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SCANDIUM 45 IN METEORITIC IRONS

Masatake HONDA¹, Hisao NAGAI¹, Nobuo TAKAOKA², Kenji YAMAMURO¹,
Takashi KUME¹, Akihiro TAKAHASHI¹ and Shigeru AKIZAWA¹

¹College of Humanities and Sciences, Nihon University,
25-40, Sakurajosui 3-chome, Setagaya-ku, Tokyo 156

²Department of Earth Sciences, Faculty of Science, Yamagata University,
Koshirakawacho 1-chome, Yamagata 990

Abstract: This report presents the results of determinations of cosmogenic ⁴⁵Sc and light noble gases in iron meteorites. Sc and REE were simultaneously determined using radiochemical neutron activation method. This method is sensitive enough for detection of as little as lower than 10⁻¹³ gSc/g, compared to the level of cosmic ray effects in irons which are usually in the range of 10⁻⁹–10⁻¹² gSc/g. The lowest concentration found in irons was 2 × 10⁻¹² gSc/g in the Campo del Cielo and similar contents were observed in the Gibeon, DRP-78008 (and Brenham-metal). Even at these low levels, Sc seems mostly attributable to cosmic ray production.

The Sc content in irons is best compared with cosmogenic ⁴He. The ratio of Sc to ⁴He, approximately 1 × 10⁻¹² gSc/1 × 10⁻⁸ cc ⁴He, or an average atomic ratio of 1:19 ± 3, was observed independent of shielding.

Technical aspects of metal phase separation from ordinary chondrites were studied. In one Peace River metal sample, 0.9 ppbSc was found. This number corresponds to 0.01% of the stone phase in the metal. This level of contamination seems still higher than the cosmogenic level of 0.2 ppbSc expected in the metal phase of this meteorite.

1. Introduction

This report presents the results of determinations performed in our laboratories of cosmogenic ⁴⁵Sc and light noble gases in iron meteorites. Brief discussions concerning the production rates of these products are presented. Technical aspects of metal phase separation from ordinary chondrites for sample preparation are also described.

In studies of cosmogenic nuclides as early as 1958, WÄNKE proposed that Sc can be determined in iron meteorites as a cosmogenic product (WÄNKE, 1958, 1960). Recently, as we have reported (HONDA *et al.*, 1984, 1985a, b), the scandium content in surface cleaned iron meteorites has proven useful as a direct measure of cumulative cosmic ray effects in meteorites because Sc has practically no natural background in these samples. Besides neutron activation methods (NAA), glow discharge mass spectrometry has also been applied to these studies (SHIMAMURA *et al.*, 1986).

Very few cosmogenic nuclides can be determined by NAA, and Sc is a non-volatile element which is free from possible loss of the products in space. The problem is then only in the natural background of Sc in the samples.

2. Determination of Scandium 45

The radiochemical neutron activation method (RNAA) was used for the determinations. Neutron doses were 10^{17} – 10^{19} n/cm². ⁴⁶Sc is produced by a ⁴⁵Sc (n, γ) ⁴⁶Sc reaction. It is then determined by measurements of 889 and 1120 keV gamma rays. As preparation, the surfaces were cleaned before and after the irradiation; using aqua regia more than 10% of the sample was removed from the surface of the original sample. After the irradiation the etched sample was weighed and dissolved in HNO₃ (1:2) or in persulfate solution (see Section 4 below); insoluble materials were removed by filtration. The Sc carrier (10 mg) and Cr hold back carrier were added to the solution, which was evaporated to a small, concentrated volume; distilled water was then added to dilute it to 100 ml/g sample. The solution was filtered and passed through a 10 m (for <1 g sample) cation exchange column in the HR form, and all cations were adsorbed once. A strongly colored band of FeR reached toward the end of the column, and the column was oversaturated with Fe³⁺ occasionally. About 50 ml of 2N HCl were passed through the column to elute completely Fe, Ni and other cations. Sc was then eluted with HOAc acidic 2N (NH₄)OAc. The effluents (about 10–15 ml of the acetate solution) were recovered starting just before the saturation of NH₄⁺. Sc and other cations which form stable complex anions with acetate were recovered simultaneously in this fraction. All REE (rare earth elements) were collected quantitatively along with Sc and Y in their carrier free states. The effluents were evaporated to dryness to remove the ammonium salt. Sc(OH)₃ was then reprecipitated with ammonium hydroxide in the presence of H₂O₂; Sc₂O₃ was recovered after ignition. The gamma ray counting was performed with the oxide, and the chemical yield was determined first by weighing the oxide and later by reactivation for Sc. In many cases, quantitative recoveries were found. The latter method gave more reliable data. This is the simplest and most convenient method for the radiochemical separation of Sc and REE in a group. Small amounts of impurities such as ¹⁹²Ir, ⁵⁰Fe, ⁶⁰Co and ⁵¹Cr were commonly present in the final sample. Sometimes the 884.5 keV γ peak of ¹⁹²Ir interfered with the 889 keV γ peak of ⁴⁶Sc. If necessary, an anion exchange purification step was performed with the 8M HCl solution and the cation exchange could be repeated. This method is sensitive enough to detect Sc contents as low as, lower than, 10^{-13} gSc/g. On the other hand, the level of cosmic ray effects on the irons is usually in the range of 10^{-9} – 10^{-12} gSc/g.

Table 1 shows the data we have obtained thus far, with some revisions from our previous determinations (HONDA *et al.*, 1984, 1985a, b). The lowest figure in the table is 2×10^{-12} gSc/g for one sample of the Campo del Cielo, one of the largest iron meteorites. Similar contents were observed in the Gibeon, DRP-78008 (and in the metal phase of the Brenham pallasite). Even at these levels, Sc seems mostly attributable to cosmic rays, as described below from comparisons with ⁴He contents. Therefore, we can assume that the Sc content in irons directly indicates the total cosmic ray exposure with no correction necessary for Sc from a natural origin. Even in the sulfide inclusions, the naturally occurring Sc seems to be lower than the cosmogenic, as shown in the table by the Odessa troilite sample. Possible interference due to ⁴⁶Ti(n, p)⁴⁶Sc is negligibly low, because Ti content has been found to be lower than 0.1 ppm in these samples

Table 1. Scandium contents found in meteoritic irons.

Sample	ID	Size (g)	Sc found ppb	⁴ He 10 ⁻⁸ cc/g	⁵³ Mn* dpm/kg
ALH-762, IA	54	0.68	1.55±0.16		556±21
ALH-78100, IIA	72	0.76	0.66±0.07		
ALH-78252, IVA	83	0.44	0.76±0.08		376±14
Boxhole, IIIA	H125-93	0.82	0.12±0.02		
Braunau, H, IIA	H. Wänke	0.11*	0.080±0.011	32.7#	512±20
		1.06	0.066±0.007		
Brenham, Pall.	metal, LJ.	1.1	0.010±0.009	<2#	<15
Campo del Cielo IA	N.	0.49	0.064±0.006		
Campo del Cielo IA	AII-215, No. 2	1.11	0.002±0.001	1.7#	
Canyon Diablo IA	H34-5031	2.5	0.20±0.02		165±40
		0.90	0.25±0.03		
Chinga, IVB	N.	0.88*	0.058±0.005	85	
Charcas, IIIA	P. Signer	0.26	2.06±0.21	2040#	370±50
DRP-78008, IIB	5	0.09*	0.013±0.001	6.4	2.1±0.2
DRP-78007, IIB	72; 77	0.29	0.21±0.02	282	144±6
Gibeon, IVA	N.	1.29*	0.007±0.001	9.8	
		0.81*	0.008±0.001		
Gibeon, IVA	LJ. USNM	0.80*	0.011±0.001		0±2
Grant, IIIB	K+100	1.08	1.84±0.18	1930#	320±40
	(surface)	0.91	1.69±0.16		
Guin, IIE	N.	1.15	0.12±0.01	110; 102#	
Henbury, IIIA	H193-488	0.86	0.60±0.08		331±20
Henbury, IIIA	H193-493	0.77	0.89±0.09		
Nativitas, IIIA	H112-30	0.64	0.34±0.20		
Odessa IA	H91-2	0.089*	0.12±0.01		20±3
Odessa IA, troilite	(H91-2)	0.95	0.13±0.02		
Odessa IA	H91-202	0.90*	0.16±0.02		
Odessa IA	H91-3	0.11*	0.91±0.09		190±25
		0.47*	0.84±0.08		
PGP-77006, IA	71	0.61	1.21±0.12		569±23
Tawallah Val., IVB	N.	0.42	0.54±0.05	978	
Toluca, IA	H128-388	0.8*	0.083±0.008		ca. 4
Tocopilla, IIA	N.	1.26	0.48±0.05	360	
Trenton, IIIA	AII-22+2	0.59	1.62±0.16	1400#	590±14
Treysa, IIIB	interior	0.88	1.80±0.18	1920#	377±14
Warburton R. IVB	N.	0.92	1.66±0.17	2550#	
Xiquipilco, IA	H128-400	1.02	0.15±0.02		
Y-75031, AN	52	0.29	2.34±0.23	2629#	474±19
Y-790517, IIIA	72	1.02	0.39±0.20		552±20
Y-790724, IIIA	81	0.54	0.20±0.16		518±18
Y-791076, (IIC)	62	0.12	2.52±0.25	2990	
Y-791694, Ni rich ataxite	68	0.18	0.088±0.009	96	

“N”: D. New; “H”: Am. Meteor. Lab.

* Neutron dose: 10¹⁹ n/cm².

Others: Data compilations by SCHULTZ and KRUSE (1983) for ⁴He and by NISHIZUMI (1987) for ⁵³Mn; data for Braunau, Y-75031, Y-790517 and Y-790724 are unpublished data by NISHIZUMI.

(SHIMAMURA *et al.*, 1986), and the effect has been estimated at 0.005 ppbSc/ppmTi, even in the RSR position of the Triga II reactor having a Cd ratio of about 3. The errors quoted are usually $\pm 10\%$, including uncertainties in the chemical yield and geometry errors in the gamma ray countings. Some larger errors were caused by a shorter counting time. The back-ground, 0.03 cpm, at 1120 keV (^{214}Bi), was corrected for the same energy peak as ^{46}Sc .

Our chemical separation scheme for Sc is entirely based on the cation exchange process, and the REE are recovered along with the Sc carrier separated from iron and nickel. When samples contain Sc from terrestrial contamination, determination of REE (and uranium through fission products) can be expected in the final gamma ray spectra of the Sc fraction. In the Gibeon and DRP-78008 samples described above, and in other samples, no significant gamma ray peaks of REE activities were detected, except in the Odessa troilite. REE content must thus be at or lower than 10^{-12} g/g. In the metal fractions separated from chondrites, some REE—such as Eu, Tb, Yb and Lu—were observed at the respective levels relative to the Sc content.

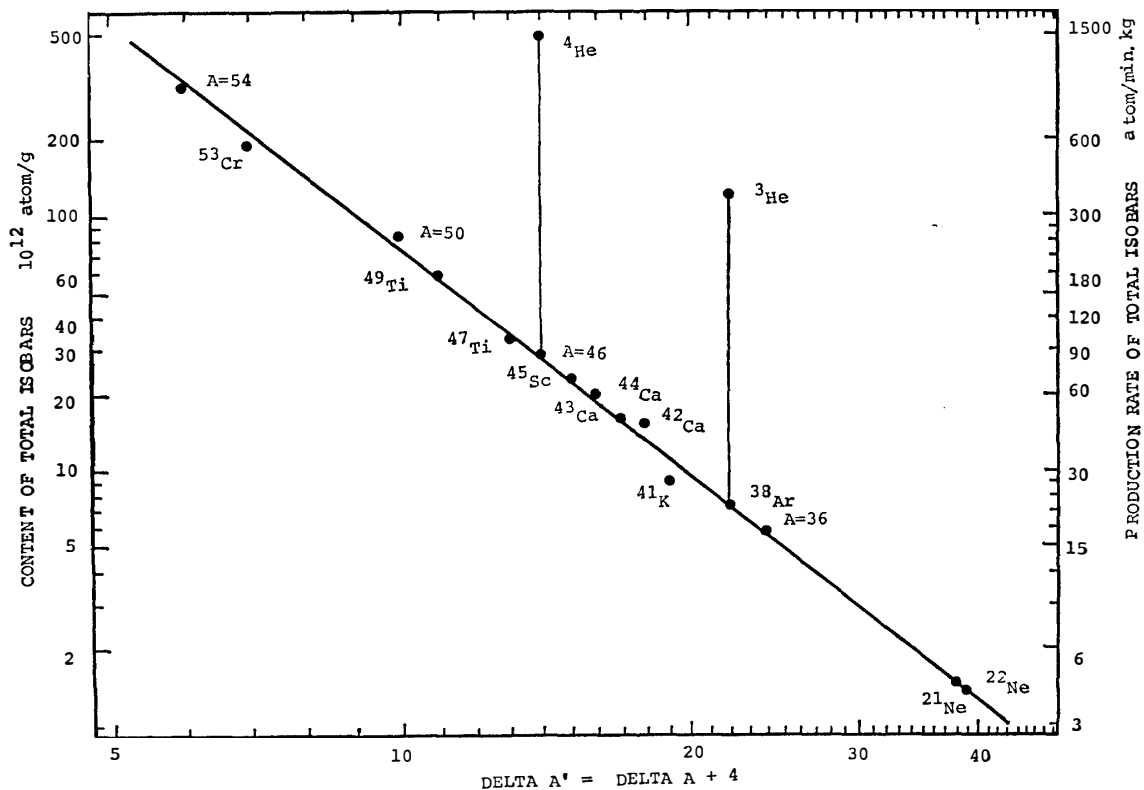


Fig. 1. Systematics among stable nuclides observed in the interior sample of the Grant iron meteorite

$$C(\Delta A) = k_1' \times T \cdot \exp(-k_2' \Delta A),$$

where C : content of total isobar; T : exposure age; $k_1' = 1.3 \times 10^5$ atom/kgmin; $k_2' = 2.91$. The productions of two helium isotopes, ^4He and ^3He , are estimated at 18.3 and 16.7 times those of $A=46$ and $A=38$ products respectively (HONDA, 1985). Therefore the production of ^{45}Sc is considered to be in parallel with that of ^4He . The left vertical scale indicates the contents and the right one is the production rates based on the exposure age, 6×10^8 y.

3. Production Rate of Cosmogenic ^{45}Sc

On the whole, the Sc content in iron meteorites is best compared with the cosmogenic ^4He content (Fig. 1). The ratio of Sc to ^4He in our data as well as the ratio in WÄNKE's data (1960) is very close to $1 \times 10^{-12} \text{ gSc} / 1 \times 10^{-8} \text{ cc } ^4\text{He}$; that is $(^{45}\text{Sc}/^4\text{He})_{\text{atom}} = 1/19 \pm 3$ ($n=13$). This ratio is due to the relation of the delta A (ΔA) values, the mass difference between the target mass and the product mass. For ^{45}Sc , the target mass (56) minus the mass of Sc (45) equals 11. The effective ΔA of ^4He has been estimated at 10 ($56-46=10$) (HONDA, 1985). The two ΔA values are close, and the ratio of the two production rates is nearly constant, evaluated at $P(^4\text{He})/P(^{45}\text{Sc})=20$ because the production rate of ^4He relative to the total isobaric yield for $A=46$ is estimated at 18.3, and the ratio of the production rates for $A=46$ to that for $A=45$ is slightly higher than one. This can also be demonstrated by plotting $^4\text{He}/^{45}\text{Sc}$ as a function of $^4\text{He}/^{21}\text{Ne}$ (ranging 200–700), a shielding index, which gives a horizontal line within experimental error. Significant discrepancies between Sc and ^4He contents were observed with Braunau, Tawallah Valley (and DRP-78008). The exposure ages of some meteorites seem relatively short; besides the Braunau, they are the Y-791694, Guin, Chinga and Tocopilla, all at 10^7 – 10^8 years.

Through the determinations of ^{45}Sc , ^{10}Be and other cosmogenic nuclides an exceptional history was detected for the Braunau iron meteorite (IIA, fell 1847). It has been pointed out already that the lower ^3He content in this object is attributable to ^3H (and ^3He) loss in space (HINTENBERGER and WÄNKE, 1964; HINTENBERGER *et al.*, 1967). The results found in this work suggest, however, that not only ^3He but also other volatile

Table 2. Comparison of estimated and experimental data for the Braunau iron meteorite.

	$\Delta A+4$	Estimated data		Experimental data	References
		model 1 T (exposure: Ma) 18	model 2 9		
^{58}Mn	7	494	418	512 ± 20 dpm/kg	Table 1
^{45}Sc	15	0.064	0.032	0.07 ± 0.01 ppb	this work
^4He	14	70	35	32.7 10^{-8} cc/g	(a)
^3He	22	20	10	3.3 10^{-8} cc/g	(a)
^{21}Ne	39	0.26	0.13	0.13 10^{-8} cc/g	(a)
^{38}Ar	22	1.16	0.58	0.64 10^{-8} cc/g	(b)
^{36}Ar	24	0.72	0.36	0.55 10^{-8} cc/g	(b)
^{10}Be	22	3.4	3.3	3.4 ± 0.2 dpm/kg	(c)
^{36}Cl	24	15.8	15.8	22 ± 1 dpm/kg	(d)
^{26}Al	34	2.4	2.4	2.4 ± 0.3 dpm/kg	(c), (e)
k_1' :		114 $\times 10^3$ atom/kgmin			
k_2' :		2.64	(see Fig. 1)		

(a) HINTENBERGER and WÄNKE (1964).

(b) COBB (1965).

(c) NAGAI *et al.* (1987).

(d) VILCSEK and WÄNKE (1963).

(e) 2.6 dpm/kg by HAMPEL and SCHAEFFER (1979).

light noble gases were lost by a factor of 2, and that the exposure age is about 20 Ma (model 1) instead of the less than 10 Ma (9 Ma by model 2) based on ^4He , Ne and Ar isotopes (Table 2).

4. Metal Phase Separation from Chondrites

To determine high energy cosmogenic nuclides produced in Fe-Ni alloys in chondrites, as well as to determine the elemental partitions for trace elements between metal

Table 3. Scandium and other elements in chondritic irons.

(A) Metal phase separation from chondrites.

Name ID	Sample wt. (g)	Crude metal (g)	HF step	Crush. step	Before dissol. (g)	Insol. stone (%)	Metal in solution	
							mgMg/g (st. %)	mgAl/g (st. %)
Bruderheim, L6								
16-2*	10.8	1.17	1	0	0.74	1.2	0.37(0.26)	0.04(0.36)
B-156	8.9	1.00	1	3	0.63	0.06	0.12(0.08)	0.0 (0.0)
Peace River, L6								
	12	1.46	3	3	0.80	0.09	0.13(0.09)	0.01(0.09)
Y-74192, H5								
	2.3	0.42	3	3	0.36	0.0	0.51(0.35)	— —
Jilin, H5								
T-E	18	6.1	3	>3	2.8	0.8	0.70(0.47)	0.04(0.36)

st. %: Stone (chondrite) content (%) estimated in the metal fractions, in the solution (by atomic absorption), and the insoluble part, taking 14%Mg and 1.2%Al in chondrites.

* Bruderheim 16-2 was not treated by the purification in Step 4. By NAA, 70 ppbSc and 28 ppmNa were found in the insoluble part, which correspond to 0.9% and 0.4% stone respectively. On the other hand, besides Mg and Al, 0.3%Sc (25 ppb) and $0.5 \pm 0.2\%$ Eu (0.35 ppb) were detected in the solution.

(B) Composition of purified metal fraction of the Peace River chondrite.

Chondrite Sc:	8.4 ± 0.8 ppm	
Metal fraction*		
Dissolved metal:**		Stone %
Sc	0.90 ± 0.08 ppb	0.011
Sm	(0.08) ppb	(0.04)
Mg	20 ppm	0.014
Insoluble residue (stone):		
Sc	2.2 ± 0.15 ppb	0.026
Na	2.1 ± 0.2 ppm	0.030
Solid metal:		
Na	2.7 ± 0.5 ppm	0.04
Mg	(110) ppm	(0.08)
Al	10 ± 1 ppm	0.08

* Recovered metal: 6% of the chondrite.

Purified from the metal sample listed in Table 3A.

** In other chondrites, 0.02 and 0.03% stones (based on their Mg contents) were found in the Bruderheim and Leedey respectively.

and other phases and to separate trace elements in metal, metal grains must be cleanly separated from the bulk matrix. Various techniques for this process have been studied at many laboratories (VILCSEK and WÄNKE, 1965; BEGEMANN and VILCSEK, 1969; BEGEMANN *et al.*, 1976; NYQUIST *et al.*, 1973). In our work, originally to determine the concentration of cosmogenic ^{26}Al and ^{10}Be in metals (NAGAI *et al.*, 1987), efforts have been made to separate clean metal grains, even if for cosmogenic Sc and REE they are not yet satisfactory. Table 3 shows some of the results obtained. Our current process is as follows:

Step 1: The chondrite fragments are gently crushed and the silicates are ground into fine grains and sieved using a nylon net with a mesh size of 50.

Step 2: The crude magnetic fraction is separated using a hand magnet. This step separates approximately 10% of an L-chondrite sample and 20–30% of an H-chondrite sample.

Step 3: Most siliceous materials and sulfides are removed from the surface of the metal grains using a 46% HF solution at room temperature for 10–15 min (BEGEMANN *et al.*, 1976; NYQUIST *et al.*, 1973). Initially, HF reacts vigorously producing H_2S . With cleaner samples the reaction is not extensive and most of the metal remains unaffected.

Step 4: The first crushing step is repeated by hitting them gently with a hammer between a stainless steel pestle and plate; eventually the surface of larger metal grains are flattened. Some extremely large grains are rejected by a hand picking at this stage. Stones evolved in this process are removed by washing with alcohol in an ultrasonic bath. The 3rd and 4th steps are then repeated for further purification until no visible evolution of stones is observed in the suspension. This cycle reduces the stone content by a factor of more than 10 without markedly decreasing the yield (usually a reduction of the recovery is 5–15% per cycle), as shown in the table. On the other hand, it presents the problem of contamination from tool surfaces. A grain size fractionation is performed before the next dissolution step.

Step 5: Metal grains are dissolved in a fresh neutral aqueous solution of ammonium persulfate, $(\text{NH}_4)_2\text{S}_2\text{O}_8$ (recrystallized from the alcoholic solution; 6 g/g metal in 10–20 ml), for about 30–50 min (BEGEMANN and VILCSEK 1969; BEGEMANN *et al.*, 1976). Although neutral this solution dissolves a considerable part, up to about one half, of the remaining stones in the metal fraction. The solution is filtered through a membrane filter and minor insoluble residue is separated into stones and remaining magnetic parts (a high nickel content phase) using a magnet.

The contents of Na, Mg, Al, Ca, Sc, V and REE were examined in the solutions, the insoluble silicates and the solid metal samples, by the atomic absorption and neutron activation. The level of chondrite (stone) contamination in the metal is estimated, simply assuming that the concentration of these elements (especially Sc and Mg) in the samples relative to that in the bulk is reflecting, though it might not be always the case. With H chondrites, the purities of the metals obtained were lower than with L chondrites. Usually finer grain size fractions have higher purities.

Some experiments with L chondrites indicate that less than 0.1% stone content was found in the metal solution (recovered 5–6% of chondrites). Beyond this level, some siliceous minerals may still remain even after the nearly complete removal of olivine and feldspar (SHIMA and HONDA, 1967). In one sample of the Peace River

(Table 3B), 0.9ppbSc was found in the solution, corresponding to 0.01% of the (Sc bearing) stony phase in the chondrite and agreeing with the value calculated from Mg, Na and other elements. This Sc concentration is still higher than the cosmogenic levels of 0.2ppbSc in the Peace River and 0.1ppbSc in the Bruderheim, but it is not necessarily being a limit because purity increases with each step.

On the other hand, the level of 0.1% is already sufficiently low as the metal sample for ^{26}Al and ^{10}Be determination with minor corrections; it is also satisfactory for determining other cosmogenic nuclides including noble gases, especially Ne, and perhaps also ^4He in cases where proper corrections can be made. In our experiences, however, this technique did not work as satisfactorily for severely weathered chondrites and E chondrites, for example, as it did for fresh ordinary chondrite specimens.

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