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Distribution of elements between different phases of Apollo 14 rocks and soils

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Abstract—Neutron activation analysis data are given for samples 14163 (soil), 14276 (breccia), 14303 (breccia) and 14310 (crystalline rock). Two polished thin sections of breccia 14303 were studied and electron-microprobe data are given on the constituent minerals. Orthopyroxene separate from rock 14310 was also analyzed by electron microprobe. Data are given on plagioclase, orthopyroxene, three glass fractions, and two rock fractions from sample 14163, and on plagioclase and orthopyroxene fractions from rock 14310. The different glass and rock fractions of sample 14163 have similar compositions. Published modal analyses of 14310 show that plagioclase plus pyroxene constitute about 95% of the rock. Most of the trace elements are concentrated in the remaining 5%. Iron is also concentrated in this fraction being probably related to the presence of metallic iron in the sample.

INTRODUCTION

Samples received

THE FOLLOWING SAMPLES were received: 14163,154 (<1 mm fines, 4.960 g); 14276,8 (breccia, 0.236 g); 14303,14 (breccia, 32.31 g) with two polished sections; and 14310,123 (rock, 4.04 g).

Sample 14276 was part of a consortium sample with Professor G. J. Wasserburg as consortium leader.

Data on sample 15601,75 (<1 mm fines, 1.01 g) were reported by us at the Third Lunar Science Conference (Brunfelt *et al.*, 1972). The analytical data are presented in the Appendix, but will not be discussed further in this paper.

Previously published papers on the received material

In addition to our abstract in the proceedings of the Third Lunar Science Conference (Brunfelt *et al.*, 1972), a determination of 36 elements in sample 14163,154 was reported by Brunfelt *et al.* (1971). A discussion of REE distributions in apatite and whitlockite from lunar rock 14310,123 and from a terrestrial occurrence (Ödegaarden, Norway) was published by Griffin *et al.* (1972) and will not be commented on further in this paper.

Sample preparation and analytical methods

Only bulk analyses have been carried out on sample 14276 while both the bulk breccia and a separated matrix fraction have been analyzed in the case of 14303. Two

polished sections (14303,48 and 14303,49) were studied microscopically and analyzed by electron microprobe.

Mineral separations were carried out on samples 14163 and 14310 after a bulk sample had been split off. In the case of 14163 a fine fraction (<0.12 mm grain size) was washed with ethanol and removed by sieving before the mineral separation. This fine fraction was analyzed separately. Plagioclase, orthopyroxene, light and dark rock fragments, glass spheres, irregular dark glass fragments, and twisted glass fragments were concentrated by handpicking from 14163 and analyzed.

Sample 14310 was split into a light and a heavy fraction by the use of acetylene tetrabromide. Plagioclase and orthopyroxene fractions of high purities were obtained from these fractions by magnetic separation and handpicking.

All the separated fractions and bulk samples were analyzed by neutron activation following the procedure of Brunfelt and Steinnes (1971). Some of the 14163 fractions were too small to permit determination of elements other than those that could be assayed without using radiochemical separation steps.

Orthopyroxene from sample 14310 was also analyzed by electron microprobe, using an ARL-EMX probe at the Central Institute for Industrial Research, Oslo. Various natural minerals were used as standards for these analyses (see Griffin *et al.* (1972) for REE determinations in whitlockite and apatite).

PRESENTATION AND DISCUSSION OF DATA

Regolith 14163,154 (<1 mm fines)

The analytical data obtained by neutron activation analysis on the regolith and separated fractions are given in Table 1. There is no significant difference between the bulk sample and the less than 0.12 mm size fraction (columns 1 and 2). Our data compare well with emission spectrographic and spark source mass spectrometric determinations by Taylor *et al.* (1971) for 19 elements. Significant differences exist only for Ga and W (4.5 and 0.66 ppm, respectively, according to Taylor *et al.*). Our data for W agree well with that of Wänke *et al.* (1972), who reported 1.95 ppm W. Baedecker *et al.* (1972), Helmke and Haskin (1972), and Wänke *et al.* (1972) found 8.7, 7.5, and 8.3 ppm Ga, respectively, in this sample. Baedecker *et al.* (1972) obtained values for Zn and Cd in good agreement with those reported by us. Of the number of elements determined both by Helmke and Haskin (1972) and ourselves, we find significant disagreement only for Ce (157 versus 203 ppm). Taylor *et al.* (1971) and Wakita *et al.* (1972) both obtained 200 ppm Ce. Hubbard and Gast (1972) obtained 176 ppm Ce. The other data given by Hubbard and Gast (1972) agrees well with our own except for Ba, which they reported as 926 ppm versus 748 ppm in our analysis. Taylor *et al.* (1971) and Wakita *et al.* (1972) found 710 and 730 ppm Ba, respectively. Other determinations of trace elements in 14163 are by Jackson *et al.* (1972); Keith *et al.* (1972); Laul *et al.* (1972); Morgan *et al.* (1972). A study of the mean values from these analyses indicates that our determination of Th (11.3 ppm) is somewhat low and that the content is more likely to be 13 and 14 ppm.

The bulk composition of sample 14163 is compared with the average composition of the bulk regolith from the Apollo 11 and 12 landing sites in Fig. 1. The higher

Table 1. Compositions of lunar regolith 14163,154 by neutron activation analysis.

Element	14163 Regolith	14163 Fine fraction	14163/1 Plagio-clase	14163/2 Glass (spheres)	14163/3 Glass (dark)	14163/4–5 Glass (twisted)	14163/9 Ortho-pyroxene	14163/12 Light rock fragments	14163/13 Dark rock fragments
Na (%)	0.54	0.55	0.88	0.39	0.46	0.45	0.11	0.60	0.56
Mg (%)	5.5	6.7	1.7	5.5	6.7	5.4	18.8	7.2	7.4
Al (%)	9.1	9.6	16.5	8.9	9.0	10.0	2.6	10.2	8.9
Cl (ppm)	47								
K (%)	0.41	0.33							
Ca (%)	7.2	7.3	11.5	8.3	7.6	7.6	1.7	8.6	7.2
Sc (ppm)	21	20.5	2.5	23.1	22.8	20.3	25.7	16.5	19.7
Ti (%)	0.90	1.11	0.13	0.82	1.05	1.08	0.35	0.71	0.84
V (ppm)	48	45	< 10	29	34	39	50	18	38
Cr (%)	0.137	0.143	0.019	0.194	0.154	0.156	0.256	0.116	0.147
Mn (%)	0.103	0.102	0.132	0.114	0.102	0.093	0.132	0.085	0.098
Fe (%)	8.1	8.16	0.95	9.16	8.65	7.75	12.0	6.62	7.92
Co (ppm)	34	34.7	4.35	77.2	34.6	25.5	36.6	19.8	24.6
Ni (ppm)			34	990	330	190	140	220	230
Cu (ppm)	10.4	13.4							
Zn (ppm)	33	40							
Ga (ppm)	7.7	8.2							
As (ppm)	0.02	0.10							
Se (ppm)	0.29								
Rb (ppm)	16	13	8.5	8.4	10	16	28	18	15
Sr (ppm)	185	170							
Pd (ppm)	0.11	~0.1							
Sb (ppm)	0.003	0.01	1.3	0.11		0.01			
Cs (ppm)	0.68	0.56							
Ba (ppm)	748	740	447	515	689	583	163	647	753
La (ppm)	67	61	14	49	68	54	23	64	81
Ce (ppm)	203		40	162	214	183		189	214
Pr (ppm)		26							
Sm (ppm)	30	27.3	4.4	20.1	32.1	27.5	8.0	30.0	37.3
Eu (ppm)	2.5	2.8	4.5	2.3	2.4	2.4	1.7	2.8	2.8
Tb (ppm)	6.3	5.8	0.9	4.2	6.4	5.3	1.2	5.8	7.1
Dy (ppm)		33.5	5.8	24.5	35.3	30.2	8.3	33.4	41.5
Ho (ppm)		8.2							
Er (ppm)		17.3							
Yb (ppm)	21	15	3.7	16	19	18	8.6	19	25
Lu (ppm)	3.1								
Hf (ppm)	19	20.6	13.4	19.0	25.4	20.7		23.3	
Ta (ppm)	2.7	2.9	0.4	2.3	3.4	2.6	0.6	2.7	3.6
W (ppm)	1.8	1.40							
Au (ppb)		2.3							
Th (ppm)	11.3	10.6	2.2	8.1	11.1	10.5	2.7	10.9	13.6
U (ppm)	3.4	3.4	0.69	2.8	3.7	3.2	1.2	4.0	4.4

concentration of most trace elements in the Apollo 14 soil is readily apparent from this figure.

The different types of glass and rock fragments separated from the bulk sample all have rather similar compositions (Fig. 2). The glass spheres tend to have the lowest concentrations of most elements, notable exceptions are Fe, Co, and Ni.

Plagioclase and orthopyroxene were the major crystalline mineral constituents in the sample. They differ significantly in composition from the other separated fragments. The chondrite normalized REE distribution patterns illustrate this point rather well, Fig. 3.

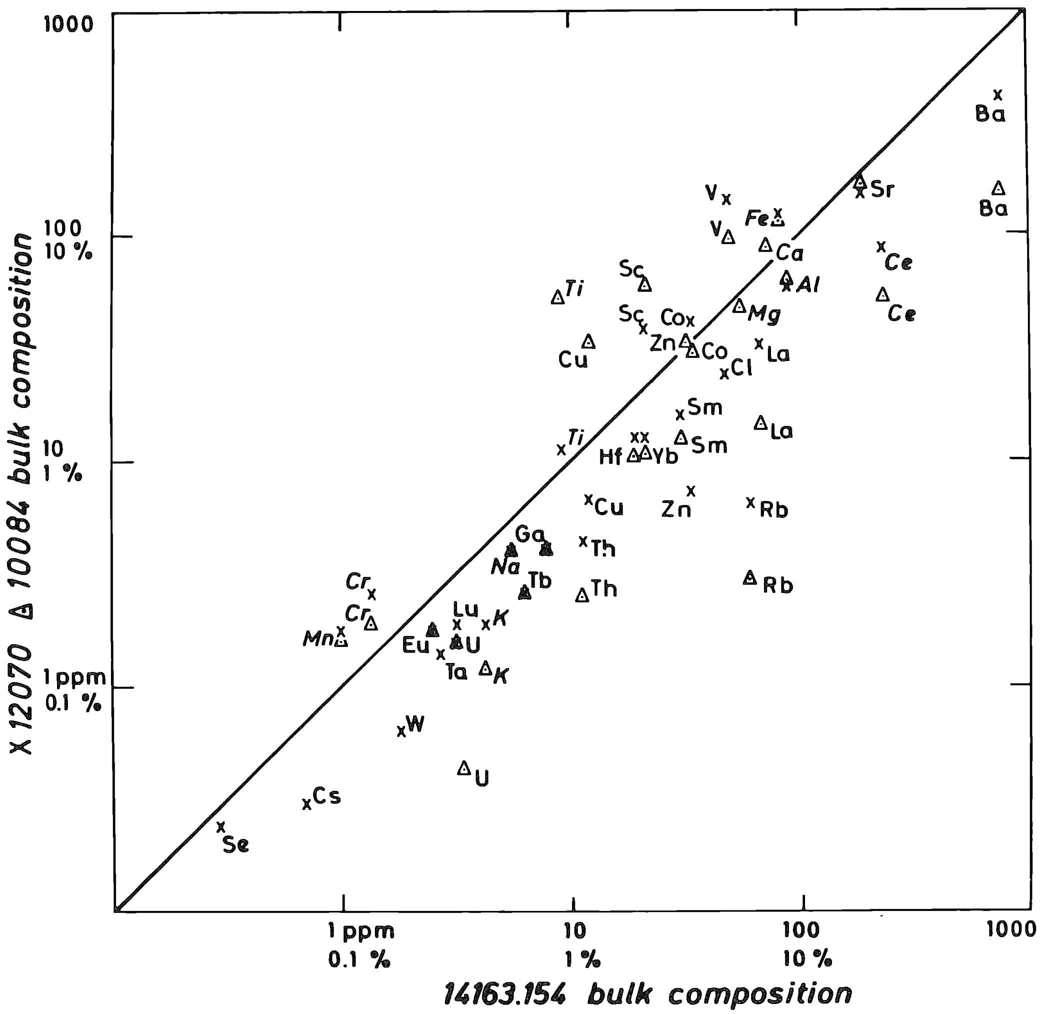

Fig. 1. Bulk composition of regolith 14163 compared with regoliths 10084 and 12070. Na, Mg, Al, K, Ca, Ti, Cr, Mn, Fe in percent; rest in ppm. Solid line indicates 1 : 1 distribution ratio.

Breccias 14276 and 14303

The neutron activation data on the breccias are listed in Table 2. The elemental compositions of the 14303 ground mass and bulk sample are very similar. The groundmass is slightly enriched in Fe, Co, and Ni. The compositions of the breccias are compared with the regolith in Fig. 4. The general similarity of regolith and breccias is apparent from this figure.

Two polished thin sections of breccia 14303 (14303,48 and 14303,49) have been studied. Electron-microprobe data on constituent minerals are shown in Table 3. The large variation in FeO/MgO ratios between individual grains indicates the complexity of mineral studies of this material.

We are not aware of any other chemical data available on samples 14276 and 14303. No information appears to be published in *Lunar Science—III* (editor C. Watkins) Lunar Science Institute Contr. 88.

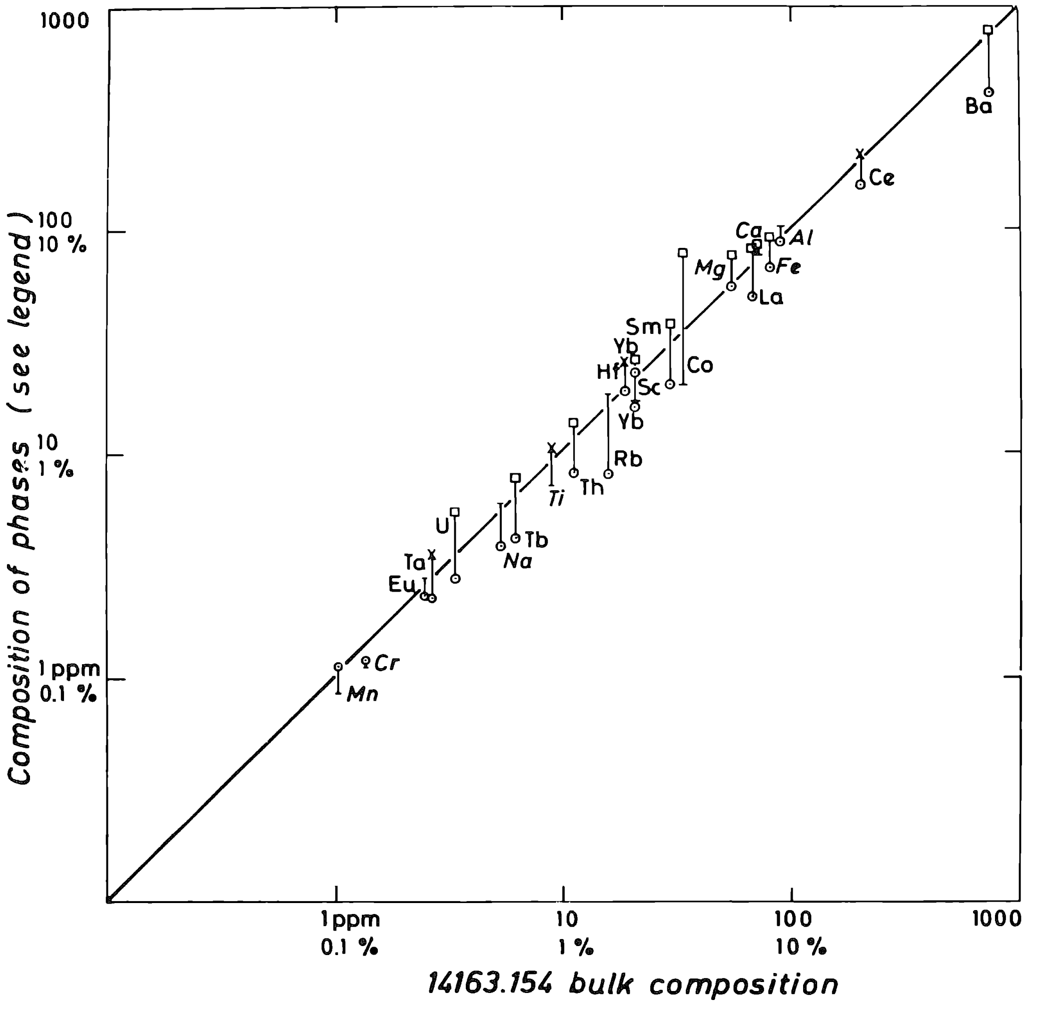

Fig. 2. Distribution of elements between different fragments of 14163,154 and bulk sample. Phases showing maximum and minimum values are indicated, remaining phases plot in the indicated range (□ dark rock fragments, - light rock fragments, x dark irregular glass fragments, v twisted glass fragments, ⊙ glass spheres).

Rock 14310,123

This is the one of the two homogeneous crystalline rocks collected on the Apollo 14 mission. Chemical data on bulk rock and separated plagioclase and orthopyroxene fractions are shown in Table 4. Electron-microprobe analyses of polished mounts of orthopyroxene separates are given in Table 5. No thin sections were made available to us for electron-microprobe analyses or microscopic studies. The sizes of the orthopyroxene grains range between 0.1 and 0.2 mm and the mineral appears to be only slightly heterogeneous. The orthopyroxene identification was verified by optical and x-ray diffraction techniques. Clinopyroxene appears to be present in only subordinate amounts in the chip received by us. This is at variance with the findings of Gancarz *et al.* (1971), who reported no orthopyroxene in the sample. Hollister *et al.* (1972) reported 23.8% augite, 11.4% hypersthene, and 1.6% pigeonite, while Melson *et al.* (1972) reported 18% low-calcium and 36% high-calcium pyroxene in 14310.

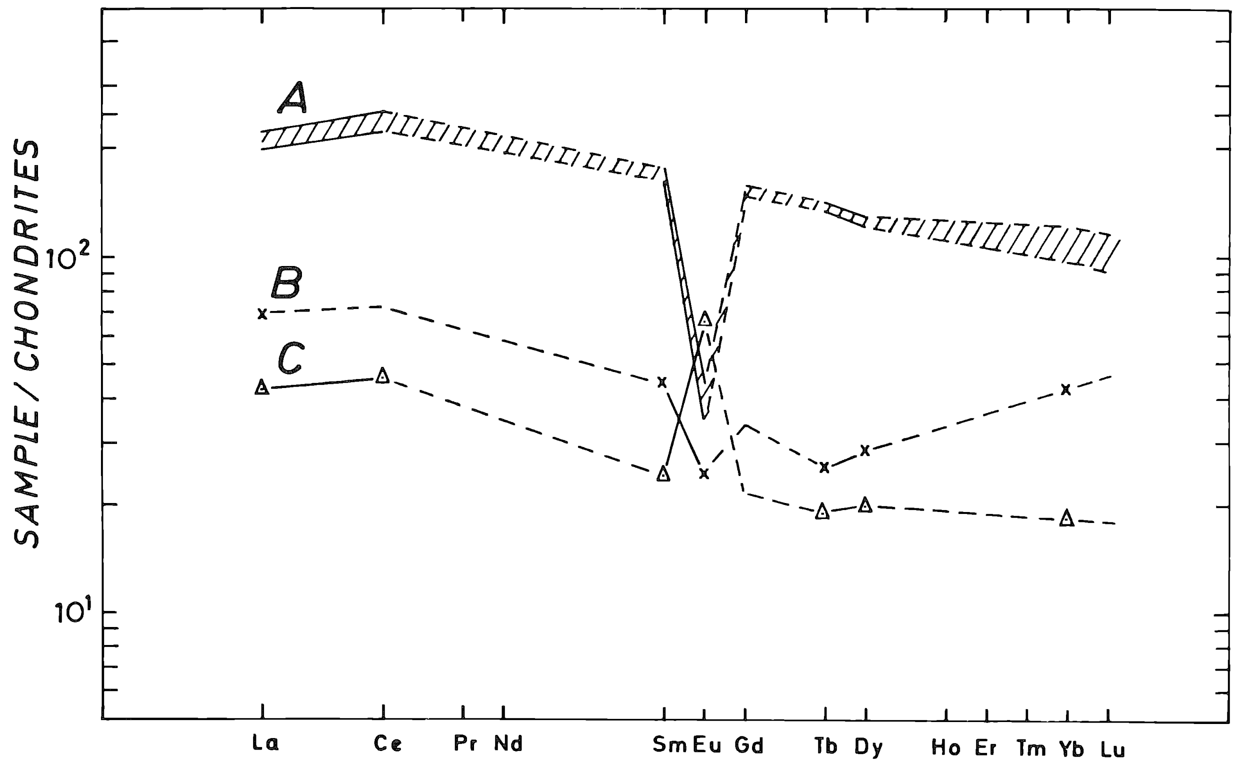

Fig. 3. Chondrite normalized REE-distribution patterns in 14163,154 pyroxene (B), plagioclase (C), bulk fines and other separated fractions (A, shaded area).

Neither of these are in agreement with our impression. Battey *et al.* (1972), on the basis of Mössbauer studies, claimed that clinopyroxene, with a composition close to $En_{46}Fs_{28}Wo_{26}$, is the major iron mineral in rock 14310, and Jagodzinski and Korokawa (1972) wrote that "orthopyroxenes are very rare in samples 14310,106." The petrology of rock 14310 and its pyroxenes is also discussed in some detail by Bence and Papike (1972); Ford *et al.* (1972); Kushiro (1972); Ridley *et al.* (1972); Ringwood *et al.* (1972); Takeda and Ridley (1972); and Walter (1972). The presence of orthopyroxene, augitic clinopyroxene, and pigeonite is reported by all these authors but without any statements being made about their relative amounts.

These very different findings regarding the nature and relative amounts of pyroxenes in rock 14310 could indicate heterogeneity in the rock. However, studies of the other published whole rock analyses by Baedecker (1972), Helmke and Haskin (1972), Hubbard and Gast (1972), Keith *et al.* (1972), Laul *et al.* (1972), and Morgan *et al.* (1972) give very little support for the idea of a heterogeneous sample. We report significantly higher concentrations of some of the heavy REE than, for instance, Helmke and Haskin, and Hubbard and Gast. Morgan *et al.* report much higher values for Au (4.31 versus 0.3 ppb) and In (130 versus 30 ppb) than we do, but again analytical error or sample contamination are the most likely explanations.

The concensus of opinion among petrologists and geochemists, who have studied this rock, is that it represents the crystallization product of a surface impact melt, i.e., Ringwood *et al.* (1972); Walker *et al.* (1972). Its similar composition to the regolith 14163 is illustrated in Fig. 5. Modal analyses by LSPET (1971) and Gancarz *et al.*

Table 2. Elemental composition of samples 14303,14 and 14276,8 (4) by neutron activation analysis.

Element	14303 Bulk rock	14303 Groundmass	14276 Split B
Na (%)	0.62	0.60	0.52
Mg (%)	6.5	6.6	5.2
Al (%)	8.77	8.53	11.7
Cl (ppm)	18		23
K (%)	1.33		0.36
Ca (%)	7.1	6.3	9.5
Sc (ppm)	23.2	23.9	18.2
Ti (%)	1.09	1.01	0.66
V (ppm)	45	47	25
Cr (%)	0.137	0.142	0.1450
Mn (%)	0.1085	0.1075	0.08
Fe (%)	8.07	8.54	6.04
Co (ppm)	30.5	34.2	13
Ni (ppm)	260	320	
Cu (ppm)	75		38
Zn (ppm)	0.8–3.7*		1.7
Ga (ppm)	5.3		3.8
As (ppm)	0.07		0.16
Rb (ppm)	20	27	14
Sr (ppm)	160		170
Cd (ppm)			< 0.02
In (ppb)			56
Sb (ppm)	< 0.03		0.024
Cs (ppm)	0.86	1.1	0.57
Ba (ppm)	890	830	540
La (ppm)	72	71	43
Ce (ppm)	210	200	
Sm (ppm)	34.6	33.3	20.0
Eu (ppm)	2.5	2.3	1.9
Tb (ppm)	7.0	7.0	4.8
Dy (ppm)	50.8	50.9	23
Ho (ppm)	9.5		7.3
Er (ppm)	30		
Yb (ppm)	28	29	11.7
Lu (ppm)	4.4	4.5	
Hf (ppm)	25.6	25.4	16
Ta (ppm)	3.2	3.4	2.1
W (ppm)	0.85		2.5
Au (ppb)			0.3
Th (ppm)	12.6	12.9	8.0
U (ppm)	3.6	3.4	2.5

* Probably inhomogeneities.

(1971) indicate 61% plagioclase and 31% pyroxene, versus 50% plagioclase and 40% pyroxene, respectively, in this rock. In Fig. 6 we have plotted the sum of the element concentrations in plagioclase and pyroxene (calculated as $\frac{2}{3}$ plagioclase and $\frac{1}{3}$ pyroxene) versus bulk rock (K in pyroxene and Ti in plagioclase taken as zero; Sr and Ca in pyroxene as 10 ppm and 1.7%, respectively). If we assume plagioclase and pyroxene to make up 95% of the rock, the average composition of the remaining 5% is (in percent): Mg (30.2), Fe (49.2), Ti (11.6), K (0.48), Mn (0.6), Cr (0.20); in ppm: Sc (132), Co (166), Rb (216), Ba (6640), La (854), Sm (360), Tb (159), Dy (134), Yb (172), Hf (266), Ta (41), Th (148), U (48.6). The high Fe content in the remaining

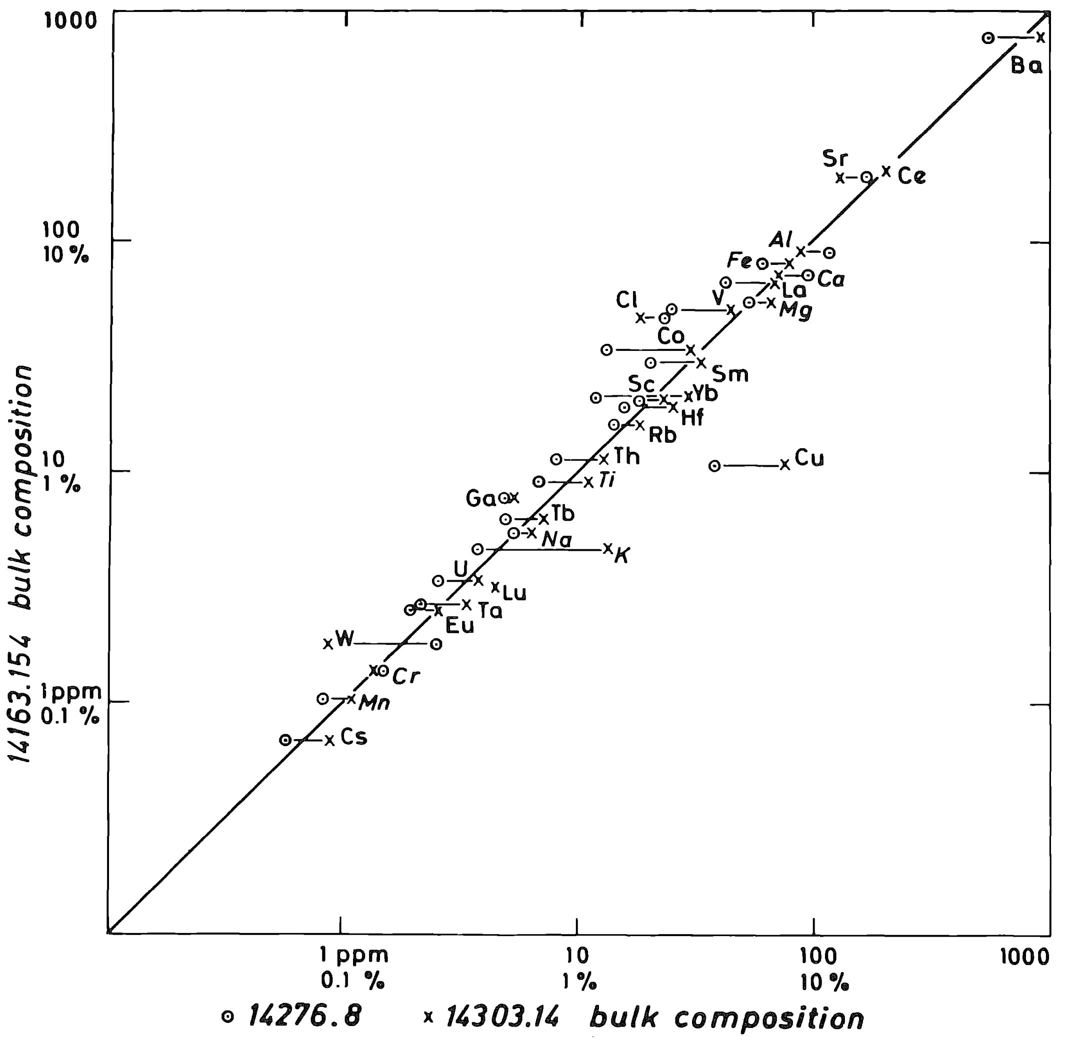

Fig. 4. Comparison of breccias 14276 and 14303 with bulk regolith 14163.

phases could be related to the presence of metallic iron (i.e., El Goresy *et al.*, 1972), while the high Mg content may partly be related to analytical error. Compston *et al.* (1972), Hubbard and Gast (1972), and Kushiro (1972) all report between 7.6 and 7.9% MgO while we have found 8.8% MgO. It is interesting that the bulk of the potassium is not to be found in the plagioclase concentrate which represents the lightest mineral fraction.

It must be emphasized that what we have termed "remaining phases" could include outer rims of the plagioclase and pyroxene grains which may have been selectively removed during the grinding and mineral separation. Some of the trace elements could be strongly concentrated in these rims.

The chondrite normalized REE patterns of the plagioclase and orthopyroxene concentrate are compared with that of the bulk rock in Fig. 7. The importance of minor phases as hosts for a number of trace elements is strikingly demonstrated by the lunar rocks.

Table 3. Electron-microprobe data on minerals in breccia 14303 (nos. 1 to 12 from 14303,48; nos. 13 to 20 from 14303,49).

	Olivines						
	1	8	14	16	17	18	20
SiO_2	37.78	38.72	39.48	40.65	37.84	40.34	37.59
TiO_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Cr_2O_3	0.13	0.12	0.15	0.16	0.13	0.07	0.11
Al_2O_3	0.38	0.54	0.21	0.39	0.41	0.39	0.46
FeO	30.21	23.44	18.36	13.55	28.03	15.17	29.30
MnO	0.32	0.23	0.18	0.13	0.30	0.14	0.28
MgO	28.97	37.00	41.67	45.80	32.96	44.01	32.38
CaO	0.51	0.36	0.0	0.06	0.39	0.15	0.25
Na_2O	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	98.30	100.41	100.05	100.74	100.06	100.27	100.37

	Pigeonites					Spinel 14303,49
	3	4	12	13	19	
SiO_2	52.72	53.15	54.04	48.58	50.29	0.0
TiO_2	0.43	0.38	0.31	0.39	0.31	0.0
Ca_2O_3	0.80	0.82	0.84	0.80	0.30	5.61
Al_2O_3	2.00	1.91	1.57	1.17	0.82	62.91
FeO	16.56	15.55	16.78	17.85	30.72	13.41
MnO	0.34	0.33	0.34	0.27	0.55	0.0
MgO	20.57	21.60	23.34	24.74	10.75	18.17
CaO	5.11	5.15	3.39	5.15	5.98	0.0
Na_2O	0.0	0.0	0.0	0.0	0.04	—
	98.53	98.89	100.61	98.95	99.76	100.10

	Orthopyroxenes				Clinopyroxenes		
	7	9	10	11	2	6	15
SiO_2	54.30	55.81	56.77	54.50	52.31	51.00	52.73
TiO_2	0.33	0.35	0.17	0.65	0.58	1.50	0.91
Cr_2O_3	0.34	0.56	0.29	0.33	0.01	0.65	0.18
Al_2O_3	1.18	0.98	0.90	1.16	1.56	1.79	1.42
FeO	17.91	12.02	11.30	18.63	9.77	8.47	15.64
MnO	0.30	0.21	0.22	0.30	0.22	0.20	0.30
MgO	23.58	29.31	30.38	22.16	14.50	16.10	12.65
CaO	1.79	1.74	0.68	2.25	21.12	19.80	16.34
Na_2O	0.0	0.0	0.0	0.0	0.08	0.05	0.11
	99.73	100.98	100.71	99.98	100.15	99.56	100.28

Analyses by Dr. W. L. Griffin.

Comparison of plagioclase and orthopyroxene from regolith 14163 and rock 14310

A rigorous comparison between these mineral separates is hampered by the larger amounts of impurities in the minerals separated from sample 14163. The 14310 orthopyroxene has the highest Al, Ca, Na contents and the highest Fe/Mg ratio. The compositions of the plagioclase are virtually identical, 14163 plagioclase being slightly more sodic. The chondrite normalized REE patterns of the minerals are compared in Fig. 8. The 14163 orthopyroxene is significantly enriched in the light REE compared with 14310 orthopyroxene while the contents of heavy REE are approximately the same in both minerals. The plagioclase have similar REE distribution patterns, the 14163 plagioclase having slightly higher absolute concentrations and a markedly higher Eu content.

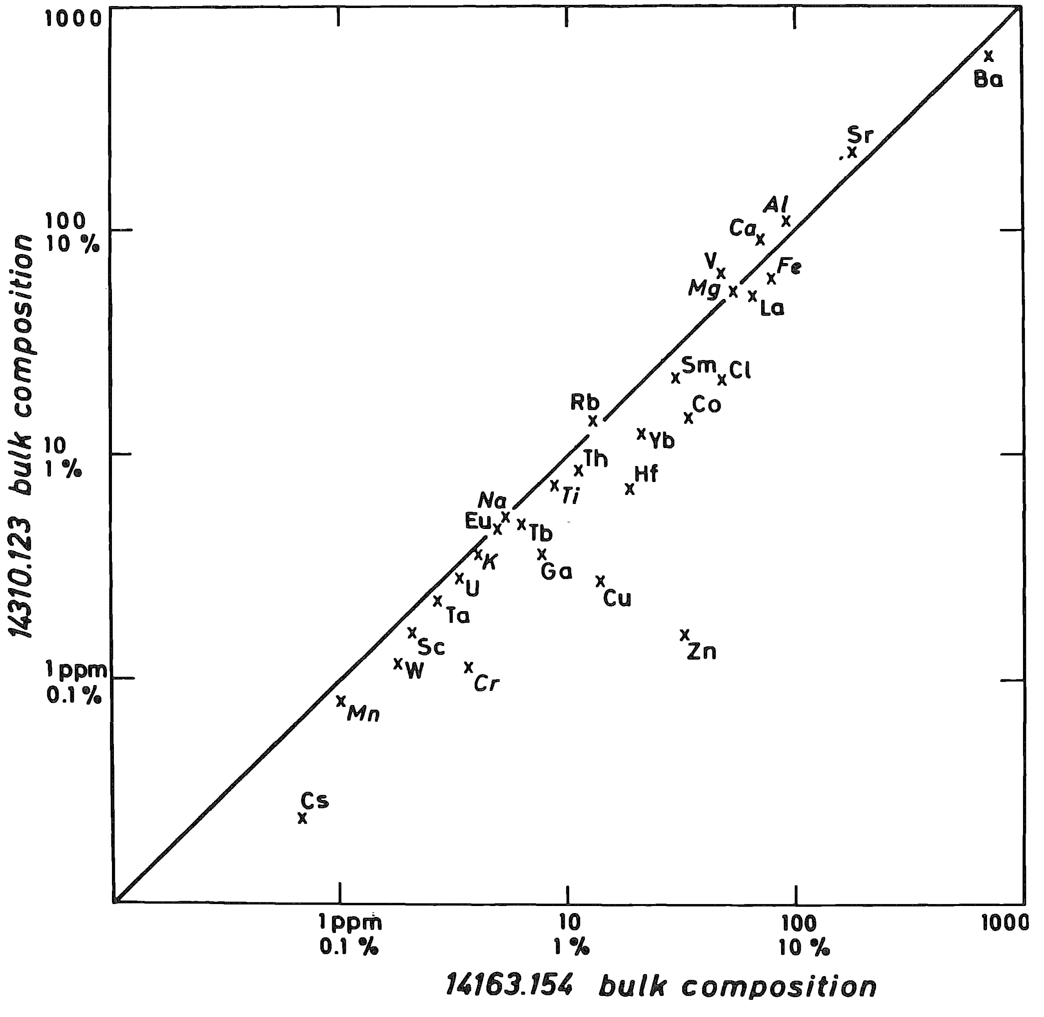

Fig. 5. Comparison of 14163,154 and 14310,123 bulk compositions.

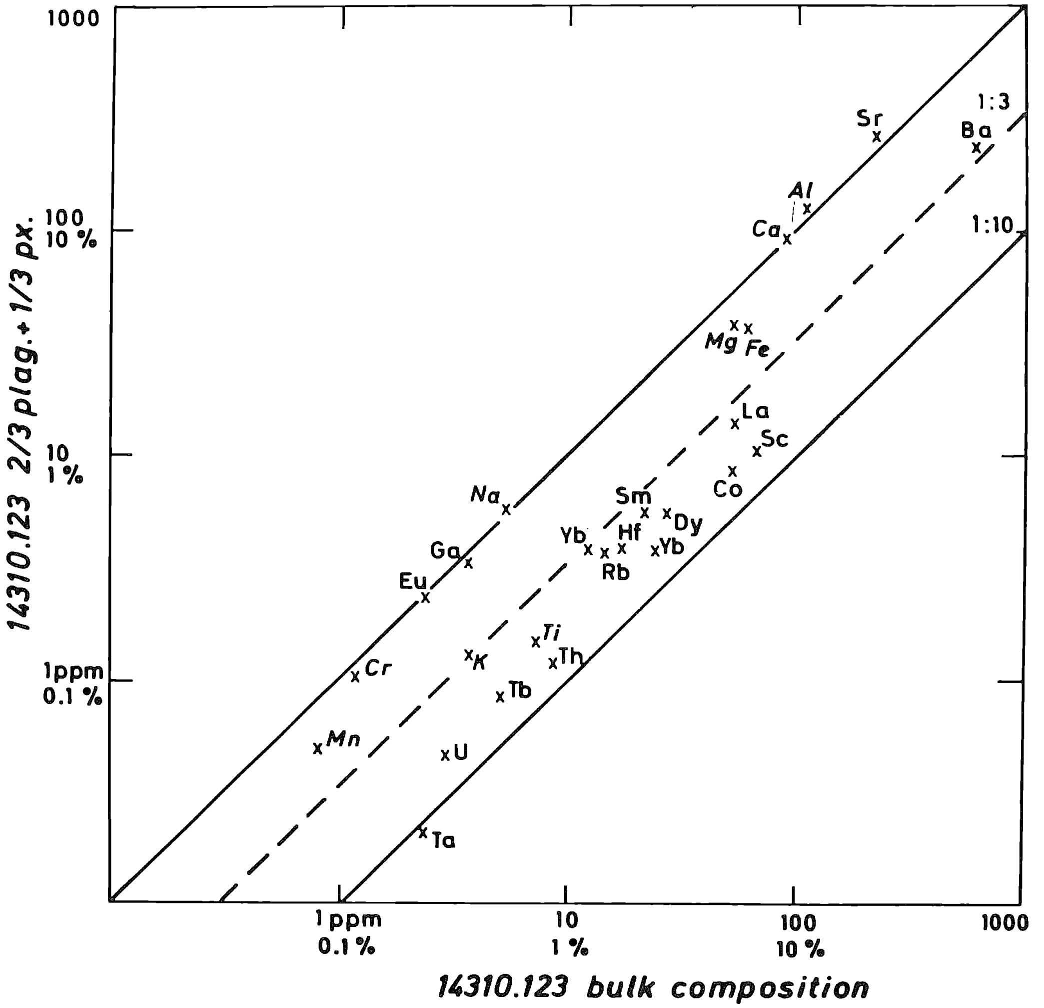

Fig. 6. Distribution of elements between plagioclase plus pyroxene and bulk rock 14310,123.

Table 4. Composition of rock 14310,123 by neutron activation analysis.

Element	14310 Bulk rock	14310/1 Plagioclase	14310/2 Pyroxene (orthorombic)
Na (%)	0.54	0.78	0.20
Mg (%)	5.3	0.5	11.3
Al (%)	11.0	16.5	3.9
Cl (ppm)	22		
K (%)	0.37	0.20	
Ca (%)	9.0	11.8	3.9
Sc (ppm)	16.7	1.0	28.2
Ti (%)	0.73	0.05	0.44
V (ppm)	56	< 50	150
Cr (%)	0.116	0.014	0.288
Mn (%)	0.0842	0.0067	0.1382
Fe (%)	6.17	0.92	9.30
Co (ppm)	15.1	1.1	17.8
Ni (ppm)		5	20
Cu (ppm)	2.8	15	
Zn (ppm)	1.6	12	
Ga (ppm)	3.7	4.6	
As (ppm)	0.03	0.6	
Rb (ppm)	15	4.1	2.8
Sr (ppm)	220	390	
Cd (ppm)	< 0.02		
In (ppb)	30		
Sb (ppm)	0.004	< 0.01	0.09
Cs (ppm)	0.4		
Ba (ppm)	595	350	90
La (ppm)	53	12	7.0
Ce (ppm)		32	17
Pr (ppm)	17		
Sm (ppm)	22.7	4.7	4.8
Eu (ppm)	2.4	3.0	1.1
Tb (ppm)	5.1	0.73	1.1
Dy (ppm)	27.3	4.3	8.1
Ho (ppm)	6.5		
Er (ppm)	16		
Yb (ppm)	12.5	2.3	7.2
Hf (ppm)	17.2	2.2	4.3
Ta (ppm)	2.3	0.22	0.23
W (ppm)	1.20	0.30	
Au (ppb)	0.3		
Th (ppm)	8.6	1.5	1.0
U (ppm)	2.9	0.50	0.40

Table 5. Electron-microprobe analysis of orthopyroxene in rock 14310,123 (average composition).

	Wt. %
SiO_2	52.28
TiO_2	0.58
Cr_2O_3	0.30
Al_2O_3	1.88
FeO	15.35
MnO	0.30
MgO	26.96
CaO	2.33
Na_2O	0.10
	100.08

Analyses by Dr. W. L. Griffin.

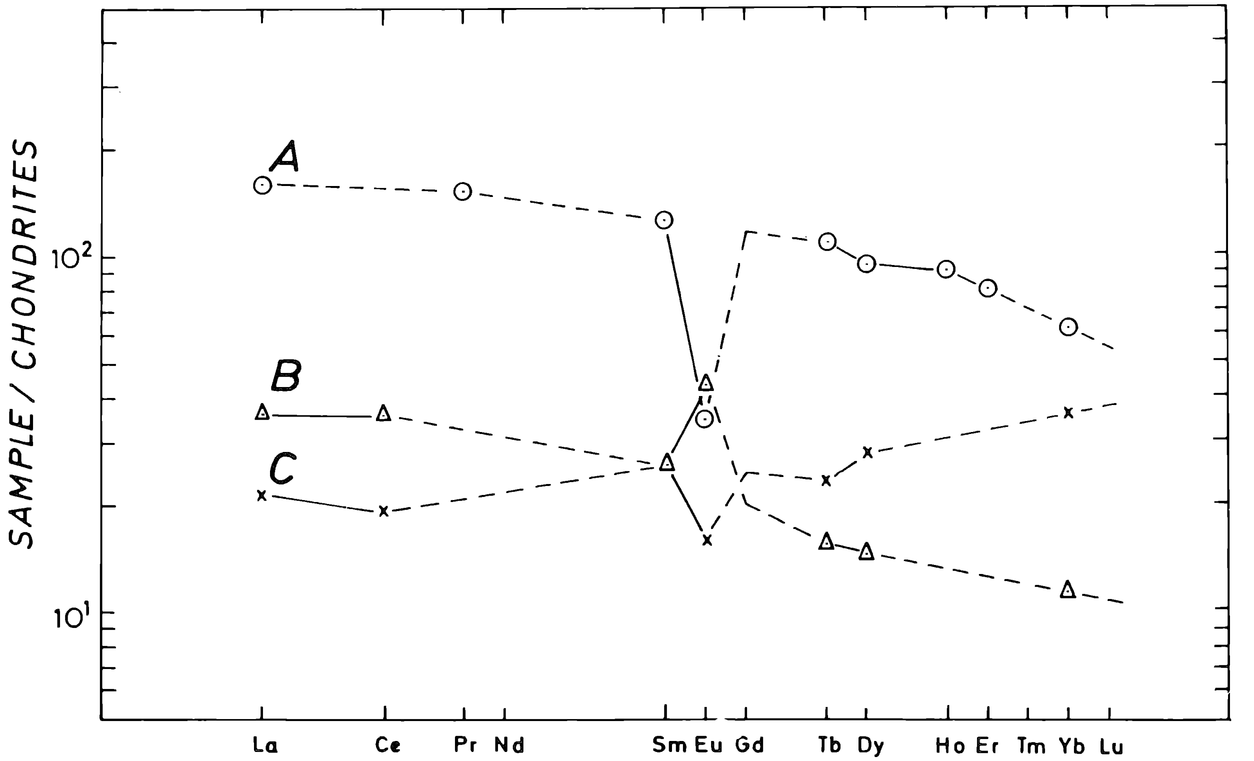

Fig. 7. REE distribution in 14310,123 bulk rock (A), plagioclase (B), and pyroxene (C).

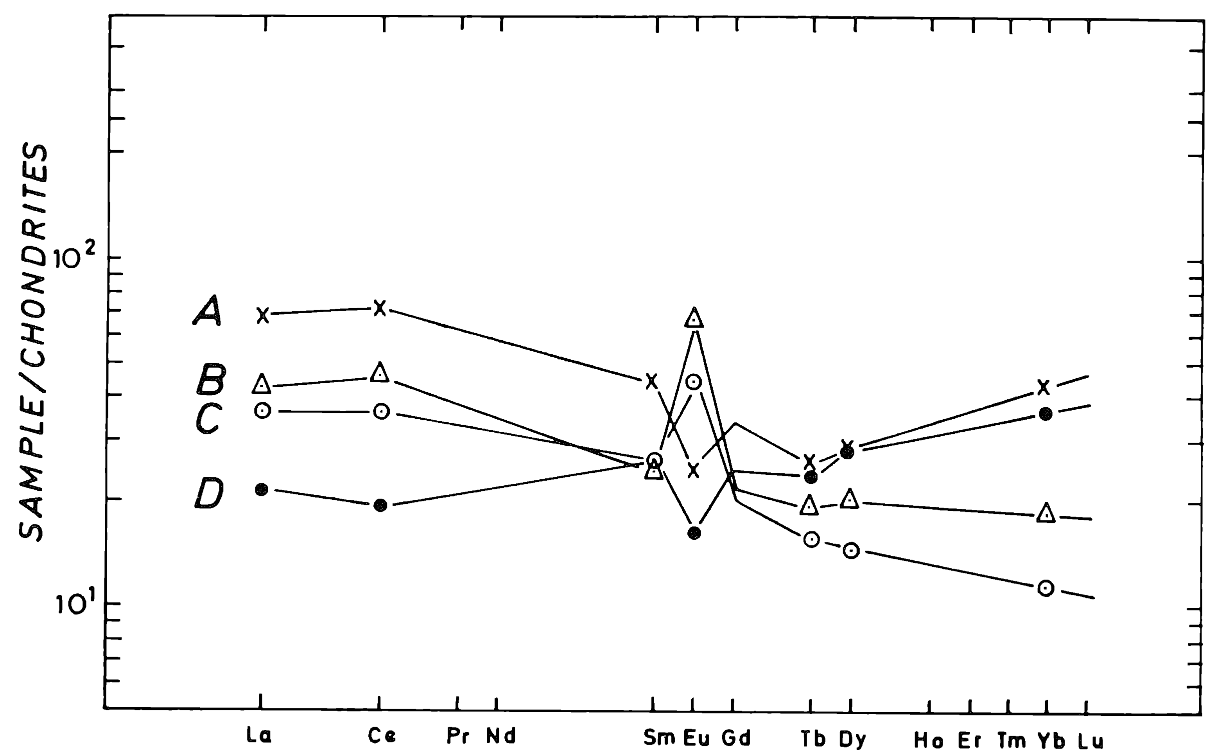

Fig. 8. Comparison of REE distribution patterns of 14310,123 and 14163,154 plagioclase and orthopyroxene. (A) Pyroxene 14163. (B) Plagioclase 14163. (C) Plagioclase 14310. (D) Pyroxene 14310.

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APPENDIX

Elemental composition of sample 15601,75 by neutron activation analysis.

Element	15601 Bulk regolith	Element	15601 Bulk regolith
Na (%)	0.24		
Mg (%)	6.9	Sb (ppm)	< 0.03
Al (%)	5.83	Cs (ppm)	0.30
Cl (ppm)	7.6	Ba (ppm)	135
K (%)	0.18	La (ppm)	11.3
Ca (%)	6.8	Ce (ppm)	29
Sc (ppm)	35.1	Pr (ppm)	3.0
Ti (%)	1.18	Sm (ppm)	6.32
V (ppm)	200	Eu (ppm)	1.0
Cr (%)	0.351	Tb (ppm)	1.3
Mn (%)	0.1962	Dy (ppm)	9.7
Fe (%)	14.6	Ho (ppm)	1.8
Co (ppm)	48.9	Er (ppm)	5.7
Ni (ppm)	170	Yb (ppm)	5.2
Cu (ppm)	6.4	Lu (ppm)	0.9
Zn (ppm)	9.8	Hf (ppm)	4.86
Ga (ppm)	3.1	Ta (ppm)	0.60
As (ppm)	< 0.005	W (ppm)	0.066
Rb (ppm)	5.3	Th (ppm)	1.52
Sr (ppm)	120	U (ppm)	0.46