VIII-Ⅱ-1. Project Research

Project 1

PR1

Trace Elemental Analysis Using Research Reactor

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Objective: Elemental analysis is the most fundamental experiment in many fields of research, for example, chemistry, physics, biology, environmental science, medical science, and there are various method for elemental analysis. Neutron activation analysis (NAA) is one of the most sensitive and accurate analytical method. But not all elements can be determined by NAA method with high accuracy. In NAA method, analysis samples are irradiated by neutrons to produce radioactive isotopes in samples, and energy spectra of gamma-rays emitted from the isotopes are measured to determine elements contained in samples. If produced radioisotopes have too short or too long half lives, obtaining enough statistics in gamma-ray measurements becomes very difficult. And if there exists 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 complicate photo peaks. In order to eliminate these disturbances, various techniques have been developed, for example, Anti-Compton method can reduce Compton background caused by short-lived products.

Research Subjects: Two types of approach, hardware development and methodological improvement, have been performed to increase the precision of NAA method in this project research. In the approach by hardware development, Prompt Gamma-ray Analysis (PGA) system and Anti-Compton Spectrometer (ACS) system have been developed and installed. The PGA system is connected with Super Mirror Neutron Guide Tube at B-4 beam hole of KUR which can supply high-quality thermal neutron beam. The ACS system is installed at next to two of pneumatic capsule transporting systems (Pn-1 and TC-Pn) of KUR to detect short-lived nuclei effectively without the Compton background. In another approach of methodological improvement, developing and applying new NAA techniques have been tried to actual elemental analysis experiments. The respective subjects of the research groups of this project are described as follows;

P1-1: Measurements of neutron spectrum transition for high-precision NAA

P1-2: Development of prompt gamma-ray analysis apparatus in KURRI

P1-3: Antimony and arsenic concentration distribution in depth direction around a smelter

P1-4: Neutron activation analysis of elements in mineral type natural medicine ‘Ryukotsu”

P1-5: Multi-element analysis of domestic green tea by neutron activation analysis

P1-6: Biodistribution of gadolinium-based contrast agent, and concentration of manganese and zinc in normal and nephrectomized mice

Results: Hardware developments have been completed. And applications of the hardware and methodological approaches for NAA experiments have been performed in the final period of this project. The brief summaries of the performed subjects are as follows;

P1-1: The gold applied PET sheets as neutron monitors enclosed in irradiation rabbits were irradiated to examine the neutron energy spectra and its variation in the irradiation hole of KUR. The five monitors were irradiated by neutrons in Pn-2 irradiation hole in 10 minutes with an interval of 1.5 hours after the thermal power of KUR reached 1 MW. The axial distributions of activities of ^{198}Au and ^{122}Sb in the rabbit were estimated by processing the picture obtained from the imaging plate. It was found that neutron distributions estimated by each isotope change over time, and magnitude of the change is different between ^{198}Au and ^{122}Sb. This means the neutron spectra at the irradiation hole became softer while the five times irradiations. The magnitude of change is small but nonnegligible in highly precise NAA.

P1-2: Both neutron radiography and elemental analysis for a pottery sample have been performed simultaneously. For the elemental analysis, PGA installed at B-4 beam line was used. Since elemental analysis for such as the pottery without the appropriate reference standards at the present stage of the installed system, a part of the sample was subjected to NAA to determine the elements.

P1-3: Antimony pollution characteristics in soil samples collected around an antimony smelter at Maibara has been estimated using NAA method and compared to the pollution characteristics of arsenic. Obtained distribution of antimony shows a decreasing tendency with increasing the depth, on the other hand arsenic concentrations are considered to be background level.

P1-4: The relationship between elemental contents of mineral type raw material, called “Ryukotsu” and effectiveness is unclear. To clarify this relationship, NAA for Ryukotsu samples were performed under several conditions. The role of Ryukotsu in herbal medicine must be clarified using the obtained results in future studies.

P1-5: To estimate the amount of elements taken from drinking green tea, several kinds of domestic green tea were analyzed by NAA.

P1-6: The distribution of Gd and concentration of some minerals in selected tissue of four experimental condition applied mice was measured by NAA method. The nephrectomized mice showed high Gd retention in each tissue compared to normal mice. No correlation of “exogenous” Gd and “endogenous” Zn was confirmed. However, it is not sufficient to achieve with statistically-warranted value of the biodistribution of “exogenous” Gd and the concentration of “endogenous” and “exogenous” trace elements in normal and nephrectomized mice to clarify the correlation of Gd with trace elements.

採択課題番号 23P1　　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

PR1-1 Measurements of Neutron Spectrum Transition for High-precision NAA

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INTRODUCTION: Distributions of neutron flux and energy spectrum at neutron irradiation fields affect estimations of nuclear reaction rate. Precise measurement of the distributions is important for neutron irradiation experiments such as neutron activation analysis, radio isotope production, etc. A new measurement method for neutron flux distributions using a gold-applied thin sheet as a neutron monitor has been developed [1]. In this method, gold solution is applied on a printing sheet by a commercially available ink-jet printer to prepare a neutron monitor materials. The prepared neutron monitor is thin enough to ignore the self-absorption in neutron irradiation and self-shielding effect in gamma-ray measurement. The distribution of neutron flux can be visualized easily by means of an imaging plate in radiation measurement for the neutron-irradiated monitor. In the previous report, the neutron distributions of neutron irradiation holes (Pn-1, -2, -3, and TC-Pn) of Kyoto University Research Reactor (KUR) has been measured by this method. The gold solution was applied on the half-side of a PET sheet to prepare the neutron monitor in the work, and distributions of radioactivity of products (^{198}Au and ^{122}Sb which is produced by neutron capture reaction of ^{121}Sb containing in a PET film) was measured using imaging plate. A difference of the production ratio of radioactivity of ^{122}Sb to that of ^{198}Au indicates a difference of spectra of irradiated neutrons. In the present report, distributions of the neutron energy spectrum were examined by the monitor.

EXPERIMENTS: The gold applied PET sheets as neutron monitors enclosed in irradiation rabbits were irradiated to examine the neutron energy spectra and its variation in the irradiation hole of KUR. The detailed preparation scheme of the monitor is described in the previous report. The five monitors were enclosed in separated irradiation rabbits and irradiated by neutrons in Pn-2 irradiation hole in 10 minutes with an interval of 1.5 hours after the thermal power of KUR reached 1 MW. After cooling time of one day, the irradiated monitor was separated to two pieces, one is applied on by gold and the other is not, and distributions of radioactivities produced in the monitor pieces (^{198}Au and ^{122}Sb for the former, and ^{122}Sb for the latter) were measured by the imaging plate for 5 min of the exposure time.

RESULTS: The axial distributions of activities of ^{198}Au and ^{122}Sb in the rabbit were estimated by processing the picture obtained from the imaging plate and shown in Fig.1. The relative radioactivities in each 3.5 mm range are shown in this figure. Activities are normalized to the average activity on reaching the thermal power 1 MW. Symbols indicate the time variation of the distributions. It is found the time variation for ^{122}Sb production is larger than that for ^{198}Au. The transition of activity ratio of ^{122}Sb / ^{198}Au shows the change of irradiated neutron spectra. The time variation of activity ratio of ^{122}Sb / ^{198}Au is shown in Fig. 2. The values are normalized to the ratio on the reactor power reaching 1 MW in this figure. The ratio decrease with the elapsed time and reaches 95% after 6 hours. This means the neutron spectra at the irradiation hole became softer while the five times irradiations. The magnitude of change is small but non-negligible in highly precise NAA.

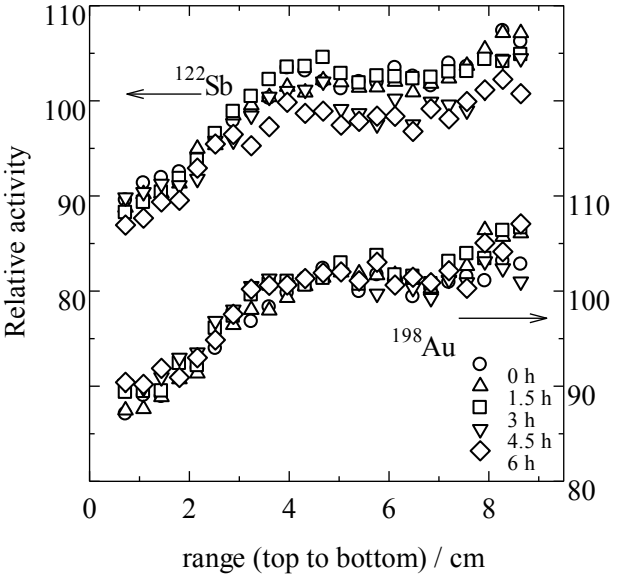

Fig. 1. The axial distributions of produced activities of ^{198}Au and ^{122}Sb in the rabbit.

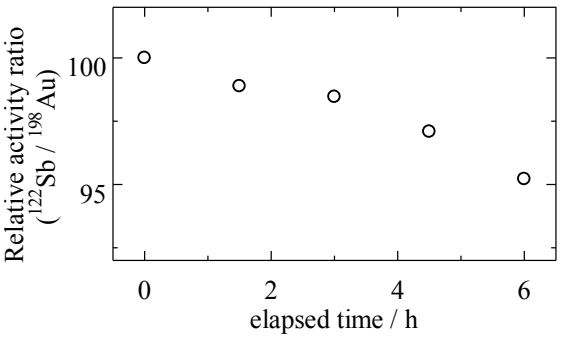

Fig. 2. The time variation of activity ratio of ^{122}Sb / ^{198}Au.

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採択課題番号 23P1-1　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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PR1-2 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: Kyoto University Research Reactor Institute has the neutron radiography facility at B-4 beam hole, where a supermirror neutron guide tube has been installed [1]. The details of neutron beam and the imaging devices for neutron radiography at B-4 beam hole were reported elsewhere [2]. At B-4 beam hole, the PGA system has been developed to make it possible to measure some light elements such as H, B, Si, S, etc. which were not able to be measured by the conventional neutron activation analysis (NAA). Since the B-4 beam hole 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. Consequently the B-4 becomes the unique facility, where not only neutron radiography but also PGA and even in nuclear fission experiments can be performed.

MOTIVATION: Neutron radiography has been applied to industrial quality tests and to various research activities as a non-destructive imaging technique utilizing neutrons. Neutrons have many advantages for imaging over visible light, X-rays or electrons. The distinctiveness of neutron radiography rests on the characteristic interactions between neutrons and nuclei. This leads to some special relationships between the material and geometrical properties of an object and the neutron radiographic image. The neutron image provides integrated absorption rates of neutron in the object along the neutron path, but no information about elemental components for material itself.

PROGRESS in 2011: In this work, we attempt to get the neutron radiographic image and also to get information about elemental components simultaneously in the pottery sample using neutron. To get information about elemental components, PGA at the B-4, which has been carried out using a single germanium detector and appropriate shielding surrounding the detector, is utilized [3]. The pottery sample is the replica of the archeological sample, supplied by IAEA. The sample is measured in this work in order to confirm the applicability of large sample analysis by PGA for inhomogeneous bulk archeological samples in the future.

Since it is difficult to get results of elemental analysis for the pottery by PGA without the appropriate reference standards at the present stage, a part of the pottery sample was crushed and pulverized, and was subjected to instrumental NAA using KUR. Measured elemental contents obtained by NAA in the five part of the pottery sample are shown in Table 1, and they will be compared to PGA results in the future.

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[2] Saito Y, Sekimoto S, Hino M, Kawabata Y (2011) Nucl Instr and Meth A. 651:36-41

[3] Sekimoto S, Saito Y, Homura Y, Ebihara M, Kawabata Y (2012) 7th International Topical Meeting on Neutron Radiography, June 16-22, Kingston, Canada.

Table 1. Measured elemental contents in the pottery sample

	Pottery_1 Contents (%)	Pottery_2 Contents (%)	Pottery_3 Contents (%)	Pottery_4 Contents (%)	Pottery_5 Contents (%)
Na	1.93 ± 0.06	2.11 ± 0.08	2.02 ± 0.08	2.10 ± 0.07	2.06 ± 0.09
Mg	2.13 ± 0.17	2.17 ± 0.15	2.11 ± 0.16	2.17 ± 0.15	2.33 ± 0.16
Al	7.81 ± 0.28	7.80 ± 0.12	8.62 ± 0.13	8.48 ± 0.13	8.76 ± 0.15
Ca	3.2 ± 0.5	2.7 ± 0.4	3.7 ± 0.5	3.4 ± 0.4	3.2 ± 0.5
Ti	0.44 ± 0.11	0.45 ± 0.08	0.40 ± 0.08	0.45 ± 0.08	0.45 ± 0.09
V	0.013 ± 0.001	0.013 ± 0.001	0.015 ± 0.001	0.014 ± 0.001	0.015 ± 0.001
Mn	0.085 ± 0.001	0.089 ± 0.001	0.087 ± 0.001	0.087 ± 0.001	0.091 ± 0.001

採択課題番号 23P1-2　　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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PR1-3

Antimony and Arsenic Concentration Distribution in Depth Direction around a Smelter

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INTRODUCTION: Around antimony smelters and mines, very high levels of antimony pollution have been detected and reported[1] [2] [3]; antimony pollution was reported to affect the health of residents living in the vicinity of a smelter [4]. Antimony smelters are usually built in areas remote from residential areas. City waste incinerators, which are now established in every local government area, are more important sources of antimony pollution than smelters for the general public. Because samples collected around antimony smelters contain high levels of antimony, design of an experimental system to measure the samples is easy. We aimed to study antimony pollution characteristics and behavior around smelters and a mine, and apply the results to antimony environmental contamination in general. In this study, we estimated antimony pollution characteristics in soil samples collected around an antimony smelter at Maibara (Shiga-pref.) by comparing with pollution characteristics of arsenic.

EXPERIMENTS: The soil sample was collected around an antimony smelter at Maibara-city in Shiga-prefecture where health hazards had been occurred in 1970's (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 Maibara soils were plotted in Fig. 2. Antimony concentrations showed a decreasing tendency with increasing depth, on the other hand arsenic concentrations at Maibara area were considered to be background level, and only little changes of concentrations along with depth were recognized.

Fig. 1 Soil sampling point

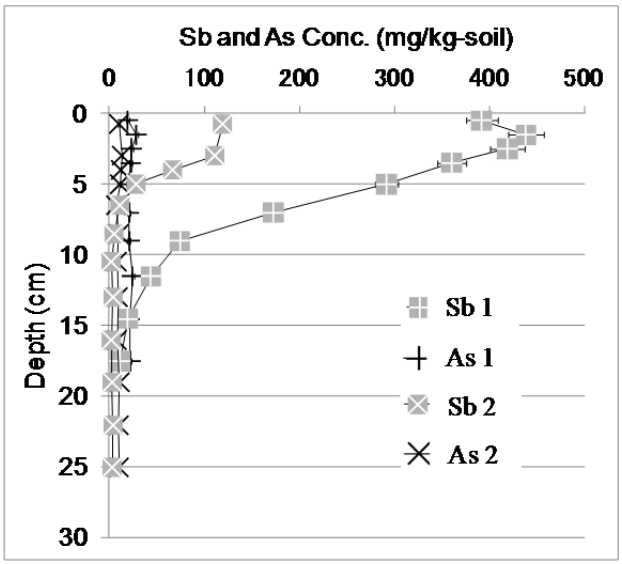

Fig. 2 Antimony and arsenic concentrations in the soil samples at Maibara area. Sb 1 and As 1, or Sb 2 and As 2 are respectively derived from the same core sampling.

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[2]Ainsworth N, Cooke J A, Johnson M S (1991) Water, Air and Soil Pollution 57-58: 193-199
[3]Wilson N J, Craw D, Hunter K (2004) Environmental Pollution 129: 257–266
[4]Ishida N, Katayama Y (1978) Kagaku 48: 509-515 (in Japanese)

採択課題番号 23P1-3　　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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PR1-4 Neutron Activated Analysis of Elements in Mineral Type Natural Medicine "Ryukotsu"

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4Museum, Osaka University

INTRODUCTION: The plant is one of the important raw materials of the herbal medicine. Moreover, the animal, reptiles, insect and minerals are also the important raw materials of the herbal medicine. The effect of the herbal medicine is largely affected by the home and the quality of the used raw materials. The metal elements are considered to be related with the quality of herbal medicine. Especially, the relationship between elemental contents of mineral type raw material, called "Ryukotsu" and effectiveness is unclear, and a study to clarify the relationship is hardly done [1].

To clarify this relationship, analyses of elements in Ryukotsu were done under several conditions. In this report, we show the obtained results.

EXPERIMENTS: The used Ryukotsu was commercial base material for herbal medicine. In preparation of herbal medicine, Ryukotsu and other raw materials were mixed and extracted by hot water. Four types of Ryukotsu were prepared depending on the extracting conditions, and subjected to activation analysis. Types 1, 2, 3 and 4 were Ryukotsu before extraction, after extraction without others, after extraction with others and type3 washed with water after extraction, respectively. In activation analysis, all samples were irradiated for 1 min and 60 min in KUR, and gamma-ray energy spectra of samples were measured for 200 seconds by Ge detector.

RESULTS: Ryukotsu is defined as the fossil of mammals at Mesozoic era. As standard sample for quantitative analysis for fossil, semi-quantitative analysis of elements in Ryukotsu was done in this study. The results about Ca and Al were shown Figs. 1 and 2, respectively.

From these results, Ca content in Ryukotsu was little changed by extraction in all conditions. However, the content of Al was largely changed depending on conditions.

Mn, Na, V, S and Cl showed behavior like the Ca. Zn showed behavior like the Al. The other elements had a large error, and their behavior did not become clear.

The role of Ryukotsu in herbal medicine must be clarified using these results in future studies.

REFERENCES:

[1] Ministry of Health, Labour and Welfare "*Nihon Yakkyokuho*" (2011), http://www.mhlw.go.jp/topics/bukyoku/iyaku/yakkyoku/

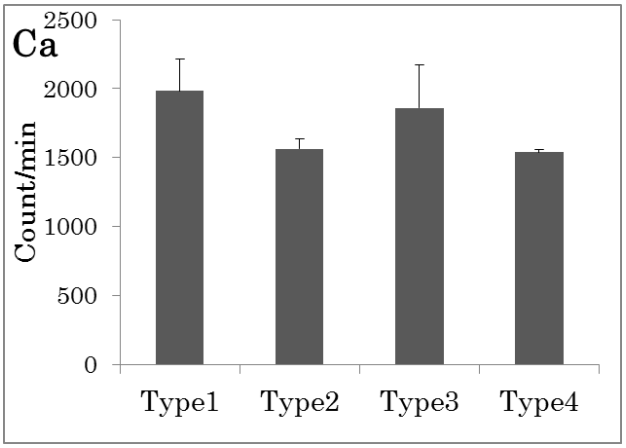

Fig. 1. Content of Ca in each type of Ryukotsu
Types 1, 2, 3 and 4 were Ryukotsu before extraction, after extraction without others, after extraction with others and type3 washed with water after extraction, respectively.

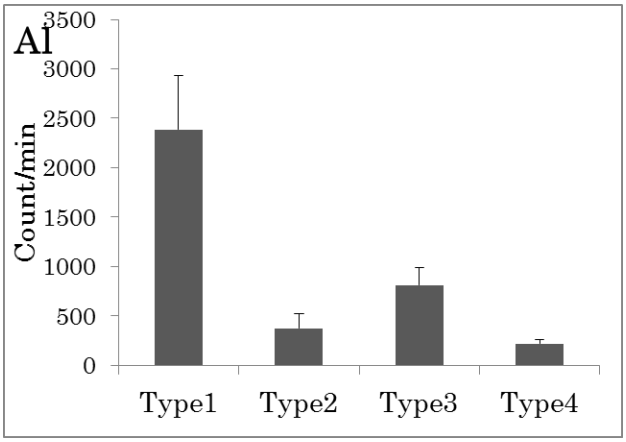

Fig. 2. Content of Al in each type of Ryukotsu.

採択課題番号　23P1-4　　放射化分析による鉱物生薬"竜骨"の元素分析　　プロジェクト
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PR1-5 Multi-element Analysis of Domestic Green Tea by Neutron Activation Analysis

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INTRODUCTION

Drinking green tea is taken daily well in Japan. To estimate the mount of elements taken from drinking green tea, several kinds of domestic green tea were analyzed by neutron activation analysis.

EXPERIMENTS:

<Samples>

Three different kinds of powder teas and seven different kinds of green teas were obtained from one green tea company. Five of different kinds of green tea were; Sayama-Cha, Uji-Cha, Uresino-Cha, Yame-Cha, and Kagoshima-Cha. Rest of two green teas were two different species of tea tree from Kagoshima; Okumidori and Asatsuyu. Powder teas were used for analysis without any treatment. Each green tea was pulverized by mill.

<Neutron activation analysis>

About 0.5 g of samples were sealed in plastic bags and irradiated. Each samples were irradiated three different condition; Ti : Td : Tc = 20 sec : 60 sec : 100 sec, 1min : 1 min : 10 min, 4 h : 1 month : 30 min. NIST SRM 1646a Estuarine Sediment, NIST SRM 1633a Coal Fly Ash, NIST SRM 1566b Oyster Tissue, BCR-60 Lagarosiphon Major, and JSO-1 Soil were used as comparator samples.

<Preparation of drinking green tea>

Drinking green teas were prepared from 2 g of green tea and 80 mL of hot water with standard ceramic tea pot. As for hot water, two kinds of tap water and distilled water were used for comparison. After making drinking green tea, the residue green tea was freeze dried, and pulverized.

RESULTS: Concentrations obtained for domestic green teas analyzed by long life nuclides are shown in Table 1. Cs, Sc, Rb, Fe, and Co levels showed differences between the location of tea production and species of green tea trees. Ratios of elements extracted in drinking green teas are shown in Table 2. Blanc cells caused that peaks were not found or ratio was bigger than 1.00.

Table 2. Ratios of elements extracted in drinking green tea

Element	U	Th	Cs	Sc	Rb	Fe	Zn	Co
Sayama		0.23		0.48	0.49		0.35	0.15
Uji			0.84	0.57	0.71	0.42	0.66	0.71
Ureshino			0.60	0.07	0.64		0.52	0.60
Yame		0.03	0.35	0.52	0.50	0.37	0.46	0.48
Kagoshima	0.27	0.17	0.62	0.59	0.73	0.69	0.62	0.43
Okumidori	0.04		0.13	0.01	0.25			
Asatusyu	0.06	0.27	0.54	0.43	0.53	0.34	0.50	0.16

Table 1 .Elemental levels of domestic green teas (unit: μg/g, dry weight).

Element	Cr	U	Th	Cs	Sc	Rb	Fe	Zn	Co
Nuclide used for analysis	Cr-51	U-238	Th-232	Cs-134	Sc-46	Rb-86	Fe-59	Zn-65	Co-60
Gamma enery	320.08	351.9	583.2	604.7	889.28	1076.6	1099.25	1115.55	1173.24
Half-life	27.7 d			2.06 y	83.81 d	18.66 d	44.5 d	243.9 d	5.27 y
Sayama	1.0	0.13	0.017	0.03	0.022	14.6	86.5	35.7	0.158
Uji	1.0	0.09	0.012	0.14	0.021	21.0	69.6	40.4	0.156
Ureshino	1.5	0.12	0.014	0.05	0.016	21.0	60.6	37.8	0.271
Yame	0.7	0.12	0.013	0.05	0.026	17.1	82.7	34.4	0.227
Kagoshima	1.1	0.12	0.012	0.12	0.011	22.7	87.7	39.0	0.095
Okumidori		0.19	0.014	0.22	0.013	30.0	125.7	42.1	
Asatusyu		0.15	0.015	0.12	0.012	25.4	145.5	35.6	0.085

採択課題番号 23P1-5　中性子放射化分析法による国産緑茶の多元素分析　プロジェクト
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PR1-6 Biodistribution of Gadolinium-Based Contrast Agent, and Concentration of Manganese and Zinc in Normal and Nephrectomized Mice

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INTRODUCTION: Gadolinium (Gd)-based contrast agents have been used in medical MR-imaging. However, human nephrogenic systemic fibrosis (NSF) is reported in Gd-based contrast agent received patients with severe renal insufficiency [1] and a depletion of endogenous Zn ion may contribute in the development of NSF [2, 3]. On the other hand, Pietsch *et al.* reported no correlation of Zn with the NSF [4].

Gadolinium has a high activation cross section (1060 barn) for thermal neutron and it is possible to detect sensitively by neutron activation analysis (NAA). NAA technique also quantitates essential trace elements *in vivo* such as Zn and so on. In this study, we measured distribution of Gd and concentration of some minerals in selected tissue of four experimental condition applied mice by using NAA.

EXPERIMENTS: Twenty male 8-week-old ICR strain mice were used in this study. Ten mice were housed as normal, and another ten mice were partially nephrectomized, which were used as pseudo renal impairment model [5].Five mice in each group was administered 2.5 mmol Gd/kg b.w. Omniscan (gadodiamide) intravenously. Two days after administration, mice were sacrificed and dissected. The blood, femur, kidney, and liver were excised and weighted. The samples were freeze- dried and sealed into polyethylene bags for NAA. The sealed samples and standard of gadolinium were irradiated in Pn-3 for 30 second and in Pn-2 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 was shown in Fig.1. The kidney showed high Gd retention behaviors due to normal process of excretion of Gd. Skeletal Gd distribution of model mice were significantly higher than that of normal mice. The nephrectomized mice showed high Gd retention in each tissue compared to normal mice.

The concentration of Mn and Zn were shown in Fig.1 and Fig. 2. Manganese concentrations in kidney of model mice decreased relative to normal mice. On the other hand, Mn concentrations in liver were comparable during four groups. Zinc concentration in the selected tissue showed no difference during four groups of mice.

We confirmed no correlation of "exogenous" Gd and "endogenous" Zn. However, it is not sufficient to achieve with statistically-warranted value of the biodistribution of "exogenous" Gd and the concentration of "endogenous" and "exogenous" trace elements in normal and nephrectomized mice to clarify the correlation of Gd with trace elements.

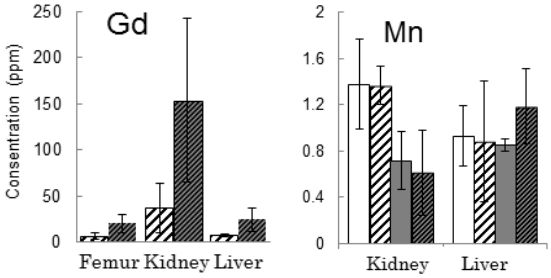

Fig. 1. Biodistribution of gadolinium and Mn in selected tissues of normal and nephrectomized mice 2 d after the omniscan administration. White bars showed normal mice and gray bars showed model mice, and the bars with striped showed the groups of Gd administration.

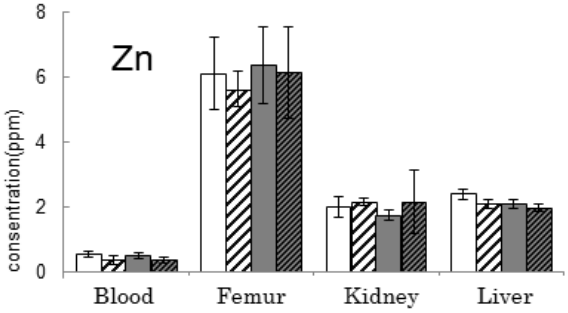

Fig. 2. Concentration of zinc in the blood, femur, kidney, and liver of normal and nephrectomized mice 2 d after the omniscan administration. White bars showed normal mice and gray bars showed model mice, and the bars with striped showed the groups of Gd administration.

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(原子炉中性子を用いた微量元素分析)

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