

The Determination of Major and Trace Elements in Iron Meteorites by Neutron Activation Analysis

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I. Introduction

When we obtain a new meteorite, first of all it is necessary to analyze the mean elemental composition, not only major elements but also trace elements if it is possible. Of course, it is better that data are accurate enough to use further cosmochemical discussion.

Because number of meteorite falls are limited, and it is often observed that some classes contain only a small meteorite, it is also desirable to use as small a sample for analyses as possible.

In Fig. 1, the part of periodic table of the elements is shown. In iron meteorites, except for the upper 5 elements and Mn, Tc, Tl, Pb and Bi which are generally known to be present at less than the ppb level or none, all of the other elements in this table should be analyzed. These are so-called siderophile elements and part of calcophile elements.

Among these elements, major elements, Fe, Ni, and Co and trace element, Cu, showing in shaded cases, could be analyzed accurately by any methods such as titration, atomic absorption, inductively coupled plasma spectrometry and so on. The concentration or the distribution of Cr in iron meteorites is very variable, <1 to ~100 ppm (Shima and Honda, 1966a and b). Higher content of Cr could be analyzed by atomic absorption spectroscopy or plasma spectrometry (Shima *et al.*, 1978). The case of Cr was, therefore made a half shade. Phosphorus could be analyzed colorimetrically or plasma spectrometrically. In these methods, however, ~1 g each of sample is necessary for accurate quantitative analysis.

For the above purpose, thermal neutron activation analysis should be the best candidate, because all of these elements could be analyzed by using only less than 0.2 g sample without any complicated chemical procedure. The methods have been developed for a single or several elements in iron meteorites by several workers (Baedecker and Ehmann, 1965; Crockett, 1972; Hamaguchi *et al.*, 1961; Kiesel *et al.*, 1967; Kimberlin *et al.*, 1968; Wasson and Kimberlin 1966). No work, however, has been carried out for covering all of these elements.

VIA		VIIA		VIII			IB	II _B	III _B	IV _B	V _B
								B	C	N	
								Al	Si	P	
Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As		
Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb		
W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi		
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Fig. 1. A part of the periodic table of the elements.

- M ; well known to be very low or none in iron meteorites.
- M ; could be analyzed by other methods rather easily.
- M ; depends on contents in iron meteorites, could be analysed by other methods.
- M ; analyzed by instrumental (non-destructive) neutron activation analysis.
- M ; depends on contents in iron meteorites, could be analyzed by instrumental neutron activation analysis.
- M ; analyzed by radiochemical neutron activation analysis. Half-life of analyzed nuclides: less than 2 days.
- M ; analyzed by radiochemical neutron activation analysis. Half-life of analyzed nuclides: longer than 2 days.

II. Method and Procedure

Depending on analyses required, four methods were planned and applied for the analyses of iron meteorites. Main component elements, Fe, Co and Ni and some other special elements such as Ga and Au are not necessary to separate chemically from each other. Some elements produce only very short-lived nuclides by (n, γ) reaction of thermal neutron and others contain nuclides only transformed to rather long-lived ones with very small efficiency by (n, γ) reaction of thermal neutron. Each method and procedure are described successively.

A. Monitors:

Most of the standard solutions used as monitors for thermal neutrons in these experiments were prepared from accurately known weight of pure metals, normally better than 4 nines, except Ir, Ag, Hg and As.

1. *Ni, Cu, In*; These were dissolved separately in concentrated HNO_3 and after brown NO and NO_2 gas was expelled, these were diluted with H_2O to the desired con-

centrations.

2. *Co, Re*; About 5N-HNO₃ was used for the dissolution of these metals.
3. *Pd, Pt, Au*; These were dissolved in aqua-regia then diluted with H₂O.
4. *Mo*; Molybdenum was dissolved in aqua-regia, evaporated to dryness and the residue, MoO₃, was dissolved in NH₄OH.
5. *W*; Tungsten was dissolved in the mixture of HNO₃ and HF, evaporated to dryness and the residue, yellow WO₃ was dissolved in NH₄OH.
6. *Cr*; Chromium was dissolved in HCl and converted to nitrate by HNO₃.
7. *Ga*; Gallium was dissolved in the mixture of HCl and H₂O₂, excess H₂O₂ expelled and diluted in H₂O.
8. *Sn*; Tin was dissolved in concentrated HCl and diluted with water.
9. *Zn*; Zinc was dissolved in 2N-H₂SO₄.
10. *Cd*; Cadmium was dissolved in the mixture of H₂SO₄ and HNO₃, excess HNO₃ expelled and diluted with H₂O.
11. *Ge*; According to Wasson and Kimberlin (1967), Ge metal was dissolved in the mixture of oxalic acid solution, NH₄OH and H₂O₂.
12. *Sb*; The 0.1 g of five nine metal was mixed with ~1 g of NaOH and ~0.5 g of Na₂O₂ and fused in Zr crucible. The melt was dissolved in HCl and the solution was made to 3N HCl.
13. *Ru*; The 0.1 g of three nine metal was mixed with the mixture of ~0.5 g of KOH and ~0.5 g of KNO₃, and fused in Zr crucible. The fused melt was extracted with H₂O and acidified by concentrated HCl. The solution was kept as 6N HCl. Ruthenium with valency state 8 is rather unstable, and reduced to valency state 3 by HCl. The reduction rate is rather slow. In the course of reduction from valency 8 to 3, Ru is passing through the valency state 4, which is insoluble in any kind of solvent. The present solution also precipitated RuO₂ some days later. The concentration of the solution was redetermined gravimetrically.
14. *Os*; The 0.1 g of 200 mesh four nine metal was mixed with the mixture of ~1 g of NaOH and ~0.5 g of Na₂O₂ and fused in Zr crucible. The fused melt was extracted with H₂O. The solution was kept as ~0.8 N NaOH. Osmium in the above solution is also meta-stable, just like Ru, gradually reduced to valency 4. A few months later, some precipitate was observed. In such case, the solution was standardized gravimetrically.
15. *As*; Arsenic solution was prepared by dissolving As₂O₃ in ~1N NaOH and acidified with H₂SO₄.
16. *Ag*; The AgNO₃ was dissolved in H₂O for Ag standard solution.
17. *Hg*; Mercury standard solution was prepared by dissolving HgCl₂ in H₂SO₄.
18. *Ir*; Two kinds of Ir standard solutions were used in this study. One of them was the complex compound, [(CH₃)₄N] [IrCl₆], solution containing 401 ppm Ir supplied by professor T. Ishimori, St Paul's University. Another was the solution dissolving IrCl₄·H₂O, assay (Ir) 54.5% minimum, in H₂O and HCl. The concentrations of both solutions were compared with each other.

For the monitors these solutions were diluted to appropriate concentration and weighed on teflon- or aluminum-foils or in quartz tube, and dried then sealed in polyethylene bag for Triga Mark II and in quartz tube for JRR-2 and JRR-4 reactors.

B. *Methods and Procedure:*

1. *Non-destructive thermal neutron activation analysis;* In Fig. 1, elements in boxes with oblique lines from upper right-hand could be analyzed by this method. As is well known besides major elements, depending on contents and thermal neutron activation efficiencies of each element in iron meteorites, some trace elements could

Table 1. Nuclear data on (n, γ) reaction used for instrumental neutron activation analyses in this work*.

Stable nuclides	isotopic abundances (%)	$\sigma_{act.}$ (burns)	radio-nuclides	half life	main γ ray energies (keV) (intensity)**
⁵⁶ Fe	0.3	1.16	⁵⁹ Fe	44.6 d	192.5 (2) 1098.6 (50) 1291.5 (40)
⁶⁴ Ni	0.9	1.48	⁶⁵ Ni	2.520 h	366.5 (10) 1115.4 (30) 1481.7 (50)
⁵⁹ Co	100	37	⁶⁰ Co	5.27 y	1173.1 (100) 1332.4 (100)
⁶³ Cu	69.2	4.4	⁶⁴ Cu	12.71 h	1345.5 (100)
⁷¹ Ga	39.9	4.7	⁷² Ga	14.1 h	630.1 (10) 834.1 (40) 2201.4 (14)
⁷⁴ Ge	36.5	0.52	⁷⁵ Ge	82.8 m	198.6 (10) 246.6 (80)
⁷⁶ Ge	7.8	0.15	⁷⁷ Ge	11.30 h	211.4 (50) 215.5 (50) 264.5 (50)
⁷⁵ As	100	4.4	⁷⁶ As	26.3 h	559.2 (75) 657.0 (10) 1215.8 (7)
¹⁹¹ Ir	37.3	300	¹⁹² Ir	74.2 d	295.8 (10) 316.5 (40) 467.9 (20)
¹⁹³ Ir	62.7	110	¹⁹⁴ Ir	19.15 h	328.0 (50) 644.6 (10) 938.4 (6)
¹⁹⁷ Au	100	98.8	¹⁹⁸ Au	2.696 d	411.8 (100)

* Data from "Chart of Nuclides" (1977).

** Data are derived from Dams & Adams, *Radiochim. Acta* **10**, 5-8, (1968).

be analyzed. The nuclear data on (n, γ) reaction by thermal neutrons used for this procedure are presented in Table 1. Due to the content of some elements in iron meteorites, some of nuclides in this table could not be detected.

For these purpose, cleaned and weighed about 50 mg iron meteorite specimen with monitors were irradiated by thermal neutron for 1, 6 and 12 hours in Triga Mark II reactor at a flux 1.5×10^{12} neutron/cm²·sec in St. Paul's University. After 3 to 20 hours cooling, irradiated meteorite samples and monitors were analyzed by Ge(Li) γ ray spectrometer.

2. *Radiochemical neutron activation analysis-1*; This was designed for elements having only short-lived nuclides, half-life is less than a day, such as ⁷⁵Ge; half-life: 82 min., ⁷⁷Ge; 11.30 hrs., ¹⁰⁹Pd; 13.43 hrs. and ¹⁹⁷Pt; 18.3 hrs. These elements are rather insensitive for (n, γ) reaction but important for the classification of iron meteorites.

About 50 mg of iron meteorite specimen was weighed accurately, sealed in quartz tube and irradiated together with monitors, Ge, Pd and Pt, sealed also in quartz tube for 3 hours in JRR 4 reactor at a flux 8×10^{13} neutron/cm²·sec in the Japan Atomic Energy Research Institute at Tokai. Irradiated samples were cooled for about 3 hours then dissolved in aqua regia. The post irradiation chemistry was composed of the precipitation of Ge with H₂S in strong, ~6N, acidic media, and the anion exchange

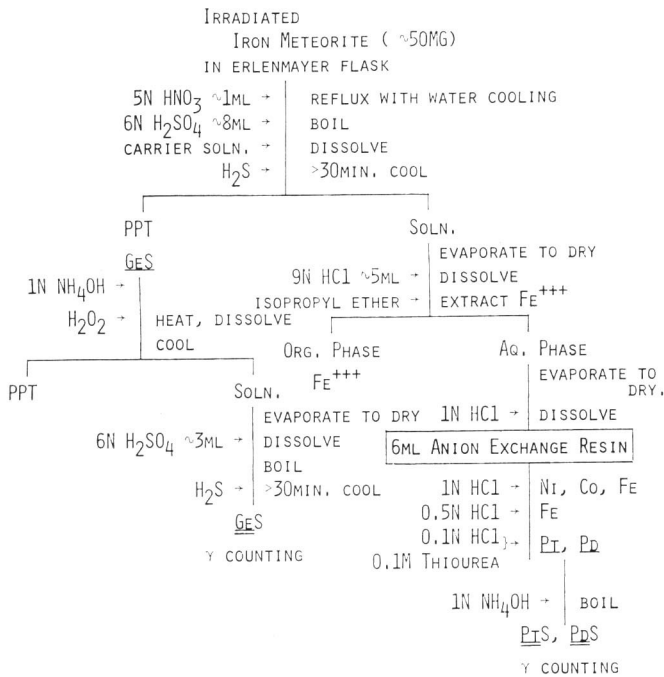


Fig. 2. Schematic diagram of the post-irradiation chemistry used for the radiochemical neutron activation analysis of Ge, Pd and Pt.

separation of Pt and Pd from Fe, Ni and Co followed by the precipitation of sulfide. The brief method is schematically given in Fig. 2.

A similar experiment was also made by irradiating about 50 mg of iron meteorites

Table 2. Nuclear data on (n, γ) reaction used for radiochemical neutron activation analyses (Method 3).*

Stable nuclides	isotopic abundances (%)	σ_{act} (burns)	radio-nuclides	half life	main γ ray energies (keV) (intensity)**
^{76}Ge	7.8	0.15	^{77}Ge	11.30 h	***
^{75}As	100	4.4	^{76}As	26.3 h	***
^{98}Mo	24.1	0.14	^{99}Mo	66.02 h	140.6 (90) 180.9 (10) 739.9 (2)
^{90}Ru	5.5	0.24	^{97}Mo	2.89 d	215.8 (90) 325.1 (10)
^{108}Pd	26.7	12.2	^{109}Pd	13.43 h	311.5 (10) 88.0 (100)
^{114}Cd	28.7	0.30	^{115}Cd	53.5 h	492.5 (30) 527.7 (60)
^{121}Sb	57.3	6.2 ₅	^{122}Sb	2.72 d	564.0 (90) 692.5 (5) 1140.5 (1)
^{186}W	28.6	38	^{187}W	23.9 h	134.3 (10) 479.3 (20) 685.7 (40)
^{187}Re	62.60	75.3	^{188}Re	16.95 h	155.1 (70) 478.0 (6) 633.0 (10)
^{192}Os	41.0	2.0	^{193}Os	30.5 h	139.0 (20) 460.4 (20) 557.7 (10)
^{193}Ir	62.7	110	^{194}Ir	19.15 h	328.0 (50) 644.6 (10) 938.4 (6)
^{196}Pt	25.3	0.7 ₅	^{197}Pt	18.3 h	77.7 (90) 191.4 (10)
^{198}Pt	7.2	3.7	^{199}Pt $\downarrow \beta^-$ ^{199}Au	30.8 m 3.14 d	158.3 (80) 208.2 (20)
^{197}Au	100	98.8	^{198}Au	2.696 d	***

* Data from "Chart of Nuclides" (1977).

** Data are derived from Dams & Adams, Radiochim. Acta **10**, 5-8 (1968).

*** See Table 1.

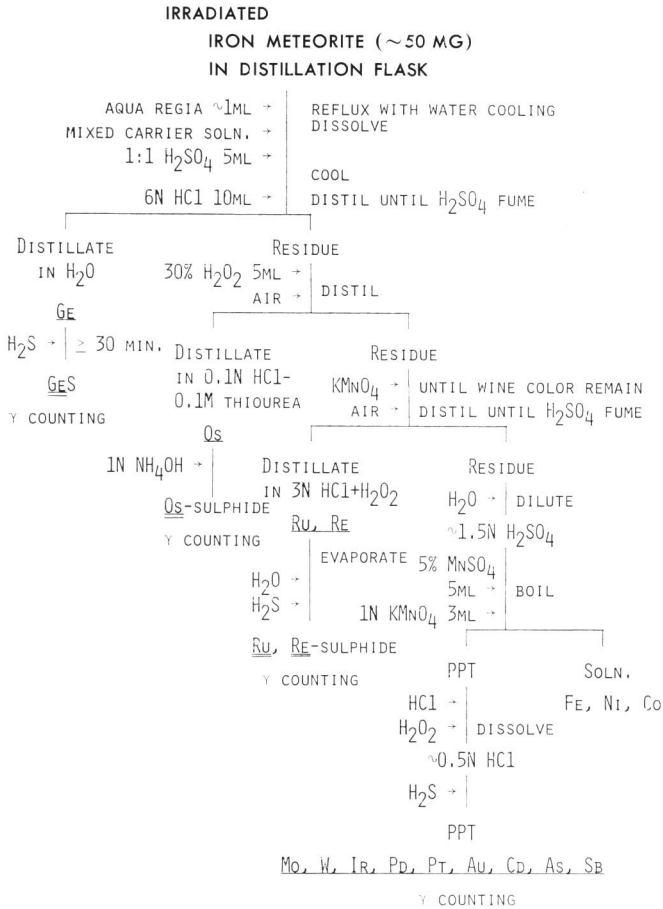


Fig. 3. Schematic diagram of the post-irradiation chemistry used for the radiochemical thermal neutron activation analysis of the elements with half-life of less than 2 days.

sealed in polyethylene bags with monitors for 12 hours in Triga Mark II reactor at a flux 1.5×10^{12} neutron/cm²·sec in St. Paul's University.

3. *Radiochemical neutron activation analysis-2*; The method was designed for the elements with short-lived nuclides, half-life; ≤ 2 days. The nuclear data of such elements on (n, γ) reaction used for this method is shown in Table 2.

Cleaned and weighed about 50 mg iron meteorite samples with monitors including all elements to be analyzed were irradiated by thermal neutron for 10 hours in JRR 4 reactor at a flux 8×10^{13} neutron/cm²·sec in the Japan Atomic Energy Research Institute at Tokai. The post irradiation chemistry is schematically shown in Fig. 3.

After 17 hours cooling, iron meteorite with carrier was decomposed by aqua regia in distillation flask to which a water cooled reflux tube was attached. This method consists of successive distillation of Ge, Os, Ru and Re. Distilled elements were

precipitated as sulfides and counted by Ge(Li) γ ray spectrometer. From the residue after distilling Ge, Os, Ru and Re, Pd and Pt were separated from Fe, Ni and Co by coprecipitating with MnO_2 . The MnO_2 precipitate also contained Mo, W, Ir, Au, Cd, As and Sb. For purifying these elements, the precipitate was dissolved in HCl and H_2O_2 , then Pd, Pt, Mo, W, Ir, Au, Cd, As and Sb were precipitated as sulfide with hydrogen sulfide in acidic solution. These were measured altogether with Ge(Li) γ ray spectrometer, because γ energies for each nuclide could easily be isolated.

4. *Radiochemical neutron activation analysis-3.* The method was planned for the nuclides having longer half lives. The nuclear data of each nuclides used for this experiments are tabulated in Table 3.

About 50 mg of accurately weighed iron meteorite specimen with monitors

Table 3. Nuclear data on (n, γ) reaction used for radiochemical neutron activation analyses (Method 4)*.

Stable nuclides	isotopic abundances (%)	$\sigma_{\text{act.}}$ (burns)	radio-nuclides	half life	main γ ray energies (keV) (intensity)**
^{64}Zn	48.6	0.76	^{65}Zn	243.8 d	1115.4 (100)
^{75}As	100	4.4	^{76}As	26.3 h	***
^{98}Mo	24.1	0.14	^{99}Mo	66.02 h	***
^{102}Ru	31.6	1.4	^{103}Ru	39.4 d	497.0 (90) 610.2 (10)
^{109}Ag	48.17	4.4	$^{110\text{m}}\text{Ag}$	252 d	657.8 (30) 884.5 (20) 937.2 (10)
^{113}In	4.3	8	$^{114\text{m}}\text{In}$	49.5 d	190.2 (100)
^{116}Sn	14.7	0.006	$^{117\text{m}}\text{Sn}$	14 d	158.4 (100)
^{124}Sn	5.6	0.13 ₄	^{125}Sn	9.65 d	332.0 (100)
^{123}Sb	42.7	4.2 ₆	^{124}Sb	60.20 d	602.6 (50) 722.8 (6) 1690.7 (25)
^{184}W	30.67	1.8	^{185}W	75.1 d	125.5 (100)
^{185}Re	37.40	111	^{186}Re	90.6 h	122.6 (6) 137.0 (90)
^{190}Os	26.4	13	^{191}Os	15.3 d	129.4 (100)
^{191}Ir	37.3	300	^{192}Ir	74.2 d	***
^{197}Au	100	98.8	^{198}Au	2.696 d	***
^{202}Hg	29.8	4.9	^{203}Hg	46.60 d	279.1 (100)

* Data from "Chart of Nuclides" (1977).

** Data are derived from Dams & Adams, *Radiochim. Acta* **10**, 5-8 (1968).

*** See Table 1 or Table 2.

in quartz tubes were irradiated for 260 hours in JRR 2 reactor at a flux 3×10^{13} neutron/cm²·sec in the Japan Atomic Energy Institute at Tokai. The post irradiation chemistry is schematically illustrated in Fig. 4.

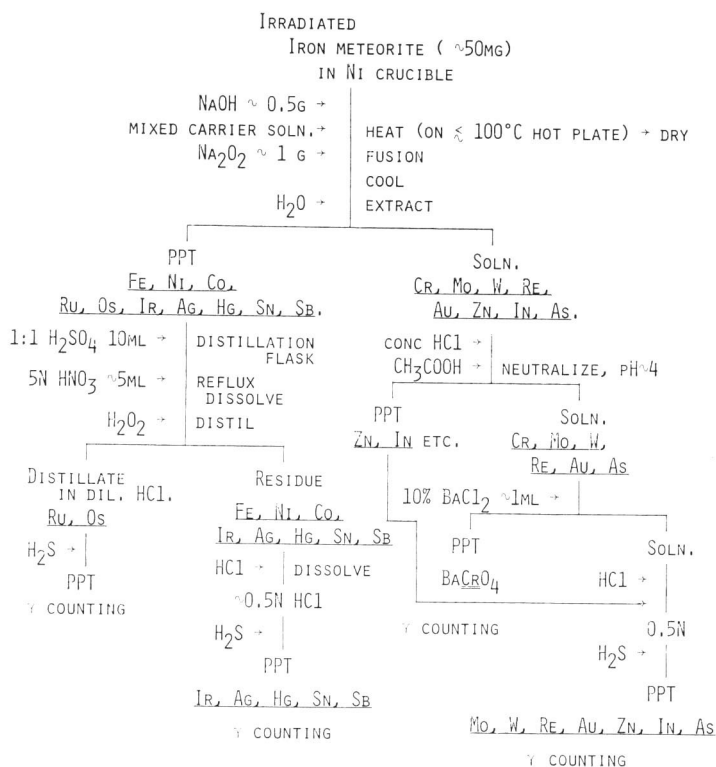


Fig. 4. Schematic diagram of the post-irradiation chemistry used for the radiochemical thermal neutron activation analysis of the elements with half-life of longer than 2 days.

The irradiated sample was fused by Na₂O₂ in Ni crucible. After extraction of the fused cake by H₂O, Ru and Os were distilled from the residue dissolved in H₂SO₄ and Ir, Ag, Hg, Sn and Sb were separated from Fe, Ni and Co by precipitation with hydrogen sulfide in acidic solution. From the solution extracted from the fused cake, Cr was precipitated as BaCrO₄ and Mo, W, Re, Au, Zn, In and As were precipitated with hydrogen sulfide in slightly acidic solution. Nuclides collected and purified in each fraction were measured by their γ energy with Ge(Li) γ ray spectrometer.

C. Chemical Yields:

The chemical yields after radiochemical separations were determined by irradiating all separated samples again by thermal neutrons, for 1 or 5 hours depending on elements at flux 5×10^{11} neutron/cm²·sec in Triga Mark II reactor in St. Paul's

University. The chemical yields of germanium were measured gravimetrically by weighing GeO_2 .

III. Results and Discussion

An example of a γ ray spectrum of the iron meteorite, Shirahagi, which was irradiated by thermal neutron for 6 hours in Triga Mark II reactor at a flux 1.5×10^{12} neutron/cm²·sec in St. Paul's University, is shown in Fig. 5. This was taken for 1000 sec after ~ 1 day cooling. One channel was adjusted for \sim one keV.

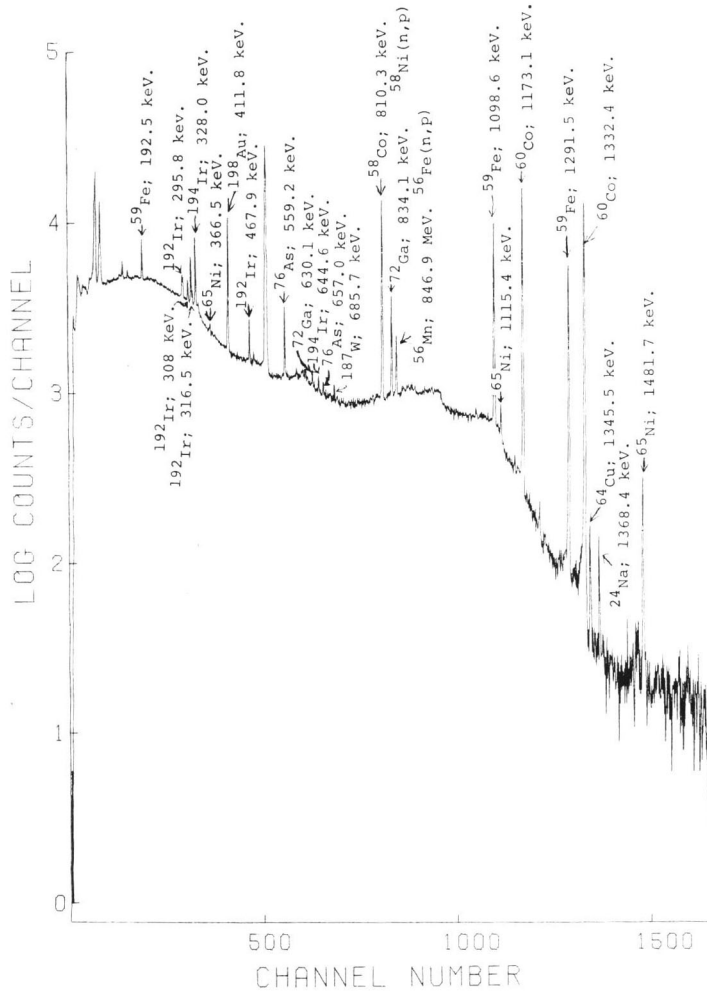


Fig. 5. An example of γ ray spectrum obtained by Ge(Li) detector for the iron meteorite Shirahagi. (non-destructive).

It is clearly shown that besides (n, γ) reactions of the main components, $^{58}\text{Fe}(n, \gamma)^{59}\text{Fe}$, $^{64}\text{Ni}(n, \gamma)^{65}\text{Ni}$ and $^{59}\text{Co}(n, \gamma)^{60}\text{Co}$, (n, p) reactions $^{56}\text{Fe}(n, p)^{56}\text{Mn}$ and $^{58}\text{Ni}(n, p)^{58}\text{Co}$ also occurred. These were expected Triga Mark II reactor contains a significant proportion of high-energy neutrons.

The γ -ray energy peaks from some trace elements such as Cu, Ga, Ir, Au, As and W are also visible, that is, these elements in Shirahagi are determined only by comparison with monitors without any chemical treatment. However, depending on the meteorite, some element could not be detected. For example, in Fig. 5, Ge peaks are not observed while in Kuga and Tendo, they appeared to some extent. Contrary to the case of Ge, Ir peaks were not detected in Kuga whereas in Fig. 5, they are clearly observed. In this method, due to the effect of main components, background is so high that the accuracy of data for trace elements remains only $\sim \pm 10\%$.

All the nuclides in samples and in standards were measured at least three times each in one irradiation to check decay rate. This procedure is necessary to confirm each nuclide against impurities. Examples for decay curves of Os and Ru in standard and in separated samples from Shirahagi and Tendo are shown in Figs. 6 and 7.

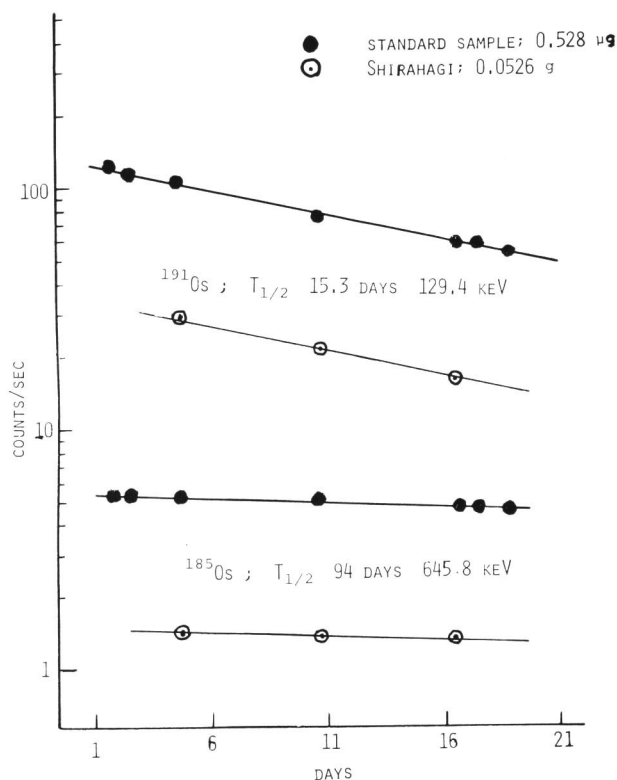


Fig. 6. Examples of decay curves of ^{191}Os and ^{185}Os .

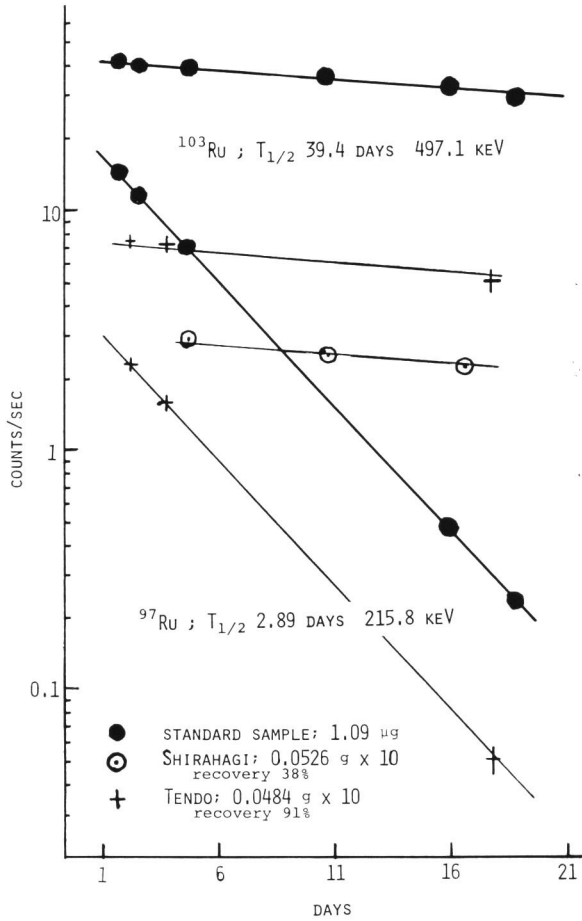


Fig. 7. Examples of decay curves of ^{97}Ru and ^{103}Ru .

The results are summarized in Table 4. Main component elements such as Ni and Co agreed very well with the values obtained by the method described in the former paper (Shima *et al.*, 1978). Wasson (1974) measured Ga, Ge and Ir in Shirahagi. Gallium and Ir in this work agreed well with his data.

Gallium, Ge and Ir have been used for the indicator elements for the classification of iron meteorites. The logarithmic concentrations of Ga, Ge and Ir in 439, 449 and 432 iron meteorites against the concentration of Ni were plotted in Tables 8, 9 and 10 respectively. The present values for Shirahagi, Tendo and Kuga in Table 4 are just in the clusters of Group IVA, IIIA and IIIB respectively. These fact confirm that these iron meteorites belong to respective groups, which have not surely been decided by the analyses of major elements and microanalytical observations (Shima *et al.*, 1978).

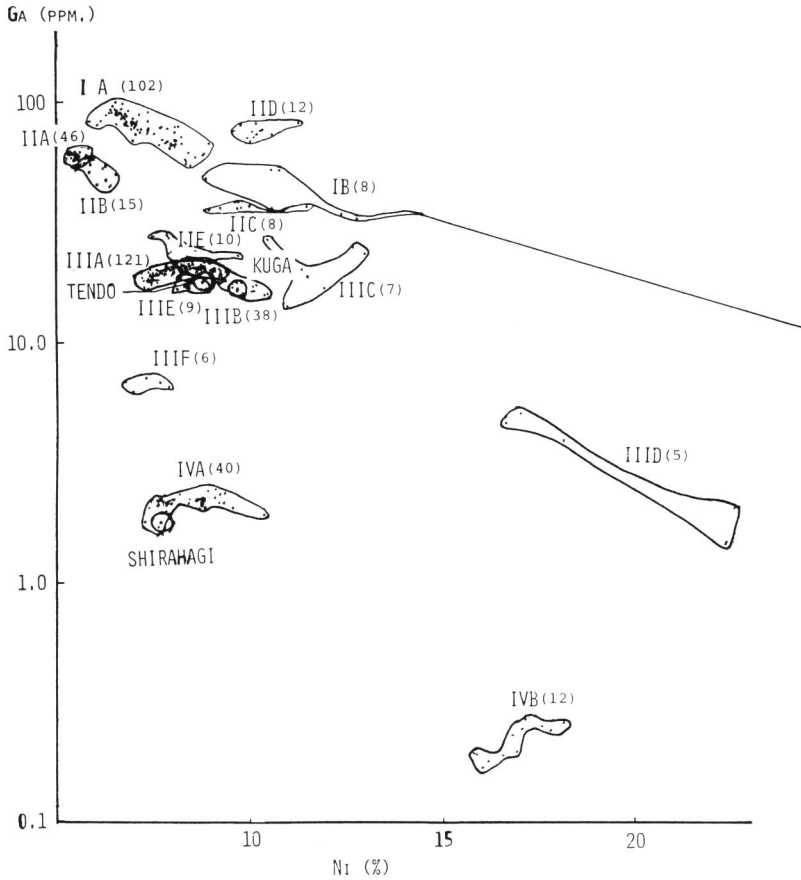


Fig. 8. The relationship between logarithmic concentration of Ga and the concentration of Ni. The data for Shirahagi, Tendo and Kuga are clearly situated in the clusters of IVA, IIIA and IIIB respectively.

The analytical results of other trace elements should have many implications for the formation of iron meteorites, the accretion of the elements from solar nebula, the recrystallization of constituent minerals during the long history of meteorite, behavior of elements in the different conditions other than the surface of earth etc. However, to discuss these facts further it is necessary to have more data for other iron meteorites.

IV. Acknowledgements

The authour expresses her sincere thanks to Professors T. Ishimori and K. Tomura in St. Paul's University, Professor Y. Ito and Mr. T. Takano in Tokai Branch, Research

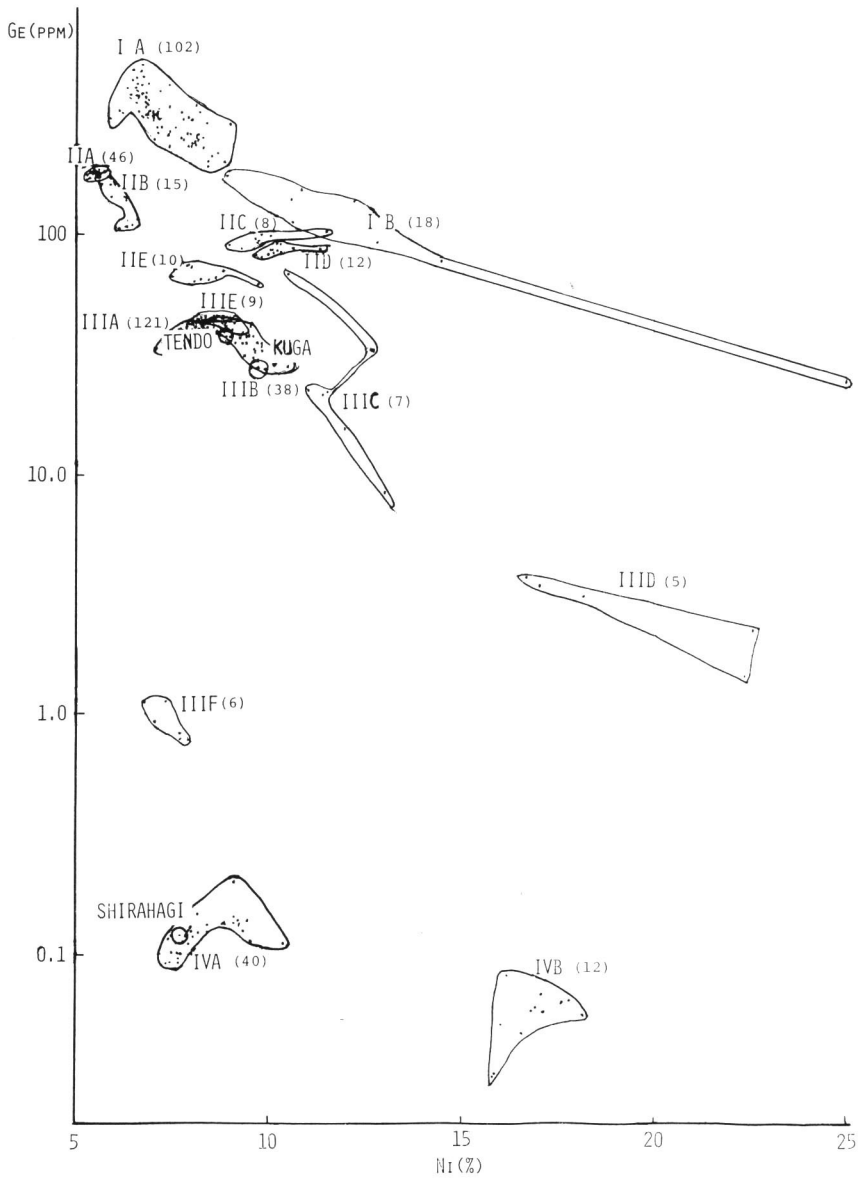


Fig. 9 The relationship between logarithmic concentration of Ge and the concentration of Ni. The data for Shirahagi, Tendo and Kuga are clearly situated in the clusters of IVA, IIIA and IIIB respectively.

Center for Nuclear Science and Technology, The University of Tokyo, and Professor M. Honda and Miss K. Horie in The Institute for Solid State Physics, The University of Tokyo, for allowing her to use facilities for chemical separation of

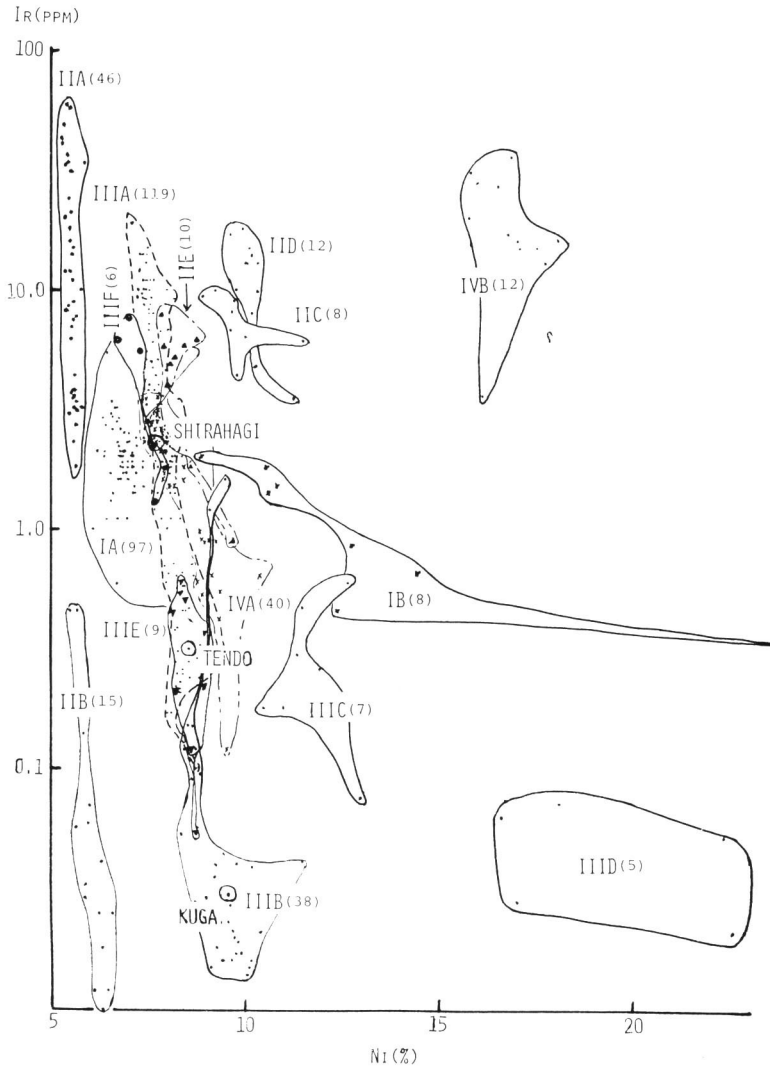


Fig. 10. The relationship between logarithmic concentration of Ir and the concentration of Ni. The data for Shirahagi, Tendo and Kuga are clearly situated in the clusters of IVA, IIIA and IIIB respectively.

radioactive samples and the Ge(Li) γ ray spectrometer. This work has been performed by the collaboration of Dr. A. Okada and Mrs. S. Yabuki in The Institute of Physical and Chemical Research, and by miscellaneous assistance of individuals in St. Paul's University, in the Japan Atomic Energy Research Institute and in Tokai branch, Research Center for Nuclear Science and Technology, The University of Tokyo.

Table 4. The composition of major and trace elements in iron meteorites Shirahagi, Tendo and Kuga.*

Element	Shirahagi		Tendo		Kuga	
	previous work	this work	previous work	this work	previous work	this work
Ni (%)	7.68**	7.65	8.89**	8.56	9.68**	9.63
Co (%)	0.39**	0.42	0.54**	0.43	0.58**	0.594
Cu (ppm)	150**	143	150**	—	110**	120
Cr (ppm)	310**	61.3	3**	3.33	<20**	5.09
Ga (ppm)	2.19***	1.89	—	18.0	—	16.5
Ge (ppm)	0.120***	—	—	37	—	26.5
Ir (ppm)	2.4***	2.37	—	0.330	—	0.034
Mo (ppm)	—	6.5	—	3.5	—	11.6
W (ppm)	—	0.44	—	0.47	—	0.41
Ru (ppm)	—	0.430	—	0.421	—	0.408
Os (ppm)	—	2.64	—	0.101	—	2.78
Pd (ppm)	—	2.1	—	7.8	—	1.2
Pt (ppm)	—	5.2	—	8.0	—	5.5
Ag (ppm)	—	0.0081	—	0.013	—	0.0041
Au (ppm)	—	0.79	—	1.08	—	2.2
As (ppm)	—	5.5	—	14	—	36
Sb (ppm)	—	0.018	—	0.13	—	0.34

* mean values of a couple of measurements.

** Shima *et al.*, (1978).

*** Wasson (1974).

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