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Ralph Stone and Company, Inc. Engineers

# INVESTIGATIONS OF LUNAR SAMPLING

AND

SAMPLE RETURN METHODS

Prepared under Contract No. NAS 9-3549
by
RALPH STONE AND COMPANY, INC.
ENGINEERS
Los Angeles, California

for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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#### FOREWORD

All work reported herein was performed for the National Aeronautics and Space Administration, Manned Spacecraft Center, Houston, Texas, Under Contract NAS 9-3549 to Ralph Stone and Company, Incorporated, Engineers.

The contract authorized a six-month study commencing September 25, 1964, and delineated work in lunar geologic and biologic sample acquisition, packaging and return. The study was conducted under the cognizance of the Advanced Spacecraft Technology Division with Mr. Donald A. Flory of the Lunar Surface Technology Branch serving as Technical Representative.

Acknowledgment is extended to members of the Early Apollo Science Teams who contributed time and advice. Others who contributed more directly to the studies include E. T. Conrad, Clifford Frondel, Ph.D., Gregory Jann, Ph.D., David Andrew Link, Michael Saleh, M.D., Ph.D., John C. Simons, Jr., Ph.D. and Ralph Stone, M. S.. E. A. Webster served as Project Manager.

Subcontractors included Ardel, Incorporated, Glendale, California, and Dynamics Technology Laboratories, Gardena, California.

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# INVESTIGATION OF LUNAR SAMPLING AND SAMPLE RETURN METHODS

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#### SUMMARY

The return of samples of lunar material acquired by Apollo astronauts implies many unique objectives. Minimum contamination from the packaging system and from outside influences such as the LEM and terrestrial atmospheres, and minimum intra-sample contact are primary requirements. Protection from physical damage during re-entry and landing are essential, yet the packaging system weight must be minimized to permit the return of as much lunar material as possible within the 70 pounds allocated for samples, containers and accessories. All objectives must be met with a system which the astronaut can use effectively despite severe limitations on time and dexterity.

Principal restrictions were those imposed by the Early Apollo Sciences Teams whose concern was that trace contaminants from packaging materials could be misleading to laboratories evaluating the returned specimens. Aluminum, copper and gold were among the few acceptable candidate materials, with stainless steel a possible alternative for special sample requirements.

The amounts and kinds of gases evolved by the samples are scientifically important, requiring that a completely passive high vacuum design be sought for the storage container. This eliminated pumps or getters from consideration, and placed further constraints on the choice of materials, seal designs and pretreatment procedures to minimize outgassing.

Configurations studied were generally limited to 8 x 11-1/2 x 19 in. drawer-like outer containers and flexible inner bags made of various polyester film and aluminum foil laminates.

Results indicated the feasibility of a packaging approach involving partial compartmentation of the sample box and ingress of large and small sample bags through a port in the top. Redundant metallic seals generally met the vacuum requirements, but placed an added burden on the latching mechanism to apply a sufficiently high unit force to effect a positive closure.

The limited time available to the astronaut for sampling and the restricted container volume were found to place significant constraints on the maximum number of individually packaged samples. To improve payload efficiency and return maximum weight it was found that with the bag configurations tested, samples smaller than 2 in. should be packaged in groups of three or more.

#### INTRODUCTION

The return of lunar surface material by the Apollo astronauts will be a scientific event of the first magnitude. Scientists throughout the world, and more specifically, those on the Early Apollo Sciences Teams are methodically preparing an exhaustive sample analysis and research program. Their concern is that the maximum amount of samples are returned, properly documented and identified, packaged to minimize physical damage and contamination, and returned to Earth in a form that will not detract from subsequent scientific experiments.

The rough outline of a plausible scheme for sample return was evolved in mid-1964, and described generally the packaging of geologic samples in flexible bags, the preservation of special purpose samples in small rigid canisters and the stowage of all sample containers within a rigid, drawer-like sample box aboard the LEM. From the beginning, the investigators were cognizant that the paramount requirement was to return representative lunar samples. Other principal objectives were minimum container weight, minimum sample contamination, simplicity, high reliability and minimum use of the astronaut's time. It was felt generally that the sample box should be a passive vacuum chamber having the best possible seal, and that the inner bags and canisters should be individually sealed to minimize intra-sample contamination and to provide redundant protection.

The scope of the investigation included development of engineering criteria pertinent to the return of lunar samples from the Apollo manned lunar missions in a form most usable to the many scientific teams and laboratories responsible for analysis and investigation. The principal project guideline was to evolve an acquisition and packaging system combining maximum sample return with optimum protection. Details of the investigation have been presented in four sections:

# PART I SAMPLE CONTAINER DESIGN REQUIREMENTS

- A. Sample Box
- B. Inner Bag
- C. Special Containers

#### PART II CONTAMINATION

PART III SAMPLE ACQUISITION AND PACKAGING PROCEDURES

#### PART IV APPENDICES

- A. Comments from Scientific Community
- B. Vibration Test Results
- C. Packaging Demonstration Test Results
- D. Inner Sample Bag Materials Test Results
- E. Bibliography

#### RECOMMENDATIONS

# SAMPLE BOX

- 1. Rigorous structural investigations and tests should be completed to achieve an optimum configuration. Elliptical and rectangular sandwich construction, and frame and plate configuration with partial bulkheads were analytically superior to the other structural systems studied.
- 2. Container material selection work indicated that several aluminum alloys may be suitable for structural, vacuum outgassing and sample contamination constraints. Final material choice will depend in part on whether manufacturing methods and alloying constituents can be closely controlled.
- 3. A two-stage redundant seal should be used consisting of a metallic inner seal (probably gold or aluminum) to minimize outgassing and diffusion, and an elastomeric outer seal to provide added protection against unexpected deflections or imperfections on the sealing surfaces.
- 4. The final configuration must accommodate an 8 x 11 inch elliptically-shaped entry port in the  $11\frac{1}{2}$  x 19 inch top surface. The latching mechanism should exert a force of at least 200 pounds/lineal inch to seat the redundant elastomeric and metallic seals.
- 5. The periphery of the box should have a metallic tear-off strip (e.g., similar to a coffee can design) to facilitate removal of samples at the receiving station.
- 6. Partial bulkheads less than two inches in height should be used to minimize movement of samples and to provide additional structural stiffening.
- 7. Additional packaging protection to prevent sample movement appears to be essential to minimize abrasion and puncture of the inner sample bags.

# INNER SAMPLE CONTAINERS

1. The biologic sample container should incorporate either a shield or probe to permit acquisition of samples with minimum contamination from the astronaut's glove or suit leakage. Except for this constraint, all inner containers are potentially applicable to the storage of samples intended for biologic examination.

# (RECOMMENDATIONS, con't)

- 2. The container for machine-driven core samples should be cylindrical with redundant seals similar to those on the sample box. The cylinders can be stored in special locations within the Command Module, and should be designed accordingly.
- 3. Containers intended for samples to be examined for surface and trapped gases should be stainless steel cylinders with metallic seals, and should be designed to connect directly to the gas analysis apparatus without intermediate processing.

# SAMPLE BAGS

- 1. Two candidate materials, copper and aluminum foil, meet most of the vacuum, packaging and contamination requirements. To provide an impervious gas barrier the bag should be a laminated foil, the number and thickness of plys to depend on the physical protection provided by the overall packaging system.
- 2. At least two sizes of bags should be supplied, one about 3 inches in diameter and the other 8 inches. Dexterity and time constraints appear to limit the total number of sample bags to 100. If small rocks (2 inches and under) are collected, greater packaging efficiency can be achieved by placing several in a bag, rather than protecting each individually.
- 3. Bag closure can best be achieved by mechanical crimping. Multiple folds proved to be awkward and ineffective, while sealants generate contaminating gases.

#### CONTAMINATION CONTROL

- 1. Materials should be selected to avoid contaminants unacceptable to scientists designing the lunar sample experimental programs. Manufacturing controls are essential to ensure quality materials of acceptable purity. Identification programs to obtain reliable signatures, and cleaning processes to remove spurious surface contaminants are secondary considerations.
- 2. Preheating techniques required to minimize outgassing for effective high vacuum storage will provide ample packaging material sterilization. The hermetically sealed sample box will ensure against biological contamination during the translunar phase, while control sampling techniques will be used to differentiate between biological growth of lunar and non-lunar origin.

# SAMPLE IDENTIFICATION AND ORIENTATION

1. Adequate sample identification can be achieved by numbering the sample

# (RECOMMENDATIONS, con't)

containers and recording the astronaut's descriptive data during explorations.

- 2. Sample orientation can be recorded by photographs taken by the exploring astronaut, showing the sample in relation to its immediate surroundings. Other photographs will be used to record the sample area in its relationship to the LEM. Orientation of the LEM to the lunar topography should be determined by comparing photographs taken during the LEM descent stage with previously prepared detailed topographic maps of the landing area.
- 3. All photographs and voice recordings should be time indexed.

# FUTURE WORK

- 1. It is recommended that an orderly program be continued for the development of breadboard, prototype, proof and flight qualification hardware for the sample box and inner containers.
- 2. A seal and structural optimization program should be implemented immediately to provide adequate lead-time for the delivery of prototype hard-ware for evaluation and test.
- 3. Extensive qualification test programs should be implemented in close coordination between NASA and the scientific community. These programs should be planned to ensure the compliance of the lunar sample container program with the overall research effort for sample evaluation and analysis.
- 4. Critical samples such as biological, gas, low-level radiation counting and surface structure will require the closest cooperation between the MSC receiving station design and the sample container development program to ensure compatible interfaces with the handling and manipulating equipment at MSC.

#### PART I

# SAMPLE CONTAINER DESIGN REQUIREMENTS

#### A. SAMPLE BOX

# OBJECTIVES AND CONSTRAINTS

The objective of this section is to present design criteria for the prototype sample container. The principal requirements are as follows:

- (1) Size and configuration. Overall envelope dimensions are 8 x 11½ x 19 in., the limits of the storage areas provided in the LEM and the Command Module. Figure 1 illustrates a rectangular parallelepiped sample box which makes optimum use of the available space.
- (2) Weight. Total weight of the boxes, inner containers and all lunar samples must be limited to 70 pounds. To optimize the sample packaging system, the weight of the container storage systems must be a minimum consistent with adequate sample protection and sealing requirements. Indications are that each of the two sample boxes will weigh not more than 6 to 8 pounds.
- (3) Physical protection. The sample container should be sufficiently rigid to prevent vacuum break or other inadvertent contamination during the return flight. In addition, the packaging system within the box should be adequate to protect the samples against damage and intra-sample contamination. Further, should it be necessary to jettison the container in the ocean, the box design should insure against salt water intrusion or damage during extended flotation in rough seas.
- (4) Contamination. Trace contaminants from the packaging and sealing materials will intermix with the samples. These traces are critical and must be easily identifiable and scientifically neutral. Generally, all organic materials and inorganics with molecular weights higher than Cu are not acceptable.
- (5) Astronaut constraints. The design of the rock box must be such that the unit may be: (a) easily removed from the Command Module and stowed in the LEM, (b) easily removed from the LEM and transferred to the lunar surface, and (c) readily opened for the storage of samples. In that regard, the entry port in the box must be large enough for

efficient and simple storage by the awkwardly gloved astronaut, and sealing must require a minimum of time and effort.

# STRUCTURAL DESIGN CONSIDERATIONS

The sample box must be capable of withstanding a design pressure differential of 20.5 psi and a proof pressure of 30 psi. It must be capable of withstanding Apollo launch and Command Module re-entry vibration requirements (Procedure IX, Environmental Specifications for Apollo Scientific Equipment) at the particular equipment location assigned for the sample containers, and should withstand the maximum anticipated (currently 78-G) shock load. This is particularly important at the seal locations, which must have sufficient stiffness to prevent vacuum break over the complete range of static and dynamic loads. See Figure 2 for the complete loading regime.

Support during storage as currently planned will be by side rails, although box strength design would be improved by uniform bottom support. Preliminary analyses of several structural systems have been completed and are summarized in Table I. Figures 3 through 6 illustrate the various structures analyzed.

# MATERIALS

An aluminum alloy, 5456-H321, has been tentatively selected because of its minimal outgassing tendencies and favorable structural aspects. Batzen and Ryan (see Bibliography Appendix V) reported an outgassing rate of 8 x  $10^{-16}$  torr-liter/cm<sup>2</sup>-sec for this material baked at  $200^{\circ}$ C. For comparison, Grove reported outgassing rates of  $10^{-15}$  torr-liter/cm<sup>2</sup>-sec for  $400^{\circ}$ C baked stainless steel as being routinely attainable.

The effect of aluminum outgassing will be to increase the box pressure at a rate of 3 x  $10^{-9}$  torr/hour. The gases evolved will be mainly hydrogen, with minor amounts of water, CO and CO<sub>2</sub>.

#### INGRESS

Ingress is planned through a port with a minimum diameter of 8 inches, located in the ll½ x 19 in. top face of the sample box. A more refined access would be an oblong-shaped port with its major axis along the 19 in. dimension, provided that problems attendant with latching can be overcome. The use of a full opening lid is unattractive from a structural strength standpoint, requiring more weight to ensure stiffness of the seal along the major box perimeters, as well as extending the seal length two to three times that of the circular port.

$\begin{pmatrix} 3 \\ \text{Vol/Wt} \\ \text{Ratio} \\ (\text{in}^3/1\text{b}) \end{pmatrix}$	(F/E)	117	220
$(2)$ Volume $(in^3)$	(F)	1200	1450
( <u>l)</u> Weight ( 1bs.)	(E)	8.8 plus 1.5 for lid hardware.	3.3 plus 3.3 for edge connections, lid hard- ware.
Size of Members (in.)	(D)	0.012 skin 2½ D. tubes, 0.58 thick framework.	0.3 core 0.012 thick plates on each side of core. Bulk- head of same ts. material.
Constraints	(၁)	Deflection of panel not to exceed 0.5 . Frames are simply supported.	ure. Max. panel de- 0.3 core ed flection 0.2, 0.012 thick assuming simply plates on supported. Max. each side of panels moment resisted core. Bulk- ame ing fixed supports. material. Manufacturing techniques of connecting sand- wich plates require study and testing.
Approach	(B)	Flexible skins carry forces as membrane stresses. Frames subject to axial loads, bending moments, transverse shear, and torsion.	ruct reat s w/ s w/ f 40 A11 the s
Construction	(A)	Membrane - Frame (rectangular box) w/one partition.	Sandwich (rectangular) w/one partition.
No.		<u>-</u>	(2)
	Size (1) (2) of Weight Volume Construction Approach Constraints Members (in.) (in.)	Size (1) (2)	Construction Approach Constraints Members (1) (2) of Weight Volume (in.) (1b.) (1b.) (in.)

	ins	SUMMARY OF PRELIMINARY	TABLE I (continued) OF PRELIMINARY STRUCTURAL ANALYSIS OF SAMPLE CONTAINER	nued) YSIS OF SAMPLE	CONTAINER		•
No.	Construction	Approach	Constraints	Size of Members (in.)	D Weight ( lbs.)	(in 3)	3 Vol/Wt Ratio (in Mb)
	(A)	(B)	(0)	(a)		(F)	( F/E )
(e)	Sandwich (elliptical) w/o partitions.	Ellipse equivalent to 5 R cylinder. Rigid structure. Max.stress of 40 K.S.I. Axial loads & bending moments resisted by shell.	Use flat ends to conserve volume. Provide access through ends. Manufacturing techniques require study & testing.	0.2 core 0.012 thick plates on each side of core.	2.5 plus 2.0 for edge connections and lid hardware.	1200	266
4	Waffle pattern w/o partition.	Rigid structure. Edges partially fixed. Individual panels resist "G" force of concen- trated samples. Primary load in bending. Membrane stresses neglected	Rounding edges formed by bending; Min. contamination. Deflection min. at port opening. Max.volume. Deflection of 0.4 in bottom panel.		10.2 complete	1525	150
(5)	Waffle pattern w/one partition.	Same as waffle pattern w/o partition, except max. deflection in bottom panel is 0.2.	pattern w/o pt max. ottom panel	0.020 plate 0.15 x 25 webs @ 1.4 o-c. 0.120 plate at edges; Bulk- head similar material	8.2 complete	1515	185

	ume $Vol/Wt$ Ratio $(in Mb)$		
	(2) Volume (in <sup>3</sup> )	i i	
CONTAINER	① Weight ( 1bs.)	(E) (J.1 complete	1
(continued)	Size of Members (in.)	(D) Bulkheads @ 3 both ways top and bottom; one way on sides045 plates. Frames .06 thick x 1.1 deep.	hardware was not somewhat less etc. ight. counting for the ls. However, a lt is expected the container will connection.
TABLE I STRUCTURAL	Constraints	Bulkheads used for compartmentation as well as stability. Assume simply supported ss plates.	ed is the calculated weight of the container edge connection and/or lid hardware was not ded as noted.  olume. Effective volume is somewhat less te to corners, accessories, etc.  inside volume to total weight.  It of container without accounting for the lid hardware is 7.1 pounds. However, a the was used in the analysis. It is expected can be safely pared from the container will sht of lid hardware & edge connection.
SUMMARY OF PRELIMINARY	Approach	Rigid structure. B Plates act in f bending only. Crippling of bulk-a head frames is critical. Frame interaction improves stability.	noted is the of edge corst added as rate to corst adde to corst and lid harmond hat can be veight of li
MNS	Construction	(A) Frame & Plate (rectangular box) w/closely spaced bulkheads	FOOTNOTES:  (1) The first weight not Where the weight of calculated, it is ad due to lost space du due to lost space du (2) This is the inside v due to lost space du (3) This is the ratio of edge connections and conservative approact that the weight that approximate the weight
	No.	9	FOO

To facilitate removal of the samples by remote control manipulation at the receiving station, as well as to provide insurance against cold welding of the lid, a tear-away strip around the major perimeter of the box should be included, as shown in Figure 7.

# SEAL REQUIREMENTS

Materials and configuration are strongly interacting elements in the seal design and cannot be regarded independently. Because of the many successful applications of fluoroelastomers in vacuum sealing applications, this material in 0-ring form was examined first. However, calculations indicated that the outgassing rate would cause a rate-of-pressure-rise higher than could be tolerated (about  $10^{-2} torr/hr$ ), and it was concluded that elastomers should not be permitted direct exposure to the inner volume of the rock box. The performance paradox of the fluoroelastomers is due to the fact that the proposed application is for a passive chamber, whereas most ultra high vacuum chambers incorporate substantial pumping systems.

Indium and lead have been rejected by members of the Early Apollo Sciences Teams (see Appendix A) as being potentially critical contaminants that might mask their investigations. Gold and aluminum are generally acceptable candidate materials, but a seal selection program should be scheduled to determine the final materials and design configuration.

A double seal configuration is recommended to minimize leakage effects and provide essential insurance in case one seal fails. The outer 0-ring seal material should be fluoroelastomers for deflection compliance, and there should be a guard vacuum space between the two seals. Since an elastomer cannot be exposed to the chamber interior, the inner seal should be metallic. Most prior experience with gold and aluminum has been in foils and solid or hollow toroidal shapes. Whether these will be acceptable depends largely upon whether sufficient force per lineal inch can be applied through a simplified closure mechanism capable of being actuated by the astronaut.

To keep pressure buildup in the sample box below  $10^{-8}$  torr in 100 hr. with a 25-liter box, total gas ingress must be kept below 2.5 x  $10^{-9}$  torr liter/hr. This corresponds to:

Surface outassing from inside of box:  $1.3 \times 10^{-16}$  torr-liter/cm<sup>2</sup>-sec.

Outgassing from surface of inner seal:  $5 \times 10^{-14}$  torr-liter/cm<sup>2</sup>-sec.

Permeation through inner seal:  $1.2 \times 10^{-14}$  torr-liter/sec.-cm of seal length

All of the preceding will be difficult to achieve, though probably attainable.

Permeation through the outer elastomeric seal should be low enough to keep guard volume rate-of-pressure rise below 1 torr/100 hr., or 7 x  $10^{-10}$  torr-liter/cm<sup>2</sup>-sec.-atm/cm. This is roughly 4 times lower than the value presently used for fluoroelastomer, but might be obtained by using strict manufacturing controls, selective quality assurance tests, effective pretreatment and sealing methodology. Permeation through inner metallic seal will be small enough to be acceptable.

# LATCHING DESIGN CONSIDERATIONS

The sample box ingress port must be simple to remove and replace, and readily operable by the astronaut with his severely limited dexterity and mobility. The lid should be hinged or articulated from the box to prevent dropping or damage. The hinge and clamping mechanism must be designed so that cold welding will not be a problem and dust will not cause jamming of the latching mechanism. The latter requirements are particularly severe with a seal such as gold, which must have a high unit load per lineal inch to be effective. The seals provide an additional restriction for the latching mechanism, which must be designed to seat and clamp the lid squarely and in a direction normal to the plane of the seal rather than by sliding or rotation.

Two latching schemes have been suggested which illustrate this approach. One, seen in Figure 8, shows a rotating taper locking device in which the loads are carried by ball bearings and the force applied by a gear. The other, using an inflatable torus to develop sealing force, is more attractive because it minimizes mechanical motion between various parts and requires that the astronaut merely break a sealed built-in inert gas capsule to actuate the seal.

A redundant clamping mechanism, which would be actuated during the transearth phase, and which would provide considerably more protection during the final minutes of re-entry and landing shock, is worth considering. Several approaches have been suggested, such as a supplementary clamping ring or a pressurized outer toroidal ring.

#### PACKAGING REQUIREMENTS

The various tubes and containers for biologic and gas sampling, and other special purposes should be secured with clips or other mounting provisions along the side and end walls of the sample box. To minimize excessive jostling of the rock samples during transfer and storage, an

egg-crate compartmentation scheme has been experimented with. The 2 in high compartment walls serve to isolate the smaller samples and reduce movement, and to support the large samples, protecting the smaller ones beneath from being pulverized.

Because the density of the material on the lunar surface is not known, it is impossible to predict the total sample volume. The packaging scheme must therefore be flexible, neither being restricted to small compartments for rocks, nor to large voids in anticipation of returning only pumice or dust samples.

Conventional padding materials are unacceptable, principally because of their high gas entrapment. Metallic springs, if used, must be sufficiently strong to hold the samples against the walls under vibration and shock conditions, yet not so stiff as to crush delicate rock sample structures which some investigators anticipate will be encountered. A cushioning scheme involving an inflatable bladder is a possibility, although if used it must be a highly redundant structure, totally devoid of gas transmissibility and leakage. As can be seen from vibration test results, a device such as the bladder or an equally confining mechanism appears to be mandatory to prevent sample movement and attendant abrasion.

# INSTRUMENTATION REQUIREMENTS

A means must be provided to determine when sufficient samples have been collected to meet but not exceed the weight limitation. Such a device must be of simple construction, similar in concept to a direct reading spring scale. One design envisions a spring attached to the box, while another proposes to attach the box to a spring on the Jacob's staff. Either method would provide sufficient accuracy for gross weight determinations.

A simple bi-metallic element with surface read-out can be used for temperature indications. If more sophisticated instrumentation is required, a standard vacuum penetration for thermistor or thermocouple connections can be accommodated easily.

An indication of vacuum failure can be provided with a Bourdon tube or aneroid gauge with a miniature surface read-out. If desired, a "well" for the insertion of an ion or Redhead magnetron gauge could be supplied. To eliminate additional seals the surfaces surrounding such optional penetrations should be continuous, having reduced thicknesses capable of being easily cut if the investigators at the receiving station find it necessary to insert additional instrumentation.

#### B. INNER BAG

# OBJECTIVES AND CONSTRAINTS

The inner sample bag or wrapper, being in intimate contact with the samples, must be of a material which can be readily identified and which will not interfere with later petrographic, physical or biological investigations. The bag material must be tough, abrasion and puncture resistant, have minimal outgassing, gas permeability and electrostatic properties.

# MATERIAL REQUIREMENTS

Gas permeability considerations have ruled out the possibility of using single layer plastics or single layers of foil for the inner bag material. Experience with high altitude balloons has shown that multiple laminations negate pinholes in individual layers and provide an effective gas barrier. This approach is recommended regardless of the material selected.

Two criteria of utmost importance for the inner liner of the flexible sample bags are (1) it must be a high purity, identifiable material which will not interfere with subsequent investigations, and (2) it must be capable of effective pre-treatment to minimize outgassing. The two materials that appear to meet these criteria best are aluminum and copper, with the latter having an edge because it can be baked at 600 to 800°C, compared with 200 to 300°C with aluminum.

If laminated materials are used, the manufacturing method must be extremely well controlled. Gas entrained between the layers could invalidate the effects of outgassing pre-treatment, and organic adhesives or other contaminating influences could do serious harm to subsequent investigations should the layer next to the samples be penetrated due to abrasion from vibration or other problems. The outer liner of the flexible container is also critical, since excessive outgassing might raise the pressure in the passive storage container to a level that could affect other samples, should the flexible container seals be penetrated.

#### CONFIGURATION DESIGN REQUIREMENTS

Two sizes of bags are desired, one for the larger samples up to 8 in. and the other for samples smaller than 3 in. Several shapes appear to be satisfactory packing alternatives, but from a storage and dispensing standpoint, only those containers capable of being folded flat are desirable. Of equal importance is the requirement that the sample bags be open when dispensed, or readily openable by theastronaut (see Appendix C).

In the event that aluminum or copper foil bag liners are used, care must be taken to ensure against bag liners sticking by cohesion or cold welding prior to sample packaging. Disposable liners appear to be one answer to this problem. Cold welding may prove useful for bag sealing, with pressure application by roller or stylus providing a vacuum closure.

#### C. SPECIAL CONTAINERS

# GAS SAMPLES

Special containers destined for experiments on the evolution of gas from the surface of lunar rocks will involve exacting design requirements. To be effective for these purposes, the interior surfaces must have absolute minimum levels of outgassing. Highly polished stainless steel cylinders generally meet these requirements, not only from an inherently low outgassing standpoint, but because they can be baked at very high temperatures (1000°C) to further reduce contamination and gas evolution. The containers may be relatively small, no more than 1 in. diameter by 6 in. long, and must be sealed with a metallic (preferably gold or aluminum) seal. A ready means of adapting the container to the ultimate experiment must be provided so that the gas sampling container can be opened directly into the experimental apparatus.

#### BIOLOGICAL CONTAINER

The design requirements for the biological sample containers encompass the requisites of pre-sterilization, control during acquisition to minimize extraneous contamination, and stowage in a reliably sealed sample box which will provide redundant protection.

There appears to be little doubt that the outgassing bake temperature of 400 to 800°C will be sufficient for sterilization purposes. The configuration which appears most likely to minimize unwanted astronaut contamination is a shielded telescoping probe which, when extended, can be used to penetrate the contaminated surface and obtain samples from a depth of several inches.

The ordinary geologic sample packaging procedures will be very nearly asceptic, with the exception of possible contamination introduced by the astronaut, or the LEM fuel combustion products. Thus, all samples can be potentially applicable to biological examination.

#### PART II

#### CONTAMINATION

# OBJECTIVES AND CONSTRAINTS

The success of the sampling program depends largely upon the degree to which the samples reflect their lunar make-up and character when returned to earth laboratories for examination. The constantly active cycles of outgassing and absorption which universally occur in hard vacuums, will inevitably produce some unwanted molecules on the surfaces of the samples. Similarly, whatever material is in contact with the samples during the transearth phase will be deposited to some degree upon the sample surfaces. Thus, control over both materials and cleaning processes is crucial.

# SOURCES OF CONTAMINATION

(1) Fuel Trace Contamination. The principal source of lunar contamination will be the LEM's rocket engines. About 4000 lbs. of a N<sub>2</sub>O<sub>4</sub>- hydrazine unsymmetrical dimethylhydrazine mixture will be burned, the exact amount to depend upon the number of seconds of hover needed. Estimates of the extent of such contamination from the point of landing range as high as a thousand miles in all directions. Taking a somewhat less conservative viewpoint, if the LEM approaches the lunar surface tangentially, contamination could be minimized by taking samples away from the LEM in a direction opposite to the approach path.

Should secondary power supplies be used to drive the rock coring device or other supplementary equipment, the combustion products of these units would be major sources of contamination and the sampling program should be oriented as far as possible from these activities.

- (2) Gloves and Suits. Because the space suit is pressurized to approximately 3.7 psi and contains many articulating joints with seals, relatively large amounts of gas leakage will accompany the exploring astronaut during his sampling program. The most serious leakage will be from the gloves and wrist joint which are in close proximity to the hand tools and samples during acquisition and packaging. Samples which have been handled by the astronaut will inevitably contain traces of glove contamination which will be particularly annoying during laboratory analysis, since it will be present in random amounts on nearly all samples.
- (3) Sample Bag Liners. Some material will inevitably be deposited by spalling or abrasion from the inside surfaces of the sample bag liners.

  Aluminum and copper are both being considered as candidate liners and both have the advantage of being readily identifiable.

# (PART II, CONTAMINATION, con't)

A more subtle source of contamination will be the evolution of gas molecules from the surfaces of the bag liners. The type and extent of such contamination will depend largely upon the effectiveness of the cleaning procedures and the storing provisions employed, which places great importance upon bake-off, vacuum stripping, and electron beam techniques for pre-cleaning.

- (4) Polyester Film. Unlike fluorocarbons, which have a known signature when viewed with the spectrometer, there is little reliable information identifying polyester films. If such a material is to be used, considerable pre-launch identification experience must be obtained. A secondary influence implied by the use of foil and laminates is the amount of gas trapped between layers by the manufacturing process. An investigation of techniques to strip and minimize this effect must be pursued if the laminating technique is employed. In addition, the type of adhesive used to bind the laminations is of extreme importance and mass spectrometer signatures and/or other identification must be obtained, as with other sample container materials.
- (5) Outgassing. Seal and wall outgassing are always potentially troublesome sources of contamination, but the materials and pre-treatment criteria established in Part I should minimize their adverse effect.
- (6) LEM Contamination. Inasmuch as the principal source of earth bacteria released on the moon will be the LEM's atmosphere, the atmosphere within the LEM should be vented through an ultra high efficiency biological filter. The redundant sealing capabilities of the sample box and the internal sample container bags and canisters will minimize the chances of contamination from the LEM and CM atmosphere during the trans-earth phase.
- (7) Receiving Station Influences. Considerable coordination will have to be effected between the sample container program and the design and construction of the facilities in the receiving station. The proceedings of the American Vacuum Society and the minutes of the meetings of Committee E-21, American Society for Testing of Materials, contain many references to excessive accidental contamination in large and small vacuum installations. These contaminants have been attributed principally to (1) backstreaming from diffusion pumps and (2) the boil-off of low ends from plastics, wire insulation, seals and electronic components. Extreme care must be exercised in all facets of the receiving station design, or the engineering, cleaning, and handling processes used for the sample container design program will be invalidated.

(PART II, CONTAMINATION, con't)

## IDENTIFICATION OF CONTAMINANTS

It is important that an orderly program be developed which will give investigators in all disciplines an opportunity to evaluate the sources and types of sample contamination. In this regard a series of control tests should be designed to consider contamination sources from: (1) basic packaging materials; (2) surfaces of packaging materials; (3) astronaut handling; (4) LEM combustion products; (5) adjacent samples; (6) materials introduced at the receiving station; and (7) materials introduced by the investigating agencies.

The aluminum used for construction of the boxes and peripheral equipment, the materials used for all seals, and the materials of the inner containers and bags should all be made in single batch processes. The batches should be sufficiently large to provide for the entire Apollo program, including control samples. This will reduce the amount of peripheral investigation required during later missions to identify trace contaminants by laboratories competent in obtaining signatures of materials through mass spectrometry and other applicable means. The control samples obtained at the time of fabrication will be used during later investigations to help identify trace contaminants.

#### PART III

#### SAMPLE ACQUISITION PROCEDURES

#### **OBJECTIVES AND CONSTRAINTS**

The details of the procedures by which the astronaut will obtain, identify and package uncontaminated lunar surface geological, biological and other samples are dependent upon the size of the samples obtained, the number of samples in each size category, the character of the samples, i.e., whether dust or hard rock, and the amount of individual separation required. The intent of this study was not to provide the criteria by which samples would be selected, but rather to examine the packaging implications of various sampling philosophies. Since the available packaging schemes are limited by the small amount of time available to the astronaut, recommendations on identification, orientations and selection criteria have been developed in conjunction with the acquisition procedures.

#### IDENTIFICATION

A numbering system for the flexible and rigid containers is the most acceptable means of sample identification. Tests indicated that black numbers 2-1/2 inches high on an aluminum foil bag were plainly visible on photographs taken from a distance of 27 feet under normal outdoor lighting conditions. When the outer liner of the flexible sample bags has been finalized, color, background, and texture of the numbers can be selected for optimum contrast. The exploring astronaut's comments while picking up rocks and his identification of bag numbers should be done by automatic voice recording indexed to real time. Photo recording by hand-held, ship's, and/or head-held cameras will provide additional sample identification. Real time marks on the film for positive indexing to the recorded voice comments can be an essential identification aid.

# ORIENTATION

The problem of properly orienting the earth's investigator to the precise position on the lunar surface at which any particular sample was acquired involves first the gross location of the lunar landing site. Work currently under way by Dr. Shoemaker and others on detailed mapping of the lunar surface will provide adequate reference material from which orbiting and descent photographs can be matched to identify major lunar landmarks. Having determined the landing location, the LEM must then be oriented to its immediate landscape, and the location of each sample oriented to the LEM. A simple procedure is outlined in Figure 9, based on the assumption that the astronaut's hand-held camera can be provided with a mil scale or other angle measuring device.

The orientation procedure will require the astronaut to do two things:

- (1) Proceed at right angles to the front of the LEM a nominal distance; for example, 100 feet, as measured by a light cord, and erect light-weight poles with colored flags.
- (2) At each sample location, take a photograph of the LEM and range pole(s) with flag(s) visible in the picture.

The unique configuration of the LEM makes it possible to determine the azimuth at which any given LEM photograph was taken. This, together with the photographically measured angle between the LEM and the flag, and the measured distance between the LEM and the flag provides all that is necessary to determine by simple triangulation the positions of samples relative to the LEM. The system is simple, requires no surveying instruments or special angle or distance measurements by the astronaut, and involves a minimum of extra weight (poles, flags and cord).

This system, or an equivalent, is recommended on the basis of studies of the time required for packaging activities. Every additional second used for identification activities will detract from the exploration program, and may mean less than the full complement of samples will be packaged and returned.

The orientation of individual samples to the topography of their immediate surroundings (5-10 ft) can again best be handled by photographic means. Prearranged packaging procedures including scribe marks on certain samples indicating lunar north and vertical orientation are essential, and the combination of voice recording and photographs will provide positive identification and adequate supplementary data records.

# ROCK HANDLING

The acquisition and packaging of large rocks presupposes no particular problem, since the flexible inner sample container bags are ideally suited for rocks of the four to six inch diameter category. For samples below three inches, individual packaging becomes prohibitively time consuming.

There seems little doubt that in the case of aggregates, gravels, pumice, or dust, that bags with hoops in the throats which can be used conveniently as scoops provide a convenient approach. In other instances, it may be desirable to acquire undisturbed surface dust or pumice samples which preserve the soil structure for later analysis, which can be accomplished by hand coring with thin metallic test tubes.

Samples from machine driven coring devices can be packaged in separate

(PART III, SAMPLE ACQUISITION PROCEDURES, con't)

tubular containers and stored in available locations outside the sample boxes. Specific handling and storage for these special samples will be covered in subsequent investigations.

# TRANSPORTATION TECHNIQUES

The sample box will be a light weight, highly engineered structure which should not be carried by the astronaut during his sampling explorations. Since it would not be desirable to expose LEM atmosphere to the highly cleaned interior of the box, it should be transported some distance (20-30 ft) from the LEM before being opened. The geologic sample bags and other special-purpose containers will then be removed from the sample box, carried in a pocket or hooked to the Jacob's staff or walker, and individually dispensed. For the return of samples to the box, a simplified bandolier or pouch will suffice, inasmuch as the total sample weight will produce a reaction equivalent to only ten earth pounds while on the lunar surface.

#### SAMPLE BOX PACKING

Packing of the various samples within the rock box will follow a logical pattern. The special containers will be inserted first into special clips on the inside faces of the container. A sufficient number of small (2 in. or less) samples will then be inserted to fill the spaces between the egg crate dividers. Intermediate sized (2-4 in.) samples will then be placed in the far corners of the box, followed by the two large (4-6 in.) samples at either end of the box. The remaining spaces still visible around the larger samples will be filled by additional small samples until the weighing device indicates a full load.

It was originally believed that the principal packaging constraint would be the weight limitation, since even relatively light rocks (sp. gr. 1.0) would exceed the 55-60 pound weight limitation in the 2 ft. 3 space allocated. The addition of individual bags for packaging each rock sample complicated the analysis by adding the tare weight of the bags and the time required to dispense the bag, drop the samples in, close, seal, and stow the package in the sample box.

Table II illustrates two approaches—one filling the available volume with individually wrapped samples and the other combining several samples per bag in the smaller sizes. As can be seen, packaging time is cut in half and sample weight is increased 40% by the latter approach. Note also that for an average sample specific gravity of 2.0, a nearly optimum 56.0 lbs. of filled sample bags can be stowed in the two boxes. The volume and weight required for gas sampling, biological and other special containers has been neglected, although they could affect the results by 20%.

TABLE II SUMMARY OF PACKAGING DATA Number Time Sample Diameter Number Total Weight with Specific Gravity (3) for of of and Packaging Packaging Scheme Samples Bags (2) 1.0 2.0 3.0 (4) (1) One inch: 100 (5.7)50 min. individual bag 100 (1.9)(3.8)3.5 5.4 7.3 (11.4)20 min. 40 (3.8)(7.6)five per bag 200 8.3 12.1 4.5 Two inch: 50 50 (7.6)(15.2)(22.8)25 min. individual bag 8.4 16.0 23.6 90 30 (13.6)(27.2)(40.8)15 min. three per bag 27.7 41.3 14.1 Three inch: 10 (5.1)(10.2)(15.3)5 min. 10 individual bag 5.3 10.4 15.5 Five inch: 2 2 (4.7)(9.4)(14.2)1 min. individual bag 14.4 4.9 9.6 Totals for indi-(19.3)(38.6)(58.0)vidually pack-81 min. 162 162 22.1 41.4 60.8 aged samples: Totals for multiple packaging (27.2)(54.4)(81.7)of samples plus 82 28.8 56.0 41 min. 3 and 5 in. 302 83.3 FOOTNOTES: Number of samples per bag. 1. 4.5 inch diameter for sample sizes up to 3 inches, 8 inch diameter 2.

# TABLE II SUMMARY OF PACKAGING DATA (FOOTNOTES, con't) for over three inches in diamter. 3. Numbers in parenthesis are sample weights. Numbers without parenthesis are sample plus bag weights. 4. This time is packaging only. It does not include time to locate sample or describe outcrop 5. For additional details, see Appendix D.

## GAS SAMPLE ACQUISITION

Experimenters who are defining tests to identify traces of gas from the surfaces and interstices of rock samples are interested in probing, if possible, the interior of each package, whether flexible or rigid before the containers are opened for other experiments. For this purpose, gas sampling nipples having a diaphragm which can be ruptured by inserting a hollow needle can be affixed to each container.

Additionally, at least two containers capable of holding 25 grams of dust or other maximum surface-to-weight ratio samples will be constructed. No special requirements are known regarding collection of the sample, but a highly reliable gas seal is mandatory. Construction details of the gas sampling container will be extremely critical, as summarized in Part I-C, "SPECIAL CONTAINERS".

There is some feeling that dry ice or other frozen gases may be trapped below the lunar surface and that samples of this material may build up significant pressures as sublimation occurs. Other considerations along similar lines cite the characteristic evolution of gas when activated charcoal, which has been saturated with gas, is thawed from a frozen state. The behavior of any lunar sample which approximates either of these examples could be troublesome from a packaging standpoint, and some thought must be given to the possibility of the astronaut pre-screening his samples to eliminate such materials, if possible. The alternative is to provide a pressure relief system which will prevent gas buildup, but this would not become effective until the protective lunar vacuum in the storage container had been overcome, and the chances of intrasample contamination would thus be greatly increased.

#### BIOLOGICAL SAMPLE ACQUISITION

The initial thinking in connection with the acquisition of biological samples was that a highly specific asceptic procedure would be required to obtain meaningful samples. Subsequent investigations, taking into consideration the severe demands upon the astronaut's time and his limited dexterity tend to put greater reliance on the configuration of the sampling device and a few rudimentary procedures. For example, since the gloves on the thermal outer garment will be highly contaminated, the astronaut should not touch the samples directly. Perhaps the most practical tool configuration would be a long (18-24 in.) lightweight telescoping tube which could probe beneath the surface.

The normal airborn vehicles for transmitting contamination from the glove to the samples will be entirely lacking in the lunar vacuum making unnecessary much of the surgical cleanliness concepts of the "sterile field", etc. The outgassing from the gloves and leakage from the articulating joint seals of the astronaut's pressure suit are potential contaminants, but

(PART III, SAMPLE ACQUISITION PROCEDURES, con't)

these sources are expected to dissipate and minimize possible transport of contaminants.

Control experiments should establish in advance the characteristic contamination emanating from the astronaut. Satisfactory pre-test situation must be simulated in a man-rated space chamber to prove control of the clean containers or canisters proposed for the lunar surface samplings.

# SAMPLE BOX SEALING

Some experts believe that the lunar surface will be quite "dusty". Low gravity particles stirred up by the LEM's landing may be attracted to the box and its sealing surfaces. To avoid this, the astronaut will strip a protective cover from the seal and its mating surface just before closing the lid. Even with this procedure some further dusting may occur, but the redundant seal minimizes the probability of vacuum failure from this cause.

#### APPENDIX A

#### SUMMARY OF OPINIONS FROM SCIENTIFIC COMMUNITY

# 1.0 INTRODUCTION

The opinions of scientific experts are an essential component in the solution of the lunar sample acquisition problem. This appendix reports a sampling of comments and suggestions by various members of the Early Apollo Sciences Teams and other persons suggested by NASA. It should be noted that the following summary is the contractor's best attempt to present often diverse opinion of the experts interviewed, each of whom spoke principally from the viewpoint of his own discipline. The material is segregated into the following four categories:

Protection from Physical Damage (Section 2.0)
Protection from Contamination (Section 3.0)
Sample Acquisition (Section 4.0)
Sample Make-up, Thermal Protection, Other. (Section 5.0)

Each section has been arranged in sub-sections according to the fields of interest of the contributors. Those contacted included:

- Dr. James R. Arnold, Univ. of Calif., San Diego, Calif.
- Dr. P. R. Bell, Oak Ridge National Laboratory, Oak Ridge, Tenn.
- Dr. Klaus Bieman, M.I.T., Cambridge, Mass.
- Dr. M. Calvin, Univ. of Calif., Berkeley, Calif.
- Dr. Clifford Frondel, Harvard Univ., Cambridge, Mass.
- Dr. V. W. Green (for Dr. Gaylord Anderson), Univ. of Minn.
- Dr. A. J. Haagen-Smit , Calif. Inst. of Tech., Pasadena, Calif.
- Dr. Kurt Hemenway, Dudley Observatory, Albany, N. Y.
- Dr. R. V. Hoffman (for Dr. C. R. Phillips) Ft. Detrich, Frederick, Md.
- Dr. R. M. Lemon, Univ. of Calif., Berkeley, Calif.
- Dr. J. Hoover Mackin, Univ. of Texas, Austin, Texas
- Dr. Brian Mason, Am. Museum of Natural History, N. Y., N. Y.
- Dr. Wayne Meinke, National Bureau of Standards, Washington, D.C.
- Dr. Peter Signor (for Dr. Paul Gast), Univ. of Minn., Minneapolis, Minn.
- Dr. Karl Turekian, Yale Univ., New Haven, Conn.
- Dr. Aaron Waters, Univ. of Calif., Santa Barbara, Calif.

Comments by individual contributors are noted by separate paragraph numbers within the disciplines noted, as with 2.1.1., 2.1.2 below.

# 2.0 PROTECTION FROM PHYSICAL DAMAGE

## 2.1 Bio-Sciences Comments

- 2.1.1 The amount of packing materials depends on the ratio of dust versus hard rocks. If primarily dust, only a small amount (100 cc) need be preserved in structural form. Changes in sample make-up with depth are important to preserve and identify.
- 2.1.2 The physical condition of the samples is important only from a surface contamination standpoint, i.e., the smaller the samples, the larger the ratio of surface area to volume.

# 2.2 Geo-Chemistry Comments

- 2.2.1 Protect samples by keeping them rigidly fixed, not cushioned.
- 2.2.2 Rock protection is not a primary concern. Broken rock samples will be acceptable.
- 2.2.3 Rock samples are apt to be either very fragile or very tough.

# 2.3 Mineralogy and Petrology Comments

- 2.3.1 Most of project objectives will be met even with unprotected samples.
- 2.3.2 At least one special sample should be protected against vibration to preserve surface texture.

# 2.4 Geology Comments

2.4.1 Abrasion important from standpoint of sample container strength, not the effect on samples.

# 2.5 Other Sources

- 2.5.1 Lunar surface may be covered with a thick dust layer which is structurally weak. Except to preserve a structural sample, packaging should not be critical.
- 2.5.2 Low density dust material is not anticipated since particles should fall back to lunar surface at high velocity due to lack of atmosphere.

# 3.0 PROTECTION FROM CONTAMINATION

#### 3.1 Bio-Sciences Comments

3.1.1 Any container contaminant should be a common identifiable material. The most undesirable contaminants would be uranium, thorium, lithium, berrilium, boron and lead. Next in importance would be rubidium, strontium and the rare earths. In general, all contamination by elements with higher molecular weights than nickel should be avoided if possible.

Iron, aluminum and silica would be satisfactory packaging contaminants. Packaging materials should be gas-free.

Catalysts used in the manufacture of many plastics may contain trace metals which would mask parts of the investigations.

A small inner container should suffice for ultrahard vacuum preservation of the biological sample. All other samples should be stowed in the vacuum sample container.

3.1.2 Inert gas atmosphere in sample box is satisfactory.
A few spores would not particularly hurt organic chemical evaluation. Silicone should be avoided.
Rocket fuel combustion products are very important.
A single batch should be made sufficiently large such that the spacecraft requirements and subsequent evaluations can be completed on the same batch.

No plastics of any kind should be used except compounds such as fluorocarbons with known signatures. Polyester films would have to be very carefully analyzed and identified. Gold, aluminum, titanium are satisfactory minerals.

3.1.3 Probably will not find any germs on moon.

Satisfactory sterilization procedure would be to heat innermost components to 135°C for 24 hours, with correspondingly higher temperatures on outer walls.

Five (5) percent of spacecraft electronic components have been found to be contaminated.

A pressurized box with inert atmosphere would not be harmful. Satisfactory inert gases are helium, argon, and nitrogen.

The exterior of sample box could be sterilized upon return to earth by washing with fuming nitric acid, then washing with distilled water.

Viruses are not likely to be found because of their specialized nature. Sterilization is aimed at bacteria, not virus.

3.1.4 The technology for sterilization and acquisition of lunar biologic samples is similar to earth procedures. Sample containers must be sterilized by classical techniques. Baking at 600-800°C is satisfactory.

Samples should be packed in containers in which experiments will later be done, rather than risk transferring samples. Ports for the insertion of a syringe for innoculation of broth directly into samples would be helpful. It would also be helpful if these sample containers were transparent. The entire box must be sterilized before shipment.

# 3.2 Geo-Chemistry Comments

- 3.2.1 Doubt that trace contamination in a reasonably protected sample will impede analysis work.
- 3.2.2 Maintain 2-10 pounds of samples in ultra-high vacuum. Remainder of samples should have reasonable protection.

  Suggest that high purity or equivalent is adequate for all but the ultra-high vacuum canister.

Contamination from packaging materials could be a serious problem. Aluminum is one of the materials which would be satisfactory for an inner bag.

3.2.3 Partial pressures of suit and gloves may be an important source of contaminating gasses.

10<sup>-7</sup> torr is adequate box pressure for most samples. Preserving the lunar environment is important for only a very small percentage of samples.

Suggest a high purity aluminum for inner bag liner.

3.2.4 Rare gas as a storage preservative should not be used. Clean nitrogen, if used, would have to be superclean, but would prefer no gas.

Should not use getters, and containers should not be pumped. Otherwise, gas production analysis would be invalidated.

Diffusion of gas from one container finding its way into an adjacent bag is not a serious problem.

Bag should be carried to the moon in a hyper-clean state. Ion bombardment is a much better method than electron bombardment for precleaning due to higher energy levels.

3.2.5 When sample boxes arrive they should be checked to see if leakage has occurred either from outside in or from inside out. Boxes should then be disinfected and placed in secondary containers at 10-6torr, to minimize chances of further terrestrial contamination.

Control bags identical to those in the sample box should be analyzed for outgassing during the mission. High vacuum samples should equal five percent of total sample weight. Would prefer these to be in many two to four-gram containers rather than one large container. Glass breakoff containers should be designed to weigh under 30 grams, and would provide a total of 50 samples in the 5% weight allowance.

A pressure relieving device is probably essential, since a possibility exists that certain samples could evolve large volumes of gas.

Effective outgassing of container cannot be done at temper

Effective outgassing of container cannot be done at temperatures below 600-800°C. Recommend copper rather than aluminum foil because of this.

# 3.3 Mineralogy & Petrology Comments

3.3.1 Interaction between samples, individually bagged in rock box is likely to be negligible.

Biological findings are likely to be prejudiced by minimal packaging protection -- but this is small part of overall objective.

If  $10^{-14}$ torr cannot be maintained in box or around samples, then  $10^{-3}$  or  $10^{-2}$  may be as acceptable as  $10^{-8}$  or  $10^{-9}$ .

3.3.2 The spacecraft appears to be the major contamination source.

Sample box should maintain  $10^{-8}$ torr vacuum. Another approach may be to use a controlled atmosphere of nitrogen.

Inner sample bags need not maintain vacuum. However, special containers should be provided for lunar gas samples.

# 3.4 Geology Comments

3.4.1 Oxidation should not be a major problem because most dry rocks are relatively inert.

#### 3.5 Other Sources

- 3.5.1 Samples may be reactive with water and oxygen. Protection must be afforded until the degree of reactivity can be determined.
- 3.5.2 Fluorocarbon or polyethylene are satisfactory as sample containers. No strict constraints except container should not generate particles. It would be a good idea to have two kinds of containers, such as stainless steel and fluorocarbon, to check one against the other. Should not use silicone or titanium.
- 3.5.3 Polyester films must be degassed before coating.

Should not use lead, gold, or indium for seal. Avoid rare gases.

Anticipate difficulty in achieving seal due to flying dust particles. Recommend seal be protected with a non-sticky gas or liquid film.

# 4.0 SAMPLE ACQUISITION NOTES

#### 4.1 Bio-Sciences Comments

- 4.1.1 During the first mission, general roconnaissance is of prime importance. Therefore, many small containers should be used so as to get representative materials. Samples must be labeled. Notation of orientation may be important to ascertain cosmic ray and solar wind activity, and the astronaut should therefore attempt to denote vertical and north orientation. Photographs should be taken showing sample orientation, bedding planes, striated structure, etc. Try to bring back coherent and semi-coherent dust layers in separate bags, if possible.
- 4.1.2 Acquisition of microbiological samples might be done with a hollow coring tube, using fluorocarbon plugs as spacers, and keeping the tube as a storage device.

It is more important to obtain reliable samples than to maximize returned sample weight. Would prefer, for biosciences purposes, 25 1-gram samples, 5 or 10 samples weighing 100 to 250 grams, and one sample weighing 1000 grams.

4.1.3 Astronaut training is a most important facet of acquiring biologic samples. Microbiological data may also be acquired from geological samples. Configuration of sample container is not a constraint, but transparency would be useful. Initial acquisition sample containers should be designed to accommodate later laboratory experiments.

#### 4.2 Geo-Chemistry Comments

- 4.2.1 Very particular about sample orientation--which side up, lunar north, etc.
- 4.2.2 Do reasonable sample orientation work--do not trade samples to gain data.

Fill one box first, then the other to simplify packaging.

4.2.3 Sample orientation (up, north) and temperature not too important.

Astronaut constraints are an important acquisition limitation.

4.2.4 Interested in analyzing gases in the sample, as well as on the sample. Therefore, would like one surface sample and several at increasing depths.

Must have several gas sample containers so experiments can be repeated to check for reproducibility and quality of work.

Suggest copper foil rather than aluminum for bags, since copper can be degassed at 800°C while the temperature limitation for aluminum is 450°C. In addition, copper is better for pinch sealing, since aluminum must have a clean surface which is highly reactive, and copper can be crimped without special surface treatment. Suggest manufacturing technique investigations involving dipping copper mesh into copper.

Signal to noise ratio for gas studies is bad with small bags, and must have several large bags for higher quality experiments. Bags must have navel with puncture diaphragm 1/8 to 3/8 in. diameter for gas sampling.

#### 4.3 Mineralogy and Petrology Comments

4.3.1 Several hundred 1-1/2 in. diameter bags should be provided. Five-gram samples (pebble size) are adequate.

Generally prefer a large number of small samples about 1/2 in. The astronaut should have a device for determining the temperature of samples.

Special sampling tubes should be provided for dust samples, gas samples, and surface texture preservation.

## 4.4. Geology Comments

4.4.1 If the lunar surface is a homogeneous plain, sample location is not too important.

Orientation (up, north, etc.) is important for geophysical but not for most geological purposes.

Calibration of grain size, if above sand size range, is best obtained from on-site photographs.

#### 4.5 Other Sources

4.5.1 Emphasis must be placed on redundancy of sample makeup so that experimentors can check their results.

Anticipate high electrostatic charge.

#### 5.0 SAMPLE MAKE-UP, THERMAL PROTECTION, OTHER

#### 5.1 Bio-Sciences

- 5.1.1 Concerning temperature preservation:
  - (1) Exposed rocks in vacuum are not likely to boil off gas.
  - (2) Permanent shadow locations near the lunar poles may trap volatile gases. This would be of great interest in later missions.
  - (3) During later missions, the sample boxes should be refrigerated.
  - (4) Try not to let sample containers get hot (150°F).
- 5.1.2 Sample temperature variations are not a primary concern.

5.1.3 Microbiologists must have access to samples first in order to examine for presence of organisms. Need only 5 or 10 samples weighing a few grams for microbiological tests. Formaldehyde and alcohol or ethylene oxide in gaseous form can be used to sterilize the outside of the container.

# 5.2 Geo-Chemistry Comments

- 5.2.1 Rather see more weight of samples brought back than trade payload for sophisticated packaging techniques. Want a representative samples.
- 5.2.2 Thermal protection between high and low temperature samples is not important for first mission.
- 5.2.3 Order of sampling is potentially important for statistical purposes.

# 5.3 Mineralogy and Petrology Comments

5.3.1 Most important to have at least one large rock 8-10 pounds.

#### 5.4 Geology Comments

5.4.1 Thermal conditions not important--temperature cycling has occurred on the moon surface.

Age determination will require a large bulk sample.

If ledge or outcropping is homogeneous, any sample will do. If not, go after peculiarities. Size of speciments should be dictated by grain size.

## 5.5 Other Sources

- 5.5.1 Samples should impart no particular radioactive hazard.

  However, warming of cold samples may anneal dislocations or crystallization due to radiation.
- 5.5.2 Specific gravity is expected towary from 1.5 to 3.0.

  Recommend coffee can type strip around box periphery
  to assist in opening at receiving station. Special
  containers must be provided for low-level counting.

#### APPENDIX B

## BREADBOARD SAMPLE BOX VIBRATION TESTS

#### 1.0 INTRODUCTION

One of the most critical requirements for the packaging system is to preserve the integrity of the samples during launch and re-entry vibration regimes.

This series of tests evaluated the sample protection provided by the compartmentation and flexible bag packaging system proposed during the current study. Vibration effects on the breadboard sample box were not considered relevant to the ultimate structural design.

## 2.0 TEST ARTICLES

- 2.1 Breadboard Samples Box, per Figures 10. Compartment edges protected with plastic tubing.
- 2.2 Six Flexible Samples Containers, 3 in. diameter by 6 in. long, constructed of a four-ply lamination of a 1/2-mil aluminum foil inner liner, a 1/2-mil polyester film second ply, a 1/2-mil polyester film third ply, and a 1/2-mil aluminum foil outer liner.
- 2.3 Polyethylene and aluminum foil bags
- 2.4 Assorted large (4 in.) and small (1 to 2 in.) rock specimens sp. gr. 2.2.

## 3.0 TEST EQUIPMENT

Ling Model A 300 B Shaker and associated calibration and readout equipment.

## 4.0 TEST PROCEDURES

## 4.1 Test No. 1

Sinusoidal vibration, 5-70 CPS (sweep) at 0.04 in. Double Amplitude, followed by 70-2000 CPS (sweep) at 10 G's. Five minutes total run time. Empty box.

# 4.2 Test No. 2

Sinusoidal vibration, 5-44 CPS (sweep) at 0.20 DA, followed by 44-1200 CPS (sweep) at 20 G's. Five minutes total run time. Ten pounds of small rocks added to box, approximately  $150 \, \text{in}^3$ . (See Figure 11)

# 4.3 Test No. 3

Sinusoidal vibration for two minutes at 30 CPS, 0.20 DA, 9.5 G's. Same rocks as Test No. 2, except one 4-pound rock sample (approximately 60 in<sup>3</sup>) added to the box.

## 4.4 Test No. 4

Sinusoidal vibration for two minutes at 30 CPS, 0.10 DA, 4.7 G's. Same rocks as Test No. 3.

## 5.0 RESULTS

## 5.1 Test No. 1

Resonance noted at 602 and 647 CPS. No damage resulted to the box. Compartmentation tack welds failed at 5 locations, but walls remained rigid and structurally satisfactory.

# 5.2 Test No. 2

- 5 CPS No sample movement.
- 15 CPS Rocks moved violently, but tinfoil and laminated bags moved much less than polyethylene wrapped samples.
- 29 CPS Rocks moved much more, jumbled from one compartment to next; tinfoil and laminated bags continued to move less than polyethylene-wrapped samples.
- 66 CPS Movement of samples reduced; foil and laminated bags did not move.
- 190 CPS Occasional movement.
- 330 CPS Movement stopped, except sand spilled from one bag and moved violently.
- 800 CPS No sample movement.
- 1200 CPS No sample movement.

#### 5.3 Test No. 3

All rocks, including large rock, tumbled violently. Plastic protection at top of egg-crate dividers worn through. Small bags punctured by contact with bolt-heads. Large sample bag punctured by top edge of compartmentation (see Figure 12).

## 5.4 Test No. 4

Significant movement of samples but much less than during Test No. 3. Bags cut and abraided, but not as extensively as illustrated in Fig. 12.

#### 6.0 DISCUSSION

Having verified that the box was structurally sound during the first test, samples were added and amplitude increased to accelerate wear during the second sweep frequency test. The maximum movement of samples occurred during the second test at about 30 CPS; the third test was concentrated at that frequency to accelerate wear on the sample container. The following table shows the comparison between specification requirements (Procedure IX, Environmental Specifications for Apollo Scientific Equipment) and this series of screening tests.

TABLE III

	EQUIPMENT WEIGHT	5-27.5 CPS	27.5 to 52 CPS	52 to 500 CPS
SPEC REQMTS	Less than 50 lbs	1.56 G	0.043 in. DA	6.0 G
TEST #1	28 lbs	0.04 in. DA	0.04 in. DA	10G at 70 CPS 25G at 600 CPS
TEST #2	38 lbs	0.2 in. DA	0.2 in. DA	20G
TEST #3	42 lbs		0.2 in. DA 30 CPS (9.5G)	
TEST #4	42 lbs		0.1 in. DA 30 CPS (4.7G)	

The bag degradation experienced during Tests 3 and 4 was much more severe than would actually occur because of (1) accelerated test conditions (approximately 5 times specification requirements) and (2) unprotected projection and egg-crate dividers (plastic tubing protection wore off in the first 1/2 minute). Nevertheless, the results do show that material tougher than the 4-ply foil and polyester film laminate is required. The movement of samples enveloped in laminated bags was less than those protected only by polyethylene, indicating that the foil damps sample vibration. However, a means of securing the samples against movement is mandatory.

With respect to the dividers, the tops of the compartment walls can be rolled over to form a generous radius, eliminating this as a potential source of wear, and also greatly increasing their value as an integral component of the structural system of the sample box.

## 7.0 CONCLUSION

7.1 The four-ply aluminum foil to polyester film laminate material used for inner sample bags during these tests was partially effective in damping rock movement during intensive vibration of the sample container.

Plastic materials were not effective for vibration damping.

7.2 The four-ply aluminum foil to polyester film laminate material (0.0005 in. per layer) was not adequate in resisting puncture and abraision under the vibration conditions of the test.

# APPENDIX C SAMPLE BOX PACKAGING DEMONSTRATION TEST

#### 1.0 PURPOSE OF TEST

The packaging concept, including placement of individual samples in flexible bags, sealing the bags and stowing them in the sample box, was subject to design constraints imposed by the limited dexterity of the astronaut. The proposed system, therefore, emphasizes simplicity and ease of handling, and numerous in-house tests were done by the contractor to simulate as nearly as possible conditions of use.

It was recognized from the beginning that the problems of limited visibility, restricted suit movements from differential pressure, and limited degrees of freedom in the astronaut's gloves would be impossible to completely simulate. Therefore more formal evaluations were conducted in the NASA-MSC Crew Systems Laboratory using a technician in a state-of-the art suit to verify the results. This report summarizes the results of the Crew Systems Laboratory tests.

# 2.0 TEST ARTICLES

- 2.1 Breadboard Sample Box, per Figure 10. Compartment edges protected with plastic tubing.
- 2.2 Six Flexible Sample Containers, 3 in. diameter by 6 in. long, constructed of a four-ply lamination of 1/2 mil aluminum foil inner liner, 1/2 mil polyester film, 1/2 mil polyester film, and 1/2 mil aluminum foil outer liner.
- 2.3 Assorted polyethylene bags, some with aluminum foil liners.
- 2.4 Assorted large (4 in.) and small (1 to 2 inc.) rock specimens, average sp. gr., 2.2.
- 2.5 Finely washed beach sand.
- 2.6 Random pebbles, 1/2 to 1 in.
- 2.7 Random sized (to 4 in.) slag rocks from NASA MSC simulated landing site.

## 3.0 TEST EQUIPMENT

3.1 Early Gemini pressure suit, together with associated communications and air pressure supply equipment.

3.2 Black and white still photographic equipment, 16 mm color motion picture equipment and associated lighting.

## 4.0 TEST PROCEDURE

#### 4.1 Sample Box Tests

The suit technician, using an open end wrench, unscrewed two bolts and removed the lid covering the 8 in. diameter port at the top of the breadboard rock box. After wrapping each individual sample (see Test No. 2) the technician stowed the samples inside the box by reaching into the far corners and then filling the closer compartments.

#### 4.2 Inner Bag Tests

The flexible sample container, an aluminum foil to polyester film laminate, 3 in. diameter at the top and 2-1/2 in. at the bottom, 6 in. long, and stiffened at the open throat with a soft aluminum ring, was tested for general utility, dispensing and sealing capability.

Utility tests performed included (1) placing various size rocks in bags and (2) scooping finely washed beach sand into the bag. Dispensing was accomplished by removing nested bags either by lifting on the handle or by pulling a tag attached to the base of the bag. Sealing was accomplished by crimping the mouth of the bag shut and folding the top over several times so that a long diffusion path, but not a vacuum type seal would be produced.

#### 5.0 RESULTS AND DISCUSSION

## 5.1 Sample Box Tests

The intravehicular gloves were not an obstacle in handling the various containers as illustrated in Figures 13, 14 and 15. Figure 16 shows the technician filling a compartment in the far corner of the box without particular difficulty. Figure 17 illustrates the uniform distribution of samples within the rock box. The thermal outer garment gloves that cover the astronaut's gloves employed during this test will reduce dexterity and increase bulkiness, making ingress somewhat more difficult.

The size of the entry port is marginal because the suit technician questioned his ability to place rocks in the far corners of the box.

#### 5.2 Inner Bag Tests

The small sample bag proved to be a satisfactory scoop in unconsolidated sand. The handle on the bag could be readily grasped and the scooping motion was easily accomplished. Samples could be easily placed in the bag by holding the bag in one hand and dropping the sample in with the other hand.

The dispensing technique tested was easily accomplished. The suit technician demonstrated equal ability in obtaining a bag by lifting up on the bag handle or by lifting on the tab fixed to the base of the bag.

The sealing method was very difficult to accomplish. Due to limited manual dexterity the top of the bag could not be easily crimped. The lack of manual and wrist dexterity made the twisting needed to complete the seal difficult, and only after effort and concentration was the suit technician able to crimp and fold a few bags.

# 6.0 CONCLUSIONS

#### 6.1 Sample Box Tests

The size of the entry port was barely adequate under the conditions of the test, and should be enlarged for the prototype box. Limited compartmentation of the configuration tested was satisfactory for sample packing purposes, but must be reduced in height so as not to limit maximum sample size.

#### 6.2 Inner Bag Tests

Fold-over sealing is not a satisfactory means of bag closure. Aluminum hoops, if used, must be ductile so as not to tend to remain open, or must be sheared off before storing in the sample box.

#### APPENDIX D

#### INNER SAMPLE BAG MATERIALS TEST

#### 1.0 INTRODUCTION

A group of tests was performed on the four-ply aluminum to polyester film laminates initially selected as likely candidates for the flexible inner containers. These tests evaluated the characteristics of the material at high vacuum, and included

Diaphragm diffusion rate tests Seal leakage tests Outgassing rate determination

#### 2.0 DIFFUSION RATE STUDIES

A four-ply laminate consisting of 1/2 mil layers of aluminum, polyester film, polyester film, and aluminum, per G. P. Schjeldahl Tompany Type 717, was placed in a test fixture with  $10^{-5}$  torr on one side of the sample material membrane and positive pressures of 5,10,15,20 and 25 psig helium on the other side. Tests were performed at 74 and  $125^{\rm OF}$ , using a C.E.C. Model 120 mass spectrometer as a leak detector.

Two samples which exhibited high diffusion rates, were later found to have minute puncture holes due to apparent mishandling after fabrication. The remaining samples exhibited no detectable diffusion during 30 minutes under the noted test conditions. The 25 psig tests were extended to 90 minutes without detection of any diffusion.

#### 3.0 SEAL TESTS

## 3.1 Twist Seal

A bag closure was simulated on an open-ended tube of the four-ply laminated aluminum to polyester film material by twisting one end of the tube to effect a closure. The opposite end of the tube was then gripped circumferentially between two flanges and a vacuum pulled simultaneously on both sides of the simulated seal. A mass spectrometer leak detector was connected to one side of the seal.

The admission of helium to the opposite side was detected immediately in the leak detector, indicating gross direct leakage. The test was repeated several times, always indicating a leakage in excess of  $10^{-3}$  std. cc/sec.

#### 3.2 Fold Seal

A second type of seal was tested by folding flat one end of a

tubular section of aluminum to polyester film laminate material and folding the flattened portion back on itself five (5) times in 1/4 in. folds. The opposite end of the tube was then clamped circumferentially between two flanges as before, and a simultaneous vacuum pulled on both sides of the seal to  $10^{-5}$ torr. With a mass spectrometer connected to one side of the seal, helium pressure approximately two decades greater than the pressure from the mass spectrometer side was applied to the seal.

No leakage was detected for eight minutes, after which a diffusion rate equivalent to  $2 \times 10^{-7}$  std. cc/sec. was noted for 15 minutes. Then the leak rate increased steadily until more than  $10^{-4}$  std.cc/sec. had been reached. When repeated, the leak rate was greater than  $10^{-4}$  std. cc/sec. almost immediately, indicating that a permanent leakage path had been established.

A second sample was cleaned with alcohol, set up in the test fixture, and folded five times as before. With  $10^{-5}$  torr on the leak detector side and  $10^{-3}$  helium on the inside of the bag, an immediate leakage of 4 x  $10^{-7}$  std. cc/sec. was detected. See Figure 18 for a comparison of leakage rates.

#### 4.0 OUTGASSING RATE DETERMINATION

Two samples, each consisting of two layers of 1/2 mil polyester film sandwiched between outer layers of 1/2 mil aluminum foil were precleaned in an ion pump for four days at  $150^{\circ}$ F and  $10^{-8}$ torr.

A background curve was obtained on the empty test chamber, pumped to the  $10^{-10} \rm torr$  range, and a second curve was obtained with the precleaned sample in the test chamber.

Comparison between the two curves are illustrated on Figure 19, showing outgassing rates as a function of pressure.

#### 5.0 CONCLUSIONS

#### 5.1 Diffusion Rate Studies

The four-ply candidate aluminum to polyester film laminates are effective barriers against gas diffusion.

#### 5.2 Seal Tests

# 5.2.1 Twist Seal

Seals performed by twisting the mouths of the sample bags closed are unsatisfactory.

## 5.2.2. Fold Seal

Seals found by folding over the flattened mouths of the sample bags are marginal and will not be effective in retaining gases evolved in significant quantities by the lunar samples.

# 5.3 Outgassing Rate Tests

Outgassing of the candidate four-ply aluminum to polyester film laminates material was excessive. Samples tested had not had optimum handling and manufacturing controls for high vacuum applications, nor was pre-cleaning technique adequate. Future sample procurement specifications must contain rigorous quality control provisions.

#### APPENDIX E

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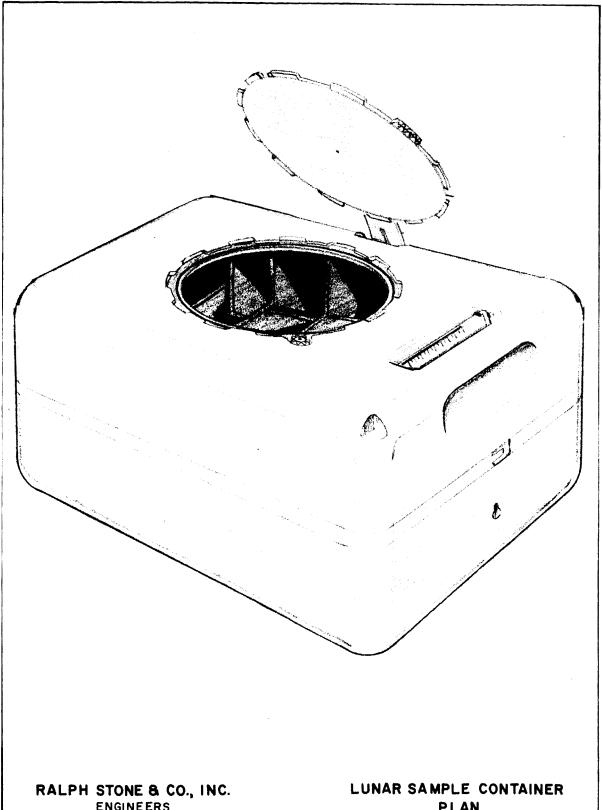
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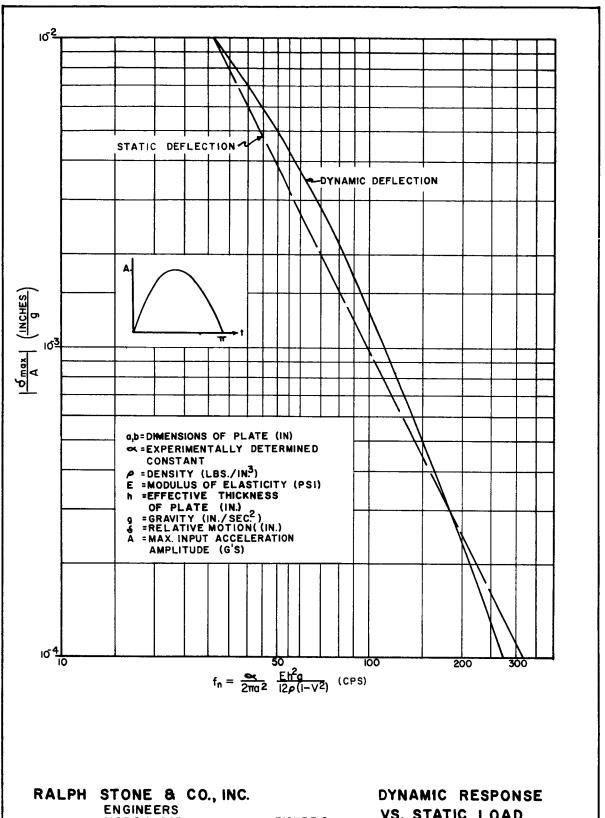
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FIGURE I

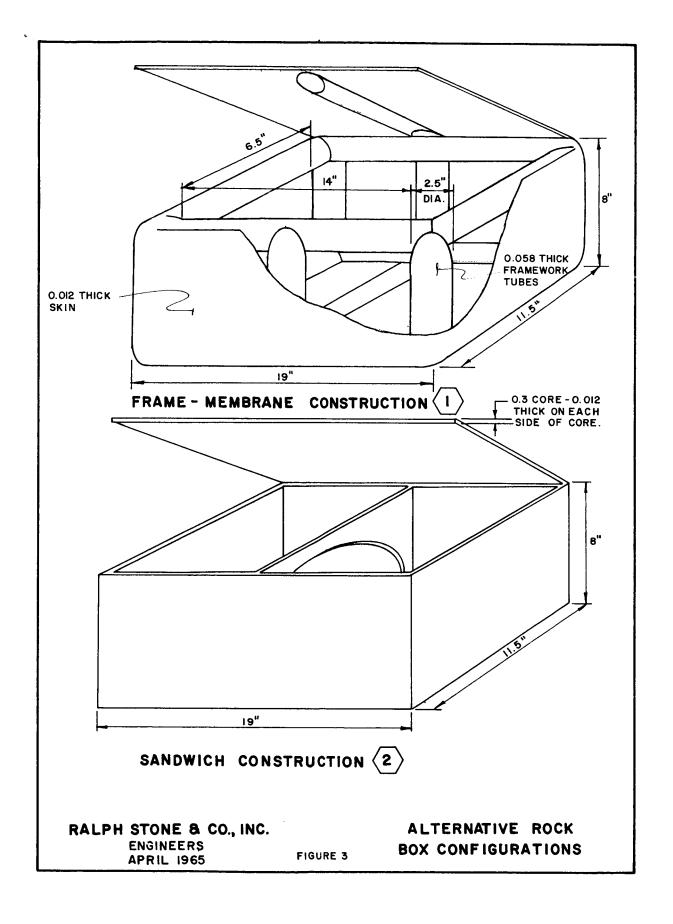
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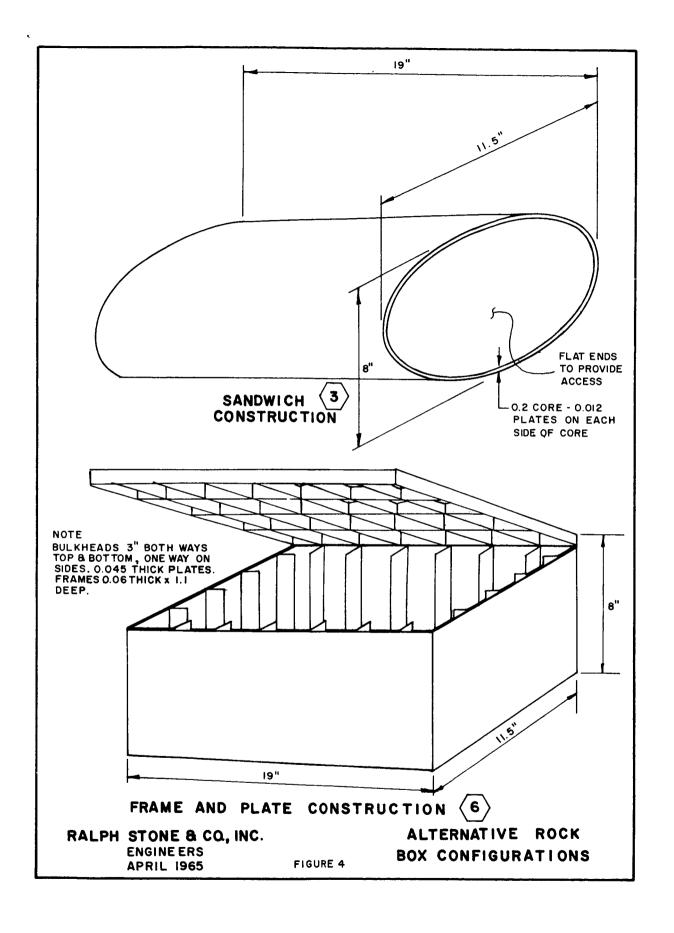


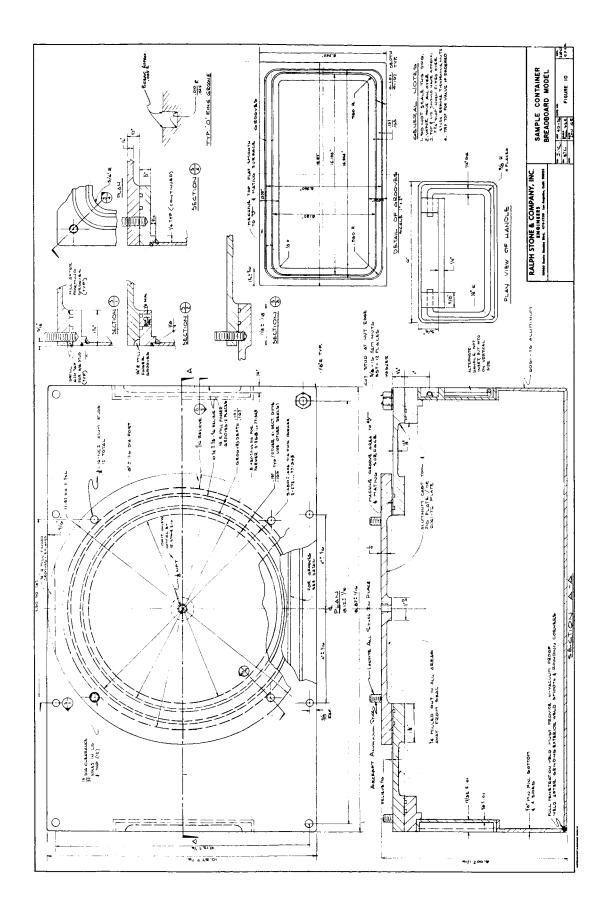
**MARCH 1965** 

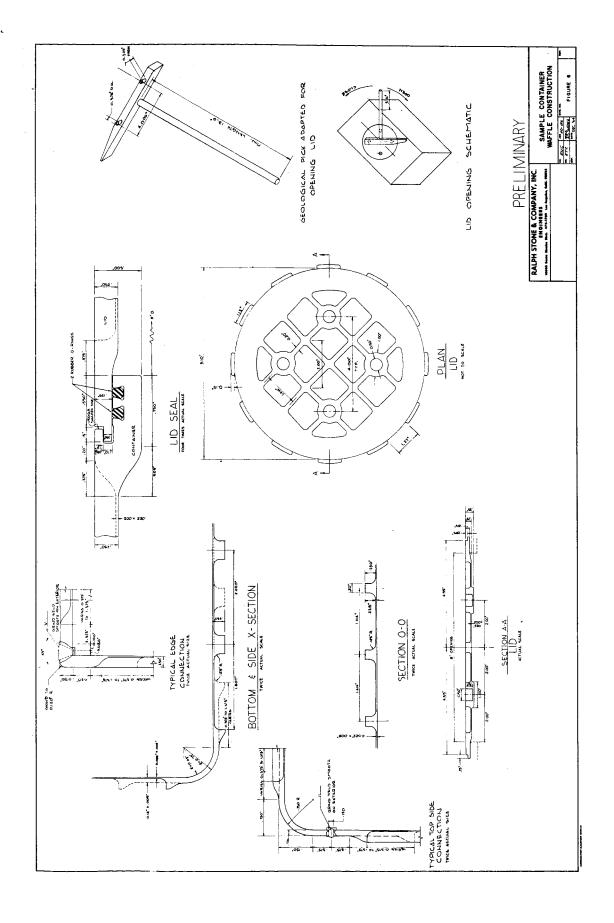
FIGURE 2

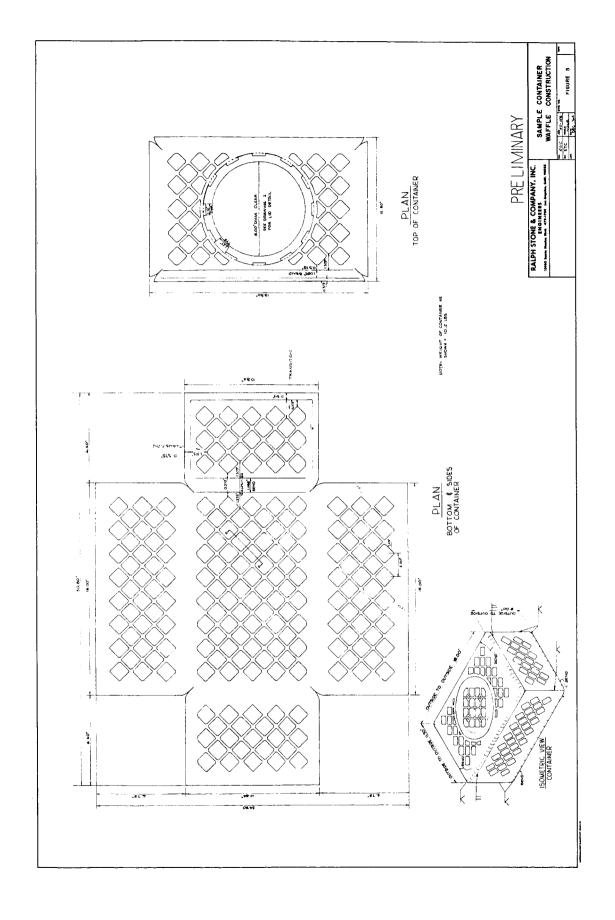
VS. STATIC LOAD

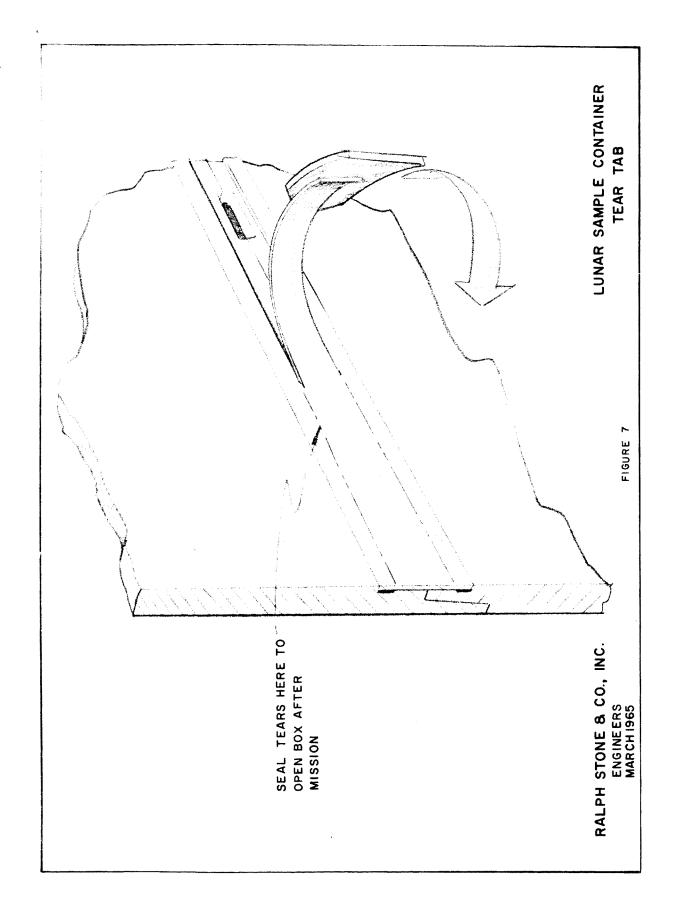


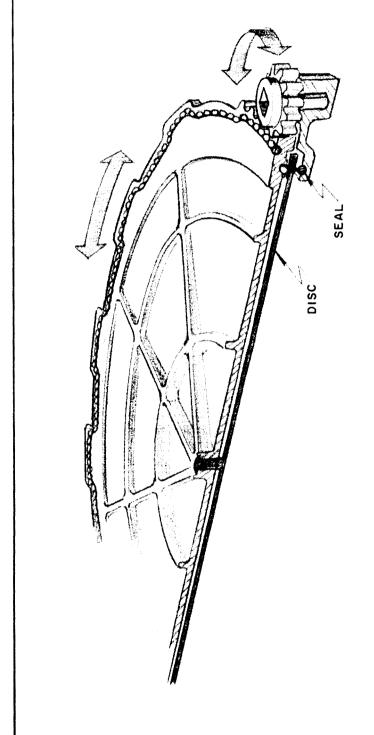










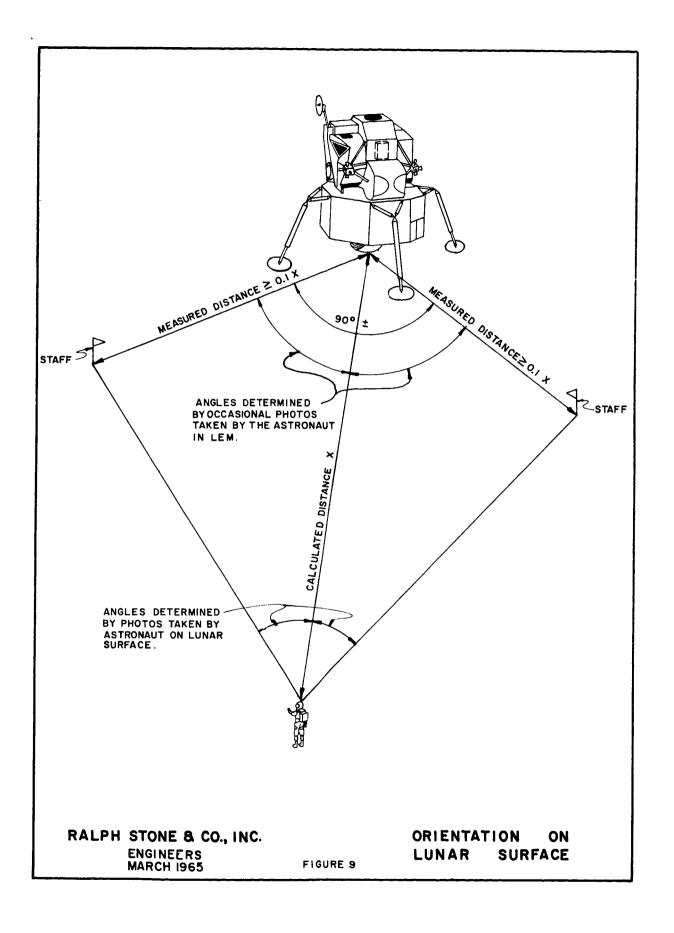


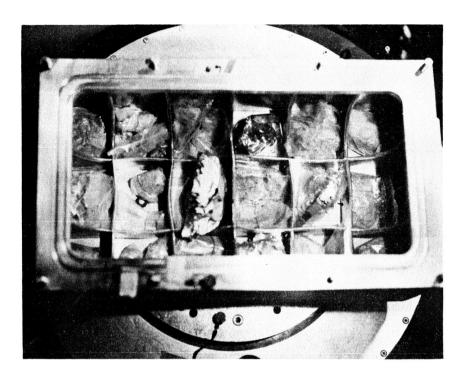
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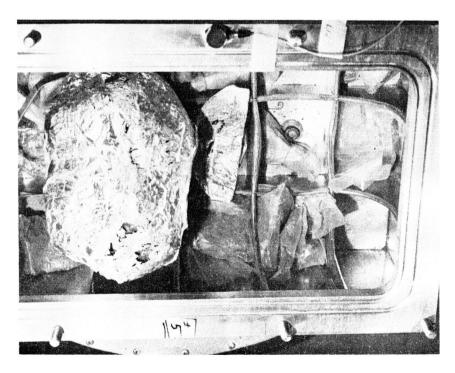
LUNAR SAMPLE CONTAINER

FIGURE 8





SAMPLE PLACEMENT DURING TEST NO. 2 FIGURE II



SAMPLE BAG DAMAGE DURING TEST NO. 3
FIGURE 12



SAND SCOOPING DEMONSTRATION FIGURE 13



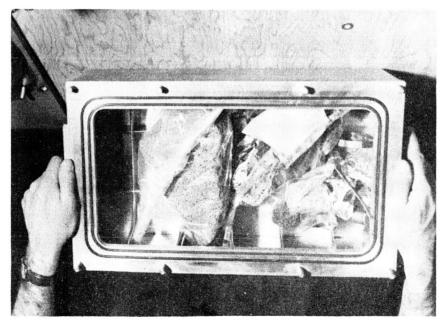
FLEXIBLE SAMPLE BAG DEMONSTRATION FIGURE 14



METALLIC TEST TUBE CORING DEMONSTRATION FIGURE 15



SAMPLE STOWAGE IN BOX CORNERS FIGURE 16



STOWAGE AS DONE BY SUIT TECHNICIAN FIGURE 17

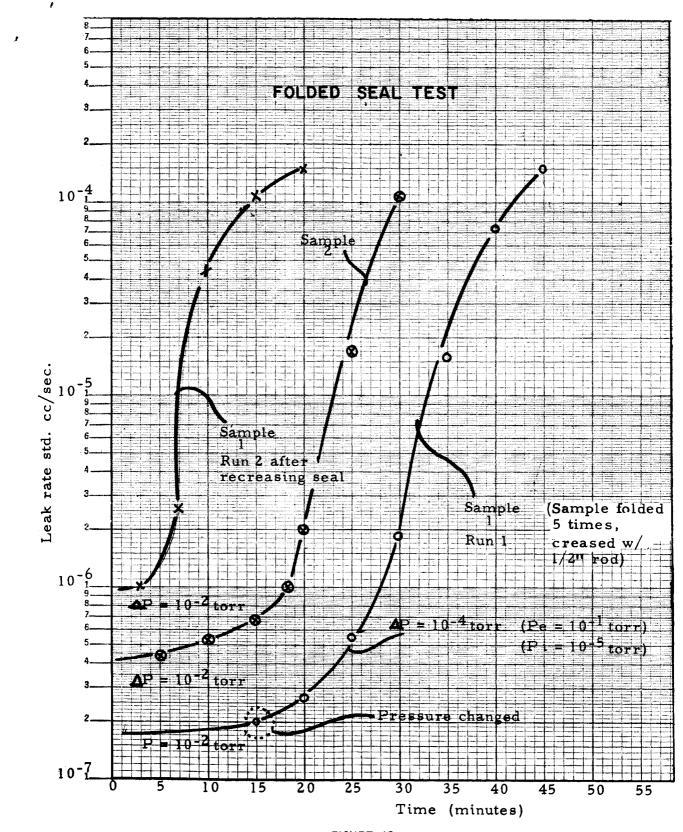


FIGURE 18

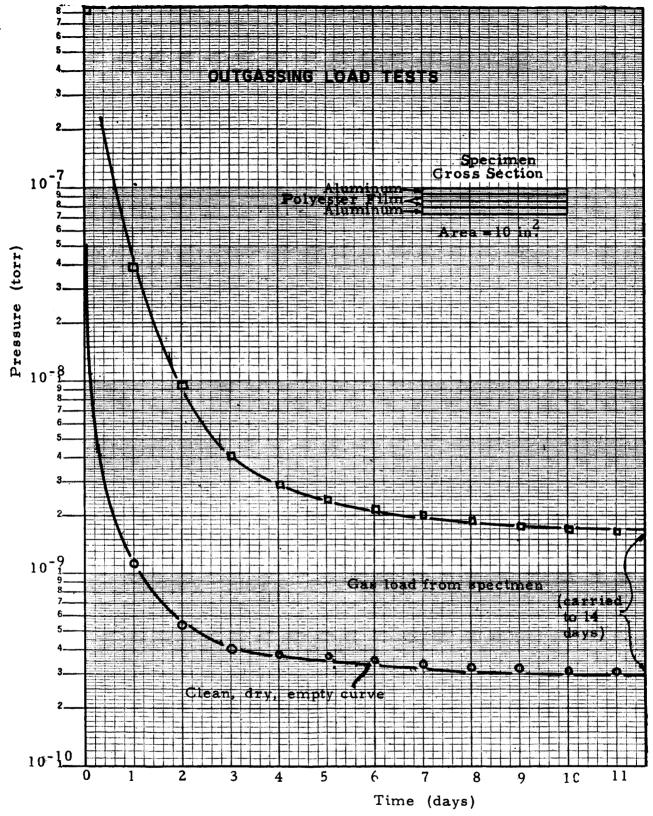


FIGURE 19