

PLANETARY MATERIALS LABORATORY
SAMPLE PROCESSING PROCEDURE

DATE: OCTOBER 24, 1991

SPP 73

PEELING AND IMPREGNATION OF CORES

1 INTRODUCTION

This procedure directs how to take a peel from a core which has been dissected and how to impregnate the core with epoxy after the peel has been taken. These two processes are combined into one procedure because they are always done together and because final weight calculations require data from both processes.

1.1 This procedure details how to remove a layer about 1 mm thick from the lengthwise surface of a dissected core. A plastic strip is coated with cement, and then laid face down on the dissection surface. After a few moments to allow the cement to penetrate the core materials, the plastic strip is removed and turned over to bring the adhering layer, the "peel", on top. When the cement sets, the grains are held in position, but unlike the impregnated material, can be removed with relative ease for individual study. The removal of this layer exposes a soil surface undisturbed by dissection. Fine details of stratigraphy are visible on both the core and the peel surfaces. Detailed photographic documentation of these surfaces follows. The peel constitutes a permanent record of core stratigraphy.

1.2 This procedure also details how to mix a low viscosity epoxy and, under vacuum conditions, slowly pour the epoxy onto a lengthwise section of lunar core soil. In this way the epoxy will penetrate into pores throughout the soil with minimum disturbance of the soil. Besides preserving fine stratigraphic detail, this procedure is the first step in a series which results in continuous thin sections along the length of the core.

1.3 A log book is used to record sims and specify recipes.

2 RESPONSIBILITY

2.1 The supervisor of the Custom Thin Section Laboratory (CTSL) is responsible for coordinating the schedule, and the CTSL personnel are responsible for preparation of the work area, and assembly of the supplies and equipment.

2.2 A curatorial representative must be present and approve all handling steps that involve picking up and transferring the unimpregnated core. Weight discrepancies and weight checks after processing must be reviewed by the curator. The TM must be present during the peeling and impregnation of the core.

2.3 The core processor who dissected the core observes the peel and pouring of epoxy. The core processor also screens the photos.

- 2.4 The CTSL personnel are responsible for performing all action described in this procedure.
- 2.5 A meeting will be held with the curatorial representative the day before transfer to CTSL to establish that everything is ready and simulations were successful.

3 SAFETY

Handle all volatile materials within the hood that is exhausting continuously to the outside, and steps involving these materials must be accomplished within the hood. Any sources of open spark such as flashbulbs, arc lights, or unshielded motors must not be in the room during operation of this procedure. Volatile materials used in this procedure are: toluene, Krylon Spray Coating, butyl glycidyl ether. Remove from the safety containers only the small amount of chemical needed. A fire extinguisher and trained operator will be present during handling of these volatile materials.

Must use OSHA respirator when using toluene or ether.

MSDS for all chemicals should be available.

CAUTIONS precede these steps:

Refer to memo NS3/86-M258 (Appendix A)

- 6.1.8
- 6.1.9
- 6.4
- 6.8

4 DEFINITIONS

ether - butyl glycidyl ether
MSDS - material safety data sheet

5 REQUIRED EQUIPMENT

5.1 Chemicals

<u>Chemicals</u>	<u>Quantity</u>
n-butyl methacrylate (Du Pont Elvacite 2004 acrylic resin)	30-40 g
Krylon Crystal Clear Acrylic Spray Coating #1301	
Araldite 506 resin	80 g
Versamid 140 hardener	25 g
Butyl glycidyl ether	50-75 g

The butyl glycidyl ether must be within shelf life and specially pretested. Exact recipe is established during simulations preceding the core processing.

5.2 Tools for peel process

These tools should be visibly clean and freon rinsed.

<u>Tools</u>	<u>Quantity</u>
Plexiglass mounting strips (Make strips the length of the core minus 3 mm and ranging from slightly wider than the core soil holder.)	variable
Epoxy plugs used to hold soil in place for impregnation.	
1 x 1 cm squares of plexiglass sheet to use for handles on back of peel (optional)	2
Plexiglass box designed for storage of peels	1 for each peel
Template to control methacrylate thickness	1
Metal tape to secure epoxy plugs	

5.3 Tools for epoxy impregnation

<u>Tools</u>	<u>Quantity</u>
250 ml plastic beaker	1
50 ml plastic beaker	3
Epoxy pouring trough	1
Vacuum chamber with baseplate and fittings	
Epoxy pouring guide (2 halves) with rubber gaskets, screws, and fittings	
500 g Mettler balance or equivalent	
1200 g Mettler balance or equivalent	
Vacuum chamber valve and instrument panel	
Incubator oven	
Curved scraper (for removing core from receptacle) SDZ 36112755-001	
End plugs	
Metal tape	

5.4 Photographic Equipment

2 Cameras and lenses
 Film holder
 Ektachrome 6117 color cut film, polaroid film

Lights capable of providing even illumination over
the entire core (strobe lights)
Color filters for lights (optional)

5.5 Miscellaneous Equipment

<u>Tools</u>	<u>Quantity</u>
Aluminum core cover plate (SDZ 36112437-003)	1
Aluminum sample number tags:	
Core number, 6000 (for impregnated portion)	1
Core number, 6001 (for peel)	1
Core number, 6002 (for second peel, if needed)	1
"TOP"	3
"BOTTOM"	3
Scalpel and blades	1
Stainless steel applicator, spoonula or equivalent	1
Dental pick	1
Aluminum foil sheets, 18 x 24 or larger	1
OSHA/MSHA-approved respirator with organic acid mist cartridges	3
Chemical resistant gloves	2 pr
Splash goggles	2
Lab apron	2

5.6 Log book

6 PROCEDURE

6.1 Preparation before core is moved to work area

6.1.1 Scheduling. A brief outline of activities is given below for the purpose of making a schedule. Fixed time requirements are underlined. The schedule should be planned so that nothing needs tending after regular work hours or on weekends.

- a) Do a full simulation of the peeling procedure a few minutes before the lunar peel is taken using the same batch of methacrylate. This allows the processor to practice handling techniques and check viscosity of the methacrylate.
- b) Full simulation of core impregnation must be done to establish epoxy mixture and times. Record in log book. This should use the exact batch of resin, hardener, and ether.

- c) Arrange for work area to be mopped and work surfaces to be freon rinsed.

6.1.2 Preparation of data packs

- a) A large number of weight measurements which cannot be repeated are made at very specific steps in this procedure. To insure that no measurement is overlooked, spaces are provided for the recording of data at appropriate steps.
- b) All operations are covered by 3 separate procedures: Peeling and primary impregnation are covered by SPP 73, secondary encapsulation, splitting and segmenting of lunar cores and drive tubes are covered by SPP 63, while preparation of TS are covered by SPP 115. All information including photo documentation are kept in one large data pack which includes F-6 used to record weight-change generated by each procedure.

6.1.3 Sample numbering and tags

- a) Obtain the tags listed in section 5.5.
- b) When the peel is taken, number the unimpregnated bulk of the soil ,6000, and number the first peel ,6001. Number additional samples in sequence of creation.

6.1.4 Assemble and set up tools in work area.

6.1.5 Install the vacuum chamber according to TSP 29 and prepare it as follows:

- a) Cover the floor of the vacuum chamber with aluminum foil to catch possible spillage.
- b) Place the baseplate, which supports the dissection receptacle, into the vacuum chamber.
- c) Check that vacuum valves are working properly.

6.1.6 Set up the photographic equipment using the following guidelines:

Since the core surface is unprotected, photographic techniques should minimize the time and amount of handling done over the core. One way of accomplishing this is to have two cameras prefocused: one on a monopod for overall view and one on a sliding rail for 1:1 magnification. Set up the camera and mark where to place the core so that the processor does not have to stand on a ladder over the core for focusing or view

finding. Set up light sources needed. Take practice shots to verify set up is correct.

6.1.7 Preparation of plexiglass strip and epoxy plugs

- a) Select a plexiglass strip of appropriate width with these considerations in mind: Because some core surfaces are uneven, it might be necessary to take more than one peel. Make sure you have two strips approximately 3 mm wider than the trough and two strips the same size for the second peel, if necessary. Attach small epoxy handles, with super glue, to back of plexiglass strip to aid in picking up the peel. The template for passing strip through to insure even thickness of methacrylate has been modified for 1/2" x 1/2" x 3/16" thick handles placed directly in the center on both ends of plexiglass strips.
- b) Cut the length of each strip to give 1-2 mm clearance between strip and epoxy plugs.
- c) Inscribe "TOP" at one end of each strip.
- d) Record weight and length of each plexiglass strip:

Weight _____ gram
Length _____ cm
- e) Epoxy plugs are fabricated by filling trough of designated baseplate with thin section recipe epoxy (described in SPP 63). Place in oven at 125° F, remove from trough next day. Depending upon where the lunar soils starts and ends these plugs can range from 1-3 inches. Each plug should have a 1/8" plexiglass strip superglued on the sample side to serve as a dam to insure a 1/8" layer of epoxy over surface of the core.

6.1.8 Mixing of methacrylate solution

CAUTION

N-butyl methacrylate, toluene, and "Krylon" are FLAMMABLE, TOXIC, and DESENSITIZING to skin. Methacrylate is an EXTREMELY HAZARDOUS ADHESIVE. Do not get these materials in eyes, on skin or clothing. Use protective eyewear. Do not breathe vapors. Wear protective gloves during application of solvents or adhesives. Use only with adequate ventilation. Keep away from open flame or spark producing devices.

- a) Make about 30-40 ml of n-butyl methacrylate solution in a squeeze bottle. Prepare the methacrylate by mixing equal weights of Du Pont Elvacite 2004 acrylic resin and toluene. (This can be quickly approximated by filling a beaker to 30-40 [B with acrylic resin and adding just enough toluene to cover the resin.) The acrylic resin dissolves slowly; therefore, wait about 10 minutes to judge viscosity. The solution should be quite viscous, but still come out of the squeeze bottle. The solution should not run off the plastic strip when poured.
- b) Adjust viscosity by adding either acrylic resin or toluene.

6.1.9 Simulation of taking a peel. This simulation must be done no more than 1 day in advance of the actual work on a lunar core and is best done immediately before the real peel is taken using the same batch of methacrylate.

Observe CAUTION note that precedes step 6.1.8.

- a) Prepare a mockup of the core using simulated lunar soil in a dissection receptacle designated for this purpose. Do not use receptacles which will later hold lunar sample.
- b) Obtain a plexiglass strip, coat it with methacrylate, and take a peel according to sections 6.4.2 through 6.4.7 given in this procedure for the lunar peel.
- c) If necessary adjust the viscosity of methacrylate as described in 6.1.8, and repeat simulation. Perform simulation until confidence in handling techniques is attained.

6.1.10 Make sure core is ready for transfer and is covered with a screw-on aluminum cover plate and bagged in one teflon bag only (cut off extra bags in storage cabinet).

6.2 Transfer core from storage or dissection cabinet to peel taking location.

Conditions of transfer: Make the transfer with every effort to avoid jarring or imparting vibrations to the core (vibrations disrupt stratification). The TM must be present before the core can be picked up for this transfer. He will document his presence on the F-4. Station persons at each door through which the route passes to open doors at appropriate times and make sure no other traffic crosses the transfer route. Designate a person to head and a person to follow the person handling the core. These persons will be responsible for clearing the halls of traffic and removing obstacles which could cause the core handler to stumble.

6.3 Pre-peel documentation

6.3.1 Weights of hardware

- a) Unbag the core and obtain a gross weight. List all items in container such as screws, guards, ram, follower, etc. Amend list below to accurately represent situation for each core. The weight of each item is to be recorded by the time the procedure is completed; therefore, each time a piece of hardware is removed from the core holder, save the hardware for weighing.

Gross weight of core and container ___g.

<u>ITEMS IN CONTAINER</u>	<u>WT.</u>
base plate	___g
cover	___g
4 cover screws	___g
ram	___g
follower	___g
2 guards	___g
2 guard screws	___g
2 clamps	___g
8 clamp screws	___g

Other:

- b) Remove the cover and save the cover and screws for weighing. Weigh the core and holder (without cover and cover screws) ___g.

- c) Prepare a diagram of the core in the holder, indicate the position of the soil in the holder and all items of hardware included in gross weight from 6.3.1b above.

Sketch:

6.3.2 Photography

Take one overall Polaroid photograph and one cut film photograph to document the condition of the core after transfer. Compare the core to photos taken before transfer. If a change has occurred or if the core has been in storage several months since dissection, take 1:1 magnification photos along the length of the core. Note changes in appearance of the core in the data pack and on the photograph.

6.4 Taking of peel

Observe CAUTION note that precedes step 6.1.8.

6.4.1 Clean dust from area of receptacle where tape will be attached. Replace ram and follower with epoxy plugs by removing end plates one at a time and sliding epoxy plug gently up against core and taping securely with metal tape. When replacing end plates carefully insert 1/8" teflon gaskets specially fabricated for extra protection against leakage once the core holder is filled with epoxy.

6.4.2 Once the simulation of taking the peel is successful according to step 6.1.9, spread a thin layer of same batch of methacrylate over designated, pre-weighed plexiglass strip.

6.4.3 Pass coated strip through template to assure uniform thickness of methacrylate over plexiglass strip.

6.4.4 Allow surface to dry for 30 seconds. Remove protective gloves.

6.4.5 Pressing strip against soil, place end of plexiglass strip marked "TOP" at the top end of the core. Keep strip oriented for remainder of procedure. Impress prepared surface of strip against core, using minimal hand pressure where necessary to insure contact. Verify by observing the soil-wetting through the strip, that contact is complete and that air bubbles are expelled as much as possible.

6.4.6 Lift peel straight up off the core surface and move it from directly over the core quickly and smoothly.

6.4.7 At this point, the TM will state by CO any further action needed to deal with any anomalies that have occurred. At this time, a second peel may be requested by the curator or core processor. There will be no more than two peels made (one only is preferred).

6.5 Post-peel documentation

6.5.1 Weight

Gross weight of dried peel on plastic strip

(a)___g

Weight of plastic strip (from 6.1.7d)

(b)___g

Calculate the weight of soil plus methacrylate by subtracting (b) from (a)

(c)___g

The weight of methacrylate is usually .18g/cm length of strip. Estimate wt. of methacrylate

(d)___g

Estimate the wt. of lunar soil in the peel by subtracting (d) from (c)

EST. wt. of lunar soil (e)___g

Weigh remaining core and holder.

(f)___g

Verify that all pieces of hardware included in this gross weight are shown in 6.3.1c.

Calculate amount of soil removed in peel by weight difference in core holder before and after peeling.

Wt. of lunar soil in peel (g)___g

Compare with weight estimated in (e).

6.5.2 Photography

- a) Take one overall view (post-peel) of the core by itself.
- b) Place the peel next to the soil in the core holder in the same orientation. Place sample number tags and "TOP" and "BOTTOM" tags in correct position. Allow scale to be in view.
- c) Photograph the core and peel together. This photographic documentation is very important for it records the least disturbed surface. Allow sufficient time to photograph the core under various lighting conditions. Comparable photos taken under different lighting can be made by setting up all light sources simultaneously (if possible before core is moved into position) and then taking photos in sequence by each technique before changing the view. Take an overall view and a set of 1:1 photos with at least 50% overlap between views (for stereo photographs). Immediately send film for processing. Verify good images before processing.

6.6 Storage of peel

- 6.6.1 Place core holder away from the peel in a safe area of the work surface. Spray one light coat of Krylon Clear Acrylic on the surface of the peel. However, the coating should be thick enough to prevent the peel from shedding dust.
 - 6.6.2 Affix peel to storage box with methacrylate. Also glue number tag and "TOP" and "BOTTOM" tags in place with methacrylate. Take overall view Polaroid photograph.
 - 6.6.3 Place a cover on the peel container.
 - 6.6.4 At this point, SPP 72 (Spectral Reflectance) may be performed if directed by CO.
- 6.7 Placement of core in vacuum chamber
- 6.7.1 Attach epoxy pouring trough to vacuum chamber control rod. Lock the trough in the level position by tightening set screw on rod. Tilt trough to vertical to allow maximum clearance.
 - 6.7.2 Carefully place the core in the vacuum chamber so that receptacle fits onto baseplate.
- 6.8 Mixing of epoxy

CAUTION

Araldite 506 resin, Versamid 140 hardener and butyl glycidyl ether are TOXIC and DESENSITIZING to skin. Do not get these materials in eyes, on skin or clothing. Use protective gloves at all appropriate steps. Use only with adequate ventilation. Keep away from open flame or spark producing devices. The prescribed respirators must be worn during exposure to vapors, during mixing and use. MSDS should be available for reference.

Preparation of epoxy must be done in early morning on the day when the pumpdown begins. On the flow bench with exhaust system in operation, prepare the following materials:

- 6.8.1 Amount of resin, ether, and hardener is determined by weight. Exact recipe is established during simulations and pot tests.
- 6.8.2 The amount and batch number of the ether is critical. Consult logbook maintained for this purpose and determine the batch number of the ether last used successfully and the amount of ether used. Each newly-used batch of ether must be tested; therefore, if the current (tested) batch is not sufficient to impregnate two cores (100-150 ml), mix a small amount of epoxy using the new, untested batch of ether and run this epoxy test through the impregnation procedure alongside the core. Enter in the

logbook the date, time, core sample number and epoxy formula used for impregnation. Specify the exact amount of each ingredient and specify the batch number of butyl glycidyl ether used. If testing a new batch of ether, write the formula used for mixing that epoxy also.

- 6.8.3 Mix 80 g Araldite 506 resin and 25 g Versamid 140 hardener in a 250 ml plastic beaker. Stir briefly until sample is visually mixed.
- 6.8.4 Add the amount of butyl glycidyl ether prescribed in the logbook and stir thoroughly.
- 6.8.5 Fill the trough with epoxy-ether mixture.
- 6.8.6 Verify that epoxy will not accidentally spill on core.
- 6.8.7 Emplace and close chamber cover. Verify that all valves work and are closed.

6.9 Pumpdown Sequence

Pumping should take a maximum of 2 hours and 45 minutes including pouring time. This sequence should begin no later than 9:00 a.m. to insure enough time for complete pump-down, removal of pouring guides and placement in incubator. Chamber pressure and mm should be monitored and recorded carefully for the first 20-30 minutes until vacuum reaches 1 mm, this is when most bubbling will occur. Be ready to vent chamber if violent bubbling threatens core. Once chamber reaches 1 mm, pump for 1 hr. and 30 minutes, logging status every 20 - 30 minutes, until pour time.

- 6.9.1 Turn on pump and solenoid valve with all valves to chamber closed. When vacuum pump reaches "0", open coarse adjust and fine adjust all the way. Open cut-off valve all the way, which initiates pump-down sequence.

Cut-off valve opened

Time _____

Pressure _____

Date _____

- 6.9.2 Continue pumping for 20-30 minutes or until chamber pressure reaches 1 mm Hg. Note appearance of bubbles also in log book.

20-30 min. pump-down

Time _____

Pressure _____

- 6.9.3 Continue pumping for 1 hr. 30 min. recording time and pressure every 20 - 30 minutes.

1 hr. 30 min. pump-down

Time	Time
Pressure	Pressure
Time	Time
Pressure	Pressure
Time	
Pressure	

6.10 Pouring of epoxy

6.10.1 After total of 1 hr. and 30 minutes pump-down, gently rock pouring trough and verify epoxy is sufficiently fluid. If it is not, abort run. Slowly turn the rod and allow the epoxy mixture to flow onto the pouring guide and then slowly and evenly cover the core. Once soil is completely covered, make sure level of epoxy is up to the top of plexiglass dam glued to surface of plug, which is approximately 1/8" thick.

Pouring of epoxy

Time _____
 Pressure _____
 Date _____

6.10.2 Allow the core to remain undisturbed for at least 5 minutes so that the epoxy can penetrate into the soil. Epoxy must have completely covered dirt before venting.

6.10.3 Release the pressure on the vacuum chamber evenly over a one to three minute period. In this way increasing atmospheric pressure helps push epoxy down into the pores.

Release of vacuum

Time _____
 Date _____

6.11 Incubation of impregnated core

6.11.1 Remove cover from chamber and remove pouring guide. Remove core from chamber and place in incubator. Set temperature at 35° C. Maintain this temperature at least until 8 or 9 a.m. the following day (18 hr. cure).

6.11.2 Test gelatin with pick. When jelled and not sticky, remove core from receptacle while in gel stage using a special curver scraper and scalpel, and place the core in a foil holder. If necessary impregnate reverse side

of core. Use Araldite and Versamid without ether and do the work on a clean bench. Place tags indicating "TOP" and "BOTTOM" in proper position.

Core removed from
receptacle:

Time _____

Date _____

Describe reverse side impregnation if required.

- 6.11.3 Return the core to the oven (35°) until it has hardened. When the core has cured sufficiently for secondary encapsulation, at least two weeks, remove it from the oven. Photograph the core with tags in place using Polaroid film. This is to document orientation.
- 6.11.4 Record in the logbook the length of time it took to reach the gel stage. Also record the time required for completion of curing.
- 6.11.5 Refer to SPP 63 for secondary encapsulation and SPP 115 for thin section preparation.
- 6.12 Post-processing documentation
- 6.12.1 After removing all epoxy from hardware, reweigh all items of hardware and record in section 6.3.1a.
- 6.12.2 Scrapings and shavings of epoxy that contain lunar residue may be placed in lunar trash. Estimate the weight of this material.
- (a) Weight _____g
- 6.12.3 Calculate the weight of soil in impregnated portion by subtracting the weight of the hardware (see section 6.3.1a for weights and 6.3.1c for configuration of hardware) from the weight of soil in holder taken after peel from section 6.5.1, line (f).
- Wt. of soil & holder
from 6.5.1 (f) _____g
- (b) Wt. of Hardware _____g
- (a) Wt. of soil in
scrapings _____g
- (d) Calc. wt. of soil
in impg. portion _____g
- 6.12.4 Complete F-6 forms for peeling and impregnation using data recorded in this procedure. Transfer original observations from the procedure to the F-6 under the heading "cal/inv" and cite the step in the procedure from

which the data came. Additional explanation of what the weight quantity represents may be needed. Examples are: "gross weight pre-peel", "gross weight post-peel". Place a xerox copy of this procedure in the impregnation data pack. The original should already be in the peel data pack. Have paperwork reviewed.

6.13 Transfer of completed samples

Slip peel container into plastic bag, and transfer to RSV.

6.14 Clean up

Return equipment and chemicals used in this procedure to proper storage areas. The core receptacle should be returned to tool control for cleaning. Equipment used for terrestrial simulation is to be kept separate from lunar.

Laboratory Manager

Planetary Materials Curator

Contamination Control Officer

3/11/80 version signed by Dudley J. Fitts

NASA Safety

This procedure replaces the 3/11/80 version of SPP 73.

The procedure takes effect on the date of the last signature.

The term of the procedure is indefinite.