Cosmogenic and Primordial Radionuclides in Lunar Samples by Nondestructive Gamma-Ray Spectrometry

Abstract. *The* ^{7}Be, ^{22}Na, ^{26}Al, ^{44}Ti, ^{46}Sc, ^{48}V, ^{51}Cr, ^{54}Mn, ^{56}Co, ^{57}Co, ^{60}Co, ^{40}K, ^{238}U, *and* ^{232}Th *were measured in lunar fines and portions of three rocks. Major production of cosmogenic radionuclides is due to solar protons, thus their concentrations are far different than those in meteorites. Surface exposures of the rocks and fines are long compared with the 0.74 million year half-life of* ^{26}Al. *Lunar fines show substantially higher concentrations of low energy reaction products. The ratios of thorium to uranium are extremely constant at 3.8, which indicates very little geochemical differentiation and are in good agreement with a common nucleosynthesis for lunar and earth materials.*

The relative and absolute radionuclide concentrations of lunar samples are far different from those observed in meteorites, which reflects substantial differences in the chemical composition of the lunar surface and in the energy spectrum of incident cosmic rays. Whereas meteorites are exposed to a somewhat similar but less intense cosmic ray spectrum, the isotopes produced by solar cosmic rays are largely lost by ablation during entry of the meteorite through the earth's atmosphere. Thus, while the cosmogenic isotopes observed in meteorites result mainly from the high energy galactic component, those on the lunar surface are produced largely from the solar component of the cosmic ray spectrum.

Preliminary gamma ray spectrometric measurements made during sample quarantine at the Lunar Receiving Laboratory provided approximate radionuclide concentrations of lunar samples (*1*). The objectives of the present work were to perform very precise measurements of the radionuclide content of lunar surface rocks and fines and interpret these observations in terms of cosmic ray exposure and the history of the specimens.

All of the radionuclide measurements were made by nondestructive gamma ray spectrometric techniques. The lunar samples were maintained in a dry nitrogen atmosphere in thin containers of stainless steel, or aluminum-lined lucite for the counting measurements. The counting was performed on anticoincidence shielded multidimensional gamma ray spectrometers (MDGRS) (*2*), employing large NaI(Tl) detectors and on a Ge(Li) spectrometer (*3*). The MDGRS which employed either two 28 cm diameter by 15 cm thick NaI (Tl) crystals or two 23 cm by 20 cm thick NaI(Tl) crystals offer a very high sensitivity for the measurement of those radionuclides which decay by emission of several gamma rays or by emission of a positron and a gamma ray [^{22}Na, ^{26}Al, ^{44}Ti, ^{46}Sc, ^{48}V, ^{56}Co, ^{232}Th (^{208}Tl) and ^{238}U(^{214}Bi)]. The anticoincidence shielded Ge(Li) spectrometer which viewed the sample with two 70 cm^{3} detectors provided an improved sensitivity for the measurement of radionuclides which emit only one photon per disintegration (^{7}Be, ^{51}Cr, ^{54}Mn and ^{57}Co) and served to verify the measurements of the more abundant cascade photon emitters (^{22}Na and ^{26}Al). The concentrations of the radionuclides in the lunar samples were based on 10,000 to 20,000 minute count intervals on both types of analyzers. Standard mockups with the same size, shape, physical density, and electron density as each lunar rock and containing known amounts of each radionuclide of interest were prepared from a mixture of plaster of paris, iron powder, and water (*4*). Mockups of the lunar fines were prepared in mixtures of 5 percent Al_2O_3 and 95 percent dunite to provide the same density as the fines. The standard radioisotopes were first pipetted into the Al_2O_3, dried, and after mixing, the labeled Al_2O_3 was homogeneously mixed with the dunite to form the mockup. Other types of mockups prepared in powdered basalt, and dunite with 25 percent Fe were shown to have essentially identical counting efficiencies to those in the dunite (*5*), thus substantiating the adequacy of the mockups.

Inhomogeneous mockups where the activity was all located in the top 25 percent produced only a 5-percent effect on the total counting efficiency but could easily be determined by differences in the ratios of the coincidence photons from ^{22}Na and ^{26}Al which were deposited in the two NaI(Tl) crystals.

The standard radionuclide solutions used in preparing mockups were all calibrated by counting them in a precisely controlled geometry on a Ge(Li) diode for which counting efficiency versus photon energy had been very accurately established (*4*). The radio-

Table 1. Cosmogenic and primordial radionuclides in lunar materials. The ± values are the standard deviations associated with all counting statistics. Rock A(57), sample 10057,30; rock B(17), sample 10017,37; rock B(3), sample 10003,25; fines 1 (84), sample 10084,41; fines 2 (84), sample 10084,113-2; fines 3 (84), sample 10084,113-1. Values for rows 1 (^{7}Be) through 11 (^{60}Co) are expressed as disintegrations per minute per kilogram; row 12 (K) is weight percent; rows 13 (Th) and 14 (U) are ppm; weights of sample and of rock are expressed in grams. Values prefaced with < represent 2σ of the gross counting rate in the photopeak area.

Radionuclides	Rock A(57)	Rock B(17)	Rock B(3)	Fines 1 (84)	Fines 2 (84)	Fines 3 (84)
^{7}Be				< 112		
^{22}Na	43 ± 2	45 ± 2	49 ± 2	64 ± 3	63 ± 2	
^{26}Al	84 ± 2	80 ± 2	75 ± 2	131 ± 4	137 ± 4	
^{44}Ti	< 2					< 1.5
^{46}Sc	11 ± 2	13 ± 2	8 ± 2	11 ± 1		
^{48}V				< 10		
^{51}Cr	< 62			< 63		
^{54}Mn	41 ± 8	46 ± 17	60 ± 7	24 ± 3		
^{56}Co	19 ± 9					53 ± 10
^{57}Co	< 3.9	< 5.0	< 4.3	1.3 ± 1.6		
^{60}Co	< 1	0.8 ± 0.6	< 1	0.3 ± 0.8		
K	0.23 ± 0.02	0.26 ± 0.01	0.046 ± 0.006	0.11 ± 0.01	0.11 ± 0.01	
Th	3.6 ± 0.1	3.2 ± 0.1	1.08 ± 0.05	2.2 ± 0.1	2.3 ± 0.1	
U	0.95 ± 0.05	0.86 ± 0.05	0.29 ± 0.02	0.54 ± 0.03	0.56 ± 0.04	
Sample weight	230	115.5	117.2	999	327.0	270.8
Rock weight	897	971	213*			

* Weight uncertain.

Table 2. Calculated production rates of ^{26}Al from galactic cosmic rays in lunar material. Rates are expressed in disintegrations per minute per kilogram, based on work of Fuse and Anders (*6*). Samples are identified fully in Table 1.

Material	Al	Si	Fe	Ca	Ti	Total		Observed	Calculated percent of observed
						4π	2π		
Fines (84)	32.9	62.0	0.3	0.6	0.1	95.9	48.0	134	36
Rock A (57)	27.6	52.1	0.3	0.5	0.2	80.7	40.4	84	48
Rock B (17)	25.2	58.0	0.3	0.5	0.2	84.2	42.1	80	53
Rock B (3)*							42.1	75	56

* Chemical composition not available but assumed to be the same as for rock 17.

nuclide content of the standard mockups were thus known within ±2 to 3 percent. The instruments used for the lunar sample measurements were calibrated by counting duplicates or triplicates of the mockups to a precision of ± 1 to 2 percent. The radionuclide concentrations in the lunar samples were calculated by direct comparison of their counting rates with those of mockup standards after application of appropriate background and Compton corrections.

A serious problem in the measurements of cosmogenic radionuclides that is not encountered in meteorite measurements resulted from the high concentrations of Th and U in the lunar material. The U and Th which were 10- to 50-fold higher in the lunar material than in meteorites produced a high Compton scatter "background" over the entire coincidence spectrum.

The radionuclide concentrations observed in samples of three lunar rocks and in lunar fines are summarized in Table 1, with standard deviations as low as ± 3 percent for ^{22}Na and ^{26}Al. One of the more striking observations of the measurements is the large differences between ^{22}Na and ^{26}Al concentrations of the rocks and fines (Table 1). This large range was not evident in the LRL preliminary measurements (*1*), perhaps because of the large statistical uncertainties in their measurements. In addition, concentrations of these radionuclides are much higher than one would have predicted from a 2π isotropically incident galactic cosmic ray flux. The expected galactic production of ^{26}Al, after buildup of secondaries, was determined for the chemical compositions (*1*) of the lunar samples (Table 2), with the method of Fuse and Anders (*6*) for stone meteorites. Since the calculated concentrations listed for the 2π isotropic flux include the contribution from secondary buildup, these values should be considered as upper limits, as should the accompanying values for the percent galactic contribution. Extensive measurements in near surface samples from the meteorite Saint Séverin (*7*) provided the first direct proof that ^{22}Na and ^{26}Al in near surface samples are almost twofold lower than in deeper regions of a large meteorite. Thus, the galactic contributions may be as little as one-half that listed in Table 2. The low ^{22}Na and ^{26}Al concentrations in the Denver meteorite (*8*) are apparently due to a lack of secondary flux buildup because of its small size (Table 3). The range in the concentrations of ^{22}Na and ^{26}Al in lunar samples shown in Table 1 is not accounted for by differences in chemical composition and is apparently due to differences in shielding. Unfortunately, documentation of the orientation of the rock on the lunar surface, the relationship of our specimens to the original rocks, and of the sampling depth of the lunar fines is not presently available to help explain the observed concentration differences. The ^{26}Al concentrations in the three rock samples range from 56 to 63 percent of that in the lunar fines while the ^{22}Na concentrations in the three rocks are also low, ranging from 67 to 77 percent of that in the fines. Since both of these radionuclides are produced by low energy reactions, a closer average distance to the surface which the fines apparently occupied could explain their higher concentrations.

The ratios of ^{26}Al to ^{22}Na are two- to threefold higher than those normally present in meteorites. This high ratio is apparently due to a low ^{22}Na production rate. The threefold higher aluminum content of the lunar samples (*1*) than of meteorites (*9*) would favor ^{26}Al production relative to ^{22}Na from the low energy solar protons. Also, Mg which is a major target element for ^{22}Na production is about threefold lower in the lunar samples and would further limit ^{22}Na production. The ^{26}Al to ^{22}Na ratios in lunar samples bear a direct relationship to exposure age and could be employed for estimating the surface exposure age in a manner similar to that used in the age dating of meteorites. However, since the relative production rates of ^{26}Al and ^{22}Na for the low energy solar component is not well known, caution should be exercised in applying this dating technique either with this isotope pair or for other cosmogenic pairs. Regardless

Table 3. Comparison of cosmogenic radionuclides in lunar sample with those in meteorites. Meteorite A_1 is an 11-cm deep sample from the Allende meteorite (*10*); meteorite A_2 is a sample from > 25 cm deep in the Allende meteorite (*10*); meteorite B is the Denver meteorite (*8*) and meteorite C is a near-surface sample from the Saint Séverin meteorite (*7*).

Isotope	Activity (dpm/kg)								Ratio of isotope to ^{22}Na							
	Lunar sample				Meteorite				Lunar sample				Meteorite			
	Rock			Fines	A_1	A_2	B	C	Rock			Fines	A_1	A_2	B	C
	(57)	(17)	(3)	(84)					(57)	(17)	(3)	(84)				
^{22}Na	43	45	49	66	69	81	61	56								
^{26}Al	84	80	75	134	57	61	43	36	1.95	1.78	1.53	2.03	0.83	0.75	0.70	0.64
^{44}Ti	<2			<1.5					<0.05			<0.02				
^{46}Sc	11	13	8	11	6	9			0.26	0.29	0.16	0.17	0.09	0.11		
^{48}V				<10		21						<0.12		0.26		
^{51}Cr	<62			<63		54			<1.9			<0.95		0.67		
^{54}Mn	41	46	60	24	99	129		48	1.0	1.0	1.2	0.36	1.4	1.6		0.86
^{56}Co	19			53				4	0.59			0.80				0.07
^{57}Co	<3.9	<5.0	<4.3	1.3		32		28	<0.12	<0.11	<0.09	0.02		0.40		0.50
^{60}Co	<1	0.8	<1	0.3	41	128	<4	11	<0.03	0.018	<0.02	0.04	0.59	1.6	<0.07	0.20

of any variations in the ^{26}Al and ^{22}Na production rates with energy, depth, solar cycle, and so forth, it is obvious that the lunar rocks and fines have surface exposure ages which are comparable to or much longer than the half-life of ^{26}Al. If one assumed that the production ratios of ^{26}Al : ^{22}Na were the same in the lunar samples, and that the ^{26}Al : ^{22}Na ratio of about two which was observed in both the lunar fines and in rock 57 (Table 3) represents a saturation value, then rocks 17 and 3 would indicate lunar surface exposure ages of 2.2 and 1.5 million years, respectively. However, in view of known differences in ^{26}Al : ^{22}Na production rates with energy it appears more likely that all of the samples are at saturation and that the ratio differences are due to shielding.

The ^{54}Mn concentrations in the lunar samples show an even wider range than ^{22}Na and ^{26}Al and do not appear to show any correlation with them (Tables 1 and 3). The main target element for ^{54}Mn production is iron and the much lower concentrations of iron in the lunar materials than in chondrites helps explain the generally lower ^{54}Mn concentrations. The efficient production of ^{54}Mn in lunar materials requires more energetic protons than the production of either ^{22}Na or ^{26}Al. The lunar fines show a much lower ^{54}Mn concentration than the rocks which is not explained by their somewhat lower iron content. It appears that while the solar protons greatly enhance the production of ^{22}Na and ^{26}Al, they contribute relatively little to ^{54}Mn production; thus one observes the low ^{54}Mn in lunar fines and the higher concentrations in rocks where significant secondary flux buildup may occur.

The titanium content of the lunar samples is about 50 times that in meteorites and one would therefore expect cosmogenic isotopes from this target element to be more prevalent. The ^{46}Sc which is produced from titanium with a cross section of 80 mb at 45 Mev (*5*) was present at concentrations of 8 to 13 disintegrations per minute per kilogram in the lunar rocks and fines. Considering that only a 2π isotropic flux is incident on the lunar surface, these values are high compared with 6 to 9 disintegrations per minute per kilogram in interior samples of the Allende meteorite (*1*) (Table 3) where iron is the principal target element. From ^{46}Sc concentrations in meteorites (*10, 11*), the 2π lunar irradiation effect, and limited secondary flux buildup in the lunar samples, a lunar surface ^{46}Sc concentration of 2 to 4 disintegrations per minute per kilogram would have been expected. The threefold higher ^{46}Sc concentration is attributed mainly to the high titanium content of the samples plus the additional solar protons in a sufficiently high energy range for its effective production. The ^{44}Ti concentrations in rock 57 and in the lunar fines sample were <2 and <1.5 disintegrations per minute per kilogram. The cross section for production of ^{44}Ti from Ti is low (*5*), comparable to that from iron, whereas the threshold is rather high. Therefore, ^{44}Ti concentrations comparable to, or lower than the observed 1.4 and 2.0 disintegrations per minute per kilogram in the Harleton and Bruderheim meteorites (*12*) would be expected.

The isotopes ^{56}Co and ^{48}V are both produced by low energy (p,n) reactions on Fe and Ti, respectively. They have almost identical excitation functions (*5*) and are thus excellent indicators of the solar particle flux intensity. Cobalt-56 concentrations of 53 and 19 disintegrations per minute per kilogram in the lunar fines and rock 57, respectively, were observed. These concentrations are more than ten times what one would predict from the reported 4 disintegrations per minute per kilogram in meteorites (*7*), indicating that >90 percent of the ^{56}Co originates from solar proton reactions. Because of the short half-life of ^{48}V (16.1 days) and the 2- to 3-month delay in sample receipt only a less than value, <10 disintegrations per minute per kilogram, in lunar fines could be measured. Based on the nearly identical nature of the excitation functions for ^{56}Co and ^{48}V formation (*5*), we would predict a ^{48}V concentration at the time of sampling of <8 disintegrations per minute per kilogram.

The ^{57}Co concentrations in the lunar samples are about tenfold lower than in meteorites. The principal target elements Fe and Ni are about two- and tenfold lower, respectively, in lunar material and this is evidently responsible for the very low ^{57}Co values.

The very low ^{60}Co concentrations (Table 1) in the lunar materials are to be expected from the low content of stable cobalt of the lunar surface which is about thirtyfold lower than that in meteorites. Only less than values were obtained for ^{7}Be (53 days) and ^{51}Cr (28 days). Beryllium-7 is a high energy reaction product and has not yet been observed in meteorites. Chromium-51 had decayed by about fivefold at the time of sample receipt and was thus well below its expected and measurable concentration.

As indicated by the very high concentration of low energy products such as ^{56}Co and ^{26}Al, the solar proton component contributes heavily to their production, and a depth profile measurement of such radionuclides would provide a basis for determining surface exposure age, accretion rate, and long and short term variations in cosmic ray flux.

Interest in the K, U, and Th content of the moon lies in its radiogenic heating together with its history in relationship to that of the earth, meteorites, tektites, and other solar system bodies. Our measurements of the natural radioactivity due to K, U, and Th are in general agreement with preliminary measurements (*1*), but they provided smaller error limits and showed a much narrower range in Th : U ratios. Our measurements extend the K : Th weight ratio to cover a range of 1600 to 3000 and reduce the Th : U weight ratio range to less than 10 percent. The lunar sample Th : U ratios of 3.8 ± 0.2 are in good agreement with the calculated ^{232}Th : ^{238}U value of 3.8 to 0.3 (*13*) for the present-day solar system and with the ratios in chondrites (*9*), and terrestrial crustal rocks (*14*). These constant Th : U ratios, which would be expected from their very similar ionic radii and identical outer electron configurations, add strong support for cessation of nucleosynthesis of lunar matter at the same time as that of the earth's and meteorite's.

The K, U, and Th concentrations of the lunar materials are considerably different than those observed in other extraterrestrial and major terrestrial materials. Compared with stony meteorites (*9*), the U and Th in lunar samples are some tenfold higher while the K ranges from comparable to fivefold higher concentrations. The lunar material has U and Th concentrations that are similar to some tektites (*15*), but its K content is about tenfold lower. Compared with the average crustal composition of the earth, the lunar materials are about two- to fourfold lower in U and Th and tenfold lower in K. Of the major terrestrial materials the lunar samples most closely resemble basalt (*11*) with similar U and Th concentrations but about a fourfold lower K content. The lunar fines represent somewhat of an integrated sample, that reflects major lunar surface processes, and since their primordial radionuclide concentrations are similar to those of the lunar rocks, the total meteorite contribution to the lunar fines must not have been large enough to significantly alter its composition. The lunar materials thus appear rather unique in their primordial radionuclide composition, which indicates a substantially different geochemical history than that of known earth and other extraterrestrial materials.

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Elemental Compositions and Ages of Lunar Samples by Nondestructive Gamma-Ray Spectrometry

Abstract. *A gamma-ray spectrometry system with low background was used to determine the radioactivity of crystalline rocks, breccias, and fine material. Nuclides identified were* ^{40}K, ^{232}Th, ^{238}U, ^{7}Be, ^{22}Na, ^{26}Al, ^{44}Ti, ^{46}Sc, ^{48}V, ^{52}Mn, ^{54}Mn, *and* ^{56}Co. *Concentrations of K, Th, and U ranged between 480 and 2550, 1.01 and 3.30, and 0.26 and 0.83 parts per million, respectively. Concentrations of thorium and uranium were those of terrestrial basalts, while the potassium concentrations were near values for chondrites. Products of low-energy nuclear reactions showed pronounced concentration gradients at rock surfaces. Concentrations of K and of* ^{22}Na *determined here were combined with concentrations of rare gases to estimate gas-retention ages and cosmic-ray exposure ages with ranges of 2200 to 3200 and 34 to 340 million years, respectively, for three rocks.*

Lunar samples from Apollo 11 were analyzed by nondestructive, gamma-ray spectrometry at the Lunar Receiving Laboratory (LRL), Houston, Texas (*1*). The preliminary data have now been refined and additional samples also have been analyzed.

The low-background, gamma-ray spectrometer used in these studies has been described (*1*). Because the samples were viewed by two detectors, gamma-ray spectrums could be recorded either as noncoincident, or singles, data if an event occurred in one detector only, or as a gamma-gamma coincidence event if a signal was produced in both detectors simultaneously. The data acquisition system included a coincidence-anticoincidence logic circuit and dual 12-bit analog-to-digital converters interfaced to an on-line, digital computer. The computer was programmed to record singles data from each detector at a resolution of 256 channels, gamma-gamma coincidence data in a folded matrix with a resolution of 127 by 127 channels but requiring only 8192 storage locations, and a sum-coincidence spectrum of 256 channels. All of these spectrums were used in the detailed analysis of each sample.

It was necessary to use a variety of sample containers (three types of stainless steel and two types of polypropylene) with different attenuation and scattering properties for gamma rays. In addition, variations in radioactive contamination caused uncertainties in the background corrections. The preliminary examination was made especially difficult because it was not known in advance of the mission that some of these containers were to become necessary. Spectrum libraries for calibration in some cases had to be acquired during the active mission period, which reduced the time available for measuring lunar samples. After the sample quarantine was lifted, a number of small samples were measured which had been exposed to atmosphere, and so did not require special containers.

Before receipt of lunar samples, response functions of 19 expected nuclides were collected. Extended sources of electrolytically reduced iron powder containing 10^4 to 10^5 disintegrations per minute of each nuclide were prepared, measured, and compiled into a library of spectrums for the preliminary examination.

Potassium, thorium, and uranium standards were calibrated by weighing. Potassium standards were prepared from reagent-grade potassium chloride, and for some sources from potassium enriched in ^{40}K. Certified standards of thorium and uranium were obtained from the U.S. Atomic Energy Commission, New Brunswick Laboratory. All other radionuclides were standardized by absolute gamma-ray spectrometry to an overall uncertainty of less than 5 percent.

Final calibrations were performed through use of replicas filled with radionuclide standards which reproduced closely the bulk and electronic densities of the lunar samples. The replicas consisted of 0.08-mm aluminum-foil shells hand-formed around a plaster cast of each lunar sample, reinforced with epoxy cement and filled with iron powder. Electronic densities were calculated from the gross chemical composition (*1*) and matched in the replicated standards by use of a mass of iron powder about 5 percent in excess of the lunar sample mass. The bulk density was reproduced by the addition of 3-mm polyurethane spheres (0.016 g/cm^3). Such exact replicas were prepared for the four crystalline rocks measured. Lunar fines were simulated with mixtures of powdered iron and dunite. Nine replicas of each lunar sample were filled with a known portion of each radionuclide found in the preliminary examination. A spectrum library was collected for each set of replicas and used for final analysis of the five samples. The breccia samples were analyzed with cyclindrical standards used in the preliminary examination.

The concentration of each nuclide