

**CARBON COATING: OPERATION OF THE LADD VACUUM EVAPORATOR
FOR CARBON COATING SAMPLES**

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1.0 PURPOSE

This procedure provides instructions to carbon coat geologic samples using the Ladd Vacuum Evaporator.

2.0 SCOPE

This procedure applies to carbon coating of any geologic sample related to work on the Los Alamos Yucca Mountain Project.

3.0 APPLICABLE DOCUMENTS

Documents referenced in this procedure are: Ladd Vacuum Evaporator Instruction Manual for Ladd Vacuum Evaporator, Cat. no. 3000, 1988; Ladd Research Industries, Inc., P.O. Box 1005, Burling, VT 05402; TWS-ESS-DP-101, Procedure for Identification and Control for Mineralogy-Petrology; TWS-QAS-QP-02.1, Los Alamos Yucca Mountain Project Personnel Selection, Training, and Certification

4.0 PERSONNEL RESPONSIBILITIES

Any individual certified to work on the Los Alamos Yucca Mountain Project and trained in this procedure may perform it.

5.0 PRINCIPLE

All geologic samples to be analyzed using either the Electron Microprobe or the Scanning Electron Microscope must be coated with a conductive material prior to analysis.

6.0 PROCEDURE

6.1 Initial start-up of Ladd Vacuum Evaporator (Cat. No 3000).

- 6.1.1 Turn MAIN SWITCH to ON (red light will come on).**
- 6.1.2 Turn MECHANICAL PUMP switch to ON (red light on).**
- 6.1.3 Turn MANIFOLD VALVE (black knob on left side to BACKING).**
- 6.1.4 Read Mechanical Pump Thermocouple (TC) Gauge, pressure should drop to 40 millitorr or less.**
- 6.1.5 Turn DIFFUSION PUMP switch to ON (red light on).**
- 6.1.6 Wait 15 minutes for diffusion pump oil to heat. Continue only after the required waiting period.**
- 6.1.7 Turn MANIFOLD VALVE (black knob) to ROUGHING.**
- 6.1.8 Read Bell Jar TC gauge. When Bell Jar TC gauge reads 40 millitorr or less, the red light above the Main Valve knob will go out Discharge Gauge will automatically come on at this pressure.**
- 6.1.9 Turn MANIFOLD VALVE (black knob) to BACKING.**

6.2 Sample insertion or change

- 6.2.1 **MAIN VALVE (Red knob) must be in CLOSED position and MANIFOLD VALVE must be on BACKING.**
- 6.2.2 **Open AIR INLET toggle to Bell Jar.**
- 6.2.3 **Raise Bell Jar to top of Jar guide and swing to right, locking it in the raised position.**
- 6.2.4 **Place sample(s) on turn table.**
- 6.2.5 **Turn Rotary Drive control on and adjust rotation speed to about 1/2 revolution per second. Check that all samples will clear carbon rod mount posts.**
- 6.2.6 **Check to make sure carbon rod is set-up correctly; if not set up or if this is the second time through this sequence, follow carbon rod set-up procedure as follows, otherwise skip to pumpdown section.**
- 6.2.7 **If you are done coating and have set the carbon rod for the next user proceed to step 6.6 for shut down.**

6.3 Carbon rod set-up

- 6.3.1 **Remove the glass shield.**
- 6.3.2 **Swing lock latch into position.**
- 6.3.3 **Loosen thumb screw .**
- 6.3.4 **Swing the carbon rod chuck counter-clockwise towards you.**
- 6.3.5 **Grasp the end of the carbon rod chuck and loosen the knurled end of the holder.**
- 6.3.6 **Remove the old carbon rod. If one end is still unused since machining, replace the rod in the holder with the unused end sticking out. If both ends have been fired, replace the used rod with a new one. Visually check that the machined tip of the new rod is 6/32 in. \pm 1/32 in. in length. Place the used carbon rod in the plastic container marked for such use.**
- 6.3.7 **Slide the carbon rod almost all the way into the carbon rod chuck but do not tighten it.**
- 6.3.8 **Loosen the other knurled chuck. Remove the carbon rod and file both ends flat, perpendicular to the length.**
- 6.3.9 **Replace carbon rod from left side back into the knurled chuck. Leave the carbon rod sticking out of the knurled chuck about 1/2 in. to 3/4 in. and tighten the knurled chuck.**
- 6.3.10 **Swing the carbon rod chuck on the right side back to its original position so that carbon rods line up with each other.**
- 6.3.11 **Tighten thumb screw.**
- 6.3.12 **Visually check that the lock latch is in position to hold the carbon rod with the tip back.**
- 6.3.13 **Pull 'tipped' carbon rod forward out of the knurled chuck until it just touches the other carbon rod and then tighten knurled chuck. Swing the release lock out and slowly let the spring push the carbon rod into contact with the other carbon rod.**
- 6.3.14 **Put a clean glass shield in place. Replace bell jar, being careful to seat jar correctly inside stainless bell jar guide.**

6.4 Pump-down

- 6.4.1 **Close AIR INLET toggle valve.**
- 6.4.2 **Turn MANIFOLD VALVE (black knob) to ROUGHING.**
- 6.4.3 **Wait until Bell Jar TC gauge reads less than 40 millitorr and red light above MAIN VALVE goes out.**

- 6.4.4 Turn MANIFOLD VALVE (black knob) to BACKING.
- 6.4.5 Turn MAIN VALVE (red knob) to OPEN.
- 6.4.6 When DISCHARGE GAUGE reads less than 5×10^{-5} proceed with evaporation.

6.5 Evaporation

- 6.5.1 Verify that the ELECTRODE SELECTOR SWITCH is set on circuit E4.
- 6.5.2 Turn ELECTRODE SWITCH to ON (red light).
- 6.5.3 Turn Rotary Drive control on and adjust rotation speed to about 1/2 rev. per second with control unit beside Bell Jar.
- 6.5.4 Check to make sure pieces of dark viewing glass (for eye protection) are on top of Bell Jar cage. If not present, find before proceeding and place them on top of Bell Jar cage in a direct line between your eyes and the carbon rods.
- 6.5.5 Turn ELECTRODE CURRENT CONTROL Knob clockwise from 0 to between 30 and 40 milliamps or until the carbon rods arc. Watch rods through dark glass until carbon rod tip is completely burnt away.
- 6.5.6 Return ELECTRODE CURRENT CONTROL Knob to 0.
- 6.5.7 Turn ELECTRODE SWITCH to OFF.
- 6.5.8 Return to part 2 (Sample change) to retrieve sample.

6.6 Final shut-down

- 6.6.1 Make sure that MAIN VALVE is closed. Remove sample, replace Bell Jar.
- 6.6.2 Turn DIFFUSION PUMP SWITCH to OFF.
- 6.6.3 Close AIR INLET toggle valve.
- 6.6.4 Turn MANIFOLD VALVE (black knob) to ROUGHING.
- 6.6.5 Wait until Bell Jar TC gauge reads 40 to 30 millitorr.
- 6.6.6 Turn MANIFOLD VALVE to BACKING.
- 6.6.7 Let DIFFUSION PUMP cool for 20 minutes.
- 6.6.8 Turn MANIFOLD VALVE (black knob) TO CLOSED.
- 6.6.9 Turn MECHANICAL PUMP SWITCH to OFF.
- 6.6.10 Turn MAIN SWITCH to OFF.

7.0 QUALITY ASSURANCE REQUIREMENTS

7.1 Acceptance Criteria

Samples should be satisfactorily coated if the carbon rod tip is completely burned away following evaporation.

7.2 Sample Storage

All samples will be stored in a locked cabinet in the sample preparation lab from time of receipt until retrieved by responsible Investigator, excluding only the time for coating. Samples must be stored in a container marked with the sample identification and the name of the responsible investigator.

7.3 Documentation

Samples received for carbon coating will be logged into a carbon coating logbook. The logbook will contain the sample identification, the name of the responsible investigator, the date the sample was received for coating, the date coated, the name of the person performing the coating, and the date retrieved by the responsible investigator.

7.4 Sample Traceability

Samples will be tracked in accordance with the Los Alamos YMP Control of samples procedure (QP-08.1).

7.5 Personnel Qualifications and Training

Training to this procedure consists of reading this DP. Evidence of training shall be documented in accordance with QP-02.1.

7.6 Calibration

This is a sample preparation procedure and produces no data. Calibration is not applicable.