

Acme Coke
11236 S. Torrence Ave.
Chicago IL 60617



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“Procedure for Preparing Coal Pellets
for Petrographic Analysis”

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Preliminary

Enter all incoming coal samples in the log book.

Check the coal for excessive moisture. If the coal feels wet and agglomerates easily, dry it at 105°C for 2 hours or air dry it overnight. The coal must be spread out on a tray for drying.

Clean six 1-inch pellet molds and coat the surfaces that slide or contact the pellets with MS-122 Release Agent Dry Lubricant (Miller-Stephenson Chemical Co., Inc. Morton Grove, Ill.) The coating is white and takes about 20 minutes to dry.

Turn on the exhaust hood over the grinder. Blow the grinder interior and the small riffle and accessories clean with compressed air and wipe with a clean towel. Clean the 20 mesh screen with a brush. All equipment must be thoroughly clean to prevent contamination.

Riffling, Screening and Grinding

Note: Riffle, screen and grind one sample at a time. Note the order in which samples are riffled and ground as a check on possible contamination in the process.

Quarter the 1-gallon sample of rail coal in the large riffle to obtain a 1-quart sample. Return the 3-quart fraction to the can. Mixer bin samples are received in quart cans.

If a substantial amount of the coal is plus 1/4 inch in size, grind the coal at the 10 notch setting on the grinder before proceeding. (Close the grinder, then back off 10 notches).

Pour the 1-quart sample of coal into the small riffle. If any lumps are too large to pass thru, break the lumps with a hammer outside the riffle, re-mix the sample and then riffle again. Riffle twice to quarter the sample. Save the 3/4 size fraction in the receiving can, continue sample preparation with the 1/4-size fraction.

Screen the minus 20 mesh (-20M) coal from the quartered sample. Grind the +20M coal and screen successively until all of the coal is -20M. Always grind only the +20M fraction. At the start of grinding adjust the grinder to the closed position, then back off 4 notches. Successively grind at the 4, 3, 2, and

1 notch settings. Depending on the coal, the sample may have to pass the grinder 3 to 10 times. Always pour the coal into the grinder slowly to prevent jamming of the grinder or tripping the circuit breaker (breaker No. 22).

Sample Size Reduction

Mixer Bin and Rail Samples. Riffle the -20M coal three times, reducing the sample to 1/8-size. (If the quart can was less than 2/3 full, riffle only twice at this stage to reduce the sample to 1/4-size instead of 1/8-size). Save the larger size fraction in a plastic bag; store in the quart or gallon can with the raw coal sample. Use the smaller size fraction for pellet preparation. This sample is sufficient to prepare one pellet. Place the smaller size fraction in a paper cup and write the name, date and source of the sample on the cup immediately. Place the sample in the covered carrying container. The -20M coal reaches the 20-30 ml mark on the Buehler Ltd. paper cups, depending on how full the original sample can was.

Blend Samples: Riffle the -20M coal two times, reducing the sample to 1/4-size. (Riffle only once if the quart can was less than 2/3-full). This is enough to make two pellets. Save the larger size fraction in a plastic bag; store in the can with the raw coal sample. Place the smaller size sample in a paper cup and write the name, date and source of the sample on the cup immediately. Place the sample in the covered carrying container.

Clean the grinder, riffle, pans and screen thoroughly before proceeding to the next sample. Improper cleaning will cause contamination and give erroneous test results. Clean the grinding area after all samples are ready for pelletizing.

Pelletizing

Note: Disposable plastic gloves should be worn to protect the hands from the epoxy binder.

Insert the bottoms into the molds that were coated with MS-122 Release Agent Dry Lubricant.

Mix binder for a maximum of 4 pellets. The 2-part epoxy binder doesn't provide enough time to make more pellets from one batch. Normally pellets are prepared in sets of 6; therefore, work them up in 2 sets of 3 pellets each.

Mix 40 ml of APCO 5313 Part A with 4.0 ml of APCO 5313 Part B (Applied Plastics Co., Inc., El Segundo, Calif.). Mix 5-10 ml of this 2-part binder thoroughly with the coal sample. Start with about 5 ml and add more if needed to wet all of the coal. Use a tongue depressor for mixing the coal with the binder. Do not use more binder than is necessary to wet all of the coal.

Using the tongue depressor, add the coal-binder mixture to the pellet mold, tamping down with the marked steel rod. Add coal-binder mixture to the mold until the mark on the steel rod reaches the top of the mold when the rod is pressed against the coal-binder mixture. Clean the rod thoroughly with a towel to prevent contamination of the next sample, insert the mold top into the mold, and immediately place a sticker label on the side of the mold to identify the sample.

Place the mold in a specimen mount press and apply a load of 10,000 psi. Let it stand under load for 5 minutes. During this time, mix two more coal samples with binder and place in other presses. After 5 minutes release the pressure, clamp the pellet mold assembly together as tight as possible manually with a 6-inch "C" clamp and place the clamp-mold assembly in an oven at 110°F for 90 minutes.

Repeat this pelletizing procedure for the second set of coal samples.

Remove the clamp-pellet mold assembly from the oven, release the clamp and push the pellet from the mold using the specimen mount press. Be sure the pellet does not fall to the bench top where it could break; catch it just below mold level. Release one pellet at a time and transfer the identification sticker from the mold to the side of the pellet immediately to avoid confusing the pellets.

When all pellets have been released from the molds, engrave the sample identification on the top and the side of the pellet using the vibrating engraver. Remove the sticker label from the pellet and discard. The end without the engraving will be polished.

Grinding and Polishing

If necessary, grind the pellet surface to be polished on the belt sander using water and a 240 grit wet/dry paper holding

the pellet by hand. Hold the pellet as level as possible. Grind to remove any air gaps and to make the pellet height approximately 3/4-inch for the Automet polishing holder. Maximum height for the Automet holder is 7/8-inch. Shorter pellets are usable if shim tabs are used to set equal pressure on the pellets in the polishing holder assembly. Round off the edge of the pellet on the side to be polished to prevent tearing of the polishing cloths and possible chipping of the pellet.

Mount the pellets in the polishing holder. If fewer than 6 pellets were prepared, use old pellets to fill the remaining pellet spacings for proper balance and load distribution. Note: the standard 1-inch pellet holder has to be drilled out to 1.014 inch to accommodate the coal pellets.

Place the pellet holder with pellets in the Automet polisher that has a speed of 550 RPM. Use water and 325 grit paper to obtain flat polishing surfaces. Start at a pressure setting half-way between the first and second lines from the bottom (10 lb.); slowly increase pressure towards the second line (20 psi), depending on the amount of grinding needed. Check after 2 minutes; samples normally require 2 to 5 minutes. Rinse the samples and polishing wheel in tap water. The 325 grit paper usually can be used for two sets of samples.

Replace the 325 grit paper with 600 grit paper on the same 550 RPM polishing unit. Using water and a pressure setting on the second mark (20 lbs.), grind for 2 minutes to produce a smoother surface for polishing. Rinse the samples and polishing wheel in tap water. The 600 grit paper usually can be used for two sets of samples.

Clean the wheel covered with Texmet polishing cloth with running tap water and a soft-bristle brush. Using a speed of 550 RPM, polish the pellets for one minute using Buehler Al₂O₃ 5 micron holding the pellet assembly in hand. Apply a pressure of about 10 pounds. Rinse the pellets in tap water, insert into the ultrasonic cleaner containing deionized water for 10 seconds, then rinse with deionized water. Clean the polishing wheel with tap water and a soft-bristle brush. The Texmet polishing cloth can be used for an extended period of time; it is good for 25 to 50 sets of pellets.

Clean the wheel that is covered with Metcloth overlaid with selected silk using running tap water and a soft-bristle brush.

Rinse with deionized water. Using a speed of 130 RPM, polish the pellets using Buehler A2 0.3 micron Al_2O_3 that has been diluted with 3 parts of deionized water. Start with the pressure on the second line from the bottom (20 lb.), increasing the pressure to half-way between the second and third lines (30 lb.) midway through the 5 minute polishing cycle. Always keep the polishing cloth wet during polishing. Use about one-half of a bottle (4-5 fluid ounces) of the diluted Al_2O_3 ; do not use running tap water. Clean the pellets using tap water, 10 seconds in the ultrasonic cleaner and a deionized water rinse.

Check the pellets for scratches and pits under the Unitron microscope (150X). Occasional scratches and pits are acceptable. If numerous scratches or pits are visible, repolish using Buehler A2 0.3 micron Al_2O_3 . If the pellets are acceptable, clean the polishing wheel with tap water and a soft-bristle brush followed with deionized water. The polishing wheel can be reused until the silk tears.

Clean the wheel that is covered with (1) Microcloth, (2) Metcloth, and (3) selected silk using running tap water and a soft-bristle brush. Rinse with deionized water. Using a speed of 130 RPM, polish the pellets using Buehler B3 0.05 micron Al_2O_3 that has been diluted with 3 parts of deionized water. Set the pressure on the second line from the bottom (20 lb.) and maintain that pressure throughout the 2 minute polishing cycle. Use about one-fifth of a bottle (2 fluid ounces) of the diluted Al_2O_3 to keep the polishing surface wet during polishing; do not use running tap water. Clean the pellets using tap water, 10 seconds in the ultrasonic cleaner and a deionized water rinse.

Check the pellets for scratches under the Unitron microscope (150X). If more than occasional scratches are visible, repolish using Buehler A2 0.3 micron Al_2O_3 and then Buehler B3 0.05 micron Al_2O_3 as described above. If the pellets are essentially free of scratches they are ready for the petrographic microscope. Place them in a desiccator.

Clean the polishing wheel using running tap water and a soft-bristle brush followed with a deionized water rinse. Remove the accumulated Al_2O_3 from the bowls beneath the polishing wheels. Clean up the pellet preparation area and put the unused portions of the coal samples in storage.

A. Romaine