

## K/Ar dating of lunar soils II

E. CALVIN ALEXANDER, JR.,<sup>a</sup> A. BATES,<sup>a</sup> M. R. COSCIO, JR.,<sup>a</sup>  
 J. C. DRAGON,<sup>b</sup> V. R. MURTHY,<sup>a</sup> R. O. PEPIN,<sup>b,c</sup> and  
 T. R. VENKATESAN<sup>b,d</sup>

University of Minnesota, Minneapolis, MN 55455

**Abstract**—Correlation techniques are applied to rare gas abundance and isotopic measurements and measured target element concentrations in grain-sized separates of 12033, 15531, 67701, 71501 and 75081 to deduce K/Ar ages, exposure ages, and isotopic compositions of surface correlated argon components. <sup>40</sup>Ar-<sup>39</sup>Ar stepwise heating experiments are applied to 12033 and two samples of 75081 to define situations in which <sup>40</sup>Ar-<sup>39</sup>Ar techniques are applicable to lunar soils. A comparison of <sup>40</sup>Ar/<sup>36</sup>Ar<sub>sc</sub> vs. <sup>40</sup>K/<sup>36</sup>Ar<sub>sc</sub> with <sup>40</sup>Ar vs. <sup>36</sup>Ar and <sup>40</sup>Ar/<sup>36</sup>Ar<sub>sc</sub> vs. 1/<sup>36</sup>Ar<sub>sc</sub> correlation techniques indicates that the latter two techniques can produce spurious ages and that their use should be abandoned. The results of <sup>40</sup>Ar-<sup>39</sup>Ar stepwise heating experiments of submature to mature soils are dominated by fractionations of the surface correlated component(s) and do not yield chronologically significant data. <sup>40</sup>Ar-<sup>39</sup>Ar stepwise heating experiments, however, can yield chronologically significant information from immature soils such as 12033.

The KREEP component in 12033 yields an age of 800<sup>+400</sup><sub>-50</sub> m.y. in agreement with Eberhardt *et al.*'s (1973a) results. To the extent that 12033 KREEP is Copernicus ejecta, the age of 12033 dates the Copernicus impact. Results from dark mantle soils 75081 and 71501 indicate old ages indistinguishable from the ages of the orange glass soil 74220. Data from 67701 from the rim of North Ray Crater indicate that the K/Ar clock in the rim material was not reset by the impact.

### INTRODUCTION

FOLLOWING THE PIONEERING WORK of Turner (1970), the <sup>40</sup>Ar-<sup>39</sup>Ar technique of K/Ar dating has proven to be one of the most versatile tools in lunar chronology. Although the recoil of <sup>39</sup>Ar can produce anomalous ages in some cases (Turner and Cadogan, 1974; Huneke and Smith, 1976), many workers now routinely date the >1 cm rock samples returned from the lunar surface using this technique. Early attempts at conventional whole rock K/Ar dating (cf. Funkhouser *et al.*, 1970) ultimately proved unsuccessful because most of the rocks had lost varying amounts of <sup>40</sup>Ar. Conventional K/Ar age determinations on mineral separates have proven useful in some cases, however (cf. Eberhardt *et al.*, 1971; Murthy *et al.*, 1972). <sup>40</sup>Ar-<sup>39</sup>Ar analyses of fragments from the coarse fines, <1 cm but >1 mm, have shown that much of this material is dateable. When combined with petrographic and chemical studies on the same fragments, the age data have identified samples

<sup>a</sup>Department of Geology and Geophysics.

<sup>b</sup>Department of Physics.

<sup>c</sup>Present address: Lunar Science Institute, 3303 NASA Rd 1, Houston, Texas 77058.

<sup>d</sup>Present address: Physical Research Lab, Navrangpura, Ahmedabad 380 009, India.

both older and younger than those previously recognized in the collection of large rocks (Schaeffer and Husain, 1973; Eberhardt *et al.*, 1976).

The class of lunar material which has proven most resistant to routine K/Ar dating is the <1 mm soils. There is no generally accepted method of dating these materials.  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  techniques have proven moderately successful in the case of some of the unique soils such as the KREEP rich soil 12033 (Eberhardt *et al.*, 1973a) and the orange glass soil 74220 (Husain and Schaeffer, 1973; Huneke *et al.*, 1973; Eberhardt *et al.*, 1973b), but have failed to yield useful chronological information for the more mature lunar soils (cf. Huneke *et al.*'s (1973) analysis of 74241). A variety of more-or-less conventional K/Ar techniques have been used to analyze lunar soils but no technique or techniques have been generally accepted as yielding reliable ages. This work is an attempt to identify those techniques which extract the most reliable chronologic information from lunar soils and to define the situations in which the best data are obtainable.

The "age" of a lunar soil is, in general, a much more nebulous concept than the age of a rock or a coarse fines fragment. The history of a rock on the lunar surface may be complicated but it is the same for all of the parts of that rock, at least since the rock was assembled. It has been demonstrated, however, that the different minerals in a given soil can have experienced different histories, (cf. the analyses of mineral separates from 10084 by Eberhardt *et al.* (1970) and Basford (1974)), and this difference probably extends to the individual grains. Until analytical techniques capable of measuring the ages of individual submillimeter particles are developed, the only quantity that is even potentially measurable is an "average age" for a soil or some physically separable component within the soil. For the rest of this paper the term age will mean the average Ar retention age of a number of particles except where explicitly noted otherwise.

Conventional K/Ar ages are always subject to uncertainties due to the presence of excess  $^{40}\text{Ar}$  and/or to  $^{40}\text{Ar}$  loss. An "age" is actually an analytically determined  $^{40}\text{Ar}/^{40}\text{K}$  ratio. The degree of correspondence between such a  $^{40}\text{Ar}/^{40}\text{K}$  ratio and any physically significant event in the history of the material being dated can be decided only by comparisons to external information such as ages from other isotopic systematics, chemical studies, etc. This is particularly true for K/Ar data from lunar soils.

The determination of a K/Ar age for a lunar soil is further complicated by the presence of nonradiogenic argon in the soil in significant quantities. The non-radiogenic argon component is usually dominated by a surface correlated argon component derived from the solar wind and lunar atmosphere. The surface correlated component varies in isotopic composition from soil to soil, from mineral to mineral within a soil and probably from grain to grain of a given mineral. The nonradiogenic component also contains a volume correlated spallogenic argon component produced by various particle irradiations during the near surface history of the soil.

In order to measure the K/Ar age of a lunar soil it is necessary to separate the various argon components. This separation is usually accomplished by measuring the concentration of argon, potassium and calcium (and if necessary other target

elements) in grain-sized separates of the lunar soil. Correlation systematics for a two-component structure of argon have been developed (Eberhardt *et al.*, 1970; Pepin *et al.*, 1974) which allow the decomposition of the argon spectrum if certain assumptions are met.

## EXPERIMENTAL PROCEDURE AND RESULTS

Grain-sized separates of samples 12033,42; 15531,42; 67701,22; 71501,27 and 75081,17 were prepared by sieving in acetone. Grain size intervals and masses of the aliquots used for rare gas analyses are given in Table 1. The rare gas analyses reported in Table 1 were made on two different systems. The 6" double focusing mass spectrometer, gas extraction system, standards and data reduction system used to analyze samples 12033,42, 15531,42, and 67701,22 have been described by Pepin *et al.* (1975). The 6" single focusing mass spectrometer, gas extraction system, standards and data reduction system used to analyze samples 71501,27 and 75081,17 are a different system and are described by Venkatesan (1976). The K and Ca concentrations reported in Table 1 were determined by isotope dilution as described by Evensen *et al.* (1973).

Unsieved aliquots of 12033,42 and 75081,17 and an aliquot of the 37–74  $\mu$  grain-sized fraction of 75081,17 were irradiated for  $^{40}\text{Ar}$ – $^{39}\text{Ar}$  analysis in the U.S.G.S. TRIGA reactor in Denver. Details of the irradiation, designated MIDT # 1, are available in Venkatesan (1976). The neutron fluence was monitored using the St. Severin standard described by Alexander and Davis (1974). Tables 2 and 3 give summaries of the results from step-wise heating experiments on the irradiated samples. Complete isotopic data for these samples are available in Venkatesan (1976).

An experimental problem affecting the analyses of the two irradiated samples of 75081 and, to a much lesser degree, the analysis of irradiated 12033 was discovered during data reduction, after the analyses were completed. A small contamination at mass 37 apparently occurred during the analyses of the samples. Since this contamination was not present in the blanks associated with the analyses, we believe the problem was due to the formation of small amounts of species like  $^1\text{H}^{36}\text{Ar}^+$  in the spectrometer. Due to a series of equipment failures, the samples were not analyzed until ~14 months after the neutron irradiation. The resulting large decay correction, ~4000, multiplied the otherwise negligible contamination at mass 37 to significant proportions. The  $^{37}\text{Ar}$  content for each temperature fraction was therefore calculated by using literature values for the Ca contents of the samples to calculate the total amount of  $^{37}\text{Ar}$  which should have been measured and then apportioning that amount of  $^{37}\text{Ar}$  among the temperature fractions according to the fractional release of spallogenic  $^{38}\text{Ar}$  by the samples. Details of this calculation are given in Venkatesan (1976).

## DISCUSSION

### 15531

*Exposure Age.* Pepin *et al.* (1974) have shown that the key correlation for determining both spallation gas concentrations and trapped gas isotopic compositions in grain sized samples of lunar fines are ordinate intercept relationships which in the case of argon have the form:

$$^{38}\text{Ar}/^{36}\text{Ar} = \frac{[\text{C}]}{^{36}\text{Ar}} \left[ \frac{^{36}\text{Ar}_{\text{vc}}}{[\text{C}]} \left\{ \left( \frac{^{38}\text{Ar}}{^{36}\text{Ar}} \right)_{\text{vc}} - \left( \frac{^{38}\text{Ar}}{^{36}\text{Ar}} \right)_{\text{sc}} \right\} \right] + \left( \frac{^{38}\text{Ar}}{^{36}\text{Ar}} \right)_{\text{sc}} \quad (1)$$

where vc and sc are the volume correlated (spallogenic) and surface correlated components respectively, and [C] the chemical concentration(s) of target elements from which volume-correlated gases are produced *in situ*. If the measured quantities  $^{38}\text{Ar}/^{36}\text{Ar}$  and  $[\text{C}]/^{36}\text{Ar}$  are plotted against each other for a suite of size

Table 1. Argon, potassium and calcium in grain-sized separates from lunar fines.

Sample	Mass <sup>a</sup> (mg)	<sup>40</sup> Ar (units of 10 <sup>-5</sup> cm <sup>3</sup> /g)	K (ppm)	Ca (%)	<sup>40</sup> Ar/ <sup>36</sup> Ar	<sup>38</sup> Ar/ <sup>36</sup> Ar
<i>12033,42</i>						
250–1000 μ	41.44	2.772 ±0.069	3782 ±76	8.88 ±0.18	4.556 ±0.033	0.2237 ±0.0006
74–250 μ	40.09	2.446 ±0.064	2757 ±56	7.69 ±0.15	3.126 ±0.026	0.2220 ±0.0006
37–74 μ	41.76	2.288 ±0.059	2584 ±52	8.16 ±0.16	1.806 ±0.011	0.2156 ±0.0005
16–37 μ	36.71	2.494 ±0.070	2597 ±52	9.34 ±0.19	1.258 ±0.008	0.2076 ±0.0005
4–16 μ	16.11	3.483 ±0.098	3079 ±62	9.20 ±0.18	0.9443 ±0.0083	0.1999 ±0.0005
<4 μ	11.23	5.36 ±0.15	3560 ±71	10.49 ±0.21	0.7236 ±0.0059	0.1947 ±0.0004
<i>15531,42</i>						
250–1000 μ	39.94	2.730 ±0.049	425 ±9	11.82 ±0.24	1.044 ±0.007	0.1982 ±0.0005
74–250 μ	47.37	4.437 ±0.075	612 ±12	7.64 ±0.15	0.8361 ±0.0040	0.1943 ±0.0005
37–74 μ	23.68	7.12 ±0.14	677 ±14	8.18 ±0.16	0.7238 ±0.0036	0.1923 ±0.0004
16–37 μ	14.12	13.03 ±0.26	844 ±17	7.92 ±0.16	0.6638 ±0.0033	0.1904 ±0.0004
4–16 μ	4.35	25.19 ±0.71	1013 ±20	8.27 ±0.17	0.6208 ±0.0045	0.1896 ±0.0004
<4 μ	4.04	67.3 ±1.1	1241 ±25	8.74 ±0.17	0.6067 ±0.0027	0.1886 ±0.0004
<i>67701,22</i>						
250–1000 μ	47.65	4.67 ±0.12	499 ±10	12.93 ±0.26	2.044 ±0.009	0.1916 ±0.0005
74–250 μ	45.18	6.14 ±0.12 ±0.26	563 ±11 ±12	12.26 ±0.25 ±0.25	1.219 ±0.005 ±0.0039	0.1907 ±0.0004 ±0.0004
37–74 μ	21.49	9.76 ±0.26	586 ±12	12.51 ±0.25	0.9523 ±0.0039	0.1897 ±0.0004
16–37 μ	15.62	19.69 ±0.89	599 ±12	12.31 ±0.25	0.8337 ±0.0039	0.1898 ±0.0005
4–16 μ	3.78	26.35 ±0.74	774 ±15	12.95 ±0.26	0.7618 ±0.0061	0.1884 ±0.0004
<4 μ	4.43	60.4 ±1.0	596 ±12	12.70 ±0.25	0.7287 ±0.0032	0.1877 ±0.0004
<i>71501,27</i>						
147–1000 μ	25.99	8.91 ±0.24	624 <sup>b</sup> ±12	— <sup>c</sup>	1.141 ±0.017	0.1919 ±0.0010
74–147 μ	17.86	7.27 ±0.32	601 ±12	—	1.017 ±0.034	0.1913 ±0.0010

Table 1. (Continued).

Sample	Mass <sup>a</sup> (mg)	<sup>40</sup> Ar (units of $10^{-5}$ cm <sup>3</sup> /g)	K (ppm)	Ca (%)	<sup>40</sup> Ar/ <sup>36</sup> Ar	<sup>38</sup> Ar/ <sup>36</sup> Ar
37-74 $\mu$	13.60	10.45 $\pm 0.33$	588 $\pm 12$	—	0.8072 $\pm 0.0094$	0.1918 $\pm 0.0010$
25-37 $\mu$	8.32	15.68 $\pm 0.50$	674 $\pm 13$	—	0.732 $\pm 0.016$	0.1914 $\pm 0.0010$
16-25 $\mu$	7.68	20.75 $\pm 0.63$	692 $\pm 14$	—	0.703 $\pm 0.015$	0.1908 $\pm 0.0010$
8-16 $\mu$	3.64	47.3 $\pm 1.8$	852 $\pm 17$	—	0.677 $\pm 0.019$	0.1902 $\pm 0.0010$
4-8 $\mu$	1.89	78.0 $\pm 3.6$	1020 $\pm 20$	—	0.700 $\pm 0.024$	0.1894 $\pm 0.0010$
Total Soil	6.66	18.23 $\pm 0.55$	507 $\pm 10$	—	0.741 $\pm 0.016$	0.1904 $\pm 0.0009$
75081,17						
147-100 $\mu$	22.93	5.28 $\pm 0.14$	605 <sup>d</sup> $\pm 18$	—	1.531 $\pm 0.019$	0.1943 $\pm 0.0006$
74-147 $\mu$	14.14	6.30 $\pm 0.17$	609 $\pm 18$	—	1.115 $\pm 0.009$	0.1936 $\pm 0.0006$
37-74 $\mu$	12.04	10.13 $\pm 0.26$	603 $\pm 18$	—	0.9112 $\pm 0.0073$	0.1916 $\pm 0.0006$
25-37 $\mu$	7.32	14.99 $\pm 0.39$	707 $\pm 21$	—	0.8298 $\pm 0.0051$	0.1909 $\pm 0.0005$
16-25 $\mu$	5.18	22.44 $\pm 0.43$	787 $\pm 24$	—	0.7945 $\pm 0.0098$	0.1904 $\pm 0.0005$
8-16 $\mu$	2.96	51.0 $\pm 1.4$	953 $\pm 29$	—	0.7320 $\pm 0.0046$	0.1896 $\pm 0.0006$
4-8 $\mu$	1.56	75.0 $\pm 2.0$	1011 $\pm 30$	—	0.7437 $\pm 0.0048$	0.1889 $\pm 0.0005$
1-4 $\mu$	0.52	67.2 $\pm 2.9$	—	—	0.816 $\pm 0.021$	0.1879 $\pm 0.0006$

The errors listed for the argon data are  $1\sigma$  and include contributions from the statistical scatter of the measured ratios, blank corrections, mass discrimination, and (for <sup>40</sup>Ar concentrations) uncertainties in gas calibrations and sensitivities—all compounded quadratically. Errors listed for the K and Ca contents, 3 and 2% respectively, are estimated errors.

<sup>a</sup>Mass of aliquot used for rare gas analysis. The aliquots used for K and Ca analysis weighed  $\sim 1$  mg.

<sup>b</sup>Evensen *et al.* (1974).

<sup>c</sup>Not determined.

<sup>d</sup>Evensen *et al.* (1973).

separates, linear correlations will result if all fractions contain the same two isotopically invariant volume and surface correlated components and the same specific concentrations of <sup>36</sup>Ar<sub>vc</sub>/[C].

Figure 1 shows a plot of <sup>38</sup>Ar/<sup>36</sup>Ar versus Ca/<sup>36</sup>Ar for the data from 15531,42. In Fig. 1 we are assuming that calcium is the dominant target element for the

Table 2. Argon from stepwise heating of 20.40 mg of neutron irradiated lunar fines 12033,42.

Temp (°C)	<sup>40</sup> Ar (units of 10 <sup>-6</sup> cm <sup>3</sup> g)	<sup>40</sup> Ar/ <sup>36</sup> Ar <sub>sc</sub>	<sup>39</sup> Ar/ <sup>36</sup> Ar <sub>sc</sub>	<sup>40</sup> Ar/ <sup>39</sup> Ar <sup>a</sup>	Apparent <sup>b</sup> age (units of 10 <sup>9</sup> yr)
450	0.0946 ±0.0056	303 ±18	0.27 ±0.12	1120 ±530	5.43 ±0.89
550	0.2282 ±0.0075	33.5 ±1.1	0.121 ±0.011	274 ±27	3.07 ±0.15
650	0.51 ±0.25	24 ±12	0.1115 ±0.0046	208 ±102	2.67 ±0.74
700	0.703 ±0.011	9.19 ±0.14	0.1099 ±0.0030	80.0 ±2.6	1.486 ±0.033
750	1.299 ±0.016	3.831 ±0.047	0.0544 ±0.0004	63.13 ±0.98	1.255 ±0.014
800	2.956 ±0.020	1.748 ±0.012	0.0242 ±0.0001	55.85 ±0.44	1.147 ±0.007
950	7.348 ±0.024	1.0418 ±0.0034	0.01853 ±0.00005	34.82 ±0.21	0.792 ±0.004
1000	2.067 ±0.011	1.3097 ±0.0070	0.02328 ±0.00008	39.22 0.33	0.872 ±0.005
1050	1.980 ±0.012	0.9422 ±0.0057	0.01298 ±0.00005	42.03 ±0.44	0.921 ±0.008
1150	6.834 ±0.039	1.0128 ±0.0058	0.00807 ±0.00003	76.36 ±0.77	1.438 ±0.0058
1350	5.194 ±0.022	1.7523 ±0.0074	0.01312 ±0.00005	103.33 ±0.69	1.764 ±0.008
1500	0.260 ±0.015	— <sup>c</sup>	— <sup>c</sup>	98.2 ±6.0	1.706 ±0.069
1650	0.2017 ±0.0019	54.84 ±0.52	0.068 ±0.015	800 ±170	4.84 ±0.38
Sum	29.67 ±0.26	1.314 ±0.012	0.01603 ±0.00004	57.22 ±0.74	1.167 ±0.012

<sup>a</sup>Calculated using  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}} = 0.397 \pm 0.012$ .

<sup>b</sup> $J = 0.01499 \pm 0.00027$ ,  $\lambda = 5.305 \times 10^{-10} \text{ yr}^{-1}$ ,  $T_m = (4.504 \pm 0.020) \times 10^9 \text{ yr}$ .

<sup>c</sup><sup>38</sup>Ar dominated by chlorine-derived <sup>38</sup>Ar and nominal calculation yields negative amount of <sup>36</sup>Ar<sub>sc</sub>. No correction for surface correlated <sup>40</sup>Ar/<sup>36</sup>Ar applied.

production of spallation argon. The four smallest grain-sized fractions form a linear array and the two coarsest fractions plot well off the line in the direction of too little <sup>38</sup>Ar (or too much <sup>36</sup>Ar). A York (1969) least-squares fit to the four smallest grain-sized fractions yields a line whose intercept,  $0.18844 \pm 0.00018$ , is from equation (1) the isotopic composition of the surface correlated component and whose slope is:

$$\text{slope} = \left[ \frac{^{36}\text{Ar}_{\text{vc}}}{[\text{Ca}]} \left\{ \left( \frac{^{38}\text{Ar}}{^{36}\text{Ar}} \right)_{\text{vc}} - \left( \frac{^{38}\text{Ar}}{^{36}\text{Ar}} \right)_{\text{sc}} \right\} \right]. \quad (2)$$

Using 1.52 as the value for  $(^{38}\text{Ar}/^{36}\text{Ar})_{\text{vc}}$  (Pepin *et al.*, 1974) and the intercept value

Table 3. Argon from stepwise heating of neutron irradiated lunar fines 75081.

Temp (°C)	<sup>40</sup> Ar (units of 10 <sup>-6</sup> cm <sup>3</sup> /g)	<sup>40</sup> Ar/ <sup>36</sup> Ar <sub>sc</sub>	<sup>39</sup> Ar/ <sup>36</sup> Ar <sub>sc</sub> (× 10 <sup>-3</sup> )
	75081,17	34.20 mg	total soil
350	0.1876 ± 0.0035	193.7 ± 5.8	184 ± 14
450	0.1322 ± 0.0045	208 ± 62	276 ± 103
550	1.646 ± 0.014	38.4 ± 2.0	42.2 ± 3.0
650	3.862 ± 0.081	10.65 ± 0.63	39.4 ± 2.4
750	24.29 ± 0.45	1.799 ± 0.044	0.853 ± 0.023
800	40.43 ± 0.47	0.883 ± 0.011	0.2517 ± 0.0039
850	35.99 ± 0.13	0.5487 ± 0.0020	0.1696 ± 0.0029
900	29.65 ± 0.23	0.6441 ± 0.0058	0.2363 ± 0.0041
950	16.77 ± 0.16	0.7876 ± 0.0082	0.4186 ± 0.0044
1000	6.538 ± 0.069	0.8505 ± 0.0097	0.657 ± 0.010
1050	6.032 ± 0.067	0.7600 ± 0.0091	0.5517 ± 0.0087
1150	27.10 ± 0.29	0.898 ± 0.010	0.4033 ± 0.0032
1250	7.833 ± 0.056	1.0047 ± 0.0083	0.6883 ± 0.0057
1350	1.642 ± 0.015	1.494 ± 0.015	1.706 ± 0.035
1450	0.4423 ± 0.0059	1.751 ± 0.025	2.343 ± 0.066
1550	0.0183 ± 0.0061	0.79 ± 0.26	3.6 ± 1.1
1650	0.077 ± 0.010	5.18 ± 0.89	12.9 ± 2.9
Sum	202.64 ± 0.79	0.8184 ± 0.0046	0.4044 ± 0.0064
	75081,17	27.40 mg	34-74 μ
350	0.0766 ± 0.0061	42.0 ± 3.7	93 ± 27
450	0.1866 ± 0.0071	20.8 ± 1.1	106.4 ± 8.3
550	1.619 ± 0.018	11.27 ± 0.27	45.3 ± 2.8
650	5.052 ± 0.027	3.144 ± 0.024	4.95 ± 0.15
750	12.11 ± 0.10	1.498 ± 0.14	1.101 ± 0.015
850	32.13 ± 0.19	0.6452 ± 0.0048	0.3473 ± 0.0025
950	18.51 ± 0.27	0.894 ± 0.014	0.6850 ± 0.0050
1050	9.680 ± 0.075	0.9532 ± 0.0086	0.8699 ± 0.0064
1150	17.61 ± 0.17	1.021 ± 0.011	0.6205 ± 0.0051
1250	10.200 ± 0.099	1.185 ± 0.013	0.7563 ± 0.0058
1350	3.493 ± 0.028	1.646 ± 0.015	1.406 ± 0.017
1450	1.160 ± 0.030	2.189 ± 0.058	3.078 ± 0.051
1550	0.022 ± 0.018	1.3 ± 1.0	29.9 ± 9.8
1650	0.134 ± 0.025	7.3 ± 1.5	9.4 ± 2.6
Sum	111.98 ± 0.41	0.9974 ± 0.0043	0.6933 ± 0.0043

The errors listed are 1σ statistical and do not include the error associated with the <sup>37</sup>Ar correction (see text) or the estimated 5% error in sensitivity.

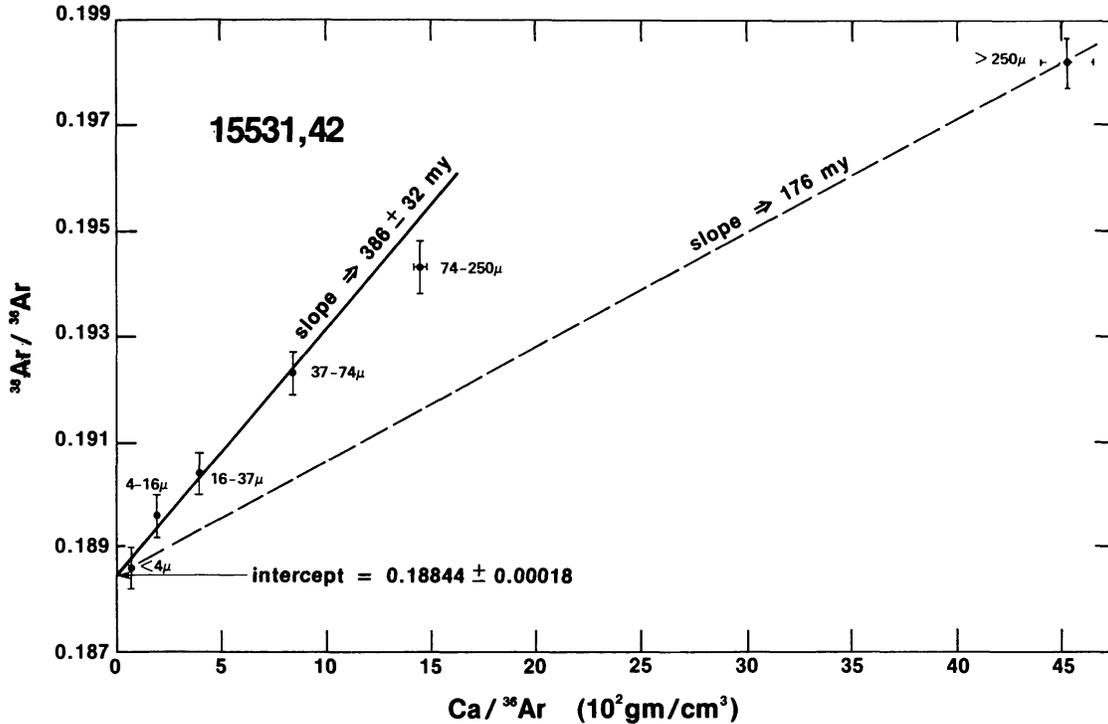


Fig. 1.  $^{38}\text{Ar}/^{36}\text{Ar}$  vs.  $\text{Ca}/^{36}\text{Ar}$  for data from grain-sized fractions of lunar soil, 15531,42. One  $\sigma$  error bars are shown where larger than the data point on this and the following figures. The slope of the lines are proportional to the exposure age of the soil and the ordinate intercept is the isotopic composition of surface correlated  $^{38}\text{Ar}/^{36}\text{Ar}$ . This plot is typical of all of the soils analyzed in this work.

for  $(^{38}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$ , the cosmic ray exposure age  $T_{38}$  is given by:

$$T_{38} = (^{38}\text{Ar}_{\text{vc}}/[\text{Ca}])/P_{\text{Ca}}^{38} = 1.52(\text{slope})/P_{\text{Ca}}^{38}(1.52 - \text{slope}) \quad (3)$$

where  $P_{\text{Ca}}^{38}$  is the production rate of  $^{38}\text{Ar}$  from Ca by cosmic rays on the lunar surface. We have adopted Turner *et al.*'s (1971) nominal value of  $1.4 \times 10^{-8} \text{ cm}^3 \text{ STP/gCa/m.y.}$  The slope of the fit to the four smallest grain-sized fractions in Fig. 1 corresponds to an exposure age of  $386 \pm 32 \text{ m.y.}$

The deviation of the coarsest fractions from the correlation line in Fig. 1 is typical of all of the samples studied in this work. This deviation is in the direction of a lower exposure age for the coarsest fractions. Assuming that the surface correlated component defined by the finer fractions is applicable to the coarser fractions, a model exposure age can be calculated for the coarsest fraction. The model exposure age is 176 m.y. for the  $> 250 \mu$  fraction from 15531,42 and is shown by the dashed line in Fig. 1.

*K/Ar Age.* Having determined the  $(^{38}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  ratio, the  $^{36}\text{Ar}$  can be apportioned between surface correlated and volume correlated components and the ordinate intercept relationship for  $^{40}\text{Ar}$  and  $^{36}\text{Ar}$  reduced to *the form of* an isochron diagram:

$$^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}} = (^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}} + (^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}) \frac{\lambda_{\text{EC}}}{\lambda_{\text{T}}} (e^{\lambda_{\text{T}}t} - 1) \quad (4)$$

where  $\lambda_T$  is the total decay probability for  $^{40}\text{K}$  and  $\lambda_{\text{EC}}$  is the decay probability of those decays which produce  $^{40}\text{Ar}$ . Equation (4) explicitly assumes that spallogenic  $^{40}\text{Ar}$  is negligible compared to radiogenic and surface correlated  $^{40}\text{Ar}$ . The conceptual model for Eq. (4) is not the same as that for normal isochron diagrams since the trapped component was presumably added over a span of time after the radiogenic  $^{40}\text{Ar}$  began to accumulate from the *in situ* decay. The slope and intercept of a linear array on a plot of  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  can be correctly interpreted in terms of an age and surface correlated  $^{40}\text{Ar}/^{36}\text{Ar}$  component if the following assumptions are met:

- A. The grains were completely degassed at  $t = 0$  and have quantitatively retained  $^{40}\text{Ar}$  ever since  $t = 0$ .
- B. No K has been added to or subtracted from the grains since  $t = 0$ .
- C.  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  is the same for all grains.
- D. All grains have the same (average) age.

If assumptions A through D are met, the resulting mixing line will have an intercept that is the correct value of  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  and a slope that is proportional to the correct age. The existence of a linear array on the diagram is a necessary *but not sufficient* criteria for deciding if the assumptions are met. Several plausible violations of one or more of the assumptions will result in linear arrays with incorrect slopes or intercepts or both. Several of these situations will be discussed in the following paragraphs.

Figure 2 is a plot of  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  for the data from grain-sized separates of 15531,42. The 4 to 250  $\mu$  fractions define a rather precise linear array with a slope corresponding to  $2.699 \pm 0.007$  G.y. and an intercept of  $0.56136 \pm 0.00072$ . The  $>250 \mu$  and  $<4 \mu$  fractions plot above the isochron. As will be seen below and as has been previously noted (Pepin *et al.*, 1972) the coarsest and finest fractions often plot off the isochrons defined by the intermediate grain-sized fractions. The most common situation is that both the finest and coarsest fractions plot above the isochron and the resulting pattern is concaved upward. In Pepin *et al.*'s (1972) analysis of 14259 the coarsest fraction plots below the isochron, however, and in Bogard *et al.*'s (1974) analysis of 76501 the finest fractions plot below the isochron. These grain-sized fractions yielding data which do not plot on the isochrons are clear evidence that one or more of the assumptions listed above are not being fulfilled by these fractions.

The deviations of the fine fractions from the isochrons seems most likely due to variations in  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$ . Pepin *et al.*'s (1972) analysis of 14149 convincingly shows a variation of  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  in the finer grain-sized fractions. It is not clear if the apparent variation is due to an actual grain size dependency of  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  or if it reflects a change in the mineral mix of each grain-sized fraction with the different minerals having different  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  and ages. Basford's (1974) analyses of 10084 tend to support the latter view. In Basford's analyses the finest grain-sized separates of bulk 10084 plot above the isochron defined by the intermediate grain-sized fractions. The finest grain-sized fractions of plagioclase and ilmenite separates plot on the isochrons, however, defined by the intermediate grain-sized

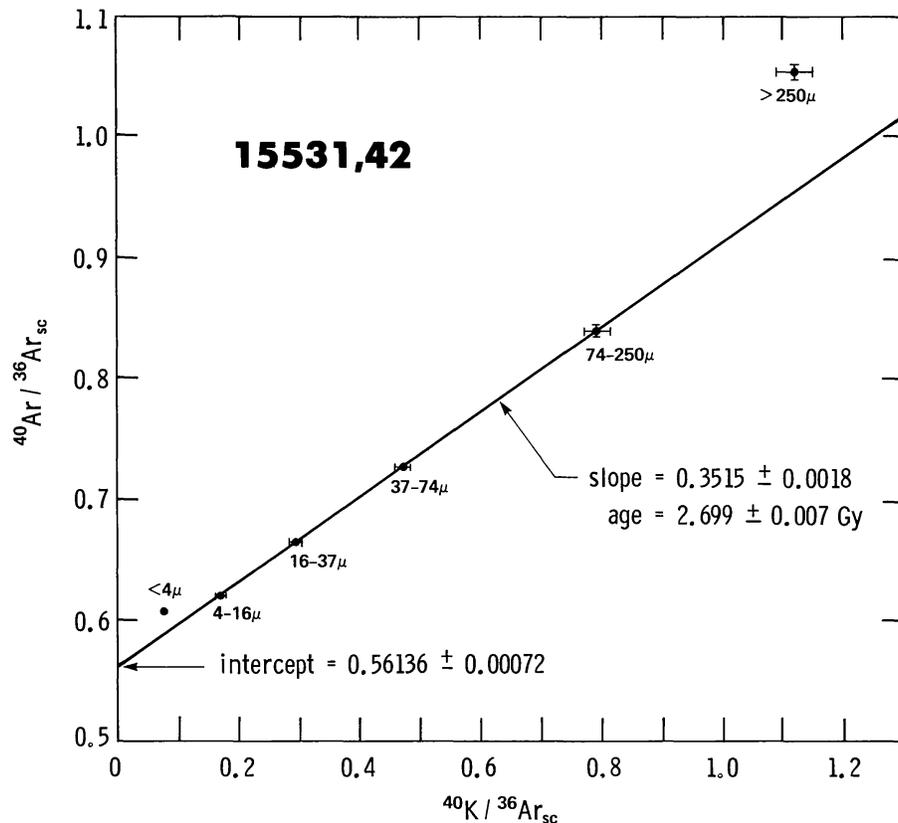


Fig. 2.  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  for data from grain-sized fractions of lunar soil 15531,42. The ordinate intercept of the line is the isotopic composition of the surface correlated  $^{40}\text{Ar}/^{36}\text{Ar}$ .

fractions of each mineral. The two minerals yield distinctly different ages and  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  from the bulk soil. The data from the finest fractions of the bulk soil can be qualitatively explained as an enrichment of plagioclase relative to ilmenite in the finest fractions.

The deviations of the coarsest fractions are more compatible with variations in age than with variations in  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$ . In general, model ages of the coarsest fraction of a soil, calculated assuming the  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  defined by intermediate fractions is applicable to the coarsest fraction, yields an age intermediate between the isochron age of the soil and the age of reasonable source rocks for the soil. For 15531, the model age of the  $>250\ \mu$  fraction is 3.03 G.y. This model age is between the 2.699 G.y. isochron age of the soil and the 3.3 G.y. crystallization age of the Apollo 15 basalts but agrees well with the bulk K/Ar ages of the basalts. The simplest interpretation is that the coarsest fractions are dominated by lithic fragments of local rocks. The coarsest fractions usually contain a significant agglutinate component (cf. Heiken *et al.*, 1973; McKay *et al.*, 1974), however, and the situation is probably much more complicated than the simple model of the preceding sentence.

At least two other formalizations have been suggested to deduce the radiogenic  $^{40}\text{Ar}$  and  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  values from lunar soils. Heymann and Yaniv (1970) and

several subsequent workers have used plots of  $^{40}\text{Ar}$  vs.  $^{36}\text{Ar}$ . Eberhardt *et al.* (1970) first suggested the use of  $^{40}\text{Ar}/^{36}\text{Ar}$  vs.  $1/^{36}\text{Ar}$  plots. Both groups explicitly note that a straight line in either space assumes that all of the samples have the same K content in addition to meeting assumptions A through D above.

The argon data from 15531,42 define straight lines in both  $^{40}\text{Ar}$  vs.  $^{36}\text{Ar}$  space and  $^{40}\text{Ar}/^{36}\text{Ar}$  vs.  $1/^{36}\text{Ar}$  space in which all six data points appear to plot on the lines. The  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  and K/Ar ages derived from these formalizations and from Fig. 2 are compared in Table 4. The same data set yields K/Ar ages which differ by 400 m.y. depending on the formalization used. The reason for this discrepancy is shown in Fig. 3. The K contents of the grain-sized fractions of 15531,42 are not constant but smoothly increase with decreasing grain size by a factor of  $\sim 3$ . The existence of a straight line in  $^{40}\text{Ar}$  vs.  $^{36}\text{Ar}$  or  $^{40}\text{Ar}/^{36}\text{Ar}$  vs.  $1/^{36}\text{Ar}$  space is not, therefore, a sufficient criterion to establish that all of a sample has the same K content. The K/Ar ages derived from such lines can be significantly in error.

The 15531,42 data shown in Fig. 3 illustrate another problem, namely, sample inhomogeneity. It is common practice to combine the radiogenic  $^{40}\text{Ar}$  measured in one laboratory with the K content measured in a different laboratory on a different allocation of the soil sample. The data given in Evensen *et al.* (1973) and this work came from different sievings of samples from the same 1 gram allocation of 15531. Although the sense of the variation in K concentrations with grain size is the same in the two sets of analyses, Evensen *et al.* measured about 50% more K than was found in this work. Since both sets of measurements were made in the same laboratory using identical techniques, spikes, etc., and since both sets are self-consistent, we believe that the difference is real and reflects K inhomogeneities within a single allocation. It is imperative that the K and  $^{40}\text{Ar}$  contents be determined on aliquots of the same sample, or better yet simultaneously on the same sample, if meaningful K/Ar ages are expected.

Sample 15531 was collected from a shallow trench, from which mare basalt 15555 was also collected, at Station 9A near the edge of Hadley Rille at the Apollo 15 site. The 2.7 G.y. age for 15531 is younger than the 3.3 G.y. age of 15555 in particular and 15 mare basalts in general but is in the range of their total K/Ar ages (Alexander *et al.*, 1972; Husain *et al.*, 1972; Podosek *et al.*, 1972; York *et al.*, 1972). The exposure age of 386 m.y. is older than the exposure age of approximately 85 m.y. for 15555 (Podosek *et al.*, 1972; York *et al.*, 1972; Marti and Lightner, 1972). The model K/Ar and exposure ages of the  $> 250 \mu$  fraction of 15531 are displaced toward the values for 15555.

Table 4. Comparison of calculated age and  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  for 15531,42 grain-sized separates using different formalizations.

	$^{40}\text{Ar}$ vs. $^{36}\text{Ar}$	$^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$ vs. $1/^{36}\text{Ar}_{\text{sc}}$	$^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$ vs. $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$
Age (G.y.)	2.299	2.345	2.699
	$\pm 0.044$	$\pm 0.050$	$\pm 0.007$
$(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$	0.6007	0.5973	0.5614
	$\pm 0.0052$	$\pm 0.0035$	$\pm 0.0007$

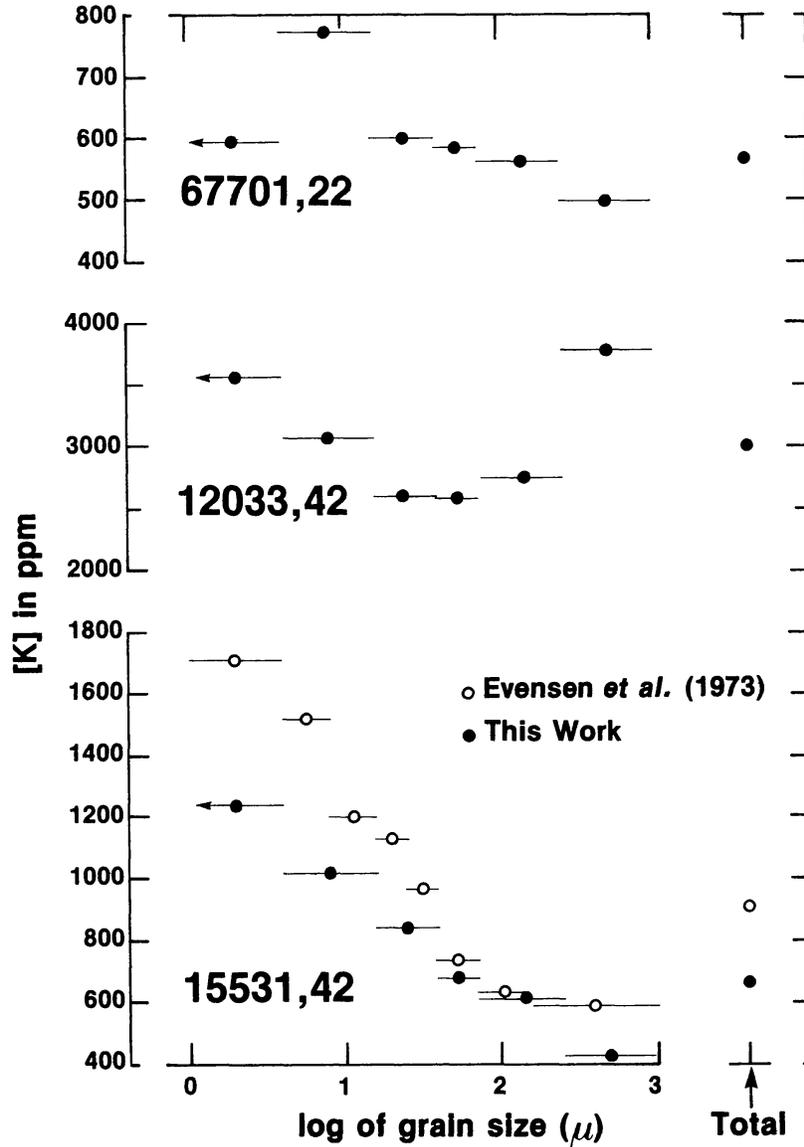


Fig. 3. Potassium concentration vs. log of grain size from grain-sized separates from three lunar soils. The potassium concentration was determined by isotope dilution on  $\sim 1$  mg aliquots. The bar through each data point shows the size interval represented by the data point. The "total" points shown on the right hand side of the figure were obtained by multiplying the potassium concentration of each fraction by the fractional mass yield of that fraction. The two analyses of 15531,42 were made on two different sievings of material from the same allocation.

### 67701

Sample 67701 was collected from a small, 40–50 cm, crater on the rim of North Ray Crater at Station 11 of Apollo 16. This soil is coarse grained and immature (Butler *et al.*, 1973; Heiken *et al.*, 1973).

*Exposure Age.* The  $^{38}\text{Ar}/^{36}\text{Ar}$  vs.  $\text{Ca}/^{36}\text{Ar}$  diagram for 67701,22 is very similar to Fig. 1 and is not shown to conserve space. The 16–37  $\mu$  fraction plots slightly above

the line defined by the  $<4$ ,  $4-16$ , and  $37-74 \mu$  fractions. The slope of that line corresponds to an exposure age of  $146 \pm 23$  m.y. The model exposure age of the  $>250 \mu$  fraction is 57 m.y. which is close to the age of 50 m.y. which is accepted for North Ray Crater (Behrmann *et al.*, 1973). The older exposure age of the finer grain-sized fractions clearly indicates that even this immature soil from the topographically high rim of a young crater contains significant amounts of material whose cosmic ray exposure age predates the crater.

**K/Ar Age.** Figure 4 is a plot of  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  for the data from grain-sized separates from 67701,22. The  $<250 \mu$  fractions scatter about a line whose slope corresponds to  $3.73 \pm 0.15$  G.y. Most of the scatter is in the  $4-16 \mu$  and  $16-37 \mu$  fractions and may be due to aliquoting or analytical problems. The K content of the  $4-16 \mu$  fraction appears to be high relative to the K content of the  $<4 \mu$  and  $16-37 \mu$  fractions. If the average of the latter two values were used as the K content for the  $4-16 \mu$  fraction, the resulting datum plots on the isochron. The  $>250 \mu$  fraction plots well above the line and has a model age of 4.17 G.y. Several of the light matrix breccias from the rim of North Ray Crater have  $^{40}\text{Ar}-^{39}\text{Ar}$  ages in excess of 4.2 G.y. (cf. Schaeffer and Husain, 1973) and so 4.17 G.y. is not an impossible age. There is no evidence that the North Ray Crater event reset the K/Ar

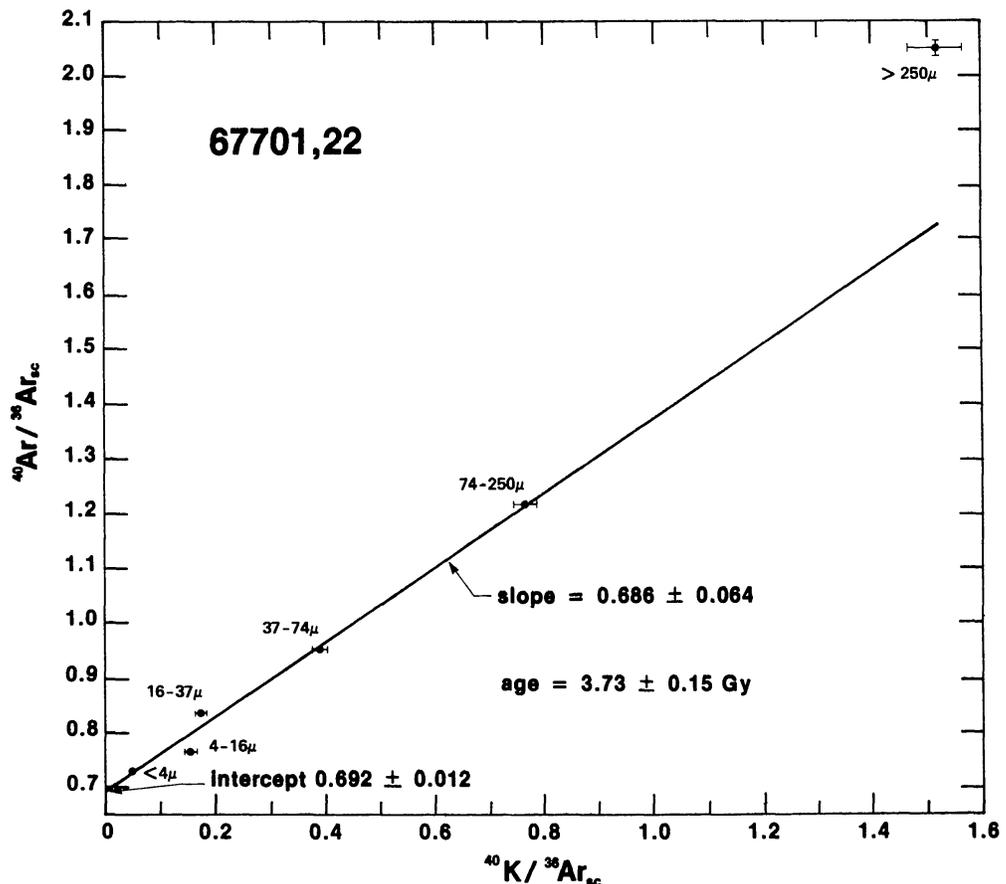


Fig. 4.  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  for data from grain-sized separates of lunar soil 67701,22.

clock to any significant degree in this material. The isochron age of 3.7 G.y. is a reasonable K/Ar age for the Apollo 16 site.

### 71501

Sample 71501 is a surface sample collected at Station 1 of Apollo 17. It is a sample from the “dark mantle” and is a submature to mature soil in McKay *et al.*'s (1974) classification.

**Exposure Age.** Calcium was not measured on the grain-sized fractions of 71501,27. The calcium content of the grain-sized fractions of the other soils in Table 1 is fairly uniform, however, and as noted by Pepin *et al.* (1975) a plot of  $^{38}\text{Ar}/^{36}\text{Ar}$  vs.  $1/^{36}\text{Ar}$  yields good estimates of  $(^{38}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  and spallogenic  $^{38}\text{Ar}$  if the target elements are uniformly distributed. The 4 through 147  $\mu$  fractions of 71501,27 define a straight line in  $^{38}\text{Ar}/^{36}\text{Ar}$  vs.  $1/^{36}\text{Ar}$  space whose slope, when combined with an average of the literature values for Ca in 71501 (Apollo 17 PET, 1973; Laul *et al.*, 1974; Rhodes *et al.*, 1974; Müller, 1975) yields an exposure age of  $269 \pm 39$  m.y. The  $> 147 \mu$  and total soil fractions are collinear with the intercept of the line defined by the other fractions. The model exposure age of the  $> 147 \mu$  fraction is 177 m.y.

**K/Ar Age.** Figure 5 is a plot of  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  for 71501,27. The 4 through 147  $\mu$  fractions scatter about a line whose slope corresponds to  $3.62 \pm 0.15$  G.y. The  $> 147 \mu$  fraction yields a model age of 4.31 G.y.

### 12033

Sample 12033 was collected from the bottom of a 15-cm deep trench about 15 m inside the northwest rim of Head Crater by the Apollo 12 astronauts (Shoemaker *et al.*, 1970). The 12033 fines contain a high proportion of KREEP glass (Hubbard and Gast, 1971; Meyer *et al.*, 1971; Marvin *et al.*, 1971). Hubbard *et al.* (1971) have suggested that the KREEP glass may have been deposited at the Apollo 12 site by a ray from the crater Copernicus. If Apollo 12 KREEP glass is Copernicus ejecta and if the impact event leaves a decipherable record in the K/Ar systematics, then the age of 12033 KREEP will be the age of the Copernicus impact.

**Exposure Age.** The  $< 4$  through 37–74  $\mu$  fractions of 12033,42 define a good line in  $^{38}\text{Ar}/^{36}\text{Ar}$  vs.  $\text{Ca}/^{36}\text{Ar}$  space. The slope of that line corresponds to an exposure age of  $333 \pm 11$  m.y. The 74–250  $\mu$  and  $> 250 \mu$  fractions plot progressively further to the right of the line and the model exposure age for the  $> 250 \mu$  fraction is 194 m.y. The latter age is in excellent agreement with Eberhardt *et al.*'s (1973a) values of 190 and 210 m.y. for two different  $> 150 \mu$  KREEP glass separates from 12033.

**K/Ar Age.** Figure 6 is a plot of  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  for 12033. Five of the six grain-sized fractions of 12033,42 listed in Table 1 define an excellent straight line whose slope corresponds to an age of  $1.208 \pm 0.017$  G.y. It is not known why the 74–250  $\mu$  fraction plots slightly off the line but it could be due to aliquoting problems of the type discussed earlier. Also shown in Fig. 6 are the total of the temperature data from Table 2 (Venkatesan, 1976) and the total data calculated

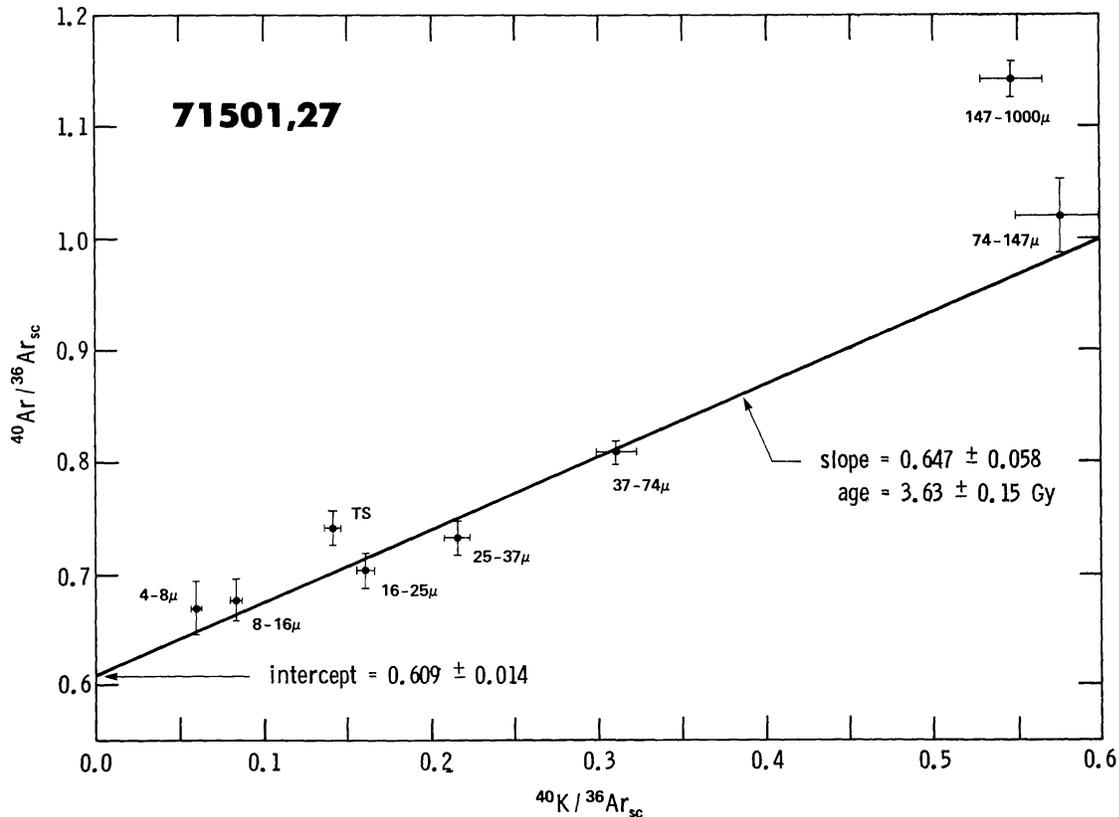


Fig. 5.  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  for data from grain-sized separates of lunar soil 71501,27.

from Eberhardt *et al.*'s (1973a) analyses. All of the  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  data are consistent with the line defined by the grain-sized data. The ordinate intercept of the line in Fig. 6,  $0.397 \pm 0.012$ , is a better estimate of  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  in 12033 than was available to Eberhardt *et al.* (1973a) and will be used below to interpret the  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  data. Note the change in scale between Fig. 6 and Figs. 2, 4, 5, 7, and 8. All of the data from 15531, 67701, 71501 and 75081 plot to the left of the finest fraction of 12033,42.

The  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  stepwise heating data from 12033, both our data from Table 2 and Eberhardt *et al.*'s (1973a) data, do not plot along the isochron shown in Fig. 6 but scatter around it in a complicated fashion. The scatter indicates: (a) that  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  is fractionating into various components in the stepwise heating experiment; or (b) that "sites" of differing ages are degassing at different temperatures; or (c) that some combination of (a) and (b) has occurred; or (d) that the data have been compromised by artifacts of the  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  technique; or (e) that some combination of (a), (b) and (d) has occurred. It is instructive to examine options (a), (b) and (d) as independent end members. Options (c) and (e), while viable, are inherently indeterminate.

Option (a) that the scatter in the temperature release data is completely due to variations in  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  is untenable for the 12033 data. Assuming that the age of each fraction is 1.208 G.y. from Fig. 6, one calculates that the  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  must vary from 16.9 to  $-0.08$  for our data and from 74.4 to  $-20.3$  for Eberhardt *et al.*'s (1973) data. Assuming that the age of each fraction is 800 m.y. (from the minimum of Fig.

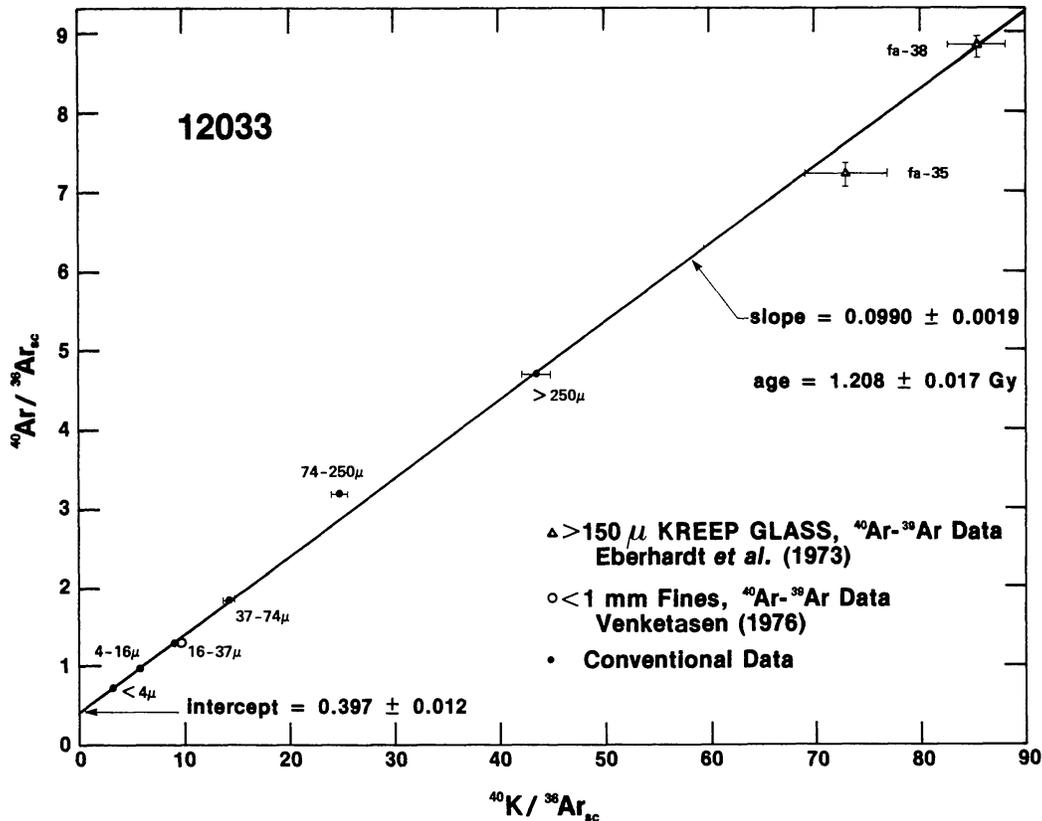


Fig. 6.  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  for data from grain-sized separates,  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  stepwise heating of bulk soil and from Eberhardt *et al.*'s (1973a)  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  analyses of lunar soil 12033. The line shown is a York (1969) fit to the  $<4$ ,  $4-16$ ,  $16-37$ ,  $37-74$  and  $>250\ \mu$  conventional data. Note the change in scale between this and Figs. 2, 4, 5, and 8.

7) removes the need for absurd negative values for  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  but moves the maximum required  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  up to 123.

Option (b) that the scatter in the temperature release data is completely due to variation in the apparent age of each fraction is the approach adopted by Eberhardt *et al.* (1973a) and is viable for the 12033 data. Figure 7 is a plot of apparent age vs. cumulative %  $^{39}\text{Ar}$  released calculated from the data listed in Table 2 with the assumption that the  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  defined by the intercept of Fig. 6 is applicable to each of the temperature fractions. The dashed line in Fig. 7 gives the average of Eberhardt *et al.*'s (1973a) results recalculated using the same  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$ . (This recalculation produces only very minor changes.) The gross features of the two curves are very similar and the lowest ages obtained in each case are identical within errors. The age pattern which Eberhardt *et al.* (1973a) identified in the  $>150\ \mu$  fractions clearly dominates the bulk soil also. Eberhardt *et al.* (1973a) interpreted their results in terms of major but incomplete degassing at  $\sim 800$  m.y. of KREEP material which was much older. They showed that Turner's (1969) theoretical treatment of Ar degassing provided a good fit to the intermediate and high temperature data but does not fit the low temperature data.

Option (d) that the pattern in Fig. 7 is an artifact produced by argon

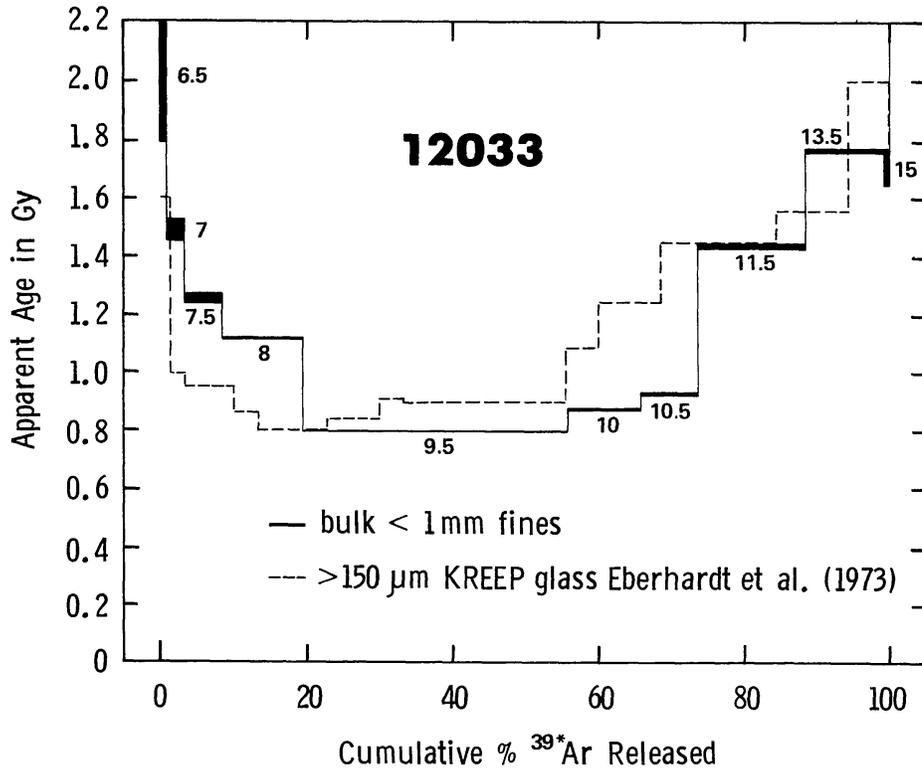


Fig. 7. Apparent age vs. cumulative %  $^{39}\text{Ar}$  released for stepwise heating data from this work and Eberhardt *et al.*'s (1973a) analyses of 12033. The numbers by the data are the release temperatures in hundreds of  $^{\circ}\text{C}$ .

redistribution during irradiation, of the type discussed theoretically by Turner and Cadogan (1974) and then experimentally documented by Huneke and Smith (1976), does not seem to be reasonable quantitatively. For example, the 950 $^{\circ}\text{C}$  fraction in our data yields an age of 792 m.y. and contains 36% of the total  $^{39}\text{Ar}$  release. In order to explain 792 m.y. as an age which has been lowered from 1.2 G.y. by the addition of  $^{39}\text{Ar}$  to the 950 $^{\circ}\text{C}$  fraction by recoil, 42% of the  $^{39}\text{Ar}$  in the fraction or 15% of the total  $^{39}\text{Ar}$  released by the sample would have to have been introduced into the 950 $^{\circ}\text{C}$  fraction by recoil. Similar amounts of  $^{39}\text{Ar}$  movement are necessary to explain the other temperature fractions. Approximately 50% of the total  $^{39}\text{Ar}$  would have to be redistributed to explain the release pattern in Fig. 7. While a 50% recoil effect is not *a priori* impossible since every  $^{39}\text{Ar}$  is moved significantly by the recoil process, Huneke and Smith (1976) only achieved a net depletion of 8.6% of  $^{39}\text{Ar}$  in their 5  $\mu$  glass in what was probably a much more favorable geometry.

The conventional K/Ar and  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  results as outlined above lend themselves to different interpretations. We emphasize that this is a matter of interpretation and that our data and Eberhardt *et al.*'s (1973a) data are gratifyingly consistent.

Interpretation  $\neq$  1, our preferred interpretation, is that the bottom of the apparent age curve in Fig. 7,  $\sim$ 800 m.y., is the best estimate of the age of KREEP in 12033 and therefore of Copernicus. This is in agreement with Eberhardt *et al.*'s (1972) interpretation. In this interpretation the straight line in Fig. 6 is produced by

incompletely degassed KREEP glass, with an effective bulk K/Ar age of 1.2 G.y., mixed with varying amounts of dilutant material. The dilutant material has a much lower K and Ar content and does not contribute significantly to the K/Ar systematics. Since all of the various fractions yield total K/Ar ages of 1.2 G.y., there is no grain size dependency to the degree of Ar loss. The simplest explanation for this lack is that the present grain size distribution is secondary and results from the break up of larger particles after the gas loss occurred. Interpretation # 1 is supported by Silver's (1971) conclusion based on U-Th-Pb systematics that the "exotic" radioactive debris in the Apollo 12 soil had been subjected to an important thermal episode  $850 \pm 100$  m.y. ago.

We are, however, unable to disprove to our satisfaction Interpretation # 2 in which the  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  curves are artifacts which fall under options (d) or (e) above. If this is the case, the age given by the slope in Fig. 6 would be the age of KREEP from 12033 and thereby of the Copernicus event. The best available age for 12033 KREEP is therefore  $800_{-50}^{+400}$  m.y. where the large upper limit includes the uncertainty introduced by Interpretation # 2.

### 75081

Sample 75081 is a surface sample collected from the southwest rim of Camelot Crater at Station 5 of Apollo 17. It is a sample of the Apollo 17 "dark mantle," a submature soil in McKay *et al.*'s (1974) classification and is similar to the 71501 sample discussed above.

**Exposure Age.** The five grain-sized fractions of 75081,17 between 8 and  $147 \mu$  define a good line in  $^{38}\text{Ar}/^{36}\text{Ar}$  vs.  $1/^{36}\text{Ar}$  space. The slope of that line corresponds to an exposure age of  $247 \pm 13$  m.y. The  $> 147 \mu$  fraction plots to the right of the correlation line and has a model exposure age of 174 m.y.

**K/Ar Age.** Figure 8 is a plot of  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  for the grain-sized data from 75081,17. The five grain-sized fractions between 8 and  $147 \mu$  define a line whose slope corresponds to an age of  $3.539 \pm 0.058$  G.y. The model age of the  $> 147 \mu$  fraction is 3.80 G.y. The total of the  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  temperature data discussed below plot on the 3.538 G.y. line.

The data from  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  stepwise heating experiments on an aliquot of the 37-74  $\mu$  grain-sized fraction and an aliquot of bulk 75081,17 are shown in Fig. 9 plotted in  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  space at the same scale as Fig. 8. These data fall into options (c) or (e) discussed above in connection with the 12033 data. In order to explain the data one must assume that the  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  is fractionating and that material with differing ages is degassing at different temperatures or that recoil has compromised the data. The 850°C fraction from the 37-74  $\mu$  aliquot and the 850° and 900°C fractions from the bulk soil sample yield  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  ratios less than  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  defined by the conventional data. These fractions are unambiguous evidence that  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  is fractionating in the stepwise heating experiment. The fractionation is not an artifact of  $^{39}\text{Ar}$  recoil since  $^{39}\text{Ar}$  recoil would move the data horizontally not vertically in  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  space. While all of the

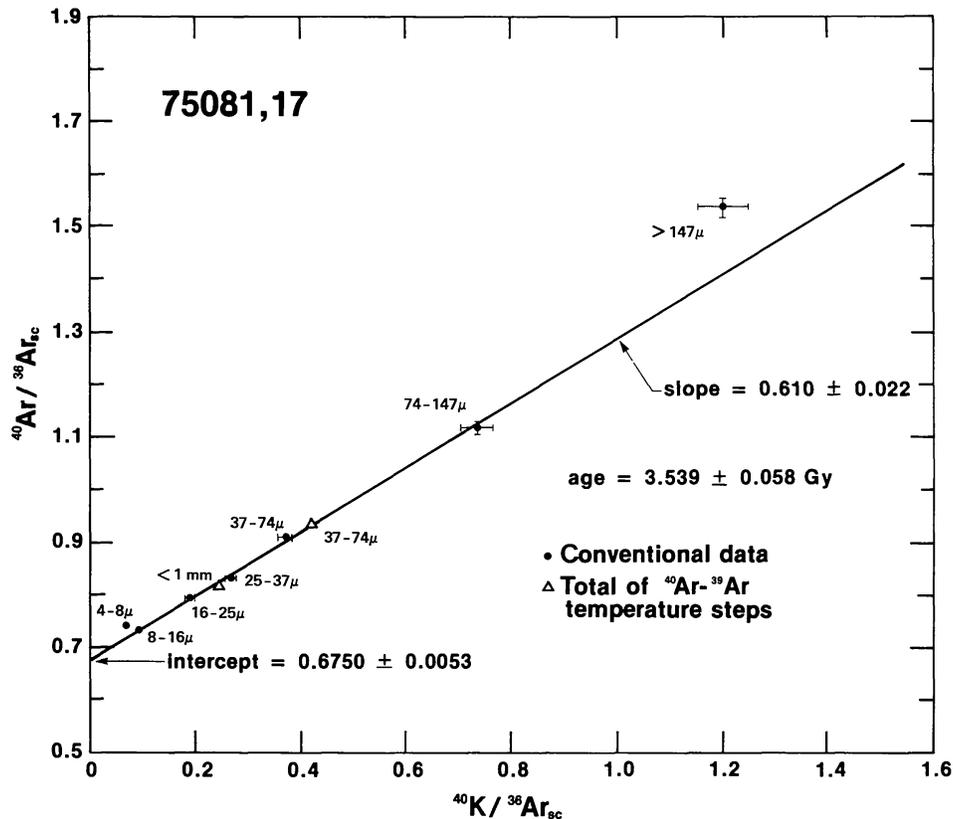


Fig. 8.  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  for data from grain-sized separates and the total of  $^{40}\text{Ar}-^{39}\text{Ar}$  stepwise heatings of  $37-74 \mu$  and bulk soil aliquots of 75081,17. The line shown is a fit to the 8 through  $147 \mu$  conventional data.

data from 75081 which plot on scale in Fig. 8 can be explained in terms of a 3.54 G.y. age and a varying  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  component, the high and low temperature data, which plot off scale in Fig. 8, cannot be so explained. Some of the high and low temperature data require negative or unreasonably high  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  values to yield a 3.54 G.y. age and must represent age differences and or recoil artifacts. Huneke *et al.* (1973) have shown that 74241 yields similarly complicated  $^{40}\text{Ar}-^{39}\text{Ar}$  stepwise heating data. Judging from these analyses of 75081 and Huneke *et al.*'s (1973) analysis of 74241, stepwise heating  $^{40}\text{Ar}-^{39}\text{Ar}$  experiments do not yield chronologically significant information from submature or mature lunar soils.

The "dark mantle" at the Apollo 17 site was interpreted in premission photogeologic studies as a recent pyroclastic unit (cf. McGetchin and Head, 1973) which was younger than Copernicus. The age of 75081 and 71501 agree within error and contain no evidence of a recent component. These "dark mantle" soils are far older than the Copernicus age, only slightly younger than the  $\sim 3.8$  G.y. age of the valley floor basalts (cf. Turner *et al.*, 1973) and are approximately contemporaneous with the orange glass soil 74220 (Husain and Schaeffer, 1973; Huneke *et al.*, 1973; Eberhardt *et al.*, 1973b) from the "light mantle" at the Apollo 17 site. These results confirm the postmission conclusions that the "dark mantle" is old (cf. Lucchitta, 1973).

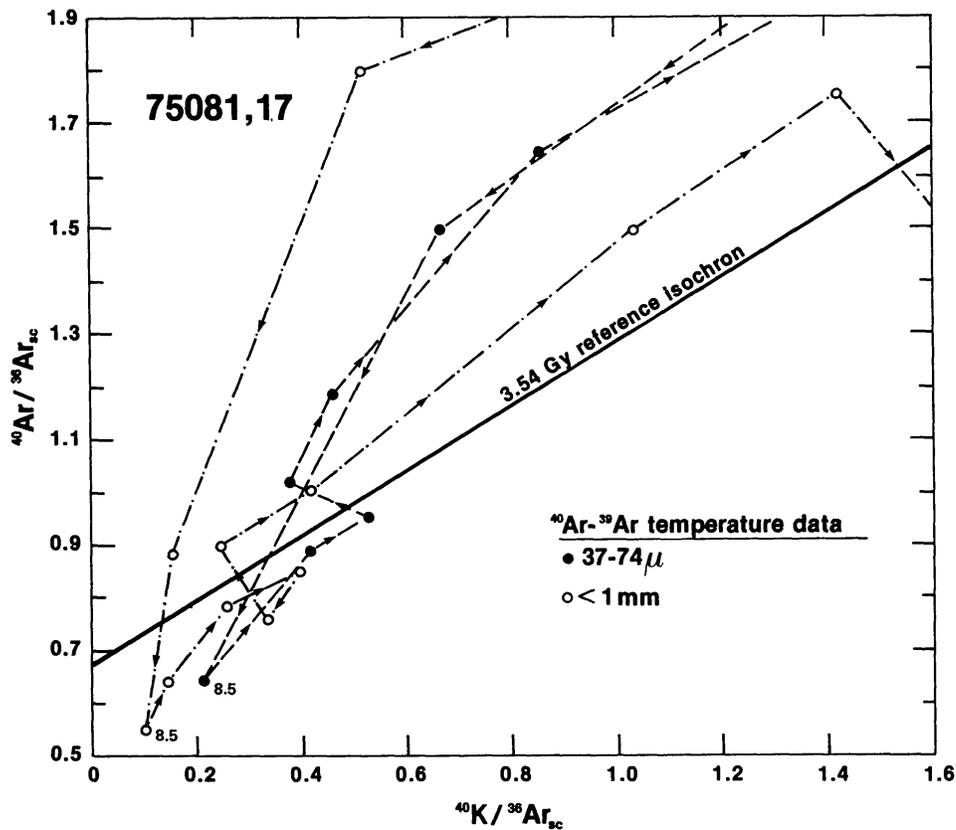


Fig. 9.  $^{40}\text{Ar}/^{39}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  for temperature data from stepwise heatings of 37–74  $\mu$  and bulk soil aliquots of 75081,17. The arrows on the lines connecting the data points indicate increasing temperature. To prevent crowding only the 850°C fraction of each analysis is identified by the number 8.5. The <750°C fractions of both aliquots and the >1350° and >1450°C fractions of 37–74  $\mu$  and bulk soil aliquots, respectively, plot off scale above and/or to the right of the Fig.

#### SUMMARY AND CONCLUSIONS

In view of the complex history of individual lunar soils, it is not surprising that no routine method for K/Ar dating lunar soils has emerged from this or other works. Conventional K/Ar measurements in which argon and potassium are measured on aliquots of grain-sized separates of lunar soils often yield data which can be interpreted in terms of an age and a single  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$ . Conventional K/Ar measurements are plagued by aliquoting and sample inhomogeneity problems, however. We are currently exploring the possibility that  $^{40}\text{Ar}$ – $^{39}\text{Ar}$  total fusion experiments on grain-sized separates can be used to overcome the sample inhomogeneity problem. The major question to be resolved is, will the total fusion  $^{40}\text{Ar}$ – $^{39}\text{Ar}$  data be compromised by recoil effects, particularly in the finest grain-sized fractions? More significant, however, is the evidence from the finest and coarsest grain-sized fractions and from mineral separates of grain-sized separates that many soils contain components with differing ages and/or  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  ratios. Much additional work needs to be done on mineralogic, morphologic, etc., separates of grain-sized separates of lunar soils. Despite these

caveats, ages deduced from  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  and analogous plots of data from grain-sized separates appear to be the best available K/Ar ages of submature to mature lunar soils.

Ages deduced from  $^{40}\text{Ar}$  vs.  $^{36}\text{Ar}$  and/or  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $1/^{36}\text{Ar}_{\text{sc}}$  or analogous plots, which assume that a soil has a uniform K content, can be significantly in error. Unless evidence is available that the K content is uniform within the soil, such “ages” should be viewed with distrust. The existence of a straight line in  $^{40}\text{Ar}$  vs.  $^{36}\text{Ar}$  or  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $1/^{36}\text{Ar}_{\text{sc}}$  space is not sufficient evidence that the K content of the fractions is constant.

Stepwise heating  $^{40}\text{Ar}$ – $^{39}\text{Ar}$  experiments do not appear to yield chronologically significant data from mature or submature soils. Such experiments can, however, yield useful information from simpler, immature soils such as 12033 where the K/Ar systematics are dominated by a single component.

Table 5 is a summary of the isotopic compositions of surface correlated argon, exposure ages and K/Ar ages derived from this work. It is evident that small but statistically real variations exist in  $(^{38}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  as well as much larger variations in  $(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$  between lunar soils. The exposure ages listed are actually for the  $<74\ \mu$  fractions. The coarser fractions uniformly have lower exposure ages. This trend is opposite from what might be expected from diffusive loss of spallogenic  $^{38}\text{Ar}$  and must reflect a longer effective exposure to cosmic rays recorded by the  $<74\ \mu$  material.

Figure 10 is a histogram of the  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  and  $^{40}\text{Ar}$ – $^{39}\text{Ar}$  K/Ar ages available for lunar soils. While the statistics are less than overwhelming, several features are noteworthy in Fig. 10. The two concentrations of ages between 2.6 and  $\sim 3$  G.y. and at  $\sim 4$  G.y. noted by Pepin *et al.* (1972) are still evident. A new concentration formed by the A17 mare soils and an A16 soil has formed between

Table 5. Surface correlated argon exposure ages, and K/Ar ages of lunar soils.

Sample	$(^{38}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$	$(^{40}\text{Ar}/^{36}\text{Ar})_{\text{sc}}$	$^{38}\text{Ar}_{\text{sp}}/\text{Ca}$ (units of $10^{-6}\text{cm}^3/\text{g}$ )	Exposure <sup>a</sup> age (units of $10^6$ yr)	K/Ar <sup>b</sup> age (units of $10^9$ yr)
12033,42	0.18902 $\pm 0.00045$	0.397 $\pm 0.012$	4.66 $\pm 0.16$	333 $\pm 11$	$\sim 0.800^{\text{d}}$
15531,42	0.18844 $\pm 0.00018$	0.56136 $\pm 0.00072$	5.40 $\pm 0.45$	386 $\pm 32$	2.699 $\pm 0.007$
67701,22	0.18756 $\pm 0.00021$	0.692 $\pm 0.012$	2.04 $\pm 0.33$	146 $\pm 23$	3.73 $\pm 0.15$
71501,27	0.18976 <sup>c</sup> $\pm 0.00025$	0.610 $\pm 0.014$	3.77 $\pm 0.55$	269 $\pm 39$	3.62 $\pm 0.15$
75081,17	0.18949 <sup>c</sup> $\pm 0.00011$	0.675 $\pm 0.005$	3.46 $\pm 0.19$	247 $\pm 13$	3.54 $\pm 0.06$

<sup>a</sup>Using  $P_{\text{Ca}}^{38} = 1.4 \times 10^{-8} \text{ cm}^3/\text{gCa}/\text{m.y.}$

<sup>b</sup> $\lambda_{\text{E.C.}} = 0.585 \times 10^{-10}/\text{yr}$ ,  $\lambda_{\beta^-} = 4.72 \times 10^{-10}/\text{yr}$ ,  $^{40}\text{K}/\text{K} = 1.19 \times 10^{-4}$ .

<sup>c</sup>Intercepts of  $^{38}\text{Ar}/^{36}\text{Ar}$  vs.  $1/^{36}\text{Ar}$  diagrams.

<sup>d</sup> $^{40}\text{Ar}$ – $^{39}\text{Ar}$  step-wise heating result. See text.

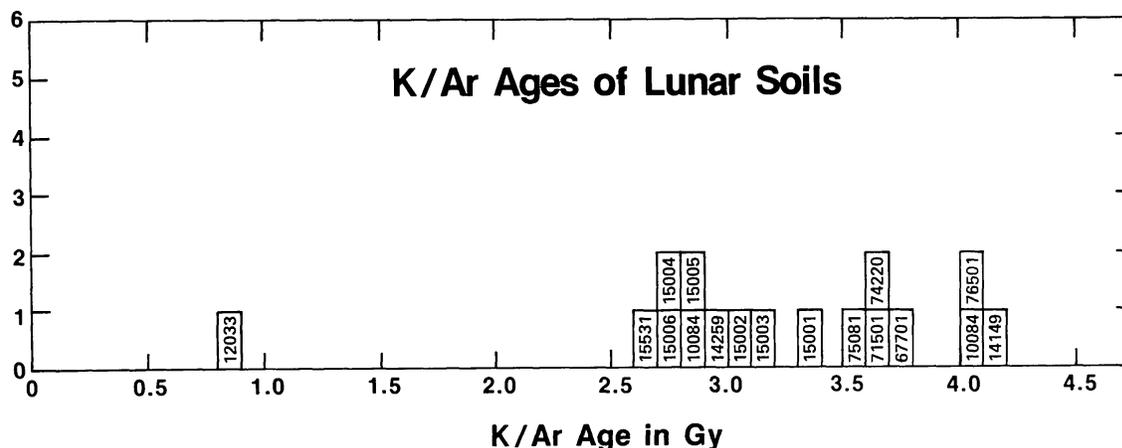


Fig. 10. Histogram of K/Ar ages of lunar soils derived from  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  studies or  $^{40}\text{Ar}/^{36}\text{Ar}_{\text{sc}}$  vs.  $^{40}\text{K}/^{36}\text{Ar}_{\text{sc}}$  diagrams on grain-sized separates. The sources of the data are: 12033—Eberhardt *et al.* (1973a) and this work; 67701 and 15531—this work; 71501 and 75081—Venkatesan (1976) and this work; 15501, 15502, 15503, 15504, 15505, and 15506—Pepin *et al.* (1974); 74220—Husain and Schaeffer (1973), Huneke *et al.* (1973), and Eberhardt *et al.* (1973b); 76501—Bogard *et al.* (1974); the two boxes labeled 10084 are from Basford's (1974) analyses of ilmenite and plagioclase separates from 10084; and 14259 and 14149—Pepin *et al.* (1972).

3.5 and 3.8 G.y. The simplest explanation of these “concentrations” is that they represent the bulk K/Ar age of their source material lowered by a small amount of Ar loss. It is striking that the regolith formation processes in general and an impact crater the size of North Ray at the Apollo 16 site in particular do not leave a recognizable signature in the K/Ar systematics. The Copernicus event, as recorded in 12033, remains the only chronologic signature less than 2.6 G.y. evident in the lunar soils analyzed so far.

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