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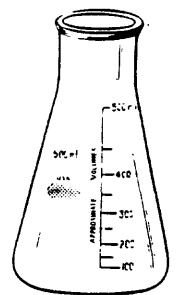
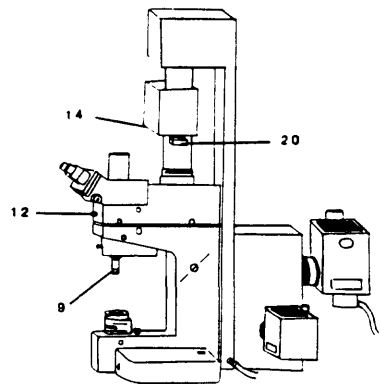
Geological Survey

COAL PETROGRAPHIC LABORATORY  
PROCEDURES AND SAFETY MANUAL II

by

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Open-file Report 94-631



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## **PREFACE**

This manual is a much-needed revision of an earlier USGS publication, *Coal Petrographic Laboratory Procedures and Safety Manual* (Moore and Stanton, 1985). The development of new sample preparation and analysis techniques has necessitated both an overhaul of many established procedures and the adoption of new ones.

This manual has a dual purpose: 1) to provide new employees, interested colleagues, industry representatives, and the public a guide to the methods and procedures used in sample preparation and analysis within a coal petrographic laboratory, and 2) to act as a reference for safety and consistency in laboratory sample preparation. Precise methodology ensures reliable quality control and provides maximum safety margins for the user. In writing this handbook, we have tried to set procedures that will allow employees to work with a minimum amount of supervision.

Many of the sample preparation procedures used in this manual follow the American Society for Testing Materials' (ASTM) recommendations. The appropriate ASTM standard number is noted at the beginning of these procedures. References are cited for sample preparation methods taken from the literature. After each procedure, necessary safety precautions are listed; these **must** be followed each time a particular technique is performed.

Although this manual is as up-to-date as possible, sample preparation and analysis techniques are constantly being improved and better materials for use in preparations are constantly being developed. Future revisions of this manual will be periodically necessary.

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## **1.0 INTRODUCTION**

### **1.1 Purpose of Laboratory**

The purpose of the USGS coal petrography laboratory is to prepare coal, rock, peat, and soil samples for physical and optical analyses. Physical analyses performed in this laboratory include low temperature ashing (LTA), high temperature ashing (HTA), X-ray radiography, and chemical determinations. Optical analyses consist of vitrinite reflectance and spectral fluorescence measurements (indicative of coal diagenesis) and maceral (organic) analysis. Subsplits of samples are also sent to analytical laboratories for chemical and mineralogical analyses.

From the results of these analyses, coal samples can be characterized by their composition, rank, and quality. Coal bed variability can also be determined when a suite of samples is analyzed from one coal bed. The detailed study of coal beds is important for both utilization and geologic studies. In addition to being of value in mine planning and utilization, coal research gives clues to the geologic processes that formed the coal bed.

### **1.2 Types of Samples**

Coal is the major rock type processed in the laboratory. Occasionally, other rocks associated with coal beds are prepared (e.g., claystone, shale, limestone, sandstone). Peat and soil samples are also periodically processed and analyzed.

When coal is received as a bulk sample in the laboratory, it is usually in one of two forms: 1) core sample or 2) coal from a channel sample. A core sample arrives as an intact, oriented unit that readily allows x-raying, description, and subsampling. A channel sample, usually collected from a fresh mine face, arrives as broken and unoriented pieces in a bag. Channel samples collected from outcrops may be weathered, greatly limiting the types of analyses that can be performed reliably. Although sample collection modes may differ, preparations for each sample are similar in many respects.

### **1.3 General Safety Rules**

- 1. All laboratory procedures must be carefully studied before any attempt is made to perform them.**
- 2. Safety glasses must be worn when performing any work in the laboratory.**
- 3. The wearing of contact lenses is not permitted when using resins, acids, or organic solvents.**
- 4. No open-toed shoes may be worn in the laboratory.**
- 5. Fume masks must be worn when working with resins and solvents.**
- 6. Rubber or disposable plastic gloves must be worn when using acetone, methanol, acids, toluene, resins, and/or colloidal silica polishes.**
- 7. The following items must be used in a fume hood: acetone, methanol, toluene, resins, and acids.**
- 8. Before any chemicals and/or resins are used, the employee MUST consult the Material Safety Data Sheet and Safety Files. These books contain important materials and hazardous waste disposal information.**
- 9. If a laboratory procedure has the potential to generate smoke or heated dust (e.g., sawing wood), a Burn Permit must be obtained ahead of time from the USGS Safety Office to avoid the accidental triggering of lab smoke alarms.**
- 10. All reagents should be clearly labelled and stored in designated cabinets. Incompatible chemicals should NEVER be stored in the same cabinet. All heavy items and chemicals should be stored below counter level when practicable.**
- 11. Any visitors and minors may be present only with the permission of the lab supervisor, and must wear safety glasses at all times.**
- 12. The last employee to leave for the day should check the status of ovens, furnaces and all other electrical devices, turn off all lights, and lock the lab doors.**

## 2.0 LABORATORY ORGANIZATION

### 2.1 Sample Flow

This laboratory follows ASTM standard D2013-86 (ASTM, 1992a) procedures for crushing, grinding, sieving, and splitting coal samples. The idealized steps in processing a sample are illustrated in Figure 1. When a bulk sample is received at the laboratory, it is initially crushed to -8 mesh (<2.4 mm) and then split into three equally sized sub-samples: 1) an analytical split, 2) a petrography split, and 3) a storage split. When there is insufficient sample (less than 100 grams) for all three splits, the storage split is omitted.

Using a large high-speed rotary grinder with a 2 mm sieve screen, the analytical split is crushed to less than 20 mesh (850  $\mu$ m). Two subsplits (an ashing split and a storage split for storage) are then obtained by using a riffle sampler (opening > 2.5 mm). Again, if there is an insufficient amount of sample for two splits, the storage split is not taken.

The ashing split is then ground to less than 60 mesh (250  $\mu$ m) using a small high-speed rotary mill grinder. Two subsplits are made from the ground sample: one for high temperature ashing (HTA) and the other for low temperature ashing (LTA) determination. Because little sample is needed for the HTA, only a very small subsplit is taken (about 2 g). If there is more sample than needed for the LTA (~ 18 g), the remaining unashed sample is retained as a storage split. The ashed remains are held for X-ray mineralogy.

Using a large high-speed rotary grinder with a 2 mm sieve screen, the petrography split is crushed to less than 20 mesh (850  $\mu$ m). Two subsplits obtained by using a riffle sampler (opening 2.5 mm), are as follows: 1) a split for pellet making, and 2) a storage split. Again, if there is an insufficient amount of sample for two splits, the storage split is not taken.

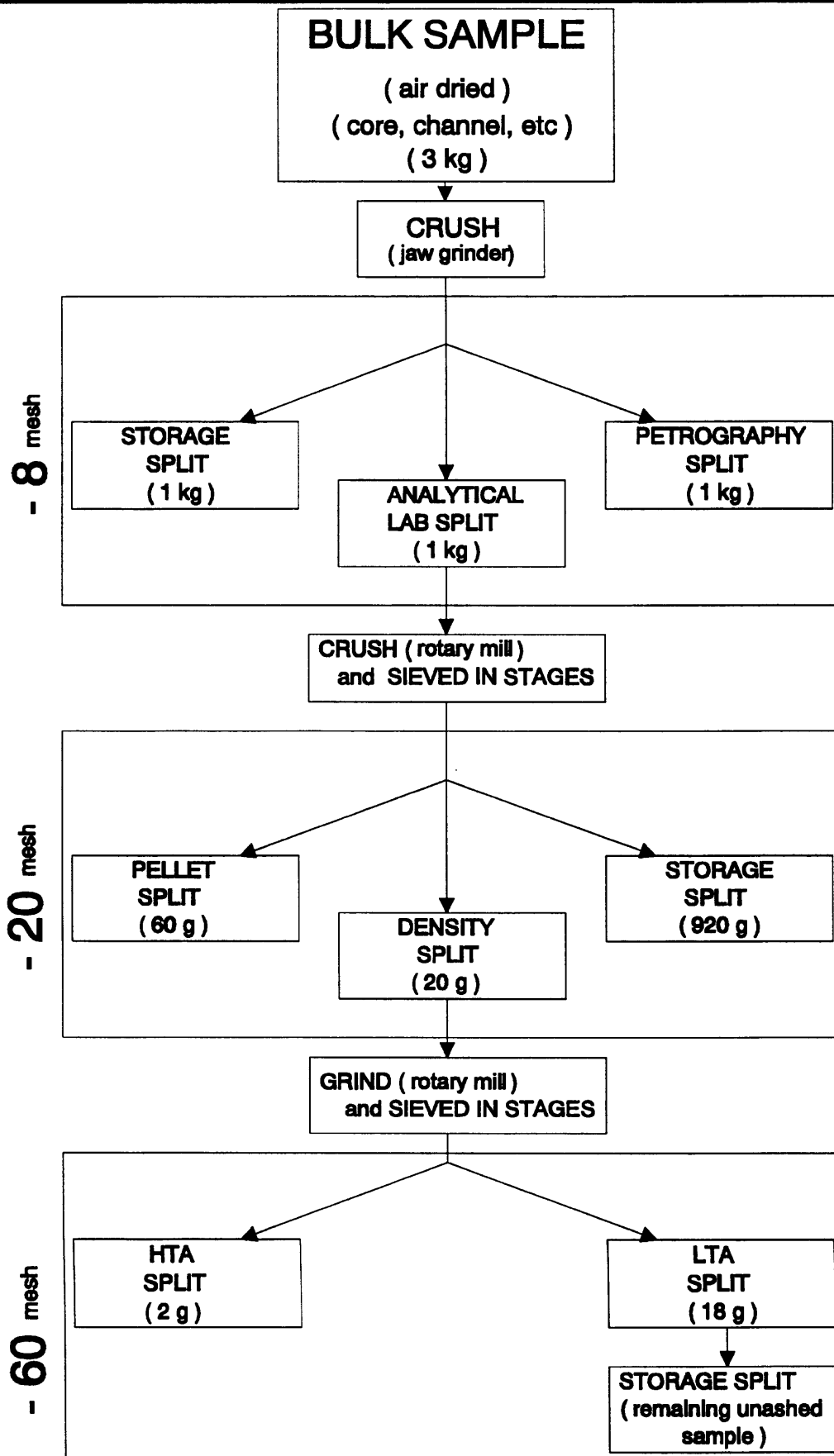
To summarize, a sample progresses through three basic preparation steps: 1) preliminary crushing and splitting at 8 mesh, 2) crushing to 20 mesh for the petrographic splits, 3) 60 mesh fine grinding of the ashing subsplit for ash determinations. This preparation procedure gives representative subsamples with the least amount of contamination.

### 2.2 Record Keeping

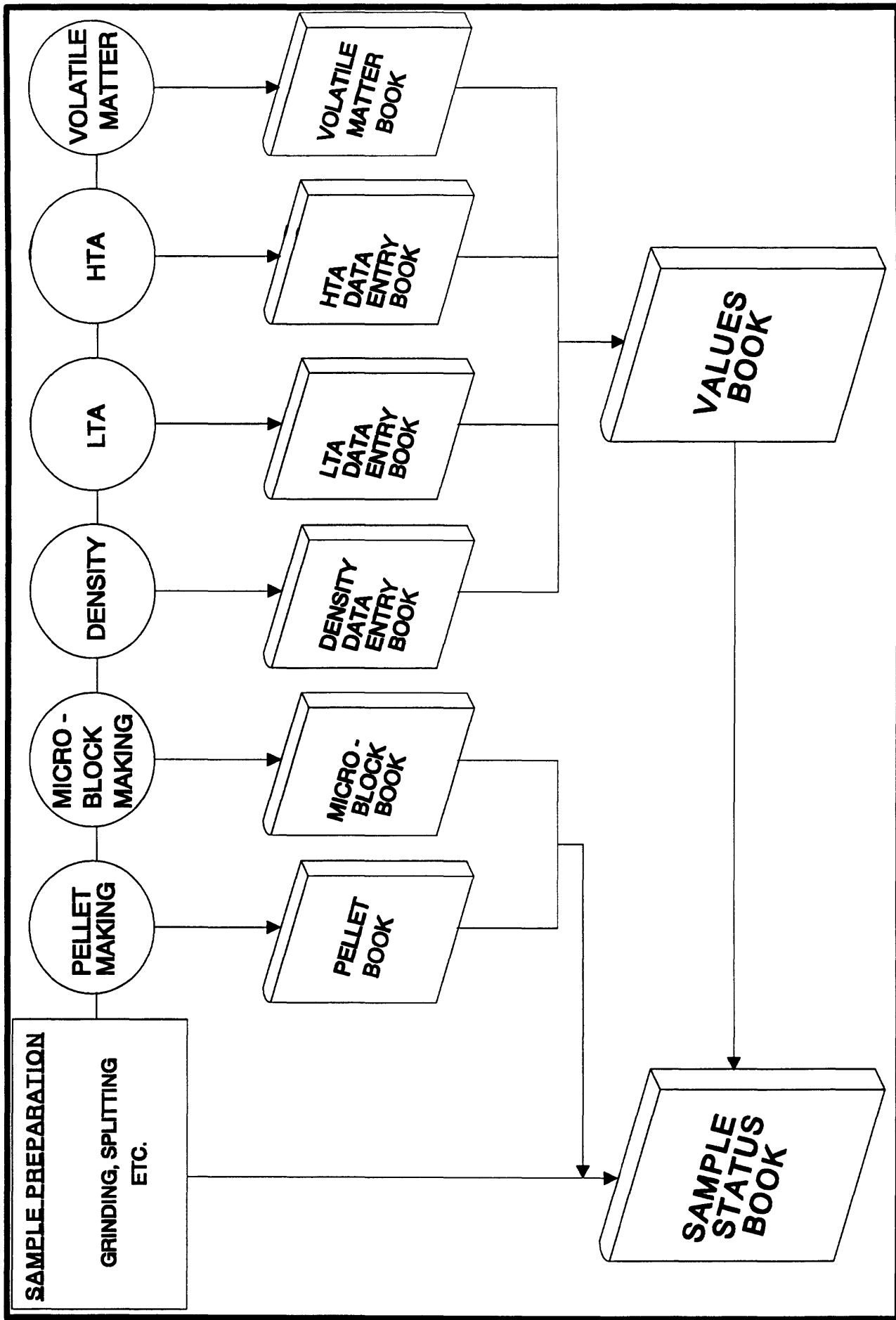
Laboratory organization not only entails the physical handling of samples, but also the maintenance of sample status records. It is important to know both a sample's stage of preparation and its location in the lab. Because this laboratory has hundreds of samples at various stages of processing at any given time, written and computerized ledgers are used to track samples.

In this laboratory, eight notebooks are maintained (Figure 2). Six of these books (HTA, LTA, Pellet, Density, Volatile Matter, and Microblock) are used primarily for on-the-spot entry of specific procedures. A sample's progress, in these and all other procedures, is noted in the Sample Status book --- a three-ring binder volume of completed and empty sample status sheets. The sample status sheets (Figure 3) were designed to logically follow the flow of sample preparation. The Values Book is used as a repository of all finalized analytical and petrographic data gathered for each sample processed in the laboratory.





**FIGURE 1: Flow diagram of idealized sample preparation**



**FIGURE 2: Flow diagram of record keeping**



### **3.0 SAMPLE HANDLING**

#### **3.1 Sample Splitting and Contamination**

All final interpretations of data depend on the quality of sample preparation in the laboratory. Ideally, any subsplit that is analyzed will be representative of the original bulk sample. To assure that the sample is representative, mechanical riffles should be used. Particle size must also be considered when subsplits are small. Improper splitting gives unrepresentative splits and leads to erroneous results.

Sample contamination is another common problem within the laboratory. Grinding machines, splitting riffles, mortar and pestles, sieves, and other equipment that come into contact with the coal or rock samples must be thoroughly cleaned between samples using compressed air, acetone, and disposable wipes. In addition, laboratory counter tops, fume hoods, and the general working area should be kept as clean as possible to prevent sample contamination from dust.

#### **3.2 Importance of Clear Labelling**

Personal notation does not work within the laboratory. Each sample bag and container must be neatly and clearly marked as to its contents so others may understand it. A mislabelled sample can have disastrous effects on interpretations. Any sample that is spilled, dropped, or lost should be immediately reported to a supervisor (an unrepresentative or mismarked sample is worse than no sample at all). If any question arises about a sample number, it should be noted on the bag or container and a supervisor should be informed.

## 4.0 SAMPLE CRUSHING AND GRINDING (ASTM 2013-86, 1992a)

### 4.1 Jaw Crusher (-8 mesh grinding)

Equipment . . . . .	jaw crusher large riffler acetone disposable wipes wire brush plastic bags permanent marker pen Sample Status Book
Safety Equipment . . . . .	dust respirator safety glasses/goggles ear mufflers rubber gloves laboratory coat and/or apron

#### 4.1A Preliminary Steps

1. Clean the crushing plates and platform of the jaw crusher, as well as the sample catch bin, with compressed air, acetone and disposable wipes.
2. Check the gap between the crushing plates to ensure that it is a minimum of 1/4" wide.
3. Place the cardboard shelf on the platform under the crushing plates.
4. Turn on the hood vacuum.

#### 4.1B Crushing the Sample

1. Turn on the jaw crusher.
2. Slowly and steadily pour the bulk sample between the crushing plates. Crush until all of the sample has passed through the plates.
3. Turn off the machine and open the crushing plates.
4. Sweep all sample residue from the plates and lower platform into the catch bin. Remove the sample catch bin and pour the crushed split into labelled sample bag.

5. Clean the plates, platform, and sample catch bin with compressed air, wire brush, acetone and disposable wipes.

#### 4.1C Splitting the Sample

1. Split the sample into thirds using a large riffler. Mark the sample bags "A-Labs," "Petrography," and "Storage" and with the sample identification. If there is insufficient sample for the three splits, do not take the storage split.
2. Clean the riffler with compressed air, acetone and disposable wipes in a fume hood.

#### 4.1D Record Keeping

Record that the sample was ground and split in the Sample Status Book.

#### 4.1E Safety Procedures

##### **1. Using the Jaw Crusher**

- **dust respirator, ear protection, and safety glasses or goggles must be worn at all times**
- **laboratory coat and/or apron must be worn**
- **keep fingers and clothing away from crushing plates**

##### **2. Using Acetone**

- **avoid breathing fumes, use in a fume hood if possible**
- **wear protective gloves**
- **avoid skin and eye contact**

## 4.2 Rotary Mill Grinder (-20 mesh grinding)

Equipment . . . . .	rotary mill grinder disposable wipes mortar and pestle permanent marker pen wire brush medium riffler 20 mesh (2 mm) sieve sample jars or bags Sample Status Book
Safety Equipment . . . . .	dust respirator safety glasses/goggles ear muffs protective gloves laboratory coat and/or apron

### 4.2A Preliminary Steps

1. Check that the proper sizing screen is in the grinding machine (use the 2 mm screen to obtain -20 mesh sample).
2. Clean the machine using compressed air, wire brush, acetone and disposable wipes.
3. Clean the sample catching cup and 20 mesh sieve with compressed air, acetone and disposable wipes in a fume hood.

### 4.2B Grinding the Sample

1. Close the mill door and plug in the machine.
2. Close the slide between the overhead sample holder and the grinding chamber.
3. Place the sample into the overhead sample holder.
4. Put the cap on the sample holder.
5. Turn on the machine and then open the slider.
6. Let the sample grind completely.
7. Turn off the machine and allow the blades to stop turning.
8. Open the mill door.
9. Check to see that all of the sample has passed through the screen. If not, close the mill door and continue to grind until all of the sample passes through the screen.

10. Shake the mill to ensure that all of the sample has fallen into the sample cup.
11. Remove the sample cup from under the mill and place the sample into a 20 mesh sieve.
12. Clean all grinder parts using compressed air, wire brush, acetone and disposable wipes.

#### 4.2C Sieving the Sample

1. Place the crushed sample into a 20 mesh sieve (step 11, section 4.2B).
2. Gently shake the sample through the screen.
3. Place any sample that does not pass through the screen into a mortar and pestle.
4. Grind by hand until all of the sample passes through the 20 mesh screen.
5. Clean the sieving and grinding equipment with compressed air, acetone and disposable wipes in a fume hood.

#### 4.2D Splitting the Sample

1. Use a medium-size riffler and obtain the following subsplits: 1) ashing split (20 g), 2) pellet split (60 g) and 3) storage split (remainder).
2. Bag or jar all of the splits.
3. Clean the riffler using compressed air, acetone and disposable wipes in a fume hood.

#### 4.2E Record Keeping

Record all grinding and splits in the Sample Status Book.

#### 4.2F Safety Procedures

##### **1. Using the Rotary Grinder**

- **dust respirator, safety glasses and ear mufflers must be worn at all times**
- **NEVER open the grinder door when the blades are in motion**
- **ALWAYS unplug the machine before cleaning**

##### **2. Using Acetone**

- **avoid breathing fumes, use in a fume hood if possible**
- **wear protective gloves**
- **avoid skin and eye contact**



### 4.3 Rotary Mill Grinder (for -60/-100/-200 mesh grinding)

Equipment . . . . .	high-speed rotary mill oven capable of maintaining 105°C acetone and disposable wipes splitting paper sample bags or jars marker pens sieve series brushes Sample Status Book
Safety Equipment . . . . .	dust respirator safety glasses/goggles rubber gloves laboratory coat and/or apron

#### 4.3A Preliminary Steps

1. Preheat the oven to 60°C for organic materials or 105°C for all other materials.
2. Place opened sample jars in the oven for one hour to dry samples.
3. Clean the mill with compressed air, brush, acetone and disposable wipes.
4. Install the 0.2mm screen on the interior grinding platform being sure that its orientation arrow matches the red arrow on the mill exterior (i.e., counterclockwise).
5. Set the grind selection switch to the "1" position.

#### 4.3B Grinding and Sieving the Sample

1. Set the mill timer to at least one minute (power will not come on otherwise).
2. Push the "ON" button to the up position.
3. Introduce the sample through the feed tube in a slow and steady manner.
4. When grinding is complete (this should only take several seconds --- listen), push the "ON" button to the down position. Let the unit sit for ten seconds to let any suspended fines settle.

5. Unscrew the mill lid; brush off and collect on splitting paper any ground sample that has accumulated on this surface (usually a fair amount).
6. Remove the 0.2mm screen and knock out any adhering sample onto the splitting paper.
7. Brush any sample adhering to the grinding wheel and platform into the sample collection pan. Remove the pan and brush its contents out onto the splitting paper.
8. Screen complete ground sample (lid, screen and pan brushings) through the desired sieve size. Any unpassed material may either be ground by hand or reprocessed through the mill.
9. Finished samples should be placed into labelled storage bottles.
10. Between each sample, a rigorous cleaning regime is needed to prevent cross-contamination of samples. The 0.2mm screen and grinding wheel must be processed as follows: compressed air, ultrasonic cleaning, oven dry, acetone wipe. All other mill parts (bowl, lid, interior and exterior feed collars and mill unit interior) should be cleaned off with compressed air, acetone and disposable wipes.

#### 4.3C Record Keeping

Enter all grinding and sample information into the Sample Status Book.

#### 4.3D Safety Procedures

##### **1. Using the Rotary Mill**

- **use in a vented hood**
- **dust respirator and safety glasses/goggles must be worn at all times**
- **laboratory coat and/or apron should be worn**
- **do not insert fingers into feed collar unless the unit is unplugged**
- **always tightly secure the unit lid before grinding a sample**

##### **2. Using Acetone**

- **avoid breathing fumes, use in a fume hood if possible**
- **wear protective gloves**
- **avoid skin and eye contact**

## 5.0 PELLET AND BLOCK SAMPLE CASTING

### 5.1 Pellet Making

Equipment . . . . .	pellet molds (ID=1", 1/4" and 1" plugs) epoxy resin and hardener stirring sticks paper cups marking pen and sample labels oven capable of maintaining 60°C masking tape and release agent toluene and acetone hand press analytical triple-beam balance Pellet and Sample Status Books
Safety Equipment . . . . .	rubber gloves rubber apron or laboratory coat organic vapor respirator safety glasses/goggles fume hood

#### 5.1A Procedure for Anthracite and Bituminous Coal (ASTM D2797-85[1990], 1992c)

##### 5.1A.1 Preliminary Steps

- a. Label two molds with sample number.
- b. Label a paper cup with sample number.
- c. Spray release agent onto the molds and plugs.
- d. Place two 1/4" plugs into each mold bottom and tape into place.
- e. Pour the reserved petrographic split (approximately 15g) into the paper cup.

##### 5.1A.2 Mixing and Pouring the Epoxy (performed in a fume hood)

- a. Mix the epoxy resin and hardener in correct proportions for one minute.

- b. If a thinner (less viscous) mixture is desired, add 2-3 ml of toluene.
- c. Add some epoxy to the coal and mix thoroughly until the coal is "just wet."
- d. Place equal amounts of this coal/epoxy mixture into each of the two molds. Insert a 1" plug into each mold and tamp down lightly by hand.

#### 5.1A.3 Pressing and Compacting the Mixture

- a. Place the pellet mold under the hand press and pump to complete compression. Release pressure.
- b. Repeat this cycle two more times. Remove the pellet mold; wipe the press stage and mold clean of any exuded epoxy.
- c. Place the pellet mold in a fume hood to cure at room temperature overnight. If the sample does not setup, it should be placed in a 60°C oven for at least one hour to complete curing.

#### 5.1A.4 Popping and Labelling the Pellets

- a. Extract the pellet and all plugs from the mold by using a hand press.
- b. Place an ID label on either surface of the pellet.
- c. Replace the bottom plugs and pellet (ID side up) into the mold.
- d. Pour fresh, clear epoxy-resin to the rim of the mold.
- e. Let cure overnight in a fume hood at room temperature (for fast curing, place in an oven for one hour at 60°C).
- f. Pop the pellet out using a hand press (to avoid damaging the pellet, place a 1/4" plug on the epoxy surface and press out the plug).

#### 5.1A.5 Cleaning the Molds (performed in a fume hood)

- a. Place the empty, used molds and plugs in an acetone bath overnight.
- b. Remove the molds from the acetone scrub them with steel wool.

- c. Rinse the molds with acetone and then let them air dry.
- d. Store the clean, dried molds and plugs away from dirt and dust.

#### **5.1A.6 Record Keeping & 5.1A.7 Safety Procedures**

See sections 5.1C.3 and 5.1C.4.

#### **5.1B Procedure for Lignite and Subbituminous Coals**

Aside from the following exceptions, the procedure for casting pellets of lignite and subbituminous coals is exactly the same as that given for anthracites and bituminous coals under 5.1A: a) Samples must be placed in a petri dish and oven-dried for 2 hours at 60°C prior to any pelleting, and b) Proper coal/epoxy mixture will appear "gummy" or "oozy."

#### **5.1C Procedure for Sands, Silts and Clays**

##### **5.1C.1 Preliminary Steps**

- a. Place the sample in a petri dish and dry in an oven for 24-48 hours at 105°C.
- b. Breakup any portions of the sample (especially clays) that may have agglomerated during drying.
- c. Label a 1" diameter plastic pellet cap and then spray it with a release agent.
- d. Pour the sample into the pellet cap until it is half filled.

##### **5.1C.2 Mixing and Pouring the Epoxy (performed in a fume hood)**

- a. Mix the epoxy resin and hardener in correct proportions. Either a low viscosity resin (e.g., Versamid [see disclaimer]) or a toluene-thinned resin must be used for proper results.
- b. Overpour the sample with the epoxy almost, but not quite, filling the pellet cap.
- c. Vacuum impregnate the sample for approximately five minutes (see 5.5). Do not let the mixture "boil over." After impregnation, remove the sample to cure at room temperature in a fume hood overnight.

- d. Cut the cured sample out of the pellet cap and label. The sample is ready for thin sectioning. For SEM use, the pellet must have an epoxy cap (see 5.1A).

#### 5.1C.3 Record Keeping

- a. Record in the Pellet Book that the pellet was made and labelled.
- b. Record in the Sample Status Book the date that the pellet was made.

#### 5.1C.4 Safety Procedures

##### **a. Using Epoxy Resin**

- **organic vapor respirator/safety glasses/rubber gloves must be worn**
- **do not wear contact lenses**
- **avoid skin and eye contact**
- **must be used in a fume hood**
- **laboratory coat and/or apron must be worn**

##### **b. Using the Hand Press**

- **safety glasses/goggles must be worn**
- **properly secure mold under press**

##### **c. Using Acetone**

- **safety glasses/goggles and rubber gloves must be worn**
- **avoid breathing fumes, use in fume hood if possible**
- **avoid skin and eye contact**

## 5.2 Microblock Casting

Equipment . . . . .	pellet molds (ID = 1" with 1/4" plugs) rock or hand saw epoxy resin and hardener stirring sticks and paper cups sample labels and marking pen oven capable of maintaining 105 °C masking tape release agent triple beam balance Sample Status and Microblock Books
Safety Equipment . . . . .	rubber gloves laboratory apron and/or coat organic vapor respirator safety glasses/goggles fume hood

### 5.2A Cutting the Sample

1. If the sample is oriented, clearly mark the orientation with marker and masking tape.
2. Using a water-lubricated rock saw (or band saw) cut a cube to fit into a pellet mold.
3. Let sample dry at room temperature for 8 hours or put it into a 60°C oven for one hour.

### 5.2B Casting the Pellet (performed in a fume hood)

1. Label a mold with sample number.
2. Place two 1/4" plugs into the mold bottom and tape into place.
3. Spray a release agent into the mold.
4. Mix a small amount of epoxy resin and hardener in the correct proportions.
5. Place the sample into the mold and overpour with a few drops of the resin mixture.
6. Let the resin set up at room temperature in a fume hood for about two hours (this will prevent subsequent floating of the sample). Place a label onto the sample in the mold.

7. Mix and pour a fresh resin mixture up to the rim of the mold.
8. Let the sample cure overnight in a fume hood or put it into a 60°C oven for one hour.

#### 5.2C Record Keeping

1. Record in the Microblock Book the date that the sample was cast.
2. Record in the Sample Status Book that a microblock was made.

#### 5.2D Safety Procedures

##### **1. Using the Rock or Band Saw**

- **safety glasses/goggles and ear mufflers must be worn**
- **laboratory coat and/or apron must be worn**
- **make sure that all saw features are tightened**
- **be sure that the proper blade for the job is used**
- **keep fingers and clothing away from all moving parts**
- **never force the saw blade to cut too fast**

##### **2. Using Epoxy Resin**

- **organic vapor respirator/safety glasses/rubber gloves must be worn**
- **do not wear contact lenses**
- **avoid skin and eye contact**
- **must be used in a fume hood**
- **laboratory coat and/or apron must be worn**



### 5.3 Microsample Casting

Equipment . . . . . pellet molds (ID = 1" with 1/4" plugs)  
epoxy resin and hardener  
stirring sticks and paper cups  
markers  
release agent  
hand drill  
Sample Status and Pellet Books

Safety Equipment . . . . . organic vapor respirator  
rubber gloves  
laboratory apron and/or coat  
safety glasses/goggles  
fume hood

#### 5.3A Preliminary Steps -- Making the Resin Blank (performed in a fume hood)

1. Make the epoxy resin blank by pouring a properly proportioned mixture into a stainless steel mold (fill to rim) spaced at the bottom with two 1/4" plugs. All mold parts should have previously been coated with release agent.
2. Let the blank cure for one day in a fume hood, or for one hour in a 60°C oven.
3. Pop out the blank with a hand press.
4. Polish one side of the blank to a minimum 5  $\mu\text{m}$  polish.
5. Drill a small, shallow hole of appropriate size (this will depend on the amount of sample) into the polished blank face. Use compressed air to blow any debris clear of the hole.

#### 5.3B Mixing and Casting the Sample

1. Combine the crushed sample with an epoxy resin mixture in either a paper cup or in the pre-drilled hole.
2. Make sure to fill the pre-drilled hole with the sample mixture level to the pellet surface. Tap the mold lightly to release any entrained air bubbles.
3. Wipe away any excess epoxy from the blank surface. Write sample ID on the pellet side with a marker.

4. Let the sample cure for one day in a fume hood, or for 2 hours in a 60°C oven.

### 5.3C Record Keeping

1. Record in the Pellet Book that the sample was prepared.
2. Record in the Sample Status Book that a microsample was made.

### 5.3D Safety Procedures

#### **1. Using Epoxy Resin**

- **organic vapor respirator/safety glasses/rubber gloves must be worn**
- **do not wear contact lenses**
- **avoid skin and eye contact**
- **must be used in a fume hood**
- **laboratory coat and/or apron must be worn**

#### **2. Using the Hand Drill**

- **safety glasses/goggles must be worn**
- **keep fingers and clothing away from all moving parts**

## 5.4 Block Casting

Equipment . . . . .	polyester resin and hardener silicone block molds paper cups mixing sticks dye/gloss (optional) toluene (optional) rock saw
Safety Equipment . . . . .	safety glasses/goggles rubber gloves organic vapor respirator laboratory apron or coat fume hood

### 5.4A Cutting the Sample

1. Friable coal blocks or core pieces should be reinforced with masking tape or a resin coat to prevent breakage.
2. Cut the sample with a rock saw (maintaining proper orientation) to produce the desired block size.
3. Dry the cut sample in a 60°C oven for 2 hours.

### 5.4B Casting the Sample (performed in a fume hood)

1. Place the labelled sample, cut face down, into a silicone mold. Note: difficult to label sample surfaces should be partially overpoured with resin; a sample label can then be affixed to this first layer before a final resin coat is poured. Alternately, the finished block may be labelled by indelible marker or engraving.
2. Mix polyester resin and hardener in correct proportions. Add dye or gloss, if desired.
3. Overpour the sample in the silicone mold.
4. Let the cast sample cure for a day at room temperature inside a fume hood. If the resin does not completely setup after a day, place in a 60°C oven for 1-2 hours. Remove and let cool; the resin should have hardened.

5. Unmold the sample and place in a storage desiccator (fresh polyester resin exudes a heavy, unpleasant odor).

#### 5.4C Suggestions

1. Some dyes and glosses can inhibit hardening; to compensate, add a higher proportion of hardener.
2. Low-density materials should not be cast in a single step as they may float. An initial thin resin layer should be poured (and setup for at least one hour) to anchor the sample to the silicone mold. This can then be followed by a final pour.
3. Low-rank and friable samples should receive a thin initial pour of toluene-thinned resin and then be vacuum impregnated for at least five minutes. Longer impregnation times can be achieved if the proportion of hardener in the resin mix is adjusted. See section 5.5 for more details on vacuum impregnation. Once it is satisfactorily impregnated, the sample is ready for a final overpour.

#### 5.4D Record Keeping

Record in the Microblock book that a block was formed and labelled.

#### 5.4E Safety Procedures

##### **1. Using the Rock Saw**

- **safety glasses/goggles and ear muffers must be worn**
- **laboratory coat or apron should be worn**
- **make sure that all saw features are tightened**
- **make sure the proper type of saw blade is used**
- **keep fingers and clothing away from moving parts**
- **never force blade to cut too fast**

##### **2. Using Polyester Resin**

- **organic vapor respirator/safety glasses/rubber gloves must be worn**
- **do not wear contact lenses**
- **avoid skin and eye contact**
- **must be used in a fume hood**
- **laboratory coat or apron should be worn**

## 5.5 Vacuum Impregnation

Equipment	vacuum chamber vacuum pump pellet molds silicone block molds polyester or epoxy resin and hardener mixing sticks and paper cups release agent
Safety Equipment	organic vapor respirator safety glasses/goggles rubber gloves laboratory coat and/or apron fume hood

It is often desirable to vacuum impregnate coals that are low-rank, "dirty," or are cores that will be slabbed (cut into 1/4"-1/2" thickness) for X-ray radiography. Vacuum impregnation causes any pores in the coal to fill with resin; the more resin that penetrates the coal, the less likely that a sample will crack or flake. Vacuum impregnation may be used with core samples, hand samples, microblocks and crushed coal.

### 5.5A Hand Samples, Core Samples and Microblocks (performed in a fume hood)

1. Place the cut and labelled sample into either a pellet mold (microblock) or a block mold (hand and core samples).
2. Prepare an epoxy resin mixture to the correct proportions and pour enough into the mold to just cover the sample (be sure not to fill the mold to capacity).
3. Put the sample into a vacuum chamber. Note: If the sample tray covers any of the chamber venting holes, raise it up on blocks or place it on a platform.
4. Close and seal the vacuum chamber.
5. Turn on the vacuum pump. Check the vacuum chamber seals. If necessary, replenish silicone sealant and then reseal the chamber seals.
6. Watch the sample and vent air into the chamber if any resin begins to boil over.
7. Run vacuum pump for 5-10 minutes.
8. Turn off the vacuum pump and vent air slowly into the chamber.

9. Remove the sample and cure overnight in a fume hood, or for 1 hour in a 60°C oven.

#### 5.5B Crushed Samples (performed in a fume hood)

1. Mix the sample with an ample amount of epoxy resin mixture in a paper cup.
2. Place this mixture into a previously prepared (labelled and sprayed with release agent) stainless steel mold with one 1/4" bottom plug and pour a small amount of resin on top.
3. Put the sample and open-topped mold into a vacuum chamber.
4. Follow steps 5 through 8 of the preceding section (5.5A).
5. Remove the sample and place a top plug in the mold.
6. Place the pellet mold under a hand press and pump to complete compression. Release pressure. Repeat two more times.
7. Let the sample cure overnight in a fume hood. If the sample still flakes or cracks, try using more resin, longer impregnation times and less pressure with fewer releases.

#### 5.5C Suggestions

In the case of low rank materials such as peat and lignite, a slower curing resin mix and longer impregnation times should be employed for preparation.

#### 5.5D Safety Procedures

##### **1. Using Polyester or Epoxy Resin**

- **organic vapor respirator/safety glasses/rubber gloves must be worn**
- **do not wear contact lenses**
- **avoid skin and eye contact**
- **must be used in a fume hood**
- **laboratory coat and/or apron must be worn**

##### **2. Using the Vacuum Equipment**

- **check oil level in pump before use**
- **remain in room during pump operation**
- **vent air into vacuum chamber slowly**

## 5.6 Freeze Drying

Equipment	analytical balance (sensitivity of 0.01 gram) freeze drying unit jars, lids, filters, filter rings & connectors Drying Book and Data Record Sheets storage containers
Safety Equipment	safety glasses/goggles laboratory apron and/or coat

### 5.6A Preliminary Steps

1. Zero check balance.
2. Label and weigh an empty drying jar (without lid). Record this value.
3. Place a sample into the drying jar being careful not to fill it past the two-thirds mark. Large samples should be split among several jars as necessary.
4. Weigh the filled jar (without lid assembly) and record this value.
5. Secure two filters into the jar lid recess using a filter ring.
6. Insert the large end of a connector into the lid port and snap this assembly onto the sample jar.
7. Place the sample in a freezer and let sit overnight. The sample must be completely frozen when hooked up to the drying unit. See Suggestions, section 5.6C below.

### 5.6B Drying Procedure

1. Remove the sample from the freezer.
2. Insert the free end of its connector into an unoccupied freeze dryer port.
3. Slowly turn the port valve until the indicator ridges on the valve and port are aligned.
4. Allow freeze drying unit to pump back down to a steady state vacuum ( $< 10$  millitorr).
5. Check the freeze drying unit pressure gauge to ensure a return to its original reading. Any drastic increase in pressure could indicate a leak in one or more of the following

interfaces: a) port-connector, b) connector-lid, and c) lid-jar.

6. If a leak is suspected, place a thumb over the valve bleed hole and turn the valve off slowly to release vacuum. Disconnect the jar from the freeze dryer port. Check all seal interfaces noted above and then reattach the jar to unit (steps 2-4 above).
7. Once per day, the sample should be removed from the freeze drying unit and weighed (without the lid). Record this weight.
8. When the change between two consecutive weighings is  $<0.01$  g, the sample is done.
9. Unfinished samples should be reattached to the freeze dryer until drying is completed.
10. Finished samples should be transferred to properly labelled storage containers. Used drying jars and associated parts should be washed with detergent, rinsed with deionized water and then dried using compressed air.

#### 5.6C Suggestions

The use of liquid nitrogen to "flash-freeze" samples may be preferable to other procedures as a method of reducing freezing-related expansion damage to microscopical coal structures. As with all super-cooled materials, liquid nitrogen should only be handled by trained personnel wearing appropriate protective gear.

#### 5.6D Record Keeping

Record data in the Drying Book and on the Data Record Sheets.

#### 5.6E Safety Procedures

- **care should be taken when inserting glass connectors into a lid or unit port**



## 5.7 X-ray Diffraction Pellet Making

Equipment	vacuum pump hydraulic press x-ray pellet mold spatulas spectrographic powder weighing paper acetone disposable wipes marking pen triple beam balance
Safety Equipment	rubber gloves safety glasses/goggles

### 5.7A Preliminary Steps

1. Clean x-ray pellet mold parts with acetone and disposable wipes. Assemble the mold.
2. Obtain the -200 mesh ashed (LTA preferable) sample split.
3. Zero check balance. Weigh out 1.6 g of spectrographic mounting powder.

### 5.7B Forming the XRD Pellet

1. Pour mounting powder into the mold. Shake mold gently to ensure even distribution.
2. Slowly insert the top plunger and tamp the powder down firmly. Cover the vacuum port by hand, withdraw plunger slightly and rotate 1/4 turn. Uncover the vacuum port.
3. Repeat step 2 three more times.
4. After the final tamp down, remove the plunger completely from the mold (being sure to keep the vacuum port sealed off throughout).
5. Carefully sprinkle the -200 mesh sample with a small spatula onto the pressed powder surface. Try to layer as evenly and fully as possible. If the sample is small, cover the central area preferentially. Put on only enough sample to cover the powder surface so that no "white" spots show through.
6. Place the 1/4" top plug and plunger into the sample mold and tamp down very lightly.

7. Turn on the vacuum pump and attach its tygon tubing to the pellet mold vacuum port.
8. Put the mold into a hydraulic press, center and tighten in place. Lower the safety door.
9. Depress and hold in the hydraulic press power button. Engage the pressure valve by rotating it clockwise until tight. Hold sample at 15,000 psi for ten seconds.
10. Release the press power button. Slowly release the system pressure by rotating the pressure valve in a counterclockwise direction.
11. Turn off the vacuum pump. Raise the safety door and remove the mold.
12. Invert the mold and lightly bang it on the floor until the sample and plugs are loosened. Remove and label the sample.
13. Clean all mold parts and spatulas with acetone and disposable wipes.

#### 5.7C Record Keeping

Record that an XRD pellet was made in the Sample Status Book.

#### 5.7D Safety Procedures

##### **1. Using the Pressing and Vacuum Equipment**

- **safety glasses/goggles must be worn**
- **check vacuum pump oil level before use**
- **press safety door must be down during operation**
- **always release pressure slowly**

##### **2. Using Acetone**

- **safety glasses/goggles and rubber gloves must be worn**
- **avoid breathing fumes, use in fume hood if possible**
- **avoid skin and eye contact**

6.0 SAMPLE POLISHING

6.1 Pellet and Microblock Polishing (Cole & Berry, 1965; ASTM D2797-85[1990], 1992c)

Equipment . . . . .	automatic polishing machine polishing cloths (various naps) 600 grit paper 15 micron diamond platen alumina and silica polishes sample holder and leveler ultrasonic cleaner and liquid Sample Status and Pellet/Microblock Books
Safety Equipment . . . . .	safety glasses/goggles laboratory apron and/or coat dust mask

6.1A Polishing Pellet Tops

1. Mount 10 pellets into a pellet holder with the epoxy side down (Note: If polishing less than 10 pellets, insert blanks into any remaining holes).
2. Level and tighten.
3. Place on the 15 micron diamond platen and grind (program 01, step 01) until flat. Use a steady stream of water. If necessary, repeat this step using 600 grit paper.
4. Rinse the samples in tap water.
5. Rough polish on a short-napped cloth (e.g., Texmet [see disclaimer]) with 1.0 micron aluminum oxide polishing compound (program 01/step 03).
6. Rinse the samples in tap water.

6.1B Polishing Sample Bottoms

1. After polishing the tops of the pellets, relevel the pellets with the coal side down.
2. Grind on the 15 micron diamond platen (program 01, step 01) until flat. A brownish slurry should occur.

3. Rinse the samples with tap water; then clean them ultrasonically for 2 minutes and rinse them again.
4. Final grind on 600 grit paper (program 01, step 02). A brownish slurry should occur.
5. Rinse samples with tap water, clean them ultrasonically for two minutes, and then rinse them with tap water again.
6. Rough polish on a short-napped cloth (e.g., Texmet [see disclaimer]) with 1.0 micron aluminum oxide polish (program 01, step 03). A brownish slurry should occur.
7. Rough buff on a short-napped cloth (e.g., Texmet [see disclaimer]) to remove excess polishing compound and any loose particles (program 01, step 04).
8. Rinse samples with distilled water, clean ultrasonically for two minutes, and then rinse them with distilled water again.
9. Final polish on a medium-napped cloth (e.g., Mastertex [see disclaimer]) with 0.06 micron colloidal silica polishing compound (program 01, step 05). A light colored slurry should occur.
10. Final buff on a medium-napped cloth (e.g., Mastertex [see disclaimer]) to remove excess polishing compound and any loose particles (program 01, step 06).
11. Rinse samples with distilled water, clean ultrasonically for two minutes, and then rinse them with distilled water again.
12. Blow dry and place the pellets in a desiccator. (Note: If the samples are low-rank and not thoroughly impregnated, **do not blow dry** ..... pat dry and store in a sealed humidity box to prevent desiccation and cracking.)

#### 6.1C Record Keeping

1. Record in the Pellet Book or Microblock Book that the sample was polished.
2. Note in the Sample Status Book that the sample was polished.

#### 6.1D Suggestions

1. Grinding times will vary for microsamples depending on the sample thickness.

2. Increased polishing times may be needed for some hard coals. Coals of differing hardness should not be polished together as this will result in a scratchy, uneven polish on the softer materials.
3. If microprobe or SEM analyses are to be conducted on the samples prepared, deionized water must be substituted for tap water and new polishing cloths must also be used.
4. Modified polishing technique for problem coals (weathered/high-clay/high-calcite)
  - a. Grind samples as usual (15 micron diamond platen followed by 600 grit paper).
  - b. Rough Polish 1: 5.0 micron alumina on short-napped paper.
  - c. Rough Polish 2: 1.0 micron alumina on short-napped paper.
  - d. Final Polish: 0.06 micron amorphous silica on medium-napped paper.
  - e. Adjust polishing times to balance the amount of relief versus polish quality.
5. Directions for Automet automated polishing system [see disclaimer]
  - a. Ecomet unit power must be on for the Automet power to come on. When the Automet power button is pressed, the indicator LED should blink four times --- continuous blinking indicates a problem.
  - b. The following items are located on the back of the Ecomet unit: water metering valve (regulates dispensing arm and bowl flush jet), Automet fuse post, and Ecomet main fuse switch.
  - c. The Ecomet motor has a default setting of 50 rpm.
  - d. The bowl flush jet water flow is regulated only by the water metering valve.
  - e. To properly emplace a platen, align the base plate pins with the platen holes. When engaged, press the platen down to create a friction fit.
  - f. The specimen holder (when attached to the Automet mounting post) should not interfere with the splash ring or paper holder. Test positioning by partially lowering the specimen holder. If interference appears imminent, press the Stop button and reposition.
  - g. Choose either the MAN or the AUTO operation mode.
  - h. Enter operational parameters: time, force, fluids, head rotation direction, speed.

- i. If in the AUTO mode, press the "Memory Enter" button to save entered parameters. AUTO LED indicator will blink to show that the step was saved. Up to 100 ten-step programs can be saved into the permanent memory. Press the "Memory Increase" button to select the next step of the program.
- j. If in the MAN mode, continue.
- k. Rapid Advance to Program/Step option: choose MAN, use the MEM INCR button to get to the desired program/step, press the AUTO button and continue.
- l. Parameters can be changed and used, but won't be saved unless the ENTER button has been pressed.
- m. The two start buttons on either side of the Automet head must be pressed in simultaneously *and held* until the CYCLE RUN LED stops blinking and remains on. When the buttons are pressed, all other functions (water flow, platen rotation, etc.) should begin.
- n. The Automet head will retract when the cycle has ended or if the CYCLE STOP button is pressed.
- o. There is no need to relevel pellets between grinding steps.
- p. When using non-adhesive backed grinding papers, the platen should be dampened slightly with water to provide a friction fit for the paper. A paper holding collar should also be used.
- q. After use, all parts of the Automet/Ecomet system (frame, bowl, splash cover, platen base, main unit, etc.) should be completely wiped down. The main bowl and drainage system should be flushed until the discharge water is clear. Grinding and polishing platens should have their metal surfaces wiped clean and then be set out to dry. After drying, the platens should be returned to their storage drawers to protect them from dust. The diamond platen should be stored as part of the main unit.
- r. The Automet memory unit can save up to 100 ten-step programs. Programs 1-9 are reserved for the laboratory's standard polishing regimes and should not be altered in any way. Program slots 10-100 are available for general use.

## 6. Standard Polishing Parameters (Program 01)

Step 01 (diamond platen grind): 2 minutes, 40 lbs, water on, clockwise rotation, 200 rpm.

Step 02 (600 grit grind): 2 minutes, 40 lbs, water on, clockwise rotation, 200 rpm.

Step 03 (initial polish): 2 minutes, 40 lbs, water on (no armflow), clockwise rotation, 200 rpm.

Step 04 (initial buff): 30 seconds, 40 lbs, water on (no armflow), clockwise rotation, 250 rpm.

Step 05 (final polish): 2 minutes, 40 lbs, water on (no armflow), clockwise rotation, 200 rpm.

Step 06 (final buff): 30 seconds, 40 lbs, water on (no armflow), clockwise rotation, 250 rpm.

## **6.1E Safety Procedures**

### **1. Using the Automatic Polishing Machine**

- **safety glasses/goggles must be worn**
- **keep fingers and clothing away from all moving parts**
- **do not insert fingers under machine when in motion**
- **make sure unit is securely seated and that the Automet head is locked into position before beginning the polishing cycle**

### **2. Using the Ultrasonic Cleaner**

- **keep fingers out of the cleaner while it is on**

### **3. Using Colloidal Silica Polishing Compound**

- **rubber gloves and safety glasses/goggles must be worn**
- **avoid skin and eye contact (basic solution)**
- **after use, the dried platen must be stored in a sealed bag in order to avoid the creation of a hazardous dust problem**

## 6.2 Block Polishing

Equipment	assorted grit papers alumina and silica polishes automatic polishing machine assorted polishing cloths 15 micron diamond wheel sample holder and leveler ultrasonic cleaner and liquid Sample Status Book Microblock Book
Safety Equipment	safety glasses/goggles laboratory apron or coat

### 6.2A Polishing Cast Blocks

1. Mount 3 blocks in the block holder with the sample side down (Note: if polishing less than 3 blocks, insert epoxy blanks into the remaining holes).
2. Level and tighten.
3. Unless otherwise stated, all block processing operations follow program 01/step 01 (see Standard Polishing Parameters, 6.1D-6).
4. Rough grind the samples on a 15 micron diamond wheel to remove any sharp edges.
5. Rinse the samples in tap water.
6. First intermediate grind with 240 grit silicon carbide paper until the samples are exposed. (Note: This step may be omitted or shortened if the samples are fully exposed.) A brownish slurry should occur.
7. Rinse the samples in tap water.
8. Second intermediate grind with 400 grit silicon carbide paper. (Note: This step may be omitted/shortened if samples are fully exposed.) A brownish slurry should occur.
9. Rinse the samples in tap water.
10. Final grind with 600 grit silicon carbide paper. A brownish slurry should occur.
11. Rinse the samples in tap water; and then clean them ultrasonically for 2 minutes.



12. Rough polish on a short-napped cloth (e.g., Texmet [see disclaimer]) with 1.0 micron aluminum oxide polishing (program 01, step 03). A brownish slurry should occur.
13. Rough buff on a short-napped cloth (e.g., Texmet [see disclaimer]) to remove excess polishing compound and any loose particles (program 01, step 04).
14. Rinse the samples with distilled water; clean them ultrasonically for two minutes and then rinse them with distilled water again.
15. Final polish on a medium-napped cloth (e.g., Mastertex [see disclaimer]) with an 0.06 micron amorphous silica polishing compound (program 01, step 05). A light colored slurry should occur.
16. Final buff on a medium-napped cloth (e.g., Mastertex [see disclaimer]) to remove excess polishing compound and any loose particles (program 01, step 06).
17. Rinse the samples with distilled water. Clean them ultrasonically for two minutes and then rinse them with distilled water again.
18. Blow dry the samples and place them in a desiccator. (Note: If samples are low-rank and are not thoroughly impregnated, **do not blow dry** .... pat dry and store in a sealed humidity box instead to prevent desiccation and cracking.)

#### 6.2B Record Keeping

Record in the Sample Status and Microblock books that the sample was polished.

#### 6.2C Suggestions

1. Grinding time at each step may vary considerably; the sample size and hardness will determine the amount of time needed.
2. See section 6.1D-5 for automatic polisher operation directions and parameters.
3. If microprobe/SEM analyses are to be done on the sample, deionized water must be substituted for tap water in all steps and new cloths must be put on the platens.

## **6.2D Safety Procedures**

### **1. Using the Automatic Polishing Machine**

- **safety glasses/goggles must be worn**
- **keep fingers and clothing away from moving parts**
- **do not insert fingers under machine when in motion**
- **make sure that the unit is securely seated and that the Automet head is locked into position before beginning the polishing cycle**

### **2. Using the Ultrasonic Cleaner**

- **keep fingers out of the cleaner while it is on**

### **3. Using Amorphous Silica Polishing Suspension**

- **rubber gloves and safety glasses/goggles must be worn**
- **avoid skin and eye contact (basic solution)**
- **after use, the dry platen must be stored in a sealed bag to avoid the creation of a hazardous dust problem**

## 7.0 SAMPLE ETCHING

Equipment	50, 100, 500 ml beakers glass stirring rod hot plate and watch glasses 25 ml graduated cylinder 100 ml graduated cylinder 47% sulfuric acid ( $\text{H}_2\text{SO}_4$ ) potassium permanganate ( $\text{KMnO}_4$ ) sodium sulfite ( $\text{Na}_2\text{SO}_3$ ) chromium trioxide ( $\text{CrO}_3$ ) ultrasonic cleaner and liquid ceramic stirring bar
Safety Equipment	safety glasses/goggles laboratory apron and coat rubber gloves organic vapor respirator

### 7.1 Lignite, Subbituminous and Bituminous Coals (modified from Stach, 1982)

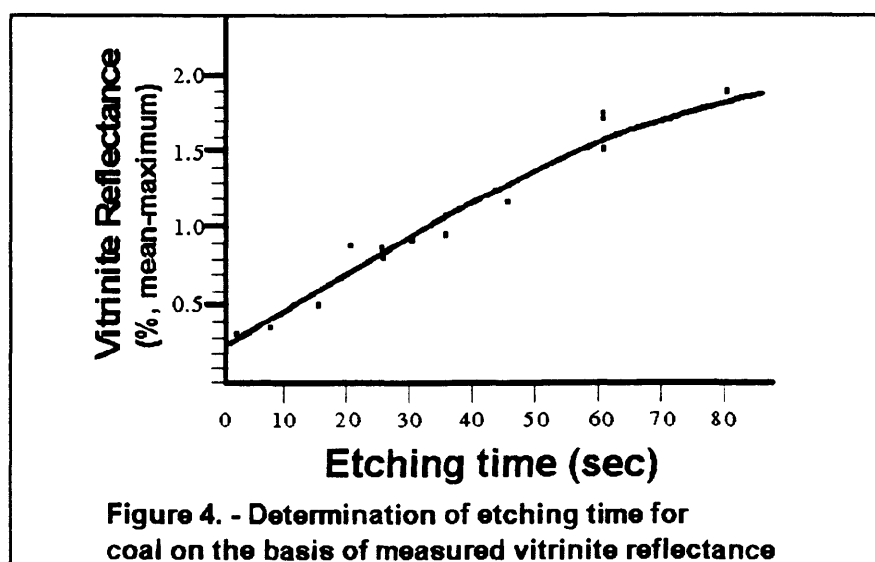
#### 7.1A Preliminary Steps

1. Mix an etching solution of 100ml water + 25g  $\text{KMnO}_4$  + 5ml  $\text{H}_2\text{SO}_4$ .
2. Mix a rinsing solution of 100ml water + 25g  $\text{Na}_2\text{SO}_3$  + 5ml  $\text{H}_2\text{SO}_4$ .
3. Stir the rinsing solution until all of the  $\text{Na}_2\text{SO}_3$  has dissolved.
4. Heat the etching solution in a water bath; boil until nearly all of the  $\text{KMnO}_4$  has dissolved.
5. Cover a small section of each pellet to be etched with a strip of scotch tape so that a portion will remain unetched for comparison purposes.

#### 7.1B Etching Procedure

1. If a dilution of the etching solution is necessary (see 7.1D-4), the solution must be returned to a boil before any etching is attempted.

2. Immediately pour part of the etching solution into a watch glass.
3. Submerge the partially masked face of a polished pellet into the decanted etching solution. Agitate pellet to dispel any air bubbles trapped beneath the pellet surface.
4. The recommended etching time for any particular sample will vary as a function of its rank, degree of weathering and the etching solution strength. See section 7.1D.
5. Remove pellet and rinse it immediately with water to remove excess etching solution.
6. Submerge the partially masked pellet face into the rinsing solution for one minute to remove all traces of purplish permanganate stain.
7. Clean the pellet ultrasonically in water for one minute.
8. Remove the tape and any residues left behind with acetone or methanol.



#### 7.1C Record Keeping

See section 7.2C.

### 7.1D Suggestions

1. The relationship between etching time and mean max vitrinite reflectance ( $\bar{R}_{o\ max}$ ) is essentially linear for reflectance values up to 1.90 (the boundary between low-vol bituminous and semi-anthracite).
2. The necessary etching time can either be estimated from Figure 4 on page 39 (Stanton & Pontolillo, unpub. data) or directly calculated using the equation :  $Y = (46.450)X - 11.549$ , where Y is etching time and X is mean max vitrinite reflectance ( $\bar{R}_{o\ max}$ ) value.
3. Without exception, weathered coals require significantly reduced etching times than would be expected from their mean-max vitrinite reflectance. Additionally, differential weathering will result in differential etching. Several attempts may be needed to achieve an optimal etching result. The samples will need to be repolished after each unsuccessful attempt.
4. When preparing low-rank coal samples, it may be necessary to dilute the etching solution with water and thereby increase the etching time proportionately. For example, the theoretical etching time required for a lignite with  $\bar{R}_{o\ max} = 0.30$  is 2.4 seconds --- hardly a replicable parameter. Diluting the etchant to a 50% solution will yield a much more usable parameter of approximately five seconds.

### 7.1E Safety Procedures

See section 7.2E.

## **7.2 Anthracites and Semi-Anthracites (modified from Seyler & Edwards, 1929)**

### 7.2A Preliminary Steps

1. Prepare a saturated stock solution of chromic acid by dissolving 50.82g of chromium trioxide ( $CrO_3$ ) in 30ml of distilled water.
2. Prepare an etching solution consisting of 10ml concentrated (47%) sulfuric acid ( $H_2SO_4$ ) and 30ml of the stock chromic acid solution. Add enough distilled water to redissolve any chromium trioxide that precipitates out.
3. Mask a small portion of the sample if desired; be forewarned: only certain tapes can withstand this etching solution. Some prior testing on dummy samples is advisable.
4. Warm the sample in an oven at 60°C (or 100°C if the sample is unmasked).

### 7.2B Etching Procedure

1. Boil the etching solution in a water bath until separation begins (i.e., chromium trioxide begins to precipitate out of solution).
2. Immerse the sample face into the etching solution and agitate slightly to disperse any trapped air bubbles. Keep the solution at a light boil throughout the etching process. Add further deionized water or etching solution as necessary.
3. The recommended etching time for any particular sample will vary with its rank and degree of weathering. See section 7.2D.
4. Remove the sample, rinse in deionized water and then ultrasonically clean for one minute. Blow dry with compressed air.
5. Remove the tape and any residues left behind with acetone or methanol.
6. The etching solution may be reused for an extended period of time. Simply add enough distilled water to put any precipitates back into solution and reheat in a water bath to the boiling point.

### 7.2C Record Keeping

1. Record in the Pellet Book that the sample was etched.
2. Record in the Etching Book the etching parameters and results (i.e., solution strength, etch time, good/bad etch).

### 7.2D Suggestions

A satisfactory relationship between rank and etching time (as exists for lower-rank coals) has yet to be resolved for anthracites and semi-anthracites. Any attempt to etch these materials will be purely experimental. In several cases, anthracites of similar rank, but different provenance, required considerably different etching times. The extent to which weathering may affect this etching process has yet to be determined.

## **7.2E Safety Procedures**

### **1. Handling Chemicals**

- all chemicals must be handled in a fume hood
- heavy rubber gloves, laboratory coat and rubber apron must be worn when handling chemicals and when etching is performed
- safety glasses/goggles and an organic vapor respirator must be worn
- avoid skin/eye contact with all chemicals
- when mixing solutions, always add acid to water

### **2. Using the Hot Plate and Water Bath**

- use in a fume hood
- keep bulk chemicals/flammables away from heat
- place the hot plate on a level surface

### **3. Using the Ultrasonic Cleaner**

- when the ultrasonic cleaner is on, keep fingers out

## 8.0 SAMPLE ASHING

### 8.1 Low Temperature Ashing (LTA)

Equipment . . . . .	plasma asher petri dishes analytical balance accurate to 0.0001 g mortar and pestle 100 mesh sieve screen oven capable of maintaining 105°C desiccator with drying agent oxygen tank 1 N ammonium acetate (pH 7) LTA, Values and Sample Status Books sample bottles cleaning solution (see below) small spatula
Safety Equipment . . . . .	safety glasses/goggles laboratory apron or coat organic vapor respirator acid-resistant gloves

#### 8.1A Preliminary Steps

1. Obtain the -60 mesh split marked for LTA.
2. Pulverize the sample with a mortar and pestle, and screen to ensure passing -100 mesh.
3. Place 1-2 grams of the ground sample into a clean, preweighed and recorded petri dish (see section 8.1E-5). Zero check balance. Weigh and record.
4. Dry the sample in an oven at 105°C for at least one hour (preferably overnight).
5. Zero check balance. Weigh the sample dish and record.
6. Calculate % moisture lost and record in LTA book. If the sample is a lignite or subbituminous coal, mix it for 1-24 hours (depending on the coal) with 1N ammonium acetate. Then reweigh the sample to determine the weight loss.



### 8.1B Ashing Procedure

1. Place the sample in the LTA ashing chamber.
2. Close the chamber door; make sure that the gasket is properly centered.
3. Close the main unit door.
4. Turn on the vacuum pump. Push the asher ON button.
5. Once the unit has evacuated to 1mm Hg (1 torr) or less, push the RF button. Note: a slight rise in pressure may occur.
6. Switch the RF selector to the Reflected/10 scale. RF meter reading should drop to zero within 10-15 seconds. If this does not occur, see section 8.1E-4.
7. When a reflected value of approximately zero is obtained, switch the RF selector as follows and verify proper power readings: 2-chamber unit (Forward scale 70 watts); 4-chamber unit (Forward scale 150 watts).
8. Adjust the RF power with designated knob as required.
9. Turn up the oxygen flow into the range of 15-20 cc/minute (0.75 - 1.00 arbitrary units) --- watch indicator ball. Note: higher flowrates may cause difficulties in maintaining a proper vacuum.
10. The ashing chamber should exhibit a pale blue plasma. As ashing progresses, the plasma color will slowly change, becoming violet when the sample is fully ashed.

### 8.1C Stirring and Weighing Samples

1. Samples should be weighed and stirred once every eight hours.
2. Push the RF button OFF. Note: the power reading should drop to zero.
3. Push the OFF button. Note: the gauge will show a quick rise to atmospheric pressure.
4. Turn off the oxygen flow.
5. Once the ashing chamber pressure has equalized, remove the sample to a desiccator and let sit for 20 minutes to reach room temperature.
6. Zero check balance. Weigh the sample and record value.

7. Stir the sample to expose any unoxidized material. This may be done with a spatula or by gently shaking the sample dish.
8. A sample is done when its weight changes by less than 0.1% of the original dry sample weight or when it begins to gain weight (by absorbing moisture).
9. Return any uncompleted samples to the LTA unit chamber for additional ashing.
10. Finished samples should be transferred to properly labelled specimen bottles. The sample weight should be noted in the LTA book.

#### 8.1D Record Keeping

1. Record % moisture in the LTA Book.
2. Record final ash % in the LTA and Values Books.
3. Record in the Sample Status Book that LTA has been done.

#### 8.1E Suggestions

1. Chambers must not be left empty when running an ashing unit. Arrange the samples accordingly if possible, otherwise dummy samples (i.e., petri dish with sand) should be employed as needed.
2. The asher should be checked occasionally for any problems (i.e., power and vacuum readings) especially when new samples are being started up.
3. If an ashing unit is off from a power outage be sure to press the RF button OFF before attempting a restart of the ashing process.
4. When the RF Reflected/10 scale does not drop to zero, the following guidelines apply:  
A) If the power reading varies by  $< 1.00$  watt, turn the C1 and C2 screws very slowly to rezero. B) If the power reading varies by  $> 1.00$  watt, then the asher's automatic tuner needs adjustment. Open the asher top lid. Inside and to the left sits the automatic tuning cylinder ridden by a tracking wheel. Careful taps to the tracking wheel switches (black with little flags sticking up) will cause the tracking wheel to change position. This should reduce the RF Reflected. If one-half hour of "fiddling around" fails to reduce the reflected power, shut the unit down to avoid damage. **AT ALL TIMES CARE MUST BE TAKEN TO TOUCH ONLY THE TRACKING WHEEL SWITCHES!**

5. Petri dishes to be used in the LTA process should be prepared ahead of time as follows:

- a) Wash (alconox in deionized water), rinse and set the dishes out to dry.
- b) Soak the dishes overnight in a cleaning solution of 900ml concentrated (47%) sulfuric acid and 100ml saturated potassium dichromate solution.
- c) Remove the dishes from the cleaning solution and rinse thoroughly at least six times with deionized water.
- d) Place the dishes in an oven at 105°C to dry for two hours. Transfer the dishes to a desiccator to cool.
- e) Zero check balance. Weigh the dishes and record on Data Sheet. Store the dishes in a desiccator until needed.

6. The vacuum pump oil must be changed after every two weeks of continuous use.

7. Whenever changing oxygen tanks, the proper procedure is as follows:

- a) Close the tank valve.
- b) Uncouple the tygon tubing from the needle valve spigot.
- c) Evacuate oxygen until both gauges read zero.
- d) Close the needle valve (turn counterclockwise until fully out).
- e) Loosen the gauge nut and remove the gauges.
- f) Clean the valve cup of the new tank with a damp cloth.
- g) Standing to one side, crack the nozzle open to blow clear any remaining debris.
- h) Close the nozzle and install the gauges onto the new tank.
- i) Wiggle the gauges while tightening replaced gauge nut to insure that they are properly seated. Tighten firmly.
- j) Open the tank valve slowly until the tank gauge reads 2500 psi. Listen for leaks; if any, repeat steps a through i.
- k) Cover the needle valve spigot by hand and then open the needle valve slowly - -- turn clockwise.

- l) Use thumb on/off method and adjustments of the needle valve to obtain two consecutive readings of 2 psi on the needle valve gauge.
- m) Couple the tygon tubing to the needle valve spigot.

#### **8.1F Safety Procedures**

- secure the oxygen tank with a chain and holder to a wall
- ammonium acetate should be used in a fume hood and requires the use of safety glasses, rubber gloves, lab coat, and an organic vapor respirator

## 8.2 High Temperature Ashing [HTA] (ASTM D3174-89, 1992b)

Equipment	muffle furnace 17 ml porcelain crucibles analytical balance accurate to 0.0001 g desiccator with drying agent oven capable of 105°C spatula tongs HTA and Sample Status Books
Safety Equipment	safety glasses/goggles heat resistant gloves laboratory coat

### 8.2A Preliminary Steps

1. Obtain the -60 mesh split marked for HTA.
2. Place the sample into a petri dish and then in an oven for one hour at 105°C.
3. Place a clean crucible into an oven with the sample.
4. After an hour remove both the sample and crucible; place them in a desiccator and let them cool to room temperature.
5. Zero check balance. Weigh the crucible and record in HTA book as dish weight (D).
6. Place one gram of sample into the crucible and record as dish and sample weight (DS).

### 8.2B Ashing the Sample

1. Place the sample into a room temperature furnace and turn on furnace fan.
2. Heat the furnace to 500°C within the first hour (8°C/min).
3. Heat the furnace to 750°C in the second hour (4°C/min) and maintain for 60 minutes.
4. Turn off the furnace and let it cool to 400°C.
5. Remove the sample with tongs and place in a desiccator. Make sure that there is nothing flammable in the desiccator ahead of time.

6. Let the sample cool to room temperature (about 8-12 minutes).
7. Zero check balance, weigh sample, and record in HTA book as dish + ash weight (DA).
8. Calculate the ash value:  $(DA-D/DS-D) \times 100 = \% \text{ Ash}$

#### 8.2C Record Keeping

1. Record in HTA book all the weights measured and % Ash.
2. Note in the Sample Status Book that HTA was performed.

#### 8.2D Suggestions

1. Input for Programmable Furnace

- Initial Rate (1st hour) . . . 8°C/min.
- Transition temperature . . . 500°C
- Hold time . . . . . 0 min.
- Final rate (2nd hour) . . . . 4°C/min.
- Final temperature . . . . . 750°C
- Final hold time . . . . . 60 min.

2. Repeatability (%)

- In Lab:
  - No carbonates present . . . 0.2
  - Carbonates present . . . . 0.3
  - Coals > 12% ash with  
carbonate and pyrite . . 0.5
- Between Labs:
  - No carbonates present . . . 0.3
  - Carbonates present . . . . 0.5
  - Coals > 12% ash with  
carbonate and pyrite . . 1.0

#### 8.2E Safety Procedures

- **heat resistant gloves must be worn when removing samples from the oven**

## 9.0 SAMPLE ANALYSES

### 9.1 X-Ray Radiography

Equipment . . . . . X-ray radiography unit  
VCR and monitor  
masking tape  
meter stick with lead markings  
lead number/letter ID set  
blank VCR tape  
X-ray logbook  
marking pen  
X-ray film and film cassette  
instant developing unit

Safety Equipment . . . . . radiation badge and ring

#### 9.1A X-raying onto Videotape

##### 9.1A.1 Preliminary Steps

- a. Insert key and turn on the X-ray unit. Turn on the VCR, insert a tape and reset the VCR tape counter to zero.
- b. Depress and hold down the X-ray ON button (and either the FWD or REV button) for one minute to properly calibrate the system. Release the button to turn X-rays off.
- c. Two indicators that X-rays are being produced: 1) the red warning light on unit top is lit and 2) the peak tube current meter shows a reading greater than zero. **NEVER** change samples, open unit doors, or lift lead curtains when X-rays are being produced.
- d. Open the left rear unit access door and set the kV dial at desired value. Standard for coals is 90% (63% for peats; lower values enhance the resolution of soft tissues). Record this setting in the X-ray logbook.
- e. Affix a leaded meter stick lengthwise onto the X-ray conveyor belt so that it will appear in the top edge of the monitor screen during taping.

#### 9.1A.2 X-raying Procedure

- a. Place a sample (core, block, etc.) lengthwise onto the conveyor belt and aligned alongside of the meter stick.
- b. Prepare a leaded direction arrow (to indicate sample orientation) and I.D. card. Affix both onto the conveyor belt in front of the sample.
- c. Set the sample feed dial on the righthand side of the console to the "S" position. This will give a size/speed ratio of approximately 1:1.
- d. Put the VCR into taping mode (i.e., press PLAY/RECORD and then PAUSE).
- e. Press the X-ray ON, conveyor REV and VCR PAUSE buttons to begin sample scanning/taping. Record beginning tape counter number in X-ray logbook.
- f. Once the entire sample has been scanned, press the X-ray ON, conveyor STOP and VCR PAUSE buttons in order to end scanning/taping. Record ending tape counter number in the X-ray logbook.
- g. Press the conveyor FWD button to back the sample up to its original position. Stop.
- h. Set the sample feed dial to the "F" position. This will give a size/speed ratio of approximately 1:4. Record beginning tape counter number in the X-ray logbook.
- i. Press the X-Ray ON, conveyor REV and VCR PAUSE buttons to resume scanning/taping.
- j. When the sample is centered on the monitor screen, press the conveyor STOP button and record this stationary image for 10-12 seconds.
- k. Press the X-ray ON and VCR PAUSE buttons to conclude sample scanning/taping. Record ending tape counter number in the X-ray logbook.
- l. Press the conveyor FWD button to back the sample out to its original position. Remove the sample and I.D. card from the conveyor belt.
- m. Repeat steps a through l for each remaining sample.

#### 9.1A.3 Record Keeping and 9.1A.4 Safety Procedures

See sections 9.1B.3 and 9.1B.4.



## 9.1B X-raying onto Radiographic Film

### 9.1B.1 Preliminary Steps

See section 9.1A.1.

### 9.1B.2 X-raying Procedure

- a. Load an 8x10 X-radiographic negative into the film cassette. Tape a lead orientation arrow onto the topside of the cassette casing.
- b. Place a sample on top of the film cassette making sure that it is inside the guides on the cassette.
- c. Place the cassette onto the X-ray conveyor belt inside of the lead curtains.
- d. Set the sample feed dial on the righthand side of the console to the "S" position.
- e. Press the X-ray ON and Conveyor REV buttons to begin sample exposure.
- f. Once the entire sample has been exposed, press the X-ray ON and conveyor STOP buttons to end scanning.
- g. Press the X-ray ON and Conveyor FWD buttons to begin a second sample exposure.
- h. Once the entire sample has been exposed, press the X-ray ON and conveyor STOP buttons to end scanning. The sample has now been returned to its original position and has been exposed twice.
- i. The number of cumulative exposures required to obtain a usable x-ray image will vary depending on the thickness and composition of the sample material. Bituminous coal of 0.5" thickness requires 10 exposures, for example.
- j. Once the necessary number of exposures have been made, remove the film cassette from the X-ray conveyor belt. Carefully remove the sample from the cassette surface and process the film cassette through an instant developing unit according to the manufacturer's directions.
- k. After the developed film has received a protective coating and dried completely, write the sample I.D. and any other desired information onto the film surface with an indelible marker.

#### **9.1B.3 Record Keeping**

Date, time of session, power level (kV), sample I.D., and tape counter numbers (when applicable) all need to be entered into the X-ray Logbook.

#### **9.1B.4 Safety Procedures**

- X-ray badge and ring must be worn at all times
- Never open unit doors/disturb lead curtains while X-rays are being generated
- X-ray unit should be inspected regularly for leaks
- Always clear the X-ray conveyor belt before turning the unit on

## 9.2 Vitrinite Reflectance (ASTM 2798-91, 1992e)

Equipment . . . . . 500X reflected light petrographic microscope  
photomultiplier with peak picker  
rotating circular stage  
immersion oil (non-drying, refract. index 1.515-1.519)  
sample leveling press  
modeling clay  
reflectance calibration standards  
2" x 1" glass slides

### 9.2A Setting Up the Microscope (Numbers refer to Figure 5)

1. Turn on the large and small digital voltmeters (1 & 2), lamp power supply (3), high voltage power supply (4) and stable [DC] power supply (5).
2. Increase the lamp power supply to maximum (6) and the stable [DC] power supply to 8.25 amps (7).
3. Switch on the peak picker (8).
4. Install oil objective lens type 50X.85p oel (9).
5. Push out the half stop (10).
6. Push in the slide; the number "8" should be displayed (11).
7. Pull out MPV 2 (12).
8. Switch the condensor out by lining up its indicator knob and reference line (13).
9. Check to see that the spectral wedge is out (14). The dial arrow should be lined up with the red indicator dot.
10. Place the switch towards the illustrated hand (15).
11. Place the switch down for through-the-eyepiece viewing during metering (16).
12. Push the red lighted button to engage the high voltage power supply (17).
13. Place the reflectance calibration standard under the objective and pick a reference glass using the coordinates from the calibration matrix (see Figure 6).

14. Engage the measuring square (push up lever 18).
15. While watching the objective and calibration standard, focus down until the two become very close.
16. Looking through the eyepiece, focus until the measuring square is sharp and defined.
17. Check the voltmeters to see that both read zero. If not, switch scale on the large voltmeter (to eliminate any static charge buildup). Adjust the dark current (19) if necessary.
18. Engage filter S542-21 (20).
19. Engage the photometer by pressing the foot pedal -- a red light should come on (21).
20. Let the photometer warm up and stabilize for at least 20-30 minutes.

#### 9.2B Centering and Calibrating the Microscope

1. Turn off any overhead lights.
2. Remove the calibration standard. Level and focus a sample.
3. Focus on the sample and engage the measuring square. Both the sample and measuring square should be sharply in focus. If not, adjust the focusing square (22).
4. To center the objective, place the cross-hair over a small object (e.g., pyrite, micrinite). Rotate the stage and note the travel path of the object selected. Correct centering with objective wrenches.
5. To calibrate the photometer:
  - a. Place the calibration standard under the objective. Focus as previously described.
  - b. Move to a reference glass using coordinates from the calibration matrix (see Figure 6, for example).
  - c. Clear the peak picker by pressing in the track button (light will blink) and then releasing.
  - d. Rotate the stage. If the voltage does not agree with the calibration matrix value, then adjust the voltage (23).

- e. Clear the peak picker and rotate the stage again. Continue this process (steps d & e) until the photometer is calibrated to the reference glass.
- f. Move to another reference glass and calibrate as per steps c-e.
- g. Return to the first reference glass and check the reflectance reading. If no adjustment is necessary, then the photometer is calibrated. If adjustment is needed, then repeat steps b-g until both glasses are calibrated.

### 9.2C Measuring Vitrinite Reflectance

1. Place a leveled sample under the microscope and focus.
2. Systematically transect the pellet measuring only the structureless vitrinites (telocollinite) that are scratch-free and pyrite-free.
3. Whenever a particle of telocollinite is found, center on a scratchless area.
4. Focus sharply and engage the measuring square.
5. Clear the peak picker and rotate the stage.
6. Record maximum reflectance ( $R_{o_{max}}$ ). Note: watch the voltmeter readings --- jerky stage motion can cause inflated values.
7. Repeat measurements until 25 counts are recorded. Check calibration. Measure another 25 particles on this mount and then check the calibration again.
8. Measure 50 counts on the next pellet for a total of 100 counts for one sample.

### 9.2D Preparation of Calibration Matrix

1. Set up the microscope as previously described in section 9.2A.
2. Place the glass calibration standard on the stage.
3. Center the objective onto the first glass and record the stage position coordinates.
4. Clear the peak picker and rotate the stage.
5. Record the reflectance value. Adjust the voltage until the calculated value of glass no.1 is read as the peak value.

6. Read and record the position and peak value of all other glasses without any adjustment of voltage.
7. Enter each value into row 1 of the calibration matrix (see Figure 6).
8. Return to the coordinates of glass no.2 and adjust voltage until the calculated value of glass no.2 is read as the peak value.
9. Repeat step 7, above, and enter the measured values into the proper row of the calibration matrix.
10. For each of the remaining calibration glasses, repeat steps 3 - 8.
11. Throw out any highly inconsistent readings (those with a difference greater than 0.2%). On this basis, glass 5 in Figure 6 should be discounted as a reliable calibration glass.
12. Average all other readings, on a glass by glass basis, except for the adjusted reading (underlined in Figure 6). The average will be the calibration value to be used at the specified coordinate of each glass.

#### 9.2E Record Keeping

1. Record data in a histogram format on an appropriate vitrinite reflectance data sheet.
2. Calculate and report the mean maximum reflectance and mean standard deviation for each set of sample measurements.

#### 9.2F Suggestions

In some circumstances, mean random reflectance ( $\bar{R}_{o, \text{ran}}$ ) measurements may be preferred. The methodology is the same as for mean maximum reflectance measurements with the following exceptions: 1) No stage rotation occurs during the measurement process; 2) The microscope polarizer must be removed for the duration of all random measurements; 3) The microscope will need to be recalibrated prior to and after taking random measurements.

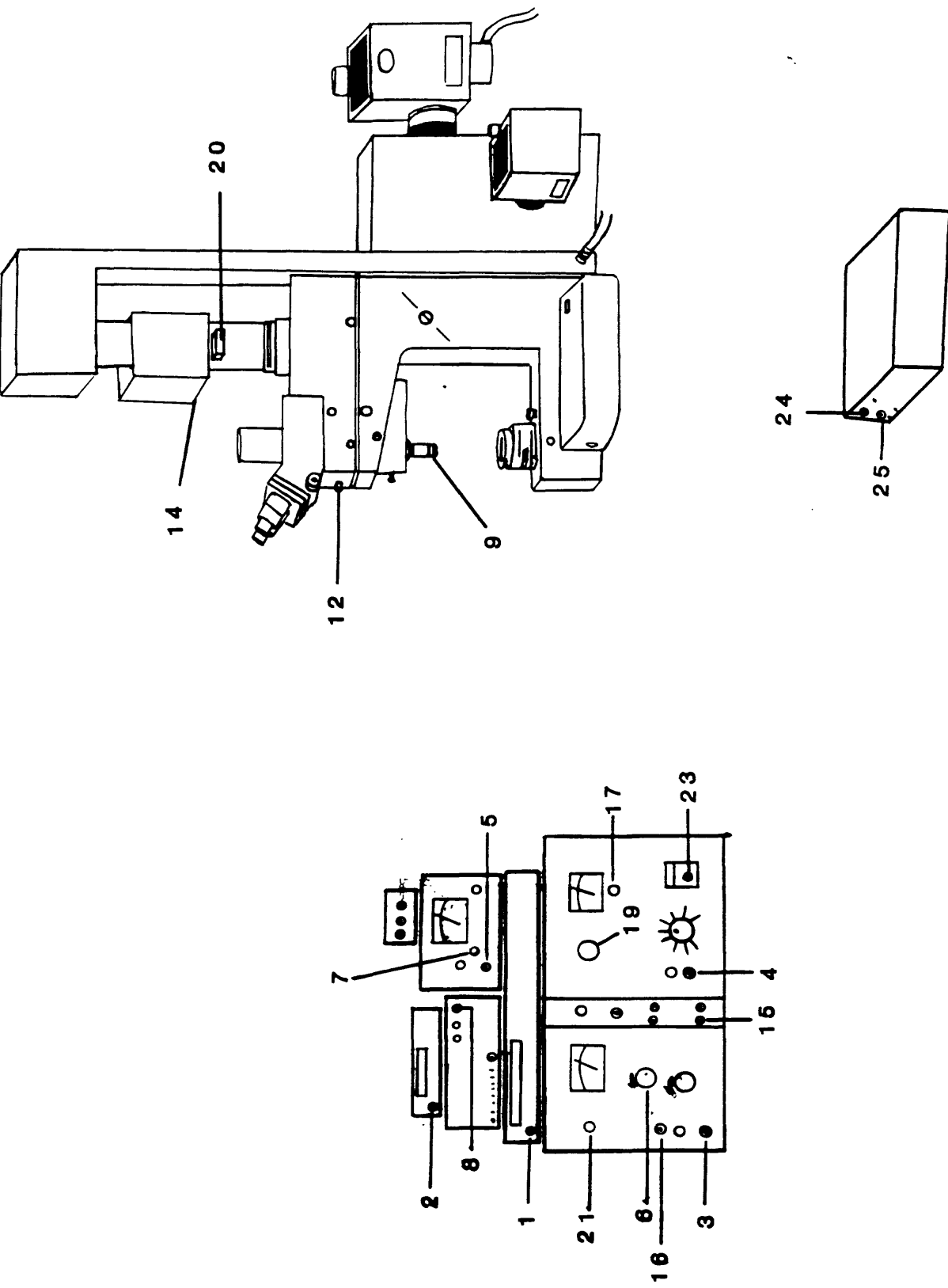


Figure 5: Diagram of microscope set-up

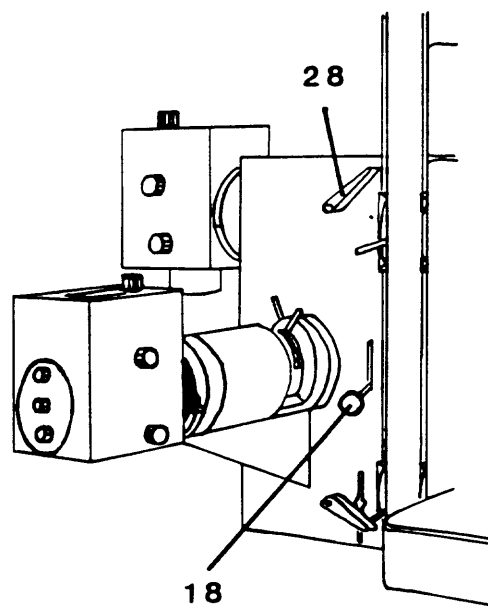
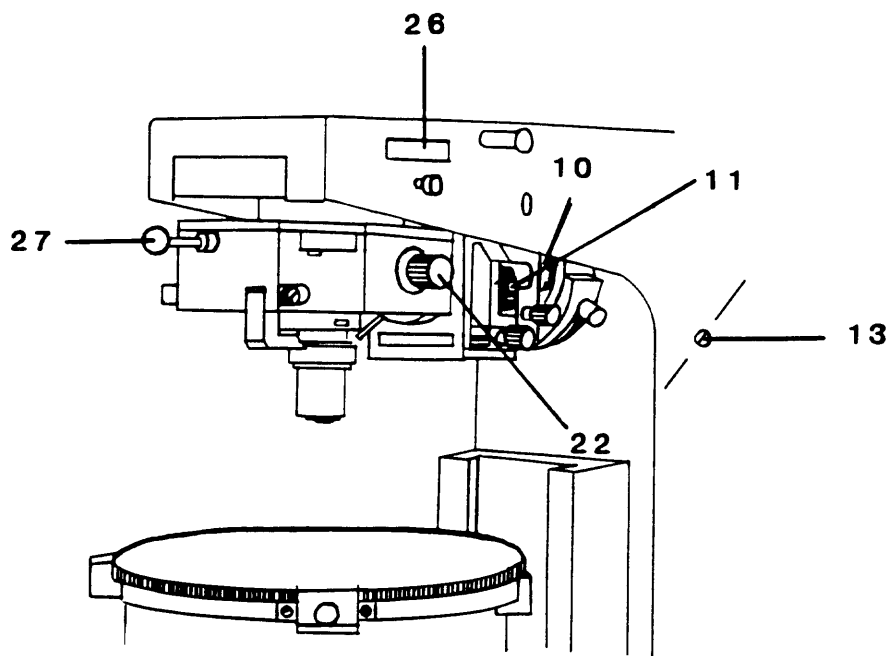


Figure 5 (continued)



**FIGURE 6: Example of a calibration matrix**

<b>GLASS #</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>5</b>	<b>6</b>
<b>1</b>	<u><b>0.300</b></u>	<b>0.508</b>	<b>0.911</b>	<b>1.024</b>	<b>1.351</b>	<b>1.649</b>
<b>2</b>	<b>0.306</b>	<u><b>0.496</b></u>	<b>0.906</b>	<b>1.022</b>	<b>1.351</b>	<b>1.654</b>
<b>3</b>	<b>0.304</b>	<b>0.504</b>	<u><b>0.921</b></u>	<b>1.007</b>	<b>1.363</b>	<b>1.650</b>
<b>4</b>	<b>0.307</b>	<b>0.508</b>	<b>0.924</b>	<u><b>1.039</b></u>	<b>1.386</b>	<b>1.670</b>
<b>5</b>	<b>0.324</b>	<b>0.554</b>	<b>0.928</b>	<b>1.049</b>	<u><b>1.413</b></u>	<b>1.703</b>
<b>6</b>	<b>0.307</b>	<b>0.509</b>	<b>0.914</b>	<b>1.028</b>	<b>1.361</b>	<u><b>1.661</b></u>
<b>Calibration Values</b>	<b>0.306</b>	<b>0.509</b>	<b>0.923</b>	<b>1.020</b>	<b>1.372</b>	<b>1.656</b>
<b>Stage Coordinates</b>	<b>4x116</b>	<b>4x109</b>	<b>4x100</b>	<b>13x116</b>	<b>13x116</b>	<b>13x100</b>
<div> <div>DATE _____</div> <div>BY _____</div> </div>						

### 9.3 Maceral Analysis (ASTM 2799-92, 1992f; ICCP, 1963)

Equipment . . . . . 500X reflected light petrographic microscope  
eyepiece with cross hair  
mechanical stage point counter  
immersion oil (non-drying, refract. index 1.515-1.519)  
sample leveling press  
modeling clay  
2" x 1" glass slides

#### 9.3A Setting-up the Microscope (Numbers refer to Figure 5)

1. Turn on the main lamp power supply (3) and the mercury vapor lamp (24) [if conducting blue-light (fluorescence) analysis].
2. Increase to maximum the main lamp power supply current (6).
3. If performing blue-light (fluorescence) analysis, ignite the mercury vapor lamp (25).
4. Install either objective lens type 50X.85p oel for white-light analysis or NPL FLUOTAR 50X.85P for blue-light analysis (no oil used --- this is an air objective!).
5. Remove 8 slide (11) and replace lensed aperture diaphragm with clear aperture diaphragm (tube behind item 10 in Figure 5).
6. Switch the condensor out by lining up its indicator knob and reference line (13).
7. Engage the half stop (10) and push in MPV 2 (12).
8. Mount and level a sample onto a glass slide using modeling clay and the sample leveling press.
9. Secure the mechanical stage onto the microscope platform.
10. For blue-light analysis, engage the yellow filtration (26), the blue filtration (27) and the fluorescing light-source (28). For white-light analysis (which should be done after blue-light analysis), place a small amount of immersion oil on the sample face.
11. Place the sample under the objective on the mechanical stage.
12. While watching the objective and sample, focus down until the two become quite close.
13. While looking through the eyepiece, focus until the image is sharp and clear.

### 9.3B Anthracite and Bituminous Coals

1. For anthracite and bituminous coals use the classification sheet displayed in Figure 7.
2. If fluorescing macerals are present, perform 500 point counts on a previously etched sample using the blue-light classification. Record counts on the appropriate data sheet.
3. Change microscope set-up to white-light mode.
4. Perform 500 point counts on a previously etched and oiled sample using the white-light classification. Record counts on the appropriate data sheet.

### 9.3C Lignite and Subbituminous Coals

1. For lignite and subbituminous coals use the classification sheet displayed in Figure 8.
2. Repeat steps 2-4 above (section 9.3B) and record data.

### 9.3D Suggestions

1. White light analyses should always be performed on etched sections unless there is an appreciable amount of micrinite in the sample. Since etching easily destroys micrinite, such samples should be analyzed in an unetched state.
2. Two pellets for each sample should be counted (500 points each), preferably by separate individuals, as a check of accuracy and reproducibility. These data should be within 2% mean variation (ASTM 2799).
3. Mineral matter is invariably counted (in the "Other" category) during blue-light analysis but not during white-light analysis where it is easily recognized and avoided. Thus, blue-light data needs to be adjusted to a mineral-matter-free (mmf) basis so as to be equivalent with white-light data. A sample's sulfur content and ash yield values should be used in conjunction with the Parr Mineral Matter Formula (ASTM 2799-92) in order to calculate its mineral matter content. This calculated mineral matter content value is then used to adjust each blue-light count maceral type total to obtain mineral matter free values. These adjusted values are used in all subsequent calculations.

### 9.3E Safety Procedures

**Never, ever, look directly at the blue-light illumination without the proper barrier filters in place! Severe eye damage can occur!**

# Bituminous Coal Data Sheet

Coal Bed: \_\_\_\_\_ Date: \_\_\_\_\_  
 Sample #: \_\_\_\_\_ Ash % \_\_\_\_\_  
 Comments: \_\_\_\_\_

	A	B	d	Totals	*OB (1000-OW)
<b>White Light Analysis</b>					
Telinite					
Eucorpo in Tel					
Poricorpo in Tel					
Gelo - gray coll					
Gelo - blk granular					
Gelo - w/mic					
Gelo - gray granular					
Eucorpo in Gelo					
Poricorpo in Gelo					
Detrocollinite					
Fusinite					
Semifusinite					
Fus corpo in fus					
Fus corpo in matrix					
Semicorpo in semifus					
Semicorpo in matrix					
Gelofusinite					
Gelosemifusinite					
Macrifusinite					
Macrisemifusinite					
Micrinite					
Inertodetrinite					
Sclerotinite					
Others (lipts) OW					

	A	B	d	Totals
<b>Blue Light Analysis</b>				
Sporinite				
Cutinite				
Resinite in tel				
Resinite in matrix				
Amorphous resinite				
Fluorinite				
Bituminite				
Exsudatinite				
Alginite				
Suberinite				
Liptodetrinite				
Others (V+I) OB				

**FIGURE 7: Data sheet for high-rank coals**

# Low Rank Coal Data Sheet

Coal Bed: \_\_\_\_\_ Date: \_\_\_\_\_  
 Sample #: \_\_\_\_\_ Ash % \_\_\_\_\_  
 Comments: \_\_\_\_\_

<b>White Light Analysis</b>	A	B	d	Totals	<b>*OB</b> (1000-OW)
Humotelinite					
Eucorpo in Telinite					
Poricorpo in Telinite					
Eu-gelinite					
Pori-gelinite					
Detrogelinite					
Eucorpo in Gelinite					
Poricorpo in Gelinite					
Fusinite					
Semifusinite					
Inertodetrinite					
Sclerotinite					
Macrinite					
Total Liptinite					
Minerals ( isolated ct )					
Total Macerals ( cts mmf )					

<b>Blue Light Analysis</b>	A	B	d	Totals
Sporinite				
Cutinite				
Resinite in tel				
Resinite in detrinite				
Suberinite				
Exsudatinit				
Bituminite				
Fluorinite				
Alginite				
Liptodetrinite				
Fluor. Clays ( isolated )				
Others, nonfluorescing				

**FIGURE 8: Data sheet for low - rank coals**

## 9.4 Spectral Fluorescence

Equipment . . . . . 500X reflected light petrographic microscope  
eyepiece with measuring rectangle  
photomultiplier  
PC with bernoulli and laser printer  
Spectra software  
sample leveling press  
modeling clay  
2" x 1" glass slides

### 9.4A Preliminary Steps (bracketed numbers refer to Figure 5)

1. Place the following filters in the rear microscope housing: BG23, BG38 and UV. Replace filter S542-21 with a blank insert.
2. Microscope body filtering should be set as per blue-light procedure (i.e., remove 8 slide [item #11 in Figure 5] and replace lensed aperture diaphragm with a clear aperture diaphragm [tube behind item #10 in Figure 5]).
3. The microscope body half-stop (28) should be in the down position.
4. MPV-2 (12) should be pushed in; the lower stop (27) should be pulled out.
5. Barrier filter K530 **must** be in place (26) during all viewing. The filter should be removed when taking measurements.
6. Turn on the mercury vapor lamp (25) and ignite (24). **No other electronics should be on at this time.**
7. Install the objective lens type NPL FLUOTAR 50X.85P (no oil used -- this is an air objective).
8. Turn on the large and small digital voltmeters (1 & 2), lamp power supply (3), high voltage power supply (4), computer and printer.
9. Increase the lamp power supply to maximum (6).
10. Change the printer selection to the "laserjet" setting. Install "Spreadsheet" bernoulli and type c:\spectra (return). Program will begin.

#### 9.4B Measuring Spectral Fluorescence

1. Center the eyepiece measuring rectangle over a maceral of interest. Remove the barrier filter K530 (26). Pull out stop MPV-2 (12).
2. Flip switch #13 on the main power unit (this is item 15 on Figure 5) to the down (or "A") position. Engage the photometer with the foot pedal.
3. In the computer program menu select "low intensity" and then the "autoscan" option.
4. Press in the white wavelength button (next to switch #15) on the main power unit. The autoscan process will begin and the button will remain lit as long as the spectral wedge is in motion. When scanning is finished, the process will auto-stop.
5. Additional measurements can be taken only after the spectral wedge has returned to zero and auto-stop has occurred. **Remember to re-engage barrier filter K530 prior to continued viewing!**
6. Further measurements can be taken by repeating steps 1 - 5, described above.
7. The number of measurements required for a representative classification will vary from sample to sample depending on such factors as the number and type of macerals available, data scattering, etc. As a general rule, a minimum of 15 measurements per maceral type should be taken when possible.

#### 9.4C Record Keeping

All data and printouts should be placed in an appropriate data file.

#### 9.4D Suggestions

1. To change over to the standard blue-light setup: replace the UV filter with filter BG12. Use barrier filter K530 during all viewing.
2. During the sample preparation phase, the sample should not be exposed to temperatures in excess of 60°C or treated with phosphoric acid, nitric acid or bromoform. Such events can lead to changes in the spectral curve and the generation of spurious data.

#### 9.4E Safety Procedures

**Never look directly at blue-light illumination! Severe eye damage can result!**

## **10.0 EMERGENCY PROCEDURES AND PRECAUTIONS**

### **10.1 Eye Wash Station**

**The eyewash station should be positioned in the laboratory free of any obstructions. The water should be changed at least once every 6-12 months. Be acquainted with the correct operation of the eye wash station before an emergency.**

### **10.2 Fire Extinguisher**

**The fire extinguisher should be located free of obstructions in the laboratory. The correct type of extinguisher should be used depending on the type of hazard that exists in the laboratory. Before using, dial x7222. If there is no answer, dial 9-911 to report the fire and then extinguish it.**

### **10.3 First Aid**

**A first aid box consisting of band-aids, antiseptic, bandages, etc. should be placed in an easily accessible location in the laboratory. Any accident, regardless of how minor, should always be reported to a supervisor. Most accidents should also be reported to the health unit.**

### **10.4 Acid Spills**

**In case of acid spills, the emergency number is x7222 and should be called IMMEDIATELY, DO NOT ATTEMPT TO CLEAN UP THE SPILL YOURSELF.**

## **11.0 ACKNOWLEDGEMENTS**

**Many people have helped to develop and improve the procedures in this manual. We wish to especially thank Frank T. Dulong, Terry Lerch, Brenda Pierce, and Leslie Ruppert. Through the years, industry and university personnel have influenced our procedures. These individuals include: J.J. Renton, Ralph Gray, Alan Davis, William Spackman, Jack Crelling, Timothy A. Moore, Cortland F. Eble, and William Grady. We also wish to thank Brian Gerardi for drafting the figures used throughout this report.**



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