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Chemical Composition of Lunar Material

Abstract. *Chemical and emission spectrographic analyses of three Apollo 11 samples, 10017-29, 10020-30, and 10084-132, are given. Major and minor constituents were determined both by conventional rock analysis methods and by a new composite scheme utilizing a lithium fluoborate method for dissolution of the samples and atomic absorption spectroscopy and colorimetry. Trace constituents were determined by optical emission spectroscopy involving a d-c arc, air-jet controlled.*

Samples 10017-29 and 10020-30, weighing 16.2 g and 9.8 g, respectively, consisted of rock chips. Sample 10084-132 was an aliquot of the <1-mm fraction of the type D fines, weighing 7.3 g. All samples were crushed to pass a 100-mesh nylon screen. Some metallic fragments were observed in the fines, but an attempt to separate a metal phase from this sample was not successful.

The conventional rock analysis methods used are those described by Maxwell (*1*), except for the determination of total carbon, CO_2, fluorine, chlorine, and total sulfur. A nondistillation method (*2, 3*) with a spectrophotometric finish was used for the simultaneous determination of chlorine and fluorine. An induction furnace was used to liberate the total carbon and sulfur (*4*) as CO_2 and SO_2, respectively, which were then determined titrimetrically, the carbon by nonaqueous titration. The CO_2 released by acid treatment of the sample was similarly determined. The USGS basalt, BCR-1, was run simultaneously with the lunar samples to provide a means of evaluating the accuracy of the analytical data. The results are given in Table 1.

The amount of the metallic phase present in 10084-132 was too small for reliable determination. Metallic iron occurring as inclusions in other minerals would not be detected by the methods used to determine metallic iron. It is also possible that some of the titanium is present as Ti(III). Measurement of these possible constituents, and of others contributing to the total reducing capacity of the sample, was attempted by carrying out the decomposition of the sample for the determination of Fe(II) by the modified Pratt method (*1*) in the presence of an added, known amount of Fe(III). Determination of the total iron content of the same solution and subtraction of the added Fe should give an equivalent Fe(II) equal to that obtained by the initial Fe(II) determination, or reveal the presence of some other reducing agent in the solution. The assumption is made that no Fe(III) is present in the sample. The results are given in Table 2; the difference found is expressed as both equivalent Ti_2O_3 and metallic Fe, although it is not possible at this time to say which, if either, is the unknown reducing agent.

Two sample portions were used for analysis by atomic absorption spectroscopy and colorimetry. A 200-mg sample was fused with a fivefold excess of lithium metaborate, the fusion dissolved in dilute hydrofluoric acid, and sufficient boric acid added to provide a solution stable toward glass for at least 2 hours (*5*). Two dilutions were prepared from this, one containing 50 mg of sample and 300 mg of Sr in 100 ml and the other 10 mg of sample and 150 mg of Sr in 100 ml. The more concentrated

Table 1. Chemical analyses (percent by weight) of lunar material [analysts, J. A. Maxwell (conventional) and S. Abbey (atomic absorption spectroscopy, AAS)].

Constituent	BCR-1 (USGS)		10017-29		10020-30		10084-132	
	Conventional	AAS	Conventional	AAS	Conventional	AAS	Conventional	AAS
SiO_2	54.48		40.78	40.14	39.92	41.00	42.28	42.44
Al_2O_3	13.62		8.12	8.17	10.04	9.83	13.76	13.66
TiO_2	2.26		11.71	11.16*	10.72	10.28*	7.35	7.19*
Fe_2O_3	3.77		0.00		0.00		0.00	
FeO	8.65		19.82	19.38†	19.35	19.03†	16.02	15.67†
MgO	3.46		7.65	7.72	7.81	7.77	7.93	7.87
CaO	6.91		10.55	10.99	11.24	11.96	12.00	12.26
Na_2O	(3.36)‡	3.36	(0.51)‡	0.51	(0.37)‡	0.37	(0.42)‡	0.42
K_2O	(1.76)‡	1.76	(0.30)‡	0.30	(0.05)‡	0.05	(0.13)‡	0.13
H_2O^+	0.66		0.00		0.00		0.00	
H_2O^-	0.86		0.01		0.01		0.01	
P_2O_5	0.347	0.35*	0.13	0.15*	0.08	0.07*	0.11	0.10*
MnO	0.165		0.22	0.25	0.24	0.27	0.20	0.21
CO_2	0.055		0.00		0.00		0.03	
F	0.039		0.00		0.00		0.00	
Cl	0.002		0.00		0.00		0.00	
S (total)	0.033		0.22		0.15		0.13	
C	0.00		0.01		0.01		0.00	
Cr_2O_3			(0.36)‡	0.36	(0.40)‡	0.40	(0.33)‡	0.33
Subtotal	100.43		100.39	99.13	100.39	101.03	100.70	100.28
Less O≡S	0.02		0.11		0.08		0.07	
Total	100.41		100.28	99.13	100.31	101.03	100.63	100.28

* Colorimetric, on solutions prepared for atomic absorption spectroscopy (AAS). † Total Fe, expressed as FeO. ‡ AAS values.

Table 2. Reducing substances (percent by weight) in lunar material. N.S., not significant.

Constituent	10017-29	10020-30	10084-132
(1) FeO	19.82	19.43	17.05
(2) Fe_2O_3 equivalent	22.03	21.59	18.95
(3) Total Fe, expressed as Fe_2O_3*	22.01	21.42	17.80
(4) FeO equivalent	19.81	19.28	16.02
(5) Difference (1–4)	N.S.	0.15	1.03
(6) Ti_2O_3 equivalent of (5)		0.15	1.03
(7) Metallic Fe equivalent of (5)		0.04	0.27

* After subtracting Fe(III) added.

Table 3. Minor and trace element content (parts per million) of lunar material (analyst, W. H. Champ).

Constituent	BCR-1 (USGS)	10017-29	10020-30	10084-132
Cr	<20	2200	3000	2300
Zr	<200	410	210	260
Ba	700	230	67	150
Sr	220	100	130	140
V	420	49	120	67
Ni	<20	<20	<20	190
Co	<20	<20	<20	<20
Cu	10	<5	<5	<5
Y	34	160	120	120
Yb	<10	<10	<10	<10
Sc	23	52	78	51
Li*	15	23	12	15
Zn*	120	49	26	47

* By atomic absorption spectroscopy (S. Abbey, analyst). All other determinations by optical emission spectroscopy.

solution was used to determine Si and Al by atomic absorption spectroscopy, with the nitrous oxide–acetylene flame, and K, Mn, and Cr with the air-acetylene flame. All measurements were made with a modified Techtron model AA-3 instrument. The more dilute solution was used to determine total Fe, Mg, Ca, and Na, all with the air-acetylene flame. Details of the method will be given elsewhere. Phosphorus and titanium were also determined photometrically on the main sample solution (*6*, *7*). The results are given in Table 1.

All determinations were based on a comparison of sample solutions with solutions similarly prepared from international reference rock samples. Values assigned to the compositions of the reference samples were based on data prepared by Abbey (*8*) and Govindaraju (*9*). For some elements, it was necessary to add standard solutions of the element to the solutions of the reference samples in order to bring the elemental concentration into the same range as was present in the samples.

An additional 500 mg were decomposed by acid attack and the resulting solution, containing free sulfuric acid and some added hydrochloric acid, was analyzed for lithium and zinc (Table 3) by atomic absorption spectroscopy (*10*). A "standard-addition" technique was used to overcome matrix effects.

The optical emission spectrographic procedure employed a d-c arc, air-jet controlled. Sample portions were mixed with a buffer consisting of alkali salts and graphite powder, at a dilution factor of 7.5, and arced to completion. The spectra were recorded in a 3.4-m Jarrell-Ash grating spectrograph. Because of the general complexity of these spectra, internal standards were not used. Instead, intensity ratios of trace element lines were related to selected iron lines in an "external standard" exposure on the same plate (consisting of Fe_2O_3 treated in the same manner as the samples). Concentrations (Table 3) were obtained from working curves, with corrections as required to correlate with standards of similar composition which were run along with the samples.

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Chemical Analyses of Lunar Samples 10017, 10072, and 10084

Abstract. *A crystalline rock, a vesicular rock, and a dust sample returned by Apollo 11 were chemically analyzed by several methods. The compositions of these samples are unlike that of any known rock or meteorite.*

The compositions of lunar samples 10017,20 (a crystalline rock), 10072,20 (a vesicular rock), and of 10084,102 (dust) are given in Tables 1–3. Three 1-g samples were used for each analysis. Wet chemical methods were used in determining the major elements. In order to obtain correct values for some major elements, especially aluminum, it was necessary to determine some trace elements as well. In determining these trace elements, emission spectroscopy, atomic absorption spectroscopy, and neutron activation were used (*1*). The experimentally obtained compositions differ markedly from the composition of all hitherto analyzed rocks and meteorites.

Table 1. Chemical composition of three Apollo 11 moon samples, expressed as oxides. Sample 10017 is crystalline rock; 10072 is vesicular rock; and 10084 is dust. G, gravimetric; C, colorimetric; AA, atomic absorption; T, titration; NA, neutron activity; Q, qualitative; and F, flame.

Oxide	Sample 10017 (%)	Sample 10072 (%)	Sample 10084 (%)	Methods used
SiO_2	40.77	40.20	42.25	G; C
TiO_2	11.82	12.28	7.24	C
Al_2O_3	7.92	7.78	13.83	G; AA
Fe_2O_3	0.0	0.0	0.0	T
FeO	19.79	19.77	15.80	T
Cr_2O_3	0.33	0.36	0.275	AA
MnO	0.22	0.22	0.20	C; NA
MgO	7.74	8.06	7.97	G
CaO	10.58	10.27	11.96	G
Na_2O	0.51	0.52	0.43	F; NA
K_2O	0.29	0.29	0.13	F; NA
P_2O_5	0.18	0.18	0.14	C
H_2O	0.00	0.00	0.00	G
CO_2	0.00	0.00	0.00	Q
Fe met.	0.0	0.0	0.0	Q
Total	100.15	99.93	100.53	

Table 2. Elemental composition of three Apollo 11 moon samples. D, by difference.

Element	Sample 10017 (%)	Sample 10072 (%)	Sample 10084 (%)	Method
Si	19.05	18.78	19.74	G; C
Ti	7.09	7.36	4.52	C
Al	4.19	4.12	7.32	G; AA
Fe	15.38	15.37	12.28	T
Cr	0.226	0.246	0.188	AA
Mn	0.17	0.17	0.155	C; NA
Mg	4.67	4.86	4.81	G
Ca	7.56	7.34	8.55	G
Na	0.38	0.385	0.32	F; NA
K	0.24	0.24	0.108	F; NA
P	0.08	0.08	0.06	C
H	0.00	0.00	0.00	G
O	(40.87)	(40.95)	(41.94)	D