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OXYGEN ISOTOPE FRACTIONATION IN APOLLO 12
ROCKS AND SOILS

(NAS 9-7888)

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ABSTRACT

Oxygen isotopic compositions of individual minerals and isotopic fractionations between coexisting minerals are remarkably similar for Apollo 11 and 12 crystalline rocks. Apollo 12 rocks show a somewhat lower plagioclase-ilmenite "isotopic temperature" of 1070°C , related to the late crystallization of ilmenite in these rocks.

Oxygen isotope analyses have been carried out on separated minerals from seven crystalline rocks (12018, 12021, 12038, 12040, 12052, 12063 and 12064), on the olivine and matrix from two olivine vitrophyres (12008 and 12009), and on four soil samples (12033, 12037, 12057, 12070).

Analytical Procedures.

The procedures for mineral separation, oxygen extraction and mass spectrometric analysis were the same as those used for the Apollo 11 samples (Onuma et.al., 1970). Results of isotopic analyses are given in Table 1. Standard error in the mean of the duplicate measurements on each sample is estimated to be ± 0.07 per mil.

Results and Discussion

As was the case with the Apollo 11 crystalline rocks, the seven Apollo 12 crystalline rocks are indistinguishable from one another in terms of the isotopic compositions (δ -values) of individual minerals (Table 1), and in terms of the mineral-pair isotopic fractionations (Δ -values) for mineral pairs (Table 2).

Comparison of isotopic fractionations in Apollo 11 rocks with those in Apollo 12 rocks shows a significant difference in mineral-pair fractionations involving olivine. Olivine was analyzed from only one Apollo 11 sample (10020), giving a δ -value of 5.14, which coincides with the values for Apollo 12 olivines. Texturally, the olivine in 10020 appears to be in reaction relation to clinopyroxene (Dence et.al., 1970), and may not be in isotopic equilibrium with the other major phases.

There is also a small difference between Apollo 11 rocks and Apollo 12 rocks with respect to oxygen isotope fractionations involving ilmenite, the latter Δ -values being about 0.15 to 0.20 greater. This is consistent with the relative position of ilmenite in the crystallization sequences of the two sets of rocks, being early for Apollo 11 rocks (O'Hara et.al., 1970), and late for Apollo 12 rocks (Biggar et.al., 1971). Although there is obvious difficulty in attaching precise thermometric significance to isotopic fractionations between two minerals which crystallized over non-coincident temperature ranges, straightforward application of the plagioclase-ilmenite thermometer (Onuma et.al., 1970) gives mean isotopic temperatures of 1120°C for Apollo 11 rocks (both A and B types), and mean temperatures of 1070°C for Apollo 12 rocks, in good agreement with the phase equilibrium data of Biggar and co-workers (1971).

Biggar and co-workers (1971) concluded that rocks 12038, 12018 and 12040 were derived from a common magma by progressive addition of cumulate olivine, and that 12064, 12021 and 12052 are similarly related by addition of cumulate pigeonite. The oxygen isotope data show no trends with respect to these groups of rocks.

The olivine vitrophyres, 12008 and 12009, appear to be simply rapidly crystallized equivalents of other more coarsely crystalline rocks. The olivine phenocrysts have the same isotopic compositions as the cumulate olivine in 12018 and 12040, and the matrix has the same composition as the whole-rock values estimated for other rocks from their mineral analyses.

In four cases the clinopyroxene fraction was separated on the basis of density into iron-rich and iron-poor portions; these were found to be isotopically indistinguishable. In 12021 and 12040, pigeonite was separated

by hand-picking, and in both cases was found to be enriched in O^{18} by about 0.2‰ relative to the Ca-clinopyroxene. Because of the separation procedure, the pigeonite analyzed is derived predominantly from the cores of large zoned crystals. The significance of the 0.2‰ difference is not clear. It may be the result of different isotopic fractionation factors for pyroxenes of different chemical composition, or may represent a departure from isotopic equilibrium resulting from either different positions in the crystallization sequence or to actual non-equilibrium crystallization. Rock 12064 also yielded an analyzable amount of pyroxferroite, which was found to have almost the same oxygen isotopic composition as the co-existing clinopyroxene. Tridymite from the same rock also appears to be in isotopic equilibrium, and has the same isotopic composition as cristobalite in Apollo 11 rock 10058 (Onuma et.al., 1970).

In comparing δ -values of separated minerals and of soil samples, there appears to be a systematic difference between Apollo 11 and Apollo 12 rocks, the latter being lower in O^{18} by about 0.2‰. This difference, if real, is certainly very small, and reflects the small oxygen isotope effects associated with fractional melting or crystallization. This suggests that the range of about two permil found for terrestrial basalts from diverse localities (Taylor, 1968; Garlick, 1966; Anderson et.al., 1971), previously considered a small range, is probably larger than can be accounted for only by processes at igneous temperatures, and reflects exchange or contamination with materials in the crust or upper mantle which have undergone low-temperature processes. Such low-temperature chemical

processes are probably absent on the moon. The uniformity of oxygen isotopic composition of rocks and soil at the two sites analyzed and of rocks of different ages (Taylor and Epstein, 1970) strengthens the assumption that this composition is typical of the lunar surface, and thus further strengthens the case against derivation of tektites or eucrites from the moon. (Onuma et.al., 1970; Epstein et.al., 1970; Friedman et.al., 1970).

The four soil samples analyzed are almost identical in isotopic composition and are enriched in O^{18} by about 0.2‰ relative to the average whole-rock compositions of the crystalline rocks, as was also the case for the Apollo 11 soils. A fine-grained fraction of soil 12070 was separated by flotation in acetone (grain-size estimated to be < 0.3 microns) and was found to have $\delta O^{18} = 7.34\text{‰}$. A sample similarly separated from Apollo 11 soil 10084 has a $\delta O^{18} = 7.60\text{‰}$. A similar enrichment in the very fine fraction of 10084 soil was noted by Epstein and Taylor (1971), who attribute the effect to a depletion of the light isotope during vaporization resulting from bombardment by high energy particles. Other reasonable possibilities are volatilization resulting from heating in impact events, or addition of a fine-grained extra-lunar component enriched in O^{18} relative to the lunar rocks.

Our analysis of sample 12033 is identical with that reported for this sample by Epstein and Taylor (1971). However, they report a δO^{18} of 6.26 for soil sample 12070, whereas, our value for 12070,46 is the same as our value for 12033 ($\delta O^{18} = 5.87$). On the basis of experiences to date

with lunar soil samples, it would be surprising if this difference were due to sample heterogeneity unless the samples analyzed were of different grain size ranges.

Conclusions.

Oxygen isotope variations in lunar igneous rocks of the mare regions are exceedingly small indicating the absence of large-scale, low-temperature processes. Isotopic fractionations within rocks correspond to equilibrium at temperatures near the solidus, independent of grain size or texture. This implies quenching of the isotopic compositions on solidification as a result of rapid cooling and the absence of water.

The main components of the regolith are derived from the crystalline rocks without change in oxygen isotopic composition, but some process has produced O^{18} -enriched material in the finest size-fractions.

Acknowledgements

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TABLE 1. Oxygen isotopic compositions* of Apollo 11-12 rocks and minerals.

[illegible]

TABLE 1 (CONT). Oxygen isotopic compositions of Apollo 12 rocks and minerals.

* δ^{18} defined as $\left(\frac{(^{18}\text{O}/^{16}\text{O})_{\text{sample}}}{(^{18}\text{O}/^{16}\text{O})_{\text{standard}}} - 1 \right) 1000$, where the standard is SMOW (Standard Mean Ocean Water)

** Abbreviations:

Tr	tridymite
Pc	plagioclase
Pg	pigeonite
Cpx	calcium clinopyroxene
Pxf	pyroxferroite
Ol	olivine
Il	ilmenite
Mx	matrix
R	whole rock

*** Corrected by 0.21‰ for olivine and pyroxene contamination.

TABLE 2. Oxygen isotope fractionations* in Apollo 11 and 12 rocks.

	<u>Apollo 11</u>	<u>Apollo 12</u>
Pc-Cpx	0.45 $\pm$.05	0.39 $\pm$.07
Pc-O1	1.04	0.78 $\pm$.04
Pc-I1	1.76 $\pm$.06	1.89 $\pm$.10
Cpx-O1	0.59	0.46 $\pm$.04
Cpx-I1	1.31 $\pm$.08	1.50 $\pm$.07
O1-I1	0.72	0.99 $\pm$.01

* Fractionations are given as $\Delta = 1000 \ln \alpha$, where,

$$\alpha_{AB} = \frac{(O^{18}/O^{16})_A}{(O^{18}/O^{16})_B} \quad \text{for two minerals A and B.}$$

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Apollo 11 rocks: Oxygen isotope fractionation between minerals, and an estimate of the temperature of formation

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Abstract.—Oxygen isotopic compositions of separated minerals from three Type A and four Type B rocks are very uniform. $\delta^{18}\text{O}$ values are: plagioclase 6.20, clinopyroxene 5.75, ilmenite 4.45 (parts per thousand relative to Standard Mean Ocean Water). The isotopic distribution corresponds to equilibrium at 1120°C. The isotopic composition of lunar pyroxenes falls within the range for pyroxenes of terrestrial mafic and ultramafic rocks, ordinary chondrites, enstatite chondrites, and enstatite achondrites, but above the range for basaltic achondrites, hypersthene achondrites, and mesosiderites. Glass isolated from the lunar soil has a $\delta^{18}\text{O}$ of 6.2, significantly richer in ^{18}O than the crystalline rock fragments in the soil.

OXYGEN isotope analyses have been done on the separated major mineral phases of seven lunar rocks of types A and B, on whole rock samples of types C and D, and on glass spherules and rock fragments separated from type D.

One-gram samples of the crystalline rocks were crushed in a steel mortar, ground in an agate mortar under acetone, and sieved through stainless-steel sieves. For the coarser-grained type B rocks, the size fraction between 325- and 500-mesh (25–43 μm) was used for mineral separation. For the finer-grained type A rocks, the fraction passing through the 500-mesh sieve (<25 μm) was used. Mineral separation was accomplished in a few cases by hand-picking grains from the coarser fractions, but usually by centrifugation in heavy liquids (bromoform, methylene iodide, and Clerici solutions). Densities of the liquids used are given for each sample in Table 1. Mineral purity was evaluated by X-ray diffraction and optical microscopy, and was >95 per cent in all samples, and usually >98 per cent.

Oxygen was extracted from 10-mg portions of the separated minerals by the bromine pentafluoride procedure (CLAYTON and MAYEDA, 1963). Manometric measurement of the oxygen yield gave the quantitative determinations of oxygen content (Tables 1 and 2). Experimental error is estimated at ± 1 per cent of the amount present. Oxygen was converted to carbon dioxide and the oxygen isotope ratio was measured with a 60°, 15-cm, double-collecting mass spectrometer. Oxygen extraction was carried out in duplicate for all samples, and each gas sample was further analyzed in duplicate. Results of isotope analyses are given in Tables 1 and 2. Standard error of the mean of the duplicate measurements is estimated to be ± 0.07 per mil.

Oxygen contents of all phases analyzed are in agreement with values calculated from the estimated major-element compositions. No evidence of nonstoichiometry with respect to oxygen has been seen.

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Table 1. Oxygen contents and isotopic compositions of minerals from Type A and Type B rocks

	Type A Rocks				Type B Rocks		
	10020-24	10022-36	10071-22	10017-21	10044-22	10047-15	10058-35
(a) Specific gravity*							
Cr†							<2.38
Pc	2.70-2.82	2.71-2.81	2.71-2.82	2.70-2.77	2.69-2.77	2.70-2.75	2.71-2.82
Cpx	3.36-3.46	3.36-3.44	3.36-3.47	3.33-3.56	hand-pick	hand-pick	3.36-3.52
Ol	3.54-3.65						
Il	>4.2	>4.2	>4.2	>4.3	>4.2	>4.3	>4.4
(b) Oxygen content (wt. %)							
Cr							53.4
Pc	45.8	46.3	47.2	47.3	46.4	46.5	46.7
Cpx	42.0	42.6	42.1	42.9	42.5	42.0	42.6
Ol	40.5						
Il	33.7	33.9	33.1	33.3	32.0	31.9	32.4
(c) $\delta^{18}\text{O}_\dagger$ (rel. to SMOW)							
Cr							7.09
Pc	6.19	6.31	6.17	6.16	6.18	6.20	6.00
Cpx	5.74	5.71	5.72	5.81	5.77	5.76	5.53
Ol	5.14						
Il	4.41	4.55	4.47	4.51	4.35	4.29	4.24
(d) $\Delta^{18}\text{O}_\S$							
Pc-Cpx	0.45	0.60	0.45	0.35	0.41	0.44	0.47
Cpx-Il	1.32	1.15	1.24	1.29	1.41	1.46	1.28
Pc-Il	1.77	1.75	1.69	1.64	1.82	1.90	1.75

* Values quoted are specific gravities of liquids which bracket the value for the separated mineral.

† Abbreviations for mineral names:

Cr—cristobalite, Pc—plagioclase, Cpx—clinopyroxene, Ol—olivine, Il—ilmenite.

‡ ^{18}O content in per mil terminology: deviation of $^{18}\text{O}/^{16}\text{O}$ ratio in parts per thousand from ratio in Standard Mean Ocean Water (SMOW).

§ Oxygen isotope fractionation, $\Delta^{18}\text{O}$, defined as $1000 \ln \alpha$, where α is a fractionation factor for two phases, e.g. $\frac{(^{18}\text{O}/^{16}\text{O})_{\text{Pc}}}{(^{18}\text{O}/^{16}\text{O})_{\text{Il}}}$.

Table 2. Oxygen contents and isotopic compositions of various fractions of Type C and Type D rocks

Sample description	O Content (wt. %)	$\delta^{18}\text{O}$ (rel. to SMOW)
Type C		
10060-12 whole rock	41.3	6.03
Type D		
10084-46 whole rock	42.6	6.18
10084-46 glass spherules	42.2	6.22
10084-46 glass fragments	42.3	5.88
10084-46 Type A fragments	41.7	5.74
10084-46 Type B fragments	40.6	5.64
10084-46 "Anorthosite" fragments	44.8	5.89

The uniformity of isotopic composition from rock to rock, for a given mineral, is very striking. Of the type A and type B rocks, only sample 10058-35 shows any systematic departure from the mean, each of its minerals having an ^{18}O content 0.2 per mil lower than the means of the other rocks.

The soil and microbreccia have ^{18}O contents similar to one another, and about 0.5 per mil greater than that of the crystalline rocks. From the approximate modal compositions of the crystalline rocks given in the preliminary reports, their whole-rock $\delta^{18}\text{O}$ is estimated to be 5.7 ± 0.1 per mil. Fragments of type A and type B rocks were hand-picked from the 50–100 mesh (150–300 μm) fraction of the lunar soil and were found to have isotopic compositions in this range (Table 2). Irregular brown glass fragments (refractive index 1.68) were found to have almost the same composition, and may have been derived by fusion of crystalline rocks.

Glassy spherules, mean dia. 300 μm , were also separated from the soil, and have an ^{18}O -content significantly greater than that of the rock fragments, implying addition of an isotopically heavier component or an isotopic fractionation in the process of formation of the spherules. An addition to the soil of 2 per cent carbonaceous-chondrite-like meteorites, as suggested by KEAYS *et al.* (1970), would cause an ^{18}O enrichment of about 0.2 per mil. Addition of other known types of meteoritic material would have a negligible effect because of the similarity of their isotopic compositions to that of the soil (TAYLOR *et al.*, 1965). Enrichment of ^{18}O in glass resulting from a loss of volatiles in very-high-temperature fusion has been shown experimentally (WALTER and CLAYTON, 1967), and may play some part on the moon.

The mean value of the $^{18}\text{O}/^{16}\text{O}$ ratio in objects of the solar system may be determined in part by the region in the early solar nebula within which the solid particles accreted. There may have been gradients in isotopic composition which are reflected today in differences among the planets, their satellites, and the meteorites and asteroids. Such differences have been observed for various classes of meteorites by TAYLOR *et al.* (1965). Lacking data for the average oxygen-isotope composition of the moon, perhaps the best we can do at this stage is to compare the isotopic composition of a particular major mineral with that of similar minerals in terrestrial rocks and meteorites. The ^{18}O -content of the Apollo 11 pyroxenes is identical with that of pyroxenes from some mantle-derived rocks: garnet peridotites and pyroxenites from kimberlite pipes (GARLICK, 1966; ANDERSON *et al.*, 1970). The lunar pyroxenes lie at the high end of the distribution for oceanic basalts and andesites (GARLICK, 1966; TAYLOR, 1968; ANDERSON *et al.*, 1970).

The lunar pyroxenes fall into group II of TAYLOR *et al.* (1965), which contains the ordinary chondrites, enstatite chondrites, and enstatite achondrites. They are distinctly richer in ^{18}O than the pyroxenes of group I meteorites: basaltic achondrites, hypersthene achondrites, and mesosiderites.

If we speculate that oxygen isotope gradients in the early solar system were in the direction of decreasing ^{18}O outward, as suggested by the data on meteorites, then the oxygen isotope results on Apollo 11 rocks indicate an initial condensation either at the same position as terrestrial material, or somewhat nearer the sun.

Isotopic fractionations between pairs of minerals can be used to determine the temperature of last equilibration of the minerals. In favorable cases, this may simply

be the temperature of their initial crystallization. Isotopic fractionations between pairs of major phases are shown in Table 1. For a given mineral pair the observed fractionations are identical, within experimental error, for all seven rocks analyzed: plagioclase-clinopyroxene, 0.45 ± 0.04 ; clinopyroxene-ilmenite, 1.31 ± 0.08 ; plagioclase-ilmenite, 1.76 ± 0.06 .

In principle, any two of these fractionations provide two independent measures of "isotopic temperature." A minimal test of the existence of equilibrium is that these

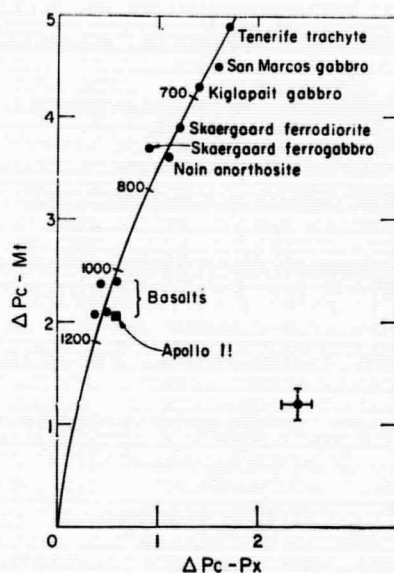


Fig. 1. Plagioclase-clinopyroxene-magnetite "concordancy diagram". Data on terrestrial rocks are from TAYLOR (1968), GARLICK (1966) and ANDERSON *et al.* (1970). Temperatures indicated along concordancy line are determined from the plagioclase-magnetite isotopic thermometer (O'NEIL and TAYLOR, 1967; O'NEIL and CLAYTON, 1964).

two temperatures agree. Unfortunately, laboratory calibration of isotopic thermometers involving pyroxene and ilmenite has not been done, so that this test cannot yet be carried out rigorously. It is, however, possible to compare the relative magnitudes of two isotopic fractionations with those found within the same mineral assemblage in various terrestrial rocks. This is done in Fig. 1, for the assemblage: plagioclase-clinopyroxene-magnetite using analytical data for terrestrial mafic igneous rocks, both intrusive and extrusive (GARLICK, 1966; TAYLOR, 1968; ANDERSON *et al.*, 1970). Because of the dependence of fractionations involving plagioclase on the chemical composition of the plagioclase (O'NEIL and TAYLOR, 1967), all data have been normalized to a feldspar composition of An₆₀. This amounts to an effective increase in the δ -value of lunar plagioclase by 0.16 per mil. Furthermore, the isotopic fractionation

between ilmenite and magnetite is very small, but not negligible, at igneous temperatures, so that the ilmenite δ -value has been reduced by 0.20 per mil to determine a hypothetical magnetite δ -value. The resulting point, representing the average fractionation for the seven Apollo 11 samples analyzed has been plotted on the plagioclase-pyroxene-magnetite diagram, and falls on the previously determined line through the data points for terrestrial rocks. It is virtually indistinguishable from the results for recent Hawaiian basalts. Hence the evidence for isotopic equilibrium among the major phases is fairly strong.

A similar "concordancy" test for the system plagioclase-clinopyroxene-olivine indicates that the olivine of sample 10020-24 was in isotopic equilibrium with the other minerals. Data for terrestrial samples are not available to make this test for cristobalite in sample 10058-35, but the magnitude of the cristobalite-plagioclase fractionation of 1.08 is within 0.1 of the value estimated by extrapolation of laboratory calibrations of the quartz-feldspar system to the isotopic temperature inferred for these rocks.

The best estimate of an isotopic temperature can be made from the fractionation between plagioclase and ilmenite. This is possible through the experimental calibration of the plagioclase-magnetite pair (O'NEIL and TAYLOR, 1967; O'NEIL and CLAYTON, 1964), along with the small ilmenite-magnetite fractionation, estimated on the basis of measurements on terrestrial basalts to be about 0.20 at the temperatures involved. The resulting isotopic temperature is 1120°C, with an analytical uncertainty of $\pm 30^\circ\text{C}$. (Somewhat larger systematic errors may exist in the calibration of this isotopic thermometer, particularly since this temperature lies outside the range of the laboratory calibration.)

The isotopic temperature is very similar to those of terrestrial mafic extrusive rocks, which in turn, are in good agreement with other estimates of extrusion temperatures. Hence it appears that the isotopic temperatures are very close to the initial temperatures of crystallization from a silicate melt. On earth, large differences in isotopic temperatures are found between fine-grained, rapidly-quenched volcanic rocks, and their coarse-grained plutonic equivalents (GARLICK, 1966; TAYLOR, 1968). This has been interpreted as resulting from postcrystallization retrograde isotopic exchange, on a scale comparable to the grain size, during the slow cooling of the plutonic rocks. No terrestrial rock as coarse-grained as the Apollo 11 type B samples has been found with a high isotopic temperature corresponding to its initial crystallization. Apparently the cooling conditions of the lunar samples were such as to prevent significant subsolidus exchange. The absence of water may have been an important factor.

The indistinguishability between type A and B rocks in terms of isotopic compositions or isotopic fractionations suggests that they were derived from magmas of the same isotopic composition, and that in both types the initial isotopic distribution at the time of crystallization has been quenched in, and has subsequently remained unchanged.

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