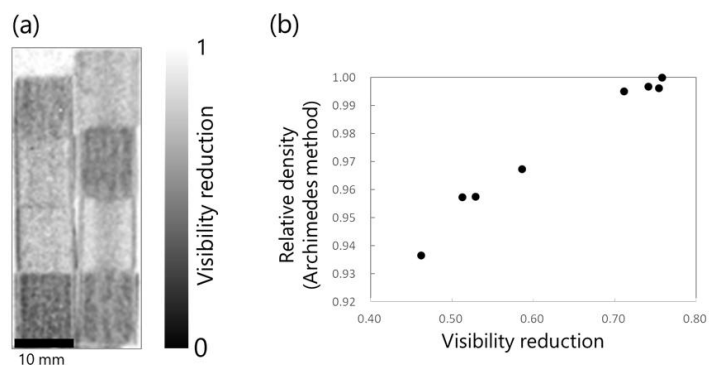


Fig. 1. (a) Neutron scattering image of the additively manufactured metal samples. (b) Correlation between the visibility reduction and the relative density evaluated by the Archimedes method.

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Testing of a Neutron Scintillation Detector at KUR CN-3

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INTRODUCTION: A flat-panel resistor-type photomultiplier (FRP) detector is a simple two-dimensional neutron detector. $^6\text{LiF/ZnS(Ag)}$ and ^6Li glass scintillators are widely used for neutron detection. The two scintillators can be purchased from Scintacor Co. Ltd. under the trade names ND Screen (ND) and GS20, respectively. GS20 outperforms ND in terms of the expected neutron efficiency and rate characteristics of the generated signal, whereas ND produces a much greater light yield. Furthermore, ND offers a cost advantage when scaled to large active areas. ND has a better positional resolution than GS20. This is because the transparent GS20 allows scintillation light from various angles of incidence to enter the photomultiplier, thereby increasing the light spread. Thus, because ND and GS20 have distinct advantages and disadvantages, they must be selected based on specific applications. In this paper, we report the results of neutron irradiation tests conducted at KUR CN-3 using GS20 (FRP-GS20) as the neutron scintillation detector.

EXPERIMENTAL RESULTS: In neutron irradiation tests using FRP-GS20, the detector was installed downstream of the disc chopper in CN-3, and the neutron efficiency and position resolution were measured using the neutron time-of-flight method. The experimental setup and measurement methods were almost identical to those used in our previous studies. The neutron intensity of CN-3 was derived to measure the neutron efficiency, as shown in Figure 1. This result was obtained using a neutron detector filled with ^3He gas. The neutron beam was collimated to $1\text{ cm} \times 1\text{ cm}$ using a B_4C resin collimator, and its wavelength dependence was calculated from the time of flight relative to the opening time of the disk chopper. The peak value of the CN-3 neutron intensity was $1.9\text{ \AA}$, and the neutron flux was $651\text{ neutrons/s cm}^2$. Figure 2 shows the measured neutron efficiency of FRP-GS20. These results were derived as the FRP-GS20 count values corresponding to the neutron intensities of CN-3 in the $1\text{--}6\text{ \AA}$ range. The thermal neutron efficiency was 66%, which was slightly lower than that provided by the manufacturer. This was attributed to the low $n\gamma$ discrimination capability in the pulse height distribution, resulting in a failure to count all neutron components. Figure 3 shows a two-dimensional image of a multi-pinhole array (pinhole diameter: 1 mm , pinhole pitch: 5 mm) fabricated from a Cd plate. The pinhole pitch was distorted at the edges owing to the use of a resistive chain readout. Based on this result, the two-dimensional image from the FRP-GS20 was corrected to absolute values, and the expected spatial resolution was 1.4 mm (full width at half maximum). This value was lower than that obtained using ND, attributed to the characteristics of the scintillator, as mentioned earlier. In the future, to improve the spatial resolution, we plan to reoptimize the configuration of FRP-GS20 and perform simulations using Geant4 to evaluate the validity of the measurement data.

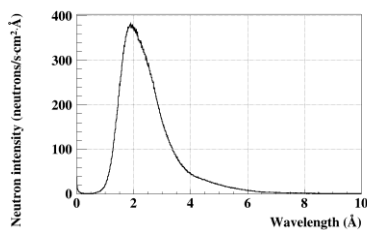


Figure 1. Neutron intensity of CN-3

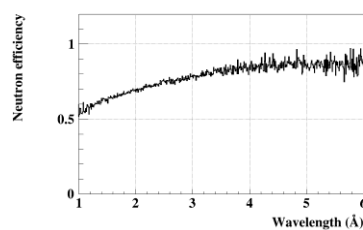


Figure 2. Neutron efficiency of FRP-GS20

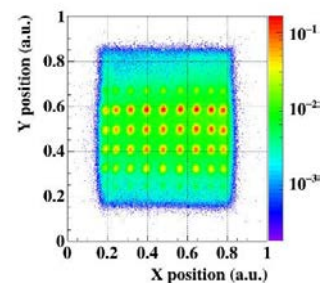


Figure 3.
2D multi-pinhole image

Study of Isotope Separation via Chemical Exchange Reaction

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INTRODUCTION: Chemical isotope separation for calcium and lithium has been studied by liquid-liquid extraction (LLE) with DC18C6 crown-ether [1]. This report describes the distribution coefficient and the isotope separation factor under the various reaction temperatures.

EXPERIMENTS: The experiment was carried out at -15, 0, room temperature (22±0.5), and 45 °C reaction temperature. 30% w/w CaCl₂/LiCl (aqueous solution) and 0.07M DC18C6 dissolved in chloroform, with a volume ratio of 1/10, as an aqueous and organic phase were mixed in the incubator with a several temperatures for 1 hour. The extraction process was conducted at room temperature for 1 minute and allowed for phase separation in the separation funnel for 10 minutes under the constant room temperature condition [1].

RESULTS: The results of temperature dependency indicated that the distribution coefficient (D) increases as the temperature decrease. The same behavior was observed on both calcium and lithium extraction. This finding was in good agreement with K. Nishizawa et al. report on the study of lithium extraction using crown-ether and temperature difference, ranging from 0 ~ 40 °C [2]. The results revealed the same behavior as our finding. However, K. Nishizawa carried out the extraction by 2M LiI, was the most applicable species due to the high distribution coefficient (D) and a high concentration of B15C5 (0.186M) under the same volume of organic and aqueous phase (20mL/20mL). The results indicated the dependency on separation factor (α) up to 1.045 at 0 °C.

Reaction Temperature °C	CaCl ₂ (aq) (M)	Distribution Coefficient (D)	The mole ratio between ion-crown complex to the total crown-ether	⁴⁸ Ca/ ⁴⁰ Ca Separation factor (α _{org})
-15	3.6	3.4 × 10 ⁻²	0.16	0.987 ± 0.003
0	3.6	3.0 × 10 ⁻²	0.14	0.989 ± 0.005
22	3.7	2.3 × 10 ⁻²	0.12	0.990 ± 0.003
45	3.6	1.6 × 10 ⁻²	0.074	0.990 ± 0.003

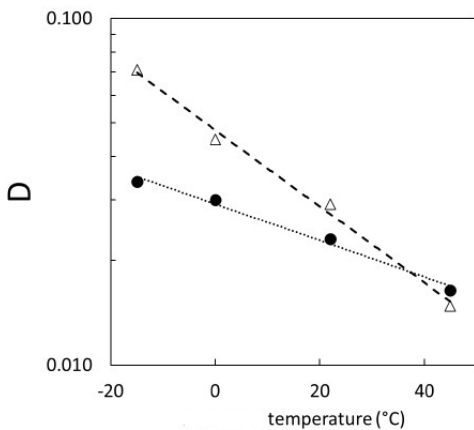


Fig. 1. The distribution coefficient (D) of calcium and lithium extraction on various reaction temperature (°C). Filled circle for Ca and open triangle for Li, respectively.

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Encapsulation of Lubricant Additives - Enhanced Lubrication Performance through Micellar Solubilization of Poorly Soluble Additives by Surfactants

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INTRODUCTION: In industrial machinery, lubrication is essential for reducing friction and wear and improving efficiency; however, conventional mineral oil-based lubricants pose challenges such as flammability and environmental pollution. As an alternative, water-based lubricants offer superior safety and environmental compatibility, but they require improvement due to issues with lubrication performance and corrosion. While additives such as nanoparticles and ionic liquids have been studied, challenges remain regarding dispersion and cost. On the other hand, organic additives are environmentally friendly and offer high lubrication performance, but they have the drawback of being poorly soluble in water. To address this issue, this study proposes a new method that utilizes micellization to encapsulate organic additives and disperse them uniformly in water.

SAMPLES: Aqueous solution with a 2HDNa concentration of 100 mM was prepared by neutralization reaction of 2-hexyldecanoate (>98%, Tokyo Chemical Industry) with NaOH (>97%, FUJIFILM Wako Pure Chemical Corporation). Our previous research demonstrated that the critical micelle concentration (CMC) of 2HDNa is approximately 25 mM [16]. Therefore, we chose a concentration of 100 mM. In addition, palmitic acid (>95 %, FUJIFILM Wako Pure Chemical Corporation) was selected as a model additive.

EXPERIMENTS AND RESULTS: FTIR spectroscopy and SAXS confirmed the successful solubilization of C16 within 2HDNa micelles, preserving its original molecular structure. Tribological experiments demonstrated that the addition of C16 significantly reduced the coefficient of friction, achieving a maximum reduction of over 33%. QCM-D and NR analyses were used to evaluate the adsorption performance. A lubrication mechanism was proposed in which micelles deform under external pressure, releasing the encapsulated C16, which then forms an effective boundary lubrication layer. These findings highlight micellar solubilization as a promising and efficient strategy for enhancing the performance of water-based lubricants.

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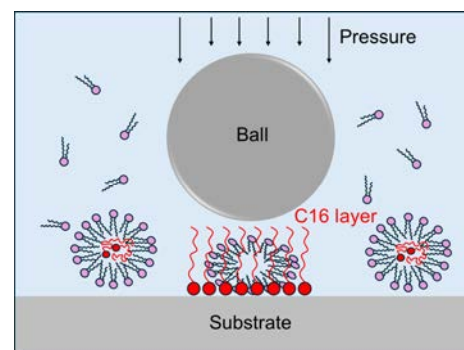


Fig. 1. Schematic illustration of the proposed lubrication mechanism by Micellar Solubilization.

Neutron shielding performance of novel lead-free iron oxide ceramics

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INTRODUCTION: Shielding is a critical safety procedure in the handling of ionizing radiation. When selecting shielding materials, it is necessary to consider appropriate options based on the type and energy of the radiation. For the shielding of γ -rays and X-rays, which are most frequently encountered, lead blocks are commonly employed. However, there is a global trend toward phasing out lead due to its inherent toxicity [1]. We have previously evaluated the γ -ray shielding performance of a novel lead-free iron-oxide-based ceramic to explore its potential as a substitute material [2]. Our prior results demonstrated its efficacy as a shielding material against γ - and X-ray radiation. To further investigate whether this novel ceramic functions as a universal radiation shield, the present study evaluates its effectiveness against reactor neutrons. Since neutrons induce activation and secondary γ -ray emission in materials, we examined not only the shielding capacity but also these associated phenomena. The findings of this study may broaden the selection of shielding materials for nuclear reactors and accelerator facilities where neutron generation is expected.

EXPERIMENTS: In the current fiscal year, additional experiments were conducted to enhance the reliability of the data by expanding the experimental dataset. While the methodology follows our previous report [3], a summary is provided below. The neutron shielding effectiveness was evaluated using the transport system at the Heavy Water Neutron Irradiation Facility (HWNIF). For this evaluation, three types of samples—ceramics, lead (Pb), and iron (Fe)—were prepared with dimensions of 10 cm $\times$ 10 cm and a thickness of 1 cm, and were utilized in a stacked configuration. The activation characteristics were assessed using the rail system at the HWNIF. For this purpose, samples of the same materials were fabricated with dimensions of 2 cm $\times$ 2 cm $\times$ 1 cm. These samples were subjected to neutron irradiation for periods of 5 and 50 minutes, and their induced activation was analyzed using a High-Purity Germanium (HPGe) semiconductor detector. Furthermore, prompt γ -ray analysis was performed using the Prompt Gamma-ray Analysis (PGA) system installed at the neutron guide tube (E-3). Samples of the same size as those in the previous experiment were irradiated at 1 MW for a duration of 5 hours.

RESULTS: The experiments conducted in the current fiscal year followed the same procedures as those of the previous year to enhance data reliability, and the results obtained were largely consistent (data not shown) [3]. Specifically, the shielding capacity of the investigated ceramics against total radiation followed the order of Fe > Ceramics > Pb. For thermal neutrons, the shielding capacity was observed to be Fe > Ceramics $\approx$ Pb. Regarding activation characteristics, the induced activity ratio followed the order of Fe > Ceramics > Pb. Furthermore, in terms of prompt γ -ray emission, the results indicated a trend of Fe $\approx$ Ceramics > Pb. These findings suggest that while the novel ceramics demonstrate a certain degree of neutron shielding capability, there remain challenges concerning material activation and prompt γ -ray emission.

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Study of neutron imaging system for ensuring safety in debris removal

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INTRODUCTION: In the debris removal for decommissioning of 1F (Fukushima Daiichi Nuclear Power Plant), the shape of debris and surrounding environment may be changed, and neutrons may be generated. Since the debris are assumed to be unevenly distributed, an omnidirectional detector that can identify the direction of incident neutrons is required. In this study, we developed a neutron imaging system based on the omnidirectional detectors for β/γ -rays [1]. In this study, irradiation tests were conducted in Kyoto University Research Reactor (KUR) to evaluate the performance of the imaging system.

EXPERIMENTS: The developing omnidirectional detector is based on the FRIE (Fractal Radiation Imaging Elements) detector, a radiation imager for β/γ -ray measurement with a Sierpinski tetrahedron shape [1]. The FRIE for neutron (nFRIE) combines neutron scintillators and shielding materials. The detector elements consist of tetrahedral acrylics and thin LiF:ZnS scintillators, and the 16 elements are installed in the fractal structure with B₄C compounds. The irradiation tests were conducted at various locations around the KUR to evaluate the direction of the neutrons and the average count rate of all elements.

RESULTS: Fig. 1 shows the photograph of the irradiation tests at the KUR. The imaging measurements were performed by rotating the detector in 30-degree increments at various positions surrounding the reactor, and high signals were obtained in the direction of the reactor at all positions. The average count rates of the 16 elements and the dose rate obtained using a neutron survey meter agreed with a deviation of 10.5%, indicating that developed detector can measure the neutron dose. In conclusion, we found that the detector we developed accurately points toward the neutron source and correlated well with the neutron dose rate.

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Fig. 1. Photograph of the irradiation test at KUR

Characterization of the KURNS-LINAC Neutron Source based on Indirect High-Energy Neutron Imaging

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INTRODUCTION: We have been developing a portable neutron source evaluation method using activation foil transfer to X-ray IP film. By utilizing threshold reactions in various metal converters, it images specific energy bands. This simple, rapid, and inexpensive method is applicable to both reactors and accelerators, assessing spatial and spectral characteristics without transporting radioactive materials. In this study, we measured the secondary particle emission profile from a photo-nuclear reaction source system at KURNS-LINAC, an electron accelerator neutron source at the Institute for Integrated Radiation and Nuclear Science, Kyoto University. The measured secondary particle emission profile from the target system in its actual operational state was evaluated by comparing it with previously measured profiles from a same kind but standalone target at the Hokkaido University neutron source (HUNS).

EXPERIMENTS: Multiple metal activation foils for neutron recording were placed at appropriate positions near the KURNS-LINAC neutron source. After two-hour irradiation with generated neutrons and secondary particles, the beta-rays emitted from the radionuclides within the activated foils were transferred to the X-ray imaging plates (IP). This allowed for the evaluation of the spatial distribution and intensity of the generated secondary particles by neutron irradiation. We used IP readers brought from Hokkaido University and, in part, SHI-ATEX equipment at Ehime. The residual activated foils were checked for product nuclides by KURRI HPGe detector.

RESULTS: Fig. 1 shows the IP transferred image from a 200 x 300 mm² Al activation foil positioned to surround the KURNS-LINAC neutron source system. This is the first time that a spatial distribution image of neutrons generated by an accelerator-based system has been observed on such a large dimension. It is clearly observed that the gradation of the image corresponds to the presence of the source cooling system, and neutrons are intensely generated along the downstream direction of the incident electron beam. In KURNS-LINAC and HUNS facilities both based on electron accelerators, neutron production relies on (γ ,n) photonuclear reactions driven by bremsstrahlung radiation generated within Ta (KURNS-LINAC) or W + Pb (HUNS) targets. This reaction is isotropic with respect to direction. Meanwhile, considering the strong neutron peak along downstream of the electron beam makes it essential to account for direct process nuclear reactions. This process evaluations using Zn activation foils checked by HPGe detector indicated that the X-ray beam and consequent neutrons spread at an angle of approximately 7 degrees along the electron beam downstream direction, which is consistent with the results obtained at HUNS. These findings suggest that the forward directionality of the generated neutrons basically corresponds well to that of the X-rays.

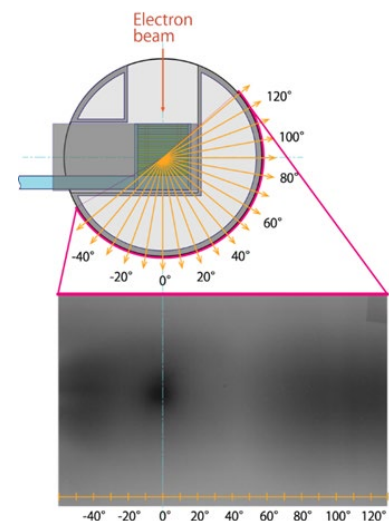


Fig. 1. Placement of a large Al activation foil (upper) and the resulting transfer image

II. PUBLICATION LIST
(APRIL 2025 – MARCH 2026)

1. Slow Neutron Physics and Neutron Scattering

Papers

Supramolecular Interface Engineering via Interdiffusion for Reusable and Dismantlable Polymer Adhesion
Yamaoka Kenji, Wada Takuma, Ogasa Iori, Komyo Takeru, Luo Chao, Ikura Ryohei, Hino Masahiro, Yamada Masako, Seto Hideki, Fujii Yoshihisa, Uetsuji Yasutomo, Takashima Yoshinori
Advanced Materials 38(6) (2026) e07939 (doi)10.1002/adma.202507939

Disorder in the Antifluorite Sulfide Cathode: $\text{Li}_{1.7}\text{Cu}_{0.15}\text{S}_2$
Miura Akira, Cheng Yi-Ting, Masahiro Takenaka, Nambu Yusuke, Sasaki Shunsuke, Nagao Masanori, Mitsudome Takato, Fujii Yuta, Tadanaga Kiyoharu, Machida Nobuya, Bartel Christopher J.
Chemistry of Materials 37(21) (2025) 8521-8528 (doi)10.1021/acs.chemmater.5c01328

Transition-Edge Sensor Spectrometer for Precision Spectroscopy of Antiprotonic Atoms
Morgan Kelsey, Azuma Toshiyuki, Baptista Gonalo, Becker Daniel, Bennett Douglas, Butin Franois, Dean Jonathan W., Eizenberg Ofir, Fowler Joseph W., Fujioka Hiroyuki, Gamba Davide, Gard Johnathon D., Garroum Nabil, Guerra Mauro, Hashimoto Tadashi, Higuchi Takashi, Hori Masaki, Indelicato Paul, Keller Mark, Machado Jorge, Mates John A.B., Nakamura Nathan, Nobles Jason, Nez Franois, Ohayon Ben, Okada Shinji, Ortiz Nathan, Rathi Shikha, Roosa Michael, Rouss  Jean-Yves, Saito Takeshi, Schmidt Daniel, Senetaire Quentin, Sommerfeldt Jonas, Szypryt Paul, Ullom Joel, Weber Joel, Yzombard Pauline, Zito Marco, Paul Nancy, Swetz Daniel
IEEE Transactions on Applied Superconductivity 36(6) (2026) 1- 6 (doi)10.1109/tasc.2026.3672351

Impact of a Reflecting Material on the Search for Neutron–Antineutron Oscillations using Ultracold Neutrons
Fujioka Hiroyuki, Higuchi Takashi
Progress of Theoretical and Experimental Physics 2026(2) (2025) 023C01 (doi)10.1093/ptep/ptaf189

In Situ High-Voltage Generation with a Cockcroft–Walton Multiplier for a Xenon Gas Time Projection Chamber
Akiyama Shinichi, Hikida Junya, Yoshida Masashi, Nakamura Kazuhiro, Ban Sei, Hirose Masanori, Ichikawa Atsuko K, Iwashita Yoshihisa, Kikawa Tatsuya, Nakajima Yasuhiro, Nakamura Kiseki D, Nakaya Tsuyoshi, Obara Shuhei, Sakashita Ken, Sekiya Hiroyuki, Sugashima Bungo, Urano Soki, Hatsumi Sota, Kobayashi Sota, Sasaki Hayato
Progress of Theoretical and Experimental Physics 2025(5) (2025) 053H03 (doi)10.1093/ptep/ptaf066

Proceedings

Development and numerical simulation of a segmented Soller Collimator for Neutron Imaging
Yunhao Gu, Masahiro Hino, Takashi Higuchi, Yoshichika Seki, Ryuto Fujitani
Proceedings of the 60th KURNS Scientific Meeting, Kumatori, Japan/Online (Jan. 29-30, 2026) 41

Development of evaluation method for metal additive manufacturing at the KUR CN-3
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Proceedings of the 60th KURNS Scientific Meeting, Kumatori, Japan/Online (Jan. 29-30, 2026) 45 (in Japanese)

Development of neutron focusing mirrors and the application for neutron beam optics in the new research reactor at "Monju" site II
Masahiro Hino, Takuya Hosobata, Masahiro Takeda, Yutaka Yamagata, Takashi Higuchi, Ryuto Fujitani, Hitoshi Endo
Proceedings of the 60th KURNS Scientific Meeting, Kumatori, Japan/Online (Jan. 29-30, 2026) 34 (in Japanese)

Exploring quantum magnetism with neutrons
Yusuke Nambu
Proceedings of the 60th KURNS Scientific Meeting, Kumatori, Japan/Online (Jan. 29-30, 2026) 7-8 (in Japanese)

Half-life measurements of sub-nanosecond isomeric state with LaBr_3 scintillator
Yui Okuda, Michihiro Shibata, Akihiro Taniguchi
Proceedings of the 60th KURNS Scientific Meeting, Kumatori, Japan/Online (Jan. 29-30, 2026) 49 (in Japanese)

Spin Phase Continuous Modulation toward inelastic scattering spectrometers
Ryuto Fujitani, Masahiro Hino, Takashi Higuchi
Proceedings of the 60th KURNS Scientific Meeting, Kumatori, Japan/Online (Jan. 29-30, 2026) 52 (in Japanese)

Others

Impact of a Reflecting Material on a Search for Neutron--Antineutron Oscillations using Ultracold Neutrons

Fujioka Hiroyuki, Higuchi Takashi

Arxiv (2025) (doi)10.48550/arXiv:2508.17725

Initial results of the TRIUMF ultracold advanced neutron source

B. Algohi, D. Anthony, L. Barron-Palos, M. Bosse, M. P. Bradley, A. Brossard, T. Bui, J. Chak, R. Chiba, C. Davis, R. de Vries, K. Drury, B. Franke, D. Fujimoto, R. Fujitani, M. Gericke, P. Giampa, C. Gibson, R. Golub, K. Hatanaka, T. Hepworth, T. Higuchi, G. Ichikawa, I. Ide, S. Imajo, A. Jaison, B. Jamieson, M. Katotoka, S. Kawasaki, M. Kitaguchi, W. Klassen, E. Korkmaz, E. Korobkina, F. Kuchler, M. Lavvaf, T. Lindner, N. Lo, S. Longo, K. W. Madison, Y. Makida, J. Malcolm, J. Mammei, R. Mammei, Z. Mao, C. Marshall, J. W. Martin, R. Matsumiya, M. McCrea, E. Miller, M. Miller, K. Mishima, T. Mohammadi, T. Momose, M. Nalbandian, T. Okamura, S. Pankratz, R. Patni, R. Picker, K. Qiao, W. D. Ramsay, W. Rathnakela, T. Reimer, D. Salazar, J. Sato, W. Schreyer, T. Shima, H. M. Shimizu, S. Sidhu, S. Stargardter, R. Stutters, P. Switzer, I. Tanihata, Tushar, S. Vanbergen, W. T. H. van Oers, N. Yazdandoost, Q. Ye, A. Zahra, M. Zhao

Arxiv (2025) (doi)10.48550/arXiv:2509.02916

Neutron EDM Experiment with an Advanced Ultracold Neutron Source at TRIUMF

T. Higuchi, B. Algohi, D. Anthony, L. Barron Palos, M. Bradley, A. Brossard, T. Bui, J. Chak, R. Chiba, C. Davis, R. de Vries, K. Drury, D. Fujimoto, R. Fujitani, M. Gericke, P. Giampa, R. Golub, T. Hepworth, G. Ichikawa, S. Imajo, A. Jaison, B. Jamieson, M. Katotoka, S. Kawasaki, M. Kitaguchi, W. Klassen, E. Korkmaz, E. Korobkina, M. Lavvaf, T. Lindner, N. Lo, S. Longo, K. Madison, Y. Makida, J. Malcolm, J. Mammei, R. Mammei, Z. Mao, C. Marshall, J. W. Martin, M. McCrea, E. Miller, M. Miller, K. Mishima, T. Mohammadi, T. Momose, T. Okamura, H. J. Ong, R. Patni, R. Picker, K. Qiao, W. D. Ramsay, W. Rathnakela, D. Salazar, J. Sato, W. Schreyer, T. Shima, H. M. Shimizu, S. Sidhu, S. Stargardter, P. Switzer, I. Tanihata, Tushar, S. Vanbergen, W. T. H. van Oers, Y. Watanabe, N. Yazdandoost, Q. Ye, A. Zahra, M. Zhao

Arxiv (2025) (doi)10.48550/arXiv:2507.05278

Novel concept for low-energy antineutron production and its application for antineutron scattering experiments

Filippi Alessandra, Fujioka Hiroyuki, Higuchi Takashi, Venturelli Luca

Arxiv (2025) (doi)10.48550/arXiv:250306972

2. Nuclear Physics and Nuclear Data

Papers

Systematic investigation of photon strength functions with monochromatic γ -ray beams

Gheorghe Ioana, Aumann Thomas, Baumann Martin, van Beek Patrick, Kuchenbrod Philipp, Lalić Nikolina, Scheit Heiko, Symochko Dmytro, Wang Hongwei, Fan Gongtao, Nishio Katsuhisa, Ohtsuki Tsutomu, Suzaki Fumi, Hirose Kentaro, Inagaki Makoto, Ari-izumi Takashi, Garrote Frank L. Bello, Bjørøen Marianne, Eriksen Tomas, Ingeberg Vetle Wegner, Paulsen Wanja, Pedersen Line G., Reaz Fardous, Belyshev Sergey, Stopani Konstantin, Lui Yiu-Wing, Filipescu Dan, Tudora Anabella, Goriely Stephane, Miyamoto Shuji, Utsunomiya Hiroaki

EPJ Web of Conferences 329 (2025) 01014 (doi)10.1051/epjconf/202532901014

Measurements of neutron capture cross-section for nuclides of interest in decommissioning (III): $^{170}\text{Er}(n,\gamma)^{171}\text{Er}$ and $^{180}\text{Hf}(n,\gamma)^{181}\text{Hf}$ reactions

Nakamura Shoji, Shibahara Yuji, Endo Shunsuke, Rovira Gerard, Kimura Atsushi

Journal of Nuclear Science and Technology 62(7) (2025) 617-630 (doi)10.1080/00223131.2025.2457589

Measurements of neutron capture cross-sections for nuclides of interest in decommissioning (IV): $^{165}\text{Ho}(n,\gamma)^{166\text{m}}\text{Ho}$ reactions

Nakamura Shoji, Shibahara Yuji, Endo Shunsuke, Rovira Gerard, Kimura Atsushi

Journal of Nuclear Science and Technology 62(11) (2025) 1086-1099 (doi)10.1080/00223131.2025.2525327

^{57}Fe Mössbauer spectrometry on $\text{SrTiO}_{3-\delta}$ perovskite oxides co-doped with dilute Fe and Sn

Nomura Kiyoshi, Takahashi Masashi, Kokubo Hiroko, Naka Takashi, Kobayashi Yasuhiro, Yoshimura Yuika, Koike Yuya, Kubuki Shiro, Okabayashi Jun, Barrero Cesar

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^{61}Ni Mössbauer Spectroscopy on Hofmann-type complex $\{\text{Fe}(\text{Ethyl isonicotinate})_2[\text{Ni}(\text{CN})_4]\}$, $\{\text{Fe}(\text{Allyl isonicotinate})_2[\text{Ni}(\text{CN})_4]\}$

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Fundamental properties of novel zero-dimensional halide Na_2HfI_6 and application for neutron detection by Li doping
Fujiwara Chihaya, Kurosawa Shunsuke, Yamaji Akihiro, Yoshikawa Akira, Matsubayashi Nishiki, Takata Takushi, Tanaka Hiroki

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Yamamoto Toshihiro, Sakamoto Hiroki

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X-Ray-Induced Quenching of the Th^{229} Clock Isomer in CaF_2

Guan Ming, Bartokos Michael, Beeks Kjeld, Fujimoto Hiroyuki, Fukunaga Yuta, Haba Hiromitsu, Hiraki Takahiro, Kasamatsu Yoshitaka, Kitao Shinji, Leitner Adrian, Masuda Takahiko, Nagasawa Nobumoto, Okai Koichi, Ogake Ryoichiro, Pimon Martin, Pressler Martin, Sasao Noboru, Schaden Fabian, Schumm Thorsten, Seto Makoto, Shigekawa Yudai, Shimizu Kotaro, Sikorsky Tomas, Tamasaku Kenji, Takatori Sayuri, Watanabe Tsukasa, Yamaguchi Atsushi, Yoda Yoshitaka, Yoshimi Akihiro, Yoshimura Koji

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A novel reduced-order model and two-stage optimization strategy for high-efficiency predicting thermal-hydraulic behavior in a 6×6 fuel bundle

Min Guangyun, Shen Xiuzhong, Wang Laishun, Jiang Naibin

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Development of controlling method of gamma rays by nuclear resonant forward scattering

Hiroyuki Yamashita

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In vivo Imaging of Gold Nanoparticles via Neutron Activation

Nanase Koshikawa, Yuka Kikuchi, Jun Kataoka, Yuichiro Kadonaga, Atsushi Toyoshima, Masashi Murakami, Hiroki Kato, Kazuhiro Ooe, Koichi Takamiya

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Present Status of Mössbauer Spectroscopy Using Sources Irradiated by KUR

Shinji Kitao, Ryo Masuda, Yasuhiro Kobayashi, Hiroyuki Yamashita, Makoto Seto

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Search for neutron-antineutron oscillations with ultracold neutrons

Takashi Higuchi, Hiroyuki Fujioka

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Saito T.Y., Niikura M., Matsuzaki T., Sakurai H., Igashira M., Imao H., Ishida K., Katabuchi T., Kawashima Y., Kubo M.K., Miyake Y., Mori Y., Ninomiya K., Sato A., Shimomura K., Strasser P., Taniguchi A., Tomono D., Watanabe, Y.

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Higuchi Takashi, Fujioka Hiroyuki

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Baptista Goncalo, Rathi Shikha, Roosa Michael, Senetaire Quentin, Sommerfeldt Jonas, Azuma Toshiyuki, Becker Daniel, Butin Francois, Eizenberg Ofir, Fowler Joseph, Fujioka Hiroyuki, Gamba Davide, Garroum Nabil, Guerra Mauro, Hashimoto Tadashi, Higuchi Takashi, Indelicato Paul, Machado Jorge, Morgan Kelsey, Nez Francois, Nobles Jason, Ohayon Ben, Okada Shinji, Schmidt Daniel, Swetz Daniel, Ullom Joel, Yzombard Pauline, Zito Marco, Paul Nancy

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Mass measurement by using newly developed doughnut-shaped helium gas cell at KISS

Y. Hirayama, M. Mukai, P. Schury, Y.X. Watanabe, S. Iimura, H. Ishiyama, S.C. Jeong, H. Miyatake, T. Niwase, M. Rosenbusch, A. Takamine, A. Taniguchi, Y. Yamanouchi, and M. Wada

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3. Reactor Physics and Reactor Engineering

Papers

Applying the derivative source method to flux derivatives in Monte Carlo fixed source problems caused by interface shifts between spontaneous fission materials

Yamamoto Toshihiro, Sakamoto Hiroki

Annals of Nuclear Energy 227(A) (2026) 111916 (doi)10.1016/j.anucene.2025.111916

Data-fusion-based variable-fidelity reduced-order model for accurate thermal-hydraulic behavior prediction in a 6×6 rod bundle

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Song Kyoseong, Pyeon Cheol Ho, Shim Hyung Jin

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Development of measurement system acquiring time-resolved energy-domain Mössbauer spectra
Yamashita Hiroyuki, Kitao Shinji, Kobayashi Yasuhiro, Fujiwara Kosuke, Mitsui Takaya, Seto Makoto
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Park Jae Hyung, Song Siwon, Kim Seunghyeon, Lee Sangjun, Kim Jinhong, Pyeon Cheol Ho, Kim Sin, Lee Bongsoo
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Kinoshita Ikuo, Hibiki Takashi, Shen Xiuzhong, Chen Shao-Wen
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Miyamoto Masanari, Higuchi Takashi, Furusawa Kentaro, Sekine Norihiko, Hayasaka Kazuhiro, Tanaka Utako
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Yiming Wang, Toshihiro Yamamoto

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Effect of Borehole Spatial Position on the Gas Concentration Field in a Goaf Area

Haifeng Ma, Xiuzhong Shen, Wenwang YANG

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Elucidation and Modeling of Flow Characteristics and Interfacial Transport Mechanisms in Gas-Liquid Two-Phase Flow

Xiuzhong Shen, Takashi Hibiki, Haomin Sun

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Neutron irradiation experiment for ITER diagnostic systems in JADA

Sin-iti Kitazawa, Masao Ishikawa, Ryota Imazawa, Eiichi Yatsuka, Tomohiko Ushiki, Kunpei Nojiri, Shigehiro Kono, Kazuhiro Totimoto, Nemoto Yoshiki, Shunsuke Tainaka, Takanori Kikuchi, Yasufumi Yoshino, Ryou Okumura, Koichi Takamiya

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Numerical Simulation and AI-Driven Prediction of Thermodynamic Performance in Curved Channel Printed Circuit Heat Exchangers

Feiran Wang, Yasushi Saito, Daisuke Ito, Naoya Odaira, Sichao Tan, Jiming Wen

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Profundity in fluid dynamics research – our vortex modeling study as example –

Kei Ito

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Relationship with Critical Assembly

Tsuyoshi Misawa

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Research on the Dynamics of Pollutants in Water, Soil, and Sediment Environments and Environmental Remediation Technologies

Yoko Fujikawa

Proceedings of the 60th KURNS Scientific Meeting, Kumatori, Japan/Online (Jan. 29-30, 2026) 70-72 (in Japanese)

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核物質を含む中性子増倍系の γ 線スペクトル測定による特性把握

Yasushi Nauchi

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Development of a bipolar 50 V output digital-to-analog converter system for ion-shuttling operations

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4. Material Science and Radiation Effects

Papers

Investigating Thermoelectric Properties of GeTe Alloys with Multi Element Doping: Insights from High-Entropy Engineering

Sun Yifan, Kurosaki Ken, Imamura Tetsuya, Torata Ryusuke, Ohishi Yuji, Palaporn Dulyawich, Nachaithong Theeranuch, Pinitsoontorn Supree, Padchasri Jintara, Kidkhunthod Pinit, Suwannaruang Methus, Tanusilp Sora-at
ACS Omega 10(29) (2025) 32112-32121 (doi)10.1021/acsomega.5c03826

Phase Transitions of Eutectic High Entropy Alloy AlCoCrFeNi_{2.1} Under Shock Compression

Parsons Sophie Emilene, Katagiri Kento, Chen Hangman, Hari Anirudh, Cubit Ernest W., Irvine Sara Jessica, Luccioni Dorian, Lin Rayen, Madril Laura Elizabeth, Reddy Tharun, McKinney William J., Ren Jie, Yang Wuxian, Ozaki Norimasa, Amouretti Alexis, Kodama Ryosuke, Nakamura Hirotaka, Nakanishi Yusuke, Ota Masato, Seto Yusuke, Takagi Sota, Okuchi Takuo, Umeda Yuhei, Inubushi Yuichi, Miyanishi Kohei, Sueda Keiichi, Togashi Tadashi, Yabashi Makina, Yabuuchi Toshinori, Li Wanghui, Specht Paul Elliot, Cao Penghui, Chen Wen, Vohra Yogesh K., Dresselhaus-Marais Leora

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Lane Harry, Kaur Guratinder, Kawamata Masahiro, Nambu Yusuke, Keller Lukas, Ewings Russell A., Voneshen David J., Williams Travis J., Walker Helen C., Viehland Dwight, Gehring Peter M., Stock Chris

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A novel liquid film shearing polishing technique for silicon carbide and its processing damage mechanisms

Chen Hongyu, Wan Hongbing, Hong Binbin, Hang Wei, Zhu Te, Zhang Peng, Cao Xingzhong, Xu Qiu, Wang Rong, Han Xuefeng, Lyu Binghai

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Research on high-quality and low-damage liquid film shearing polishing of 4H-SiC and its surface/subsurface damage evolution

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Applied Surface Science 720 (PartA) (2026) 165117 (doi)10.1016/j.apsusc.2025.165117

Effect of P₂O₅ addition on the structure of Na₂O–Y₂O₃–SiO₂ glasses and glass-ceramics

Maekawa Ryuichi, Kawada Koji, Yazawa Koji, Deguchi Kenzo, Ohki Shinobu, Yamashita Kimihiro, Hashimoto Hideki, Niitsu Kodai, Onodera Yohei, Okura Toshinori

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Multiscale Insights into Sodium Storage in Hard Carbon from Operando Small- and Wide-Angle Neutron Scattering Measurements

Y. Umemoto, K. Ohishi, D. Igarashi, R. Tatara, C. C. Lin, K. Nakamoto, Y. Kawamura, K. Hiroi, S. Takata, Y. Nambu, Y. Tateyama, S. Komaba

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Refining hydrogen positions in α -FeOOH through combined neutron diffraction and computational techniques

Yusuke Nambu, Akihide Kuwabara, Masahiro Kawamata, Seira Mori, Megumi Okazaki and Kazuhiko Maeda

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Effect of soft X-rays on vacancy-type defects in highly hydrogenated DLC films

Kanda Kazuhiro, Niwa Daisuke, Mishima Tomohiro, Hori Fuminobu, Yabuuchi Atsushi, Kinomura Atsushi

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Defect structure evolution in early-stage neutron irradiated F82H and its model alloys at 363 K and 563K

Wang Pinyao, Huang Shaosong, Sato Koichi, Xu Qiu, Yoshiie Toshimasa

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Kobayashi Seiyo, Katayama Kazunari, Isogawa Hiroki, Matsuura Hideaki, Akashi Kenta, Inuma Yuto
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Investigation of the high-temperature tensile properties and helium ion irradiation resistance of W- La_2O_3 composites prepared by hot rolling
Xu Dang, Fu Kaichao, Sang Changcheng, Chen Ruizhi, Chen Pengqi, Lu Yingwei, Zhu Dahuan, Xu Qiu, Cheng Jigui
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Fu Kaichao, Xu Dang, Sang Changcheng, Chen Ruizhi, Chen Pengqi, Zhu Dahuan, Xu Qiu, Cheng Jigui
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Tritium transfer from neutron-irradiated lithium to yttrium and gas phase
Mori Yuuka, Katayama Kazunari, Haruta Junnosuke, Kuniyoshi Mario, Katayama Shota, Fukada Satoshi, Akashi Kenta, Takeishi Toshiharu, Oyaidzu Makoto, Inuma Yuto
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A Sr–Ga Oxy-Hydroxide with High Thermal Stability: Unraveling Its Characteristic Hydrogen-Bond Network
Asai Yusuke, Nishihara Yuto, Kokubo Yoko, Arai Kenji, Ohishi Kosaku, Ogawa Satoshi, Saito Miwa, Nambu Yusuke, Avdeev Maxim, Kimoto Koji, Goo Zi Lang, Sugimoto Kuniyoshi, Inada Miki, Hayashi Katsuro, Motohashi Teruki
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Debye temperatures of Au nanoparticles supported on TiO_2 and SiO_2 estimated from ^{197}Au Mössbauer spectroscopy
Kobayashi Yasuhiro, Ohashi Hironori, Seto Makoto
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Sang Changcheng, Fu Kaichao, Xu Dang, Chen Ruizhi, Chen Pengqi, Lu Yingwei, Xia Yonghong, Xu Qiu, Cheng Jigui
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Effect of L_{12} precipitates on hydrogen-induced defects and hydrogen embrittlement behavior in CoCrFeNi-based high-entropy alloys
Ye Fengjiao, Chen Wei, Wu Haibiao, Zhu Te, Zhang Peng, Xu Qiu, Cao Xingzhong
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Hiroyasu Sasaki, Yojiro Oba, Kosuke Hiroi, Masato Onuma, Jun Uzuhashi, Tadakatsu Ohkubo
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Sang Changcheng, Fu Kaichao, Xu Dang, Chen Ruizhi, Chen Pengqi, Lu Yingwei, Xu Qiu, Cheng Jigui
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Enhanced mechanical properties of a copper nanoparticle-reinforced low-activation high-entropy alloy and its implications for irradiation resistance

Zhu Te, Xia Yuanhang, Wang Qianqian, Fan Ping, Zhang Qiaoli, Ma Hailiang, Yuan Daqing, Wang Baoyi, Xu Qiu, Cao Xingzhong

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Enhanced thermal stability and irradiation resistance of ODS-W/CuCrZr joints by interlayer employ and interface improvement

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Changes in defect structure in mechanically polished A533B steels by ion irradiation using slow positron beam techniques

Sato Koichi, Yabuuchi Atsushi, Kinomura Atsushi, Xu Qiu, Tsuchida Hidetsugu

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Yoshie T., Kinomura A., Nishimura T., Hori F.

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In-situ measurement of stress relaxation process in hydrogenated bulk Fe by gamma-ray induced positron annihilation spectroscopy

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Local structure analyses of oxygen impurities and vacancy-type defects in oxygen doped CrN epitaxial films

Kitaura Mamoru, Michishio Koji, Kinomura Atsushi, Masai Hirokazu, Hashimoto Yusuke, Yamamoto Yuta, Matsushita Tomohiro, Watanabe Shinta, Happo Naohisa, Kimura Koji, Hayashi Koichi, Ishizaki Manabu, Suzuki Tsuneo

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Nanostructural Evolution in Stainless Steel fabricated by Laser-based Powder Bed Fusion: A Combined SEM/EDX and Positron Lifetime Study

Kobayashi Keita, Kumagai Kazuhiro, Kasashima Haruka, Morishita Kazuyuki, Yasokawa Yukie, Yabuuchi Atsushi, Ito Kenji

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Dendritic crystals of α -MoO₃

Yang Ying, Nanko Makoto, Chu Ngo Minh, Do Thi Mai Dung, Nakayama Tadachika, Niihara Koichi, Suematsu Hisayuki

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Temperature variation of the magnetic properties of mechanochemically synthesized (Fe₂O₃)_{1-x}(Al₂O₃)_x solid solutions through the Mössbauer spectroscopy

Luo Dong, Nakaishi Hayato, Yabutsuka Takeshi, Kitao Shinji, Seto Makoto, Takai Shigeomi

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Exploration for the physical origin and impact of chemical short-range order in high-entropy alloys: Machine learning-assisted study

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Engineering Fe(II) spin-crossover/2D reduced graphene oxide heterostructures for tunable cooperativity, magnetic coupling, and conductance switching

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Precipitate-enhanced deuterium retention and desorption resistance in a $W_{10}Ta_{20}Ti_{30}V_{35}C_5$ refractory high-entropy alloy
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Radiation-Induced Fe²⁺-Dependent Oxidative Membrane Damage and Pedagogical Framework in Environmental Dynamics (Faculty of Agriculture)

Shinya Kato

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Papers

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Dosimetric performance of cone beam CT-guided adaptive carbon-ion radiotherapy with daily replanning for pancreatic cancer
Klassen Lukas Fabrizio, Hirashima Hideaki, Iramina Hiraku, Iwai Takahiro, Yoshimura Michio, Tanaka Hiroki, Mizowaki Takashi, Nakamura Mitsuhiko
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Preparation of ¹⁰B-enriched nitroimidazole derivative by click reaction as a functional drug for hypoxia-targeting boron-neutron capture therapy

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Zinc-dependent prothrombin activator from Rhabdophis tigrinus venom with extended substrate specificity

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Base Pair Contributions to DNA Duplex Thermodynamics

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Association-dissociation behavior of clock protein complexes in solution

Ken Morishima, Rintaro Inoue, Masaaki Sugiyama

Proceedings of the 60th KURNS Scientific Meeting, Kumatori, Japan/Online (Jan. 29-30, 2026) 25 (in Japanese)

Conformational dynamics of plant R-SNARE proteins revealed by small-angle X-ray scattering and analytical ultracentrifugation

Hirokazu Yagi, Shunsuke Asai, Maho Yagi, Ken Morishima, Rintaro Inoue, Masaaki Sugiyama, Koichi Kato

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Elucidation of the As(III)-specific aggregation process of the novel As(III)-aggregating protein GatB

Shigeko Kawai-Noma, Chiho Goto, Koji Ootsuka, Tatsuya Niwa, Yu-ki Tanaka, Takuho Saito, Natsuki Suda, Ken Morishima, Masahiro Shimizu, Atsuyuki Ito, Satoshi Ogasawara, Takeshi Murata, Masaaki Sugiyama, Katsumasa Kamiya, Shiki Yagai, Yasumitsu Ogura, Hideki Taguchi

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Masahiro Shimizu

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7. Neutron Capture Therapy

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Yasushi Saito

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Shimizu Miho, Sugai Wataru, Ohto-Fujita Eri, Atomi Aya, Nogawa Norio, Takamiya Koichi, Yoshinaga Hisao, Asano Yoshihide, Yamashita Takashi, Sato Shinichi, Enomoto Atsushi, Hatakeyama Nozomi, Yasuda Shunsuke, Tanaka Kazuya, Atomi Tomoaki, Harada Kenji, Hasebe Yukio, Watanabe Toshiyuki, Atomi Yoriko

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Development of synthetic method for partially oxidized phthalocyanine complexes extensible to minor actinides
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Lu-177 preparations

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Synthesis and adsorptivity of novel bifunctional monoamide-diamide resin for selective separation of actinyl Ions
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Hiroki Nakada, Yoshitaka Kasamatsu, Koichi Takamiya, Makoto Inagaki

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Tsutomu Miura

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Neutron instruments study for the new research reactor at the “Monju” site Nobuhiro Sato, Makoto Inagaki, Daisuke Ito, Yusuke Nambu, Yuji Kawabata, Koichi Takamiya, Masahiro Hino, Masaaki Sugiyama

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高見清氏を偲んで

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