VIII-Ⅱ-1. Project Research

Project 5

PR5 Trace Elemental Analysis Using Research Reactor

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Objective: Various elemental analysis methods have been performed in research fields such as chemistry, physics, biology, geochemistry, environmental science. Neutron activation analysis (NAA) is one of the most sensitive and accurate method for the elemental analysis. Many users apply NAA method for elemental analysis because it requires no sample preparation and is multi-elemental analysis method. Many kinds of elements can be determined using this method, but it is difficult to analyze if a radioisotope which is produced by neutron induced reaction has too short or too long half-life. And if there exist too many elements in a sample matrix such as biological samples, the accuracy of gamma-ray spectrometry decrease because of the Compton effect and overlap of photo peaks. In order to negotiate these disturbances, various techniques have been developed, for example, Anti-Compton method can reduce Compton background caused by short-lived products.

Research Subjects: In this project research, various efforts to increase the precision of NAA method were planned. One side of the efforts is developing and installing new instruments for NAA experiments. Prompt Gamma-ray Analysis (PGA) system and anti-Compton counting system have been developed at KURRI. The PGA system will be connected with Super Mirror guide tube which can supply high-quality thermal neutrons. The anti-Compton counting system will be installed at next to a pneumatic capsule transporting system to detect short-lived nuclei effectively without the Compton background. Another side of the efforts is developing and applying new NAA techniques to actual elemental analysis experiments. The respective subjects of the research groups of this project are described as follows;

P5-1: Neutron distribution measurements for high-precision NAA

P5-2: Development of prompt gamma-ray analysis apparatus in KURRI

P5-3: Antimony and arsenic concentration distribution in soil samples around a mine and a smelter

P5-4: Neutron activation analysis of elements in mice hair dosed green tea extract

P5-5: Neutron activation analysis of Ag and Se in biological samples by short life nuclides

P5-6: Biodistribution of gadolinium-based contrast agent, and concentration of trace elements in normal and nephrectomized rats

Results: The development of the anti-Compton counting system has been completed and most appropriate measurement conditions of the system has been examined. In the research theme of P5-5, determination of short-lived products in biological samples has been tried using the counting system. PGA system has been developed in the research group of P5-2. And actual elemental analysis experiments have been performed by groups of P5-3 (environmental science), P5-4 (biology), and P5-6 (biomedical science). The brief summaries of the performed subjects are as follows;

P5-1: Measurements of neutron distributions at neutron irradiation holes of KUR has been tried using developed neutron irradiation targets. The neutron distributions of Pn-1, -2, -3, and TC-Pn have been measured. In the cases of Pn-1, -2, -3, it is found that there are several percents of variation in neutron flux along the irradiation capsule. On the other hand, there is about 10% difference in neutron flux between top for each adjacent irradiation position.

P5-2: The electronics for detecting prompt gamma-rays with anti-coincidence technique has been checked, and reduction of background gamma-rays emitted from the supermirror neutron guide tube and neutron irradiated materials surrounding a Ge-detector has been tried. For the background gamma-rays from the guide tube, lead shield was added. Tiles of ^{6}LiF and ^{nat}LiF were placed as a neutron shield. And also cadmium and boron-containing polyethylene plates were placed for neutron shielding. Because the background gamma-rays observed by the Ge-detector were reduced by those arrangements, the detection sensitivity of the PGA system was improved.

P5-3: Characteristics of antimony contamination in soils around an antimony smelter in contradistinction to arsenic which is the homologous element of antimony has been studied by NAA method.

P5-4: A promotion of the exhaust of Cu from the inside of mice body by polyphenol was confirmed by NAA experiments performed at KUR and HANARO before. In the present work, the check experiments have been carried out using mice's hair at KUR.

P5-5: In neutron irradiations of biological samples, short lived nuclei such as ^{24}Na are produced and disturb the gamma-ray spectrometry by Compton effect. For avoiding this problem, measurements using the gamma-ray counting system conjugated with Compton suppression system has been tried.

P5-6: Distribution of gadolinium and concentration of some minerals in selected tissue of two type rats has been measured. One is normal, and the other is partially nephrectomized, which is used as pseudo renal impairment model. As a result, it is not sufficient to evaluate and discuss the relationship between Gd and in-vivo trace elements due to small number of animals. Therefore, this study will be continued to achieve with statistically-warranted value of the biodistribution of "exogenous" Gd and the concentration of "endogenous" trace elements in normal and nephrectomized rats.

採択課題番号 22P5　　原子炉中性子を用いた微量元素分析　　プロジェクト

（京大・原子炉）高宮幸一

PR5-1 Neutron Distribution Measurements for High-precision NAA

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INTRODUCTION: Measurement of a neutron flux and distribution for a neutron irradiation facility is important for neutron irradiation experiments such as neutron activation analysis, radioisotope production, etc. In the case of the measurement at irradiation holes of research reactors, activation method is most convenient because placing and retrieving monitor materials is easy [1]. Metallic wires and foils are often used as monitor materials in the activation method [2] [3]. In this method, monitor materials are transported to neutron irradiation holes using such as pneumatic transport tubes. After neutron irradiation at the irradiation hole, activated monitor materials are sent back to a laboratory room. A neutron flux can be determined by measuring activities of the irradiated monitor materials. In the previous report [4], new preparation method of a neutron monitoring material using an ink-jet printer has been developed. In this method, gold solution is applied on a printing sheet using an ink-jet printer to prepare a neutron monitor material. The prepared monitor is thin enough to ignore the self-absorption in neutron irradiation and self-shielding effect in gamma-ray measurement and easy to handle. Because a target material of gold for neutron activation was applied on a printing sheet using the ink-jet printer in this method, the position of the target material can be adjusted easily and correctly. In this work, measurements of neutron distributions at neutron irradiation holes of KUR (Kyoto University Research Reactor) has been tried.

EXPERIMENTS: A gold standard solution, 2 mL of 1,000 mg Au/L solution, and 2 mL of a mixed organic solvent were mixed. The mixed solution was poured in an empty ink cartridge, and the cartridge was attached to an ink-jet printer. The mixed solution in the cartridge was sprayed out to a PET sheet under control of a PC as usual document printing. As a neutron monitor for neutron distribution measurement, a rectangular PET film on which the mixed solution were applied on one side was prepared. Because a PET film contains antimony uniformly, two kinds of activities, ^{198}Au and ^{122}Sb, were produced in the neutron monitor by neutron irradiation.

A measurement of axial neutron distribution at the position of irradiation rabbits in neutron irradiation holes of KUR (Pn-1, -2, -3 and TC-Pn) were performed. The PET pieces were inserted into the rabbits. After the neutron irradiation at the irradiation holes, the distribution of produced activities were measured by imaging plate method.

RESULTS: The measurements of axial neutron distributions at the irradiation holes of KUR were tried. The neutron distributions observed in this work are the distributions of the neutrons in the energy ranges which can induce the neutron capture reactions of ^{197}Au and ^{121}Sb. The activity variations of ^{198}Au and ^{122}Sb which were produced in the neutron monitor irradiated by TC-Pn are shown in Fig. 1. In the case of TC-Pn irradiation, three rabbits can be irradiated in series at the same time. In this figure, the horizontal axis shows the range from the irradiation-tube-end of TC-Pn. The vertical axis indicates the relative activities of produced ^{198}Au (open circles) and ^{122}Sb (closed). As shown in this figure, the detailed neutron distribution can be observed in this method, and it was found that there is about 10% variation in the neutron flux at each irradiation position of TC-Pn.

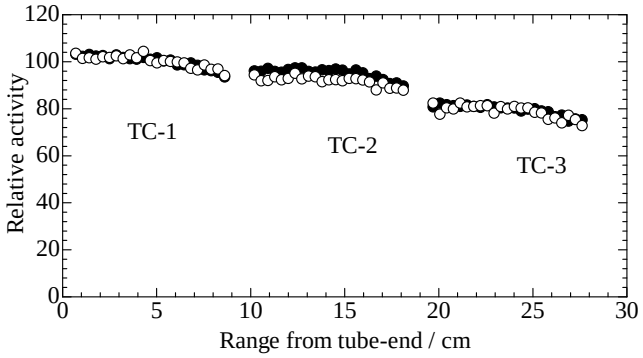

Fig. 1. Neutron distributions at three irradiation position of TC-Pn observed by neutron capture reactions of gold and antimony.

Acknowledgements: This work was supported by JSPS KAKENHI 21760707.

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採択課題番号 22P5-1　中性子放射化により生成する短寿命核種の定量法の開発　プロジェクト
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PR5-2 Development of Prompt Gamma-ray Analysis Apparatus in KURRI

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INTRODUCTION: Prompt gamma-ray analysis (PGA) apparatus has been developed in KURRI from 2008. This development is promoted by Nuclear Energy Research Initiative, Research Reactor and Hot Laboratory Utilization program, which is titled as "Improvement of Utilization on KUR and HL in KURRI", under the project in Ministry of Education, Culture, Sports, Science and Technology. In PGA, it is necessary to observe the gamma rays emitted promptly from the samples through neutron induced reaction. The neutron irradiation for PGA in KURRI will be carried out at B4 reactor site of Kyoto University Reactor (KUR). The purpose of this project is to install the PGA in KURRI and to apply the total micro-element analysis system including the PGA and the short-lived nuclides measurement system, which has been developed under the same program, to the research on medical science, archeology, cosmo- and geo-science etc., in the future. In terms of the field of nuclear energy, this project is also directed toward the ideal model of small neutron source in university. In this report, the state of preparation for installation of the PGA system in KURRI is described.

PREPARATION: In 2008, the signal processing system for the anti-coincidence circuit was built with the NIM electronics. The anti-coincidence circuit consists of timing filter amplifier (TFA), constant fraction discriminator (CFD), gate and delay generator (GDG), Quad Logic (f-in/f-out) and time-to-amplitude convertor (TAC), as shown in Fig. 1. This circuit will follow the gamma-ray spectroscopic instrument which consists of an n-type high-purity germanium detector (Ge) and bismuth germinate scintillation detectors (BGO) with high voltage (HV), photo multiplier (PM) and pre amplifier (pre-AMP). The signals from this circuit will be finally processed by pulse height analyzer (PHA).

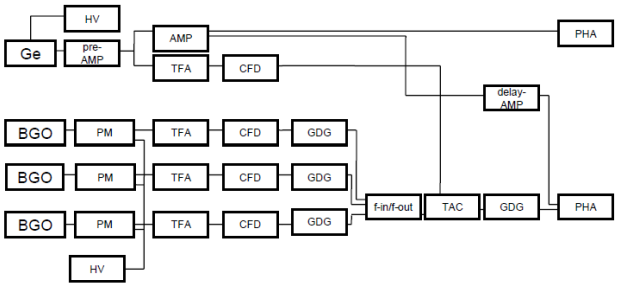

Fig. 1. Anti-coincidence system in KURRI-PGA

In 2009, n-type high-purity germanium detector (CANBERRA GR3019), multi channel analyzer (MCA, SEIKO EG&G, 7600-000) including HV (7600-300) and PHA (7600-510), and spectroscopy amplifier and gated integrator (ORTEC 673) were installed for the PGA system. The lead and LiF shieldings from background gamma rays and scattered neutrons, respectively, were also prepared for the gamma-ray spectroscopic instrument.

PROGRESS in 2010: After restart of KUR, the development of Prompt Gamma-ray Analysis (PGA) system was planned to make it possible newly using KUR to measure some elements such as H, B, Si, S, etc. which were not able to be measured by the conventional NAA. The new PGA system has been developed at the B-4 beam hall, where a supermirror neutron guide tube was installed in 1991 [1]. Since the B-4 beam hall has the end of the supermirror neutron guide and is located outside the reactor room, the B-4 is appropriate for the installation of the PGA system which requires low gamma-ray background. The supermirror neutron guide is extended by 11.7 m and has a cross section of 1×7.4 cm^2 [1]. Using the gold-activation method, the neutron fluence rates in the B-4 at 1 MW and 5 MW operation of KUR are measured to be 1×10^7 n· cm^{-2}· s^{-1} and 5×10^7 n · cm^{-2}· s^{-1}, respectively. Additionally, in the B-4 beam hall which has relatively large experimental area, a neutron radiography facility as well as PGA has been installed and then, the other experimental studies in neutron physics and even in nuclear fission experiments also has been performed [2].

At the present stage, PGA at B-4 has been carried out using single germanium detector and the shielding surrounding the detector. The shielding consist of ^{6}LiF, ^{nat}LiF, boron-containing polyethylene, and Cd for neutron shielding and Pb for gamma, and made the spectral background lower. The sensitivity of B, which is the most sensitive for PGA, was estimated to be 1.1 cps· mg^{-1} with an uncertainty of 1.5% using a chemical reagent of B. To improve the sensitivity of B and to apply PGA to other elements; H, Si, S and so on, compton –suppression system is going to be equipped to the PGA system at KUR. To enable the utilization of both PGA and the conventional NAA complementarily using KUR, further studies along this line are in progress.

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採択課題番号 22P5-2　　京大炉における即発ガンマ線分析装置の開発　　プロジェクト

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PR5-3 **Antimony and Arsenic Concentration Distribution in Soil Samples Around a Mine and a Smelter**

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INTRODUCTION: Antimony (Sb), a naturally occurring trace element in soil, is becoming a pollutant because of its increasing industrial use. Flame retardants, paints and pigments are important industrial products, and 13,279 t of antimony trioxide (Sb_2O_3) was in demand at 2006 in Japan [1]. It is pointed out that these important products, flame retardants, paints and pigments, are end up in the garbage and loaded in the environment. Antimony is categorized as items to be monitored in Japan, and the guideline values of antimony are under 0.02mg/L and 0.015mg/L for the public water quality and the tap water quality, respectively. As just described, antimony is needed to be studied about its behavior in the environment because of its large amount of usage and the strict guideline values. In this study we attempt to analyze characteristics of antimony contamination in soils around a antimony smelter in contradistinction to arsenic which is the homologous element of antimony.

EXPERIMENTS: The soil sample was collected around an antimony mine and smelter at Tsugu-city in Aichi-prefecture where were closed in 1960s (Fig. 1). The collected soil was dried in air and sieved through a 2mm sieve. About 100 mg soils of these pretreatment soils were prepared for INAA. Soil samples were irradiated by neutron at Pn-2 of KUR (thermal neutron flux: 5.5×10^{12} n･cm^{-2}･s^{-1}). BCR-176 (city waste incineration ash; certified value of antimony is 412 mg･kg^{-1}) for comparison standard of antimony and SRM-2711a (Montana II soil, moderately elevated trace element concentrations; certified value of arsenic is 107 mg･kg^{-1}) for that of arsenic were used. Irradiation time was 10 minutes, and after about one day for cooling, γ-ray spectrometry was conducted. Concentrations of antimony and arsenic were calculated by using comparison method.

RESULTS: Measurement results about antimony and arsenic in the soils were plotted in Fig. 2. Antimony concentrations were 7.2×10^2 to 2.6×10^3 (counting errors were within the range of 0.59 - 0.90 %) mg･kg^{-1} and the concentration levels are similar to previously reported values[2]. The changes in antimony concentration with depth differed between at Maibara reported before [3]

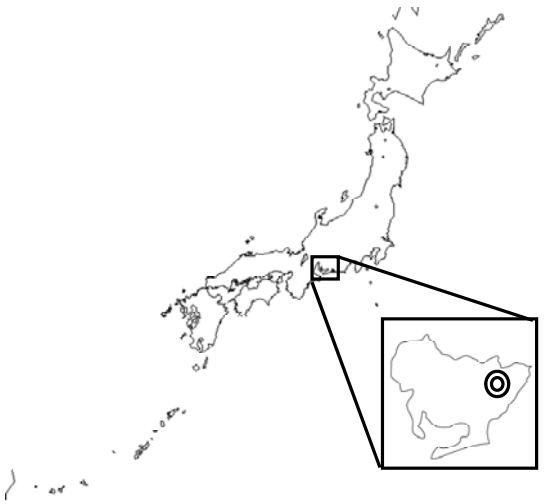

Fig. 1. Soil sampling point.

and at Tsugu. At Maibara, antimony concentrations

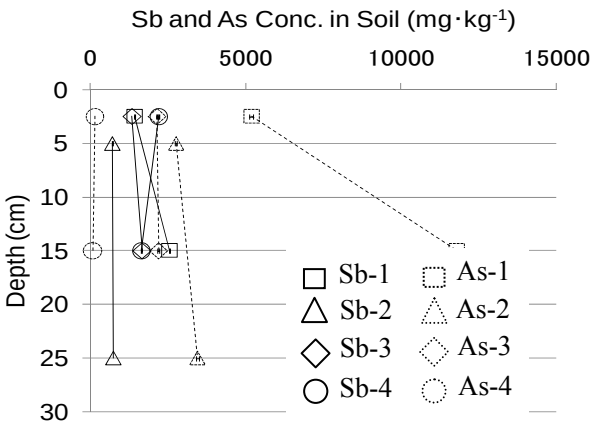

Fig. 2. Antimony and arsenic concentrations in soil samples at Tsugu area.

tended to decrease with increasing depth, but this concentration gradient did not occur at Tsugu. Arsenic soil concentrations were 8.1×10^1 to 1.2×10^4 (counting errors were within the range of 1.0 - 1.4 %) mg･kg^{-1}, and equal to or greater than that of antimony.

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採択課題番号 22P5-3　　土壌中微量環境負荷元素の汚染経路の解明に関する研究　プロジェクト
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PR5-4 Neutron Activation Analysis of Elements in Mice Hair Dosed Green Tea Extract

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INTRODUCTION: The plant is one of important raw material of the herbal medicine. Moreover, the effect of the herbal medicine is largely affected by the home and the quality of a used plant. The trace elements are considered to be related with the quality of herbal medicine, but the study about the trace elements in plant is not done widely. Therefore, the role of trace elements in herbal medicine is little clarified.

We confirmed that the polyphenol promotes the exhaust of Cu from the inside of the mice body by the activation analysis at KUR at HANARO in Korea before. We analyzed mice's hair again at KUR to confirm the previous result, and the results are reported here.

EXPERIMENTS: The green tea extract was administrated to the C57BL/6C (6 week age) constantly 4 weeks. The used concentration of green tea extract was 0.2 g/l (L group) and 2.0 g/l (H group). Water administration group (W group) was also examined as control. The body hair was collected as a reaping analytical sample on the 0th day of the intake beginning and at 7, 14, 21, and 28 days. The samples were subjected to the activation analysis at KUR after washing. In activation analysis, all samples were irradiated for 1 min in Pn-1, and gamma-ray energy spectra of samples were measured for 200 seconds by Ge detector.

RESULTS: One of the observed gamma-ray energy spectra was shown in Fig. 1 (hair from H group for 2 weeks after 2.0 g/l green tea extract administration). In Fig. 2, time-dependent change of Cu content in hair of each group was shown. From Fig. 2, it was observed that Cu was exhausted in H and L groups at early time by green tea extract administration.

From these results, polyphenol accelerated the excretion speed of Cu from the inside of the mice body. The mechanism of the acceleration in excretion of Cu is considered to be as follows, catechins in green tea extract formed the complex with Cu and accelerated the excretion speed of Cu from the inside of the mice body.[1,2] After 3 weeks passed, the same amount of Cu in hair was detected in W group. The reason of this result will be clarified in future study.

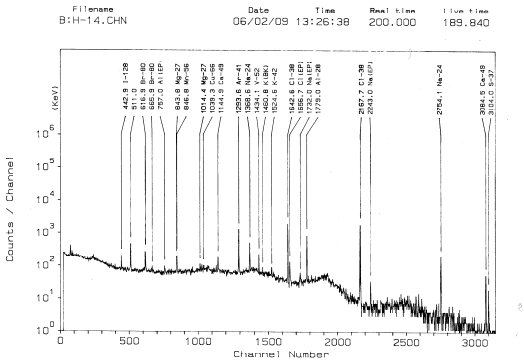

Fig. 1. Observed gamma-ray energy spectrum. Spectrum of H group of mice at 14 th day was shown.

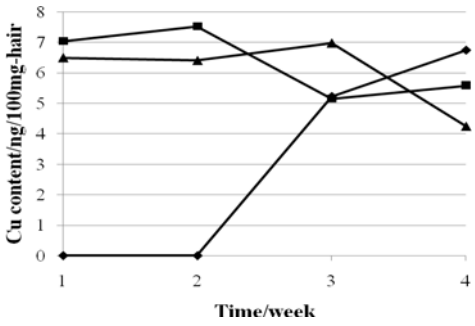

Fig. 2. Time-dependent change of Cu content in hair of mice ◆: W group, ▲: H group, ■: L group

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採択課題番号 P5-4 放射化分析による緑茶抽出物を摂取したマウス体毛中の元素分析プロジェクト
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PR5-5 Neutron Activation Analysis of Ag and Se in Biological Samples by Short Life Nuclides

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INTRODUCTION

Se and Ag are ones of trace elements in biological samples. There are not many analytical methods for these elements. This is one of the reason why the role of these elements for biology are not well known. Neutron activation analysis has high sensitivity for both Ag and Se. Usually Se-75 (half life = 119.77 d) and Ag-110m (half-life = 249.76 d) are used for analyzing Se and Ag. At the same time, both elements can produce Se-77m (half life = 17.45 s, gamma energy = 161.93 keV) and Ag-110 (half life = 24.6 s, gamma energy = 657.76 keV). Analytical results can be obtained quickly by using these short life nuclides. When biological samples are irradiated very shortly and gamma counting are done immediately after the irradiation, we will face the serious problem of high dead time induced from Na-24 mainly. For avoiding this problem, we have tried to use gamma counting system conjugated with Compton suppression system in KUR.

Fig.1. Photograph of Compton Suppression System

EXPERIMENTAL

Comparative standards were prepared from 1000 ppm Ag or Se standard solution for atomic absorption spectrometry (Wako Chemical Co., Japan) properly. 400 μL of each standard solution was dropped onto filter paper (1 cm $\times$ 1 cm) and dried under IR lamp. Then, the dried filter papers were wrapped doubly by polyethylene sheet. Comparative standard was irradiated for 10-30 seconds at Pn or Tc-Pn site in KUR. Immediately after the irradiation, irradiated standard was taken from plastic rabbit and brought to detector.

RESULTS

When we tried to use CSS system, at shortest 60 seconds were needed for taking sample out from rabbit, bringing it to the detector, fixing the detector to proper position, and start counting. It meant 4-5 and 2 half-lives for Se-77m and Ag-110, respectively. Even when comparative standards containing 10 μ g of Se or Ag, no effective peak for analysis could be found in gamma spectrum. CSS itself is powerful tool for analyzing trace elements in samples which contain high levels of NaCl in it. We should look for the condition both for inducing the activities of Se-77m and Ag-110, and for reducing the activities from matrix elements such as Na, Cl, Br, and so on. At the same time, cooling time should be set as shortest as possible. For analyzing short life nuclides such as Se-75 and Ag-110m, we should add some changes for CSS for making cooling time shorter than 30 seconds at longest. For another choice, other nuclides which half lives are longer than I minute should be the target for the analysis.

採択課題番号 22P5-5　短寿命核種による生物体中の Ag および Se の中性子放射化分析 プロジェクト
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PR5-6 Biodistribution of Gadolinium-Based Contrast Agent, and Concentration of Trace Elements in Normal and Nephrectomized Rats

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INTRODUCTION: Gadolinium (Gd)-based contrast agents have been used in medical MR-imaging. Recently, human nephrogenic systemic fibrosis (NSF) is reported in Gd-based contrast agent received patients with severe renal insufficiency [1, 2].

Gd has relatively high activation cross section for thermal neutron. Therefore, it is possible to detect sensitively by neutron activation analysis (NAA). NAA technique also quantitate essential trace element *in vivo*.

For prevention and protection of NSF, it is important to understand the mechanism of interaction between Gd and minerals. In this study, we measured distribution of Gd and concentration of some minerals in selected tissue of two type rats. One is normal, and the other is partially nephrectomized, which is used as pseudo renal impairment model [3].

EXPERIMENTS: Male 9-week-old Sprague‐Dawley rats were used in this study. Three rats were housed as normal, and another three rats were partially nephrectomized as pseudo renal impairment model. Each rat was administered 2.5 mmol Gd/kg b.w. Omniscan (gadodiamide) intravenously. Two days after administration, rats were sacrificed and dissected. The blood, femur, kidney, large intestine, liver, muscle, small intestine, and spleen were excised and weighted. The samples were freeze-dried and sealed into polyethylene bags for NAA. The sealed samples and quantitative standard of gadolinium were irradiated in Pn-3 for 2 minutes and for 1 hour, for short and long half-life radioisotopes production, respectively. The distribution of Gd and concentration of minerals were determined by using γ-ray spectrometry.

RESULTS AND DISCUSSION: The biodistribution of gadolinium 2-d after administration could be determined by ^{153}Gd ($T_{1/2}$ = 240.4 d), and was shown in Fig.1. The kidney showed high Gd retention behavior due to normal process of excretion of Gd. Skeletal Gd distribution of model rats was significantly higher than that of normal rats. All of three nephrectomized rats showed high Gd retention in each tissue compared to normal rats.

The concentration of minerals and halogens such as Na, Cl, K, Mn, Ca, Fe, Zn, and Br could be determined here. Among them, zinc had been considered to have relevance to unchelated Gd (free Gd^{2+} ion). Figure 2 compared the concentration of zinc in selected tissue between model and normal rats. Concentration of zinc in femur of model rats was higher than that of normal rats.

In this study, it is not sufficient to evaluate and discuss the relationship between Gd and in-vivo trace elements due to small number of animals. Therefore, this study will be continued to achieve with statistically-warranted value of the biodistribution of “exogenous” Gd and the concentration of “endogenous” trace elements in normal and nephrectomized rats.

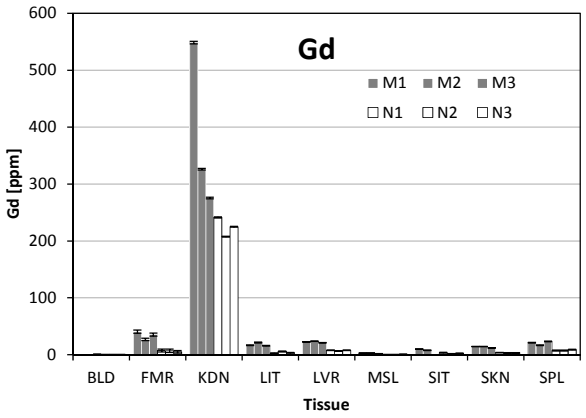

Fig. 1. Biodistribution of gadolinium in the blood, femur, kidney, large intestine, liver, muscle, small intestine, and spleen of three nephrectomized and three normal rats 2 d after agent administration.

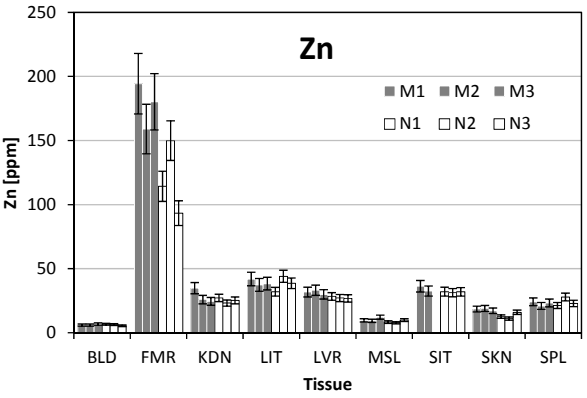

Fig. 2. Concentration of zinc in the blood, femur, kidney, large intestine, liver, muscle, small intestine, and spleen of three nephrectomized and three normal rats 2 d after agent administration.

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-アクチバブルトレーサー法の応用-（原子炉中性子を用いた微量元素分析）
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