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PREPARATION OF FUSED BEADS
FOR ELECTRON MICROPROBE ANALYSIS OF ROCK POWDERS

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David Carman

Prepared By

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Date

Arthur J. Key

Technical Approval

18 Nov. 1982

Date

J. J. O'Leary

Technical Project Officer

11/22/82

Date

Peter L. Bussolini

Quality Assurance Manager

11/24/82

Date

**PREPARATION OF FUSED BEADS
FOR ELECTRON MICROPROBE ANALYSIS OF ROCK POWDERS**

1. PURPOSE

The purpose of fluxless fusion is to reduce a rock powder to a homogeneous glass, to quench that glass without developing crystallites (inhomogeneities), and to do all of this so rapidly (within 20-30 seconds) that volatile metals (e.g., Na) are not lost. This type of fusion can be performed on the iridium-strip furnace. The process is simple and rapid, once a sample powder has been prepared (described in procedure TWS-ESS-DP-19, Sample Preparation: Rock Powders). However, it is important to note that satisfactory fluxless fusions generally are possible only with mafic rock types (basalts) and not with more siliceous rock types (rhyolite, granite), where inhomogeneity and volatile-loss problems may be severe.

2. SCOPE

This procedure applies to any fused beads prepared for microprobe analysis for the NNWSI project.

3. PROCEDURE

3.1 Begin by clamping a 1 x 5 x 0.005-cm strip of iridium foil in the furnace electrodes. Allow the center of the foil to bow slightly, in order to form a shallow trough. With a spatula rinsed with deionized water and alcohol, place 10-30 mg of sample in the bowed iridium strip. Bring the rheostat quickly to a low reading of ~150, remain there briefly until the sample begins to glow, and then bring the rheostat up sharply to a reading of ~400. The sample will start to bubble as water vapor is lost; after bubble formation has stopped (about 15 seconds at 400-425), bring the rheostat abruptly to zero while immediately starting the air jet over the iridium strip to quench the glass. The entire melting process, from beginning zero position on the rheostat to quenching at the final zero position,

should take no more than 20-30 seconds in order to prevent volatile-metal loss. Dark-tinted welders' goggles must be worn for the fusion operation, because the sample will have a white-hot incandescence while melting.

- 3.2 Continue cooling the fused sample under the air jet for at least one minute to insure thorough quenching and to cool the iridium strip to a point where it can be handled. Unclamp the strip, with the fused sample attached, and roll the strip gently back and forth over a cylindrical Pyrex tube. The rolling process will flex the iridium strip under the sample, causing the glass to pop off. When the glass has been removed, place it in a plastic tube with tungsten-carbide end caps, insert a tungsten-carbide ball, and vibrate this assembly for 10 minutes in the Spex mill. The re-powdered glass sample is then re-fused by exactly the same process outlined above. (Note: the re-powdering and re-fusion are performed to insure sample homogeneity). The final bead is then examined under the petrographic microscope to insure that no crystallites (commonly olivine or magnetite) have formed. If crystallites are seen, the sample should be re-powdered and fused again. If no crystallites have formed, the sample is ready to be polished and coated for electron microprobe analysis.
- 3.3 The plastic tube of the ball mill is discarded, but the tungsten-carbide end caps and the tungsten-carbide ball must be cleaned for reuse. These parts are cleaned by using the sonic bath, first immersing them in deionized water and then in alcohol. The iridium strip is cleaned for the next sample by immersion for one-half hour or longer in concentrated HF (a fume hood is required for this process). With careful use, an iridium strip should last for 15-20 sample fusions. Used iridium is valuable metal with a cost/weight ratio comparable to platinum or gold. Never throw away a used or torn iridium strip, but save all iridium scraps for recycling.

3.4 Rock powders may be analyzed in greater detail by instrumental neutron activation analysis (INAA) and by atomic absorption (AA). These procedures are carried out through a cooperative arrangement with Group H-8. These methods are detailed in Ref. 1.

4. QUALITY ASSURANCE

4.1 Personnel

Only qualified personnel shall be allowed to perform this work. Evidence of qualification shall be documented in a certification record to be kept in the ESS-Division resident file.

4.2 Calibration

Not applicable.

4.3 Documentation

Samples used in this procedure and related data shall be documented in a bound laboratory notebook or a L.A. Notebook, and controlled in accordance with Document Control Procedure TWS-CMBQA-QA-03. Sample identification shall be traceable throughout this process to the original whole rock sample.

5. REFERENCES

1. E. S. Gladney, D. R. Perrin, J. W. Owens, and D. Knob, "Elemental Concentrations in the United States Geological Survey's Geochemical Exploration Reference Sample - A Review," Anal. Chem. 51, 1557-1569 (1979).

6. ATTACHMENTS

Not applicable.

MICROPROBE OPERATING PROCEDURE

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David Broster for Roland Hagan
Roland Hagan
Preparer

5/30/89
Date

David Vaniman
David Vaniman
Technical Reviewer

May 26, 1989
Date

Henry Paul Nunes
Henry Paul Nunes
Quality Assurance Project Leader

5/30/89
Date

R. J. Herbst
R. J. Herbst
Technical Project Officer

5/30/89
Date

(12)

8912190174

MICROPROBE OPERATING PROCEDURE

1.0 PURPOSE

The purpose of this procedure is to allow an investigator to bring the Electron Microprobe from a standby condition to an analysis condition, perform one or more analyses, and when finished, return the system to a standby condition.

2.0 SCOPE

This procedure describes the start-up, calibration, quantitative analysis and shut-down routines for the Electron Microprobe. Requirements for Los Alamos Yucca Mountain Project (LA YMP) analytical work are listed throughout this procedure.

3.0 APPLICABLE DOCUMENTS

Documents referenced in this procedure are:

Cameca Technical Manual, Option A-21

Tracor Northern Operators Manual, 1988

SANDIA TASK8: A Subroutined Electron Microprobe Automation system, Sandia Report 2037, 1985

CONFIG8 A Configuration File Generator for SANDIA TASK8, Sandia Report 2035, 1985

TWS-ESS-DP-06, Operating Instructions For the Ladd Vacuum Evaporator For Carbon Coating

TWS-ESS-DP-122, Preparation of Electron Microprobe Standard Mounts

TWS-ESS-DP-101, Procedure for Identification and Control for Mineralogy-Petrology

TWS-QAS-QP-02.1, Los Alamos YMP Personnel Certification Procedure

TWS-ESS-DP-125, Certification of Standards for Microanalysis

TWS-QAS-QP-3.1, LANL, Yucca Mountain Project Computer Software Control

4.0 RESPONSIBILITIES

4.1 It is the responsibility of the Operator to ascertain that all Electron Microprobe systems are operational before an Investigator begins a microanalysis session.

4.2 It is the responsibility of the Investigator to determine acceptance or rejection of data generated by the Electron Microprobe analysis routines. Acceptance criteria will be entered in the Investigators controlled notebook.

4.3 Any deviations from this procedure must be documented by the responsible Investigator in his/her LA YMP notebook.

5.0 PRINCIPLE

The basic principle of the Electron Microprobe is the generation of a highly focused beam of electrons which strike and interact with the constituent elements of the target specimen. These interactions produce characteristic x-ray lines which are then detected by a set of wavelength spectrometers as well as an energy dispersive system. The intensity of each x-ray line is proportional to the concentration of the element present. Through comparison of the relative intensities of each element to those from specimens of known concentration, (i.e., Standards),

unknown compositions may be analyzed if present in abundances above a detection limit of approximately 0.1 weight percent. Minimum spatial resolution of elemental variation is limited to about 1 micron due to the actual specimen-beam interaction volume. The electron beam, stage motion, spectrometer motion, Energy Dispersive System, and entire quantitative routine are controlled by a dedicated computer and accompanying software. Reference is made to the Procedure for Standard Certification for details concerning standard selection.

6.0 DEFINITIONS

- 6.1 **Operator.** Operators are those persons that are LA YMP certified and are trained to operate, perform maintenance on, and make adjustments to the microprobe. Operators may also perform YMP microprobe analysis.
- 6.2 **Investigator.** Investigators are those persons that are certified to perform LA YMP Mineralogy/Petrology or related or similar evaluations. Investigators may perform only the operations described in this procedure.
- 6.3 **Primary Standards.** Standards from the National Bureau of Standards (NBS) or from a source where the standard was purchased using LA YMP Quality Assurance Level 1 designation for procurement, or standards for which the composition and homogeneity have been documented in published technical journals and are widely distributed, are defined as Primary Standards.
- 6.4 **Samples.** Most samples will be polished petrographic thin sections. Polished epoxy mounts and thin slabs may also be used.
- 6.5 **Sy Quest.** Sy Quests are the ten megabyte formatted removable disc cartridges used by the Tracor Northern 5500 computer.
- 6.6 **Abbreviations**
- 6.6.1 TN, Tracor Northern
 - 6.6.2 PI, Primary Investigator
 - 6.6.3 YMP, Yucca Mountain Project
 - 6.6.4 LA, Los Alamos National Laboratory
 - 6.6.5 EDS, Energy Dispersive System
 - 6.6.6 WDS, Wavelength Dispersive System
 - 6.6.7 NBS, National Bureau of Standards
 - 6.6.8 VAX, Trademark of Digital Equipment Corporation for Virtual Address Extension
 - 6.6.9 RS1, Integrated database, graphics, statistics, and programming language package and is a registered trademark of BBN Software Products Corporation.

7.0 SYSTEM DESCRIPTION

- 7.1 The Electron Microprobe is a Cameca Model MBX (purchase date 1976). Refer to the Cameca Operating Manuals for description of this system.
- 7.2 The Analyzer (EDS) is a Tracor Northern Series II X-Ray Analyzer (purchase date August 1988). Refer to the Tracor Northern Operator's Manual for this system.

7.3 Software supplied by TN and by Sandia Laboratories is not modified and is defined as Commercial Software (reference TWS-QAS-QP-3.1, section 8.0).

8.0 PREPARATORY VERIFICATION

The Operator will perform a start-up check by following the steps given in section 11.1 through 11.4 in preparation for a LA YMP microprobe analysis session. The operator will then verify and note in the LA YMP microprobe logbook that all microprobe systems are operational.

9.0 ENVIRONMENTAL CONDITIONS

Normal interior building temperatures in the range of 70 to 80 degrees Fahrenheit are acceptable for the operation of the Electron Microprobe. If the interior building temperature rises above 80 degrees Fahrenheit, the Investigator will not perform YMP work. Cooling water in the range of 40 to 60 degrees Fahrenheit for the microprobe vacuum system is supplied by the building utilities system. Building utilities are maintained by Engineering (ENG-5) and a computer generated alarm is set off at ENG-5 if unacceptable conditions are detected.

10.0 DOCUMENTATION

10.1 All current LA YMP operating conditions and a list of primary standards will be kept in a controlled logbook located near the microprobe. The operator will update this logbook as changes occur.

10.2 The LA YMP Investigator must enter in this logbook his or her name, date, and each LA YMP sample number analyzed during each analysis session. Operating conditions, samples analysed, and observations made during analysis will be noted in the Investigator's notebook.

10.3 At the beginning of a LA YMP analysis session the operator will record in the logbook the operational condition of the Electron Microprobe.

10.4 The printed output of the analysis routine is to be retained by the investigator and it shall be the investigator's responsibility to include these data in the appropriate LA YMP reports as he or she sees fit.

10.5 The floppy disks and/or diskettes assigned to the investigator are to be considered as temporary storage for analysis data and will be kept in the microprobe room. It will be the investigator's responsibility to edit, archive, and/or delete these files as needed.

10.6 All LA YMP microanalysis data will be stored permanently on the VAX in a RS1 database.

11.0 PROCEDURE

11.1 Samples will be prepared for Electron Microprobe analysis following instructions in the detailed procedure for carbon coating, TWS-ESS-DP-06.

11.2 Microprobe start-up. The microprobe is in Stand-by condition after shut down at the end of the previous work day.

- 11.2.1 Fill the liquid nitrogen cold trap until it just overflows. If you are the first to fill the cold trap for the day, refill it in approximately 30 minutes. While the microprobe is in use, the cold trap should be filled every four hours.
- 11.2.2 Activate the Vacuum System by switching the toggle on the left front of the vacuum chassis to OPERATE.
- 11.2.3 Turn on the Light Microscope Illuminator by pushing the Illuminator button In. Switch the toggle switch below it to high.
- 11.2.4 Switch the white toggle switch located on the column above the stage indicators to reflected light. Up is reflected light and down is transmitted light.
- 11.2.5 Turn on the TV camera power supply located above the TV monitor. The green light will now be on. Turn on the TV monitor.
- 11.2.6 Turn on the TN 5500 monitor power switch located on far lower right side of the Monitor console, next to the brightness dial.
- 11.2.7 Verify that the vacuum system has achieved suitable vacuum (less than 2×10^{-5} torr) and push the silver reset button just to the right of the operate button. The secondary vacuum light will now come on.
- 11.2.8 Verify that the program TASK is running by looking for the "TASK8": prompt on the TN 5500 monitor. If the prompt is correct proceed to step 11.4. If the prompt is not correct, type X 'TASK' (CR). If the prompt is now correct proceed to step 11.4, if it is still not correct continue in sequence and reboot the computer.
- 11.3 Boot computer
- 11.3.1 Press the restart toggle plate located just below the power switch on the TN 5500 console (just to the right of diskette drives 5 and 6) and step through the following sequence of inputs.
- . (prompt) 28 START?
 - . (type) DL (then carriage return, or CR)
 - . press the PROGRAM soft key
 - . select the LOAD FLEXTRAN option (option # 0) from the menu and (CR).
 - . press the RUN soft key
 - . (type) X 'START' (CR)
 - . (prompt) START 20 June 1988
- 11.3.2 The system will take about two minutes to initialize the RAM DISK and load libraries, system files, and general subroutines. It will then display the message:
- . "Use CONFIG file (CR or new #) 4000 ?"
 - . (type)(CR)
- 11.3.3 The program TASK will then display the current spectrometer and stage positions and ask "OK". The current idle positions will be listed in the logbook and all spectrometers are parked at .50 overnight.
- 11.3.4 Check the Dial Indicators on the stage and spectrometers and determine if the readouts are approximately the same as those on the LED Displays. If the values are the same type (CR) and proceed to step 11.4.1. If the values are not the same type NO (CR) and continue with this procedure.
- 11.3.5 Each time a (CR) is entered, the computer will read out one of the LED displays in the following order: X axis, Y axis, Z axis, spectrometer 1, spectrometer 2, spectrometer 3, and spectrometer 4.
- 11.3.6 If the LED and the dial indicators match then hit a (CR). If they do not match, type in the value as read from the dial indicators and then hit a (CR).

- 11.3.7 After listing spectrometer 4 the computer will again type out all values and ask again if "OK". If the positions are now correct type a (CR) and skip to step 11.4.1. If the positions are not correct type NO and repeat the above step until they are correct.

11.4 Saturating the filament

- 11.4.1 Verify that the SECONDARY VACUUM LED is now on. If it is not, verify that the vacuum reading on the secondary gauge is less than 2×10^{-5} torr and press the silver reset button. The Secondary vacuum LED should then come on.
- 11.4.2 Type GET STA THO (CR). This will move the stage to the thorium standard and allow the Investigator to see the beam when it is turned on.
- 11.4.3 Type DEF BEA -1 (CR). This will release computer control of the filament circuit.
- 11.4.4 Turn the Absorbed Current Range Selector to 10^{-7} ampere.
- 11.4.5 Push in Filament Power Supply Button .
- 11.4.6 Saturate the filament in the following manner:
- . Slowly turn the Filament Temperature Knob clockwise to about 4 to 8. The meter will read a fast rise in signal and then level out. Do not turn past this level out point. Verify that the beam is visible on the thorium standard via the TV monitor. Wait 15 minutes before proceeding to the next step; this will allow the beam to stabilize.
- 11.4.7 Type DEF BEA 15 (CR). This will allow the computer to define the beam to fifteen nano amperes, the most commonly used beam current. However, the Investigator may choose the appropriate beam current and note it in the Microprobe logbook.
- 11.4.8 Type MAN (CR). This will unblank the defined beam and allow viewing.
- 11.4.9 Press the Raster Size Control button labeled "Spot".
- 11.4.10 Use Lens 3 to make the spot as small as possible.
- 11.4.11 Use the Z axis on the joystick control to recheck the focus.
- 11.4.12 Press the large square of the Raster Size Control.
- 11.4.13 Center the beam raster on the TV Monitor crosshairs by moving the two Optical Microscope Adjusters on the column.
- 11.4.14 Type LOAD SCHED 10 (CR)
- 11.4.15 Type RUN START (CR)
- 11.4.16 The stage will move to the focus point located on the brass standard holder just below the thorium standard (see photograph displayed near the TV monitor.) If the position is correct skip to step 11.5. If the position is not correct, move to the focus point using the joystick in the following manner:
- . Switch the joystick speed control to SLOW
 - . Press the White button on the joystick.
 - . Check the position and focus. Use the joystick to correct any mis-position and use the Z axis on the joystick box to correct the focus. Repeat until the focus is correct.
 - . Switch the joystick speed control to HIGH.
 - . Press the RED button on the joystick box
 - . At the prompt, compare the displayed stage position values with the current values listed in the microprobe logbook.
 - . As each value is listed compare it with the listed value; if it is correct type a (CR). If it is not correct type the correct value and then a (CR).

11.5 Calibration

- 11.5.1 Use a schedule or create a schedule appropriate to the material being analyzed; e.g. oxides, carbonates, sulfides, etc. The following is given as an example.
- 11.5.2 Type LOA SCH 10 (CR).
- 11.5.3 Type RUN LAYMP (CR). This schedule will set the machine parameters for LA YMP work and will print out a list of the current machine and software settings. Compare this list to the one in the LA YMP notebook and verify that they are identical. Other settings may be selected but all changes must be recorded in the Investigator's notebook.
- 11.5.4 Type DEF BEA 15(CR). This will define the beam to 15 nA (or use the appropriate beam current) and after a ten second period will print out the beam value. The acceptable range in beam values is ± 1.5 nA.
- 11.5.5 Type GET STA FO (CR). The stage should move to the standard focus spot. If it does not, go to step 11.4.16.
- 11.5.6 Type GET STA THO (CR).
- 11.5.7 Type MAN (CR). This will unblank the beam for viewing. Use the optical microscope centering knobs and center the beam on the reticle of the TV monitor.
- 11.5.8 Select a spot or raster size with the raster size controls.
- 11.5.9 Type CAL (CR). This will calibrate the element table or individual elements. As each standard is moved into position for calibration the computer will request the investigator to press the white joystick button when ready. The investigator should verify that the cross hairs lie on the standard and that it is in focus and then push the white button. When the entire element table has been calibrated the TASK: prompt will return to the monitor.

11.6 Quantitative Analysis Routine

11.6.1 Calibration

- 11.6.1.1 Select a Primary standard from the standard list. The selected standard should be similar chemically (e.g., carbonate, oxide, or silicate) to the mineral to be analyzed.
- 11.6.1.2 Type GET STA XXXX (CR), (where XXXX is the one to four character label of the selected standard). This will move the stage to the selected standard.
- 11.6.1.3 Insert the investigators personal 5-1/4 in. diskette into Drive 5.
- 11.6.1.4 If desired type CAT (CR). When prompted enter the drive number used (5). This will list a catalogue of the disk. Note the last file used and the next unused file on a piece of scratch paper.
- 11.6.1.5 Press the QUANT soft key.
- 11.6.1.6 Press the AUTO soft key.
- 11.6.1.7 Press the BA (for Bence-Albee) soft key. When prompted for the BA Definition File number, enter 10 (or other desired file) and (CR). The file settings will be printed and the computer will ask if "OK?". Check to determine that the settings are identical with the current listings in the LA YMP notebook. If they are not the same or do not meet the analysis requirements type a NO (CR) and follow the prompts to correct the file. Record all changes in the Investigators notebook. If the file is correct type a YES (CR).

- 11.6.1.8 When prompted for the Setup file number, enter 10 or other desired file (CR). Again the file settings will be printed and the computer will ask if "OK?". Again check the printed settings against the current listing in the LA YMP notebook. If they are not the same, or do not meet the analysis requirements, type NO (CR) and follow the prompts to change the file. Record all changes in the Investigator's notebook. If the file is correct type YES (CR).
- 11.6.1.9 When prompted "BEAM SIZE, μM ?" type the beam size selected: spot = 1 μm , small square = 5 μm , large square = 20 μm .
- 11.6.1.10 When prompted "PRINTER LABEL" type the sample number to be analyzed.
- 11.6.1.11 When prompted "ANALYST", the Investigator will type his or her complete last name and a (CR).
- 11.6.1.12 When prompted "RESERVE FILE SPACE FOR 50 PTS (NEW # OR CR)?" type the estimated number of analyses desired from this section. A number less than 50 will save file space; however, a larger number may be entered.
- 11.6.1.13 At the prompt "1 FILES ARE REQUIRED. START AT #?(NEW OR CR)?" type the next unused file number as noted in step 11.6.4 (CR).
- 11.6.1.14 At the prompt "DISK LABEL" check that the correct LA YMP sample is listed; if correct type a (CR), if incorrect type NO (CR) and, when prompted type the correct label.
- 11.6.1.15 The computer will now open the file on the investigator's disk, print the available mineral codes, and measure and print the beam current. When the system is ready to perform a quantitative analysis it will prompt; "<WHITE> for WDS, <RED> for EDS, <CNTRL G> for END"
- 11.6.1.16 Pressing the white button will start a WDS analysis; pressing the red button will start a ten second EDS analysis, and typing a control-G will close the QUANT routine and retrun to the main TASK program.
- 11.6.1.17 The investigator may at the above prompt, type a . (period) with no (CR) which will return the TASK prompt and allow one execution of any TASK command. At the completion of the TASK command there will be no prompt and the system will wait for a joystick button to be pressed or another . (period) to be typed.
- 11.6.1.18 Check that you have focused optically on the standard you wish to analyze and then press the white joystick button.
- 11.6.1.19 If using Schedule 10, the computer will ask "ENTER THE MINERAL CODE:". Type the three letter mineral code of your choice and (CR).
- 11.6.1.20 When the analysis is complete, verify that the results are within acceptable limits of the printed values for the standard analyzed. If the published value is within \pm one sigma (as printed with the analysis) of the analyzed value, the calibration may be considered acceptable; however, it is the investigators responsibility to define his or her acceptance criteria.
- 11.6.1.21 If, in the investigators judgement, the system is not in calibration, then return to step 11.5.7. Individual elements may also be

- calibrated. If the investigator deems the system to be calibrated then analysis results from the appropriate standards must be stored on disk and analysis of the YMP thin sections may proceed.
- 11.6.1.22 Some potential sources of uncertainty will be revealed by the inability to achieve an acceptable calibration as evidenced by running the standards as unknowns and comparing the calculated values to the listed standard values. To change to another sample perform steps 12.1 through 12.15.
 - 11.6.1.23 To store an analysis the investigator must answer the prompt, "SAVE THIS ANALYSIS?" with a YES (CR). The computer will then ask for a TAG for this point. The investigator may enter an identifying tag of his or her own choosing. A maximum of 23 characters may be used. Identify all standards saved on disk as STD_XXXX, where XXXX is the four letter standard label. Caution: use no spaces or comas in either the standard or the sample tags.
 - 11.6.1.24 Standards should be run several times throughout an analysis session or whenever the Investigator questions the validity of an analysis.
 - 11.6.1.25 Type CH_X (CR). This will move the stage to the sample insertion location. Before proceeding compare the stage indicator positions with the listed sample change positions. Be sure to note the sample number and the orientation of the sample in the sample holder. Load the sample in the sample holder and screw the holder into the airlock with the handle in the 12 o'clock position.
 - 11.6.1.26 Press the pump-down button and wait for the valve driving LED to come on.
 - 11.6.1.27 Open the airlock valve by rotating the airlock lever counter-clockwise. Push the sample holder handle all the way in, and then turn the sample holder handle to the right 90 degrees (3 o'clock).
 - 11.6.1.28 Pull the sample holder handle out, still in the 3 o'clock position.
 - 11.6.1.29 Close the airlock valve by rotating the lever airlock clockwise.
 - 11.6.1.30 Press the Air Inlet button and wait about 3 seconds. Press the OFF button.
 - 11.6.1.31 Type GET POINT 46 (CR). This will move the stage to the approximate center of the thin section holder.
 - 11.6.1.32 Begin analysis of this sample.
 - 11.6.1.33 Standards should also be run at any time during an analysis session when the Investigator questions the validity of the analyses.
 - 11.6.1.34 When finished with this sample, type CONTROL G and close out the file. Open another file (steps 11.6.1.5 to 11.5.1.14) for successive samples.

12.0 ANALYZE ANOTHER SAMPLE OR SKIP TO STEP 13.0 FOR SYSTEM SHUTDOWN

12.1 Sample

- 12.1.1 Type CH (CR). The stage will move to the change position.
- 12.1.2 Verify that the dial indicators match the sample change position listed in the microprobe logbook. If they do not, contact the operator.
- 12.1.3 Press the Airlock Pump Down button.
- 12.1.4 Wait until the valve driving LED is on.

- 12.1.5 Open the Airlock valve by rotating the airlock lever counter-clockwise.
- 12.1.6 Insert sample holder handle with the handle in the 3 o'clock position and when in completely, rotate the handle counter-clockwise to the 12 o'clock position.
- 12.1.7 Pull the sample holder handle out.
- 12.1.8 Close the Airlock valve by rotating the airlock lever clockwise.
- 12.1.9 Press the Air Inlet button.
- 12.1.10 Press the Off button.
- 12.1.11 Unscrew the sample holder from the Airlock.
- 12.1.12 Change sample holders.
- 12.1.13 Screw the sample holder back into the Airlock
- 12.1.14 Repeat steps 12.1.3 to 12.1.10.

13.0 SHUT DOWN OF MICROPROBE

- 13.1 Remove the thin section from probe following steps in section 12.0.
- 13.2 If still in a QUANT routine, terminate it by typing a CONTROL G.
- 13.3 Type SAV EL 10 (CR) or the number of the element table being used. This will save the current calibration. Answer the following question prompts.
 - 13.3.1 "10 LA YMP DATE USED OK?" The question is asking if you intend to write over the old file. If your answer is yes, then continue. If your answer is no, then skip to step 13.3.4.
 - 13.3.2 Type a (CR)> This will update the calibration.
 - 13.3.3 When prompted by the program, "ENTER LABEL," type 10 LA YMP and the current date and then a (CR).
 - 13.3.4 If you intend to store the current calibration under a new file number type a NO (CR).
 - 13.3.5 Type the new file number. The computer will check to determine if the new number is used. If not, a title will be requested. The title is limited to 23 characters. Use your last name, some identifier mnemonic, and the date.
- 13.4 Put the probe into an idle mode
 - 13.4.1 Type LOA SCHED 10 (CR). This will load schedule 10.
 - 13.4.2 Type RUN BYBY (CR). This schedule will drive each spectrometer to a safe position (.50), and park it there, position the stage to the STANDARD FOCUS, and release the beam to an unregulated mode.
 - 13.4.3 Wait until the absorbed current meter indicates the beam is on and then turn the Filament Temperature knob gently to zero.
 - 13.4.4 Release the Filament Power Supply button.
 - 13.4.5 Turn off the TV camera Power Supply.
 - 13.4.6 Turn off the power switch on the TV Monitor.
 - 13.4.7 Release the Light Microscope Illuminator button and toggle the switch below it to low.
 - 13.4.8 Deactivate the Vacuum System by flipping the toggle switch on the left front of the Vacuum Chassis to STANDBY.
 - 13.4.9 Remove your floppy disk or diskette and place it in the storage box.
 - 13.4.10 Power down the SyQuest if it is in use.

- 13.4.11 Turn off the power to the TN Monitor by depressing the POWER switch located on the lower right side of the monitor (just next to the brightness control).

14.0 CALIBRATED INSTRUMENTATION

- 14.1 The Tracor Northern EDS is Operator calibrated on a monthly basis by following the instructions for calibration in the TN Operators Manual. The results, date, and name of Operator will be recorded in the Electron Microprobe Logbook.
- 14.2 The WDS is either Operator or Investigator calibrated during the course of this procedure as per section 11.5.
- 14.3 The Operator will check the accuracy of the Faraday Cup monthly with a calibrated current source (a 1.35 volt Mercury battery). Acceptable values are 9.95×10^{-9} amps, plus or minus 1×10^{-9} amps.

15.0 ACCEPTANCE/REJECTION CRITERIA

It is the responsibility of the Investigator to determine the acceptability of the data generated by microanalysis routines using the Electron Microprobe. If the Investigator chooses to define his or her own criteria for acceptability then these criteria must be noted in the Investigator's notebook.

16.0 SAMPLE STORAGE

16.1 Permanent storage

Following completion of EMP analytical investigation, samples will either be maintained in the custody of the Investigator or returned to the permanent YMP sample storage room in accordance with the procedure for Sample Identification and Control for Mineralogy-Petrology Studies (TWS-ESS-DP-101).

17.0 QUALITY ASSURANCE

17.1 Personnel Qualifications

Only qualified personnel shall be allowed to perform this work. LA YMP Investigators must undergo an orientation on the microprobe given by an Operator prior to performing this procedure. Records of certified and trained Investigators will be kept in the LA YMP personnel certification files. The Operator's signature is all that is necessary to document qualification of Investigators.

17.2 Records

- 17.2.1 The Investigator will retain all printed output from analytical sessions with the Electron Microprobe. Acceptance criteria will be entered in the Investigators controlled notebook.
- 17.2.2 The date, sample number, and Investigator's name will be entered in the Microprobe Logbook. Operating conditions, samples analysed, and observations made during analysis will be noted in the Investigator's notebook.

18.0 SAMPLE TRACEABILITY

Samples will be tracked in accordance with the procedure for Sample Identification and Control for Mineralogy-Petrology Studies, TWS-ESS-DP-101.