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Work Instruction: Coal/coke testing

Dated: 1996



# Acme Steel Company

## Work Instruction

STATUS: Revised

DEPARTMENT: Petrographic Laboratory  
 TITLE: Preparation of a Coal Pellet for Reflectance Measurement (Coal Preparation)  
 EFFECTIVE: June 10, 1996

Number: PLWI:002

Pages: 2

**Purpose** To define how a coal pellet is prepared and polished.

**Scope** Applies to all incoming coals and new coals to be evaluated.

| Step | Description of Work Activity  | Record of Activity |
|------|---|--------------------|
| 1    | Much of this work instruction is based on ASTM D 2797.  |                    |
| 2    | Air-dry a representative sample of the coal.  | Label tray         |
| 3    | Crush the sample using the grinder until it passes a No. 20 screen.   |                    |
| 4    | Form the sample into a pellet using epoxy binder , a steel mold and a pellet press at 10,000 ±1,000 lb. for at least one minute. The sample must be held under pressure overnight either in the press or in a large "C" clamp to cure at room temperature. Alternatively, a sample in a "C" clamp may be cured in two hours in an oven set at 60°C. | Label mold         |
| 5    | Press the pellet from the mold and engrave the coal identity on the surface using a vibrating marker.   | Engraved pellet    |
| 6    | Grind one end of the pellet step by step using sand papers of 240 grit, 320 grit, 400 grit and 600 grit and the automatic grinder/polisher. Grinding times are 30-60 seconds for each step.   |                    |
| 7    | Polish the pellet on fabric support step by step using alumina slurries of 1 micron, 0.3 micron and 0.05 micron particle sizes and the automatic grinder/polisher. Polishing time is approximately one minute for each step.  |                    |
| 8    | The pellet now has a flat scratch-free surface and is ready to be analyzed.   |                    |
| 9    | Store the pellet in one of the desiccators before analysis and in a pellet storage cabinet after analysis.  |                    |



**Measures**

No. 20 screen size, hydraulic pressure in lb.

**Records**

| Rev# | Rev Date | Reason for Document Change  |
|------|----------|---|
| 001  | 10/21/96 | Name keyed title to process flow chart.<br>Removed engraved pellet as record. |

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| Revision #: 001  | Revision Date: 10/21/96                                    | Author: C. Lin & V. Beaucaire                        |
| Last Review Date: 10/21/96                               | Date of Next Review:<br>07/01/97                           | Issued By: C. Lin                                    |
| Approval & Date<br>Dept. Hd. <i>V. Beaucaire 1/17/97</i> | Approval & Date<br>Dir M&QS <i>P. H. [unclear] 1/22/97</i> | Approval & Date<br>Other Party <i>C. Lin 1/17/97</i> |





# Acme Steel Company

## Work Instruction

STATUS: Revised

**DEPARTMENT:** Petrographic Laboratory  
**TITLE:** Determining the Fluidity of a Coal (Coal Storage/Coal Preparation)  
**EFFECTIVE:** June 10, 1996

Number: PLWI:003

Pages: 1

**Purpose**

To provide a relative measure of the plastic behavior of a coal or coal blend when heated under prescribed conditions.

**Scope**

Applies to all incoming coals, new coals and coal blends.

| Step | Description of Work Activity   | Record of Activity                       |
|------|--|--|
| 1    | Much of this work instruction is based on ASTM D 2639.   |  |
| 2    | Air-dry a representative sample of the coal.   | Label tray                               |
| 3    | Crush the sample using the grinder until it passes a No. 40 screen.  |  |
| 4    | Weigh $5.0 \pm 0.2$ grams of the prepared coal sample.   |  |
| 5    | Mount the stirring rod in the Plastometer crucible, charge the coal sample into the crucible and pack it according to the manufacturer's instructions.   |  |
| 6    | Run the Plastometer following the manufacturer's instructions.   |  |
| 7    | The fluidity of coals or blends is measured in dial divisions per minute (ddpm). Each rotation of the stirring rod equals 100 dial divisions. Temperature and ddpm are printed by the Plastometer every minute.  |  |
| 8    | The Plastometer indicates the key fluidity parameters--the temperature of initial softening (first temperature greater than 1 ddpm), the temperature of maximum fluidity, and the final hardening temperature (the last temperature at which the fluidity is still greater than 1 ddpm). | Plastometer printout mounted in log book |

**Measures**

No. 40 screen size, weight in grams, temperature in °C, fluidity in ddpm.

**Records**

Log book.

| Rev# | Rev Date | Reason for Document Change              |
|------|----------|---|
| 001  | 10/21/96 | Name keyed title to process flow chart. |

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| Revision #: 001  | Revision Date: 10/21/96                                | Author: C. Lin & V. Beaucaire                        |
| Last Review Date: 10/21/96                               | Date of Next Review:<br>07/01/97                       | Issued By: C. Lin                                    |
| Approval & Date<br>Dept. Hd. <i>V. Beaucaire 1/17/97</i> | Approval & Date<br>Dir M&QS <i>P. H. Green 1/21/97</i> | Approval & Date<br>Other Party <i>C. Lin 1/17/97</i> |



# Acme Steel Company

## Work Instruction

STATUS: Revised

DEPARTMENT : Petrographic Laboratory  
 TITLE: Oxidation Test for Coal (Coal Preparation)  
 EFFECTIVE: June 14, 1996

Number: PLWI:004

Pages: 1

**Purpose** To outline the procedure used to check the degree of coal oxidation.

**Scope** Applies to all coals.

| Step | Description of Work Activity   | Record of Activity |
|------|--|--------------------|
| 1    | Obtain a polished pellet of the coal to be tested.   |                    |
| 2    | Prepare a fresh 1 N KOH solution saturated with safranin-O.  |                    |
| 3    | Dip the polished coal pellet into the KOH solution for 15 minutes at room temperature.   |                    |
| 4    | Rinse the pellet with deionized water, dry it with a tissue and apply two drops of immersion oil to the dipped end of the pellet.  |                    |
| 5    | Place the pellet on the stage of the petrographic microscope and bring it into focus.  |                    |
| 6    | Estimate the degree of oxidation by observing both the color of the stain and the amount of the stain. A light green color indicates slight oxidation, dark green indicates moderate oxidation and yellow or brown indicates severe oxidation. Determine the relative amount of oxidized coal in the sample using a point count. | Log book           |
| 7    | When desired, forward a sample of the coal to a commercial laboratory to quantify the oxidation by the alkali extraction method (ASTM D 5263).   | Log book           |

**Measures** Color distinction, point count.

**Records** Log book.

| Rev# | Rev Date | Reason for Document Change              |
|------|----------|---|
| 001  | 10/21/96 | Name keyed title to process flow chart. |

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|---|--|--|
| Revision #: 001   | Revision Date: 10/21/96                                  | Author: C. Lin & V. Beaucaire                        |
| Last Review Date:<br>10/21/96                             | Date of Next Review:<br>07/01/97                         | Issued By: C. Lin                                    |
| Approval & Date<br>Dept. Hd. <i>V. Beaucaire</i> 11/17/97 | Approval & Date<br>Dir M&QS <i>P. H. Stevens</i> 1/22/97 | Approval & Date<br>Other Party <i>C. Lin</i> 1/17/97 |





## Work Instruction

# Acme Steel Company

STATUS: *Revised*

DEPARTMENT : **Petrographic Laboratory**  
 TITLE: **Determining the Mean Maximum Reflectance of Coal (Manual Method) (Coal Preparation)**  
 EFFECTIVE: **June 14, 1996**

Number: **PLWI:005**  
 Pages: **2**

**Purpose**

To define how to measure the mean maximum reflectance of the vitrinite portion of coal using the manual petrographic microscope.

**Scope**

Applies to all coals.

| Step | Description of Work Activity   | Record of Activity |
|------|--|--------------------|
| 1    | Much of this work instruction is based on ASTM D 2798.   |                    |
| 2    | Obtain a polished pellet of the coal to be tested.   |                    |
| 3    | Turn on the photometer and the light source and allow the microscope to warm up for at least 1/2 hour.   |                    |
| 4    | Apply two drops of immersion oil onto the glass standard. Use No. 4 glass for high-and mid-vol coals and No. 6 glass for low-vol coals.  |                    |
| 5    | Place the standard on the microscope stage and bring it into focus. Calibrate the microscope by rotating the stage 360 degrees and adjusting the percentage of reflected light to match the standard value for the glass used. | Log book           |
| 6    | Apply two drops of immersion oil onto the polished surface of the pellet and focus the pellet under the microscope.  |                    |
| 7    | Rotate the stage 360 degrees and record the maximum reflected light value indicated on the instrument's display.   | Log book           |
| 8    | Using a systematic method of transversing the surface of the pellet, obtain a total of 50 maximum reflectance values, checking the instrument's calibration after every 15-20 measurements.                                    | Log book           |
| 9    | Determine the mean (average) of these 50 readings.   | Log book           |



**Measures**      Percentage of reflected light.

**Records**              Log book.

| Rev# | Rev Date | Reason for Document Change              |
|------|----------|---|
| 001  | 10/21/96 | Name keyed title to process flow chart. |

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|--|--|---|
| Revision #: 001  | Revision Date: 10/21/96                                | Author: C. Lin & V. Beaucaire                       |
| Last Review Date: 10/21/96                               | Date of Next Review:<br>07/01/97                       | Issued By: C. Lin                                   |
| Approval & Date<br>Dept. Hd. <i>V. Beaucaire 1/17/97</i> | Approval & Date<br>Dir M&QS <i>D. H. Jones 1/22/97</i> | Approval & Date<br>Other Party <i>C. Lin 1/7/97</i> |



# Acme Steel Company

## Work Instruction

STATUS: Revised

DEPARTMENT : **Petrographic Laboratory**  
 TITLE: Estimating the Mean Maximum Reflectance of Coal (Automated Method) (Coal Preparation)  
 EFFECTIVE: **June 20, 1996**

Number: **PLWI:006**  
 Pages: **2**

**Purpose** To define how to estimate the mean maximum reflectance (MMR) of the vitrinite portion of coal using the automated petrographic microscope.

**Scope** Applies to all coals.

| Step | Description of Work Activity  | Record of Activity                    |
|------|---|---------------------------------------|
| 1    | Obtain a polished pellet of the coal to be tested.  |                                       |
| 2    | Turn on the photometer and the light source and allow the microscope to warm up for at least 1/2 hour.  |                                       |
| 3    | Apply two drops of immersion oil onto the glass standard. Use No. 4 glass for high-and mid-vol coals and No. 6 glass for low-vol coals.   |                                       |
| 4    | Place the standard on the microscope stage and bring it into focus. Calibrate the microscope according to the manufacturer's coal quality program.  | Log book                              |
| 5    | Apply two drops of immersion oil onto the polished surface of the pellet and allow the pellet to stand for 20 minutes before focusing it under the microscope.  |                                       |
| 6    | Use the manufacturer's coal quality program to measure the mean maximum reflectance of the coal. The microscope obtains 25,000 readings in 5 minutes, selects roughly 11,000 useful readings and composes a histogram which shows the range of reflectance measurements and the frequencies of measurement. | Computer printout mounted in log book |
| 7    | Visually estimate the center of gravity (CG) of the histogram and record this as the CG MMR value.  | Log book                              |
| 8    | For high-vol coals, multiply the CG MMR by 1.04. For mid-and low-vol coals, multiply the CG MMR by 1.02. Report these corrected MMR values.   | Log book                              |
| 9    | Check the instrument's calibration every 40-45 minutes and at the end of the analysis period.   | Log book                              |





**Measures** Light reflectance in percent, time in minutes

**Records** Computer printout, log book.

| Rev# | Rev Date | Reason for Document Change                               |
|------|----------|--|
| 001  | 10/21/96 | Name keyed title to process flow chart.<br>Added step 9. |

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|--|--|--|
| Revision #: 001  | Revision Date: 10/21/96                                | Author: C. Lin & V. Beaucaire                        |
| Last Review Date: 10/21/96                               | Date of Next Review: 07/01/97                          | Issued By: C. Lin                                    |
| Approval & Date<br>Dept. Hd. <i>V. Beaucaire 1/17/97</i> | Approval & Date<br>Dir M&QS <i>P. H. Jones 1/22/97</i> | Approval & Date<br>Other Party <i>C. Lin 1/17/97</i> |



# Acme Steel Company

## Work Instruction

STATUS: : Revised

DEPARTMENT: Petrographic Laboratory  
 TITLE: Coke Reactivity Index And Coke Strength After Reaction (Ship to Furnace/Stock)  
 EFFECTIVE: June 25, 1996

Number: PLWI:007

Pages: 2

### Purpose

This work instruction defines the measurements of coke reactivity index (CRI), which indicates the rate at which coke carbon is consumed by reaction with carbon dioxide, and coke strength after reaction (CSR), which indicates coke strength at high temperature.

### Scope

Applies to all coke.

| Step | Description of Work Activity   | Record of Activity |
|------|--|--------------------|
| 1    | Much of this work instruction is based on ASTM D 5341.   |                    |
| 2    | Obtain a representative sample of the coke weighing approximately 15 lb. A 5-gallon container is suitable.   | Label container    |
| 3    | Select representative pieces and break off approximately 250 grams of plus 3/4 inch, minus 7/8 inch coke for testing.  |                    |
| 4    | Dry the sample at 150°C for 2 hours.   |                    |
| 5    | Weigh 200 ± 2 grams of the dry coke for testing to the nearest 0.1 gram.   |                    |
| 6    | Preheat the furnace to a temperature that will allow the coke sample inside the reactor, when loaded into the furnace, to reach 1100°C ± 5°C in 30 minutes.  |                    |
| 7    | Purge the reactor with N <sub>2</sub> gas for 5 minutes at 5 liters/min. and lower the reactor into the furnace.   |                    |
| 8    | Heat the sample to 1100°C under a N <sub>2</sub> flow of 5 liters/min. Total heatup time is 40 minutes.  |                    |
| 9    | Turn off the N <sub>2</sub> gas and turn on CO <sub>2</sub> gas, setting the flow rate at 5.00 ± 0.05 liters/min. Expose the coke sample to reaction with the CO <sub>2</sub> gas for exactly 120 minutes while maintaining a temperature of 1100°C ± 5°C.   |                    |
| 10   | After 120 minutes, turn off the CO <sub>2</sub> gas and switch to N <sub>2</sub> gas at a flow rate of 5 liters/min. to purge the reactor of CO <sub>2</sub> . Subsequently remove the reactor vessel from the furnace and allow the sample to cool to 100°C while the N <sub>2</sub> gas continues to flow. |                    |



|    |  |          |
|----|--|----------|
| 11 | After cooling, remove the coke from the reactor and weigh it to the nearest 0.1 gram. The percentage of weight loss is the coke reactivity index (CRI).  | Log book |
| 12 | Transfer the reacted coke into the l-type coke tumbler and tumble 600 rotations at 20 ± 1 rpm.   |          |
| 13 | Screen the coke using a 3/8" screen. Weigh the plus 3/8" coke to the nearest 0.1 gram. Based on the total weight of coke in the tumbler, the percentage of plus 3/8" coke is the coke strength after reaction (CSR). | Log book |

**Measures**                      Weight in grams, screen size in inches, temperature in °C, flow rate in liters/min., time in minutes.

**Records**                      Log book.

| Rev # | Rev Date | Reason for Document Change  |
|-------|----------|---|
| 001   | 10/21/96 | Revise steps 2 and 10.<br>Name keyed title to process flow chart. |

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|--|--|--|
| Revision #: 001  | Revision Date: 10/21/96                              | Author: C. Lin & V. Beaucaire                        |
| Last Review Date: 10/21/96                               | Date of Next Review: 07/01/97                        | Issued By: C. Lin                                    |
| Approval & Date<br>Dept. Hd. <i>V. Beaucaire 1/17/97</i> | Approval & Date<br>Dir M&QS <i>P. H. Lee 1/22/97</i> | Approval & Date<br>Other Party <i>C. Lin 1/17/97</i> |