Table 2. Isotope-dilution analyses for Rb, Sr, and <sup>s7</sup>Sr/<sup>s6</sup>Sr of lunar rocks and mineral concentrates. t.r. denotes total-rock; plag., plagioclase; pyrox., pyroxene; and meso., mesostasis.

		Rb (ppm)	Sr (ppm)	<sup>87</sup> Rb/ <sup>86</sup> Sr	<sup>87</sup> Sr/ <sup>86</sup> Sr
		Group	1 lavas		
10024,20	t.r.	5.75	169.5	.0980	.70464
10072,30	t.r.	5.60	168.5	.0959	.70467
	plag.	3.87	531.0	.0210	.70063
	pyrox.	1.87	85.9	.0629	.70306
10017,68	t.r.	5.59	167.1	.0966	.70460
., .	meso. A	19.5	282.7	.1993	.71034
	meso. B	20.9	292.8	.2066	.71058
	plag.	0.92	499.9	.0053	.69970
		Group	2 lavas		
10003,20	t.r.	0.63	159.9	.0114	.69996
10062,36	t.r.	0.88	190.0	.0133	.70003
10045,20	t.r.	0.62*	137.7*	.0130*	.70005
,	plag.	1.33	299.0	.0129	.69999
	pyrox.	0.65	86.7	.0220	.70036
10047,21	t.r.	1.11*	209.2*	.0153*	.70009
,	plag.	0.53	574.6	.00268	.69949
		Bre	eccias		
10061,25	t.r.	3.69	166.1	.0631	.70306
10018,20	t.r.	3.64	166.1	.0633	.70299
		2	Soil		
10084,44	t.r.	2.65	164.4	.0466	.70233

\* X-ray fluorescence result.

which need not have the same age or the same primary <sup>87</sup>Sr/<sup>86</sup>Sr. Nor can precise total-rock isochrons be defined within each group, as the internal dispersion in Rb/Sr is too low. Analyses of separated minerals were thus essential. Nevertheless, assuming that both groups are precisely the same in age and initial <sup>87</sup>Sr/86Sr, we obtain a total-rock age of  $3.80 \pm 0.12$  billion years and an initial <sup>87</sup>Sr/<sup>86</sup>Sr of  $0.69935 \pm 0.00012$ . Using the x-ray spectrometric values alone for <sup>87</sup>Rb/  $^{86}$ Sr, these parameters become 3.77  $\pm$ 0.20 billion years and 0.69934  $\pm$ 0.00017. In both cases, the scatter of points slightly exceeds their assigned experimental errors, and the lines were fitted by assigning greater y variance.

By heavy liquid and magnetic separation, mineral concentrates that ranged from 50 to 200 mg in weight were made from two samples of each group (Table 2); sample 47 of plagioclase was handpicked. Data for plagioclase, pyroxene, and total-rock from sample 72 are well correlated, which suggests an age of crystallization of  $3.8 \pm 0.3$ billion years. The same age is given by the mineral data for sample 17:  $3.80 \pm 0.11$  billion years with an initial  $^{87}$ Sr/ $^{86}$ Sr of 0.6994 ± 0.0002. Most of the Rb in the rock is located in the glassy mesostasis (Table 2), and the big difference in <sup>87</sup>Rb/<sup>86</sup>Sr between this phase and the plagioclase produces a precise result. The age of the group 2 rocks is comparatively poorly defined as there is much less spread in <sup>87</sup>Rb/ <sup>86</sup>Sr between the mineral concentrates. Both groups were included in the seven specimens dated by the preliminary examination team as  $3.0 \pm 0.7$ billion years (3), which also implies a single value of age for both.

Equality in initial <sup>87</sup>Sr/<sup>86</sup>Sr for both groups is consistent with their cogenesis. In general, it now seems likely that the lava tie line of Fig. 1 does have time significance and that both groups have the same age and the same source region. Combining all rock and mineral data on this model, we obtain 3.81  $\pm$  0.07 billion years as our best present estimate for this age, with initial <sup>87</sup>Sr/  $^{86}$ Sr of 0.69936  $\pm$  0.00008.

The data for the soil and breccias (Fig. 1) are displaced from the rock isochron toward lower 87Rb/86Sr. This might be due to loss of a few percent of original Rb during the production of glass in the soil or to the presence of a transferred Sr component having a higher <sup>87</sup>Sr/<sup>86</sup>Sr.

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## Rubidium-Strontium Age and Elemental and Isotopic Abundances of Some Trace Elements in Lunar Samples

Abstract. Data on six lunar crystalline rocks give an apparent Rb-Sr isochron age of  $4.42 \pm 0.24 \times 10^9$  years (95 percent confidence limits) and initial  ${}^{87}Sr/{}^{86}Sr$ ratio similar to that in a basaltic achondrite. Relationships between K, Rb, Sr, and Ba and depletion of Eu in these samples point to plagioclase separation from the melts that produced these rocks. The abundance of 157Gd in the three lunar samples is similar to terrestrial abundance within < 0.2 percent, thus setting a limit of  $< 6 \times 10^{15}$  neutrons per square centimeter for the integrated thermal neutron flux difference between lunar and terrestrial materials.

This paper deals with the abundance and distribution of K, Rb, Sr, and Ba and the isotopic composition of strontium and the rare earth elements Sm, Eu, and Gd in Apollo 11 lunar samples. Six crystalline rocks of types A and B (1), a breccia, and the lunar soil sample were analyzed for K, Rb, Sr, and Ba abundances by stable isotope dilution methods. We measured the isotopic composition of Sr to a precision of 0.6 to 2.6 parts in 10<sup>4</sup> by the use of a digital data acquisition system in conjunction with programmed magnetic-field switching with a field-sensitive device in a 60° solid source mass spectrometer of 30.5cm radius. Repeat analyses of two lunar samples and multiple analyses of the Eimer and Amend standard reagent (Table 1) show that a reproducibility of about 2 parts in 10<sup>4</sup> has been achieved. The abundances of the rare earth elements were determined by neutron activation analyses, and their isotopic composition was determined in a manner similar to that used in Sr-isotopic analyses.

The data for the abundances of K,

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Table 1. Elemental abundances of K, Rb, Sr, and Ba and the <sup>sr</sup>Sr/<sup>se</sup>Sr ratios in Apollo 11 lunar samples.

Lunar sample	K (µg/g)	Rb (µg∕g)	Sr (µg/g)	Ba (µg∕g)	K/Ba	K/Sr	Ba/Sr	$^{87}$ Rb/ $^{86}$ Sr (× 10 <sup>2</sup> )	<sup>87</sup> Sr/ <sup>86</sup> Sr*	$(\stackrel{\Delta^{\dagger}}{\times} 10^{4})$
10045,23(B)	424	0.823	109	69.4	6.1	3.9	0.63	2.17	0.69905	0.92
10044,21(A)	816	1.15	224	128	6.4	3.6	0.59	1.48	0.69876	0.56
10058,29(B)	853	1.23	201	124	6.9	4.1	0.59	1.72	0.69911	0.69
10084,38(D)	1020	2.88	162	162	6.3	6.3	1.00	5.13	0.70093	0.85
10065,19(C)	1406	3.69	168	200	7.0	8.4	1.25	6.39	0.70212	0.77
10017,11(B)	2207	5.33	169	290	7.6	13.1	1.67	9.18	0.70387	1.6
10022,30(A)	2289	5.57	163	277	8.3	14.0	1.67	9.90	0.70404	0.65
10069,22(A)	2295	5.60	165	277	8.3	13.9	1.67	9.84	0.70397	2.6
				Nuevo	) Laredo					
	433	0.437	85.1			5.1		1.49	0.69882	1.8
			Eim	er and Ame	end reagen	t SrCO <sub>3</sub>				
					0	5			0.70734	2.1

 $\dagger \Delta = 2 \times \sum_{i=1}^{n} (\overline{X}_{i} - m)^{2}$ 

scans each, and  $(\overline{X}_{i-m})$  is the difference between the mean value of the *i*th set and the mean of all sets.

Rb, Sr, and Ba in eight lunar samples are shown in Table 1. There is excellent coherence between data for K, Ba, Sr, and, to a lesser extent, Rb. The ratios K/Ba, K/Sr, and Ba/Sr increase systematically with increase in K content. The K/Rb ratios range from 350 to 700; although no pronounced trends are seen, there is a suggestion of a decrease of the K/Rb ratio with increase in K content.

The trends shown by the various trace element ratios in the lunar samples could be due to mixing relationships between two end members. The two end members themselves (type A and type B rocks) can be products of fractionation processes involving liquid-solid equilibria from an original source material. Until data on the major mineral phases of lunar rocks are available, the choice between fractional crystallization and partial melting seems equivocal. However, we suggest that the pronounced increase in the K/Sr ratio with the increase in K content is suggestive of trends produced by fractional crystallization of plagioclase from the melts. Data on the distribution coefficients for K and Sr between liquid and basaltic plagioclase (2) show that plagioclase concentrates Sr relative to K and to a lesser extent relative to Ba, whereas the liquid tends to be enriched in K relative to plagioclase. Thus, a simple mechanism that can account for the K/Sr and Ba/Sr trends in lunar rocks is the separation of significant amounts of plagioclase from the melts which formed these rocks. Model calculations along these lines will be reported later. Plagioclase separation is also suggested by the pronounced Eu depletion observed in the lunar rocks as discussed below.

If plagioclase separation is the cause of the above trace element trends, the original chemical composition of the lunar melts would be richer in Ca and Al than was the case in the samples analyzed. The major elemental composition of such melts would approach that of gabbroic anorthosite as suggested by Olsen (3).

The Rb-Sr isotopic data for the lunar samples are shown in Table 1. The total range observed in the 87Sr/86Sr ratio is 0.8 percent. From these data, we have attempted to determine the solidification ages of the lunar rocks. The data have been grouped in different ways on the basis of assumptions regarding their genesis and internal relationships and checked for the requirements of isochronism, that is, a uniform initial <sup>87</sup>Sr/<sup>86</sup>Sr

ratio and chemical closure for the same time. If these assumptions are satisfied, we should obtain a linear relationship between the present values of <sup>87</sup>Sr/<sup>86</sup>Sr and <sup>87</sup>Rb/<sup>86</sup>Sr ratios. From the best-fit lines corresponding to various groupings, both the initial <sup>87</sup>Sr/<sup>86</sup>Sr ratio and the age can be obtained.

The initial <sup>87</sup>Sr/<sup>86</sup>Sr values, the slopes of the best-fit lines, and the ages thus obtained for various groups of lunar rocks are shown in Table 2. Although there is no a priori reason to group the data, this was deliberately done to search for any differences between the initial Sr-isotopic compositions and ages of the various types of rocks.

All the lunar samples studied here can



Fig. 1. Strontium evolution diagram for Apollo 11 lunar samples. The straight line represents the least-squares fit of all the lunar sample data points. The data point for the basaltic achondrite Nuevo Laredo falls on the lunar sample regression line (see Table 2).

Table 2. Values of initial  ${}^{s_1}Sr/{}^{s_0}Sr$  ratios and ages for various groupings of lunar samples. The errors shown are 1 standard deviation.

Group of samples	( <sup>87</sup> Sr/ <sup>86</sup> Sr) <sub>i</sub>	Slope	Age $(\times 10^{\circ} \text{ years})$	
Eight samples (all samples received in this study)	$0.69784 \pm 0.00012$	$0.06356 \pm 0.0018$	$4.43 \pm 0.13$	
Eight lunar samples and Nuevo Laredo	$0.69785 \pm 0.00010$	$0.06346 \pm 0.0016$	$4.43 \pm 0.11$	
Six crystalline rocks of types A and B	$0.69785 \pm 0.00012$	$0.06330 \pm 0.0018$	$4.42\pm0.12$	
Seven rock samples of types A, B, and C	$0.69788 \pm 0.00014$	$0.06350 \pm 0.0021$	$4.43\pm0.14$	
Three rock samples of type A	0.69783 ± 0.00023	$0.06252 \pm 0.0003$	$4.36\pm0.02$	
Three rock samples of type B	$0.69780 \pm 0.00023$	$0.06598 \pm 0.0043$	$4.60\pm0.29$	
Five samples of types B, C, and D	$0.69776 \pm 0.00019$	$0.06623 \pm 0.0034$	$4.61\pm0.23$	

fit a linear relationship on the Sr-evolution diagram (Fig. 1) with an age of  $4.43 \pm 0.13 \times 10^9$  years and an initial <sup>87</sup>Sr/<sup>86</sup>Sr ratio [(<sup>87</sup>Sr/<sup>86</sup>Sr)<sub>i</sub>] of 0.69784  $\pm$  0.00012 (Table 2). If the breccia and lunar soil are excluded on the grounds that they may have had a more complex chemical history, the data for six crystalline rocks of types A and B yield a similar age and a value for (87Sr/ <sup>86</sup>Sr)<sub>i</sub> that is virtually identical to that obtained for all samples. Higher age values of 4.61  $\pm$  0.23  $\times$  10<sup>9</sup> years are obtained for groups that include type B samples, the breccia, and the dust, but the errors, both in the age and the value of (87Sr/86Sr), become much larger, probably implying that the conditions for isochron are not adequately met.

The initial <sup>87</sup>Sr/<sup>86</sup>Sr values for the lunar samples reported here are lower by about 0.07 to 0.1 percent than values based upon initial <sup>87</sup>Sr/<sup>86</sup>Sr values from basaltic achondrites (4). This appears to be due to differences in instrumental bias between different laboratories. Our measurements on the achondrite Nuevo Laredo and on the Eimer and Amend reagent SrCO<sub>2</sub> are shown in Table 1. The value of the initial <sup>87</sup>Sr/<sup>86</sup>Sr ratio obtained for Nuevo Laredo, after correction for a growth of  $4.5 \times 10^9$  years, is 0.69786 and is identical with the initial value obtained for all the lunar samples, or the six crystalline rocks. The point described by Nuevo Laredo in the Srevolution diagram is located on the lunar data best-fit line (Fig. 1).

The precision of the present Srisotopic analyses permits an evaluation of the time interval between the formation of the lunar rocks and that of the achondritic meteorites, on the basis of certain assumptions concerning the Rb/ Sr ratio of the reservoir from which these objects were made. For example, for a reservoir Rb/Sr ratio similar to that of chondrites ( $\sim 0.25$ ), the present data suggest that the achondrites and the lunar rocks are coeval within about  $1.5 \times 10^7$  years. On the other hand, if achondrites originate from the surface of the moon, with a Rb/Sr ratio of  $\sim$ 0.02, the time interval corresponds to no more than  $1.8 \times 10^8$  years.

The data on the Rb-Sr age indicate that melting processes on the lunar surface date back to the early history of the solar system. By whatever mechanism the earth acquired the moon, by fission from the earth or by capture, this process must have taken place in the earliest history of the earth, probably contemporaneously with the terminal stages of accretion of the earth.

The elemental and isotopic abundance ratios for Sm, Eu, and Gd in two lunar crystalline rocks and in the soil sample are shown in Table 3. Because of the large thermal neutron capture cross sections of <sup>151</sup>Eu, <sup>153</sup>Eu, <sup>149</sup>Sm, and, in particular, <sup>157</sup>Gd (254,000 barns) (5),

the abundances of these isotopes in lunar samples serve as sensitive indicators of the integrated thermal neutron flux to which these samples were exposed (6). Data (Table 3) show that the <sup>157</sup>Gd/ <sup>158</sup>Gd ratios in the lunar samples are similar to the values for terrestrial rocks to within 0.2 percent. These results set an upper limit of  $6 \times 10^{15}$  neutrons per square centimeter for the differential integrated thermal neutron flux between terrestrial and lunar materials. In models that postulate a primordial irradiation of only a fraction of the solar system material and subsequent mixing with unirradiated material before accretion of planetary bodies, the present data show that the lunar material and the earth contain the same proportions of irradiated and unirradiated material to within 1 to 2 percent, if originally 5 to 10 percent of the solar system material was irradiated (7).

The inferred differential flux for the lunar materials can be discussed in terms of secondary neutrons produced in the lunar material by cosmic ray interactions. The lunar neutron flux has been estimated to be about 18 neutron  $cm^{-2}$  $sec^{-1}$  with a surface leakage of 30 percent for basaltic composition with a H/Si atom ratio of 0.22 (8). The lunar rocks are entirely devoid of hydrous minerals (1), and probably a lower H/Si ratio is appropriate, which would lead to higher leakage and a lower thermalto-nonthermal flux ratio. The flux limit inferred from the Gd data on the lunar soil is compatible with an exposure age of  $\sim 10^8$  years. We conclude that the flux limit estimated from the data is more in keeping with secondary neutron flux on the lunar surface; there seems to be no reason to call on differential primordial irradiation effects.

The Sm/Eu and Gd/Eu elemental ratios in the lunar samples analyzed (Table 3) are distinctly higher than those observed in chondrites and Ca-rich achondrites (9), thus indicating that the Eu is depleted in these samples. In terrestrial rocks, Eu depletion is due to fractionation effects produced by plagioclase which tends to concentrate Eu relative to Sm and Gd (9). The trace element trends discussed earlier and the Eu depletion in lunar samples indicate that a significant amount of plagioclase has separated from the melts that produced the analyzed lunar rocks. Therefore, we suggest that there will be a direct correlation between the K content and the magnitude of the Eu depletion anomaly, and that rocks rich in

Table 3. Elemental and isotopic abundance ratios of Sm, Eu, and Gd. Errors are 1 standard deviation of the mean.

Sample	nple Sm/Eu Gd/Eu		<sup>151</sup> Eu/ <sup>153</sup> Eu	<sup>149</sup> Sm/ <sup>148</sup> Sm*	<sup>157</sup> Gd/ <sup>158</sup> Gd <sup>†</sup>	
Reagent			$0.9147 \pm 0.0013$	$1.232\pm0.001$	$0.6307 \pm 0.0002$	
10069,22(A)	7.4	13.7	$0.9144 \pm 0.0027$	$1.235\pm0.002$	$0.6306 \pm 0.0003$	
10045,23(B)	4.5	5.48	$0.9147 \pm 0.0007$	$1.235\pm0.001$	$0.6304 \pm 0.0004$	
10084,38(D)	5.2	10.9	$0.9127 \pm 0.0007$	$1.231\pm0.001$	$0.6299 \pm 0.0007$	

\* <sup>149</sup>Sm/<sup>148</sup>Sm normalized to <sup>152</sup>Sm/<sup>154</sup>Sm = 1.1770.  $^{+157}$ Gd/<sup>158</sup>Gd normalized to <sup>150</sup>Gd/<sup>160</sup>Gd = 0.9361.

plagioclase (gabbroic anorthosite and anorthosite) may be found on the moon in future Apollo missions.

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## Age Determinations and Isotopic Abundance Measurements on Lunar Samples

Abstract. A K-Ar age of  $2300 \times 10^6$  years has been determined for a sample of type A crystalline rock (57,34). The presence of an anomalously large quantity of  ${}^{40}Ar$ , in a sample of type C breccia (65,35) precluded the calculation of its K-Ar age. Both of the rock types are characterized by low Rb/Sr ratios and consequently low  ${}^{87}Sr/{}^{86}Sr$  values. The U-Th-Pb results for a sample of type D fines (84,33) yield a  ${}^{207}Pb/{}^{206}Pb$  age of 4760  $\times$  10<sup>6</sup> years, but ages based on U-Pb and Th-Pb ratios are anomalously high. Isotopic compositions of Li, K, Rb, Sr, U, and Th are very close to the accepted values for terrestrial materials.

Isotopic determinations were undertaken for several geochronologically important elements found in three types (A, C, and D) of lunar material acquired during the flight of Apollo 11. Two additional samples are currently being studied, and the results will be reported elsewhere. The object of the study was (i) to precisely determine the concentrations of certain parent-daughter elemental pairs; (ii) to measure isotopic abundances for comparison with values for terrestrial elements; and (iii) to determine the geological age of the specimens.

Our routine experimental procedures were slightly modified to facilitate the measurement of isotopic abundances in addition to the determination of elemental concentrations. Isotope dilution techniques were exclusively used in determining the latter. Analyses of Li, K, Rb, Sr, Pb, U, and Th were carried out on two (one 15-cm radius and one 25cm radius) 90-degree deflection, solidsource mass spectrometers, both of which are equipped with triple-filament source assemblies and electron multiplier ion detection systems. A modified M.S.-10 mass spectrometer (Associated Electrical Industries), operated in the static

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mode, was used for all argon analyses. All chemical procedures and vacuum fusions were monitored for contamination through the analysis of "blanks." Contamination corrections applied for Rb, Sr, and Ar were < 0.5 percent; corrections for U and Th amounted to about 1 percent, as did those for K, with the exception of one extraction of the latter which required a 2 percent correction; a 10 percent correction was required for terrestrial Pb contamination. Our determinations of the concentrations of Rb and Sr in the Cal. Tech.



Fig. 1. Concordia diagram; U-Pb data for type D fines.

standard solutions supplied with the lunar samples agree with the stated values to within 0.25 percent.

Argon was extracted from two samples of type A crystalline rock (57,34) under slightly differing experimental conditions. In the first instance (extraction No. 2, Table 1) the purified gas was collected in two calibrated volumes having ratios of about 1:2. The smaller volume of gas was equilibrated with <sup>38</sup>Ar tracer on the mass spectrometer inlet line just before isotopic analysis, while the larger portion was used unspiked to determine the relative isotopic abundances. The <sup>36</sup>Ar/<sup>38</sup>Ar ratio determined for the latter was used to correct the results of the spiked analysis. In calculating the age it was assumed that there was no primordial <sup>40</sup>Ar component. In the second instance (extraction No. 3, Table 1) the gas was equilibrated with the <sup>38</sup>Ar tracer in the usual manner during sample fusion. The <sup>36</sup>Ar/<sup>38</sup>Ar ratio measured for the unspiked portion of extraction No. 2 was again used to correct the observed abundances for this sample.

The K measurements were made on chips of material broken from the larger samples fused for Ar extraction and should therefore provide a realistic estimate of the K content of the latter. An examination of the  $^{40}$ Ar and K values (Table 1) reveals that the concentrations of both are slightly different in the two portions of this sample.

The greatest variation however was noted for the <sup>36</sup>Ar content. While both samples yielded essentially the same quantity of <sup>40</sup>Ar, extraction No. 2 contained nearly twice as much <sup>36</sup>Ar as extraction No. 3. In spite of this variation the calculated ages for the two samples are very close at 2300  $\times$  10<sup>6</sup> and 2270  $\times$  10<sup>6</sup> years, respectively.

Two extractions were carried out for a sample (65,35) of type C breccia; the first was equilibrated with the <sup>38</sup>Ar tracer in the routine fashion during fusion, and the second was not mixed with tracer. The <sup>36</sup>Ar/<sup>38</sup>Ar ratio determined for the latter was to be used to correct the results obtained for extraction No. 1. However, this material contained very large quantities of both <sup>36</sup>Ar and <sup>40</sup>Ar, and consequently it has not been possible to calculate a realistic age for the sample. This is in agreement with preliminary data reported for breccia samples (1). The K concentration was again determined on numerous chips from the larger sample used for the Ar extraction in an attempt to ensure the

<sup>4</sup> January 1970