NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

## Technical Memorandum 33-521

# Fixation of Virgin Lunar Surface Soil

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# PREFACE

The work described in this memorandum was performed by the Space Sciences Division of the Jet Propulsion Laboratory.

# ACKNOWLEDGMENTS

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# ABSTRACT

Two systems have been shown to be suitable for fixing loose particulate soils with a polymer film without visually detectable disturbance of the soil particle spatial relationships. A two-component system uses a gas monomer condensible at the soil temperature and a gas-phase catalyst acting to polymerize the monomer. A one-component system uses a monomer which polymerizes spontaneously on and within the top few millimeters of the soil.

The two-component system may result in a simpler apparatus, but has been demonstrated to operate over a narrower temperature range, approximately -40 to -10 °C. Other two-component polymer systems have been identified which may operate at soil temperatures as high as +100 °C, but still over relatively narrow temperature ranges of approximately 30 °C.

The one-component system has been demonstrated to operate successfully with initial soil temperatures from -70 °C or lower to +150 °C.

# I. INTRODUCTION

The microstructure of the lunar surface is known only to the extent revealed by stereo photographs at a resolution of approximately 80 µm (Ref. 1). The Surveyor surface sampler operation, with television monitoring, furnished soil mechanics data at depths greater than several millimeters (Ref. 2), but the soil mechanics and morphology of the top few millimeters are still largely unknown. Some physical characteristics have been inferred from visible, infrared, and radio-frequency measurements, both active and passive (Refs. 3 to 5), but several interpretations of the data are possible.

Sampling of the lunar surface for sample return has unfortunately resulted in destruction of the microstructure of these top few millimeters of the soil sample which are expected to be extremely tenuous and perhaps influenced by static charge, interlocking reentrant surfaces, and Van der Waals' forces. Preservation and sample return of the lunar surface soil in its virgin configuration would be of interest not only in and of itself, but also as an aid in the interpretation of other phenomena. Fixation (in the sense of microscopy) of the soil requires that it be at least partially encapsulated by a fluid which conforms to the virgin configuration without disturbing it. Pouring of a liquid would certainly destroy the fine structure, so that deposition of a fixative from the gas phase is the only appropriate method of fixation. Evaporation or sputtering of a metal onto the surface has been considered, but due to the high sticking coefficient of the metal, only line-of-sight deposition would be achieved, resulting in fixation of only the top monolayer of soil.

As a result of these considerations two polymer systems in which organic monomer vapors are polymerized during or after condensation on and within the soil have been investigated and tests to fix soil under simulated lunar conditions have been successful.

At least one of these organic vapors has an extremely low sticking coefficient, of the order of $10^{-3}$ at room temperature, and penetrates into the soil to a depth of 2 to 3 mm before condensing upon and fixing the soil.

## II. OBJECTIVES

Since it would seem to be impossible to return an undisturbed sample of what must be an extremely fragile structure, fixation is the only technique (other than difficult in situ experiments) that might be used to study the microstructure of the surface and its significance relative to other experiments.

A large proportion of the experiments performed on soil samples returned by the Apollo spacecraft involves the sample depth and/or orientation; therefore, documentation of the sample by fixation is applicable to many of these experiments. Also of interest is potential applicability to Earth-based measurements of lunar surface properties and to other studies such as lunar erosion processes.

Examples of specific applications are:

### A. Lunar Surface Physical Properties

The structure and composition of the top few millimeters of lunar soil have been topics of investigation since Epstein (Ref. 6) first proposed a porous surface material in explanation of the rapid lunar eclipse cooling first observed by Pettit and Nicholson in 1927 (Ref. 7). However, infrared measurements alone do not provide adequate information for determination of a thermal model of the lunar surface. Independent measurements of photometric properties, thermal conductivity, density, and specific heat as a function of depth would be required to define completely a near-surface thermal model. Current theoretical models do not fit the infrared data without physically meaningless parameter adjustment (Ref. 8). Studies of the morphology and composition of the surface could be conducted on fixed samples. Soil mechanics and other aspects of the physical properties would, of course, not be preserved, although studies of the sample configuration might allow inferences with respect to these properties.

### B. Gardening

Studies of nuclear track densities and solar wind abundances as a function of depth will be possible with fixed samples, thereby eliminating any uncertainty in the results of these experiments due to mixing in the upper part of the sample.

### C. Erosion

This heading is differentiated from gardening in that the erosion processes might be studied by means of fixation of key features such as the boundaries between rocks and soil.

## III. TECHNIQUES AND SYSTEMS CONSIDERED

### A. Fixation Techniques

Several polymer systems and three techniques for application of the polymer to the soil have been considered.

The first and most thoroughly tested technique utilizes an inverted funnel, the rim of which is pressed into the soil so that some degree of sealing is effected. The funnel is capable of sustaining an inside pressure of approximately $1.3 \times 10^3$ N/m$^2$ (10 torr) relative to the ambient pressure without blowout of the soil. A basalt rubble, crushed from lava flow material from Little Lake, California, was used for all of the fixation tests. The particle-size distribution for the soil is shown in Fig. 1. Although the distribution is appreciably coarser than that of the lunar nominal soil, the native physical configuration of lunar soil could not be reconstructed in any case, so that matching the particle-size distribution was not considered critical to the validity of the fixation tests. Tests with such material would, of course, be required prior to flight application of a fixation experiment.

The second technique employs a nozzle to direct the gaseous fixative at the soil. It was anticipated that, should this technique prove feasible, it might be employed in places inaccessible to a funnel, such as an erosion fillet adjacent to a rock.

A third technique which has been considered, but not tested, would employ a tube with porous walls that is pressed into the soil. The fixative would be deposited through the walls of the tube and the tube would be withdrawn with the sample adhering to it. Since it is probable that insertion of the tube would considerably disturb the soil, and since the primary application of fixation is considered to be the top few millimeters of soil, this technique has not been investigated.

### B. Soil Permeability

The major part of the work reported here involved the inverted funnel technique. It appears most likely to be successful, since it is closest to duplicating Earth laboratory conditions (i.e., pressure and temperature) on the lunar surface. Accordingly, prior to the fixation tests the gaseous conductivity (permeability) of vacuum-baked soil was measured with air utilizing the funnel technique. Due to apparatus limitations and the fact that working above 53 $N/m^2$ (400 mtorr) differential was not anticipated, the measurements were restricted to that pressure range. Figure 2 is a sketch of the apparatus used for the measurements and Fig. 3 is a plot of the soil gaseous throughout as a function of differential pressure for two cases; the funnel rim being pressed lightly into the soil, and the soil being piled up around the outside of the rim. If one assumes that the soil below the funnel edge can be considered infinite in extent, the specific conductance of the soil, neglecting edge and end effects, is

$$G = \frac{Qd}{Ap}$$

where Q is the throughput, d the path length to the funnel edge (Fig. 3), A the exposed soil area, and p is the funnel pressure.

$$G = \frac{0.44\ (N/m^2)\ m^3/min \times 5 \times 10^{-3}\ m}{1.33 \times 10^{-2}\ m^2 \times 53\ N/m^2}$$

$$= 3.1 \times 10^{-3}\ m^3/min$$

where the area of the soil exposed to the funnel interior was 133 $cm^2$ and the funnel rim was buried 0.5 cm.

Data presented in Section IV of this report indicate that 5% by weight of polystyrene is more than adequate to produce good soil fixation. If it is assumed that 1% fixative is adequate for preservation of the lunar soil morphology for sample return and that the <u>in situ</u> density of the soil is $1.5 \times 10^{-3}\ kg/cm^3$, then it is necessary that $0.005 \times 10^{-3}\ kg/cm^2$ of fixative permeate the soil for fixation to a depth of 0.3 cm. Assuming the permeability of the soil for the gaseous fixative to be the same as that for air, and utilizing the equation:

$$Q = \frac{m}{t} = \frac{G_M A \Delta P}{d}$$

where m/t is the flux rate, the specific conductance for M = 104 (the molecular weight of both of the organic monomers of interest) is given by:

$$G_M = 3.1 \times 10^{-3} \frac{(N/m^2)\ m^3/min}{N/m^2} \times 4.58 \times 10^{-5} \frac{kg}{(N/m^2)\ m^3}$$

$$= 1.42 \times 10^{-7} \frac{kg/min}{(N/m^2)}$$

$$\frac{m}{A} = 5 \times 10^{-2}\ kg/m^2$$

$$\Delta P = 67\ N/m^2\ (500\ mtorr)$$

$$d = 3 \times 10^{-3}\ m$$

$$t = \frac{md}{G_M A \Delta P}$$

$$= 15\ min$$

The above calculation is of limited value since, relative to air, the fixative is condensible and of vastly different adsorptive properties. Moreover, the point has been made that permeation of fine soil by a gas near the soil surface will eventually seal the surface, thus limiting the depth of penetration which can be achieved under given conditions.

It would be expected that the depth of penetration could be increased by maintaining the surface relatively hot during the initial phases of deposition, thus preventing the fixative from sealing the surface and allowing greater penetration, then cooling the surface during deposition to complete the process. However, such a process is not quite so straightforward. Both permeation and deposition rates are functions of temperature, and so, for a fixed pressure, the

$$\text{permeation rate} = \frac{m}{t} = f(T)$$

$$\text{deposition rate} = g(T)$$

Penetration can then be expected to increase with temperature in the region where

$$\frac{\delta f}{\delta T} > \frac{\delta g}{\delta T}$$

Deposition times will, of course, increase with temperature in any case.

The function f(T) can be expected from kinetic theory to be proportional to $T^{-1/2}$ if the flow can be considered molecular, but may be proportional to $T^{1.5}$ to $T^{2}$ if the process is diffusive. The deposition rate dependence on temperature is of the form

$$g(T) \propto \alpha/T$$

It would be necessary to determine both functions in order to infer the effectiveness of temperature gradients on depths of penetration. For the purpose of the work reported here a direct measurement of the effects of temperature on depth of penetration is preferable. However, it should be noted that, for permeation and deposition rates of the forms mentioned, there will be an optimum temperature for maximum penetration.

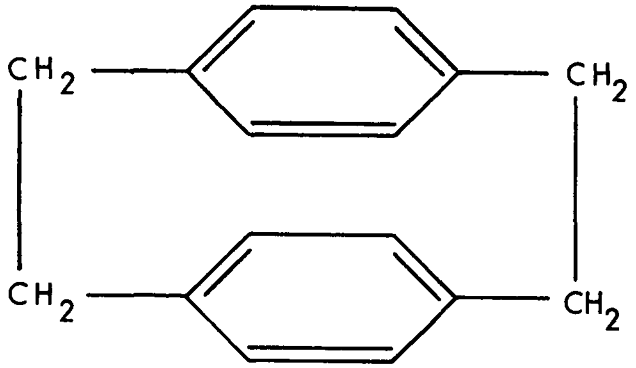
## C. Polymer Systems Considered

Brief descriptions of each of the systems which have been considered are:

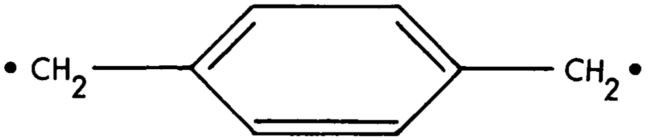
1. Monomer/Catalyst Systems. Although a large number of both monomers and catalysts exist in the vapor phase and can be condensed onto soils to form polymers, only the styrene/$BF_3$ system has been tested during this program. In monomer/catalyst systems the polymerization reaction

takes place upon exposure of the monomer to the catalyst, and the reaction has proceeded best when the monomer is first condensed onto the soil in the liquid phase and then exposed to the catalyst.

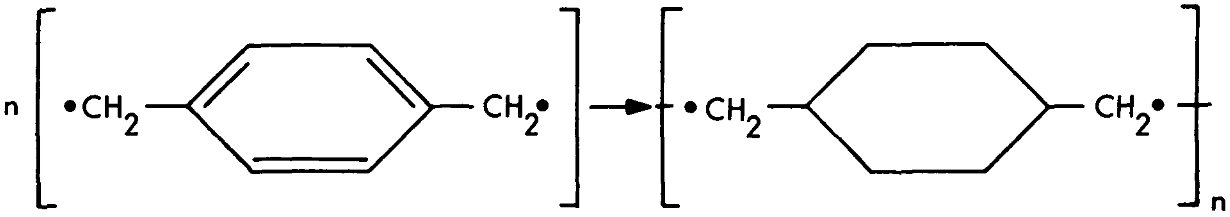
2. Parylene.[1] The Parylene system which has been used in these studies is Parylene-N. The Parylene dimer (di-p-xylylene) is available from Union Carbide Corp. as a stable white crystalline material:



This pure chemical is sublimed and then pyrolyzed in the vapor phase to yield the Parylene monomer divalent radical:



Cooling of the monomer vapor to a temperature below ~100 °C results in spontaneous polymerization to yield the Parylene polymer, poly-para-xylylene:



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[1] Trademark of Union Carbide Corp.

The number of repeating units, or "n," is generally in the range of 5000; the molecular weight, therefore, is about 500,000. Further details on the properties of Parylene can be found in Ref. 10.

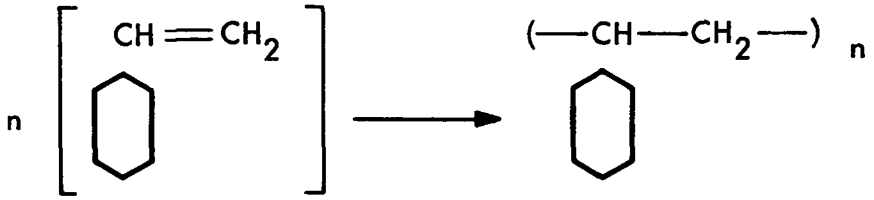
3. Photopolymerization. A wide variety of monomers has been polymerized by exposure to ultraviolet light. Several tests were performed in which a monomer was condensed into the soil and then exposed to ultraviolet rays. Although polymerization was obtained for a thin surface film, the reaction did not propagate into the soil sample.

4. Polymerization by High-Energy Radiation. Polymerization by means of exposure of a monomer to X- or $\gamma$-rays has been suggested but has not been investigated due to an assessment that this system might prove to be less attractive than others from a size and weight standpoint and due to manned flight personnel safety considerations.

## IV. MONOMER/CATALYST PROCESSES AND RESULTS

### A. Styrene Polymerization

The polymerization of styrene is a chain reaction that can be readily initiated thermally or by the aid of various catalysts. The reaction can be depicted schematically as follows:



During polymerization the double bonds disappear and heat is evolved amounting to $160 \times 10^3$ cal/kg which is almost equivalent to the heat of hydrogenation of the double bonds. The density increases from about $0.90 \times 10^{-3}$ kg/cm$^3$ to about $1.045 \times 10^{-3}$ kg/cm$^3$ for the polymer. The molecular weight of the polymer may range from a few thousand to over one million, depending on the polymerization conditions.

Thermally initiated polymerization of styrene proceeds rather slowly unless fairly high temperatures are used, in which case the reaction is apt to become uncontrollable. Peroxide and other free radical catalysts are able to speed up the polymerization reaction at moderate temperatures but still yield only fairly slow reactions.

Catalysts such as $BF_3$, $SnCl_4$, and $AlCl_3$ cause the polymerization reaction to proceed by an ionic rather than a free radical mechanism. These catalysts are extremely reactive, even at low temperatures. Because of its high activity in catalyzing polymerization of styrene at low temperature, and its ease of handling as a gas, $BF_3$ was chosen as the preferred catalyst in this investigation.

### B. Mechanism of Soil Fixation With Styrene

A hypothesis for the mechanism of soil fixation by styrene polymerization is proposed as follows: When the soil mass is exposed to styrene vapor at a pressure approaching the saturation pressure at any given temperature, styrene is physically adsorbed on all the available surfaces to a thickness of a few monolayers. More importantly, capillary condensation takes place in the sharply reentrant angles near the points of contact between grains. Polymerization of the condensed styrene in situ will form solid bridges at grain interfaces. The important feature of the mechanism is that capillary condensation can take place below the saturation pressure of styrene at the correct sites to effect bonding. Hence, it is unnecessary to saturate the soil mass with an excessive amount of styrene to achieve the desired consolidation.

Condensation takes place in the reentrant region near contact points by virtue of the reduction in vapor pressure over a concave liquid meniscus. The extent of lowering of vapor pressure over a curved meniscus finds expression in the Kelvin equation:

$$\ln (P/P_0) = \frac{-2\gamma V}{rRT} \cos \theta$$

Where r is the radius of curvature of the meniscus, $\gamma$ is the surface tension of the liquid, V is the molecular volume, $\theta$ is the contact angle of the liquid against the solid, P is the equilibrium vapor pressure over the curved meniscus and $P_0$ is the vapor pressure of the bulk liquid. Although the Kelvin equation was originally derived for condensation in a cylindrical capillary tube, it holds in general for any kind of pore structure, including slots and parallel plates.

If we depict two grains in contact as shown in Fig. 4, the diameter of the neck, D, is a function of the radius of curvature, r, which in turn is determined by the relative pressure of styrene vapor $(P/P_0)$ in the system.

It is assumed here that the significant radius of curvature is the one in the plane cutting the center of the two spheres. The height of the spherical segment, h, is taken as approximately equal to r, the so-called Kelvin radius.

The Kelvin radius required to bring about a reasonable reduction in vapor pressure is quite small. Nevertheless, in fine-particle systems vapor pressure reduction is quite easily observed. Using the Kelvin equation, relative lowering of vapor pressure for different radii are calculated as in the following:

$$r = \frac{-2\gamma M}{\rho R T (\ln P/P_0)} \cdot \cos\theta$$

For styrene at 0 °C

$$\gamma = 3.45 \times 10^{-2}\ \mathrm{J/m^2}$$

$$M = 104$$

$$\rho = 9.24 \times 10^{2}\ \mathrm{kg/m^3}$$

$$\cos\theta = 1 \text{ (assuming complete wetting)}$$

$$R = 8.31 \times 10^{3}\ \mathrm{J/deg\text{-}kg\ mole}$$

from which

$$r = -\frac{1.49 \times 10^{-9}}{\log (P/P_0)}$$

Kelvin radii for various values of relative pressure for styrene at 0 °C.

| $P/P_0$ | r, μm |
|---|---|
| 0.7 | $0.96 \times 10^{-2}$ |
| 0.8 | $1.54 \times 10^{-2}$ |
| 0.9 | $3.24 \times 10^{-2}$ |
| 0.95 | $6.68 \times 10^{-2}$ |
| 0.99 | $3.42 \times 10^{-1}$ |
| 0.999 | $3.5 \times 10$ |

The neck diameter (or the neck area) can, therefore, be approximated for any idealized particle system for a given relative pressure of styrene vapor. For contact between two spherical particles with radii $\mathscr{R}$,

$$r \cong h = \mathscr{R} - \frac{\left(4\mathscr{R}^2 - D^2\right)^{1/2}}{2}$$

For 10-μm radius particles, the following values have been calculated:

| $P/P_0$ | Neck diameter D (μm) | Neck diameter, % of particle radius |
|---|---|---|
| 0.7 | 0.8 | 8 |
| 0.8 | 1.1 | 11 |
| 0.9 | 1.6 | 16 |
| 0.95 | 2.3 | 23 |
| 0.99 | 5.2 | 52 |

Thus significant styrene bridges can be formed at pressure well below the saturation pressure of styrene.

It is believed that the mechanism described should be generally applicable whether the catalyst required to effect polymerization is added after the capillary condensation or is present in the system during condensation. There is no evidence for extensive vapor-phase polymerization; hence, it is proposed that condensation takes place as described, but the styrene is rapidly polymerized as it condenses by catalyst diffusing into the liquid phase. Initially the $BF_3$ is thought to be chemisorbed on the solid surfaces if added <u>before</u> the styrene is introduced. Adding the $BF_3$ and styrene <u>simultaneously</u> need not change the basic mechanism. Styrene will condense and be polymerized by $BF_3$ dissolving in it from the vapor phase.

Experimental evidence indicates that addition of the styrene <u>first</u> to the system, followed by catalyst addition, results in a more uniform distribution of polymer in the soil system. It is believed that this is due to the self-regulating properties of the system. The soil mass at the surface receives the styrene vapor first and styrene will condense there first. However, the heat liberated will increase the vapor pressure at this region and the vapor will tend to seek out cooler portions of the system. Therefore, when fixed equilibrium conditions are achieved, there will be a tendency for fairly uniform distribution of the styrene in the condensed phase. This process is upset if polymerization takes place fairly rapidly as styrene condenses, because the vapor pressure is reduced by polymerization. The net result is that the distribution is likely to be uneven; in fact, this is observed experimentally.

## C. Apparatus

The apparatus shown schematically in Fig. 5 was constructed for use in this phase of the investigation. A high-speed oil diffusion pump was used to evacuate the bell jar. An external manifold, with separate vacuum system, was used for feeding measured quantities of monomer vapor and catalyst into the bell jar.

The base plate of the bell-jar system contained a brass disk that served as a heat sink for the Peltier cooler which was affixed to it. The heat sink was equipped with an internal coil through which water or other coolant circulated. A metal tray or cup was placed on top of the Peltier cooler and contained a layer of simulated lunar soil.

Temperature control of the soil sample was effected by regulating the temperature of the heat sink by circulation of coolant and by adjusting the power input to the Peltier cooler. By reversing the current through the Peltier device it was used to heat the sample as well as cool it. The temperature of the heat sink, top plate of Peltier cooler, and soil sample were measured with thermocouples and temperatures were continuously recorded during experiments. Because of the low thermal conductivity of the soil, about 500 $N/m^2$ (4 torr) helium pressure was usually established temporarily in the bell jar to assist in cooling the soil sample.

## D. Experimental

### 1. Open-Cup Experiments.

This phase of the investigation was designed to determine feasibility of polymerizing styrene in a soil mass to effect its consolidation. A weighed quantity of soil was placed in a metal cup and placed on the top plate of the Peltier cooler. The configuration is shown in Fig. 6.

The soil sample was heated to about 60 to 80 °C in a vacuum to degass it. Then it was cooled below room temperature, the valve to the vacuum pump and cold trap was closed, and styrene vapor admitted to the bell jar.

After 10 min, $BF_3$ gas was added to the bell jar to a pressure of several hundred $N/m^2$ (several torr).

The operating parameters and results of the various open-cup experiments are tabulated in Table 1. The first two experiments failed to yield significant fixation of the soil, but the poor results are attributed to accidental displacement of the thermocouple during assembly, therefore the soil temperature never reached the indicated value. Styrene vapor condensed preferentially on the Peltier cooler and copper plate and hardly at all in the soil. In later experiments, judicious placement of radiation shields, and some slight warming of the Peltier cooler immediately before adding the styrene vapor, insured that the soil was initially the coldest part of the system; hence, styrene would preferentially condense on and in it. Under these conditions good consolidation of the soil was effected in several runs. (Run 7 in Table 1 is cited as a typical good run).

The best results were obtained when the styrene was added to the system first and polymerized in situ. The progress of addition of the styrene and its subsequent polymerization was readily followed by observing the temperature of the soil (Fig. 7). During condensation of the styrene, there is a steady increase in temperature due to liberation of the heat of condensation. Upon addition of $BF_3$, there is a steep exotherm due to the heat of polymerization.

2. Closed-Cup Experiments. Although the open-cup experiments described in the preceding section demonstrated the feasibility of soil fixation with polystyrene, the approach does not represent a practical method of soil fixation in the lunar environment, because pressure of reactants cannot be maintained in high vacuum. Attention was directed, therefore, to an approach in which a small inverted cup was pressed into the soil in vacuum, and the reactants introduced to the interior of the cup.

The closed-cup experiments were generally successful in effecting consolidation of the soil, except when poor sealing around the edge of the cup allowed the reactants to escape too rapidly. This problem led to design of three different cups. The type shown in Fig. 8, and to some extent a similar type (not shown) involving an added baffle in the soil, failed to provide reliable sealing. The weakness of these designs appears to be lateral displacement of the cup when lowered into the soil as the knife edge encounters large particles. Unable to penetrate a large particle, the cup tends to slide sideways with excessive disturbance of the soil. The closed cup of the type shown in Fig. 9 was generally more successful. Less soil is disturbed and a longer diffusion path is presented to the reactants. Further reliability was insured by screening the soil to remove large lumps.

The various parameters investigated are outlined in Table 2 for the closed-cup experiments. Good soil fixation was obtained at various temperatures from 0 to -33°C (initial soil temperature). Again the best results were obtained by addition of styrene first followed by $BF_3$. When the catalyst was added first, the tendency was to obtain nonuniform distribution of polymer.

3. Open-Nozzle Experiments. The next phase of the investigation involved use of a supersonic nozzle to introduce the monomer-catalyst mixture. The experimental nozzle and relationship to the condensing surfaces are depicted schematically in Fig. 10. The first two experiments were conducted with a polished aluminum plate instead of the tray holding the soil sample.

In Run 1 (Table 3) a 12-mm ID nozzle was used and only a trace of polystyrene was collected on the plate which was maintained at -60°C during the run. Reduction of the nozzle diameter to 8 mm for Run 2 led to collection of 50 mg of polystyrene on the plate. This amounted to about 10% of the total styrene fed through the nozzle in 60 min. After this encouraging result, the experiment was repeated with a thin layer of simulated lunar soil. Unfortunately, no significant fixation of the soil was obtained The polystyrene content of the soil was 0.1% by weight, which is insufficient to fix the soil. It is probable that the soil temperature, at least at the surface, does not remain at low enough temperature during the experiment.

Later experiments involved increasing the nozzle pressure and attendant flow of reactants. It was necessary to install a liquid-nitrogen-cooled shroud around the walls of the bell jar to maintain the system pressure at a low level with the high reactant flow rate. In the final experiment, boron trifluoride etherate was used as a catalyst. Being less volatile than $BF_3$, it was thought that it might be more effective under these rapid pumping conditions. None of the open-nozzle tests proved successful.

## E. Other Two-Component Systems

The utility of a two-component system depends on the monomer condensing from the gas phase. This implies that the substrate be near the condensation temperature but not so cold as to freeze the monomer and thus inhibit its polymerization.

The material (styrene) chosen for this study satisfies these conditions for the immediate range of the lunar surface temperature, but would not be suitable for a higher temperature substrate, since it would pass through the soil as a gas without condensing. This constraint prompted a brief search for candidate materials whose boiling points would be near 100°C at the prevailing low pressures.

The search revealed a family of at least 17 styrene derivatives with boiling points ranging from a low of 60°C at $2.26 \times 10^3$ $N/m^2$ (17 torr) ($\alpha$ methylstyrene) to a high of 277°C at $10^5$ $N/m^2$ (2-nitro-styrene). The literature was meager in boiling point data at other pressures, but extrapolation suggests that several materials would be candidates for the 100°C range.

All the styrene derivatives would be catalyzed by boron trifluoride and this suggests the possibility of a phased system using two or more monomers. If a low boiling monomer were released and did not produce the typical heat of condensation, a progressively higher boiling material could be released until the condensation was noted by the operator, or was used to automatically trigger the catalyst release.

It follows that any device designed to utilize these materials must be maintained at a temperature high enough to avoid condensation in the valving, manifolds, and nozzles. This could reasonably be accomplished by proper thermal design of the apparatus.

The epoxies were identified as another attractive family of materials. One of these, phenyl glycidyl ether, has a boiling point of 138°C at 400 $N/m^2$. Its handling parallels that of the styrenes, but catalysis might best be accomplished by an amine compound, again in the gas phase.

A system of this class would have the possible advantage of matching the chemistry and refractive index of the post-mission embedding for thin sections. A disadvantage might stem from the need for heating the catalyst source (not required with boron trifluoride).

It is reasonable to expect that other two-component systems exist and should be studied along with those already identified.

# V. PARYLENE PROCESS AND RESULTS

## A. Parylene Process

It was stated in Section III that the Parylene monomer polymerizes "spontaneously" upon cooling to a temperature below 100°C. The mechanism of polymerization is not known in detail, but the material does not pass through the liquid phase during polymerization and thus should exhibit no effects due to surface tension, as might be expected with processes in which a liquid contacts the soil. The quantitative significance of this detail is not presently known.

## B. Apparatus

Figure 11 shows the apparatus used in the Parylene fixation tests employing the funnel technique. The pyrolyzer is maintained at a temperature of 500 to 680°C, and the evaporator (sublimator) is controlled at a temperature less than 200°C such that the indicated pressure measured at the pyrolyzer vacuum gauge is approximately 13 $N/m^2$. In order that the reaction process proceed properly, it is also necessary that the Parylene flow rate through the pyrolyzer be correct. It has been found experimentally that the 10-mm vent shown on the funnel results in a flow rate of approximately 0.003 kg/h at a pyrolyzer pressure of 13 $N/cm^2$ and that a polymer of good quality is produced under these conditions. From the conductance equation given in Section III, exposure of the soil to the monomer under these conditions will result in a permeation rate of approximately 0.0002 kg/h for the 50 $cm^2$ sample (8.0-cm dia), $P = 13\ N/m^2$ and a funnel depth of 3 mm. Thus, only about 6% of the monomer generated by a properly operating pyrolyzer of 2.5-cm dia can be used in fixing a soil sample 8 cm in diameter. Although the percent Parylene in fixed samples has not been determined because of such factors as escape through the vent, deposition on the funnel, and general unsuitability of the experimental setup for quantitative measurements, those measurements which have been made indicate that less than 5% polymer by weight is required.

The pyrolyzer pressure gauge was maintained at 265°C in order to prevent deposition of polymer on the gauge elements. A bend in the tubing joining the pyrolyzer to the funnel or nozzle thermally isolates the pyrolyzer from the soil sample. The $LN_2$ shroud serves two purposes, simulation of the space thermal environment and as a cryogenic pump for condensible gases, particularly the large quantities of Parylene generated. The bell jar was typically held below an indicated pressure of $10^{-3}$ N/m$^2$ during all runs.

Control of the initial temperature of the soil was facilitated by means of adding about $1.3 \times 10^3$ N/m$^2$ (10 torr) of $N_2$ to the system. For the low-temperature runs $LN_2$ was circulated through the shroud until the temperature indicated by thermocouples buried in the soil reached the desired temperature. For the high-temperature runs an incandescent spot lamp located within the shroud was directed at the soil until the buried thermocouples reached the desired temperature. The lamp was then turned off. The gaseous $N_2$ was evacuated after the desired temperature had been reached and the run begun.

## C. Experimental

1. Open-Cup Experiments. The initial Parylene soil fixation experiments were conducted at Union Carbide Corp. facilities with existing equipment. With this equipment a deposition chamber follows the pyrolyzer and precedes a cold trap and mechanical vacuum pump. The deposition chamber pressure is controlled by the source output and by the ratio of the chamber input to output conductance. Only the source output is controlled, by controlling the temperature of the evaporator. The soil sample was contained in an open cup placed in the deposition chamber. The initial tests were successful but did not simulate conditions achievable for *in situ* samples on the Moon. However, these initial tests were encouraging enough that tests of the type described in Section III of this report were conducted.

2. Inverted Funnel Experiments. Previous experience had indicated that the seal formed at the rim of a funnel inverted in soil is capable of withstanding several hundreds of Newtons per square meter of pressure without blowout of the soil. The initial inverted funnel experiments were conducted without the funnel vent, but the soil conductance was inadequate to maintain flow through the pyrolyzer; thus, the dimer cracking reaction did not proceed properly. It was possible under these low flow rate conditions to maintain an indicated pressure of 13 to 27 N/m$^2$ in the pyrolyzer for several hours without measurable evaporation of the dimer or detectable deposition of Parylene on the soil. In order to establish conditions in the pyrolyzer such that the reaction would proceed normally, as in the open-cup experiments,

the funnel was vented to the high vacuum within the shroud through a 10-mm ID tube for runs at pyrolyzer pressures of 13 N/m$^2$. For some later runs the vent tube was further restricted in order to operate at higher pyrolyzer (and funnel) pressures in an attempt to increase the penetration of the fixative. The soil surface was also heated. All attempts at increasing the penetration failed for the specific soil used in these tests. A coarser soil would undoubtedly allow greater penetration but no runs were made with coarse soils. Run 52671 was made with a finer cut (~300 μm) of the same soil in an attempt to duplicate conditions of the styrene/$BF_3$ runs and resulted in somewhat less penetration.

Table 4 is a summary of key runs made using the inverted funnel technique. Only a representative number of runs have been tabulated. Approximately 10 runs either yielded negative results or were incomplete and are not tabulated, although the general results of such runs have been discussed above.

In a few cases the pyrolyzer pressure did not increase in a normal manner until about a half hour after the run had begun. It was found for these cases that the initial seal of the funnel to soil had been poor but that Parylene had deposited at the leak, finally sealing it and allowing the deposition to proceed normally.

3. Open-Nozzle Experiments. Three types were conducted. The nozzle exhaust was directed at:

(a) A cold copper plate.

(b) A pressure gauge of the same type used in the pyrolyzer.

(c) Soil samples.

The nozzle was offset from the pyrolyzer to break the thermal radiation path, and the apparatus was operated in the same manner as with the funnel. The nozzle ID was 9.2 mm, so that operating parameters were nearly identical. Nozzle standoff distance was 2.5 cm.

The copper plate was thermally attached to the $LN_2$ shroud and was held at or below -40°C throughout the deposition. The interior of the $LN_2$ shroud was again held at or below $10^{-3}$ N/m$^2$ except in the nozzle wake. Evaporation and pyrolysis of $10^{-2}$ kg of Parylene yielded a disk of polymer about 2 cm in diameter by 0.12 mm thick.

Measurement of the recovery pressure at a distance of 2.5 cm from the nozzle indicated that approximately 10 to 20% of the pyrolyzer pressure was recovered at gauge port in a flat plate normal to the flow.

Two soil samples were fixed under conditions identical to the above tests. Fixed, but somewhat friable, samples 2 cm dia × 2 mm thick were obtained.

# VI. SUMMARY

## A. Accuracy of Fixation

The goal of the work reported here is preservation of the morphology of the top few millimeters of the lunar soil. However, no requirement for accuracy of fixation has been given, and no measurement of the accuracy achieved has been made. Although a large number of techniques for measurement of the accuracy of fixation can be listed, the most definitive test would be to simulate the appropriate sample return experiment. For example, for nuclear track studies the sample could be exposed to radiation while in the simulator and then fixed. The normal or proposed sample return experiments could then be conducted on the sample. Until such tests are conducted or accuracy criteria otherwise established, no work can be done to determine if sufficient accuracy is being achieved. Two means for determining accuracy of fixation have been employed. In the first, sand grains were dropped into soil samples prior to fixation. Both polymer systems left vertical walls where the sand had dropped into the soil. In the second, a light coat of aerosol paint was deposited on the soil surface, and no disturbance could be observed after fixation.

## B. Removal of Fixative

The choice of the best fixation system must hinge not only on the sample-fixing constraints, but on the intended use of the returned material. Parylene and epoxies are good materials for thin sectioning since they are highly inert and are also a good match for the refractive index of the embedment. Their inert nature may be a disadvantage to the goal of removing the matrix. Likewise, the release or exposure of the surface of discrete particles may be complicated by such an inert film.

Styrene and its derivatives might prove to be advantageous in returned material analysis due to their high solubility. Another possibility for the removal of fixative is by localized heating with an electron beam in vacuum. This method has the multiple effect of breaking chemical bonds, heating, and pumping away the volatile products.

A similar method which should be effective on styrene and might be effective on Parylene is bombardment with active ions. Such a gas-phase "etching" should be investigated as a means of preparing samples for observation in a scanning electron microscope. Indeed, the reaction would be best investigated in a microscope that is equipped with an ion gun so as to understand the action of the "etchant" on the fixative and the underlying lunar material.

Simulated soil samples fixed by both the Parylene and monomer/catalyst processes have been embedded in epoxy and thin sectioned.

The thin sectioning was done using customary materials and techniques with one exception: the fixed sample was placed in a vacuum system with the embedding material[2] in an externally tiltable cup above. The vessel pressure was reduced to below 7 $N/m^2$ until outgassing of the resin subsided, voids in the sample were clear of gas, and any unreacted materials pumped away.

The resin mix was then poured by remote control into the sample cup, totally immersing the sample. No bubbling was noted, indicating that no gas was displaced from pores in the sample. The system was then returned to ambient pressure and the infused sample transferred to an oven to cure.

The resulting block was diamond sawed, mounted, and lapped in the conventional way. The thin sections were examined in the microscope and no evidence of voids was seen.

## C. Other Performance Factors

What may be an advantage for one experiment may be a disadvantage for another. Based on the assumption that an inverted funnel technique would be used for a flight experiment, some of the apparent advantages and disadvantages of the two polymer systems are listed as follows:

Advantages

| Monomer/catalyst | Parylene |
|---|---|
| Low weight and power | No liquid phase |
| Short deposition time | Wide temperature range |
| Easily removed | No fluid handling |

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[2] Resin, Shell Chemical Co. #825, 100 parts.
Catalyst, Emerson & Cummings #11, 25 parts.

## Disadvantages

| Monomer/catalyst | Parylene |
|---|---|
| Forms liquid phase | High weight and power |
| Narrow temperature range | Long deposition time |
| Fluid handling required toxic catalyst | Not easily removed |

# VII. CONCLUSIONS

The feasibility of soil fixation with no visually apparent disturbance has been established for two basic polymer types, the monomer/catalyst type and the Parylene type. Both employ gaseous deposition of the fixative. The fixatives can be deposited at pressures of less than $1.33 \times 10^3$ N/m$^2$ (10 torr), thus allowing a seal to be made between an inverted "funnel" and the lunar soil.

The fixed samples are tough and readily handled. Considerable effort is required to break off a piece of a sample, and it is necessary to free the sample from the funnel with a knife or spatula.

Completion of tradeoff studies between the two polymer types and design of flight instruments would require additional work and determination of the specific experiments to which the technique would be applied.

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## Table 1. Open-cup experiments

| Run No | Weight of soil, g | Initial bell jar pressure, N/m$^2$ | Styrene addition |  |  | $BF_3$ addition |  |  | Results | Percent of polystyrene in soil |
|---|---|---|---|---|---|---|---|---|---|---|
|  |  |  | Initial soil temperature, °C | Final soil temperature, °C | Final styrene pressure, N/cm$^2$ | Initial soil temperature, °C | Final soil temperature, °C | Final $BF_3$ pressure, N/m$^2$ |  |  |
| 1 | 8.86 | $10^{-2}$ | 0 | 1.5 | 160 | ---- | ---- | 1100 | Only trace of polymer in soil. No radiation shields used and soil probably never reached temperature indicated by thermocouple | Trace |
| 2 | 11.2 | ---- | 4 | 11 | 320 | 11 | 20 | 930 | Poor consolidation — aggregates formed | Trace |
| 3 | 12.4 | $10^{-2}$ | 2 | 10 | 280 | 11 | 14.5 | 5600 | Styrene added in two cycles with $BF_3$ addition after each |  |
|  |  |  | -3 | 10 | 210 | 12 | 15 | 800 | Fair consolidation except some loose grains on surface | 2.7 |
| 4 | 13.9 | $1.3 \times 10^{-2}$ | -9<br>-18 | 7.5<br>2 | 200<br>170 | 7.5<br>6 | 20.5<br>11 | 400<br>410 | Styrene added in two cycles with recooling. Large exotherm due to polymerization after first $BF_3$ addition. Slight exotherm at second addition. Good consolidation but slightly nonuniform. | 2.3 |
| 5 | 13.9 | $10^{-2}$ | -8<br>-10 | 10<br>10 | 510<br>310 |  |  |  | $BF_3$ added first 400 N/m$^2$ (3 torr). Two cycles styrene addition. Fair consolidation slightly sticky polymer | 1.3 |
| 6 | 9.39 | $9 \times 10^{-3}$ | -10 | 19 | 800 | 19 | 25 | 400 | Good consolidation of soil. Used heavy copper cup for better heat transfer | 5.91 |
| 7 | 9.43 | $7 \times 10^{-3}$ | -14 | 11 | 530 | 13 | 22 | 400 | Good consolidation of soil. Uniform distribution of polymer | 4.73 |

## Table 2. Closed-cup experiments

| Run No | Initial soil temperature, °C | Final soil temperature, °C | Styrene pressure in manifold, N/m$^2$ | Weight of styrene added, g | Time of addition, min | Bell jar pressure during addition, N/m$^2$ | $BF_3$ gas added, ml | Results | Percent of polystyrene in soil sample |
|---|---|---|---|---|---|---|---|---|---|
| 8 | -15 | --- | 330 | 1.6 | 11 | 6.7 | ~50 | Temperature record erratic due to improperly placed thermocouple. Some fixation around periphery of cup (Cup A). | Not measured |
| 9 | -14 | 3 | 400 | 1.0 | 10 | 9.3 to 13 | ~50 | Pronounced exothermic reaction and temperature rise of about 10 °C when $BF_3$ added. Good fixation but not uniform (Cup A) | Not measured |
| 10 | -14 | -12 | 530 | 2 | 10 | 5.3 | ~50 | Very little fixation. Believe bulk of styrene escaped around edge of funnel (Cup A) | Not measured |
| 11 | -8 | 2 | 530 | 1.5 | 9 | 2.7 | ~50 | Good consolidation but sample stuck in funnel. Exotherm of 15 °C when $BF_3$ was added (Cup B used through Run 16). | Not measured |
| 12 | -12 | -1 | 530 | 2 | 12 | 1.3 to 4 | ~50 | Extensive consolidation but strength of compact poor. 5 °C exotherm when $BF_3$ was added. | Not measured |
| 13 | -13 | -4 | 530 | 1.0 | 10 | 2.7 | 50 | Second addition of monomer after first. 2.0 g styrene added on second cycle. Very poor consolidation. Most of the styrene escaped | Not measured |
| 14 | -6 | -5 | 530 | 1.5 | 10 | 6.7 | 50 | Two cycles. No consolidation. Styrene pumped off and was lost. | Not measured |
| 15 | -10 | +2 | 530 | 1.0 | 8 | $2.7 \times 10^{-1}$ to 5.3 | 50 | Large exotherm of 20 °C when $BF_3$ added. Good consolidation. | 5.4 |
| 16 | -8 | -6 | 530 | 1.0 | 9 | 4 to 5.3 | 50 | No exotherm. No consolidation. Styrene lost due to poor sealing. | Not measured |
| 17 | -6 | 7 | 530 | 1.0 | 9 | 0.67 | 50 | Good consolidation. Large 27 °C exotherm when $BF_3$ added. Cup C used for rest of runs | 2.75 |
| 18 | 0 | 3 | 530 | 1.5 | 10 | 6.7 | 25 | $BF_3$ added first but it blew out soil at edge of cup and styrene was lost on subsequent addition. No fixation of soil | Not measured |
| 19 | -7 | 6 | 600 | 1.0 | 7 | 0.67 | 25 | Two cycles with cooling between. Total of 2.0 g styrene added. Large exotherm of 13 °C after first $BF_3$ addition; smaller one of 4 °C after second addition. Good consolidation but somewhat non-uniform | 6.6 |
| 20 | -1 | 22 | 600 | 1.0 | 6 | $<10^{-1}$ | See remarks | $BF_3$ added first in 4 additions of about 2 ml each. Then styrene added. Finally more $BF_3$ added in 2-ml increments. Small exotherm consolidation. Some soft polymer | Not measured |

## Table 2 (contd)

| Run No | Initial soil temperature, °C | Final soil temperature, °C | Styrene pressure in manifold, $N/m^2$ | Weight of styrene added, g | Time of addition, min | Bell jar pressure during addition, $N/m^2$ | $BF_3$ gas added, ml | Results | Percent of polystyrene in soil sample |
|---|---|---|---|---|---|---|---|---|---|
| 21 | -1 | 10 | 530 | 1.0 | 8 | $<10^{-1}$ | See remarks | Styrene added first  $BF_3$ added in 2-ml increments. Large 16°C exotherm after 1st addition  Smaller exotherm with each addition until 5th addition produced nothing  Good consolidation. Harder and more uniform than in Run 20 | 3.7 |
| 22 | -10 | 10 | 600 | 2.0 | 19 | 0.67 | See remarks | Styrene added first  $BF_3$ added in 5 increments of 2 ml each  Large 18°C exotherm on first addition  Small exotherm after 2nd addition and almost nothing after that Good consolidation but side of cake wet with gummy polymer | Not measured |
| 23 | 0 | 26 | 530 | 1.0 | 8 | $<10^{-1}$ | See remarks | $BF_3$ added first in 4 increments at 2 ml each. After styrene was added, 2 more increments of 2 ml $BF_3$ added Very small exotherm after 1st one only  Fair consolidation | 2.9 |
| 24 | -32 | -9 | 460 | 1.0 | 10 | $<10^{-1}$ | 10 ml | Low temperature run. Best fixation to date  Large exotherm of 23°C observed when $BF_3$ added | Not measured |
| 25 | -33 | 0 | 600 | 1.0 | 7 | $<10^{-1}$ | See remarks | $BF_3$ added first — 10 ml  Nonuniform distribution of polymer. Polymer soft and yielding. | Not measured |
| 26 | -33 | -5 | 560 | 1.0 | 10 | 0.27 | See remarks | $BF_3$ added in very small increments  Diluted with He 100 1  Added 5 ml at a time of mixture. Each addition caused small exotherm  After 6 additions, a large amount (~5 ml) $BF_3$ added which caused another small exotherm Good fixation | Not measured |
| 27 | -33 | -4 | 530 | 1.0 | 10 | 0.27 | --- | A repeat of Run 26 with same results as above. | Not measured |
| 28 | -32 | -5 | 530 | 1.0 | 7 | $<10^{-1}$ | --- | Styrene added then system recooled to -32°C before addition of $BF_3$  Best consolidation noted to this time | 3.17 |
| 29 | -22 | 7 | 600 | 1.0 | 9 | $<10^{-1}$ | 5 | Styrene and $BG_3$ added simultaneously  Good consolidation | Not measured |
| 30 | -33 | -13 | 530 | 2.0 | 15 | 0.27 | 10 | 1 g styrene added then system recooled and another 1 g added before $BF_3$ addition  Very large 28°C exotherm on $BF_3$ addition  Much styrene migrated to the bottom of the cup because of long residence time required for recooling. Consolidation at surface good but polymer in bottom of cup was soft | Not measured |

Table 3. Open-nozzle experiments

| Run No | Initial system pressure, N/m$^2$ | Estimated nozzle pressure, N/m$^2$ | | Bell jar pressure during addition, N/m$^2$ | Styrene added, g | $BF_3$ added, cm$^3$-stp | Duration of test, min | Apparent soil temperature during run, °C | Remarks |
|---|---|---|---|---|---|---|---|---|---|
| | | $BF_3$ | Styrene | | | | | | |
| 1 | $1 \times 10^{-3}$ | 2 7 | 13 | $5\ 3 \times 10^{-2}$ | 0 6 | 15 | 60 | -60 | Polished Al plate only Trace of polystyrene on plate Nozzle redesigned for next run |
| 2 | $1\ 3 \times 10^{-3}$ | 2 7 | 13 | $9\ 3 \times 10^{-2}$ | 0 5 | 33 | 60 | -64 | Considerable deposition of polystyrene on plate (50 mg) |
| 3 | $5\ 3 \times 10^{-4}$ | 2 7 | 20 | $1\ 3 \times 10^{-2}$ | 0 8 | 25 | 60 | -65 | Soil depth 3 mm No apparent fixation Final polystyrene content of soil was 0 1% by weight |
| 4 | $5\ 3 \times 10^{-4}$ | 4 | 93 | $4 \times 10^{-3}$<br>Fluctuated | 5 3 | 65 | 60 | -70 | $LN_2$ shroud in place Insignificant fixation of soil Some polymer on sides of cup (semisolid) |
| 5 | $6\ 7 \times 10^{-4}$ | 2 7 | 93 | $6\ 7 \times 10^{-3}$ | 4 6 | 20 | 60 | -70 | Basically a repeat of Run 4 with longer cooldown to get soil to lower temperature No fixation of soil |
| 6 | $5\ 3 \times 10^{-4}$ | 2 7 | 93 | 0 13 | 4 6 | 0 2 cm$^3$ $BF_3$ etherate as liquid | 60 | -70 | $BF_3$ etherate vapor used as catalyst Poor polymerization even on $LN_2$ shroud and trap No soil fixation Bell jar pressure high due to leak in system |

## Table 4. Summary of inverted funnel Parylene runs

| Run No | Initial soil temper-ature, °C | Final soil temper-ature, °C | Indicated pyrolyzer pressure, $N/m^2$ | Vent diam-eter, mm | Weight of dimer evaporated, g | Run time, h | Approxi-mate sample thickness, mm | Weight of fixed sample, g | Comments |
|---|---|---|---|---|---|---|---|---|---|
| 51471 | +150 | +27 | 110 | 9 5 | 4 5 | 3 3 | 2 8 | 21 3 | The upper surface of the sample is light in color, approximately the same as the unfixed soil The initial temperature of this sample was 150 °C and the quartz/halogen lamp, used to heat the soil, was maintained on for more than one hour of the three hour deposition time in an attempt to increase the depth of penetration The penetration was essentially the same as for other samples |
| 51871 | -70 | +22 | 16 | 9 5 | 7 5 | 3 3 | 2 8 | 21 8 | The initial temperature of this sample was ~ -70 °C and the heat lamp was not used All sample heating was due to radiation from the Parylene apparatus and convective transfer via the hot Parylene monomer gas stream Even though the initial conditions of this run were at the extreme opposite of those of run 5-14-71, the penetration was approximately the same. However, the dark appearance of the sample was indicative of the much thicker Parylene deposit at the surface. It can be noted by viewing a broken edge that this thick deposit extends only a fraction of a millimeter into the sample This sample should correspond closely to fixation in an unlighted region of the Moon |
| 52071 | +101 | +22 | 16 | 9 5 | 9 5 | 3 1 | 2 8 | 25 0 | This run was similar to the 5-14-71 run except that the initial temperature was ~100 °C and the heat lamp was turned off at the beginning of deposition It should correspond to fixation in sunlit area with shadowing beginning at the start of deposition There is a thin layer of heavy deposition near the surface |
| 52571 | +115 | +32 | 20 | 9 5 | 10 0 | 3 3 | >2 8 | 32 2 | This run was similar to the 5-20-71 run, but a higher pyrolyzer pressure was used to increase penetration Some slight increase was achieved |
| 52671 | +101 | +42 | 40 | 6 0 | Not recorded | 3 1 | 2 0 | 21 3 | Soil for this run was sieved to ~300 μm to duplicate the soil used for styrene runs |

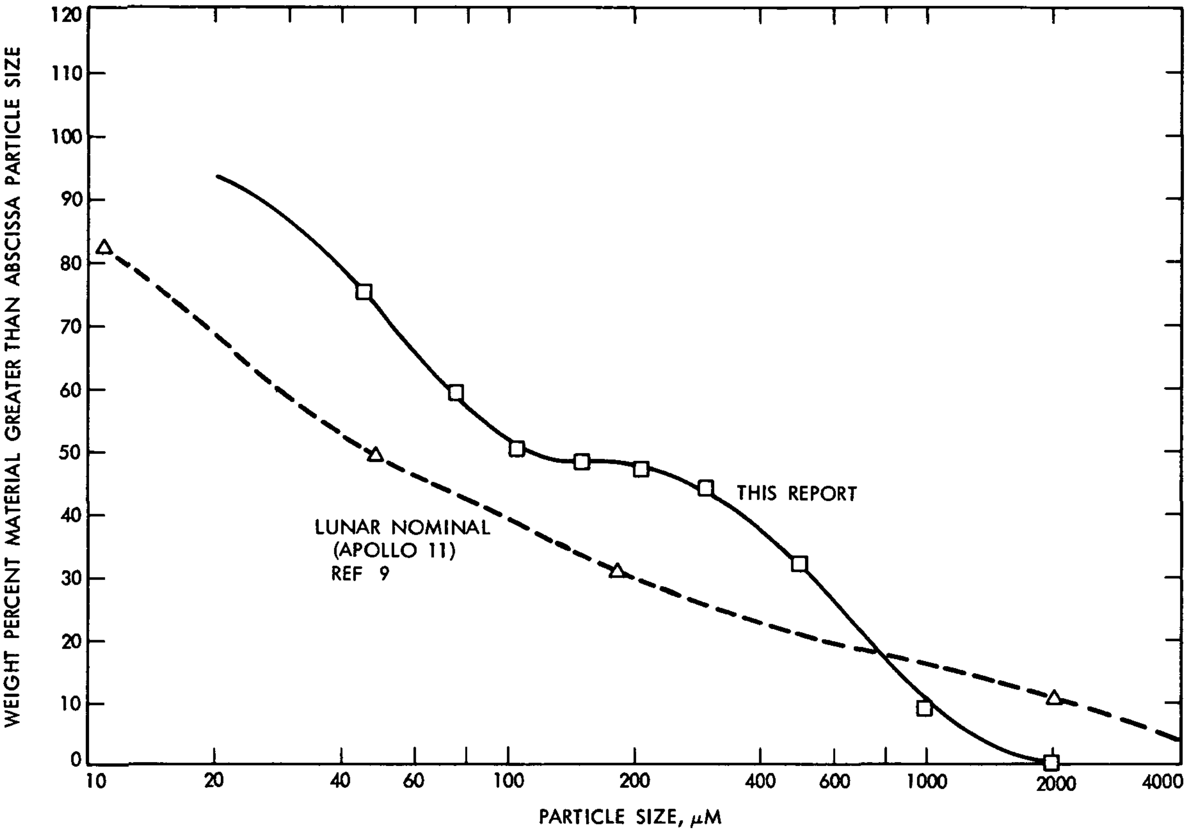


Fig. 1. Basalt rubble particle-size distribution

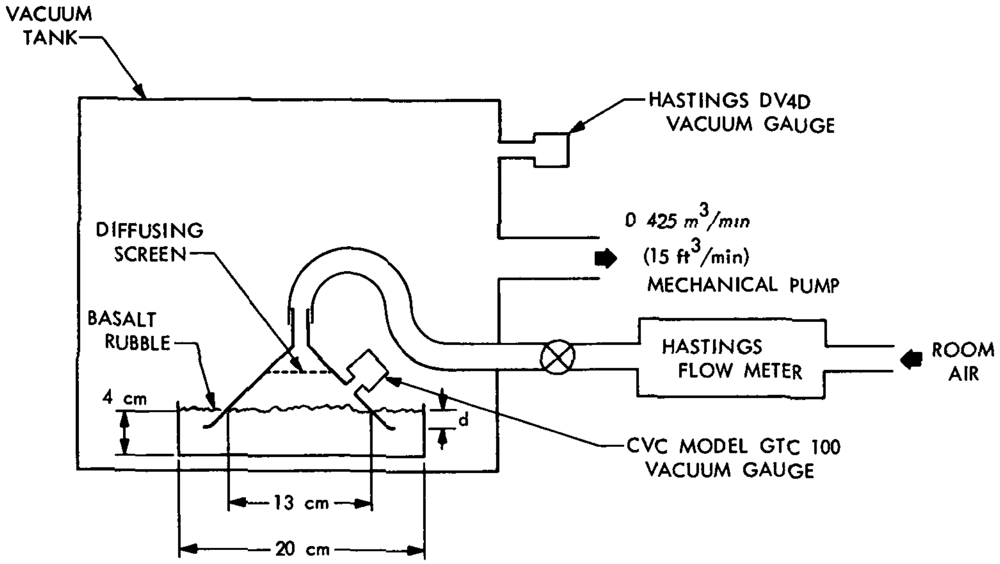


Fig. 2. Soil gaseous conductance apparatus

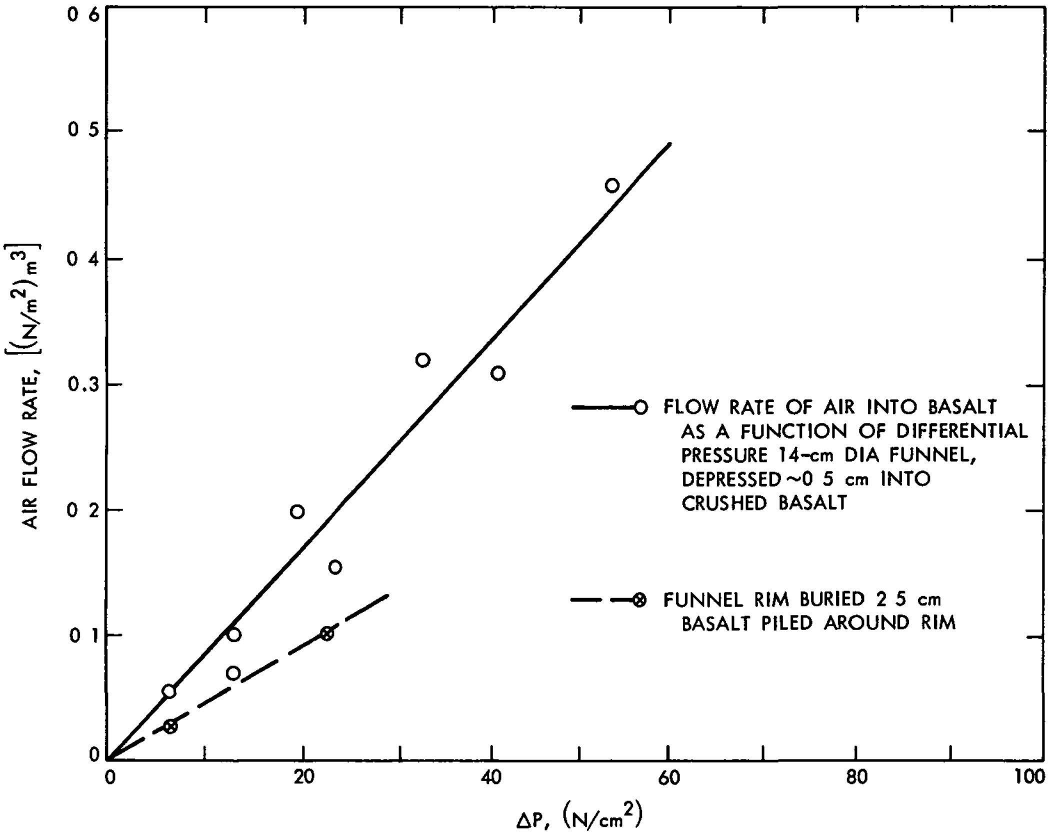


Fig. 3. Soil gaseous throughout versus pressure

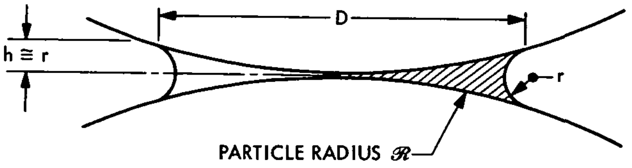


Fig. 4. Capillary condensation geometry

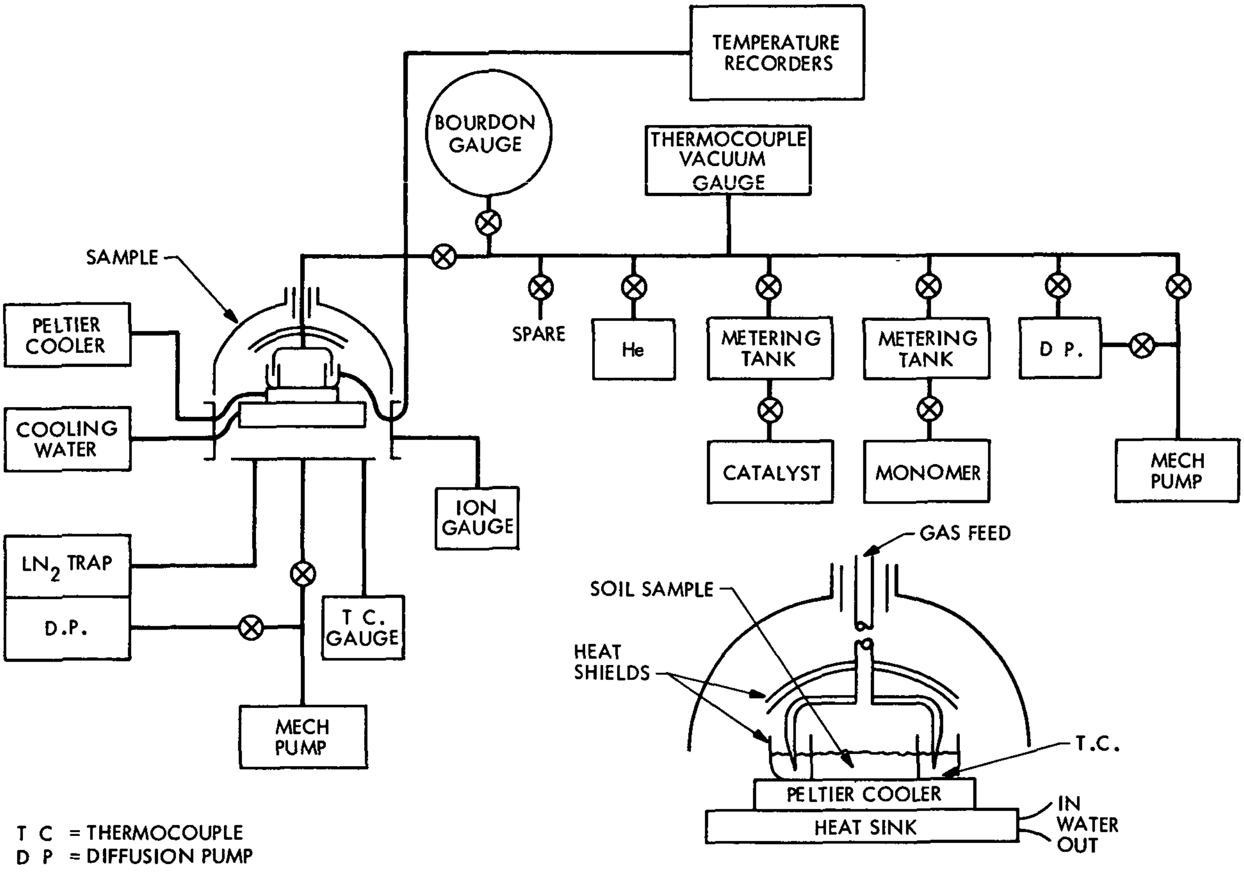


Fig. 5. Schematic of styrene fixation apparatus

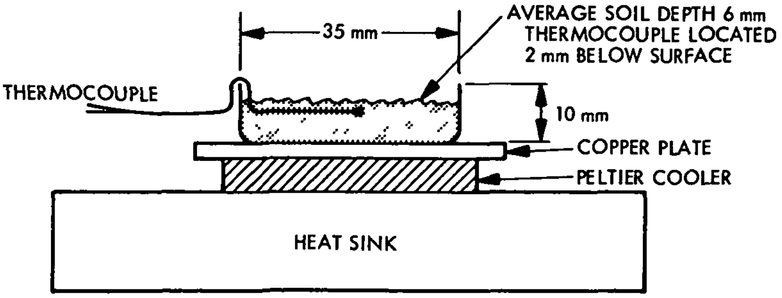


Fig. 6. Open-cup configuration

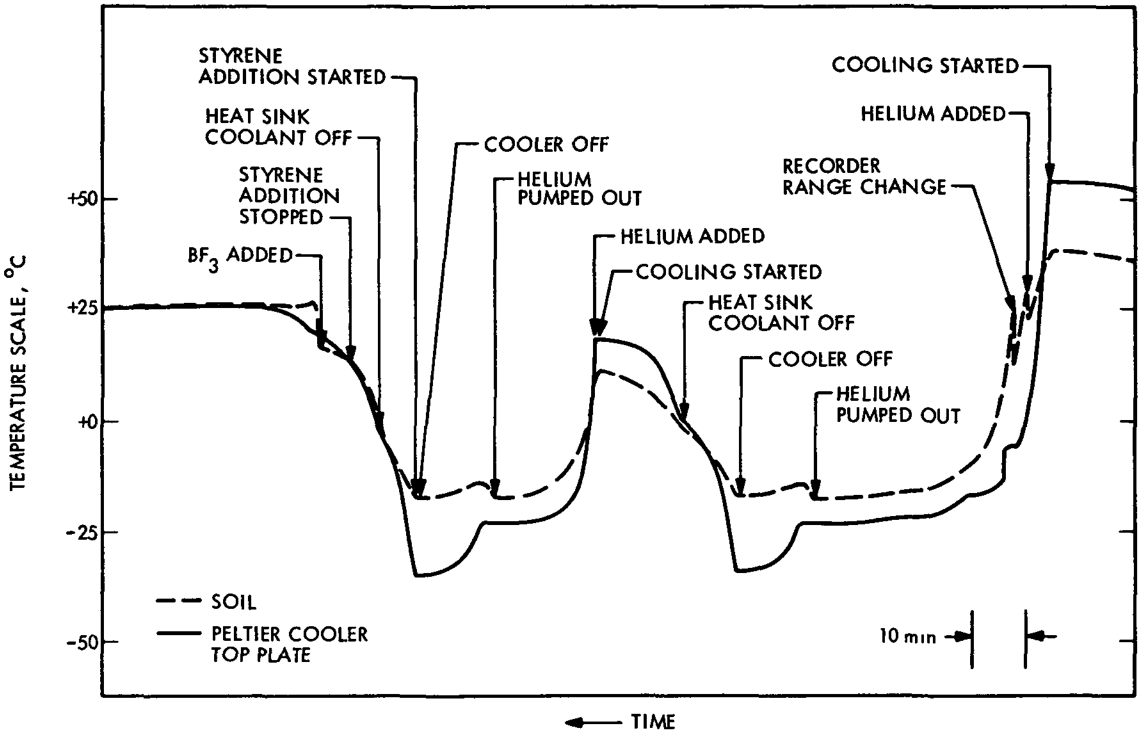


Fig. 7. Temperature history run #7 (see Table 1)

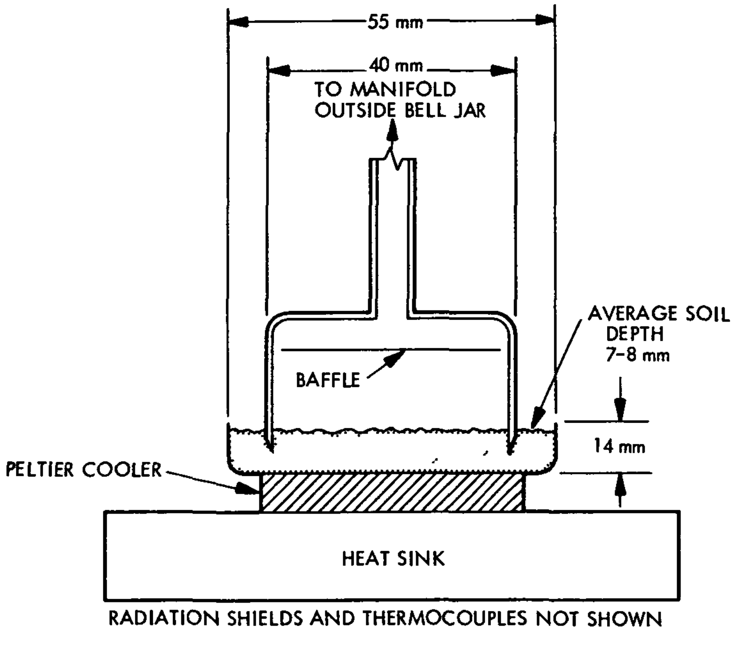
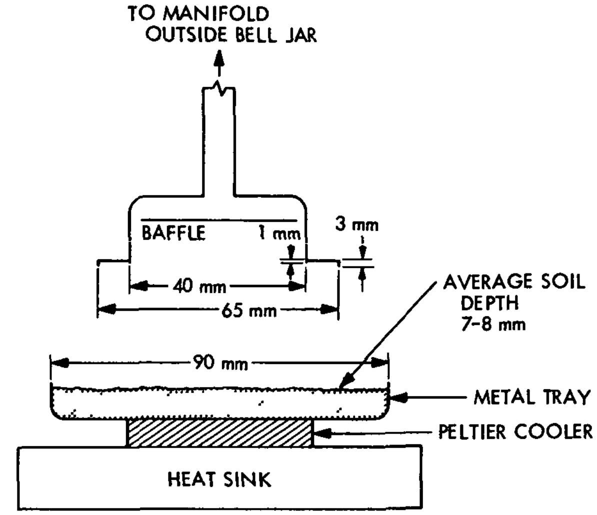


Fig. 8. Closed cup (single seal)



CUP SHOWN IN RAISED POSITION. RADIATION SHIELDS AND THERMOCOUPLES NOT SHOWN.

Fig. 9. Closed cup (double seal)

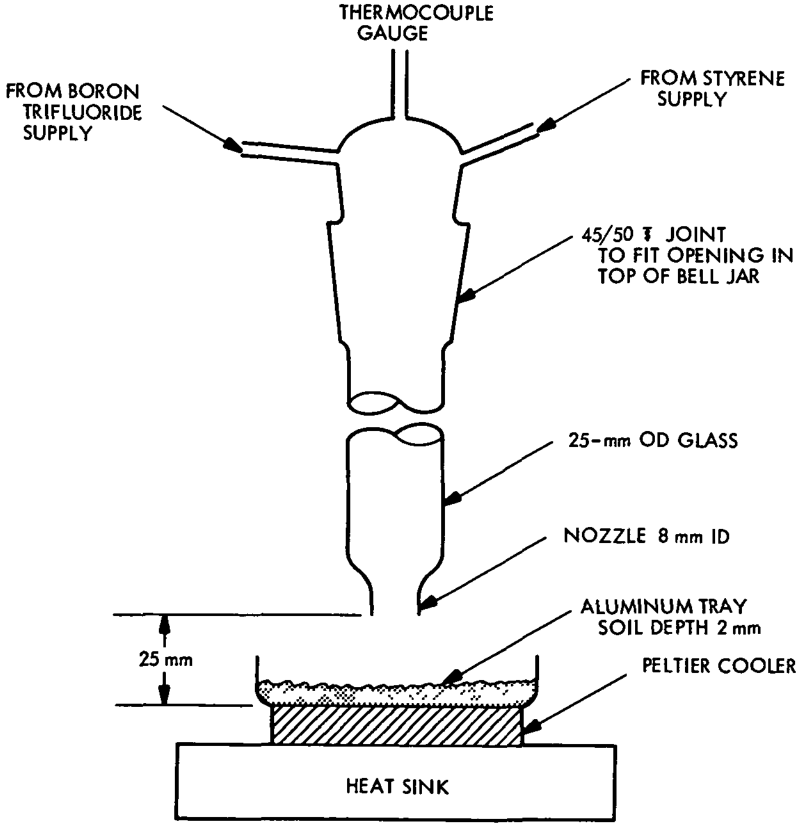


Fig. 10. Open-nozzle configuration

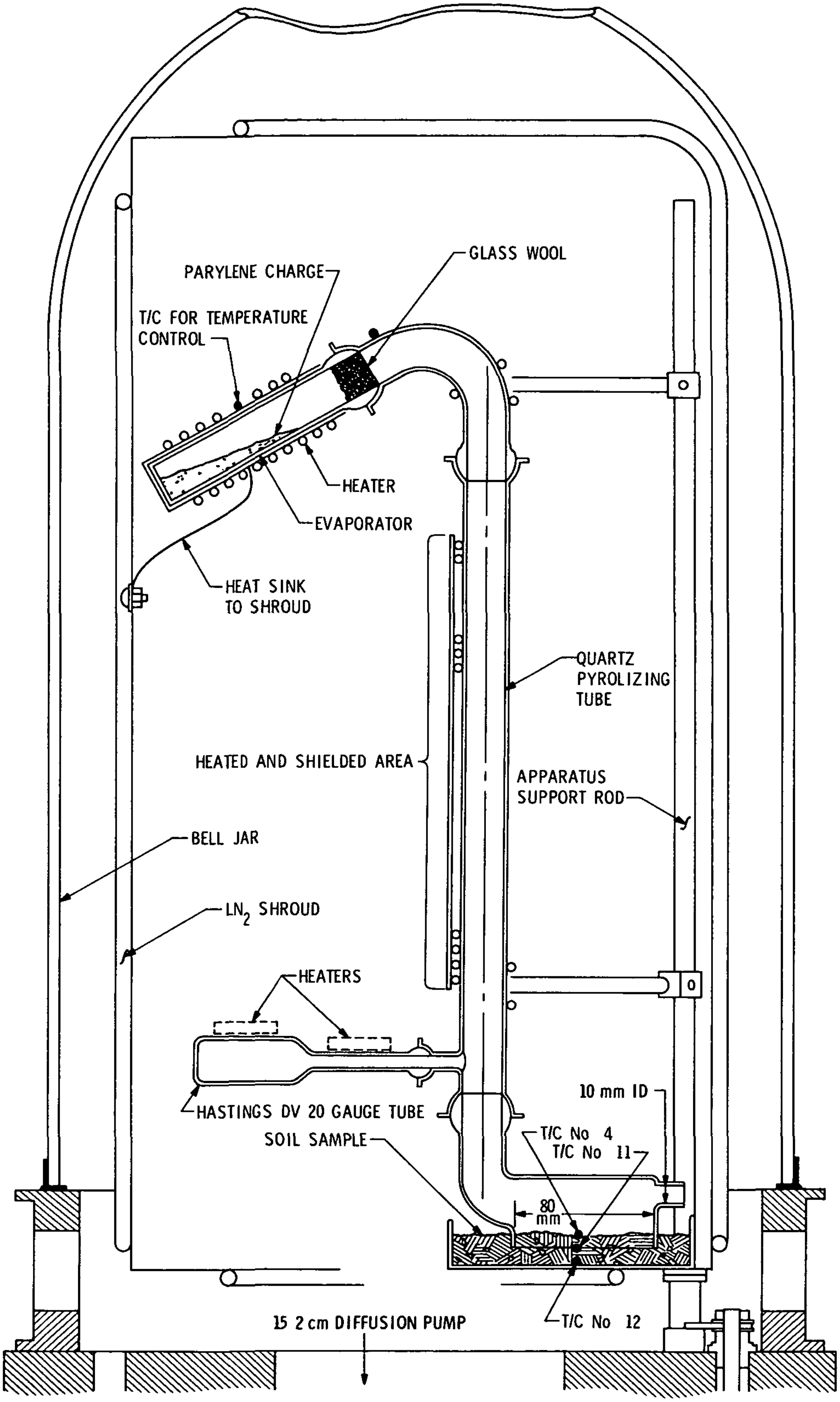


Fig. 11. Parylene soil fixation apparatus

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