TRACE ELEMENT COMPOSITION AND DISTRIBUTION OF YAMATO-691, AN UNEQUILIBRATED ENSTATITE CHONDRITE

Mitsuru Ebihara

Department of Chemistry, Faculty of General Studies, Gunma University,
2, Aramaki-cho 4-chome, Maebashi 371

Abstract: Twenty one trace elements (Re, Ir, Pd, Au, Ag, Se, Te, Zn, In, Cd, Tl, La, Ce, Nd, Sm, Eu, Gd, Tb, Tm, Yb and Lu) were determined by radio-chemical neutron activation analysis for the bulk sample and acid (nitric acid and aqua regia)-soluble and residual phases of the Yamato-691 (EH3) meteorite. No fractionation was confirmed in siderophile elements among Y-691, EH4–5s and EL6s. Among volatile chalcophiles, Zn and In in Y-691 are in the ranges of their abundance variations in EH4–5s, whereas Cd and Tl were observed to be more enriched in Y-691 than in EH4–5s.

About 15% of light rare earth elements (REEs) and 30% of heavy REEs were confirmed to be distributed in acid-residual phases (pyroxene-dominant silicates). A small but difinite negative anomaly of Yb was found in acid-residues, but neither Eu nor Ce anomalies were confirmed. Distributions of the REEs with a negative Yb anomaly in acid-residues must be a strong constraint on formation hypotheses for enstatite chondrites.

1. Introduction

Enstatite chondrites contain a large variety of unique minerals, most of which are thought to have formed under the highly reduced conditions (Mason, 1966; Keil, 1968). For instance, calcium and magnesium are well known to be present for the most part in the form of sulfides (oldhamite and niningerite, respectively), hence these elements must have behaved as chalcophile elements rather than lithophiles at the formation of enstatite chondrites.

Enstatite chondrites are classified into two groups: high-iron EHs, which are petrologic types 3–5, and low-iron ELs, which are petrologic type 6 (Sears *et al.*, 1982). In spite of rather large differences in mineralogical features between EH3 and EH4, 5 chondrites (Prinz *et al.*, 1984; McKinley *et al.*, 1984), only a faint difference in chemical composition has been recognized between them (Weeks and Sears, 1985; Kallemeyn and Wasson, 1986).

Host phase(s) of rate earth elements (REEs) in enstatite chondrites have been examined for many years by several investigators. Using a dissolution method, Shima and Honda (1967) were possibly the first to try to make clear this problem and suggested that REEs were not hosted by oldhamite. Sears *et al.* (1983) and Frazier and Boynton (1985) tried to separate oldhamite mechanically. Though they could not isolate the mineral, they came to the conclusion that oldhamite must be the host mineral for the REEs.

Recently LARIMER and GANAPATHY (1987) succeeded in isolating oldhamite in Indarch (EH4) and found that REEs were present in this mineral with enrichment factors of 100 and 30 for light REEs and heavy REEs, respectively, relative to the bulk abundances.

In this study, concentrations of 21 trace elements including 10 REEs were determined by radiochemical neutron activation analysis for bulk sample, and acid (nitric acid and aqua regia)-soluble and -residual fractions of the Y-691 chondrite (EH3). Additionally applying a stepwise dissolution method, modified after a Shima and Honda's method, REEs were studied for their distribution in Y-691 and their host phase(s) were inferred.

2. Experimental

2.1. Sample

Several chips were delivered from the National Institute of Polar Research (NIPR) for a consortium study on the Y-691 enstatite meteorite. Weathering degree of this meteorite is not listed in the catalog issued by NIPR. The sample (Y-691,84) alloted was stated to be taken from the center portion, hence it looks relatively fresh.

A total of 400 mg of the meteorite was ground in a clean agate mortar.

2.2. Radiochemical neutron activation

Basically the radiochemical procedures of University of Chicago (KEAYS *et al.*, 1974; ANDERS *et al.*, 1987) were employed for siderophile and chalcophile elements. Ten REEs were determined in the same manner as that described in EBIHARA (1987).

To analyze the bulk sample, an aliquant (about 70 mg) of the powdered sample was sealed in a synthesized quartz tube. Meteorite samples including the Allende meteorite for a reference standard were irradiated in the JRR-4 reactor of the Japan Atomic Energy Research Institute (JAERI) for 12 h at $5 \times 10^{13} \, \text{n} \, \text{cm}^{-2} \text{s}^{-1}$. After a cooling for 4 days, chemical procedures were started.

2.3. Phase separation by acid

About 100 mg of the powdered meteorite sample was sealed and irradiated in the same manner as for the bulk analysis. Being transferred into a glass vial (5 m*l*), the sample was treated with 1 m*l* of 1 M nitric acid at 90°C for 30 min at first and the residues were again treated twice with 2 m*l* of 1 : 1 aqua regia at 90°C for 30 min and 1 h. Each leachate was united, neutralized and dried. Being washed with water 3 times, insoluble materials (referred to as *acid*-residues in the following) were dried, weighed, and measured for their gamma-ray activities nondestructively. Acid-residues were then transferred quantitatively into a zirconium crucible and subjected to radiochemical separation, along with acid-leachate.

In these treatments, metal and sulfides are expected to be mostly dissolved. Acid residues consist mainly of pyroxene. Some glass and plagioclase would consistently be present because of their difficulties in acid dissolution (SHIMA, 1974).

2.4. Stepwise dissolution

About 200 mg of the powdered sample was leached twice with 10 m*l* of acetate

buffer solution (0.1 M ammonium acetate—0.1 M acetic acid, pH 4.7) for 1 h at room temperature. The residues were then treated twice with 10 ml of 0.1 M ammoniacal EDTA (ethylenediaminetetraacetic acid) (pH 9.5) at room temperature. The residues were succeedingly leached thrice with nitric acid under different conditions. Each leachate and acid residues were analyzed nondestructively by means of instrumental neutron activation using a Triga II reactor at the Atomic Energy Research Institute, Rikkyo University.

3. Results and Discussion

3.1. Elemental abundances of trace elements in Y-691

3.1.1. Siderophiles and chalcophiles

Analytical data on siderophiles and chalcophiles in Y-691 are tabulated in Table 1, along with the data for Allende and their literature values. Elemental abundances of these elements normalized to C1-chondrite values (C1's) (Anders and Ebihara, 1982) are illlustrated in Fig. 1, in which ranges of abundance variations of each element for EH4-5 and EL6 chondrites (Hertogen et al., 1983) are also shown. Except for Te and In, all the elements analyzed are found to have higher abundances than C1's.

Table 1. Siderophile and chalcophile elements in Y-691 and Allende.

Meteorite	Re ppb	Ir ppb	Pd ppb	Au ppb	Ag ppb	Se ppm	Te ppm	Zn ppm	In ppb	Cd ppb	Tl ppb
Y-691	66.2	647	838	347	261	25.4	1.63	382	46	1450	170
(lit.*)[1]	—	557	—	281	—	21.5	1.95	—	—	—	—
Allende	83.0	863	709	153	85.7	9.9	0.903	112	25.4	449	65.6
(lit.)[2]	65.0	744	697	134	94.9	7.4	0.954	115	29.6	446	62.9
(lit.)[3]	—	—	—	145	98.4	8.77	1.020	116	30.7	508	61.2
(lit.)[4]	—	785	—	145	—	8.23	—	119	35	436	—

* literature: 1. Kallemeyn and Wasson (1986), 2. Wolf et al. (1983), 3. Dennison et al. (1986), 4. Kallemeyn and Wasson (1981).

Siderophile elements (Re, Ir, Pd and Au) in Y-691 were found not to be fractionated from those in EH4-5s and EL6s, although the H and L designations imply a difference in siderophiles (Sears et al., 1982). Among moderately volatile chalcophiles (Ag, Se and Te), Ag in Y-691 had an abundance similar to those in EH4-5s, whereas both Se and Te are depleted in Y-691 compared with those in EH4-5s. A depletion of moderately volatile elements in EH3s compared with those in EH4-5s was first pointed out by Weeks and Sears (1985), and later confirmed by Kallemeyn and Wasson (1986). Among volatile chalcophile elements, Zn and In are in the ranges of their abundance variations in EH4-5s, whereas much volatile Cd and Tl were observed to be relatively more enriched in Y-691 than in EH4-5s, suggesting that the Y-691 meteorite had escaped from events causing volatile loss such as chondrule formation and metamorphic reheating.

3.1.2. Rare earth elements

Analytical data on REEs for Y-691 are listed in Table 2, along with the data for Allende and their literature values. REE abundance patterns for the whole rock sample
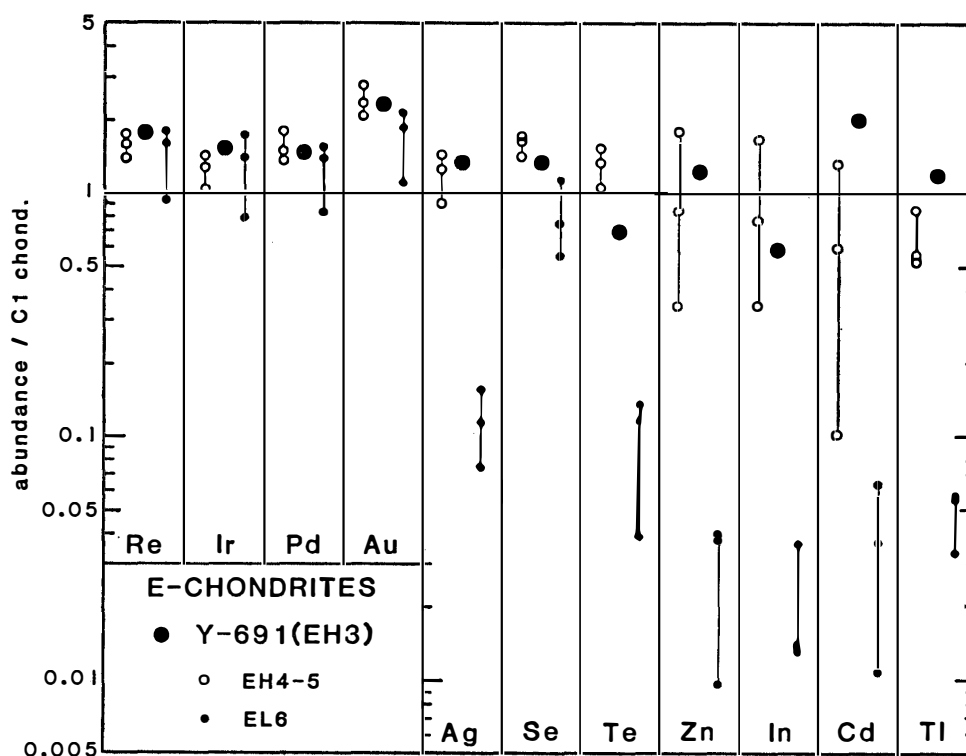
Fig. 1. C1-normalized abundances of siderophile and chalcophile elements in Y-691. Ranges of abundance variations for some EH4-5 and EL6 chondrites (HERTOGEN et al., 1983) are also shown for comparison. Siderophiles were found not to be fractionated among all petrologic classes of enstatite chondrites. The most volatile elements, Cd and Tl, were observed to be more enriched in Y-691 than in EH4-5s, which seems to be consistent with the primitiveness of Y-691.

Table 2. Rare earth elements (in ppb) in Y-691 and Allende.

Meteorite	La	Ce	Nd	Sm	Eu	Gd	Tb	Tm	Yb	Lu
Y-691	259	645	453	149	57.7	234	42.6	30.1	185	29.6
(lit.*)[1]	224	633	473	153	55.1	217			170	27.2
Allende	478	1248	909	298	109.5	396	71.8	49.4	297	43.4
(lit.)[2]	440	1250	910	290	107	430	74	49	320	58
(lit.)[3]	510	1300	970	340	100	420	80	50	310	

* literature: 1. MASUDA and TANAKA (1978), 2. WAKITA and SCHMITT (1970), 3. MARTIN and MASON (1974).

of Y-691 normalized to unfractionated-chondritic values (ANDERS and EBIHARA, 1982) are shown in Fig. 2. In addition to the pattern determined from the bulk analysis, those based on the material balance calculation for acid-soluble and -residual phases (shown as S + R, see Section 3.3) and literature data (MASUDA and TANAKA, 1978) also are shown. No apparent difference can be seen among these patterns except for La, data of which obtained in this work seem to be systematically high, suggesting that some analytical problems were involved in this element.

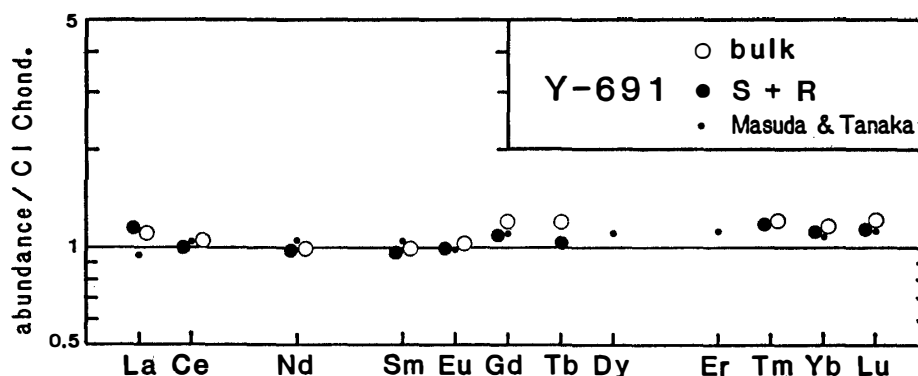All the REEs analyzed were confirmed to be scarcely fractionated from each other,

Fig. 2. Rare earth abundance patterns for the whole rock sample of Y-691, normalized to unfractionated-chondritic values (ANDERS and EBIHARA, 1982). In addition to the pattern from bulk analysis, those based on the material balance calculation for acid-soluble and -residual phases (S+R), and literature data (MASUDA and TANAKA, 1978) also are shown. No apparent difference was confirmed among these abundance patterns. It can be commonly noticed that heavy REEs are slightly more enriched than light REEs, suggesting that light REEs had been leached due to weathering on Antarctica.

being in the Cl level. Although the difference is small, it may be noticed that heavy REEs (Gd–Lu) are slightly more enriched than light REEs (La–Eu) with a discontinuity between Eu and Gd. Selective dissolution of light REEs due to weathering on Antarctica could be suggested as a possible cause.

3.2. Siderophile and chalcophile elements in acid-soluble and -insoluble phases in Y-691

Abundances and distributions of siderophile and chalcophile elements in acid-soluble and -insoluble phases are listed in Table 3. To calculate the abundances in acid-soluble phases, weight difference between bulk and acid-insoluble samples is used.

In enstatite chondrites, the great part of iron is known to be present as metal and sulfides. These minerals are easily dissolved in such acids as the ones used in this

Table 3. Abundances and distributions of siderophile and chalcophile elements in acid-soluble (S) and -residual (R) phases in Y-691.

Phases*	wt/ fraction	Re	Ir	Pb	Au	Ag	Se	Te	Zn	In	Cd	Tl
Abundances**												
S	31.7 mg	160	1190	1950	883		44.2		875	93.8	3420	
R	41.5	n.d.***	51	n.d.	45		0.44		12.8	n.d.	n.d.	
bulk 1		71	530	860	400		20		390	41	1500	
bulk 2		66.2	647	838	347	261	25.4	1.63	382	46	1450	170
Distribution****												
S	42%	<98.9	94.5	98.9	93.6	96.2	98.7		98.1	>95	>96	82.9
R	58	<1.1	5.5	1.2	6.4	3.8	1.3		1.9	<5	<4	17.1

* bulk 1: contents from material balance calculation, bulk 2: contents from whole rock analysis.
** the same units as in Table 1.
*** not determined (below detection limit).
**** in %.

work (nitric acid and aqua regia), suggesting that iron could be one of key elements for evaluating how effective the phase separation has been performed. Only 1.6% of the total iron was found to be distributed in acid-residual phases. Cobalt could be another, and possibly a better key element. Essentially no Co was detected in the acid residues. The data on Fe and Co suggest that separation of acid-insoluble fraction from acid-soluble ones was highly satisfactory.

Except for Tl, more than 90% of the bulk contents for siderophile and chalcophile elements analyzed were confirmed to be present in acid-soluble phases, possibly metal and sulfides. Essentially no Re was present in acid-residual silicates, which is in contrast with the distribution of Ir. Apparently Ir and Re are highly fractionated in acid-residual phases.

As much as 17% of Tl was observed to be distributed in acid-residues. The condensation calculation of Tl does not allow this element to be distributed into acid-residual silicates (LAUL et al., 1973; LARIMER and BARTHOLOMAY, 1979). Thallium is geochemically known to be lithophile as well as chalcophile in igneous rocks of the earth crust. The lithophile nature of Tl may explain its presence in acid-residual silicates. Indeed, the distribution fractions of Tl into acid-residues were observed to be correlated with the degree of metamorphism implied by petrologic types (M. EBIHARA, unpublished data), suggesting that Tl has migrated into silicates during a metamorphic event(s) on the parent body. However, considering that the Y-691 meteorite is thought to be the *least* metamorphosed enstatite chondrite ever collected, the transference of Tl into silicates caused by the later metamorphism sounds unlikely. Thallium is thus suggested to have been either lithophile rather than chalcophile during the condensation of nebula or much more movable than supposed in the weak metamorphic activities at the later stage.

3.3. REE abundances and distributions in acid-soluble and -residual phases of Y-691

Abundances and distributions of REEs are summarized in Table 4. Most of total REEs were found to be present in acid-soluble phases, possibly in sulfides. The fractions distributed into acid-soluble phases, however, are smaller for REEs than those for siderophile and chalcophile elements. About 15% of light REEs and 30% of heavy REEs were confirmed to be allocated to acid-residual phases.

Table 4. Rare earth abundances and distributions in acid-soluble (S) and -residual (R) phases of Y-691.

Phases*	La	Ce	Nd	Sm	Eu	Gd	Tb	Tm	Yb	Lu
Abundances**										
S	527	1200	858	276	108	378	64.4	46.1	309	43.7
R	65.3	165	123	39.7	14.8	83.2	15.0	15.8	70.6	15.2
bulk 1	269	620	448	144	56.0	214	36.9	29.4	176	28.0
bulk 2	259	645	453	149	57.7	234	42.6	30.1	185	29.6
Distribution***										
S	86.0	84.7	84.2	84.2	84.7	77.7	76.6	69.1	76.9	68.7
R	14.0	15.3	15.8	15.8	15.3	22.3	23.4	30.9	23.1	31.3

  * see footnotes in Table 3.
 ** in ppb.
*** in %.

Figure 3 shows the C1-normalized abundance pattern of REEs in acid-soluble and -insoluble phases. With consideration of material balance, REE abundances for the bulk sample were calculated and its REE pattern is shown in Fig. 3 as well as Fig. 2.
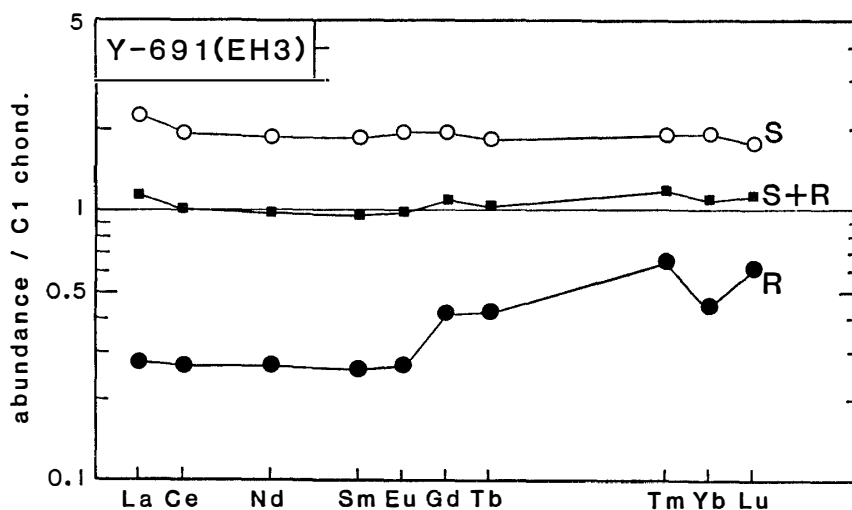

Fig. 3. C1-*normalized abundance patterns of REEs for acid-soluble* (S), *-residual* (R) *phases and calculated bulk sample* (S+R). *In acid-residual phases, REEs are present at levels of 0.3×C1's for La to 0.7×C1's for Lu, with Yb being depleted compared with the neighboring elements, Tm and Lu. Considering that heavy REEs except Yb are more refractory than light REEs, it is suggested that the acid residues of Y-691 trapped and/or preserved refractory REEs.*

For acid residues, the abundance pattern is almost flat in the light REE span, whereas the REE pattern gradually increases from Gd to Lu. As easily noticed in Fig. 3, acid residues have a small but definite negative Yb anomaly. Neither Eu nor Ce anomalies can be observed. A mirror image of these features can faintly be seen in the REE abundance pattern for an acid-soluble fraction. Based on the thermodynamic calculation, BOYNTON (1975) demonstrated that the heavy REEs are generally more refractory than the light ones and that, among the heavy REEs, Yb is exceptionally volatile compared with the neighboring REEs, Tm and Lu. Combining these theoretical predictions with the observation stated above, we come to the interesting conclusion that the acid residue trapped more refractory REEs.

One possible scenario to explain the distribution of REEs in acid-residual silicates is that, during a condensation sequence of enstatite chondrites, REEs, especially refractory REEs, were condensed in pyroxene (possibly Ca-Mg-pyroxene, diopside) and/ or some glasses at the earlier stage, whereas the remaining volatile REEs must have condensed into sulfides at the later stage. It is neither clear whether this condensation sequence was continuous nor whether the condensation conditions have changed during a sequence. Considering that the REE abundances in the bulk meteorite sample of Y-691 are almost in C1 level and that oldhamite and diopside can coexist during a condensation sequence between 1000 and 1160K at P_t (total pressure) $= 10^{-4}$ atm and $C/O = 0.92$ (LARIMER and BARTHOLOMAY, 1979), the REEs must have been distributed in acid soluble and insoluble minerals in a closed system.

The second possibility is that all the REEs were condensed in silicates (or their precursor materials), but not in sulfides at the earliest stage of REE condensation. Such silicates were then heated to the extent that silicates were not completely evaporated. Refractory REEs must have been apt to remain in solids, whereas volatile REEs were evaporated to some degree and were recondensed into sulfides as the temperature fell. The second condensation of REEs must have happened under a slightly reduced condition.

It seems to be premature to define which possibility is more plausible at present. However, any hypothesis for the formation of enstatite chondrites must explain the fact that some REEs, especially refractory REEs, are distributed in acid-residual silicates, possibly in pyroxene (diopside?), of the most primitive enstatite chondrite.

3.4. Host phase(s) of REEs in Y-691

Host phase(s) of REEs in enstatite chondrites remains still somewhat enigmatic in spite of a large effort taken so far. Most people who are concerned with this problem seem to be in favor of oldhamite as a major host phase of REEs. As discussed in the previous section, some acid-insoluble silicate (possibly pyroxene) must be one of host phase(s) of REEs, especially heavy REEs, in Y-691, although this mineral is not a major host phase. To search for a major host phase of REEs in Y-691, the stepwise dissolution experiment was carried out.

The results of stepwise dissolution are summarized in Table 5. Dissolution patterns of Sm (on behalf of the light and middle REEs) and some selected elements, Ca, Fe, Mg and Mn, which could be sulfide-forming elements in enstatite chondrites, are illustrated in Fig. 4 only for acetate buffer and EDTA steps, because a large amount of Sm was recovered in these steps while Sm was found to be scarcely leached in the acid (HNO_3) treatment. In this illustration, figures in parentheses indicate the total % fractions of elements recovered in these steps relative to bulk contents and the scale of a vertical axis is expanded to such a degree that the total % stated above is normalized to 100% for each element. As much as 77% of the total Sm was recovered in the first three leaching steps. The total fraction (14.4%) recovered at the 3rd step of HNO_3 and residue is in good agreement with the result for acid-residues (R, 15.8%) shown in Table 4.

Table 5. Fraction (%) of elements leached in each treatment of stepwise dissolution of Y-691.

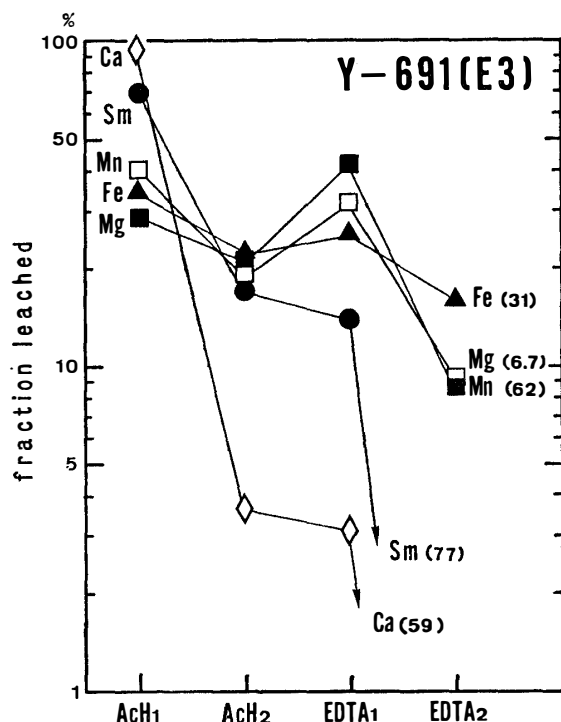| Dissolution step	Sm	Ca	Mg	Mn	Fe
Acetate 1	53	55	2.7	18	11
2	13	2.1	1.3	13	6.8
EDTA 1	11	1.9	2.1	26	8.0
2	0.04	—	0.59	5.3	5.0
HNO_3 1	8.0	0.69	2.1	8.1	46
2	0.07	3.5	1.7	1.0	10
3	2.4	8.2	16	6.3	10
Residue*	12	29	74	23	2.7

* undissolved phases.

Fig. 4. Dissolution pattern of the sulfide-forming elements in stepwise dissolution. AcH and EDTA mean acetic acid-ammonium acetate buffer and ammoniacal ethylenediaminetetraacetic acid, respectively. Figures in parentheses indicate the total % of elements recovered in the first 4 dissolution steps relative to bulk contents. The scale for each element is enlarged to the extent that the total % is normalized to 100%. Dissolution pattern of Sm is similar to that of Ca, but not identical, suggesting that most REEs recovered in these steps are present to be closely correlated with CaS in the form of sulfides.

If any two elements are hosted in a common mineral and this mineral is stepwisely dissolved with some solution(s), the dissolution patterns of these two elements should be parallel with each other in such an illustration as Fig. 4. A total of 59% of the bulk Ca was collected at the first 3 leaching steps, most of which was recovered at the first treatment with acetate buffer. One of the major Ca-containing minerals in enstatite chondrites is oldhamite, which is known to be easily dissolved even in water. The dissolution pattern of Sm is similar to that of Ca, but not identical. Samarium was found to be leached to a larger extent at the second and third steps compared with Ca. The distribution pattern of Sm is also somewhat similar to those of Fe and Mn, though essentially no Sm (and Ca) were recovered at the 2nd step of EDTA in spite of apparent leachings of Fe and Mn at that step. These results suggest that most REEs recovered in acetate buffer and EDTA treatments are present to be closely correlated with CaS, possibly in the form of REE-sulfide (REE_2S_3). It is, however, not clear whether these REEs are present in CaS as solid solutions.

Acknowledgments

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