U. S. DEPARTMENT OF INTERIOR

GEOLOGICAL SURVEY

Coal Petrographic Laboratory Procedures and Safety Manual

by

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This report is preliminary and has not been reviewed for conformity with USGS editorial standards and stratigraphic nomenclature.

PREFACE

This manual has a dual purpose: 1) to provide to new employees, interested colleagues, the industry, 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 sample preparation in the laboratory. Precise methodology ensures reliable quality control and provides maximum safety potential. In writing this handbook we have tried to set procedures that will allow employees to work under a minimum 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, new techniques for sample preparation and analysis are constantly being improved and better materials for use in preparations are constantly being developed. Therefore, updating of this manual will be periodically necessary.

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1. INTRODUCTION

1.1 Purpose of Laboratory

The purpose of this laboratory is to prepare coal and associated rock samples for physical and optical analyses. Physical analyses performed in our laboratory consist of chemistry determinations, low temperature ashing (LTA), high temperature ashing (HTA), volatile matter determinations, and X-ray radiography. Optical analyses consist of vitrinite reflectance and spectral fluorescence (measures of coal diagenesis) measurements and maceral (organic) analysis. Subsplits of samples are also sent to the Branch of Analytical Laboratories (USGS) for chemical and mineralogical analyses.

From the results of these analyses, coal samples can be characterized by their composition, rank, and washability potential. 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, 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 that are associated with coal beds are prepared (i.e. claystone, shale, limestone, sandstone). Peat 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 subsampling and description. A channel sample, usually collected from a fresh mine face, arrives as broken and unoriented pieces in a bag. Channel samples collected from outcrops can be weathered, greatly limiting the types of analyses that can be performed reliably.

Although the samples may differ in mode of collection, sample preparations for each type are similar.

1.3 General Safety Rules

1. Safety glasses are mandatory for any work performed in the laboratory.

2. Contact lenses are not permitted when using potting resins, acids, or organic solvents.

3. Any visitors and minors may be present only with the permission of the lab supervisor, and must wear safety glassas at all times.

4. The last employee to leave for the day should lock the doors, check status of ovens, furnaces, and all other electrical devices.

5. All reagents should be clearly labeled and stored in designated places.

6. All heavy items and chemicals should be stored below counter level.

7. Fume masks must be worn when pouring resins.

8. Rubber or disposable plastic gloves must be worn when using acetone, methanol, acids, toluene, resins, or colloidal silica polishing compound.

9. No open-toed shoes may be worn in the laboratory or grinding room.

10. The following must be used under the fume hood:
    a. Acetone (used for cleaning molds)
    b. Methanol (used for cleaning molds)
    c. Toluene (used for adding to epoxy and removing lap cloths)
    d. Epoxy-Resins (used in preparing pellets)
    e. Shellac (used for spraying core)
    f. Polyester Resins (used for potting block and core samples)
    g. Acids (used for special preparations)

N.B.: Acids must not be used or stored with any organic compounds (items a-f).

2 LABORATORY ORGANIZATION

2.1 Sample Flow

Our laboratory follows ASTM standard D2013 (ASTM, 1983a) for the correct procedures of crushing, grinding, sieving, and splitting coal samples. The steps in processing of an idealized sample are illustrated in Figure 1. When a bulk sample is received in the laboratory, it is first crushed to minus 8 mesh (<2.4 mm), then split into three subsamples; one-third is sent to the Branch of Analytical Laboratories (USGS) for mineralogical and chemical analyses; one-third is retained as a laboratory split that is processed further by us; and the remaining third is kept as a storage split. When insufficient sample (less than 100 g) is available for three splits, the storage split is omitted.

Using a large high-speed rotary mill grinder with a 2 mm sieve screen, the laboratory split is crushed to less than 20 mesh (850 um). Three subsplits obtained by using a riffle sampler (opening > 2.5 mm), are as follows: (1) a split for pellet making, (2) a split for density determination, and (3) a split for storage. Again, if insufficient sample is available for all three splits, the storage split is not taken.

After a density determination has been performed on the density subsample, it is ground to less than 60 mesh (250 um) using a small high-speed rotary mill grinder. Two subsplits are made from the ground sample: one for a high temperature ash (HTA) determination and the other for a low temperature ash (LTA) determination. Because little sample is needed for the HTA, only a very small subsplit is taken (about 2). After the LTA has been performed, the remaining unashed sample is retained as a storage split. The ashed remains are held for X-ray mineralogy.

To summarize, e sample progresses through three basic preparation steps: (1) preliminary crushing and splitting at 8 mesh, (2) crushing to 20 mesh for the petrographic splits, and (3) 60 mesh fine-grinding of the density 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 the samples, but also the maintenance of sample status records. It is just as important to know a sample's stage of preparation as it is to know where it is physically located. Because our laboratory, at any particular time, may have 200 to 300 samples at nine or ten different stages of processing, written ledgers are used to note sample progression.

In our laboratory, eight notebooks are kept (figure 2). Six of these notebooks are used primarily for on-the-spot data entry for specific procedures. These are the Density, HTA, LTA, and Volatile Matter data entry books and the Pellet and Microblock data books. A sample's progress in these and all other procedures, is noted in a seventh notebook, the Sample Status Book. The sample status sheets (figure 3) were designed to logically follow the flow of sample preparation. The eighth notebook, the Values Book, compiles the final data from the LTA, HTA, Density, and Volatile Matter determinations, thus allowing easy access and comparison of data for any sample.

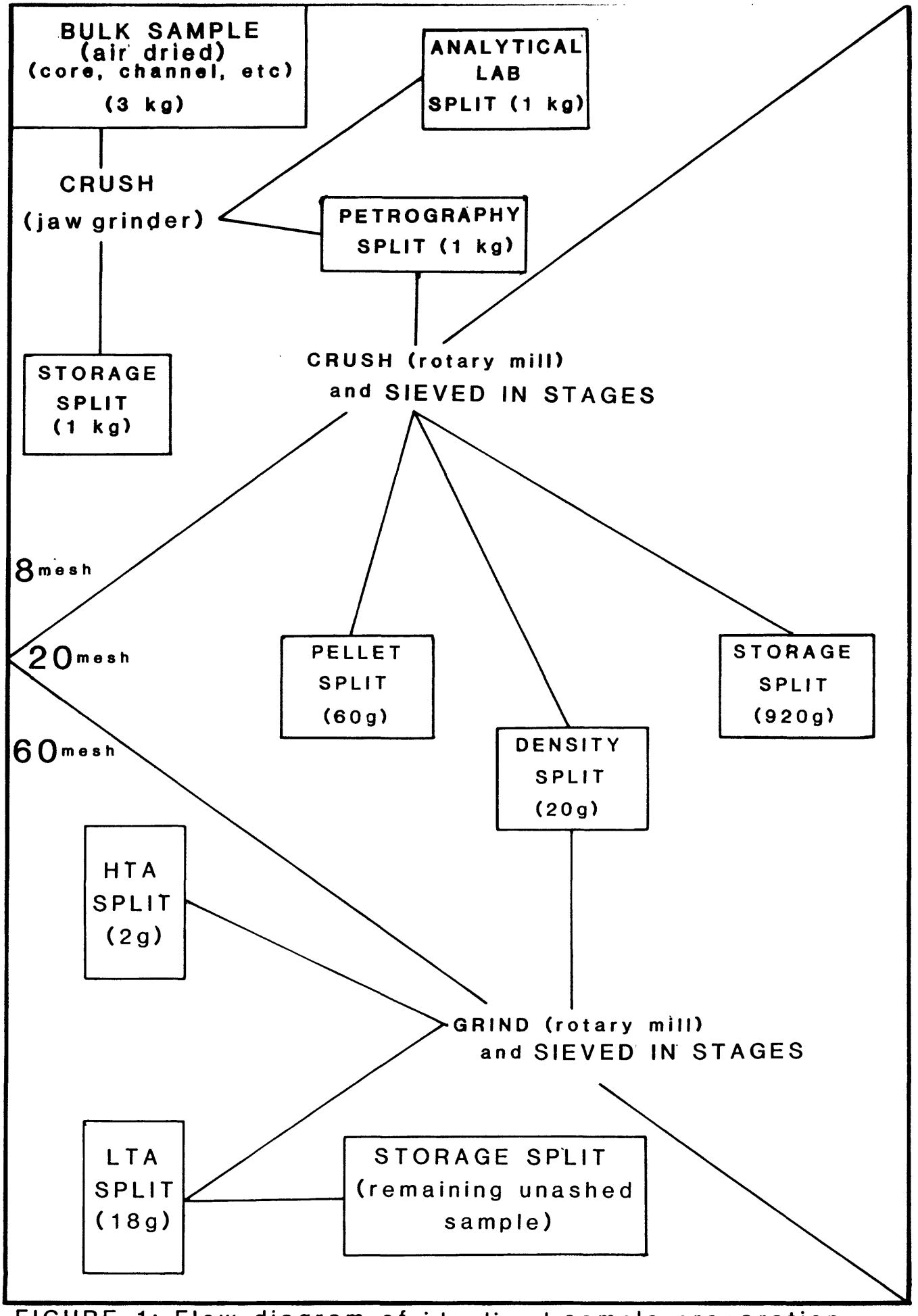

FIGURE 1: Flow diagram of idealized sample preparation

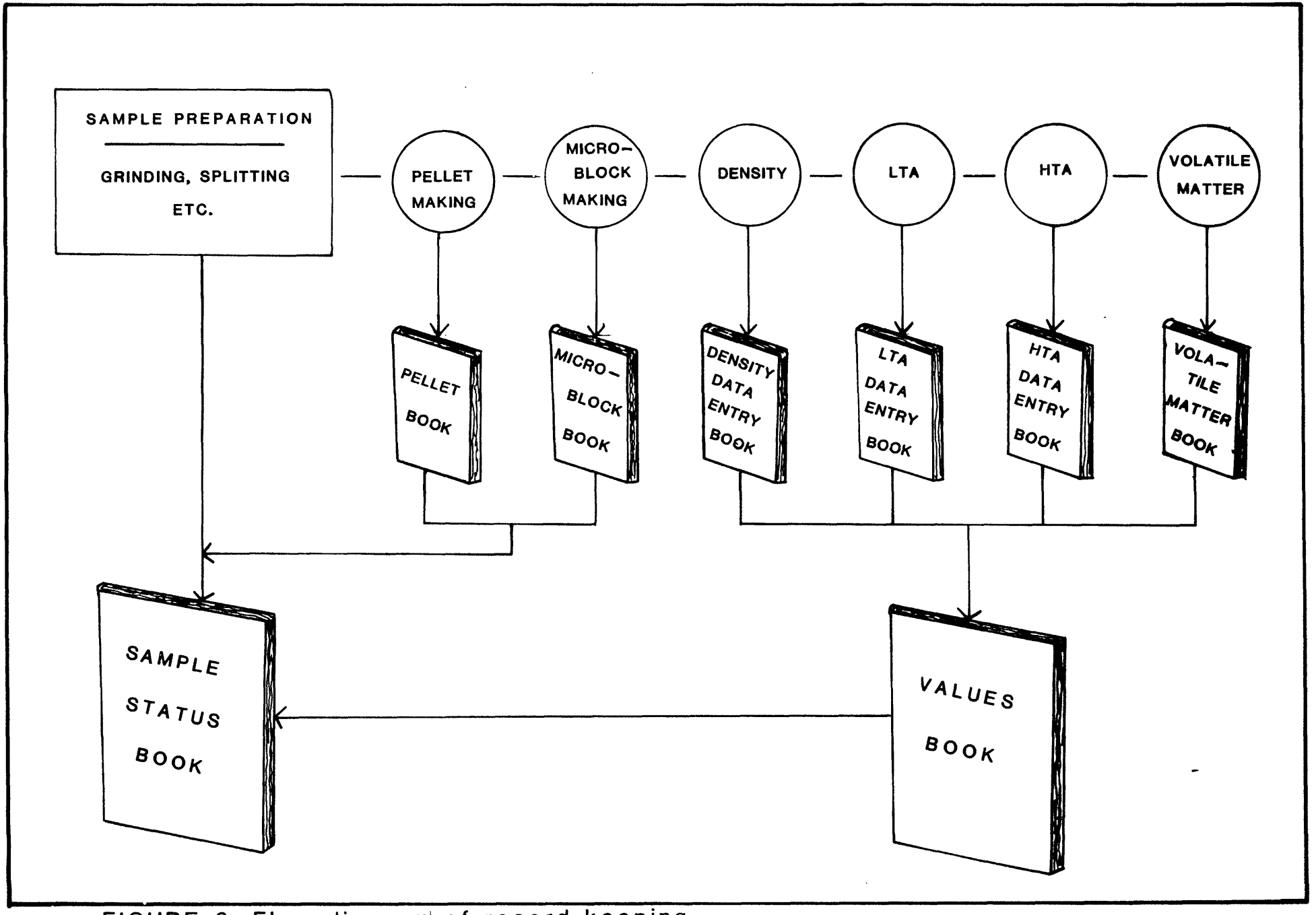

FIGURE 2: Flow diagram of record keeping

Status Sheet

Sample #	-8 mesh		-20 mesh			-60 mesh				
	Storage	Analyt. Labs	Storage	Pellet	Density	HTA	LTA	Storage	Micro-block	Other
	split	split / sent	split	split / formed	split / done	split / done	split / done	split	sample / formed	

FIGURE 3: Sample status sheet

3 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 rifflers should be used. Particle size must also be considerad when subsplits are small. Improper splitting gives unrepresentative splits and leads to erroneous results.

Sample contamination is another common problem within the laboratory. Grinding machine, splitting rifflers, mortar and pestles, sieves, and other equipment that come into contact with the coal or rock samples must be thoroughly cleaned between samples (this means using acetone and disposable wipes). In eddition, laboratory counter tops, fume hoods, and the general working area should be kept as clean as possible to prevent dust contamination of samples.

3.2 Importance of Clear Labeling

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

4 SAMPLE PREPARATION, ANALYSIS, AND SAFETY PROCEDURES

4.1 Sample crushing and grinding (ASTM 2013, 1983a)

4.1A Jaw Crusher (8 mesh grinding)

Equipment	jaw crusher large riffler (3/8"-1/2" chutes) acetone disposable wipes plastic bags permanent marker pen Sample Status Book
Safety Equipment	dust respirator safety glasses/goggles ear mufflers rubber gloves closed-toe shoes laboratory coat and/or apron

4.1A.1 Preliminary

a. Thoroughly clean crushing plates of jaw crusher with acetone and disposable wipes.

b. Check minimum gap between plates to insure the spacing is 1/4".

c. Place cardboard shelf under crushing plates.

d. Clean and place plastic sample catcher bin under plates and on top of cardboard shelf.

e. Turn on hood vacuum.

4.1A.2 Crushing

a. Turn on jaw crusher.

b. Place bulk sample between crushing plates. Crush until all sample has passed or the bin becomes full.

c. Turn off machine and open crushing plates.

d. Sweep all residue sample off the plates and lower platform into bin.

d. Thoroughly clean plates and platform with acetone and high pressure air.

4.1A.3 Splitting

a. Split sample into thirds using large riffler. Mark the bags "Analytical labs", "Petrography", and "Storage". If there is insufficient sample for the 3 splits omit the storage split.
b. Clean machine with acetone.

4.1A.4 Record Keeping

Record all grinding and splitting in the Sample Status Book.

4.1A.5 Safety Procedures

a. Using the Jaw Crusher

- **dust respirator, ear protection, and safety glasses/goggles must be worn at all times.**
- **laboratory coal and/or apron must be worn**
- **no open-toed shoes or sandals may be worn**
- **keap fingers and clothing away from crushing plates**

b. Acetone

- **avoid breathing fumes**
- **wear protective gloves**
- **avoid skin and eye contact**

4.1B Large High-Speed Rotary Mill Grinder (for 20 mesh grinding)

Equipment	large high-speed rotary mill grinder disposable wipes mortar and pestle permanent marker pen riffler (medium, 1/4" chutes) 20 mesh sieve 2 oz. jar(s) 4 oz. jar(s) razor knife Sample Status Book
Safety Equipment	dust respirator safety glasses/goggles ear mufflers protective gloves closed-toe shoes laboratory coat and/or apron

4.1B.1 Preliminary

a. Check that the proper sizing screen is in the machine (2 mm screen for 20 mesh).

b. **Unplug machine** and clean machine thoroughly using high pressure air, acetone, and disposable wipes.

c. Clean the metal sample-catching cup.

4.1B.2 Grinding

a. Close mill door and plug in machine.

b. Close slide between overhead sample holder and grinding chamber.

c. Place sample into overhead sample holder (do not fill over 3/4 full), proceed through step 6, then close slider and pour in the remaining sample.

d. Put cap on sample holder.

e. Turn on machine.

f. Open slider.

g. Let sample grind completely.

h. Turn off machine and listen for "click" that indicates the blades have stopped.

9. Open mill door.

h. Check to see that all of the sample has passed through the screen (if not, close door and continue to grind until <u>all</u> of the sample passes through the screen).

i. Shake spring-loaded bottom of mill to assure that all of the sample has fallen into metal cup.

j. Remove metal cup from under the mill and place sample into a 20 mesh sieve.

k. Clean machine and metal cup thoroughly with high pressure air and acetone.

4.1B.3 Sieving

a. Place crushed sample into 20 mesh sieve (step 12 under 4.1b.2 above).

b. Gently shake sample through screen.

c. Place remaining sample which does not pass through the screen into a mortar and pestle.

d. Grind by hand until all of the sample passes through the 20 mesh screen.

e. Clean sieving equipment and mortar and pestle with high pressure air and acetone.

4.1B.4 Splitting

a. Use medium-size riffler (>1/4" riffle chutes) and obtain successive subsplits:
Split for Density (use 4 oz. jar and make a subsplit of about 20 g).
Split for Pellet (use 2 oz. jar, for a 60-g subsplit).

b. Bag and heatseal remainder and mark as Storage split -20 mesh.

c. Clean riffler equipment using high pressure air.

4.1B.5 Record Keeping

Record all grinding and splits in Sample Status Book.

4.1B.6 **Safety Procedures**

a. Using the large High-Speed Rotary Knife Grinder
- **dust respirator, ear protection, and safety glasses/goggles must be worn at all times**
- **laboratory coat and/or apron should be worn**
- **no open-toed shoes or sandals may be worn**
- **do not ever open grinder door while blades are still moving**
- **always unplug machine before cleaning inside of the chamber**

2. Acetone

- **avoid breathing fumes**
- **avoid skin and eye contact**
- **wear protective gloves**

4.1C Small High-Speed Rotary Mill Grinder (for 60 mesh grinding)

Equipment	small high-speed rotary mill grinder belt set for high speed grinding (pulley ratio 2x motor) 20 and 40 mesh delivery tubes collection jar with special lid oven, capable of maintaining 105°C acetone disposable wipes 60 mesh sieve with bottom pan plastic bags and heatsealer vials permanent marker pens microsplitter (1/8" chutes) brush Sample Status Book
Safety Equipment	dust respirator safety glasses/goggles closed-toe shoes rubber gloves laboratory coal and/or apron

4.1C.1 Preliminary

a. Preheat oven to 60°C for subbituminous coals or 105°C for higher rank coals.

b. Place opened sample jars (density jars) in oven for one hour.

c. Clean high speed rotary mill and other equipment thoroughly with high pressure air and a brush.

d. Thoroughly clean 40 mesh delivery tube and collection jar.

4.1C.2 Grinding and Sieving

a. Turn on grinder.

b. Place dried sample in hopper letting the inverted sample jar act as a cover.

c. Let machine grind for about 45 seconds.

d. Turn off machine and remove the collection jar.

e. Place the sample from the collection jar into a 60 mesh sieve.

f. Place collection jar back into grinder and turn on grinder again.

g. Sieve sample.

h. Place the portion that does not pass sieve back into grinder (do this while grinder is still on).

i. Continue to process until all of the sample passes the 60 mesh sieve.

j. Turn off machine and with the sieve placed below the discharge tube remove and clean glass plate and discharge tube.

k. Clean machine thoroughly with acetone and air.

4.1C.3 Splitting

a. Use small-sized riffler (microsplitter, 1/8" riffle chutes).

b. obtain a successive split for high temperature ash (HTA) (use small vials to split off about 2-3 grams).

c. Mark vial HTA split -60 mesh.

d. Place remainder of sample in plastic bag.

e. Heatseal and mark bag LTA split -60 mesh.

f. Clean riffle equipment using high pressure air.

4.1C.4 Record Keeping

Enter all grinding and splitting information in Sample Status Book.

4.1C.5 Suggestions

a. It is important to thoroughly clean the mesh on the delivery tube. If there are problems cleaning the mesh, place tube into an ultrasonic cleaner. Dry tube by placing in oven.

b. If the mesh of the delivery tubes consistently clogs, try heating the tube before grinding.

c. Grinder and tube should be cleaned with acetone often when grind ing shaley end dirty coals.

e. Rotary blede gap should be 0.015 inch.

4.1C.6 Safety Procedures

a. Using the small High-Speed Rotary Mill Grinder

- use in vented hood
- dust respirator and safety glasses must be worn at all times
- laboratory coat and/or apron should be worn
- no open-toed shoes or sandals may be worn
- do not remove glass plate while blades are moving
- do not insert fingers into chamber for any reason unless machine is unplugged
- after adjusting blades always chack to see if rotor will turn 360^{o} before switching machine on.

b. Acetone

- avoid breathing fumes
- avoid skin and eye contact
- protective gloves must be worn

4.2 Casting of pellet and block samples

4.2A Pellet Making

Equipment	stainless steel pellet molds (I.D.=1") with 1/4" plugs and 1" plugs epoxy-resin wooden stirring sticks paper cups plastic mixing rod marking pen heating oven masking tape release agent (fluorocarbon) hydraulic press analytical balance (triple beam) Pellet Book Sample Status Book
Safety Equipment	rubber gloves rubber apron or laboratory coal organic vapor respirator safety glasses/goggles fume hood

4.2a.1 Procedure for Anthracite and Bituminous Coal (ASTM D2797, 1983b)

a. Preliminary

1. Label 3 molds with sample number.
2. Label 2 paper cups with sample number.
3. Spray release agent (fluorocarbon) on molds of the 2 cups.
4. Pour equal (approximately 30 g) amounts of coal into each of the 2 cups.

b. Mixing and Pouring of Epoxy (performed in fume hood)

1. Mix epoxy-resin in correct proportions (e.g. 4 parts resin (20 g) to one part activator (5 g) (by weight).
2. Add 2-3 ml of toluene to epoxy (this will decrease the viscosity of the epoxy).
3. Mix epoxy with coal in cup until coal is "just wet."
4. Place plug into the bottom of the mold.
5. Place equal amounts of coal/epoxy mixture into each of the 4 molds.

c. Pressing and Compacting the Mixture

1. Place pellet mold under hydraulic press and pump to 7000 p.s.i. and release; continue to pump and release until dial stabilizes (approximately 8–10 times).

2. Pump to 7000 p.s.i. again and let set for at least 3 min. under pressure.

3. Remove, repeat for each mold.

4. Put aside to cure overnight.

d. Popping and Labeling of Pellets

1. Extract pellet from mold by using the side plunger on the hydraulic press.

2. Persist in removal of all plugs.

3. Replace pellet into the same mold, remembering to space the bottom 1/2" (i.e. use two thin plugs for proper spacing).

4. Place appropriate typed color-coded label on top surface of pellet.

5. Pour fresh, clear, epoxy-resin to the rim of the mold.

6. Let cure overnight (for fast curing, place in oven for one hour $60^{o}C$).

7. Pop pellet out using hydraulic press (to avoid damaging the pellet, place 1/4" plug on coal surface and press out on plug).

e. Cleaning of Molds (performed in fume hood)

1. Place empty, used molds and plugs in an acetone bath overnight.

2. Scrub molds with steel wool in fume hood

3. Rinse with acetone.

4. Let dry.

5. Store the clean, dried molds and plugs away from dirt and dust.

f. Record Keeping

1. Record in Pellet Book the date that pellet was made and labeled.

2. Record in Sample Status Book that pellet was made.

4.2A.2 Procedure for Lignite and Subbituminous Coals

a. Preliminary

1. Dry sample in oven in a petri dish for at least 2 hours at 60°C.

2. Label 3 molds with sample number and place in oven with sample for 2 or more hours.

3. Label 2 paper cups with the sample number.

b. Mixing and Pouring of Epoxy

1. Mix epoxy-resin in correct proportions (e.g. 4 parts resin to one part activator (by weight) (about 20 g to 5 g).

2. Add 2-3 ml of toluene to epoxy of dry, (to reduce viscosity).

3. Pour approximately equal amounts of dry, heated coal into each of the 2 cups (total 60 g).

4. Mix apoxy into coal until the mixture appears "gummy" or "oozy".

5. Remove molds and plugs from oven.

6. Spray release agent (fluorocarbon) on molds and plugs.

7. Place equal amount of "gummy" coal mixture into each of the 4 molds.

c. Pressing and Compacting the Mixture

1. Place pallet mold under hydraulic press and pump to 5000 p.s.i. and release; pump again until dial stabilizes (approximately 8-10 times).

2. Pump again to 5000 p.s.i. and leave for 2-3 min. under pressure.

3. Remove, repeat for each mold.

4. Put aside to cure overnight.

d. Popping and Labeling of Pellets

Same as for anthracite and bituminous coals

e. Cleaning of Molds

Same as for anthracite and bituminous coals

f. Record Keeping

1. Record date pellet was made and labeled in Pellet Book.

2. Record in Sample Status Book that pellet was made.

4.2a.3 Safety Procedures

1. Handling Epoxy

   - organic vapor respirator, safety glasses/goggles, and rubber gloves must be worn
   - do not wear contact lenses
   - avoid breathing fumes, use under fume hood
   - laboratory coat and/or apron should be worn
   - avoid skin and eye contact

2. Hydraulic press

   - safety glasses/goggles should be worn
   - avoid overloading press
   - properly secure mold under press
   - make sure press features are tightened

C. Cleaning Molds - use of acetone

   - rubber gloves must be worn
   - safety glasses/goggles should be worn
   - use acetone under fume hood, avoid breathing fumes
   - use hand lotion or cream on hands to prevent excessive drying

4.2B Microblock Casting

Equipment	stainless steel pellet molds (I.D.=1") with 1/4" plugs rock saw epoxy-resin wooden stirring sticks paper cups marker pen masking tape release agent (fluorocarbon) analytical scale (triple beam) Sample Status Book Microblock Book
Safety Equipment	rubber gloves laboratory apron or coat organic vapor respirator safety glasses/goggles fume hood

4.2B.1 Cutting

a. If sample is oriented, clearly mark orientation.

b. Using a water lubricated rock saw (or band saw) cut a cube the correct size to fit into a stainless steel pellet mold (I.D. = 1").

c. Let sample dry at room temperature for 8 hours or put into oven at 60^{o} for one hour.

4.2B.2 Casting

a. Label mold with sample number.

b. Place two 1/4" plugs in bottom of mold and tape into place.

c. Spray release agent (fluorocarbon) into mold.

d. Mix a small amount of epoxy-resin in the correct proportion (4 parts Armstrong C-4 to one part activator "D" (by weight)).

e. Place sample into mold and pour a few drops of epoxy into the bottom.

f. Let set up (about 2 hours--this will prevent "floating" of the sample).

g. Place label on sample in mold.

h. Mix and pour fresh epoxy-resin to the rim of the mold.

i. Let cure overnight or in an oven at $60^{o}C$ for one hour.

4.2B.3 Record Keeping

a. Record in Microblock Book date sample was prepared.

b. Record in Sample Status Book that a microblock was made.

4.2B.4 **Safety Procedures**

a. Rock Saw or Band Saw

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

b. Handling Epoxy

- **organic vapor respirator, safety glasses/goggles, and rubber gloves must be worn**
- **avoid breathing fumes, use under fume hood**
- **avoid skin and eye contact**
- **laboratory coat and/or apron should be worn**
- **do not wear contact lenses**

4.2C Microsample Casting

Equipment	stainless steel mold (I.D. = 1") with plugs epoxy-resin wooden stirring sticks paper cups release agent (fluorocarbon) hand drill or drill press Sample Status Book Pellet Book
Safety Equipment	organic vapor respirator rubber gloves laboratory apron or coat safety glasses/goggles fume hood

4.2C.1 Preliminary - Making Epoxy "Blank"

a. Make epoxy-resin blank by pouring epoxy mix into a stainless steel mold spaced at the bottom with two 1/4" plugs.

b. Let cure for one day or one hour in an oven at 60°C.

c. Pop out epoxy blank.

d. Polish one side of epoxy blank to at least 5 micrometer polish.

e. Drill a small, shallow hole of appropriate size (depending on the amount and the dimensions of the sample grain(s),

4.2C.2 Mixing and Casting

a. Mix crushed sample with epoxy either in a peper cup or in the prepared, drilled hole (depending on amount of sample).

b. Make sure to fill hole with mixture level with the surface of the pellet and tap pellet mold on counter surface gently to bring air bubbles to surface.

c. Wipe away any excess epoxy from the surface.

d. Let cure one day or 1-2 hours in an oven at 60°C.

4.2C.3 Record Keeping

a. Record in Pellet Book that sample was prepared.

b. Record in Sample Status Book that a microsample was made.

4.2C.4 Safety Procedures

a. Handling Epoxy

- organic vapor respirator, safety glasses/goggles, and rubber gloves must be worn
- avoid breathing fumes, use under fume hood
- avoid skin and eye contact
- laboratory coat and/or apron should be worn
- do not wear contact lenses

b. Using Drill

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

4.2D Hand Sample Casting

Equipment	polyester or epoxy resin and hardener cake/pie pans(s) release agent (silicone) paper cups dye (optional)
Safety Equipment	organic vapor respirator rubber gloves safety glasses/goggles laboratory apron or coat fume hood

4.2D.1 Cutting

a. Coal block or core piece should be taped with masking tape to prevent breakage.

b. Sample should be cut to produce desired sample size.

c. Cut sample should be dried in oven (60°C) for 2 hours

4.2D.2 Casting

a. Spray tray with silicone release agent.

b. Mix polyester (or epoxy resin) in appropriate proportions.

c. Add dye, if desired.

d. Place sample, cut-face down, in pan and pour plastic. (Note: depending on the rank of the sample, it may be best to pour plastic over all or only part of the block.

e. Let sample cure for a day or more at room temperature inside a fume hood.

f. After removing sample from pan, excess sample and plastic can be trimmed off edges and surface (opposite cut face). A rim can be made be using masking tape around edges and a label and another layer of clear resin can be applied to the first cut face side.

4.2D.3 Suggestions

A. If the polyester or epoxy-resin does not completely setup after a day, place in an oven for an hour or two at 60°C. Remove and let cool; plastic should have hardened.

B. Addition of some dyes may retard hardening process; to compensate, add a higher proportion of hardener.

4.2D.4 Safety Procedures

1. Rock Saw or Band Saw

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

2. Handling of Plastic-Resin

   - organic vapor respirator, safety glasses/goggles and rubber gloves must be worn
   - avoid breathing fumes, use under fume hood
   - avoid skin and eye contact
   - laboratory coat and/or apron should be worn
   - do not wear contact lenses

4.2E Vacuum Impregnating

Equipment	vacuum bell jar vacuum pump stainless steel mold (I.D. = 1") and 1/4" plugs cake/pie pan(s) polyester or epoxy-resin
Safety Equipment	organic vapor respirator safety glasses/goggles rubber gloves laboratory apron or coat 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" thickness) for X-ray radiography. Vacuum impregnating causes 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 impregnating may be used with core samples, hand samples, microblocks, and crushed coal.)

4.2E.1 Procedure for Hand Sample, Core Sample and Microblock Impregnation

a. Place cut and prepared sample into tray (for hand samples and core sample) or stainless steel mold (for microblock).

b. Mix correct amount of resin (either polyester or epoxy). Pour mixture over sample but do not fill to capacity.

c. Put sample into vacuum chamber (Note: If sample tray covers evacuating/venting hole, raise it up on blocks or place on a platform).

d. Place vacuum bell jar over sample.

e. Turn on vacuum pump. Check seal around vacuum bell jar. If there is no seal, check that the bell jar is seated securely on the rubber mat.

f. Watch sample and vent air into chamber if "boiling" over of resin occurs.

g. Evacuate for 5-10 minutes.

h. Turn off vacuum pump and vent air into chamber slowly.

i. Remove sample to allow to let it cure overnight under a fume hood.

4.2E.2 Procedure for Crushed Samples

a. Mix -20 mesh, dried, crushed sample with epoxy-resin in a paper cup (see section 4.2a). Use copious amounts of epoxy.

b. Place sample into a stainless steel mold (with one 1/4" bottom plug) and pour a small amount of epoxy on top.

c. Put sample and open-topped mold into vacuum chamber.

d. Follow steps 5 through 8 of preceding procedure.

e. Remove sample and place top plug into mold.

f. Using the hydraulic press, place 3000 p.s.i. on sample. Release immediately and repeat 3-4 times.

g. Let cure overnight. If the sample still flakes or cracks, try using more epoxy and longer evacueting time and less pressure with fewer releases).

4.2E.3 Safety Procedures

a. Handling of Polyester or Epoxy-Resin

- **organic vapor respirator, safety glasses/goggles, and rubber gloves must be worn**
- **avoid breathing fumes, use under fume hood**
- **avoid skin and eye contact**
- **laboratory coat and/or apron should be worn**

b. Vacuum Bell Jar and Pump

- **check oil level in pump**
- **ramain in room while evacuating**
- **vent air into bell jar slowly**

4.3 Polishing of pellets, microblocks, microsamples, and hand samples

4.3A Pellet, Microblock, and Microsample Polishing

(Modified from Cole and Berry, 1965; ASTM D2797, 1983b)

Equipment	automatic polishing machines short-napped polishing cloth 600 & 400 grit paper, 8" diam. 15 micrometers diamond wheel 1.0 micrometers aluminum oxide polishing compound 0.06 micrometer colloidal silica polishing suspension sample holder sample leveler ultrasonic cleaner Sample Status Book Pellet or Microblock Book
Safety Equipment	safety glasses/goggles laboratory apron or coat

4.3A.1 Polishing Pellet Tops

a. Mount 6 pellets into pellet holder with the tops down (Note: If polishing less than 6 pellets, insert epoxy 'blanks" into remaining holes).

b. Level and tighten.

c. Place on 15-micrometer diamond wheel or 400 grit paper for 1-2 min. or until surface is completely flat. Use a steady stream of water. For proper sequence of turning on and off of the lapidary wheel see suggestion B.

d. Rinse in tap water.

e. Relevel pellets in holder.

e. Rough polish for 2 min. on short-napped cloth with 1.0 micrometer aluminum oxide (50:1 mixture of distilled water and polishing compound).

f. Rinse with tap water.

4.3A.2 Polishing of Sample Bottoms

a. After polishing the tops of 6 pellets, relevel with the bottoms down.

b. Grind for 2 min. on a 15-micrometer diamond wheel. Start with an initial pressure of 30 pounds and increase by 5 pound increments until a brownish slurry occurs. Use a slow stream of tap water while the wheel is turning.

c. Relevel.

d. Rinse with tap water. Clean ultrasonically for 2 min. then rinse.

e. Final grind on 600 grit paper for 2 min. Start with an initial pressure of 30 pounds and increase by 5 pound increments until brownish slurry occurs. Use a slow stream of tap water.

f. Rinse with tap water. Clean ultrasonically for 2 min. then rinse.

g. Rough polish for 2-3 min. on short-napped cloth with 1.0 micrometer aluminum oxide (1:1 mixture of polishing compound and distilled water). Start with an initial pressure of 30 pounds and increase by increments of 5 pounds until a brownish slurry appears.

h. Without changing the pressure or stopping the machine, flush with distilled water for 1 1/2 min. to remove particles and excess polishing compound.

i. Without releasing pressure, turn off motor.

j. Rinse with distilled water. Clean ultrasonically for 2 min. then rinse.

k. Final polish for 2 min. on short-napped cloth with 0.06 micrometer colloidal silica. Start with an initial pressure of 30 pounds and increase by increments of 5 pound until slurry occurs.

l. Without changing the pressure or stopping the machine, flush with distilled water for 1 1/2 min.

m. Turn off motor without releasing pressure.

n. Rinse in distilled water. Clean ultrasonically (2 min). Rinse again.

o. Blow dry and place pellets in desiccator.

4.3A.3 Record Keeping

a. Record in Pellet Book or Microblock Book that the sample was polished.
b. Note in Sample Status Book that sample was polished.

4.3A.4 Suggestions

A. Grinding times may vary for polishing of microsamples depending on thickness of sample.

B. Sequence for turning on and off polishing laps are as follows:

a. turn on water with nothing on the wheel
b. turn on motor and run hand over the lap to remove any dirt or excess grit
c. turn off motor
d. place pellet holder under automatic polishing unit and secure

e. turn on water
f. place a little pressure on pellet holder then turn on motor
g. place appropriate pressure on pellet holder
h. polish for the recommended time
i. turn off motor
j. release pressure
k. turn off water.

For hard coals, increased polishing times may be needed.

If microprobe or SEM analyses are to be conducted on the Sample dionized water must be substituted for tap water in all steps and new cloths must be put on laps.

4.3A.5 **Safety Procedures**

A. Automatic Polishing Machine

- **safety glasses/goggles should be worn**
- **keep fingers and clothing away from moving parts, do not insert fingers under machine when in motion**
- **make sure automatic polishing machine is securely seated and the shaft is locked into position before turning on wheel.**

B. Ultrasonic Cleaner

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

C. Colloidal Silica Polishing Suspension

- **rubber gloves and safety glasses/goggles must be worn**
- **avoid skin and eye contact (basic solution)**
- **after use, lap must be kept moist to prevent drying of polishing suspension and creating hazardous dust and seal in plastic bag in drawer.**

4.3B Hand Sample Polishing

Equipment	12" diameter lapidary wheel 60, 120, 240, 320, 600 silicon carbide grit paper and/or powders 5.0, 3.0, 0.05 micrometers aluminum oxide polishing compounds band saw or rock saw
Safety Equipment	safety glasses/goggles laboratory apron or coat

4.3B.1 Polishing cut surface

a. Remove excess plastic from side of sample to be polished using a band saw or rock saw (be sure to make a straight, even cut).

b. Use 60 grit silicon carbide powder or grit paper to remove any saw marks or plastic from face of sample. Grind until flat, uniform surface exists (use a slurry of water of grit).

c. Clean ultrasonically for 2 min. (place sample face down but raised up off the bottom).

d. Use 120 grit paper or powder and grind surface until all scratches from the previous step have been removed and a uniform surface exists.

e. Clean ultrasonically for 2 min.

f. Continue grinding through 240, 320, and 600 grit, removing all scratches from the previous step and clean ultrasonically after each step.

g. Rough polish on short napped cloth with 5.0 micrometer aluminum oxide.

h. Rinse. Clean ultrasonically (2 min.). Rinse.

i. Polish on short napped cloth with 0.3 micrometer aluminum oxide.

j. Rinse. Clean ultrasonically (2 min.). Rinse.

k. Final polish on short napped cloth with 0.05 micrometer aluminum oxide or 0.06 colloidal silica polishing suspension.

l. Rinse. Clean ultrasonically (2 min.). Rinse and blow dry.

4.3B.2 Suggestions

a. Grinding time at each step will vary considerably between samples; degree of hardness and size of sample controls the amount of time needed.

b. When polishing, a clockwise rotation (opposite that of the turning lapidary wheel) around the wheel will give the best polishing results.

4.3B.3 Safety Procedures

a. Band Saw or Rock Saw

- safety glasses/goggles and ear protection must be worn
- laboratory coat and/or apron should be worn
- make sure all saw features are secure and tight
- keep fingers and clothing away from blade and moving parts
- never force blade to cut too fast
- make sure proper blade is used

b. Lapidary Wheels

- never place fingers under wheel while moving

c. Ultrasonic Cleaner

- keep fingers out of cleaner when on

d. Colloidal Silica Polishing Suspension

- rubber gloves and safety glasses/goggles must be worn
- avoid skin and eye contact
- after use, lap must be thoroughly rinsed of all polishing suspension

4.3C Etching of Polished Pellets and Other Mounts

(Modified from Stach, p. 338, 1982)

Equipment . . . . . 50, 100, 500 ml beakers
glass stirring rods
bunsen burner or hot plate
watch glass(es) and/or petri dish(es)
10ml graduated cylinder
sulfuric acid (H_2SO_4 concentrated)
potassium permanganate ($KMnO_4$)
sodium sulfite (Na_2SO_3)

4.3C.1 Preliminary

a. Mix etching solution of 100 ml water + 25g $KMnO_4$ +5ml H_2SO_4 (concentrated).
NOTE: always add acid to water

b. Mix rinsing solution of 100ml water + 25g Na_2SO_3 + 5ml H_2SO_4

c. Stir rinsing solution until all Na_2SO_3 has dissolved.

d. Heat the etching solution in a water bath until all the $KMnO_4$ has dissolved.

4.3C.2 Etching

a. If a dilution of the etching solution is necessary: (i.e. for low rank coals)
-- dilute to the desired concentration
-- reheat to boiling for one min. then continue with the etching procedure.

b. Immediately pour part of the heated etching solution into a watch glass.

c. Submerge face of polished pellet (or other mount) into etching solution (see table below for recommended etching times).

d. Remove mount and rinse immediately with water to remove excess etching solution (1-2 sec.).

e. Submerge pellet (or other mount) into rinsing solution for one min. or until purplish stain has been completely removed.

f. Clean ultrasonically with water for one min.

g. Blow dry with air immediately.

4.3C.3 Suggestions

A. Recommended etching times for different ranks of coals

Rank	Rel. % Sol.	Approx. Time (sec.)	Example of Coal bed
Low Vol. Bit.	100	55-60	Pocahontas
Med. Vol. Bit.	100	45-55	Upper Freeport
Hi Vol. Bit.	100	20-30	Waynesburg
Subbituminous	75	4-5	Powder River coals

Note: These are only recommended times; different coals beds may require variations in time and solution concentrations.

4.3C.4 **Safety Procedures**

a. Handling of chemicals

- **all chemicals must be used under 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 organic vapor respirator must be worn**
- **avoid skin and eye contact with all chemicals** H_2SO_4 **will cause severe burns)**
- **when mixing solutions always add acid to water**

b. Use of Bunsen Burner or Hot Plate

- **use under fume hood**
- **keep all chemicals and flammables away from heat**
- **place burner or hot plate on a level surface**

c. Ultrasonic Cleaner

- **when the ultrasonic cleaner is working keep fingers out**

4.3D Concentration and Preparation of Dispersed Organic Matter

(Modified from Barrows and others, 1979)

Equipment	vacuum oven mortar and pestle acid resistant polyethylene or teflon containers hot plate or bunsen burner 500 ml beaker(s) Hydrochloric Acid, 10% (HCl) Sodium Carbonate (Na_2CO_3) Zinc Bromide ($ZnBR_2$) Hydrofluoric Acid, 48% (HF) centrifuge tubes centrifuge glass bottles
Safety Equipment	organic vapor respirator safety glasses/goggles heavy rubber gloves laboratory coat rubber apron fume hood

4.3D.1 Preliminary

a. Crush sample through jaw crusher (-8 mesh) (see section 4.1 for proper procedure).

b. Place sample in polyethylene or teflon container.

4.3D.2 Chemical Disaggregation

BEFORE USING, READ FOLLOWING MATERIAL SAFETY DATA SHEETS ON PAGES 36 - 39...

a. Treat sample with HCl for 1-3 days to dissolve carbonate minerals.

b. Place sample in acid resistant polyethylene or teflon container resting in a water bath at 50°C under a fume hood.

c. Decant and rinse with water (3 rinses).

d. Treat sample with 48% HF for 1-3 days to dissolve silicate minerals.

e. Decant and add Na_2CO_3 solution to neutralize unspent acid. Rinse with water (7 rinses).

f. Dissolve HF reaction products with 10% HCl (one day).

4.3D.3 Mechanical Separation

a. Transfer sample to centrifuge tubes.

b. Centrifuge and decant water.

c. Float kerogen fraction in $ZnBr_2$ solution (s.g.=2.0), centrifuging for a short time.

d. Decant $ZnBr_2$ solution and kerogen.

e. Discard heavy fraction (unreacted shale, pyrite, and other acid insoluble minerals).

f. Dilute $ZnBr_2$ with water.

g. Centrifuge to sink kerogen and decant.

h. Rinse kerogen with water.

9. Centrifuge and decant water (3 rinses).

4.3D.4 Pellet Preparation

a. Transfer sample to glass bottles and dry in a vacuum oven at 50°C (one day).

b. Scrape kerogen from bottles and carefully grind in mortar and pestle.

c. If sample size is large, follow procedure 4.2a for making pellets and then polish according to procedure 4.3a.

d. If sample size is small follow procedure 4.2c for microsample mounting then polish according to procedure 4.3a.

4.3D.5 **Safety Procedures**

a. **Using Hydrofluoric Acid (HF)**
BEFORE USING, READ FOLLOWING MATERIAL SAFETY DATA SHEETS ON PAGES 36 - 39...
- **WARNING: Hydrofluoric acid is an extremely hazardous chemical at any concentration or temperature. Physical contact will cause severe burns and inhalation of the vapor will irritate the respiratory system. Use all necessary measures to prevent physical contact with the acid. Consider any accident with hydrofluoric acid a serious accident. LOCATION OF CALCIUM GLUTONATE GEL SHOULD BE KNOWN IN ADVANCE OF ANY AND ALL USE OF HF ACID.**
- **Heavy rubber gloves, safety glasses/goggles, laboratory coat and rubber apron must be worn at all times.**
- **Organic vapor respirator must be worn**
- **HF must be kept and used under a fume hood. Use hood's glass door in halfway closed position .**
- **do not allow HF to stand in glass containers—it is very corrosive of glass.**

Should physical contact with HF result, affected area must be flushed with water immediately for 15 minutes and help called for. Calcium glutonate gel should be applied and massaged into affected area. Victim should then be taken to nearest hospital Emergency Room for treatment.

c. **Hydrochloric Acid and Zinc Bromide**
- **heavy rubber gloves, safety glasses/goggles, laboratory coat and rubber apron must be worn**
- **organic vapor respirator must be worn**
- **avoid skin and eye contact**
- **must be used under a fume hood**

National Safety Council
RESEARCH AND DEVELOPMENT SECTION

SAFETY AND HEALTH INFORMATION SHEET

Title: HYDROFLUORIC ACID EXPOSURE

Hydrofluoric acid (hydrogen fluoride, HF) and substances that can form HF as a result of hydrolysis by moisture are toxic on inhalation and severely corrosive to the skin, eyes and mucous membranes in both the vapor and the liquid states. Concentrated aqueous solutions may cause immediate pain upon contact with the skin. Contact with dilute solutions will result in delayed pain up to 24 hours later. HF penetrates the skin barrier and continues to cause damage until proper treatment is completed. HF forming substances include:

1. Hydrogen Fluoride (HF)
   Boron Trifluoride (BF_3)
   Phosphorous Pentafluoride (PF_5)

   Form copious HF fumes in moist air; visible; extremely irritating; corrosive; early warning of presence.

2. Bromine Pentafluoride (BrF_5)
   Bromine Trifluoride (BrF_3)
   Iodine Pentafluoride (IF_5)

   Form colored vapors from halogen but not HF type fumes; visible; irritating; corrosive; warning of presence.

3. Carbonyl Fluoride (COF_2)
   Phosphorous Trifluoride (PF_3)
   Fluorine (F_2)
   Chlorine Trifluoride (ClF_3)
   Sulfur Tetrafluoride (SF_4)
   Silicone Tetrafluoride (SiF_4)

   Little if any color; generally bad odors; irritating; corrosive; give litle warning; can oxidize and hydrolyze in the body.

Precautionary Measures

Because of the extremely hazardous nature of HF the following measures are recommended to protect employees from exposure:

1. Training in the proper use of HF or HF-producing materials should be given before any exposure. The use of HF or HF-producing materials should be limited to laboratories with total exhaust ventilation systems. All work should be accomplished within a fume hood or otherwise be protected by

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an exhausted enclosure unless prohibited by size of apparatus. In the latter case, local exhaust pick-ups should be located in areas where accidental release of materials is most likely to occur. Shields or see-through plastic wraps are also recommended for likely splash points (valves, fittings, etc.).

2. Quantities of HF or HF-producing materials on hand in the work area should be kept to an absolute minimum consistent with project needs.

3. Protective clothing/devices should be used. Chemical splash goggles are mandatory for any use. Face shields and impervious clothing (gloves, aprons, coats, etc.) should also be used depending on the degree of risk to exposure. Respiratory equipment should be considered for escape/rescue purposes.

4. A means for continuous flushing of the eyes and body with water must be provided. A hand-held aerated emergency spray nozzel that is connected to a continuous supply of water by means of a short (5 ft) (1.52m) length of hose is recommended within the lab. A safety shower should be accessible, preferably within 25 feet (7.6m) of the work area.

5. Persons working with HF or HF-producing materials should not work alone, i.e., when not within audio or visual range of another individual for more than a few minutes at a time.

6. Persons working with or supervising work that involves HF or HF-producing materials should request a review of the project by safety/medical staff before work begins. All project participants should be fully familiar with the hazards of HF; review of the manufacturer's Material Safety Data Sheet is recommended.

Emergency Procedures

Whenever anyone has been seriously exposed to HF or HF-producing materials, emergency action must be initiated immediately. Any person performing first aid must avoid contaminating themselves when assisting an exposed individual. First aid treatment must be administered by the injured coworkers at once as follows.

Liquid or Vapor Contact With Skin

Individuals who have had contact with HF or HF-producing materials must be showered immediately at the work site using available emergency shower facilities. Contaminated clothing should be removed as rapidly as possible. It is essential that all exposed areas of the body be washed with copious amounts of water for at least 15 minutes.

SAFETY AND HEALTH INFORMATION SHEET

Title: HYDROFLUORIC ACID EXPOSURE

Immediately begin soaking the area in an iced solution of 0.13 percent benzalkonium chloride ("Zepharin Chloride"). If the burned area cannot be immersed in the solution, use saturated compresses changed every two minutes. Call a physician and continue soaking until further instruction. If no physician is available, continue soaking for two hours and then apply a generous quantity of calcium gluconate gel.

Contact With Eyes

If liquid HF has entered the eyes or if the eyes have been exposed to high concentrations of vapors, they should be flushed with large quantities of clean water for 15 minutes or more using emergency facilities at the accident site. The eyelids should be held apart during irrigation to ensure contact of water with all tissues on the surface of the eye and lids. A physician, preferably an eye specialist, should be called in attendance at the first possible moment. DO NOT USE ZEPHARIN CHLORIDE OR CALCIUM GLUCONATE GEL IN THE EYES.

Inhalation

Any person who has inhaled HF or HF-producing materials must be moved immediately to an area with clean atmosphere. He should remain quiet, perferably lying down, and be kept warm and comfortable. If breathing has stopped, begin artificial respiration (mouth-to-mouth resuscitation). Oxygen rich air (approximately 40 percent oxygen) should be started as soon as possible. Prompt professional attention is required. Under no circumstances should a patient be permitted to return to work or to go home until examined and discharged by a physician who is aware of the nature of the exposure.

Ingestion

Encourage the exposed person to drink a large quantity to water without delay. Then, administer milk or two ounces of Milk of Magnesia.

Storage

Only quantities of HF or HF-producing materials which are needed for a one day use, should be in the work area. Additional supplies should be kept in a chemical storage room.

Spill, Leak, or Disposal

Notify safety personnel, provide adequate ventilation, and remove ignition sources since hydrogen may be generated by reactions with metals. Use protective clothing and equipment. HF vapor should be passed through a packed tower scrubber. Spills should be covered with lime to form a slurry. Do not flush to sewers or waterways.

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The neutralized slurry can be scraped up for disposal in an approved hazardous waste landfill. Liquid wastes may be neutralized in an impoundment or open tank with lime in a remote location away from buildings and people. Follow Federal, State, and local regulations.

References

"Occupational Health Guidelines for Hydrogen Fluoride," September, 1978.

NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, DHHS (NIOSH) Publication No. 81-123.

U.S. Governmental Printing Office, Washington, DC 20402.

4.4 Petrographic and other physical analyses

4.4A Density Determination (Stanton, 1982)

Equipment . . . . . gas comparison pycnometer
analytical balance (sensitivity of 0.01g, with a 200 g capacity)
desiccator and drying agent
oven, capable of obtaining and holding 105°C
Helium
gas drying tube, drying agent
vacuum pump
disposable wipes
acetone
Density Book
Values Book
Sample Status Book

4.4A.1 Preliminary

a. Obtain prepared density split (20 - 30 grams, minimum 5 grams at 20 mesh).

b. Place sample into petri dish and label.

c. Dry in oven for at least 4 hours at 105 - 110°C.

d. After drying, place in desiccator and permit to cool to room temperature before making volume determination.

e. Turn on balance and allow it to warm up for about 20 min.

f. Helium gas source for pycnometer should be set for 3 p.s.i. Gas should be run through lines and drying cylinder before use each day to purge system

g. A zero measurement with 4 separate determinations must be made at the beginning of each day. The average zero correction that results will be checked by one determination after each sample. The procedure for a zero measurement check is identical to that for a sample except the sample cup is empty. Also, after the initial zero check, a volume determination should be made on one of the two known volume ball standards.

4.4A.2 Density Determination

a. Clean sample cup with acetone.

b. Tare cup on balance.

c. Place all of the prepared density split in cup.

d. Weigh cup and sample and record.

e. Make sure reference wheel on pycnometer is at clockwise stop.

f. Rotate measuring wheel to estimated volume of sample.

g. Firmly lock sample cup into unit.

h. Open purge valve and coupling valve.

i. Turn on vacuum pump.

j. Open vacuum valve for 60 sec. then close valve and turn off pump, in that order (Note: with large and very powdery samples (shales) it may be necessary to have vacuum introduced gradually to avoid material getting sucked up into machine).

k. Open gas valve for 10 sec. then close.

l. Open vent valve for 10 sec. then close.

m. Rotate reference wheel counterclockwise to stop.

n. Rotate measuring wheel counterclockwise past the starting number by 5 cubic centimeters.

o. Rotate measuring wheel clockwise to starting number.

p. Wait 10 sec.

q. Close coupling valve.

r. Turn both wheels (reference and measuring) clockwise simultaneously so that pointer is kept just left of zero on scale. Continue until reference wheel stop is reached.

s. Rotate measuring wheel counterclockwise to bring pointer to zero.

t. Open coupling valve. If no significant movement of needle occurs then record value to nearest 0.01 cm^3.

u. Open purge valve and remove sample cup.

v. Repeat until 3 values are obtained from one sample.

4.4A.3 Record Keeping

a. Record humidity, temperature, zero correction, and date for each sample in the Density Book.

b. Record all volume measurements in the Density Book.

c. Record final average corrected density measurement in the Values Book.

d. Record that a density determination was made in the Sample Status Book.

4.4A.4 Suggestions

a. The zero correction factor is determined by finding the number that must be added to the zero check to equal zero. This number is then added to the average volume of sample to equal corrected volume.

b. For the daily zero measurement check, for values should be obtained consecutively that are within 0.03 cm^3. For zero measurement checks, after each sample, the value should be with 0-.03 cm^3 of the average daily zero check.

c. This machine requires patience, consistency and practice by the operator to give repeatable results. For this reason, best results are obtained from one operator doing large groups of samples.

d. Some possible sources of error are:

- leakage around cup lip
- sample absorption emittance of gases (incomplete drying of sample)
- above average room humidity and temperature
- incomplete evacuation
- interruption or slowing of procedures after coupling valve is closed
- sticky zero pointer

4.4A.5 **Safety Procedures**

a. High Pressure Gas Cylinder

- **never bump or knock valve stem**
- **always turn off gas at valve stem when finished**
- **when transporting cylinders the safety cap must be over the stem**
- **cylinder has to be securely anchored to wall with chains**

b. Acetone

- **avoid breathing fumes**
- **always wear rubber gloves**
- **avoid skin and eye contact**

4.5B Low Temperature Ashing (LTA)

Equipment . . . . . plasma asher
petri dished
analytical balance accurate to at least 0.0001g
mortar and pestle
100 mesh sieve screen
oven, capable of heating to end maintaining 105°C
desiccetor with drying agent
oxygen tank
1 N ammonium acetate (pH7)
LTA Book
Values Book
Sample Status Book

Procedure of Anthracite and Bituminous Coals

4.5B.1 Preliminary

a. Obtain the -60 mesh split marked or LTA.

b. Pulverize sample with mortar and pestle to -100 mesh.

c. Place in bottle and label.

d. Zero check balance.

e. Weigh out 3g of sample in a clean, preweighed petri dish (dishes are cleaned, oven dried, and desiccated at least 24 hours prior to use). Record weight in LTA Book.

f. Dry semple in oven at 105°C for one hour.

g. Zero check balance.

h. Weigh sample dish and record.

i. Calculate % moisture and record in LTA Book.

4.5B.2 Ashing Procedure

a. Place sample in asher chamber.

b. Close chamber door.

c. Close main door.

d. To turn on LTA - push "on" button.

e. Then turn on pump (switch box is on pump).

f. Wait about 5-10 min. for pump down to evacuate to a 1mm vacuum.

g. Push "RF" button.

h. One 1mm is reached:
   - turn up RF power to 125 watts (or about 30 watts per chamber)
   - turn up oxygen flow to 1.5 cc

9 .Chambers should exhibit pale blue plasma.

4.5B.3 Stirring and Weighing Samples

a. Turn "RF" power knob counterclockwise.

b. Push "RF" button off.

c. Open main door.

d. When vacuum has been purged, open chamber doors.

e. Remove and stir sample by gently shaking sample dish to expose the unoxidized grains (stirring should be done 3 times daily).

f. Weighting of sample is done one a day.

g. To weigh, remove and place glass over dish..

h. Take into balance room and wait 5 min.

i. Weigh, after first zero checking balance.

j. Sample is done when the weight changes by less than 0.0001 gram or begins to gain weight (i.e. by absorbing moisture).

k. Either remove completed sample or note sample weight in the LTA Book.

4.5B.4 Record Keeping

a. Record % moisture in LTA Book.

b. Record final ash % in LTA Book and in the Values Book.

c. Note in Sample Status Book that LTA has been done.

Procedure for Lignite and Subbituminous Coals
(Modified from Given and Yarzab, 1978)

4.5B.1. Preliminary

a. Obtain the -60 mesh split marked or LTA.

b. Further pulverize sample to -100 mesh with mortar and pestle.

c. Place in bottle and label.

d. Zero check balance.

e. Weigh out 3g of sample in a clean, preweighed petri dish (dishes are cleaned, oven dried, and desiccated at least 24 hours prior to use).

f. Dry sample in oven for one hour at 105°C.

g. Zero check balance.

h. Weigh sample and dish and calculate moisture.

i. Mix and stir sample for 1-24 hours (depending on coal) with 1 N ammonium acetate (pH7).

j. Reweigh to determine weight loss.

4.5B.2 Ashing Procedure

Same as for Anthracite and Bituminous coals

4.5B.3 Stirring and Weighing Sample

Same as for Anthracite and Bituminous coals

4.5B.4 Record Keeping

a. Record % moisture in LTA Book.

b. Record final ash % in LTA Book and the Values Book.

c. Note in Sample Status Book that LTA has been done.

4.5B.5 **Safety Procedures**

a. Oxygen Tank

- **secure oxygen tank with a chain and tank holder to a wall**
- **never increase tha flow of oxygen over 2.0 cc**

b. 1 N Ammonium Acetate

- **avoid skin end eye contact**
- **rubber gloves must be used**
- **organic vapor respirator must be used**

4.5C High Temperature Ashing (HTA) (ASTM D3174, 1983c)

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

4.5C.1 Preliminary

a. Obtain the -60 mesh split marked for HTA.

b. Place samples into petri dishes and then into an oven for one hour at 105°C.

c. Place clean crucibles (17ml) into oven with samples.

d. After an hour remove sample and crucibles; place into desiccator and let cool to room temperature.

d. Zero check balance.

e. Weight crucible and record in HTA Data Book as dish weight (D).

f. Place about a gram of sample into crucible and record as dish and sample weight (DS).

4.5C.2 Ashing

a. Place sample into cold furnace (i.e. room temperature, about 25°C).

b. Turn on fan for furnace.

c. Heat furnace to 500°C within first hour (8°C per min.)

d. heat furnace to 750°C in the second hour (4°C per min).

e. Maintain temperature at 750°C for 60 min).

f. Turn off furnace.

g. Let furnace cool to 400°C.

h. Remove sample and place in desiccator (Note: make sure nothing in desiccator is flammable).

i. Let cool to room temperature (about 8-12 min.).

j. Zero check balance.

k. Weigh crucible and ash, then record in ledgar as dish and ash weight (DA).

l. Calculate ash: (DA-D/DS-D) x 100 = % Ash.

4.5C.3 Record Keeping

a. Record in HTA Book % Ash.

b. Record Final % in Values Book under HTA %.

c. Note in Sample Status Book that HTA was performed.

4.5C.4 Suggestions

a. Input for Programmable Furnace

- Initial rate (1st hour) . . . . . $8^{o}C$/min.
  Transition temperature . . . . . $500^{o}C$
  Hold time . . . . . 0 min.
  Final rate (2nd hour) . . . . . $4^{o}C$/min.
  Final temperature . . . . . $750^{o}C$
  Final hold time . . . . . 60 min.

b. Repeatability (%)

- In Lab:
  No carbonates 0.2
  Carbonates present 0.3
  Coals with more that 12% ash
  with carbonate and pyrite 0.5

- Between Labs:
  No carbonates 0.3
  Carbonates present 0.5
  Coals with more that 12% ash
  with carbonate and pyrite 1.0

4.5C.5 **Safety Procedures**

Operating Furnace

- **safety glasses/goggles must be worn**
- **laboratory coat should be worn**
- **when removing sample, heat resistant gloves must be worn**

4.5D Volatile Matter Determination (ASTM D3173, 1983d; D3175, 1983e)

Equipment	platinum/fused quartz crucibles analytical balance, sensitivity at least 0.0001g electric muffle furnace tongs oven capable of reaching and maintaining 105°C hydrofluoric acid, (HF), 10% Volatile Matter Book
Safety Equipment	heat resistant gloves laboratory apron and coat safety glasses/goggles fume hood heavy rubber gloves organic vapor respirator

4.5D.1 Preliminary - Moisture Determination (ASTM D3173, 1983d)

a. Zero check balance.

b. Weigh out approximately one gram of -60 mesh coal.

c. Place sample in a preweighed, dried, and cleaned platinum or fused quartz crucible (Note: If using the fused quartz crucible NEVER touch with bare hands, ALWAYS use tongs or lens paper.

d. Place in heating oven for one hour at 105°C.

e. Reweigh and determine % moisture using the following equation:

$$\% \text{ Moisture} = \{(A-B)/A\} \times 100$$

where: A = grams of sample used, and
B = grams of sample after heating

4.5D.2 Volatile Matter Determination (ASTM D3175, 1983e)

a. Heat muffle furnace to 950°C + 20°C (Note: It is best to heat furnace to near 970°C to compensate for rapid heat loss when opening furnace door).

b. Place crucible with top in place into heated furnace.

c. After 2-3 min. inspect crucible to verify that the lid is still properly seated (if necessary, reseat the lid).

d. After heating for exactly 7 min., remove crucible from furnace and without disturbing lid, allow to cool in desiccator.

e. Zero check analyticel balance.

f. Weigh as soon as cold

g. Determine % volatile matter as follows:

% volatile matter = C-D

where: C = % weight loss and
D = % moisture

4.5.3 Cleaning of Fused Quartz Crucibles

BEFORE USING HF... READ PAGES 36 - 39.

a. **Prepare a solution of 10 % hydrofluoric acid. (Note: Hydrofluoric acid is Extremely Dangerous, use every precaution to assure safety as outlined on page 29. If you have never used this acid before, seek help initially).**

2. **Rinse crucible in hood in dilute HF to remove carbon deposits.**

3. **Continue rinsing for 3-5 min. (or until clean) followed by a thorough rinsing with deionized water and complete drying.**

4.5.4 Record Keeping

Record volatile matter determination in Volatile Matter Book and in Values Book.

4.5D.5 Suggestions

a. **Washed and rinsed crucibles must be dried as quickly as possible. Water droplets left on the crucible's surface will accumulate atmospheric contaminants that are likely to cause localized devitrification.**

b. **Temperature regulation during the procedure is critical; maintain it as exactly as possible.**

c. **In-lab repeatability should be as follows:**

Anthracite	0.3%
Semianthracite and Bituminous	0.5%
Subbituminous	0.7%
Lignite and Peat	1.0%

4.5D.6 Safety Procedures

a. Handling Hydroflouric Acid
- WARNING: Hydrofluoric acid is an extremely hazardous chemical at any concentration or temperature. Physical contact will cause serious burns and inhalation of the vapors will irritate the respiratory system. Use all necessary measures to prevent physical contact with the acid. Consider any accident with hydrofluoric acid a serious accident. -READ PAGES 36 - 39.
- heavy rubber gloves, safety glasses/goggles, laboratory coat, and rubber apron must be worn at all times
- organic vapor respirator must be worn
- must be used under a fume hood

B. Operating Furnace
- safety glasses/goggles must be worn
- laboratory coat and/or apron should be worn
- when removing samples, asbestos gloves must be worn

4.4E X-Ray Radiography

Equipment	X-ray radiography unit band saw or rock saw masking tape (wide) marker pen X-ray film (e.g. instant positive transparent radiographic 8 x 10 film) instant developing unit 13" x 11" plastic core tube holder 13" PVC (3" diam.) or acrylic (2 1/4" diam.) core tubes
Safety Equipment	safety glasses/goggles ear protection laboratory coat and/or apron radiation badge dust respirator

4.4E.1 Preliminary

a. If the core is not received in plastic tubes, carefully place it into appropriate sized tubes.

b. For 2 1/4" core, place into split acrylic tubes. Seal ends with plastic caps and mark orientation on tube (i.e. the "up" arrow).

c. For 3" core place into a half tube of PVC and tape core into place; note orientation on tape and core.

4.4E.2 X-Ray Radiography

a. Place closed 2 1/4" core tubes or taped 3" half tubes into plastic core tube holder.

b. Tape two pieces of instant X-radiograph film together so they measure 15 1/4" x 11 1/2".

c. Place film in chamber at the appropriate level.

d. Position plastic tube holder with tubes over X-radiograph film.

e. Secure all portals to chamber area with lead partition.

f. Close main door to chamber area and place lead partition around unit.

g. Set unit's controls (see table below).

h. Step back from X-ray machine until exposure has been made, remove tubes.

i. When exposure has been made, remove tubes.

j. Process film through instant processor according to instructions.

4.4E.3 Core Cutting

a. For 2 1/4" cores, remove end caps and half of the split tube; tape exposed core and note orientation on the taped portion.

b. Cut the 2 1/4" taped cores using band saw.

c. Cut the 3" cores (already taped) using band saw.

d. Annotate radiograph using observations that can be seen both on the cut surface and the radiograph.

e. If samples are taken, note sample intervals on radiographs.

4.4E.4 Suggestions

a. Sequence for turning on X-ray radiograph unit

1) turn on plug switch near socket
2) turn key to "on" position
3) select MA setting
4) slowly turn KV knob to desired setting
5) to turn off unit, first, slowly turn KV knob to zero
6) turn key to "off" position
7) turn off plug switch.

b. X-Ray Exposure Table:

coal[1] contrast typed	core diameter	KV	MA	exposure time (min.)	source to sample distance (in.)
Med High	3"	60	3	24	40
Med High	2 1/4"	60	3	12	40
Med High	slabbed (1/4")	60	3	2	16
Low	slabbed (1/4")	40	3	12-15	16

[1]Refers to amount of contrast between the "lighter" (less dense) and "darker" (more dense) components in the coal.

4.4E.5 Safety Procedures

a. X-Ray Unit

- X-ray badge and ring must be worn at all times
- all portals must be sealed off before turning on X-rays
- lead shield must be in front of door before turning on X-rays
- never open door while X-rays are on
- never stand close to machine while X-rays are on
- hava machine periodically checked for radiation leaks

b. Band Saw or Rock Saw

- dust respirator and safety glasses and ear protection must be worn
- laboratory coat and/or apron should be worn
- keep fingers and clothing away from blade and other moving parts
- never force the semple to feed too fast
- ear protection should be worn with the band saw
- make sure the proper blade is being used

4.4F Vitrinite Reflectance (ASTM D2798, 1983f)

Equipment . . . . . petrographic microscope with reflected light capabilities, capable of 500X magnification, photomultiplier with peak picker, and rotating circular stage
immersion oil, nondrying, refractive index 1.515 - 1.5.19
sample-leveling press
modeling clay
glass reflectance calibration standards
glass slide (2"X1")

4.4F.1 Setting-up the Microscope
(Numbers refer to figure 4)

a. Turn on:
large digital voltmeter (1)
small digital voltmeter (2)
lamp power supply (3)
high voltage power supply (4)
stable power supply (5)

b. Increase Current to:
lamp power supply (to highest setting) (6)
stable power supply (to 8.25 amps) (7)

c. Switch on peak picker (8)

s. Check object lens type (50X.85p) (9).

f. Push out half-stop(10).

g. Push in slide and check that number "8" is displayed (11).

h. Pull out MPV 2 (12).

i. Switch condenser out (line up knob with reference line) (13).

j. Check to see that spectral wedge is out (rotate dial to red dot) (14).

k. Place switch towards illustrated hand (15).

l. Place switch down for through-the-eye-piece-viewing during metering (16).

m. Push red lighted button to engage high voltage power supply (17).

n. Place glass reflectence calibration standard under scope and pick a reference glass using the coordinates for calibration matrix.

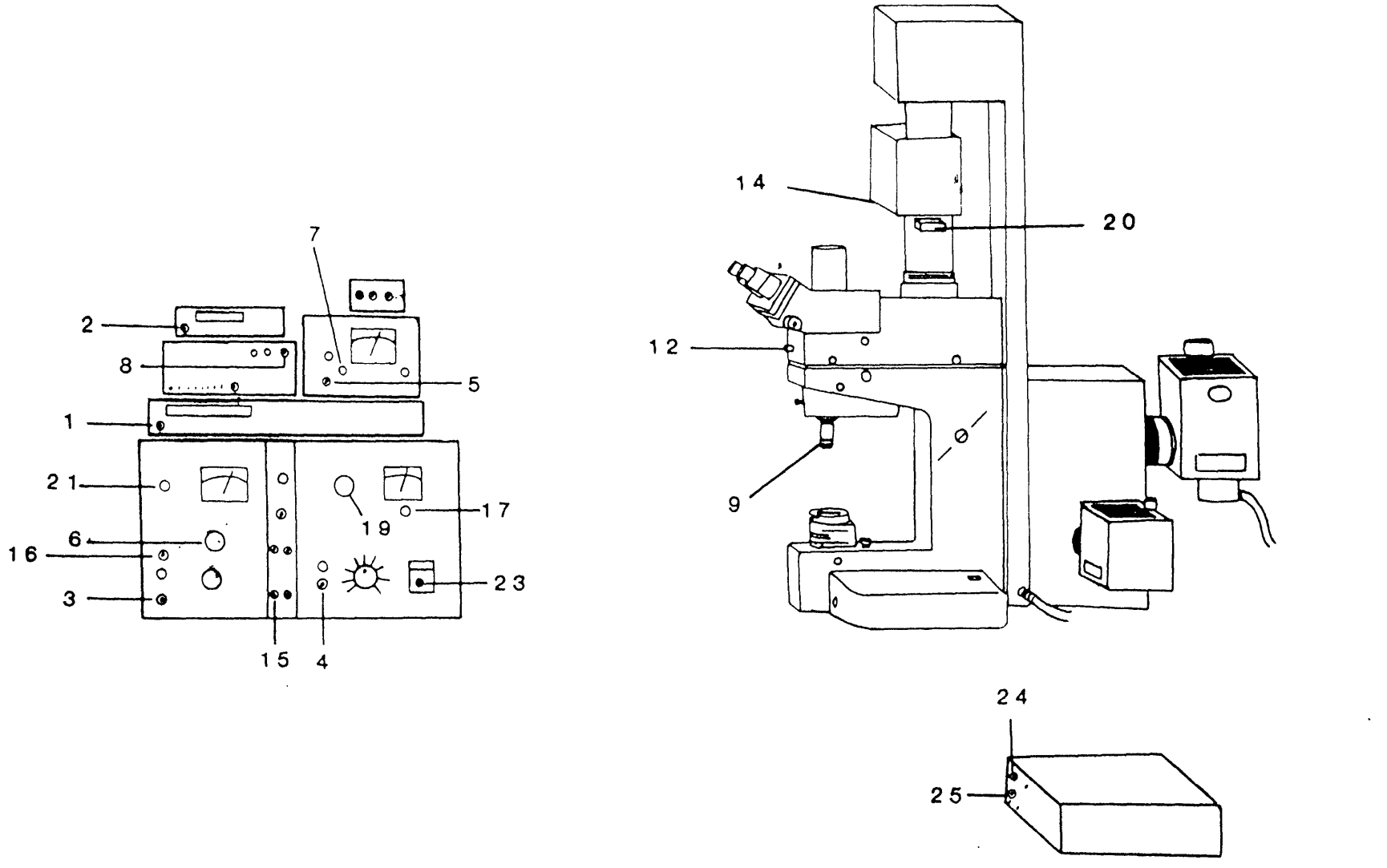
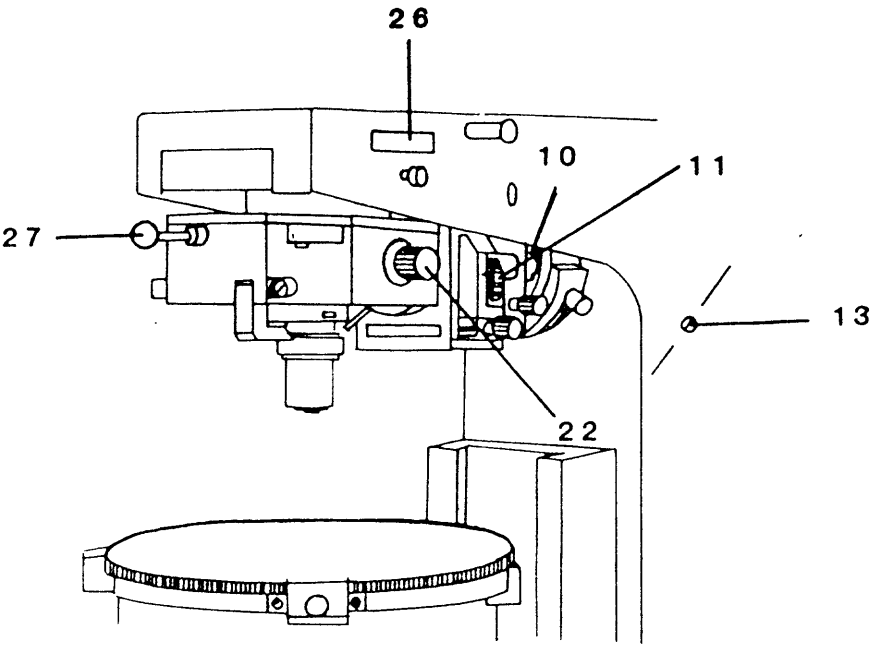


FIGURE 4: Diagram of microscope set-up (refer to text for explanation)



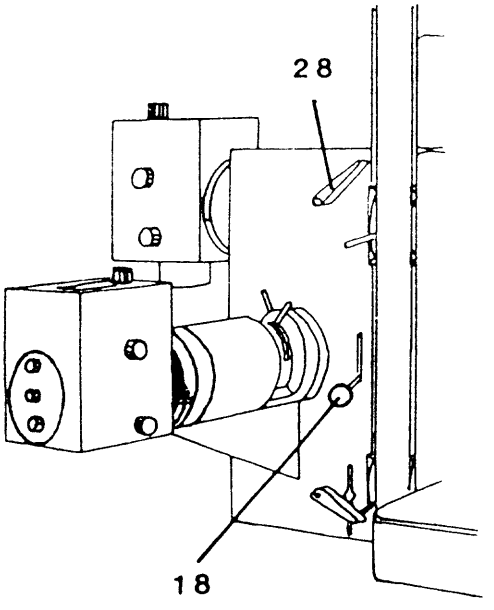


FIGURE 4: (cont'd)

GLASS *	1	2	3	4	5	6
1	0.300	0.508	0.911	1.024	1.351	1.649
2	0.306	0.496	0.906	1.022	1.360	1.654
3	0.304	0.504	0.921	1.007	1.363	1.650
4	0.307	0.508	0.924	1.039	1.386	1.670
5	0.324	0.554	0.928	1.049	1.413	1.703
6	0.307	0.509	0.914	1.028	1.361	1.661
Calibration Values	0.306	0.509	0.923	1.020	1.372	1.656
Stage Coordinates	4x116	4x109	4x100	13x116	13x109	13x100

DATE_____ BY_____

FIGURE 5: Example of a calibration matrix

o. Engage measuring square (18).

p. while watching the objective and calibration standard, focus down until the two become very close.

q. While looking through the eye piece, focus up until measuring square is sharp and defined.

r. Check volt meters to see that both read zero and adjust dark current if necessary (19).

s. Engage filter S542-21 (20).

t. Engage photometer by pushing foot switch--red light should turn on (21).

u. Let machine warm-up for 20-30 min.

4.4F.2 Centering and Calibrating Instrument

a. Turn off overhead lights.

b. After machine has warmed-up, level a sample and focus as before.

c. To assure sharp focus of measuring square, focus on sample and engage focusing square. Both sample and measuring square should be sharply in focus. If not adjust the focusing square (22).

d. To center objective,
   -place cross-hair over a small object (e.g. pyrite, micrinite)
   -rotate stage and note path of object
   -center accordingly.

e. To calibrate photometer,
   -place glass calibration standard under scope and focus as before
   -move to a reference glass using coordinates from matrix
   -clear peak picker by pressing in track button (light will blink) then release
   -rotate stage
   -if voltage does not agree with matrix-calibration value, adjust voltage (23)
   -clear peak piker and rotate stage again; continue this until photometer is calibrated to reference glass
   -move to another reference glass and calibrate as before
   -return to first reference glass and check reading; if into adjustment is necessary, instrument is calibrated; if adjustment is needed, then proceed as before until both glasses are calibrated.

4.4F.2 Measurement of Vitrinite Reflectance

a. Place leveled sample under microscope and focus as before.

b. Systematically transects the pellet measuring only the structureless vitrinites (telocolinite) that are scratch-free and pyrite-free.

c. Whenever a particle of telocollinite is found, center on a scratchless area.

d. Focus sharply and engage measuring square (mirror housing lever).

e. Clear peak picker.

f. Rotate stage (by pressing foot pedal that engages motor).

g. Record maximum reflectance.

h. Continue measurements until 25 counts are recorded.

i. Check calibration.

j. Measure another 25 particles on this mount; then check calibration.

k. Measure 50 counts on next pellet (a total of 100 counts for one sample).

4.4F.3 Preparation of Calibration Matrix

a. Set up microscope according to steps 1 to 20 in A and 1 to 4 of B.

b. Place glass calibration standard on stage.

c. Center objective onto the first glass.

d. Record position coordinates on stage.

e. Clear peak picker.

f. Rotate stage.

g. Recore value--adjust voltage until calculated value of glass No. 1 is read as peak.

h. Read and record both position and peak of all other glasses without any adjustment of voltage.

i. Enter each value into row 1 of matrix (figure 5).

j. Return to coordinates of glass No. 2 and adjust voltage to read calculated value of glass No. 2.

k. Move stage to coordinates of each glass and measure the maximum peak on each value on next row of matrix (figure 5).

l. Repeat steps 10 and 11 until matrix is completed.

m. Throw out values associated with highly inconsistent readings (greater that 0.2% difference) (such as glass 5 in figure 5).

n. Average all other readings except the adjusted reading (underlined in fig. 5).

o. The average will be the calibration value to be used at the specified coordinate of each glass.

4.4G Maceral Analysis (ASTM D2799, 1983g; ICCP, 1963)

Equipment . . . . . petrographic microscope with reflected light capabilities, 500X with eye piece with cross hair and mechanical stage point counter
immersion oil, nondrying, refractive index 1.515 to 1.519
sample leveling press
modeling clay
2" X 1" glass slides

4.4G.1 Setting-up the Microscope
(Numbers refer to figure 4)

a. Turn on:
lamp power supply (3)
mercury vapor lamp (24).
(for blue-light (fluorescence) analysis only)

b. Increase current to lamp power supply (to highest setting) (6).

c. Ignite mercury vapor lamp (25).

d. Check object lens type (50X.85p) (9).

e. Engage half stop (10).

f. Switch condenser out (line up knob with reference line) (13).

g. Push on MPV 2 (12).

h. Level sample using modeling clay and sample leveling press.

i. Secure mechanical stage on pletform.

j. Place immersion oil on pellet face and place under objective on the mechanical stage.

k. While watching the objective and pellet, focus down until the two become quite close.

l. While looking through the eye piece, focus up until the image is sharp and clear.

m. For blue-light (fluorescence) analysis engage:
yellow filtration (26)
blue filtration (27)
fluorescing light source (28)

4.4G.2 Anthracite and Bituminous Maceral Analyses

1. For anthracite and bituminous coals use the classification sheet displayed in figure 6.

2. Perform 500 point counts using white-light and classification in section A of figure 6.

3. If fluorescing macerals are present, perform 500 counts on the sample pellet using the blue-light classification (section B of figure 6).

4. Finally, perform 500 counts on the etched pellet ("c" pellet) using white-light and the classification for etched macerals (section C of figure 6).

5. Recalculate and combine the analyses and record in section D of figure 7.

4.4G.3 Lignite and Subbituminous Maceral Analyses

a. For lignite and subbituminous coals use the classification sheet displayed in figure 7.

b. Perform 500 point counts using white-light and the classification in section A. of figure 7.

c. Perform 500 counts on the same pellet using the fluorescing light source and the classification in section B of figure 7.

d. finally, perform 500 counts on the etched pellet ("c" pellet) using white light and classification for etched macerals (section C. of figure 7).

e. Recalculate and combine the analyses and record in section D of figure 7.

4.4G.4 Safety aspects

Never, ever, look directly at the blue light illumination without the barrier filter in place.

U.S.GEOLOGICAL SURVEY COAL LAB

**********BITUMINOUS COAL DATA SHEET**********

A. WHITE LIGHT ANALYSIS

MACERAL	(A1)	(A2)	(A3) Totals (A1+A2)	
Vitrinite----				
Vitrodet.____				(A4)_
Sporinite----				(tot. Vitrinite)
Cutinite-----				
Resinite-----				(A5__
"Resodet." __				(tot. Liptinite)
Alginite-----				
Liptodet.----				
Fusinite ----				
Semifusinite-				
Micrinite----				(A6)-
Macrinite----				(tot. Inert.)
Inertodet.---				
Sclerotinite-				

****

D. SUMMARY OF DATA

0.001(B6 - A6) = D1

0.001(____ - ____ = _____

WHITE LIGHT TOTALS	MACERAL VARIETY	CRYPTO C5 X D1 =	REVISED %	REPT %	
	Telinite	____X____			
	Corpo in telo	____X____			Recal.
_____	Desmocollinite	____X____			- From
	Detrocollinite	____X____			Analysis
	Corpo in desmo	____X____			C
	Pcoll in desmo	____X____			
_____	Sporinite	__/////__			
_____	Cutinite	__/////__			
_____	Resinite	__/////__			
_____	"Resodet."	__/////__			From
_____	Exsudatinite	__/////__			- Analysis
_____	Fluorinite	__/////__			B
_____	Bituminite	__/////__			
_____	Alginite	__/////__			
_____	Liptodetrinite	__/////__			
_____	Fusinite	__/////__			
_____	Semifusinite	__/////__			
_____	Micrinite	__/////__			From
_____	Macrinite	__/////__			- Analysis
_____	Inertodet.	__/////__			A
_____	Sclerotinite	__/////__			

PELLET ANALYSTS Lta_____ SET_____

Hta _____

A. by _____ Density _____ SAMPLE NO._____

Pyr S _____

B. by _____ Tot S _____ DATE _____

FIGURE 6: Maceral classification sheet for high-rank coals

C. USGS DATA SHEET FOR ETCHED PELLETS

MACERAL (crypto)	(C1) by	(C2) by	(C3)Total [1+2]	(C5)=[C3÷C4]*100 tot ea/tot vit.
Telinite				
Corpo in telinite				
Desmocollinite				
Detrocollinite				
Corpo in desmo				
Poricoll.in desmo				
all others				XXXXXXXXXXX

(C4)Total Vitrinite Counts____

****

B. BLUE LIGHT ANALYSIS

MACERAL	(B1)	(B2)	B3=B1+B2	comments
Sporinite				
Cutinite				
Resinite				
"Resodetrinite"				
Exsudatinite				
Fluorinite				
Bituminite				
Alginite				
Liptodetrinite				
Vit + INERT.			(B6)	

(B4)TOTAL LIPTINITE CTS_____

SAMPLE NO._____

FIGURE 6: (cont'd)

U.S.GEOLOGICAL SURVEY COAL LAB
********** LOW RANK COAL DATA SHEET**********
A. WHITE LIGHT ANALYSIS

MACERAL	(A1)	(A2)	(A3) Totals (A1+A2)	
Huminite-----				(A4) (tot. huminite)
Sporinite ___				
Cutinite-----				
Resinite-----				
"Resodet."___				
Suberinite---				(A5)- (tot Liptinite)
Alginite-----				
Liptodet.----				
Fusinite ----				
Semifusinite-				
Micrinite----				(A6)- (tot. Inert.)
Macrinite----				
Inertodet.---				
Sclerotinite-				

****

D. SUMMARY OF DATA

0.001(B6 - A6) = D1
0.001(____-____) = ____

WHITE LIGHT TOTALS	MACERAL VARIETY	CRYPTO C5 X D1 =	REVISED %	REPT %	
	Humotelinite	____X____			
	Eucorpo/telo	____X____			
	Poricorpo/telo	____X____			Recal. From Analysis C
	Eugelinite	____X____			
	Porigelinite	____X____			
	Detrogelinite	____X____			
	Eucorpo/gelo	____X____			
	Poricorpo/gelo	____X____			
	Sporinite	__////___			
	Cutinite	__////___			
	Resinite	__////___			
	"Resodet."	__////___			
	Suberinite	__////___			From
	Exsudatinite	__////___			Analysis
	Fluorinite	__////___			B
	Bituminite	__////___			
	Alginite	__////___			
	Liptodetrinite	__////___			
	Fusinite	__////___			
	Semifusinite	__////___			
	Micrinite	__////___			From
	Macrinite	__////___			Analysis
	Inertodet.	__////___			A
	Sclerotinite	__////___			

PELLET ANALYSTS
A. by _____
B. by _____

Lta _____
Hta _____
Density _____
Pyr S _____
Tot S _____

SET _____
SAMPLE NO. _____
DATE _____

FIGURE 7: Maceral classification sheet for low-rank coals

C. USGS DATA SHEET FOR ETCHED PELLETS

MACERAL (crypto)	(C1) by	(C2) by	(C3)Total [1+2]	(C5) =[C3÷C4]*100 tot ea/tot hum.
Humotelinite				
Eucorpo in telo				
Poricorpo in telo				
Eugelinite				
Porigelinite				
Detrogelinite				
Eucorpo/gelo				
Poricorpo/gelo				
all others				XXXXXXXXXXXX

(C4)Total Huminite Counts_____

****

B. BLUE LIGHT ANALYSIS

MACERAL	(B1)	(B2)	B3=B1+B2	comments
Sporinite				
Cutinite				
Resinite				
"Resodetrinite"				
Suberinite				
Exsudatinite				
Fluorinite				
Bituminite				
Alginite				
Liptodetrinite				
HUM + INERT.			(B6)	

(B4)TOTAL LIPTINITE CTS_____

SAMPLE NUMBER _____

FIGURE 7: (cont'd)

5 EMERGENCY PROCEDURES AND PRECAUTIONS

5.1 Eye Wash Station

The eye wash station should be positioned in the laboratory free of 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.

5.2 Fire Extinguisher

The fire extinguisher should be located free of obstructions within the laboratory. The correct type of extinguisher (CO_2, water air pressure, dry chemical, etc.) should be used depending on type of hazard that exist in the laboratory. Before using dial X 7222, if no answer, 9–911 to report fire then extinguish.

5.3 First Aid

A first aid box consisting of band–aids, antiseptic, bandages, etc., should be placed in an easily accessible place in the laboratory. Any accident should always be reported to a supervisor regardless of how serious.

5.4 Acid Spills

In case of acid spills the emergency number is X 7222 and should be called IMMEDIATELY—DO NOT ATTEMPT TO CLEAN UP YOURSELF.

6 ACKNOWLEDGMENTS

Many people have helped to develop and improve the procedures in this manual. We wish to thank particulary Derek Widmayer, Leslie Ruppert, Peter Swallow, and Philippa Benson who contributed to the refinement of these sample preparation procedures. Also, our thanks to Brenda Pierce who helped in the drafting and reviewing of this manual. Through the years other industry and university personnel have through conversation and their gracious hospitality influenced many of the details in our procedures. These individuals include: Dr. J. J. Renton, Mr. Ralph Gray, Dr. Alan Davis, Dr. William Spackman, Dr. Jack Crelling and Mr. William Grady.

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