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# CORNELL UNIVERSITY

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T. Gold, E. Bilson, R. L. Baron



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T. Gold, E. Bilson, R. L. Baron

Center for Radiophysics and Space Research

Space Science Building  
Cornell University  
Ithaca, New York 14853

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## ABSTRACT

The surface iron, titanium, calcium and silicon concentration in numerous lunar soil and rock samples was determined by Auger electron spectroscopy. As reported previously all soil samples show a large increase in the iron to oxygen ratio (and thereby of the surface concentration of iron) compared with samples of pulverized rock or with results of the bulk chemical analysis. The surface titanium concentration of the soil is also significantly increased vs. the bulk concentration whereas the surface calcium and silicon concentration is not significantly different from the bulk concentration in these elements.

A solar wind simulation experiment using 2 keV energy  $\alpha$ -particles showed that an ion dose corresponding to approximately 30,000 years of solar wind increased the iron concentration on the surface of the pulverized Apollo 14 rock sample, 14310 to the concentration measured in the Apollo 14 soil sample 14163 and the albedo of <sup>the</sup> pulverized rock decreased from 0.36 to 0.07.

The low albedo (as compared to that of pulverized rock) of the lunar soil is related to the iron + titanium concentration on its surface. A solar wind sputter reduction mechanism is discussed as a possible cause for both the surface chemical and optical properties of the soil.

## INTRODUCTION

In recent years increasing attention has been paid to the study of the chemical composition and chemical state of the outermost few atomic layers of lunar soil and rock grains. A knowledge of the surface chemistry may yield interesting information on exposure to processes that have altered only the outermost layer of the lunar surface material. The development and rapidly spreading use of surface analytical methods such as Auger electron spectroscopy (AES), X-ray photo-electron spectroscopy (ESCA), Secondary ion emission spectroscopy (SIMS) and others also contributed to the increased interest in such studies.

Using an Auger electron spectrometer with a retarding grid analyzer, we determined the surface iron, titanium and calcium concentrations in a great variety of soil and rock samples from all the Apollo sites (Gold et al., 1974 and 1975). We demonstrated a two to three fold increase in the iron/oxygen ratio on the surfaces of lunar soil grains, compared with their bulk composition, implying the presence of iron reduced to the metallic state. We presented a correlation between the surface iron concentration of soil samples and the depression of their optical albedo, and discussed a solar wind sputtering mechanism which would be most likely to cause the observed chemical and optical effects. This process, along with impact induced vaporization, followed by the deposition of material enriched in heavy metals has been discussed extensively also by Hapke et al., (1970, 1974, 1975) in conjunction with the optical properties of the lunar surface cover.

Yin et al. (1975, 1976) have studied by ESCA the mechanism of ion bombardment reduction of Fayalite ( $\text{FeSiO}_4$ ) and a number of metalhalides. In these studies an ion dose corresponding to 850 years of solar wind reduced the chemically bound iron to the metallic state on the surface of  $\text{FeSiO}_4$  powder, simultaneously a distinct visual darkening of the sample occurred.

Vinogradov et al. (1972) and also Housley and Grant (1975) demonstrated the existence of metallic iron on the surface of Apollo 11 soil sample grains.

These recent findings all point to a chemically altered surface skin on lunar soil grains especially as regards the concentration and/or chemical state of iron. More information is needed however on the chemical composition of this outer skin, and its thickness in order to deduce the exact mechanism that must be held responsible.

In this paper we present more detailed surface chemical information on lunar samples, and report our latest results on changes produced by simulated solar wind bombardment of lunar rock powders.

## EXPERIMENTAL

The Auger spectrometer used for our measurements and described earlier (Gold et al., 1974) has been modified. The retarding grid analyzer was replaced by a Varian single pass cylindrical mirror analyzer (CMA) with a standard fifteen stage BeCu electron multiplier. An external electron gun provides a primary electron beam at a grazing incidence angle

to the sample. All our data were taken with a 3 V r.m.s. modulation voltage applied to the cylindrical mirror (the actual electron energy modulation has not been determined). The change in the analyzer greatly increased the sensitivity of the measurements. Even more importantly, the cylindrical mirror analyzer detects electrons of a selected, narrow energy range, as opposed to the retarding grid system that detects all electrons with energies above a selected cutoff energy. This results in a suppression of the background continuum. The rapidly changing background continuum made analysis of low energy Auger peaks very difficult with the retarding grid analyzer. Using the CMA our low energy limit of detection of Auger peaks is approximately 85 eV in the case of lunar samples, thus we can detect and measure the 92 eV silicon Auger peak. The sample preparation techniques have been unchanged since our last publication (Gold et al., 1975). All our results presented here were obtained with a 1500 V primary electron energy and with the primary beam current between 0.5 and 1  $\mu$ A.

The samples of lunar fines were analyzed in the same state as received from the curator (air exposed); the rock samples were pulverized in a boron carbide mortar to approximately the same mean particle size as the fines. Sputter cleaning of the samples was not used nor were they immersed in any solvents. All our data presented here were obtained from Auger spectra that showed no significant contamination, having only minor carbon peaks (the peak to peak height ratio of the 290 eV calcium peak and 270 eV carbon peak was in most cases greater than 4:1).

The solar wind simulation experiment was performed in the apparatus and in the experimental circumstances described earlier (Gold et al., 1975).

## RESULTS

### Surface Chemistry

We performed Auger Spectroscopy, using the CMA, on thirteen lunar soil samples and seven rock powder samples. The results reported below contain Auger data of two additional rock samples, obtained with the retarding grid analyzer. (Due to a lack of a sufficient quantity of sample, analysis of those two rock powders could not be repeated with the CMA). Of the major elements present we observed clearly distinguishable peaks due to silicon, oxygen, calcium, titanium and iron in the Auger spectra of lunar material. The low energy detection limit imposed by the nature of our samples does not permit us to observe the low energy aluminum and magnesium Auger peaks. On the other hand the 1500 eV electron excitation energy is not sufficient for the observation of the high energy aluminum and magnesium peaks. (A 1500 eV primary electron energy was used because at this energy sample charging is largely avoided.) We measured the peak height (in the derivative mode of the Auger spectrum) of the 92 eV silicon peak, the 290 eV calcium peak, the 387 eV titanium peak (in Ti rich samples) and the 650 eV iron peak, and tabulated the peak-to-peak height ratios of each of the above peaks and the 510 eV oxygen peak. Due to the similar chemical structure of the lunar samples examined (this point will also be discussed later) the peak height ratios are good approximations of the surface chemical

concentrations in these samples. The Auger observations were calibrated by the bulk chemical concentration reported for the rock samples, specifically the bulk silicon, calcium and iron content of rock 60017 and the bulk titanium content of rock 71935. The calibration formula used and the assumptions involved were described previously (Gold et al., 1975). The elemental surface concentrations determined by this method are reported in Tables 1 and 2 along with the bulk concentrations obtained from the literature.

Table 1 shows no great differences between the surface and bulk concentrations of any of the four elements we measured. In two cases (for samples 61016 and 79135) the surface iron concentrations measured were 1.5 and 2 times greater than the reported bulk iron concentrations. We suppose this discrepancy is due to the large scale heterogeneity of the samples, our subsamples in the above two cases substantially differing from those used for the determination of the bulk chemical composition. Excluding these two samples the average surface iron/bulk iron concentration ratio is 1.11. The other average concentration ratios are (including all our rock samples): surface titanium/bulk titanium = 0.99, surface calcium/bulk calcium = 0.89 and surface silicon/bulk silicon = 0.88. Considering the deviations in the surface concentration data, primarily due to heterogeneity of even a single sample, the experimental uncertainty of any one measurement is  $\pm 25\%$ . Therefore the deviation from unity of all the surface/bulk concentration ratios for rock samples are within this experimental uncertainty. Table 2 reveals quite a different behavior, especially in the surface iron concentration of soil samples. The average surface iron/bulk iron concentration

ratio is 2.5. The other average concentration ratios are: surface titanium/bulk titanium = 1.4, surface calcium/bulk calcium = 0.74 and surface silicon/bulk silicon = 0.97.

In order to avoid biasing the surface concentration data by the choice of rock samples used for normalization, we also calculated ratios of the average elemental concentrations on soil surfaces to the average elemental concentrations on rock surfaces. These are as follows:

Element	<u>Soil surface concentration</u> <u>Rock surface concentration</u>
Fe	2.25
Ti	1.41
Ca	0.83
Si	1.1

Our recent Auger results therefore confirm the 2 to 3 fold increase of iron concentration on the surface of most soil samples examined and a significant increase in iron concentration on all the soil surfaces. A smaller but significant increase in the surface titanium concentration is also observed. However we observed no increase of the surface titanium concentration in the case of the most titanium rich soil samples: 10084 and 75061. Due to the uncertainty in our measurements the slight average decrease of calcium concentration and the slight average increase of silicon concentration cannot be taken as significant, although Housley et al., (1976) reported<sup>a</sup> similar decrease and increase respectively in these elements observed by ESCA with soil sample 10084.

In Figure 1 the albedo of ground-up rock samples and that of soil samples is plotted against the surface (Auger) iron + titanium concentration and also against the bulk iron + titanium concentration in the samples. The data points (with the exception of the iron poor, very high albedo soil samples 73241, 67601, 63501) are fitted to the exponential law:  $A = A_0 e^{-n\sigma}$ , where A is the observed albedo,  $A_0$  is the hypothetical albedo at  $n = 0$ , (the law does not seem to be valid for soil samples at very low n values), n is the iron + titanium concentration (surface or bulk) observed and  $\sigma$  is the absorption coefficient. These are three distinct curves.

1. Albedo of soil samples vs. bulk iron + titanium concentration.
2. Albedo of soil samples vs. surface iron + titanium concentration.
3. Albedo of ground-up rock samples vs. surface (approximately same as bulk) iron + titanium concentration.

#### Solar-Wind Simulation Experiments

We have reported already (Gold et al., 1975) the results of a series of proton and  $\alpha$ -particle irradiation experiments, in which the surface chemical composition of the samples was determined before and after irradiation. A 3.2 coulomb/cm<sup>2</sup> dose of proton irradiation, at 2 keV energy, (corresponding to an approximately 3000-yr. dose of the proton component to the

solar wind, assuming a proton flux on the Moon of  $2 \times 10^8$  protons/sec/cm<sup>2</sup>) changed the surface chemistry of pulverized lunar rock samples to that of the soil. In particular the iron/oxygen ratio on the surface of rock sample 14310 increased to a value which is within experimental uncertainty the same as that measured on the surface of soil sample 14163. We have not reported on the albedo change due to ion bombardment. Visual darkening was observed in the above experiments but irradiation doses of the order of a few coulombs/cm<sup>2</sup> were clearly insufficient to lower the albedo of ground-up rock to the albedo of the soil of similar bulk chemical composition.

Recently we performed an experiment with sample 14310 using a large dose of  $\alpha$ -particles. Both the Auger spectrum and the albedo of the sample were determined before and after irradiation with a 30 coulombs/cm<sup>2</sup> dose. There was a two fold increase of iron concentration on the surface of the ground-up rock sample due to the above ion dose (corresponding to approximately 30,000 years of solar wind), similar to the increase observed already with the 3.2 coulombs/cm<sup>2</sup> proton dose. The albedo (at 5500 Å) however changed from 0.36 to 0.07 in this case. The albedo of our Apollo 14 soil samples ranged from 0.10-0.13. The 30 coulombs/cm<sup>2</sup> irradiation dose thus darkened the rock powder to a somewhat lower albedo than that of lunar soil of similar bulk chemical composition.

## DISCUSSION

Auger analysis of a representative number of presently available soil and rock samples from the Moon, has shown that in the case of soil samples the surface concentration of iron and possibly of titanium is significantly greater than the bulk concentration of these elements. No significant change has been found however in the surface vs. bulk concentration of calcium and silicon. Our solar wind simulation experiments indicate that the chemical change on the surface of rock powders induced by positive ion bombardment is similar to the change from bulk to surface chemical composition in lunar soil samples. The increase of iron concentration on both the surface of soil grains and the surface of ion bombarded rock powder grains are considered to correspond to the reduction of iron (due to the loss of oxygen by sputtering) observed by Yin et al., with ESCA (1976). These authors view the iron reduction mechanism as a complex process not simply related to physical sputtering but more chemical in nature. They suspect that the reduction mechanism is strongly dependent on the nature of the transition metal involved. Our results indicate that the elemental concentration changes on the surface of the soil samples and ion bombarded material are more complex than simply mass dependent as suggested in earlier works. Actually, so far it seems that a significant bulk to surface concentration change only exists for transition metals and that the effect indeed is strongly dependent on their nature (see difference in the behavior of iron and titanium). Accordingly one could suppose that all the elements but the transition metals sputter off

with approximately equal efficiency during ion bombardment, while the sputtering efficiency for iron and titanium is lower (but not equal for these two). Clearly, an understanding of the mechanism of ion sputtering of lunar like material is not yet available. It is not yet clear how the above surface chemistry of the soil can be explained in terms of the impact vaporization-deposition process or by the combination of solar wind sputtering and impact vaporization. Auger electron spectroscopy does not reveal the chemical state of the element detected. The use of AES for quantitative analysis of the elemental surface composition requires much caution. The peak to peak height magnitude in the derivative of the electron energy distribution spectrum (used by us as a measure of the quantity of the elements present on the surface) could be influenced by the chemical environment of the atoms being studied (see for example Grant et al., 1973). Housley et al., (1976) showed that in the Auger spectrum of metallic iron the Fe peaks are twice as sharp as in the spectrum of iron oxide,  $Fe_2O_3$ . Indeed we would expect a stronger signal from the pure element than from one of its compounds in which it is present at less than 50 atomic percent. Housley et al. do not mention whether their data takes this effect into account. We have not integrated our Auger spectra and have not determined the true electron energy distribution. We are not in the position thus to determine to which extent the iron Auger data are an indication of iron reduction or an actual increase of iron concentration on the surface.

To some extent the two processes should be related and the reduction of iron down to a hundred atomic layers from the surface for example would also manifest itself in a considerable increase in the concentration of iron.

Our interest in the surface chemistry of lunar material was triggered by the desire to explain the optical behavior of the soil. Our results have shown a clear correlation between surface iron concentration of soil samples and their albedo. It is possible that a correction factor should be introduced in the surface concentration data but this would only quantitatively alter the correlation.

In Figure 1 the albedo and concentration data were fitted to the exponential law  $A = A_0 e^{-n\sigma}$  supposing that iron and titanium provide absorption centers. As we have seen the albedo of soil and of ground-up rock samples is clearly correlated with both the bulk iron + titanium concentration and the surface iron + titanium concentration in these samples. (Of course in the case of ground-up rock samples the bulk and surface concentrations are the same within experimental error.) The three distinct curves, 1, 2 and 3 show that:

- a. The albedo of the soil samples is approximately three times lower than that of ground-up rock samples having the same bulk iron + titanium concentration, see curves 1 and 3. Concurrently the soil samples have higher (2-3 times in most cases) iron + titanium concentration on their surface than the bulk concentration in these elements, see curves 1 and 2.

The soil must have suffered a treatment that affected both its albedo and its surface iron and titanium concentration, and the two effects are seen to be quantitatively related. We consider therefore that the albedo of soil is indeed intimately related to the surface chemistry.

b. There is a different relationship between the albedo of the soil samples and their surface chemistry from that existing between the albedo of the ground-up rock samples and their surface chemistry, see curves 2 and 3. A different mechanism must therefore be responsible for light absorption on the surface of soil samples and on the surface of freshly ground rock powders. In the case of soil samples it is possible that the albedo - surface iron + titanium concentration correlation means that there exists a layer on most grains, which is thick enough to be optically significant, and in which similarly increased concentration of heavy metals exists as in the outermost few angstrom layer analyzed by Auger spectroscopy. The ESCA results mentioned earlier suggest that at least some of the iron (there is no data for titanium) in this outer layer is reduced to a lower oxidation state than the state in the bulk. (For iron this is the metallic state.) Absorption centers might have been created by alteration of the chemical state of a

surface layer. It is also possible that the chemically altered skin on soil grains is too thin to be optically significant and the increased light absorption in soil samples is due to a crystallographic change in the lattice. This change then seemingly goes in step with a chemical change on the outer surface and is very probably due to the same surface weathering agent. This crystallographic change, also would more effectively increase light absorption in iron (+titanium) rich, intrinsically darker grains than in iron poor, light rock powders.

Acknowledgment

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Table 1

## Rock Samples

## Elemental concentrations in atomic percent

Sample	Fe		Ti		Ca		Si	
	Bulk	surface (Auger) normalized to 60017	Bulk	surface (Auger) normalized to 79135	Bulk	surface (Auger) normalized to 60017	Bulk	surface (Auger) normalized to 60017
10044	6.0 <sup>a</sup>	7.0	2.6	2.8	5.1	4.4	16.5	14.6
10057	6.6 <sup>b</sup>	7.2*	3.3	3.3*	4.2		15.7	
10062	6.6 <sup>c</sup>	7.9*	3.4	2.9*	5.7		15.7	
14310	2.5 <sup>d</sup>	3.3	0.3		4.8	4.4	19.0	13.6
15556	7.0 <sup>e</sup>	7.6	0.7	0.7	4.1	3.4	17.5	12.3
60017	1.1 <sup>f</sup>	1.1	0.1		6.4	6.4	15.6	15.6
61016	1.5 <sup>g</sup>	3.2	0.2		5.9	4.7	15.6	15.1
76315	2.8 <sup>h</sup>	2.6	0.4		4.3	4.9	16.9	18.4
79135	4.8 <sup>i</sup>	7.2	1.6	1.6	4.6	3.1	16.4	13.3

Bulk chemical concentrations were obtained from the following sources:

a Engel and Engel (1970)

b *ibid.*

c Goles et al. (1970)

d Philpotts et al. (1972)

e Strasheim et al. (1972)

f Laul and Schmitt (1973)

g Wänke et al. (1973)

h Rhodes et al. (1974)

i Wänke et al. (1974)

\* Data obtained with the retarding grid.

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Table 2

## Soil Samples

## Elemental concentrations in atomic percent

Sample	Fe		Ti		Ca		Si	
	Bulk	surface (Auger) normalized to 60017	Bulk	surface (Auger) normalized to 79135	Bulk	surface (Auger) normalized to 60017	Bulk	surface (Auger) normalized to 60017
10084	4.9 <sup>j</sup>	10.6	2.1	2.0	4.7	3.3	16.5	15.3
12070	5.1 <sup>k</sup>	11.0	0.7		4.2		18.0	
14003	3.4 <sup>l</sup>	9.2	0.6	0.8	4.5	4.5	18.9	14.8
14163	3.1 <sup>m</sup>	5.4	0.5	0.8	3.8	3.3	18.7	17.9
15301	4.2 <sup>n</sup>	7.8	0.3	0.6	4.0	3.1	16.2	16.4
61500	1.7 <sup>o*</sup>	4.3	0.2		6.1	3.8	16.3	15.9
62240	1.7 <sup>p*</sup>	4.4	0.2		6.1	3.6	16.3	15.6
63501	1.3 <sup>q</sup>	4.4	0.2		6.5	3.9	15.4	15.9
66041	1.9 <sup>r</sup>	5.5	0.2		5.9	3.7	15.5	17.9
67601	1.3 <sup>s</sup>	5.5	0.1		6.0	4.4	15.8	17.6
73241	2.5 <sup>t*</sup>	3.3	0.4	0.7	5.2	4.0	16.5	14.1
75061	5.2 <sup>u</sup>	11.2	2.7	2.1	3.6		14.8	
76501	3.3 <sup>v</sup>	7.7	0.9	1.1	4.6	3.7	16.1	14.3

Bulk chemical concentrations were obtained from the following sources:

j Wänke et al. (1970)

p Rose et al. (1973)

k Wänke et al. (1971)

q Brunfelt et al. (1973)

l Ehmann et al. (1972)

r Laul and Schmitt (1973)

m ibid.

s Haskin et al. (1973)

n Strasheim et al. (1972)

t Rhodes et al. (1974)

o Wänke et al. (1973)

u Brunfelt et al. (1974)

v Rose et al. (1974)

\* Literature data were not available for these very samples.

Data in this table were found for 61501, 62241 and 73141 and used for 61600, 62240 and 73241 respectively.

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## Figure Caption

Figure 1. Albedo vs. iron + titanium concentration in ground-up rock and soil samples. The data points (with the exception of that of the iron poor, very high albedo soil samples 73241, 67601, 63501) are fitted to the exponential law:  $A = A_0 e^{-n\sigma}$ , where  $A$  is the observed albedo,  $A_0$  is the hypothetical albedo at  $n = 0$ ,  $n$  is the iron + titanium concentration (surface or bulk) observed and  $\sigma$  is the absorption coefficient. The concentration error bars indicate the Auger concentration extremes obtained by taking spectra on various spots of the same sample, the albedo error bars refer to the lowest and highest albedo measured with different sample orientations. (Since very small (8 mm diameter) samples were used for the albedo measurements, they were repeated three times with three different sample orientations — in the same plane — under the light beam.) The albedo was measured at 5500 Å wavelength, at 8° illumination angle and was normalized to MgO.

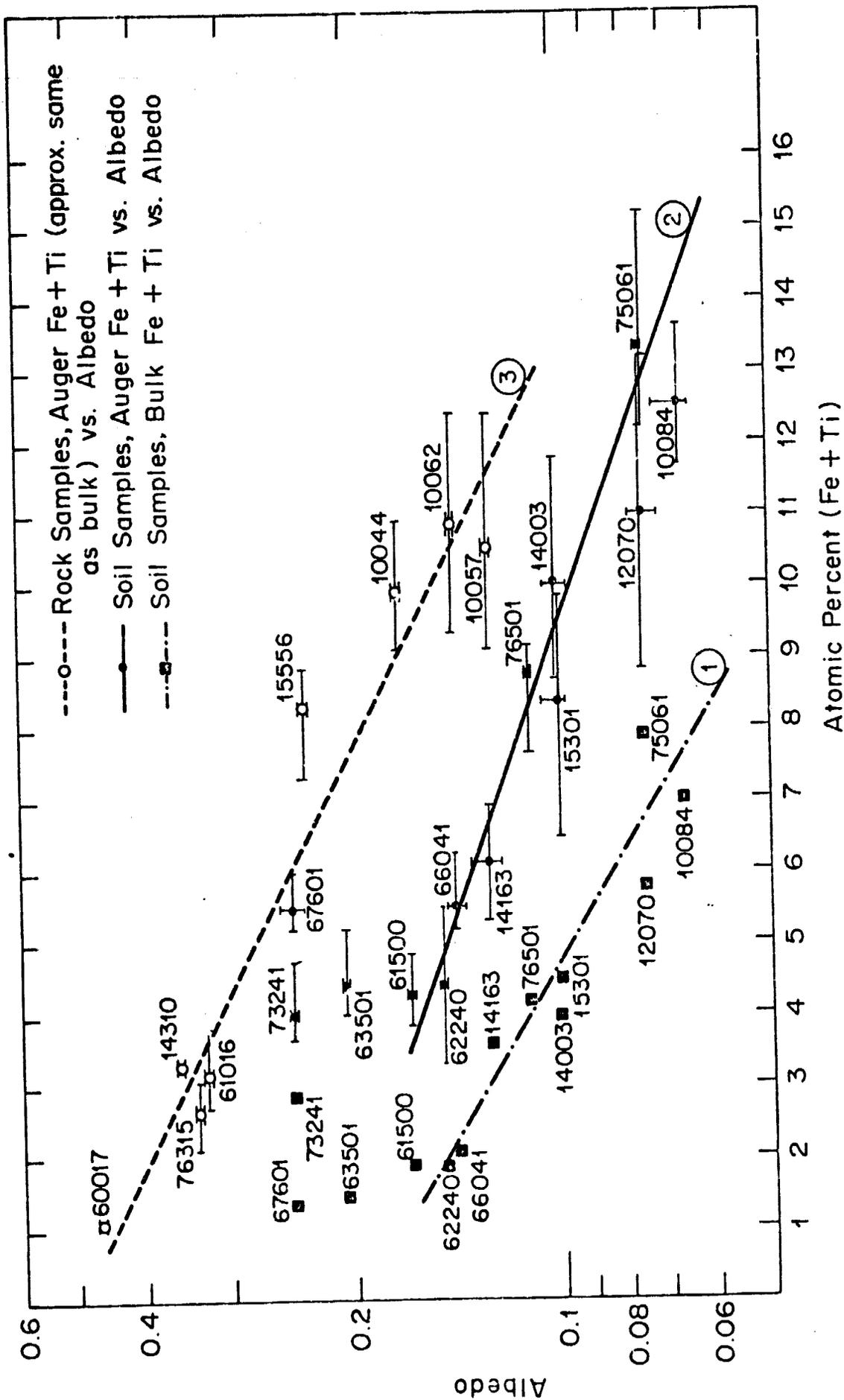


Figure 1