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A Revised Method for the Determination of Major and Trace Elements in Iron Meteorites by Neutron Activation Analysis

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I. Introduction

The distribution of trace elements in iron meteorites is an important factor for the discussion of the evolution of iron meteorites through condensation, accretion and/or fractionation processes of the elements. For such a purpose it is necessary to obtain data of similar quality for each element belonging to the same group, such as Cr-Mo-W, Cu-Ag-Au or so. Recently a number of superior analyses of trace elements in iron meteorites have been reported (COBB, 1967: CROCKET, 1972; KRACHER *et al.*, 1980; REED, 1972; SCHAUDY *et al.*, 1972; SCOTT *et al.*, 1973, 1976; SEARS, 1978; SEITNER *et al.*, 1971; SMALES *et al.*, 1967; WASSON *et al.*, 1971; WILLIS, 1981). Most of these studies are, however, limited to the determination of single elements or of several indicator elements for the classification of iron meteorites.

We have been engaged in the determination by neutron activation analysis of trace elements in Japanese iron meteorites in order to obtain useful data for cosmochemical discussions as well as to help in their classification. Since the analytical method was reported by one of the authors two years ago (SHIMA, 1979), the procedures have been revised in such a way as to give better results.

In this paper, we present the improved method of neutron activation analysis of iron meteorites.

II. Experimental Procedure

To cover all major and trace elements to be analyzed, three different methods of neutron activation analysis, instead of four as in the previous paper (SHIMA, 1979), were planned. Some elements are determined by only one of these methods and some others can be analyzed by all three. The analytical results obtained from different methods can be used not only for the comparison of each method but also for checking the homogeneity of the elements in the meteorites.

A. *Monitors*

Thermal neutron monitors were prepared in the same way as described in the previous paper. An attempt was also made to calculate γ-ray activities using only one or two representative elements. This aim only secceeded in the case of experimental conditions with rather long irradiation periods and with nuclides showing comparatively simple γ-decay.

B. *Methods and Procedure*

In these experiments neutron irradiation were performed in the following four reactors: JRR-2, JRR-3 and JRR-4 of Japan Atomic Energy Research Institute, Tokai, and TRIGA Mark II of St. Paul's University, Yokosuka.

1. Non-destructive thermal neutron activation analysis

Besides major elements, *i.e.* Co and Ni, some trace elements can be determined by this procedure. The radiochemical data on (n, γ) reactions by thermal neutrons used for this procedure are presented in Table 1. Because of the low contents of some elements in some meteorites analyzed, some of the nuclides in this table could not be detected in the practical measurements.

In the mean time, it was noticed that higher specific activities could be obtained for the required elements by irradiation with a higher neutron flux for a shorter time, because ^{59}Fe and ^{60}Co produced from main component of iron meteorites have relatively long half lives.

The neutron irradiation condition was changed from a thermal neutron flux of 1.5×10^{12} neutrons/cm^2·sec for 1 and 6 hours (TRIGA Mark II) to 8×10^{13} neutrons/cm^2·sec for 40 minutes (JRR-4). About 50 mg of each meteorite sepcimen was used in this procedure. After 1 to 2 hours cooling, the irradiated meteorite samples and monitors are analyzed with a Ge (Li) γ-ray spectrometer.

2. Radiochemical neutron activation analysis—1

Because one of major purposes of this method is the determination of trace amounts of germanium using ^{75}Ge (half life 82.78 min.), it is necessary to count its γ-ray activity within 1 or 2 hours after thermal neutron irradiation of iron meteorites. The Al-capsule commonly utilized for neutron irradiation is not adequate for this purpose because of high specific activities produced from impurity elements in Al. Therefore, polyimid capsules were used in this experiment.

The radiochemical data for the elements analyzed by this procedure are listed in Table 2.

About 50 mg of sample was weighed accurately, sealed in a quartz tube and irradiated at a flux of 8×10^{13} or 5.5×10^{13} neutron/cm^2·sec for 6 or 12 hours in JRR-4 or at a flux of 2×10^{13} neutron/cm^2·sec for 24 or 30 hours in JRR-3. Monitors were also sealed in quartz tubes and irradiated simultaneously with samples. Soon after irradiation, chemical separation of meteorite samples and Ge monitors were performed as quickly as possible.

Fig. 1 shows the schematic diagram of the post-irradiation chemistry which has

Table 1. Nuclear data on (n, γ) reaction used for instrumental Neutron activation analysis in this work.

Stable nuclides	Isotopic abundances* (%)	Therm. neut. cross section** (burns)	Radionuclides produced	Half life**	Energies used for determination** (kev)	Relative intensity**
^{58}Fe	0.3	1.14	^{59}Fe	44.56 d	1099.2 1291.6	(56.5 %) (43.5 %)
^{64}Ni	0.91	1.49	^{65}Ni	2.520 h	366.3 1115.5 1481.8	20.3 64.4 100 (23.5 %))
^{59}Co	100	37	^{60}Co	5.2719y	1173.2 1332.5	100 100 (100 %)
^{50}Cr	4.35	15.9	^{51}Cr	27.701 d	320.0	(10.2 %)
^{63}Cu	69.17	4.4	^{64}Cu	12.700 h	1345.6	(0.6 %)
^{71}Ga	39.9	4.6	^{72}Ga	14.12 h	629.9 834.0 894.2 2201.6 2491.0 2507.7	25.5 100 (95.6 %) 10.3 27.3 7.8 13.4
^{74}Ge	36.5	0.53	^{75}Ge	82.78 m	264.6	(11.1 %)
^{76}Ge	7.8	0.16	^{77}Ge	11.30 h	264.6	(53.3 %)
^{75}As	100	4.4	^{76}As	26.33 h	559.1 657.0 1216.0	100 (45.2 %) 13.5 7.57
^{108}Pd	26.46	11	^{109}Pd	13.427 h	88.05	(3.6 %)
^{115}In	95.7	71	^{116m}In	54.13 m	138.3 818.7 1752.4	(3.33%) (11.6 %) (2.44%)
^{185}Re	37.40	111	^{186}Re	90.65 h	137.2	(9.4 %)
^{187}Re	62.60	75	^{188}Re	16.98 h	155.0	(15.3 %)
^{191}Ir	37.3	941	^{192}Ir	74.18 d	295.9 316.5 468.1	34.64 100 (82.9 %) 58.1
^{193}Ir	62.7	110	^{194}Ir	19.15 h	328.4 645.1 938.7	100.0 (13.0 %) 8.94 4.57
^{198}Pt	7.2	3.7	^{199}Pt	30.8 m $\downarrow \beta^-$		
			^{199}Au	3.148 d	158.2	(39 %)
^{197}Au	100	98.8	^{198}Au	2.697 d	411.8	(95.5 %)

* Data obtained from "Atomic Weight of the Elements 1979." by HOLDEN (1980).

** Data obtained from "Table of Isotopes." WILEY (1978). Values in parentheses in the 7th column are absolute γ-ray intensities of respective nuclides.

been modified in several points from the previous work. Before starting the sample decomposition, carrier mixture solutions were treated with conc. NH_4OH and dried with moderate heat in a distillation flask. The procedure consists of the distillation of groups of Ge and Re, and Ru and Os and precipitation of Mo, W, Ir, Pd, Pt, Au, Cd, As and Sb by H_2S in dilute acidic media followed by anion exchange separation.

Table 2. Nuclear data on (n, γ) reaction used for radiochemical neutron activation analyses—1.

Stable nuclides	Isotopic abundances* (%)	Therm. neut. cross section** (burns)	Radionuclides produced	Half life**	Energes used for determination** (kev)	Relative intensity
^{74}Ge	36.5	0.53	^{75}Ge	82.78 m	198.56 264.6	103 1000 (11.1 %)
^{76}Ge	7.8	0.16	^{77}Ge	11.30 h	210.9 215.7 264.6	58 53 100 (53 %)
^{75}As	100	4.4	^{76}As	26.33 h	559.1 657.0 (1216.0)	100 (45.2 %) 13.5 7.57
^{98}Mo	24.13	0.13	^{99}Mo	66.02 h	140.5 181.1 739.4	704 49.9 100 (12.7 %)
^{96}Ru	5.52	0.26	^{97}Ru	2.88 d	215.7 (324.6)	100.5 (86.1 %) 11.9_5
^{102}Ru	31.6	1.3	^{103}Ru	39.36 d	497.1 (610.3)	100 (86.4 %) 6.3
^{104}Ru	18.7	0.48	^{105}Ru	4.44 h	469.4 676.4 724.2	37.1 33.1 100 (48 %)
^{108}Pd	26.46	11	^{109}Pd	13.427 h	88.05	(3.6 %)
^{114}Cd	28.72	0.30	^{115}Cd	53.38 h	336.2 492.1 527.7	148 (50.1 %) 31.2 100.5
^{121}Sb	57.3	6.2	^{122}Sb	2.681 h	564.1 692.8	100 (70.4 %) 5.57
^{186}W	28.6	38	^{187}W	23.86 h	134.2 479.5 685.8	40.5 100 (≈21 %) 125
^{185}Re	37.40	111	^{186}Re	90.65 h	137.2	(9.4 %)
^{187}Re	62.60	75	^{188}Re	16.98 h	155.0 (478.0) (633.0)	930 (15.3 %) 64.9 77.7
^{192}Os	41.0	2.0	^{193}Os	30.6 h	73.0 138.9 460.5	82 109 $100._5$ (3.95%)
^{191}Ir	37.3	941	^{192}Ir	74.18 d	(295.9) 316.5 468.1	34.64 100 (82.9 %) 58.1
^{193}Ir	62.7	110	^{194}Ir	19.15 h	328.4 (645.1) (938.7)	100.0 (13.0 %) 8.94 4.57
^{196}Pt	25.3	0.76	^{197}Pt	18.3 h	77.4 (191.4)	100 (17 %) 20.5
^{198}Pt	7.2	3.7	^{199}Pt	30.8 m $\downarrow \beta^-$		
			^{199}Au	3.148 d	158.2 (208.2)	100 (39 %) 22.1
^{197}Au	100	98.8	^{198}Au	2.697 d	411.8	(95.5 %)

* Data obtained from "Atomic Weight of the Elements 1979." by HOLDEN (1980).

** Data obtained from "Table of Isotopes." WILEY (1978). Values in parentheses in the 6th column are energies used not for all samples, and in the 7th column are aboslute γ-ray intensities of respective nuclides.

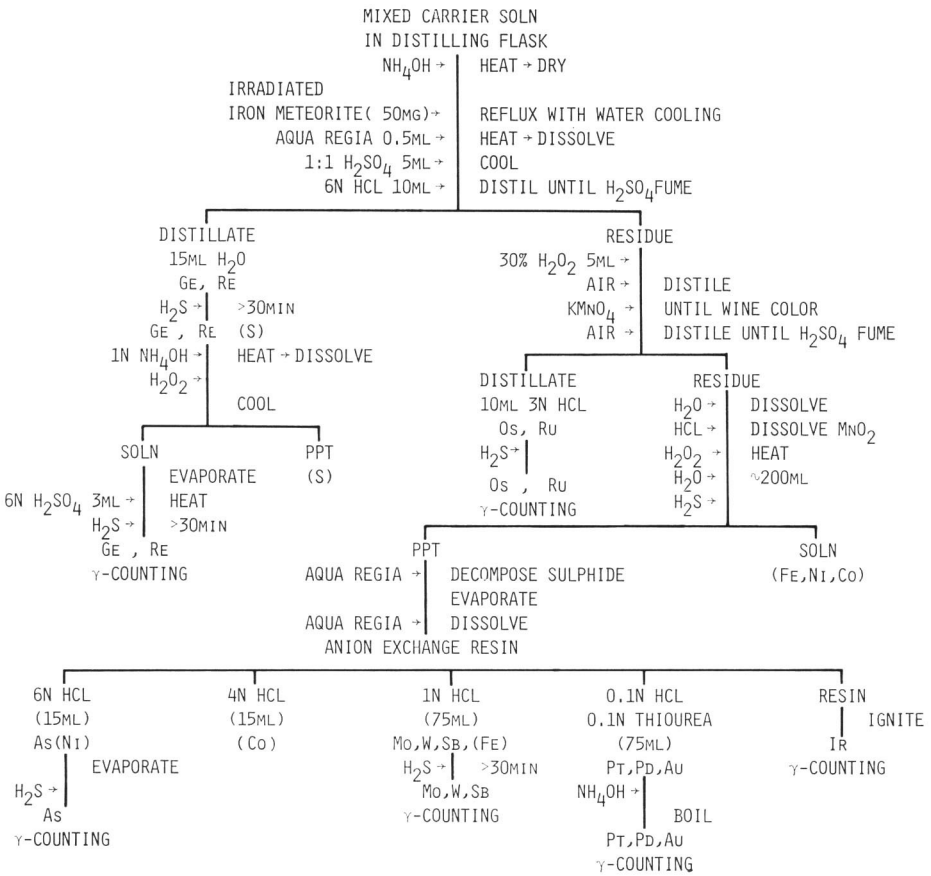

Fig. 1. Schematic diagram of the post-irradiation chemistry used for the radiochemical neutron activation analysis—1.

The distillation apparatus used in this work are shown in Fig. 2. Separated fraction of Ge-Re, As, Ru-Os, Mo-W-Sb and Pt-Pd-Au are precipitated as sulfides and analyzed with a Ge (Li) γ-ray spectrometer. The Ir fraction was obtained by igniting the anion exchange resin to ash after eluting all of other elements.

3. Radiochemicnal neutron activation analysis—2

Nuclides with half lives longer than 2 days were analyzed as follows and the radiochemical data are tabulated in Table 3.

Accurately weighed samples and monitors sealed in quartz tubes were irradiated in JRR-2 reactor at a flux of 3×10^{13} neutrons/cm^2·sec for about 266 hours.

The post irradiation chemistry is schematically illustrated in Fig. 3. The same distillation apparatus as the former method was also used here. Though the new method is still composed of several steps of distillation, precipitation, Na_2CO_3 fusion

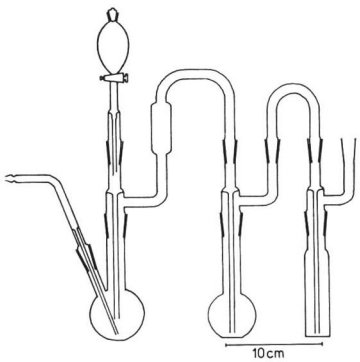

Fig. 2. Distillation apparatus for Ge, Ru, Os and Re.

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

Stable nuclides	Isotopic abundances* (%)	Therm. neut. cross section** (burns)	Radionuclides produced	Half life**	Energies used for determination*** (kev)	Relative intensities**
^{50}Cr	4.35	15.9	^{51}Cr	27.701 d	320.0	(10.2 %)
^{64}Zn	48.6	0.78	^{65}Zn	244.0 d	1115.5	(50.75%)
^{75}As	100	4.4	^{76}As	26.33 h	559.1 657.0 (1216.0)	100 (45.2 %) 13.5 7.57
^{98}Mo	24.13	0.13	^{99}Mo	66.02 h	140.5 181.1 739.4	704 49.9 100 (12.7 %)
^{96}Ru	5.52	0.26	^{97}Ru	2.88 d	215.8 (324.6)	100.5 (86.1 %) 11.9_5
^{102}Ru	31.6	1.3	^{103}Ru	39.36 d	497.0 610.3	100 (86.4 %) 6.3
^{109}Ag	48.17	4	^{110m}Ag	252.2 d	657.7 884.7 937.5	1000 (94.4 %) 771 363
^{121}Sb	57.3	6.2	^{122}Sb	2.681 d	564.1	(70.4 %)
^{123}Sb	42.7	4.0	^{124}Sb	60.20 d	602.7 722.8 1691.0	(98.3 %) (11.3 %) (49.1 %)
^{185}Re	37.40	111	^{186}Re	90.65 h	(122.6) 137.2	15.7 246 (9.4 %)
^{184}Os	0.020	3.0×10^3	^{185}Os	93.7 d	646.1 874.8 880.3	10000 (81 %) 849 677
^{190}Os	26.4	13	^{191}Os	15.4 d	129.4	(26 %)
^{191}Ir	37.3	941	^{192}Ir	74.18 d	295.8 316.5 468.1	34.64 100 (82.9 %) 58.1
^{197}Au	100	98.8	^{198}Au	2.697 d	411.8	(95.5 %)

* Data obtained from "Atomic Weight of the Elements 1979." by HOLDEN (1980).
** Data obtained from "Table of Isotopes." WILEY (1978). Values in parentheses in the 6th column are energies used not for all samples, and in the 7th column are absolute γ-ray intensities of respective nuclides.

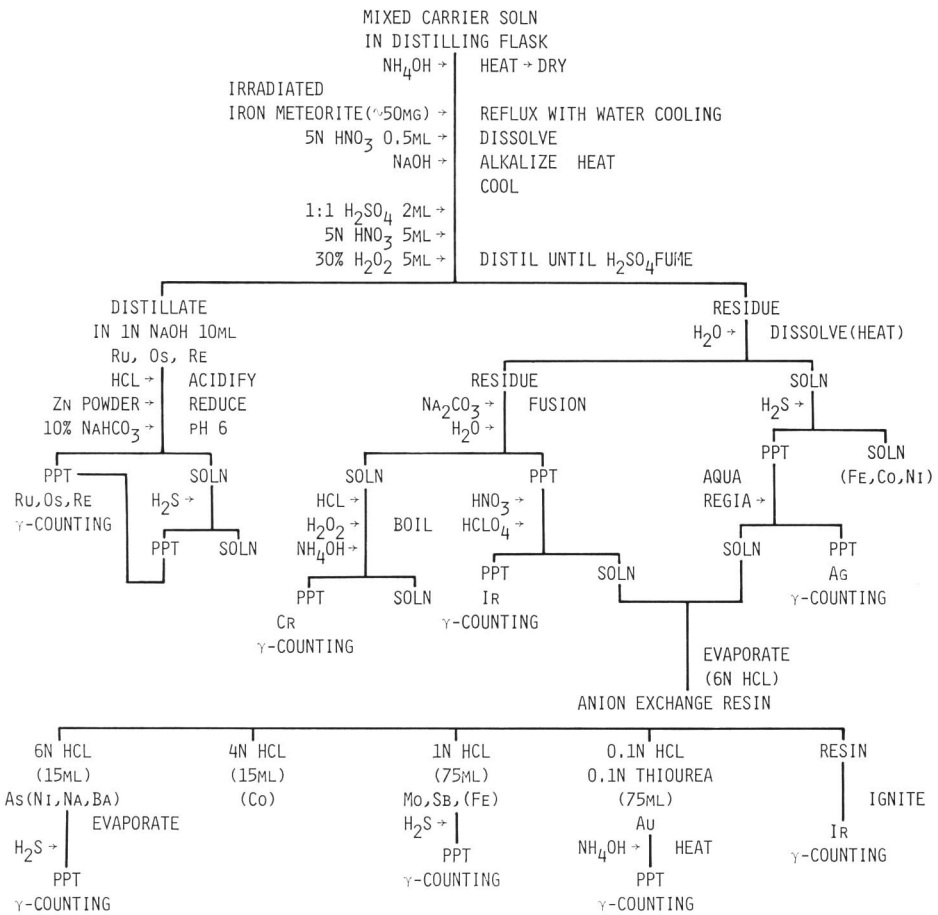

Fig. 3. Schematic diagram of the post-irradiation chemistry used for the radiochemical neutron activation analysis—2.

and anion exchange separation, it is simplified and improved compared with the previous method (SHIMA, 1979).

The separated fractions of Ru-Os-Re and Cr were precipitated as oxide, that of Ag as chloride and those of As, Mo-Sb and Au as sulfide, for counting. Finally the anion exchange resin was ignited to ash for the Ir fraction.

C. *Chemical Yields*

After radioactivities in separated fractions were diminished, chemical yields were determined by irradiating the separated samples again at a flux of 5×10^{11} neutron/cm^2·sec for one hour in TRIGA Mark II reactor. The radiochemical data used here are listed in Table 4. The produced γ-rays from carrier elements in each fraction

Table 4. Nuclear data on (n, γ) reaction used for measurements of chemical yields.

Stable nuclides	Isotopic abundances* (%)	Therm. neut. cross section** (burns)	Radionuclides produced	Half life**	Energies used for determination*** (kev)	Relative intensities
^{50}Cr	4.35	15.9	^{51}Cr	27.701 d	320.0	(10.2 %)
^{68}Zn	18.8	0.072	^{69m}Zn	13.76 h	438.7	(94.8 %)
^{74}Ge	36.5	0.53	^{75}Ge	82.78 m	198.56 264.6	103 1000 (11.1 %)
^{76}Ge	7.8	0.16	^{77}Ge	11.30 h	210.9 215.7 264.6	58 53 100 (53 %)
^{75}As	100	4.4	^{76}As	26.33 h	559.1 657.0 1216.0	100 (45.2 %) 13.5 7.57
^{98}Mo	24.13	0.13	^{99}Mo	66.02 h	(140.5) 181.1 739.4	704 49.9 100 (12.7 %)
^{96}Ru	5.52	0.26	^{97}Ru	2.88 d	215.7	(86.1 %)
^{104}Ru	18.7	0.48	^{105}Ru	4.44 h	(316.4) 469.4 676.4 724.2	23.6 37.1 33.1 100 (48 %)
^{103}Pd	26.46	11	^{109}Pd	13.427 h	88.05 (311.41)	1.48×10^4 (3.6%) 131
^{110}Pd	11.72	0.02	^{111m}Pd	5.5 h	169.5 391.3	14.3 100 (5.3 %)
^{109}Ag	48.17	4	^{110m}Ag	252.2 d	657.7 884.7 937.5	1000 (94.4 %) 771 363
^{110}Cd	12.51	0.10	^{111m}Cd	48.6 m	(149.6) 246	34.6 100 (94.2 %)
^{114}Cd	28.72	0.30	^{115}Cd	58.38 h	336.2 492.1 527.7	148 (50.1 %) 31.2 100.5
^{116}Cd	7.47	0.051 0.025	^{117}Cd ^{117m}Cd	2.43 h 3.32 h	273.3 1576.8 (1066.0) 1997.5	100 (29 %) 38.6 100 (23 %) 110
^{121}Sb	57.3	6.2	^{122}Sb	2.681 d	564.1 (692.8) (1140)	100 (70 %) 5.57 1.17
^{186}W	28.6	38	^{187}W	23.86 h	134.2 479.5 685.8	40.5 100 (≈21 %) 125
^{187}Re	62.60	75	^{188}Re	16.98 h	155.0 (478.0) (633.0)	930 (15.3 %) 64.9 77.7
^{192}Os	41.0	2.0	^{193}Os	30.6 h	73.0 138.9 (280.4) 460.5 (557.4)	82 109 31.5 100 (3.95%) 33
^{193}Ir	62.7	110	^{194}Ir	19.15 h	(293.5) 328.4 645.1 938.7	19.6 100.0 (13.0 %) 8.94 4.57

^{196}Pt	25.3	0.76	^{197}Pt	18.3 h	{(77.4) { 191.4	100 20.5	(17 %)
^{198}Pt	7.2	3.7	^{199}Pt	30.8 m ↓ β^-			
			^{199}Au	3.148 d	{158.2 {(208.2)	100 22.1	(39 %)
^{197}Au	100	98.8	^{198}Au	2.697 d	411.8		(95.5 %)

* Data obtained from "Atomic Weight of the Elements 1979." by HOLDEN (1980).

** Data obtained from "Table of Isotopes." WILEY (1978). Values in parentheses in the 6th column are energies used not for all samples, and in the 7th column are absolute γ-ray intensities of respective nuclides.

Table 5. Results of INAA.

Element	Okano	Tendo	Kuga	Tanakami	Shirahagi	Shirahagi No. 2. (Saotome)	Zaisho
Ni (%)	4.62±0.06	8.70±0.25	9.12±0.45	—	7.86±0.19	7.86±0.29	7.2±0.4
Co 〃	0.44±0.01	0.435 ±0.007	0.59±0.05	0.49±0.01	0.455 ±0.011	0.4±0.1	0.53±0.01
Cr (ppm)	—	29±3	14.9±1.2	70±6	278±12	231±5	6.55±0.14
Cu 〃	614±25	149±7	116±9	76±5	194±4	165±12	182±6
Ga 〃	56±1	24.8±2 1	16.7±0.5	15.6±0.4	2.6±0.2	2.5±0.1	13.1±0.3
Ge 〃	174±11	{54±23 {22.9±0.6	29.5±0.5	25.5±0.6	—	—	—
As 〃	5.6±1.3	7.0±0.3	30.6±0.2	7.4±0.2	4.4±0.2	3±1	24.7±0.5
Re 〃	0.72±0.01	0.033 ±0.004	—	<0.08	0.27±0.02	0.57±0.03	<0.06
Ir 〃	11.6±0.2	0.27±0.07	0.028 ±0.004	0.11±0.01	2.89±0.08	2.7±0.7	0.034 ±0.005
Pd 〃	1.8±0.2	3.44±0.10	3.9±0.4	2.2±0.2	3.3±0.4	3.1±0.1	3.9±0.1
Pt 〃	—	7.0±0.8	7.7±0.3	6±1	—	—	—
Au 〃	0.47±0.01	1.86±0.02	2.2±0.3	0.85±0.02	0.90±0.02	1.1±0.3	2.09±0.07
In 〃	—	0.019 ±0.004	—	—	<0.025	0.022 ±0.002	—

were determined with a Ge (Li) γ-ray spectrometer.

III. Results and Discussion

The results obtained by the procedures 1 to 3 are summarized in Table 5, 6 and 7, respectively. Although the results from procedure 3 are still preliminary ones, data obtained by the three methods agree rather well. Some elements in certain iron meteorites give distinct values, possibly due to inhomogeneity of the sample.

It has been said that two iron meteorites were found on the bed of Shirahagi river in Toyama Prefecture, Japan, in 1890 and 1892. They were named after the localities, Shirahagi and Saotome. Saotome was long hidden and was recently found again. Metallographical observation shows that both of them belong to the fine octahedrite

Table 6. Results of RNNA–1 (ppm).

Element	Tendo	Kuga	Tanakami	Shirahagi	Shirahagi No. 2 (Saotome)	Zaisho
Ge	24.8±0.9 53±2	28.6±0.4	48±2	0.12	0.127±0.007	85±3
As	9.0±0.6	24.0±2.4	7.0±0.1	4.3±0.6	4.7±0.1	32.9±0.9
Sb	0.2±0.1	0.23±0.02	0.046±0.005	0.022±0.002	0.018±0.004	0.37±0.02
Mo	7.0±0.3	7.6±0.5	8.5±0.6	5.7±0.4	6.3±1.4	5.8±0.2
W	0.53±0.06	0.21±0.1	2.9±0.2	4.9±1.1	3.8±0.3	1.2±0.3
Re	—	—	0.012±0.004	0.39±0.10	0.26±0.01	0.0016 ±0.0006
Ru	1.8±0.4	1.2±0.6 0.43±0.05	2.73±0.13	2.0±0.2	1.7±0.4	0.37±0.04
Os	0.35±0.03	0.4±0.1	0.26±0.02	8±2	6.0±0.3	0.20±0.03
Ir	0.37±0.10	0.07±0.01	0.56±0.02	3.2±0.1	3.72±0.02	0.18±0.03
Pd	3.3±0.3	3.8±0.2	3.50±0.05	3.3±0.3	3.2±0.6	6.0±0.2
Pt	9.6±0.4	7.9±0.1	6.8±0.2	8.5±2.4	6.6±0.1	1.4±0.1
Au	1.18±0.07	2.3±0.2	1.00±0.02	0.97±0.03	0.96±0.03	1.5±0.1
Cd	—	—	0.009±0.003	—	—	0.044±0.002

Table 7. Preliminaly results of RNAA–2 (ppm).

Element	Tendo	Kuga	Shirahagi
Cr	3±1	1.3±0.1	103±10
As	8.5±0.8	3.65±0.09	3.9±0.1
Sb	0.130±0.002	0.37±0.11	0.018±0.002
Mo	8.9±0.5	8.3±0.5	6.0±0.8
Re	0.2±0.1	—	0.36±0.04
Ru	5.9±0.2	1.24±0.19 0.4±0.1	—
Os	0.12±0.02	0.2±0.1	—
Ir	—	0.04±0.01	2.4±0.6
Ag	0.012±0.001	0.005±0.001	0.011±0.002
Au	1.19±0.03	2.4±0.1	0.97±0.06

class. Judging from the resemblance of their chemical compositions determined in this work together with metallographic observation between Shirahagi and the newly found meteorite, the so-called Saotome, we conclude that these meteorite must have been originated from the same mass in space. From this point of view, Saotome should be renamed Shirahagi No. 2.

Ga, Ge and Ir have been usually used as the indicator elements for the classification of iron meteorites. The present results confirm the classification made by one of us (SHIMA *et al.*, 1978; SHIMA, 1979), *i.e.* Tendo, Kuga, Tanakami and Shirahagi as IIIA, IIIB, IIIE and IVA, respectively. Other two meteorites Okano and Shirahagi No. 2 (Saotome) are classified by the present data as IIA and IVA, respectively.

Cosmochemical discussions and reports of analyses of other iron meteorites will

be made elesewhere.

IV. Acknowledgement

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