CENTER FOR NUCLEAR WASTE REGULATORY ANALYSES TECHNICAL OPERATING PROCEDURE	Proc. TOP 004-01 Revision 0 Page 1 of 10

Title: TOP 004-01
PROCEDURE FOR PREPARATION OF THIN SECTIONS OF GEOLOGICAL MATERIALS

EFFECTIVITY AND APPROVAL

Revision 0 of this procedure became effective on June 27, 1989. This procedure consists of the pages and changes listed below.

Page No.	Change	Date Effective
All	----	6/27/89

Supersedes Procedure No. None

Approvals

Written By	Date	Technical Review	Date
Roberto T Patalon	6/19/89	John J Russell	6/23/89
Quality Assurance	**Date**	**Cognizant Director**	**Date**
[signature]	6/26/89	Allen R. Whiting	6/27/89

CNWRA Form TOP-1

PROCEDURE FOR PREPARATION OF THIN SECTIONS OF GEOLOGICAL MATERIALS

1. Purpose

The purpose of this procedure is to describe the methods, equipment, and supplies to be used in making thin sections of geological materials.

2. Scope and Application

This procedure describes the techniques, equipment, and materials which may be utilized in the preparation of thin sections of rock and mineral specimens. The thin sections prepared using this procedure may be used for petrographic analyses of the geological materials using transmitted light, reflected light, and/or electron microscopy.

2.1 Applicable Documents

The following documents form a part of this procedure, as applicable:

(1) Center Technical Operating Procedures
(2) Center Quality Assurance Manual
(3) Operating Manual for Rock Saw/Grinder
(4) Operating Manual for Grinding/Polishing Lapidary Wheel
(5) Epoxy Material Data Sheet

3. Responsibility

(1) The cognizant Principal Investigator of the project shall be directly responsible for the implementation of this procedure and for determining acceptability of thin sections prepared using this procedure. In cases where the Principal Investigator is not a member of the Center, the Project/Element Manager shall retain this responsibility.

(2) The Center Director of Quality Assurance is responsible for providing independent surveillance, review or audits to verify implementation of this procedure.

4. Equipment and Supplies

Listed below are pieces of equipment and supplies that may be utilized in the preparation of thin sections:

(1) Rock saw/grinder

(2) Grinding/polishing lapidary wheel

(3) Convection oven

(4) Vacuum bell jar

(5) Vacuum pump

(6) Vacuum/pressure chamber

(7) Ultrasonic cleaner

(8) Petrographic microscope

(9) Binocular microscope

(10) Nitrogen tank with pressure regulator

(11) Epoxy

(12) Silica Carbide grit #220, 400, and 600

(13) Corundum grit #1000 or 1500

(14) Plastic embedding mold

(15) Petroleum jelly

(16) Metal clamp

(17) Glass plate

(18) Glass petrographic microscope slide and cover slip

(19) Glass beaker

(20) Wash bottle

(21) Ethyl Alcohol

(22) Razor blade

5. Procedures

A. Rock Preparation

1. If a rock is cohesive and firm, saw it with a diamond saw into a chip approximately 3-10 mm thick depending upon rock type, i.e. hardness and friability, and trim it square to approximately 24 x 24 mm; a larger specimen may be cut as a rectangle of approximately 24 x 34 mm, while a smaller sample may be left untrimmed. A width of 24 mm and length of 34 mm are upper limits due to the size of the glass slides.

2. A large-diameter diamond blade may be used initially for sawing large hand specimens into smaller slabs. Final trimming should be done with a thinner blade, such as a 127x 0.38 mm or similar sized diamond saw blade. The thinner blade is desirable because the cut is considerably smoother and it also conserves material.

3. If the rock is extremely porous or friable, the rock should be epoxy impregnated to bind the rock together (see Part B of this procedure). For hydrous rock or mineral specimens, special procedures are required (see Section 6. Special Procedures for Hydrous Rocks and Minerals).

4. Make sure the sample is properly labeled with its identification number(s) and/or letter(s). If necessary, the sample should be marked or labeled to indicate its orientation with respect to bedding planes, structures, or other suitable reference criteria.

B. Impregnation of a Porous or Friable Rock

1. The rock is cut to the smallest dimension possible while retaining cohesion. The rock is then dried for at least 12 hours at approximately 75°C in a convection oven.

2. In a glass beaker of suitable size, mix an appropriate epoxy designed for impregnation. The epoxy may be mixed with an appropriate epoxy dye to facilitate porosity recognition. Follow the protocol for the particular epoxy recommended by the manufacturer in the epoxy material data sheet. Line the bell jar with petroleum jelly to aid in epoxy removal should spillage occur. Place the beaker in a vacuum bell jar for about 15 minutes until the frothing ceases. Break and restore the vacuum several times. A vapor trap must be present between the bell jar and the vacuum line to trap any gas or moisture coming from the bell jar.

3. Using plastic breakaway embedding molds, immerse the rock chip in the epoxy. Label each mold with the respective sample number and, if necessary, sample orientation. Place the specimen in the vacuum bell jar for about 10 minutes until frothing ceases. Then transfer the specimen into a vacuum/pressure chamber. Line the chamber with petroleum jelly to aid clean-up should spillage occur. Evacuate the pressure chamber for about 30 minutes. Then slowly bleed nitrogen from a nitrogen gas tank into the chamber over a ten minute interval until the pressure in the chamber reaches approximately 1500 psi. After 45 minutes shut off nitrogen supply. Leave specimen under pressure in the chamber until epoxy cures, usually 24-48 hours. The curing time is given in the epoxy material data sheet.

   Note: This protocol for impregnation may be modified depending on the type of epoxy used and its respective curing time.

4. After epoxy has cured, slowly vent the nitrogen gas from the chamber. Remove the specimen from the mold.

Trim the specimen to the proper dimensions recommended in Section A above.

C. Mounting the Specimen.

1. Use a petrographic microscope frosted-glass slide approximately 26 X 50 mm in size to mount the rock specimen.

2. The specimen surface to be mounted is ground on a cast-iron lap wheel or a glass plate. First, in a water slurry of #220 silica carbide grit, the sample is ground until all saw marks, pits, and imperfections which are visible to the naked eye or with the aid of a 10X lens are removed and a smooth flat surface is obtained. The sample is then rinsed, cleaned in an ultrasonic bath for about 1 minute, and reground in a water slurry of #400 silica carbide grit until a smooth finish is reached, confirming surface finish with the use of at least a 10X lens. Again the sample is rinsed, ultrasonically cleaned for about 1 minute, and reground in a water slurry of #600 silica carbide grit until a polished surface is obtained. Rinse and clean the specimen in an ultrasonic bath and then check it under the binocular microscope to assure that the specimen is polished and contains no imperfections.

3. The thoroughly washed sample is placed, polished surface upward, on a tray to dry. Allow time for sample to dry completely (24-48 hours). Sample may be placed in a dessicator or oven (approximately 25^{o}C) to facilitate drying.

4. In a small beaker, mix an appropriate amount of mounting epoxy. Allow epoxy to set for approximately 5 minutes. An epoxy with a refractive index of 1.55 is advantageous for the distinction of common rock-forming minerals--quartz, alkali feldspar and plagioclase.

5. A thin even coat of the epoxy is spread on the polished side of the sample. Lay the sample on a flat surface. The glass slide, frosted side down, is then slowly pressed against the specimen, starting at one end from

an angle of approximately 45 degrees. Pressure is applied to the surface of the slide to remove any trapped air bubbles. The glass slide is pressed against the sample for about one minute. Using the metal clamp, carefully clamp the glass slide and sample together to allow the epoxy to cure under pressure.

Note: For specimens containing no quartz or feldspar, judging the thickness of the thin section is difficult; therefore, quartz grains can be added to the mounting epoxy at each corner of the glass slide. This method helps keep the thin section level during grinding, and allows the thin section thickness to be checked based on the interference colors of quartz.

6. After epoxy has cured completely, remove metal clamp. Any traces of epoxy on the glass can be removed gently with a razor blade.

D. Sawing and Grinding

The procedure for sawing and grinding the slide-mounted specimen, whether performed manually or by machine, is as follows:

1. Saw the mounted chip with a diamond saw 0.38 mm thick to a thickness of approximately 0.5 mm if an automated machine is used or to a final thickness of approximately 1-2 mm if held by hand. The section should be fed slowly through the saw to prevent plucking the chip from the glass slide.

2. The section is further reduced either on an automatic machine, or hand-held on a lap wheel or glass plate. A diamond impregnated wheel is recommended. If a machine is used, the microscrew adjustment is positioned so that the chip barely touches the grinding wheel. The sample is slowly advanced against the wheel. From time to time the sample is removed and examined with a petrographic microscope under crossed polars for thickness. When the specimen is about 40 microns (.04 mm) thick, grinding ceases. At this point quartz should show first-order yellow-to-orange birefringence.

3. After ultrasonic cleaning, further grinding is done by hand on a glass slide with #600 silica carbide in a water slurry. Grind the sample applying light even pressure. After 15 to 20 seconds the slide is rinsed with clear water and examined under the petrographic microscope for thickness. This process continues until the proper thickness is obtained (i.e., when quartz exhibits first-order gray birefringence).

4. The final step is done on a glass plate with #1000 or #1500 corundum grit in a water slurry after cleaning the samples from Step 3 in an ultrasonic bath for about 1 minute. This hand grinding provides good control over the final thickness which should be approximately 30 microns (.03mm), as indicated by birefringence colors. Also, hand grinding practically eliminates plucking.

5. The use of a cover slip is optional and depends upon the intended uses of the specimen. If the slide is to be stained for mineral identification, to be used for the study of opaque minerals under reflected light, or to be examined in the electron microscope, a cover slip should not be used. If the specimen is for normal petrographic work, then a cover slip is desirable.

6. Special Procedures for Hydrous Rocks and Minerals

A. Rock Preparation

1. Special procedures are required to make thin sections of hydrous geological materials such as zeolites. The zeolites are soft rocks, thus the initial chip cut for a thin section with a diamond saw should be approximately 8-10mm thick. After trimming the chip, it should be placed in an oven (at approximately 25°C) or dessicator to dry. The dry chip is then ready to be impregnated (Section 5.B) or to be ground for mounting.

2. If impregnation is not desired, the protocol is as follows: The chip is first ground in a slurry of ethyl alcohol and #220 silica carbide grit. The sample is ground until all saw marks, pits, and imperfections

which are visible to the naked eye or with the aid of a 10X lens are removed and a smooth flat surface is obtained. The sample is then rinsed, cleaned in an ultrasonic bath of ethyl alcohol for about 1 minute, and reground in an ethyl alcohol slurry of #400 silica carbide grit until a smooth finish is reached, confirming surface finish with the use of at least a 10X lens. Again the sample is rinsed with ethyl alcohol, ultrasonically cleaned for about 1 minute, and reground in an ethyl alcohol slurry of #600 silica carbide grit until a polished surface is obtained. The specimen is rinsed and cleaned in an ultrasonic bath, then checked under the binocular microscope to assure that the sample is polished and contains no imperfections.

3. The thoroughly washed sample is placed, polished surface upward, on a tray to dry. Allow time for sample to completely dehydrate (24-48 hours). Sample may be placed in a dessicator or oven (approximately 25°C) to facilitate dehydration.

4. After sample is completely dry, check again to ensure the the surface is flat. The sample is now ready to mount to a thin section slide. The protocol to follow is the same as described in Section 5.C.

5. If the thin section is to be impregnated, follow procedures described above in Sections 5.B and 5.C.

6. The protocol to follow to finish the impregnated sample is given below.

B. Sawing and Grinding

The procedure for sawing and grinding the slide-mounted specimen, whether performed manually or by machine, is as follows:

1. Follow the steps given in 5.D.1.

2. Follow the steps given in 5.D.2.

3. After ultrasonic cleaning allow the thin section to dry. Further grinding is done by hand on a glass slide with #600 silica carbide in an ethyl alcohol slurry. Grind the sample applying light even pressure. After 10 to 15 seconds the slide is rinsed with ethyl alcohol and examined under the binocular microscope for thickness. This process continues until the proper thickness is obtained (e.g., quartz exhibits first-order gray birefringence).

4. The final step is done on a glass plate with #1000 or #1500 corundum grit in an ethyl alcohol slurry after cleaning the samples from Step 3 in an ultrasonic bath of ethyl alcohol for about 1 minute.

7. Identification and Storage

1. The prepared thin sections shall be stored in a glass slide tray or cabinet or other appropriate container. Each slide will be properly labeled using a diamond-tipped stylus, or other suitable material.

2. Records of identification numbers or labels and source locality of the geological material, the name of the individual(s) who prepared the thin section, and the date it was prepared, are to be kept at the Center in the project files.

8. Records

No records other than sample identification and source locality are required to be generated or maintained regarding preparation of thin sections of geological materials. The thin sections are either acceptable for characterization work (e.g., petrographic analysis, porosity studies, etc.), or not acceptable, and will be utilized only if acceptable as determined by the cognizant Principal Investigator or his Project/Element Manager.

9. Reference

The above procedures are based on the techniques given in Laboratory Handbook of Petrographic Techniques by C.S. Hutchison (1974, Wiley and Sons, New York, pp. 1-9).