Investigation of the Composition of the Luna 16 Lunar Sample

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The concentrations of aluminum, manganese, sodium, chromium, iron, cobalt, and 12 rare earth elements were determined by neutron activation analysis using slow neutrons, and oxygen and silicon were determined using a fast-neutron generator.

Mössbauer spectroscopy was used to investigate iron compounds in Luna 16 regolith samples from the upper part of the core.

A Luna 16 lunar sample was received in 1971 from the Institute of Geochemistry and Analytical Chemistry of the U.S.S.R Academy of Sciences for study of its composition. The sample consisted of small particles and was taken from the upper part of the lunar soil (from a depth of 7 cm), which has been called zone A (ref. 1). The following investigations were carried out at Central Institute of Physical Research:

1. Neutron activation analysis was used to determine the content of rare earth elements and those elements which cause the major radioactivity of the irradiated sample.
2. The oxygen and silicon concentrations were determined by activation analysis using a neutron generator.
3. Iron compounds were determined by Mössbauer spectroscopy.

Activation Analysis Using a Nuclear Reactor Apparatus

The samples were irradiated in a type VVR–SM nuclear reactor, with a slow-neutron flux of 2×10^{13} n · cm^{-2} · s^{-1}. Prolonged irradiations were done in the vertical channels of the reactor; short irradiations in channels serviced by pneumatic shuttles.

Gamma spectra were measured by a gamma spectrometer using a GeLi detector with a working volume of 45 cm^3 and a resolution of 3.5 keV, at an energy of 1332 keV (^{60}Co).

Determination of Aluminum, Manganese, Sodium, Chromium, Iron, and Cobalt

The above-listed elements account for most of the radioactivity after irradiation and

were determined by nondestructive methods. Sample sizes were 2 to 10 mg. Concentrations of Al, Mn, and Na were determined after 2-minute irradications using ^{28}Al, ^{56}Mn, and ^{24}Na and counting after cooling periods of 2 minutes, 1 hour, and 1 day, respectively. Concentrations of chromium, iron, and cobalt were determined using the isotopes ^{51}Cr, ^{59}Fe, and ^{60}Co after a 24-hour irradiation and a 2-week cooling period.

The results of nondestructive analysis are presented in table 1.

Table 1.—*The Concentration of Elements in Luna 16 Samples*

Element	Content %
Al	7.1 ± 1.0
Mn	0.17 ± 0.02
Na	0.28 ± 0.01
Cr	0.22 ± 0.03
Fe	14.3 ± 1.9
Co μg/g	32.5 ± 0.4

NOTE: The measurement error designates the maximum deviation from the average value.

Determination of Rare Earth Elements

The rare earth elements are very similar to one another in chemical properties and are of great interest from the point of view of the history of the lunar soil. Determination of the concentration of individual elements was specially important. For this purpose, 10-mg samples were irradiated for a period of 10 hours. The rock was fused with sodium peroxide in the presence of a lanthanum carrier, and the rare earth fraction was extracted from dilute hydrochloric acid in the form of oxalates.

Then, after various decay times, the gamma spectra of the resulting precipitates were measured. The content of 12 rare earth elements in the lunar rock was determined by this method (ref. 2); the analytical results are presented in table 2. Also indicated are certain data that characterize the analytical conditions.

Table 2.—*The Concentration of Rare Earth Elements in the Luna 16 Sample (ref. 1)*

Element	Isotope	Cooling Time (days)	Peak Used for Analysis (keV)	Concentration (μg/g)
La	^{140}La	0, 1, 4	486.8, 815.5, 1595.4	12.3 ± 0.4
Ce	^{141}Ce	5, 8, 20	145.4	32.4 ± 1.4
Pr	^{142}Pr	0, 1	1575.5	4.6 ± 2.5
Nd	^{147}Nd	8, 20	91.4	25.4 ± 2.5
Sm	^{153}Sm	5, 8	103.2	8.5 ± 0.3
Eu	^{152m}Eu	1, 4	841.6	2.20 ± 0.11
	^{152}Eu	20	344.2	
Gd	^{159}Gd	1	363.5	7.3 ± 1.2
Tb	^{160}Tb	8, 20	298.6	1.6 ± 0.2
Ho	^{166}Ho	0, 1	80.6	1.9 ± 0.2
Er	^{171}Er	0, 1	308.1	5.5 ± 0.6
Yb	^{175}Yb	5, 8, 20	282.6, 396.1	6.0 ± 0.3
Lu	^{177}Lu	5, 8, 20	208.4	0.86 ± 0.05

NOTE: The measurement error designates the standard error, calculated statistically.

We also investigated the uniformity of distribution of the rare earth elements in the rock by determining the europium and dysprosium concentrations. Six samples were used in this test and each was irradiated for 10 minutes. Then the rock was dissolved in a mixture of perchloric acid and hydrofluoric acid, and the rare earth elements were extracted with dilute hydrochloric acid by a previously prepared precipitate of lanthanum oxalate (ref. 3). The gamma spectra of the precipitates were taken several times, and the europium and dysprosium concentrations were calculated from several peaks of the ^{152m}Eu and ^{165}Dy isotopes.

The analytical results for each sample, as well as the average europium and dysprosium concentrations, are presented in table 3. Standard rock BCR 1 was taken as a material containing uniformly distributed rare earth elements, and the results of analyzing this sample are also given. As is evident from the table, the lunar sample contains both these elements in a rather uniform distribution because no significant differences in reproducibility of results between the lunar and standard rocks are obtained by using this method.

Activation Analysis Using a Neutron Generator Apparatus (ref. 4)

A neutron generator, with a neutron output of 10^{10} n $\times$ s^{-1}, was used as the fast neutron source. The gamma spectrometer had a NaI/Tl detector 75 cm $\times$ 75 cm in size.

Determination of Silicon and Oxygen

In these measurements, a 77.2 mg sample was used. Silicon was determined from the isotope ^{28}Al (irradiation 30 s, measurement 420 s) and oxygen, from the isotope ^{16}N (irradiation 8 s, measurement 15 s). The average value and standard error of the mean were calculated from the series of measurements: 20.6 $\pm$ 2.0 weight percent for silicon and 41.2 $\pm$ 1.2 weight percent for oxygen.

Mössbauer Measurements

The lunar dust sample that we measured weighted 60 mg. Sample thickness was 40

Table 3.—*Uniformity of the Europium and Dysprosium Distribution in Rocks*

	BCR1			Luna 16	
Sample (mg)	Eu (μg/g)	Dy (μg/g)	Sample (mg)	Eu (μg/g)	Dy (μg/g)
6.168	2.215	6.51	6.865	2.329	10.23
6.909	2.159	6.08	6.837	2.414	10.71
6.191	2.171	6.74	3.186	2.230	10.70
4.033	2.138	6.26	2.571	2.201	10.26
6.062	2.137	6.22	12.861	2.399	10.64
3.702	2.162	6.37	8.450	2.537	10.31
Mean	2.16	6.36	Mean	2.35	10.53
Root Mean Deviation	0.03	0.22	Root Mean Deviation	0.11	0.20

Table 4.—*Mössbauer Results*

Components	Concentration (atom %)	Hyperfine Magnetic Field (KO.)	Quadrupole Splitting (mm/s)	Chemical Shift (mm/s)
Metallic iron	4.5 ± 0.5	331.4 ± 1.0	− 0.021 ± 0.031	0.181 ± 0.015
Silicates (M1)	− 28.5 ± 0.9	—	2.748 ± 0.006	1.321 ± 0.002
Silicates (M2)	59.5 ± 1.8	—	1.956 ± 0.005	1.243 ± 0.006
Ilmenite	7.3 ± 0.4	—	0.71 (fixed)	1.202 ± 0.006

mg/cm^2. Twenty-five millicuries of ^{57}Co in Cr was the source of resonance gamma rays. The measurements were conducted at room temperature. A constant acceleration Mössbauer unit was used (ref. 5). The spectrum consisted of one hyperfine sextuplet and three poorly resolved quadrupole doublets. The parameters obtained are presented in table 4.

The values of the parameters indicate that the components of the spectrum correspond to metallic iron, ilmenite, and the M1 and M2 lattice sites of iron-containing silicates.

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section 2

Thermal History of the Moon