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# NASA TECHNICAL MEMORANDUM

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## MICROELECTROPHORESIS OF SELECTED MINERAL PARTICLES

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## TECHNICAL MEMORANDUM

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### INTRODUCTION

Electrophoresis is a technique for purifying materials based upon the characteristic surface charges of those materials. In addition to biological applications of electrophoresis and the use of electrophoresis for analysis of materials such as cloth fibers and clays [1], interest has developed in the process within NASA for the separation of insoluble minerals such as those found in lunar soil, or regolith (the term used to describe the fine-grained debris layer that overlies much of the surface of the moon) [2]. A particular mineral of interest for production of specific refined products, such as anorthite for production of aluminum and silicon or ilmenite for production of titanium and iron, must first be isolated before any method of refining can be applied [3]. The purpose of this study is to measure single particle electrophoretic mobilities of selected insoluble, small-grained minerals and to characterize the separable features. The suitability of electrophoresis as a process for a possible future space manufacturing facility that would separate lunar soil components and/or for a facility that would separate fine-grained soil on Earth can then be better evaluated.

In separation of minerals high resolution as well as large throughput are necessary if quantities sufficient for refining processes are to be isolated. A high degree of automation is also desirable. Currently available soil fractionation methods involving electrostatic, magnetic, flotation, and density characteristics do not achieve acceptable levels of efficiency when applied to fine-grained soils, nor are these methods easily automated [4]. The properties of lunar soil which reduce the effectiveness of conventional mineral separation methods should have positive effects in low-gravity electrophoretic separations [3]. Lunar soil, with an average bulk density of  $1.66 \text{ g cm}^{-3}$  for samples taken from depths of 0 to 60 cm, and with component densities as high as  $4.9 \text{ g cm}^{-3}$  in the case of ilmenite, would remain suspended longer in a low- or near-zero gravity environment. Mean grain size for most lunar soils falls between 45 and 100  $\mu\text{m}$ . Grain size fractions and general petrography of particle types for typical lunar soil samples are shown in Figures 1 and 2, respectively [5]. Successful separation is dependent upon whether this fine-grained particulate matter remains suspended for an adequate time period in the bulk medium. With eventual processing on the Moon, or in near-zero gravity, the rapid settling of particles which occurs on Earth would be avoided. Weightlessness would eliminate buoyancy-induced convection caused by thermal gradients resulting from heat produced by passage of current through the medium.

Construction of space structures such as solar power satellites will require large amounts of silicon and aluminum and small amounts of other elements such as titanium, iron, magnesium, and oxygen. Analysis of cost estimates for transporting materials from Earth to space suggests that the success of construction of large space structures may depend directly on the processing of nonterrestrial resources such as lunar soil to obtain materials [2].

<u>GRAIN SIZE</u>	<u>WEIGHT %</u>	<u>CUMULATIVE WT. %</u>
< 20 $\mu\text{m}$	26.85	26.85
20-45 $\mu\text{m}$	18.02	44.87
45-75 $\mu\text{m}$	12.40	57.27
75-90 $\mu\text{m}$	4.01	61.28
90-150 $\mu\text{m}$	11.51	72.79
150-250 $\mu\text{m}$	8.23	81.02
250-500 $\mu\text{m}$	7.72	88.74
500 $\mu\text{m}$ - 10 mm	11.27	100.0

FROM HANDBOOK OF LUNAR MATERIALS, NASA REFERENCE PUBLICATION 1067, P-84.

Figure 1. Grain size fractions for Apollo 11 soil No. 10084, 853.

PARTICLE TYPES	PETROGRAPHIC DESCRIPTION, VOLUME % FOR EACH SIZE RANGE								
	20 $\mu\text{m}$	20-45 $\mu\text{m}$	45-75 $\mu\text{m}$	75-90 $\mu\text{m}$	90-150 $\mu\text{m}$	150-250 $\mu\text{m}$	250-500 $\mu\text{m}$		
AGGLUTINATES**	17.0	17.3	13.0	17.3	9.3	11.8	10.0		
BASALTS/BRECCIAS	NON AGGLUTINATES**		11.9	23.2	25.4	42.2	60.4		
MINERALS			43.6	36.6	42.9	29.7	21.6		
GLASSES			83.0	82.7	28.3	22.4	21.8	15.6	8.4
OTHER			3.0	---	0.3	---	0.5		
TOTAL	100	100	100	100	100	100	100		
TOTAL NO. OF GRAINS COUNTED	300	161	300	300	300	178	130		
WT. % OF TOTAL SAMPLE FOR EA. SIZE FRACTION***	17.98	12.21	8.39	3.00	8.65	7.04	7.08		

\* FROM HANDBOOK OF LUNAR MATERIALS, NASA REFERENCE PUBLICATION 1067, P-87.

\*\* AGGLUTINATE VS. NON-AGGLUTINATE GRAINS WERE IDENTIFIED USING A SCANNING ELECTRON MICROSCOPE FOR PARTICLES < 45  $\mu\text{m}$  IN SIZE.

\*\*\* 26.40% BY WEIGHT OF THE PARTICLES IN THIS SAMPLE WERE IN THE 500  $\mu\text{m}$  TO 10 mm SIZE RANGE.

Figure 2. General petrography of particle types from a typical Apollo 17 mare soil No. 71061, 1.

Existing metallurgical processing options for extraction of these elements include: (1) Alcoa electrolysis or carbothermic reduction for production of aluminum from anorthite, (2) treatment in molten sodium hydroxide or carbochlorination followed by reduction of iron and titanium from ilmenite, (3) aluminothermic or silicothermic reduction of olivine for production of magnesium, (4) recovery of carbon and oxygen from carbon monoxide generated in the metal extraction processes, and (5) hydrofluoric acid leaching for dissolution and extraction of elemental constituents [3,6]. There are, however, some limitations inherent in these extraction processes which must be overcome. They include: importation of some reagents, potential leakage problems, need for recycling, high temperature of reaction, and thermal dissipation [3]. Since almost all extraction processes require that input material consist of essentially one mineral, isolation of each desired mineral (such as those found in lunar soil) is of great importance if any refining method is to be applied.

## BACKGROUND

Electrophoresis involves the transport of dissolved, or suspended, charged particles by an electric field. The electrostatic potential between the surface of shear surrounding the particle and the bulk suspension medium (i.e., between the layer of stationary ions and the bulk suspension medium), called the zeta potential, determines the migration rates of the particles. If the zeta potential is zero, the condition defined as the isoelectric point, the particle remains stationary. Electrophoretic separation is based on differential rates of migration of charged particles in a bulk suspension medium upon application of an electric field through the medium. Since the isoelectric points of various typical lunar minerals have been reported to differ appreciably, electrophoretic separation of mineral particles appears feasible [3].

The net charge at the surface of a particle in contact with a polar medium occurs as a result of the degree of ionization, ion adsorption, and/or ion dissolution. The ionization of surface groups, e.g., carboxyl and amino groups in the case of proteins, depends on the pH of the solution. Isoelectric points and mobilities will change when ion adsorption has a significant effect on the net surface charge of a particle. In the case of ionic substances, a surface charge may be the result of unequal dissolution of the oppositely charged ions of which the substances are composed [7].

Measurement of mobility is an analytical tool commonly used to indicate the expected separability of particles for preparative electrophoresis. If the distributions of mobilities of two sample constituents do not overlap appreciably under given experimental conditions, effective electrophoretic separation of the two minerals can be expected. The mobility of a particle is defined as the velocity, or rate of migration expressed in  $\mu\text{m s}^{-1}$ , per unit electrical field strength expressed in  $\text{V cm}^{-1}$ . By definition, negatively charged particles which migrate toward the anode have a negative mobility. A generally accepted standard is the human red blood cell in 0.15 M NaCl solution at pH 7.3, which has an average mobility of  $-1.08 \mu\text{m s}^{-1} \text{V}^{-1} \text{cm}$  (with a standard deviation of  $\pm 0.05$ ) [8,9].

In the past, electrophoresis of insoluble clays and minerals has been carried out using, almost exclusively, aqueous solutions of electrolytes as suspension media. The surface chemistry of vitreous silica and alumina has been investigated by using

both streaming potential and microelectrophoresis methods [10]. The variation with pH of the electrophoretic mobility of suspended particles has been determined by microelectrophoretic techniques for  $\text{Fe}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{SnO}_2$ ,  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , and a number of synthetic aluminosilicates [11]. A Beckman continuous particle electrophoresis system has been used to separate mixtures of kaolinite and montmorillonite clays into five or more fractions [12]. The influence of temperature on the electrophoretic mobility of quartz particles in phosphate-buffered saline has also been studied [13].

Although consideration has been given to the use of non-aqueous suspension media for electrophoretic separation of mineral particles, a survey of the open literature indicates that very little work has been done using non-aqueous media such as silanes or organics. The complexity of preparation of silanes and the instability and volatility of many of these compounds may make their use as fluid media for electrophoresis impractical. Work with silanes must be carried out in scrupulously dry, closed glass systems, rigorously protected from air, since silanes are flammable and hydrolyze readily. Volatility of silanes increases as molecular weight decreases, whereas stability of the compounds decreases toward the higher members. Light and heat cause an increase in the rate of decomposition of these higher members [14,15].

Organic liquids<sup>1</sup> might be used effectively depending on such properties and conditions as degree of solvation of electrolytes, reactivity, dipole moment, conductivity, ionic strength, viscosity, dielectric constant, and trace water content [16-19]. Among organic liquids which have been used to study electrokinetic properties of minerals are: n-pentanol with  $\text{TiO}_2$  particles [16]; n-heptane, cyclohexane, and benzene with carbon and  $\text{BaSO}_4$  suspensions [17]; heptanol and nitro-benzene with  $\text{TiO}_2$  and alumina-coated  $\text{TiO}_2$  [18]. Mobilities of clay particles have demonstrated increased separation potential when measured in an aqueous sodium carbonate buffer containing 5 percent ethylene glycol [12].

## MATERIALS AND METHODS

This study involves the measurement of mobilities of selected mineral components of simulated lunar soil in various fluid media. Minerals tested include ilmenite, labradorite plagioclase, olivine, and enstatite pyroxene, all of which also occur on Earth. The samples used were prepared by J. Dunning of Indiana University by mechanized agate grinding followed by sieving to isolate particles with dimensions less than 63  $\mu\text{m}$ . The general composition of each mineral is shown in Figure 3 [5,20].

Mobilities were measured in  $\mu\text{m s}^{-1} \text{V}^{-1} \text{cm}$  using a static analytical microelectrophoresis apparatus with a cylindrical chamber, manufactured by Rank Brothers, Cambridgeshire, England (illustrated in Figures 4 and 5) [21]. The chamber assembly was immersed in a controlled-temperature water bath. Temperature changes were effected by means of a Grant Instruments heater with a propeller and a metal plate for rapid heat distribution. Each suspension sample was introduced from a syringe into the electrophoresis chamber. The chamber consists of a glass capillary tube approximately 10 cm in length and 2 mm in diameter with parallel optical flats in the

1. Refers to those organics that occur as liquids at room temperature.

ORIGINAL PAGE  
BLACK AND WHITE PHOTOGRAPH

- |                               |   |
|-------------------------------|---|
| ● ILMENITE                    | $\text{FeTiO}_3$  |
| ● LABRADORITE<br>(A FELDSPAR) | $\text{Ca Al}_2 \text{Si}_2 \text{O}_8$ AND<br>$\text{Na Al Si}_3 \text{O}_8$<br>(SOLID SOLUTION) |
| ● OLIVINE                     | $\text{Mg}_2 \text{Si O}_4$ AND<br>$\text{Fe}_2 \text{Si O}_4$<br>(SOLID SOLUTION)                |
| ● PYROXENE                    | $\text{Ca Mg}^* \text{Si}_2^{**} \text{O}_6$  |

\* Cr, Mn, Fe, Ti, OR Al MAY BE SUBSTITUTED

\*\* Al MAY BE SUBSTITUTED

Figure 3. Lunar minerals.

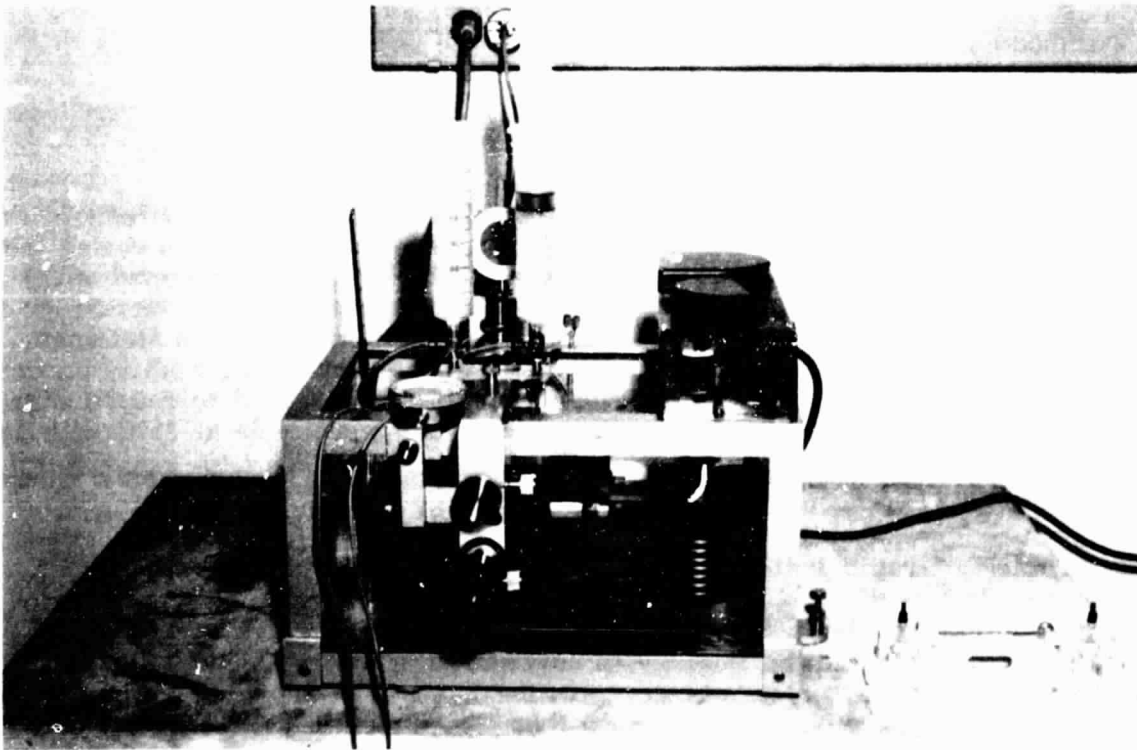


Figure 4. Rank Brothers microelectrophoresis chamber.



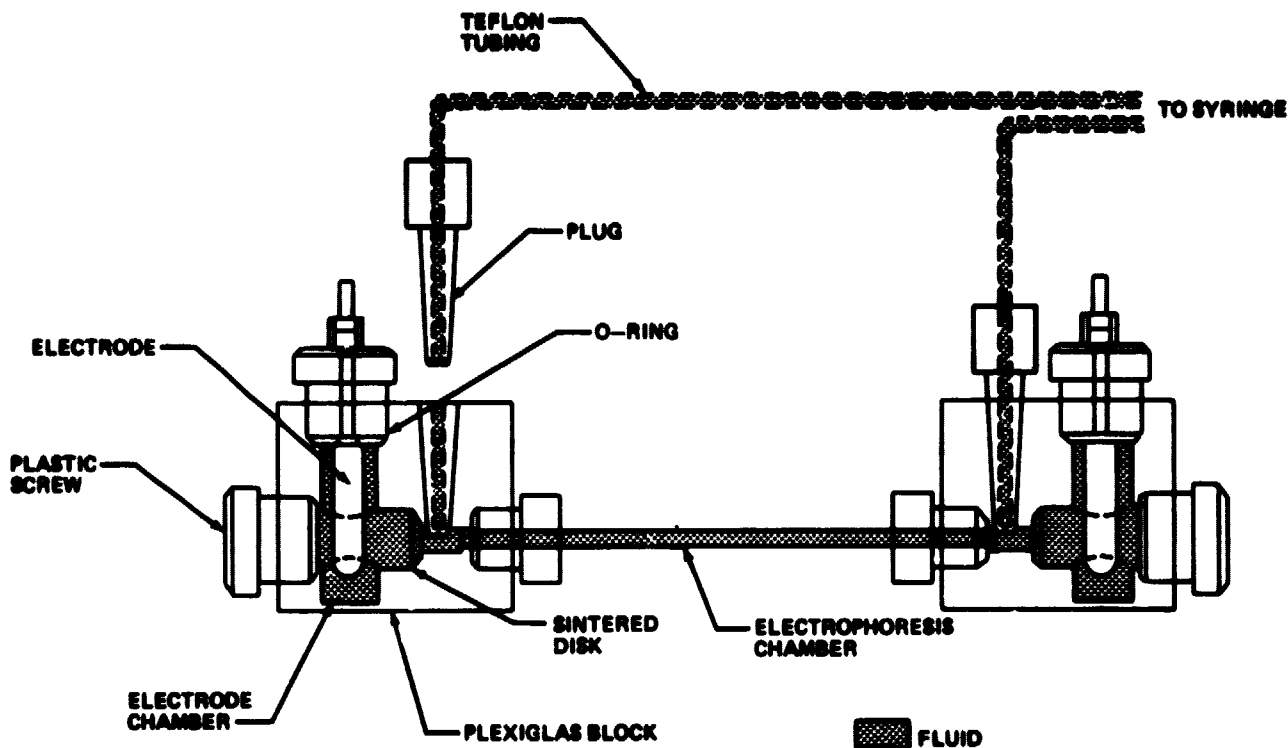


Figure 5. Rank Brothers microelectrophoresis chamber assembly.

outside walls through which a microscope can be focused. This electrophoresis chamber is separated from the electrode chambers by two sintered disks and is sealed from the sample insertion syringes by means of valves. The system is equipped with Ag-AgCl electrodes and a d.c. power supply. A 400X Bausch and Lomb microscope is focused onto the sample suspension in the electrophoresis chamber at the stationary level, i.e., the depth in the chamber where no net flow of suspending medium occurs when a voltage is applied. A timer which measures to 0.1 s was used to record migration times of mineral particles. All measurements in this study were made at 25°C with an electric field of  $3.4 \text{ V cm}^{-1}$  across the electrophoresis chamber. Measurements were made immediately after suspension of mineral particles in each fluid medium.

The problem of rapid settling which occurs with larger particles in Earth gravity restricted observation to mineral particles with dimensions of 1 to 4  $\mu\text{m}$ . This mineral particle size is in the same size range as that used in some earlier studies [13,22]. In this range, mobility is considered to be independent of particle size [23,24]. Although distorted velocity profiles have been reported for cylindrical cell microelectrophoresis caused by settling of larger particles [22], this phenomenon is not considered a problem in the present study because the concern is for relative rather than absolute mobilities.

## DISCUSSION AND EXPERIMENTAL RESULTS

The experimental procedure was divided into four parts.

### Part I

Previous experimental results indicated that pH values of 0.15 M NaCl solutions adjusted to pH 7.3 with 0.1 M HCl, 0.1 M NaOH [11], or 0.10 M NaHCO<sub>3</sub> [8] did not remain constant upon addition of mineral samples. Therefore, it was decided to use a buffer solution of 0.15 M NaHCO<sub>3</sub> (in equilibrium with the atmosphere at a pH of 9.4), as the fluid medium for initial measurements. To provide equal volumes of each mineral suspended for these and subsequent determinations, the following masses of each mineral component were suspended in 5 ml of each fluid medium:

Ilmenite	10 mg
Labradorite plagioclase	6 mg
Olivine	8 mg
Eustatite pyroxene	7 mg

Mobilities of 50 particles of each of these minerals were determined in the NaHCO<sub>3</sub> buffer.

The results are shown in the histogram illustrated in Figure 6. The number of particles is plotted against mobility range. All four minerals show good central tendency of distribution under these conditions. Good separability is indicated between labradorite plagioclase and olivine. Each of these in turn shows partial separability from both ilmenite and eustatite pyroxene. However, ilmenite and pyroxene appear inseparable under these conditions.

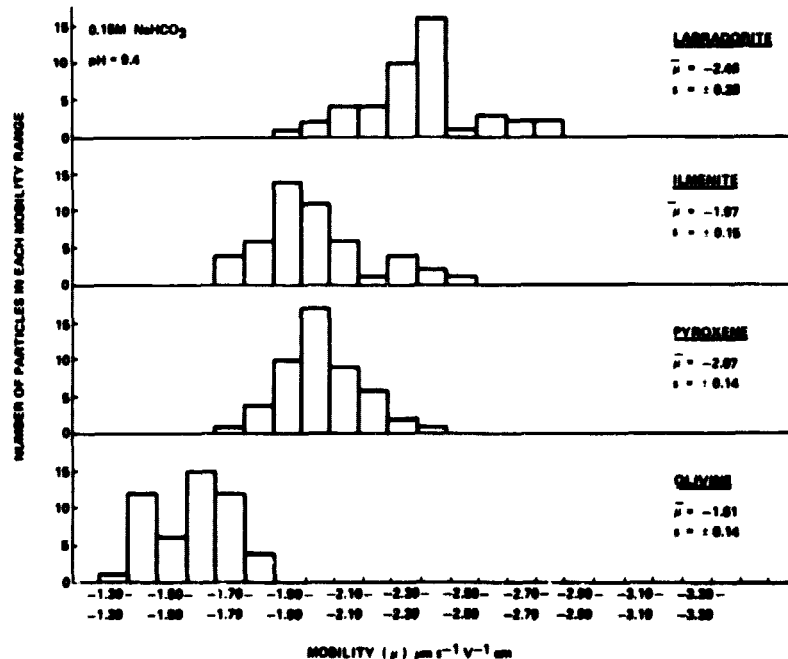


Figure 6. Electrophoretic mobilities of minerals.

## Part II

To show the effect of a medium with lower ionic strength on these four minerals, mobilities of 50 particles of each mineral were determined in 0.015 M  $\text{NaHCO}_3$  buffer solution with a pH of 9.6. As shown in Figure 7, the mean absolute mobility for each mineral differs from the previous determinations at the higher ionic strength; but the relative mobilities for ilmenite, labradorite plagioclase, and enstatite pyroxene do not indicate enhanced separability; they, in fact, indicate poor separability and less central tendency of particle distribution. Olivine, under these conditions, appears to have good separability from the other three minerals.

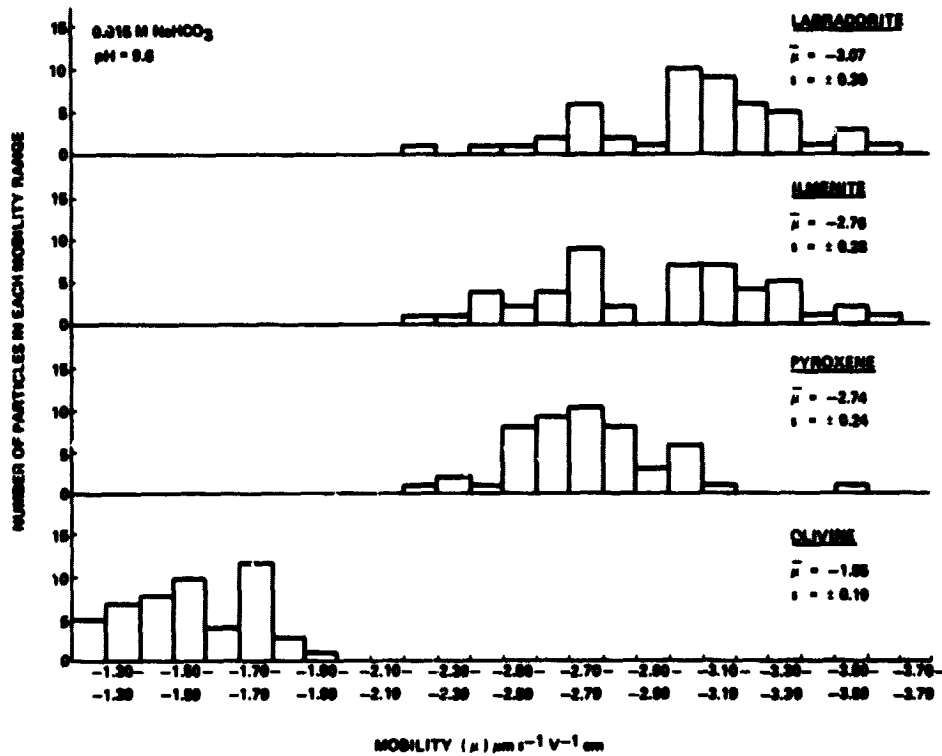


Figure 7. Electrophoretic mobilities of minerals.

## Part III

Since the cationic surfactant, dodecyl trimethyl ammonium bromide (DTAB), has proven effective in reducing surface energy of minerals by increasing the ratio of adsorbable species to surface sites, there was an interest in determining whether DTAB would affect mobility of mineral particles [25]. Mobilities of 50 particles of both ilmenite and enstatite pyroxene were determined in 0.15 M  $\text{NaHCO}_3$  buffer medium (at a pH of 9.4) which was  $1 \times 10^{-4}$  M in DTAB. As shown in Figure 8, results indicate that although there is a change in mean absolute mobilities, separability is not enhanced.

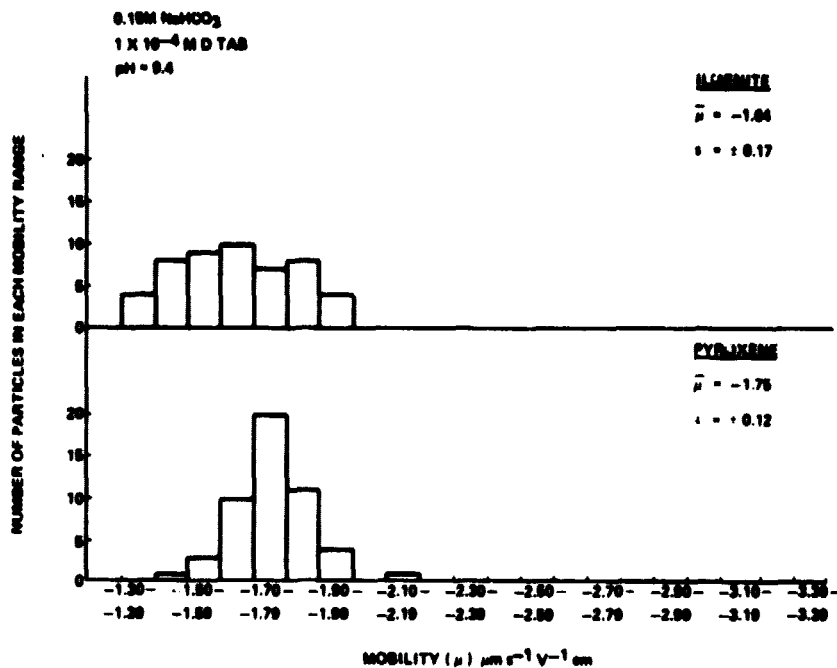


Figure 8. Electrophoretic mobilities of minerals.

#### Part IV

Buffer additives which might be differentially adsorbed by the minerals, resulting in modifications of their relative mobilities, were next considered. Based upon a report of improved separation capability of clay particles when measured in buffer containing ethylene glycol [12], it was decided to determine mobilities of ilmenite and enstatite pyroxene particles using a bulk medium of 0.15 M NaHCO<sub>3</sub> with pH of 9.4 containing 2 percent (v/v) ethylene glycol. Mobilities of 50 particles of each mineral were measured. Again, the mean absolute mobilities changed; but the relative mobilities of ilmenite and enstatite pyroxene do not indicate good separability, as shown in Figure 9. The viscosities of the original buffer solution and the buffer solution with 2 percent ethylene glycol were the same for all practical purposes, both measuring 1.1 centipoise at 25°C with a Brookfield microviscometer.

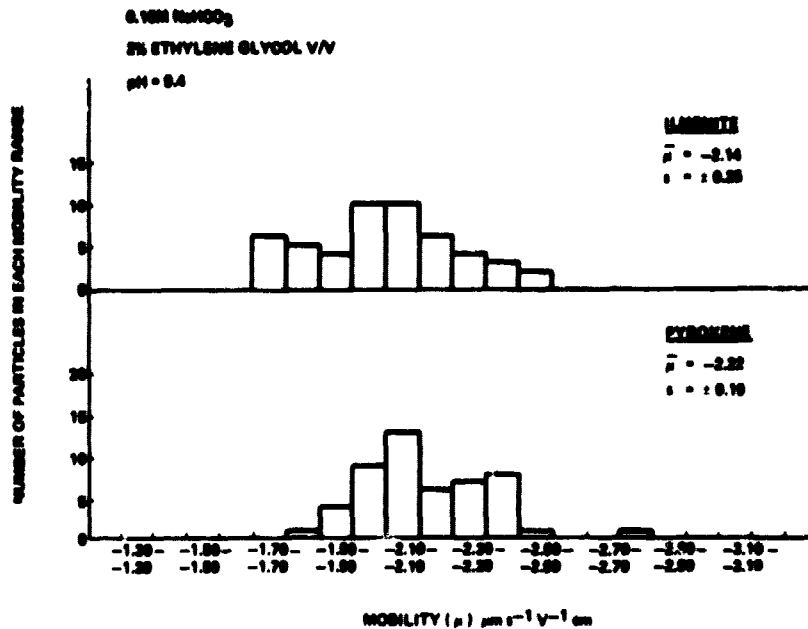


Figure 9. Electrophoretic mobilities of minerals

## CONCLUSIONS

Mineral particles are more versatile than biological materials for electrophoretic experiments because they are less fragile, i.e., are not subject to the traumas experienced by biological materials with changes in conditions such as pH or osmolarity of suspension media. The results indicate that several important mineral species are separable under the given conditions. Since mobility is a function of pH, it is important to use an effective buffer system as the suspension medium. Variation of experimental conditions - decrease in the ionic strength of the medium, addition of a surfactant (DTAB), or addition of an organic liquid (ethylene glycol) - caused changes in the absolute particle mobilities of the minerals studied but did not enhance mobility differences (separability) between species. It is to be expected the particle surface properties, and hence properties of the electrical double layer in aqueous media, could be considerably influenced by the previous history of a particular mineral specimen [11]. Mobilities of mineral components of actual lunar soil samples may, therefore, differ from the prepared samples used in this study.

It is anticipated that the separability of the four minerals used in this study and that of simulated lunar soil will be investigated further by fractionation of suspensions in a continuous flow electrophoresis system using selected buffers.

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# APPROVAL

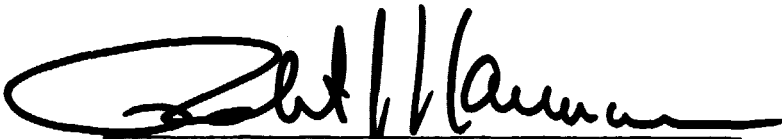
## MICROELECTROPHORESIS OF SELECTED MINERAL PARTICLES

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The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.



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