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PROMPT GAMMA-RAY ANALYSIS USING JRR-3M COLD AND THERMAL NEUTRON GUIDE BEAMS

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ABSTRACT

A permanent and stand-alone neutron-induced prompt gamma-ray analysis (PGA) system, usable at both cold and thermal neutron beam guides of JRR-3M has been constructed. Neutron flux at the sample positions were 1.4×10^8 and 2.4×10^7 n cm^{-2} s^{-1} for the cold and thermal neutrons, respectively. The γ-ray spectrometer is equipped to acquire three modes of spectra simultaneously: single mode, Compton suppression mode and pair mode, in an energy range up to 12 MeV. Owing to the cold neutron guide beam and the low γ-ray background system, analytical sensitivities and detection limits better than those in other PGA systems have been achieved. Analytical sensitivity and detection limit for 73 elements were measured. Boron, Gd, Sm and Cd are the most sensitive elements with detection limits down to 1 to 10 ng. For some elements such as F, Al, V, Eu and Hf, decay γ-rays are more sensitive compared to their respective prompt γ-ray. Analytical sensitivity of several heavy elements through detection of characteristic X-rays was higher than that through the prompt γ-ray detection. Analytical applicability of some sensitive elements such as B, H, Gd and Sm were examined. Isotopic analysis of Ni and Si were also examined.

INTRODUCTION

A neutron-induced prompt γ-ray analysis (PGA)[1,2] is an elemental and isotopic analytical technique in which prompt γ-rays emitted within 10^{-14} s after neutron capture reactions are measured. The PGA is characterized by its capability for nondestructive multi-elemental analysis and by its ability for analyzing H, B, N, S, P, Si and Cd which are difficult to be analyzed by instrumental neutron activation analysis (INAA). Owing to poor beam quality previous PGA[3-5] had disadvantages of low analytical sensitivity and high detection limit. The PGA has thus been regarded only as complementary to INAA. Earlier studies[6-8] indicated that PGA using a neutron guide beam has improved analytical sensitivity and detection limit. A permanent and stand-alone PGA system, usable at both cold and thermal neutron beam guides of JRR-3M has been constructed[9-11], similar to the National Institute of Standards and Technology (NIST)[12] and Research Center (KFA) Jülich[13]. The design concept is to achieve the lowest γ-ray background. To achieve this, 1) neutron shielding materials, lithium fluoride (LiF) tiles and fluorocarbon resin, are used as construction materials at positions near the sample, 2) the sample box is filled with He gas, and 3) Ge-BGO detectors are used to lower the

Compton background. In this paper, characteristics of the system and application of PGA for elemental and isotopic analysis are described.

EXPERIMENTAL

Apparatus

The PGA system can be set either at the cold (C2-3-2) or thermal (T1-4-1) neutron beam guide of JRR-3M (20 MW). The specification of the system is shown in Table 1. The cold neutrons are guided from a liquid hydrogen cold neutron source to the PGA system through a 51 m Ni coated glass mirror tube. The thermal neutrons are guided from the heavy water region through a 60 m Ni coated glass mirror tube and a 3.17 m evacuated guide tube. Initially, the present system was set behind a 4 m extended guided tube at the cold neutron beam port (C2-3-2)[10,11]. For improving neutron flux and homogeneity, the system has been modified and set directly to the cold neutron guide without the extended guided tube.

Table 1 Specifications of the prompt γ-ray analyzing system of JRR-3M

	Cold neutron	Thermal neutron
Neutron beam		
Flux, n cm^{-2} s^{-1}	1.4×10^8	2.4×10^7
Energy, meV (Å)	3.0 meV (5.2 Å)	15 meV (2.3 Å)
Beam size, mm^2	20 x 20	20 x 20
Sample to Ge detector distance, cm	29.5	24.5
γ-ray spectrometer		
Ge detector	n-Type HpGe, FWHM: 1.75 keV (at 1332 keV), Relative efficiency: 23.8 % (at 1332 keV)	
BGO detector (Crystal size)	Main: 171 mmϕ x 206 mm long, 20~39 mm thick Catcher: 60.3 mmϕ x 60 mm long	
Pulse height analyzer	MCA: SEIKO EG &G model 7800, 4 8 k ADCs	
Measurement mode	Single, Compton suppression; Pair	
Atmosphere in the sample box	He	

The system consists of a neutron beam shutter, a neutron beam collimator, a sample box, a neutron beam stopper, shielding for neutrons and γ-rays, and a multi-mode γ-ray spectrometer. The neutron beam collimator is made of lithium fluoride (LiF) tiles and its hole size is 20 x 20 mm^2. The neutron beam can be stopped by the beam shutter and beam stopper made of 10 mm thickness of sintered boron carbide (B_4C). The 478 keV prompt γ-rays from B are absorbed by Pb in the shutter and stopper. An air-tight sample box made of polytetrafluoroethylene (PTFE, Teflon) is placed behind the collimator. The emitted prompt γ-rays are detected through a 5 mm thick ^{6}LiF (95.4 % enrichment) tile. In order to reduce the γ-ray background, which is formed by neutrons scattered at the sample and absorbed by the construction materials, shielding was provided using 2 and 3 cm thick LiF tiles of natural isotopic composition and 5 and 10 cm thick Pb. Neutron and γ-ray shielding around the beam stopper and beam collimator is provided with 1 cm thick rubber containing 20 % B_4C, 5 or 8 cm thick Fe, and 3 or 5 cm thick Pb. The detector system is surrounded by 10 cm thick Pb shielding. The prompt γ-rays from the sample pass through a 2.0 cm diameter Pb collimator of 10 cm thick and enter the detector.

The multi-mode γ-ray spectrometer consists of a closed end coaxial type high purity Ge detector, BGO (bismuth germanate, $Bi_4Ge_3O_{12}$) shielding detectors, and a pulse height analyzer (PHA) system controlled by a personal computer. The BGO shielding detectors are

composed of a main shield, which surrounds the detector axially, and a back-shield (catcher). Three modes of prompt γ-ray measurements: a single mode, a Compton suppression mode, and a pair mode, in the energy range of 0 to 12 MeV, can be performed simultaneously. The Ge detector is set with its axis perpendicular to the neutron beam at a distance 29.5 cm (for the cold neutron beam) and 24.5 cm (for the thermal neutron beam). The Ge detector is surrounded by BGO main and catcher detectors which are used as guard detectors for Compton suppression and pair mode spectra. Signals of the three modes are fed into a 8192-channel analog to digital converter (ADC, SEIKO EG&G 1850). All the signals from the ADCs are sorted by a multi-channel analyzer (MCA, SEIKO EG&G model 7800). A more complete description of the PGA system can be found elsewhere[10]. Energy calibration of the γ-ray spectrometer was carried out using decay γ-rays of ^{56}Co and prompt γ-rays of Fe.

Neutron beams were monitored continuously by measuring scattered neutrons with a ^{3}He counter set beside the sample box and also by measuring prompt γ-rays with 0.05 and 0.5 mm thick of Ti plates for the cold and thermal neutrons, respectively. Prompt γ-ray energies and intensities of elements were taken from data compiled by Lone et al.[14].

Analytical procedure

A known amount of sample is wrapped in 25 μm thick fluorinated ethylenepropylene resin (FEP) film of an area smaller than 15 x 15 mm^2 and heat sealed. The wrapped sample is set on a PTFE sample holder by using PTFE string of 0.3 mm (diameter) and is then placed in an air-tight PTFE sample box. The air in the box was purged using He gas flow. The three modes of γ-ray spectra in the energy range of 0 to 12 MeV are acquired for 500 to 50000 s while passing He into the box at a flow rate of about 1000 ml/min.

RESULTS AND DISCUSSIONS

Neutron beam

The beam position and the beam shape were determined by a neutron radiographic method using Gd converter and X-ray film. The neutron flux at the sample position was measured by setting a 0.0254 mm thick Au foil at 45 degrees to the beam and irradiating for 30 min (cold neutron) or 3 to 15 hr (thermal neutron). The values of activation cross sections of 329 and 163 barns were used for the cold and the thermal neutrons, respectively, determined from neutron energy spectra measured by the time of flight method. The average neutron flux of the cold and thermal neutron beams in the 20 x 20 mm^2 area were 1.4 x 10^8 and 2.4 x 10^7 n cm^{-2} s^{-1} with a coefficient of variation of 18 and 5 % for the cold and thermal neutrons, respectively. Formerly when an extended guide tube was used the neutron flux was 1.1 x 10^8 n cm^{-2} s^{-1} with a coefficient of variation of 34 % for cold neutron beam[10]. The neutron flux and homogeneity of the neutron beam were improved by positioning the system to the beam port of the cold neutron directly. The flux fluctuation at the sample position of the neutron beam during one reactor operation cycle (26 days) was 1.6 and 1.0 % for the cold and thermal neutrons, respectively.

The gamma-ray spectrometer

The time distribution of Fe prompt γ-rays was measured by a time-to-amplitude converter (TAC, ORTEC 567). The start and stop signals were provided by Ge and BGOs, respectively. FWHM and FWTM of the distribution curve were 83.0 and 210 ns, respectively.

In the single mode spectrum full energy peaks, together with the single and double escape peaks, are observed. In the Compton suppression mode the Compton continuum is effectively suppressed to about 13 %. The single and double escape peaks are also decreased to about 10 and 17 %, respectively. Small peaks which are not distinct in the single mode spectrum can clearly be seen in the Compton suppression mode. In the pair mode spectrum only the double and single escape peaks were detected. Ideally, the pair mode is set for the detection of double escape peaks only, however, due to incomplete optical isolation of the nose portion of the main

BGO, single escape peaks can still be detected. Peak assignment of complex prompt γ-ray spectrum can be resolved by comparing the three modes of spectra.

The absolute efficiency was determined using standard sources of ^{56}Co, ^{57}Co, ^{60}Co, ^{133}Ba, ^{137}Cs, ^{152}Eu, and ^{241}Am. At high energies, prompt γ-rays of Fe were used. The high energy efficiency values were normalized to create a continuous absolute efficiency curve. The efficiency of full energy peaks and double and single escape peaks of single mode spectrum is higher by a factor of 2 to 3 than that of other PGA systems[3,4]. However, at energies below 100 keV no published data is available to compare with ours because other PGA systems cannot detect such low energy γ-rays. The detection efficiency of double escape peak of the pair mode is also higher by a factor of two than reported by other[4].

Gamma-ray background

The present system was designed to achieve the lowest possible γ-ray background by placing the sample in a He atmosphere because the scattering and absorption cross section of He is small. The flux measurements carried out with and without air (in He gas atmosphere) indicated that scattering by air can be as much as 8.5 %. The γ-ray background was reduced drastically as a result of the purging for air.

Table 2 Comparison of γ-ray background counts of the present system with others

Element or nuclide	Eγ keV	Background count rate, cps			
		Univ. Md-NIST[a]	Univ. Mo[b]	JRR-3M Cold	JRR-3M Thermal
H	2223	1.56	0.51	0.0094	0.0019
B	478	0.1	<0.1	<0.0040	<0.0021
C	1262	0.0014	0.0350	0.0020	<0.0007
N	1885	0.072	<0.008	0.0025	<0.0012
Na	472	0.057	0.349	<0.0035	<0.0012
Al	1779	0.027	0.268	0.0064	<0.0005
Cl	517	0.007	0.009	<0.0019	0.0012
Ti	342		0.063	<0.0025	<0.0016
Fe	352	0.007	0.136	<0.0028	<0.0016
^{207}Bi	569			0.0242	0.0327
^{207}Bi	1063			0.0081	0.0097

a: University of Maryland-NIST (Ref.15), b: University of Missouri (Ref.15),

The γ-ray background count rate of the system under cold and thermal neutron beams in a He gas atmosphere are tabulated in Table 2 together with the values of other systems[15]. The background count rates are attributed to prompt and decay γ-rays of elements in a construction materials and in air. Even though these γ-ray lines appear in the Compton suppression spectrum that was acquired for 50000 s in a He atmosphere, these data are the lowest compared to earlier data. The H background count rate of the cold neutron beam is lower by factors of 166 and 55 compared to the data of the University of Maryland-NIST and University of Missouri, respectively. Furthermore, for N, the present data is lower by a factor of 29 compared to the University Maryland-NIST data. The data of the University of Missouri quoted a detection limit of less than 0.008 cps, which is still higher than that of the present system. The ^{207}Bi background peak in the present system comes from BGO. In other PGA systems background prompt γ-rays of H and other elements of the construction materials higher than the present data are reported. This is mainly due to the presence of γ-rays, fast and epithermal neutrons in the beams of the other systems. In those systems, a large amount of moderating and shielding materials would be required to eliminate undesired neutrons. In the present system, however, such large shielding is not necessary.

For wrapping the sample, materials that produce the lowest possible prompt γ-ray backgrounds have to be used. Fluorocarbon resin is the most suitable for this purpose because the constituent elements C and F will emit prompt γ-ray at low count rates. FEP film was chosen from this viewpoint. Another advantage of using FEP film lies in the ease of sealing by heating.

Analytical sensitivity and detection limit of elements

Standard samples heat-sealed in FEP film were irradiated and the prompt γ-rays were measured for a period of time to produce a statistically sufficient count. Interference-free full energy peaks of high intensity were used for the calculation of sensitivity (cps/mg). From the Compton suppression spectrum of a known amount of FEP film, the 3σ background count in the region corresponding to the prompt γ-ray line of a specified element was estimated to calculate the detection limit. The analytical sensitivities and detection limits of 73 elements were measured. The analytical sensitivities of selected elements are shown in Table 3 and compared with those of other systems[3,4,8]. The detection limits of the selected elements are also shown in Table 4 and indicates a very great improvement compared to other systems by reducing γ-ray background at the detector.

Table 3 Comparison of analytical sensitivity of the present system with others

Element	E_γ keV	Analytical sensitivity, cps/mg Univ. Md-NIST[a]	Univ. Mo[b]	ILL[c]	JRR-3M Cold	JRR-3M Thermal
H	2223	0.86	1.23		3.74	0.417
B	478	530	759	2700	2620	212
Cd	558	170	247		405	47.5
Sm	334	640	740	3700	749	142
Gd	182	680	956	6900	1560	224
Flux, n cm^{-2} s^{-1}		$2x10^8$	$5x10^8$	$1.3x10^8$	$1.4x10^8$	$2.4x10^7$
Sample to detector distance, cm		50	75	18	29.5	24.5

a: University of Maryland-NIST (Ref.4), b: University of Missouri (Ref.3), c: ILL (Ref.8).

The analytical sensitivities of these elements using cold neutron irradiation are 5.3 to 12 times higher than those using thermal neutron irradiation. The analytical sensitivities of these elements at the cold neutron beam are 1.4 to 2 times higher than those obtained using the extended guided tube[10,11]. The sensitivity of the present system at the cold neutron beam is generally higher, up to about 5 times that reported values of the University of Maryland-NIST[4] and the University of Missouri[3] systems, except for Sm. Since the neutron capture resonance cross section of Sm is large at the epithermal region, the Sm sensitivity of the beam type systems is higher than the present cold neutron beam system. Kerr et al.[8] reported higher sensitivity values than the present system for B, Sm, and Gd. The main reason for this is that a Ge detector was used without a Compton suppression detector, and thus it could be positioned nearer to the sample. In the present system we can also remove the Compton suppression detectors and put the Ge detector nearer to the sample, but the prompt γ-ray background count will rise drastically resulting in a poorer detection limit.

Boron, Sm, Gd, and Cd are the elements having the highest analytical sensitivity with detection limits down to 1~10 ng and 10~100 ng, at the cold and thermal neutron beams,

respectively. Mercury, Dy, Eu, Er, Nd, In, Hf, Cl, Sc, Ti, Co, and H can be detected to 0.05~1 μg and 0.3~4 μg, at the cold and thermal neutron beams, respectively. Lead, F, C, and Bi have the lowest sensitivity with detection limits in the range of 0.2~2 mg and 1~10 mg, at the cold and thermal neutron beam, respectively. Other elements are considered to have a medium sensitivity, such as V, Yb (and most of the other lanthanoids), Mn, Ag, Ni, Na, K, S, Fe, Si, P, Sn, and N and so on with detection limits ranging from 1~100 μg and 5~600 μg at the cold and thermal neutrons, respectively.

The decay γ-rays at 1634 keV from ^{20}F ($T_{1/2}$:11.03 s), 1779 keV from ^{28}Al ($T_{1/2}$:2.2406 min), 1434 keV from ^{52}V ($T_{1/2}$:3.75 min), 90 keV from ^{152m2}Eu ($T_{1/2}$:1.600 h), and 214 keV from ^{179m1}Hf ($T_{1/2}$:18.68 s) have much higher sensitivities than the corresponding prompt γ-rays. Characteristic X-rays were detected for heavy elements. The emission of the X-rays is possibly due to internal conversion following the emission of prompt γ-rays. The analytical sensitivity of the X-rays for the elements with atomic weights larger than Ta is higher than that of the prompt γ-rays except for Pt, Hg, Pb, and Bi. Measurement of the characteristic X-rays is therefore useful for the analysis of these heavy elements.

Table 4 Detection limits of selected elements

Element	Eγ keV	Detection limit, μg Cold	Thermal
H	2223	1.1	2.8
B	478	0.0022	0.0097
N	1885	119	304
P	513	54	242
S	841	15	49
Cl	517	0.74	2.3
Mn	84	2.5	8.1
Fe	352	28	63
Ni	465	5.1	22
Cd	558	0.011	0.028
Sm	334	0.0071	0.013
Gd	182	0.0064	0.020
Hg	368	0.055	0.26

Applications of elemental analysis

The gamma-ray spectra of standard reference material Coal Fly Ash (NIST 1633a) was measured as a means to assess the ability of the present system. Prompt γ-ray peaks of Gd, Sm, Fe, and B are found in the spectrum. The S/N ratio of the present data as compared to that from the University of Maryland-NIST[16] system for the same sample was found to be higher by a factor of 4 to 8 for Gd, Fe and B, but for Sm it was only 1.3. A beam type PGA system like that of the University of Maryland-NIST contains γ-rays, fast and epithermal neutrons in the beam, thus the γ-ray background is high, especially in the low energy region. However, the guide beam type system is free of γ-rays, fast and epithermal neutrons, so that S/N ratios for Gd, Fe, and B are much higher. As described by Mackey et al.[17] for the case of Sm, its higher resonance capture cross section for epithermal neutrons results in a S/N ratio no better than the data of the University of Maryland-NIST system.

Due to its large neutron capture cross section, the B content in reactor materials must be as low as possible. As mentioned already, the present system was found to have the highest sensitivity for B, thus the analysis of B in reactor grade graphite and Be has been performed. The Doppler broadened 478 keV prompt γ-ray of B which is emitted as a result of ^{10}B(n, α)^{*7}Li $\longrightarrow$ ^{7}Li (σ = 3837 barn) was observed with a good S/N ratio. The determination of B was done by measuring the counts under the 478 keV photo peak. The 2.0 ppm of B thus determined is in good agreement with the certified value (2 ppm) of the material. The detection limit of B was estimated to be 40 ppb. Further improvement of the detection limit down to 4 ppb is achievable if 1 g of sample is used instead of 0.1 g because the spectrum is almost similar to the FEP background spectrum. The B content in Be which is used as the reflector frame of the Japan Material Testing Reactor (JMTR) has also been measured. The B content was determined by measuring 1.0 g Be for 50000 s in the thermal neutron beam. The result of the

analysis, 3.2 ppm, is in good agreement with the value (2.7 to 3 ppm) determined by emission spectrographic method. The detection limit of B in the sample was 24 ppb.

The analytical sensitivity of rare earth elements such as Gd, Sm, Eu, and Dy is higher than other rare earth elements such as Ce, Y, La, Pr, and Tb by 4 orders of magnitude. Therefore, the present system is useful for the determination of these rare earth elements with high sensitivity which are present in the rare earth elements that have lower sensitivity. High purity cerium(IV) oxide (CeO_2 99.99 %, 0.18 g) was analyzed at the cold neutron beam for the purpose. The prompt γ-rays at 182 and 199 keV from Gd, 334 and 439 keV from Sm, 186 keV from Dy and Er, and the decay γ-ray at 90 keV from ^{152m2}Eu were detected in the spectrum. The content of Gd and Sm was determined to be 0.41 and 0.62 ppm, respectively, using the interference free prompt γ-ray peaks at 182 and 334 keV for Gd and Sm, respectively. The detection limits of Gd and Sm in the sample were calculated to be 0.11 and 0.14 ppm, respectively.

As was mentioned by Lindstrom et al.[12], neutron guide beam PGA has a potential usefulness for trace determination of H in various types of materials. Our system has detection limits for H of 1.1 and 2.8 μg at the cold and thermal neutron beam, respectively. The trace determination capability was examined by analyzing H in Zr and zircaloy. By measuring 0.3 g of zirconium hydride (H content: 1.9 %) for 1000 s at the cold neutron beam, the H content was determined to be 1.6 %. The detection limit of H in 0.12 g of reactor material zircaroy-2 (JAERI-Z1) which was measured for 10000 s at the cold neutron beam, was estimated as 8.4 ppm.

The analytical sensitivity of Hf at the cold and thermal neutron beams are 863 and 544 times higher, respectively, than that of Zr. Therefore, the present system can also be used for trace determination of Hf in Zr, which is generally difficult due to their similar chemical properties. The analytical result of Hf which was determined using the decay γ-ray at 214 keV of ^{179m1}Hf in the above mentioned zirconium hydride was 2.0 %, with the detection limit being 58 ppm. The allowable level of Hf in reactor grade Zr is 100 ppm, therefore this system is useful for this purpose.

The system is also useful for trace determination of Cd and Hg these in environmental samples. Apart from trace determination of sensitive elements, the system is also applicable to the determination of major and minor constituent element such as S, P, N, and Si in various samples. The analysis can be carried out non destructively, thus samples that cannot be dissolved or destroyed can be analyzed. Furthermore, this technique is a multi-elemental analytical method. With all these advantages this system can be applied to various types of samples in various fields such as environmental science, material science, geology, archeology, biology, and so on.

Isotopic analysis

Since γ-rays from various nuclear transition are detected, PGA can be used for isotopic analysis. The isotopic analysis of Ni and Si which is difficult to perform by mass spectrometer has been examined. Nickel consists of 5 stable isotopes of ^{58}Ni, ^{60}Ni, ^{61}Ni, ^{62}Ni and ^{64}Ni. Compton suppression prompt γ-ray spectra of natural isotopic composition of Ni and ^{58}Ni enriched (99.66 % enrichment) isotope are shown in Fig.1 Prompt γ-ray lines of Ni in the spectrum could be assigned by measuring enriched isotopes of ^{58}Ni, ^{60}Ni, ^{61}Ni, ^{62}Ni and ^{64}Ni. Isotopic analysis of ^{28}Si, ^{29}Si and ^{30}Si were also examined by the PGA. Usefulness of PGA for isotopic analysis has been demonstrated from the experiments.

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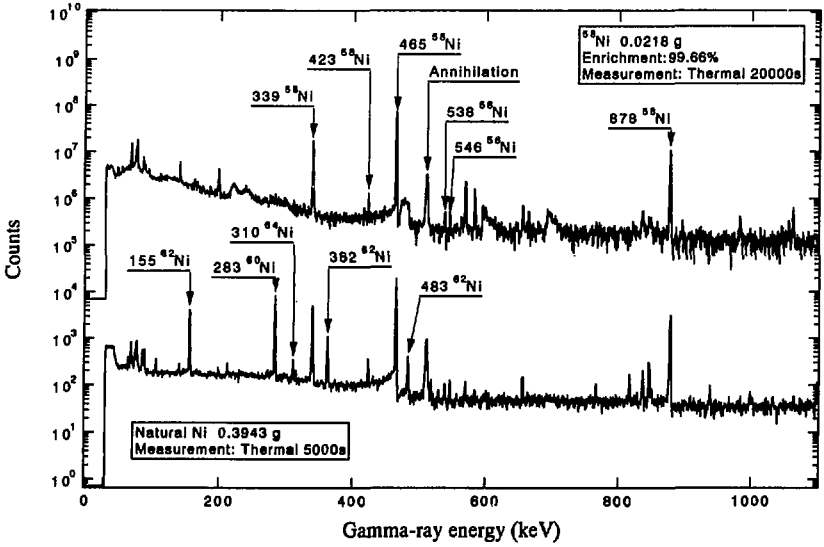

Fig. 1 Prompt γ-ray Compton suppression spectra of ^{58}Ni and natural Ni

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