

Chemical composition of Apollo 11 lunar samples 10017, 10020, 10072 and 10084

J. A. MAXWELL

Geological Survey of Canada, Ottawa, 4, Ontario

L. C. PECK

United States Geological Survey, Denver, Colorado 80225
and

H. B. WIIK

Finnish Research Council for Sciences, Helsinki, Finland

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Abstract—Major, minor and trace element analyses, by three separate laboratories, are reported for the lunar rocks 10017, 10020 and 10072, and for the fine surficial material, 10084. Brief details of the analytical procedures used are also given. The analyses confirm in general the results previously reported for lunar material, especially the high titanium content; water, carbon dioxide, fluorine, chlorine and Fe(III) are either absent or present in negligible amounts. It was not possible to determine metallic Fe but the anomalous reducing capacity of some lunar material has been demonstrated. The composition of the samples differs markedly from that of known rocks and meteorites.

INTRODUCTION

THREE samples of Apollo 11 rocks, and one of the lunar surficial material, were split and distributed among three Principal Investigators concerned with making bulk chemical analyses, as follows: Maxwell received 10017-29, 10020-30 and 10084-132, Peck was given 10020-23, 10072-32 and 10084-131, and Wiik received 10017-20, 10072-20 and 10084-102. Individual reports of the resulting analyses have already appeared (MAXWELL *et al.*, 1970; PECK and SMITH, 1970; WIIK and OJANPERÄ, 1970) but it has seemed advantageous to combine the data and analytical details into one paper, to facilitate comparison and use of the results obtained in three widely-separated laboratories.

Sample 10084 is an aliquot of the <1 mm fraction of the lunar surficial material, of which 20 g was split into three portions by Wiik in the Lunar Receiving Laboratory. The three rock specimens were broken into smaller pieces in the LRL and approximately equal portions of each (10–20 g) were distributed to each of the Principal Investigators selected to receive them. Further preparation of the samples for analysis was done in the individual laboratories.

ANALYTICAL TECHNIQUES

A. At the Geological Survey of Canada (JAM)

The conventional rock analysis methods used to determine most of the constituents are those described by MAXWELL (1968), with the exceptions of those for total carbon, carbon dioxide, fluorine, chlorine and total sulfur. The non-distillation, spectrophotometric method of HUANG and JOHNS (1967), as modified by SEN GUPTA (1968), was employed for the simultaneous determination of chlorine and fluorine. Total carbon and sulfur, liberated as CO₂ and SO₂ respectively by induction

Table 1. Chemical analyses (wt.%) of lunar material

Constituent	10017		10020		10072		10084		USGS
	GSC*	GSF*	GSC	USGS*	GSF	USGS	GSC	GSF	
SiO ₂	40.78	40.77	39.92	39.95	40.20	40.53	42.28	42.25	42.20
TiO ₂	11.71	11.82	10.72	10.52	12.28	11.74	7.35	7.54	7.32
Al ₂ O ₃	8.12	7.92	10.04	10.19	7.78	8.52	13.76	13.83	14.07
Fe ₂ O ₃	0.00	0.0	0.00	0.03	0.0	0.00†	0.00	0.0	0.00‡
FeO	19.82	19.79	19.35	19.14	19.77	19.76	16.02	15.80	15.81
Cr ₂ O ₃	0.36	0.33	0.40	0.38	0.36	0.35	0.33	0.28	0.28
MgO	7.65	7.74	7.81	7.87	8.06	7.68	7.93	7.97	7.93
CaO	10.55	10.58	11.24	11.31	10.27	10.42	12.00	11.96	12.01
Na ₂ O	0.51	0.51	0.37	0.39	0.52	0.54	0.42	0.43	0.46
K ₂ O	0.30	0.29	0.05	0.05	0.29	0.27	0.13	0.13	0.12
H ₂ O ⁺	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.05
H ₂ O ⁻	0.01		0.01	0.00		0.00	0.01		0.00
P ₂ O ₅	0.13	0.18	0.08	0.07	0.18	0.14	0.11	0.14	0.08
MnO	0.22	0.22	0.24	0.27	0.22	0.24	0.20	0.20	0.21
S	0.22	ND§	0.15	0.18	ND	0.24	0.13	ND	0.14
CO ₂	0.00	0.00	0.00	ND	0.00	ND	0.03	0.00	ND
F	0.00	ND	0.00	ND	ND	ND	0.00	ND	ND
Cl	0.00	ND	0.00	ND	ND	ND	0.00	ND	ND
C	0.01	ND	0.01	ND	ND	ND	0.00	ND	ND
Fe ⁰	ND	0.0	ND	ND	0.0	ND	ND	0.0	ND
Sub total	100.39	100.15	100.39	100.35	99.93	100.43	100.70	100.53	100.68
Less O≡S	0.11	—	0.08	0.09	—	0.12	0.07	—	0.07
Total	100.28	100.15	100.31	100.26	99.93	100.31	100.63	100.53	100.61

* GSC—Geological Survey of Canada: analysts, J. A. Maxwell, S. Abbey (Cr₂O₃, Na₂O, K₂O) and J. G. Sen Gupta (total C and S).

GSF—Geological Survey of Finland: analysts, H. B. Wiik and P. Ojanperä; P. Kauranen, P. Puumalainen, E. Häsänen and R. Rosenberg (Mn, Na, K).

USGS—United States Geological Survey: analysts, L. C. Peck and Vertie C. Smith.

† Actual value obtained, —0.14.

‡ Actual value obtained, —0.12.

§ ND—not determined.

furnace heating (SEN GUPTA, 1970), were determined titrimetrically, the CO₂ by non-aqueous titration (SEN GUPTA, 1969). The CO₂ released by heating the samples with acid was similarly determined. The results are given in Table 1; a reference basalt sample, USGS BCR-1, was run simultaneously with the lunar samples, to provide a means for evaluating the precision and accuracy of the data (Table 2).

The optical emission spectrographic determination of minor and trace elements was made with an air-jet controlled d.c. arc and a Jarrell-Ash 3.4 m grating spectrograph; 30-mg portions of the

Table 2. Chemical analysis (wt.%) of reference basalt (USGS BCR-1)*

SiO ₂	— 54.48	H ₂ O ⁺	— 0.66
TiO ₂	— 2.26	H ₂ O ⁻	— 0.86
Al ₂ O ₃	— 13.62	P ₂ O ₅	— 0.347
Fe ₂ O ₃	— 3.77	MnO	— 0.165
FeO	— 8.65	S	— 0.033
MgO	— 3.46	CO ₂	— 0.055
CaO	— 6.91	F	— 0.039
Na ₂ O	— 3.36	Cl	— 0.002
K ₂ O	— 1.76	C	— 0.00
Sub total	— 100.43		
Less O≡S	— 0.02		
Total	— 100.41		

* Analysts, J. A. Maxwell and S. Abbey (Na₂O and K₂O), Geological Survey of Canada.

Table 3. Minor and trace element content (ppm) of lunar material

Constituent	10017		10020	10072	10084		BCR-1
	GSC*	GSF*	GSC	GSF	GSC	GSF	GSC
Cr	2200	2260	3000	2460	2300	1880	<20
Zr	410	430	210	460	260	380	<200
Ba	230		67		150		700
Sr	100		130		140		220
V	49	70	120	82	67	71	420
Ni	<20	<5	<20	<5	190	200	<20
Co	<20	30	<20	30	<20	32	<20
Cu	<5	20	<5	21	<5	13	10
Zn	49	47	26	24	47	36	120
Li	23	ND	12	ND	15	ND	15
Sc	52	77	78	77	51	59	23
Y	160	160	120	175	120	120	34
Yb	<10	19	<10	19	<10	12	<10
Sm	ND	25	ND	21	ND	18	ND
Eu	ND	2.1	ND	2.0	ND	1.8	ND
Tb	ND	4	ND	3	ND	3	ND
Lu	ND	5	ND	5	ND	3	ND
Hf	ND	12	ND	12	ND	8	ND
Ta	ND	5	ND	5	ND	3	ND
Th	ND	4.6	ND	4	ND	3	ND

* GSC—Geological Survey of Canada: all determinations by emission spectroscopy (analysts, W. H. Champ, K. A. Church, D. A. Brown, Joanne Crook, G. A. Bender), except Zn and Li (atomic absorption spectroscopy, S. Abbey, analyst); < = less than; ND = not determined.

GSF—Geological Survey of Finland: analysts, H. B. Wiik and P. Ojanperä (atomic absorption spectroscopy, *AAS*); A. Löfgren and A. Savola (emission spectroscopy, *ES*); P. Kauranen, P. Puumalainen, E. Häsänen and R. Rosenberg (neutron activation, *NA*); Sc, V, Cu, Y and Zr by *ES*; Sm, Eu, Tb, Lu, Yb, Hf, Ta and Th by *NA*; Zn by *AAS*; Co by *ES* and *NA*; Ni by *ES*, *NA* and *AAS*.

samples were mixed with a buffer at a dilution factor of 7.5, and 45-mg charges of this mixture taken. No internal standard was used, but intensity ratios of trace element lines were related to selected iron lines in an “external standard” (Fe_2O_3) exposure on the same photographic plate. Standard samples having compositions generally similar to the samples were run simultaneously and used to correct the working curves. The data are given in Table 3, together with the results obtained for the USGS basalt, BCR-1.

During preparation of the samples for analysis, a very small amount of a metallic phase (metallic Fe?) was noted only in sample 10084-132. Although it could not be determined by known methods for the determination of metallic Fe, it would, however, contribute to the value obtained for Fe(II) by the modified Pratt method (MAXWELL, 1968). The possibility of the presence of some Ti(III) has also been suggested and, in order to determine the magnitude of the error likely to be introduced by these constituents into the determination of Fe(II), the procedure was further modified by the addition of a known quantity of an oxidant, Fe(III), to each sample before its decomposition with acid. Following the potentiometric determination of the “Fe(II)”, the total Fe was similarly determined in the same solution. The values obtained are given in Table 4 and it is seen that small anom-

Table 4. Reducing capacity (wt.%) of lunar material*

Sample no.	(1)	(2)	Difference (1 — 2)
	“Fe(II)” as FeO	Total Fe as FeO	
10017-29	19.82	19.81	+0.01
10020-30	19.43	19.28	+0.15
10084-132	17.05	16.02	+1.03

* Analyst, J. A. Maxwell, Geological Survey of Canada.

alies were found for the two rock samples, 10017-29 and 10020-30, and a significantly large one for the fines, 10084-132. If it is assumed that the difference in Fe(II) was caused only by reduction of the Fe(III) added, then the metallic Fe content of 10084-132 is about 0.3 per cent; similarly, the difference could be caused by the presence of about 0.7% Ti(III). While the source(s) cannot be stated with certainty, it is suggested (JAM) that metallic iron is the more likely candidate.

The use of atomic absorption spectroscopy and other techniques requiring small sample weights was given early consideration in case the quantity of sample available for analysis should prove to be limited. Although the latter situation did not arise, it was decided to apply a new composite scheme, developed by Sydney Abbey at the Geological Survey of Canada, to the determination of some major and minor constituents of the lunar samples, as a test of its future applicability to samples of this nature; two sample portions were used for the analysis, and the results are given in Table 5.

Table 5. Chemical analyses (wt.%) of lunar material by new composite scheme*

Constituent	10017-29	10020-30	10084-132
SiO ₂	40.14	41.00	42.44
TiO ₂	11.16	10.28	7.19
Al ₂ O ₃	8.17	9.83	13.66
Total Fe, expressed as FeO	19.38	19.03	15.67
MgO	7.72	7.77	7.87
CaO	10.99	11.96	12.26
Na ₂ O	0.51	0.37	0.42
K ₂ O	0.30	0.05	0.13
P ₂ O ₅	0.15	0.07	0.10
MnO	0.25	0.27	0.21
Cr ₂ O ₃	0.36	0.40	0.33
Total	99.13	101.30	100.28

* Analysts, S. Abbey (atomic absorption spectroscopy) and J. L. Bouvier (TiO₂ and P₂O₅, by colorimetry, on solutions prepared for AAS).

A 200-mg sample was fused with a fivefold excess of lithium metaborate and the fusion dissolved in dilute hydrofluoric acid; sufficient boric acid was added to provide a solution which was stable towards glass for at least 2 hr (BERNAS, 1968). Two dilutions were prepared, one containing 50 mg of sample and 300 mg Sr, and the other 10 mg and 150 mg, in 100 ml. The more concentrated solution was used for the determination by atomic absorption spectroscopy of Si and Al, using a nitrous oxide-acetylene flame, and K, Mn and Cr with the air-acetylene flame. Fe, Mg, Ca and Na were determined in the second solution, using an air-acetylene flame. Details of the method will be published elsewhere. Two more aliquots of the original sample solution were used for the photometric determination of phosphorus and titanium.

An additional 500 mg sample was decomposed by acid treatment and the resulting solution, containing free sulfuric acid and some added hydrochloric acid, was analyzed for Li and Zn by atomic absorption spectroscopy, using the method of "standard addition" (ABBEY, 1967).

All determinations were based upon a comparison of sample solutions with solutions similarly prepared from international reference rock samples, using assigned values based upon data prepared by ABBEY (1970) and GOVINDARAJU (1969). All atomic absorption measurements were made with a modified Techtron Model AA-3 instrument.

B. At the United States Geological Survey (LCP)

Except where otherwise noted, the classical procedures used are those described by PECK (1964), and the page numbers given in the following brief discussion refer to this publication. The results obtained are given in Table 1.

The procedure for the determination of SiO₂ (p. 62) was modified to eliminate complications caused by the high titanium content of the samples; the separated silica was fused with Na₂CO₃

and the dehydration and filtration repeated. The two filtrates were combined for the precipitation of the ammonium hydroxide group. The determination of titanium was done colorimetrically (p. 70) with a probable precision of ± 0.1 per cent absolute.

The known presence of troilite (FeS) and metallic Fe makes the results obtained for Fe(II) uncertain (pp. 39–42); metallic Fe is counted as FeO in the summation. Because Fe(III) is determined by difference after the separate determinations of total Fe and Fe(II), the cumulative errors in both of these determinations are therefore reflected in the value found for Fe(III). The small negative and positive values obtained for Fe(III) are probably the result of such errors, and, within the accuracy limits of the method, it is likely that the samples do not contain any Fe(III).

The value for H_2O^+ reported for sample 10084-131 is probably for water which was adsorbed after the sample was exposed to the terrestrial atmosphere; strongly adsorbed water is not expelled when a silicate is heated at 105°C (H_2O^-).

It is likely that most of the chromium present is in the mafic minerals. A small amount of black material that might have been chromite was observed in sample 10084-131 but there was no evidence of its presence in the other samples. The procedures for the determination of chromium, and of sulfur, are those described by KOLTHOFF and SANDELL (1952).

Metallic iron was not determined because of doubts about the reliability of published methods, and it might be more useful to use sample material for an examination of a separated magnetic phase by other techniques such as Mössbauer spectroscopy. The presence of metallic iron, which is reported as FeO, could account for the unacceptably high summation given in Table 1 for sample 10084-131 (similarly high summations are given for the other splits by both the GSC and GSF). There would have to be about 3 per cent of it, however, and data from other sources suggest that 1 per cent or less is probably present. Other factors, such as powder segregation in samples containing grains having a high density, may be contributing as well to the high summation.

C. *At the Geological Survey of Finland (HBW)*

The constituents given in Table 1 were determined by the following methods: MgO, CaO and total H_2O , gravimetric; TiO_2 and P_2O_5 , colorimetric; SiO_2 , gravimetric and colorimetric; Al_2O_3 , gravimetric and atomic absorption spectroscopic; FeO and Fe_2O_3 , titrimetric; Cr_2O_3 , atomic absorption spectroscopic; MnO, colorimetric and neutron activation; Na_2O and K_2O , flame emission spectroscopic and neutron activation. The determination of CO_2 and metallic Fe are qualitative. The methods used to determine other trace constituents are given, together with the results, in Table 3.

SUMMARY AND CONCLUSIONS

The compositional data given for the four lunar samples thus confirm, in general, the data previously presented by LSPET (1969), with the exception of the values for Cr, Zr and Ba. It is significant that the best agreement between the replicate results is found in the three analyses for sample 10084, reflecting the greater degree of homogeneity of this fraction of the fine surficial material. The occasional differences which are observed in the duplicate values for the three rocks may in part be caused by inhomogeneity in the distributed portions.

The composition of these four samples do not resemble any known terrestrial rock, nor any known meteorite. The very low nickel content is especially peculiar, and would make a common origin for lunar, terrestrial and meteoritic material very doubtful.

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