



Designation: D 5114 – 90 (Reapproved 1998)

## Standard Test Method for Laboratory Froth Flotation of Coal in a Mechanical Cell<sup>1</sup>

This standard is issued under the fixed designation D 5114; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### INTRODUCTION

Froth flotation of coal, the separation of ash-bearing minerals from combustibles via differences in surface chemistry, has been steadily increasing in use as a means to treat 600- $\mu\text{m}$  (No. 30 U.S.A. Standard Sieve Series) or finer coal. The process is one in which many variables need to be monitored and regulated. Because of this complexity, rigorous laboratory testing is difficult to standardize.

This test method outlines the types of equipment and procedures to apply on a laboratory scale to isolate key process variables and minimize the variations associated with the design and execution of a froth flotation test. The objective of the test method is to develop a means by which repeatable grade/recovery results are ascertained from froth flotation testing of coal without imposing unnecessary limitations on the applicability of the test results in coal preparation practice.

It is recognized that sample preparation, particularly comminution, has a significant impact on froth flotation response. This test method does not attempt to define sample preparation and size reduction practices as part of a froth flotation testing program.

This test method also does not completely cover specific procedures for the investigation of flotation kinetics. Such a test is specialized and is highly dependent upon the end use of the data.

### 1. Scope

1.1 This test method covers a laboratory procedure for conducting a single froth flotation test on fine coal (that is, nominal top size of 600  $\mu\text{m}$  (No. 30 U.S.A. Standard Sieve Series) or finer) using a defined set of starting point conditions for the operating variables.

1.2 This test method does not completely cover specific procedures for the investigation of flotation kinetics. Such a test is specialized and highly dependent upon the objective of the data.

1.3 Since optimum conditions for flotation are usually not found at the specified starting points, suggestions for development of grade/recovery curves are given in Appendix X1. Such a procedure is very case-specific and involves running a series of flotation tests in which some of the operating variables are changed in order to optimize conditions for either yield or grade.

1.4 Laboratory flotation results need not be representative of the flotation response of coal in full-scale situations, but a consistent baseline can be established against which full-scale performance can be compared.

1.5 The values stated in SI units are to be regarded as standard. The values in parentheses are provided for informa-

tion only. The values stated in each system may not be exact equivalents; therefore, each system must be used independently of the other, without combining values in any way.

1.6 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.7 Material Safety Data Sheets (MSDS) for reagents used are to be obtained from suppliers who are to be consulted before work with any chemicals used in this test method.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 121 Terminology of Coal and Coke<sup>2</sup>

D 2013 Method of Preparing Coal Samples for Analysis<sup>2</sup>

D 2015 Test Method for Gross Calorific Value of Coal and Coke by the Adiabatic Bomb Calorimeter<sup>2</sup>

D 2234 Practice for Collection of a Gross Sample of Coal<sup>2</sup>

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke<sup>2</sup>

D 3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal<sup>2</sup>

D 3177 Test Methods for Total Sulfur in the Analysis Sample of Coal and Coke<sup>2</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.07 on Physical Characteristics of Coal.

Current edition approved Aug. 31, 1990. Published December 1990.

<sup>2</sup> Annual Book of ASTM Standards, Vol 05.05.

D 4239 Test Methods for Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Methods<sup>2</sup>

D 4749 Test Method for Performing Sieve Analysis of Coal and Designating Coal Size<sup>2</sup>

### 3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, see Terminology D 121.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *collector*—a reagent used in froth flotation to promote contact and adhesion between particles and air bubbles.

3.2.2 *combustibles*—the value obtained by subtracting the dry weight (in percent) of the ash (as determined in Test Method D 3174) from 100 % representing the original weight of the analyzed sample.

3.2.3 *concentrate*—the froth product recovered in coal froth flotation.

3.2.4 *conditioning agents*—all chemicals that enhance the performance of the collectors or frothers. Conditioning agents change the characteristics of the surface of the minerals or the environment. There are many subgroups according to function: activators, depressants, emulsifiers, dispersants, flocculants, chelating reagents, froth depressants, pH modifiers, and so forth.

3.2.5 *flotation cell*—the vessel or compartment in which the flotation test is performed.

3.2.6 *froth*—a collection of bubbles and particles on the surface of a pulp in a froth flotation cell.

3.2.7 *froth flotation*—a process for cleaning fine coal in which hydrophobic particles, generally coal, attach to air bubbles in a water medium and rise to the surface to form a froth. The hydrophilic particles, generally the ash-forming matter, remain in the water phase.

3.2.8 *frother*—a reagent used in froth flotation to control the size and stability of the air bubbles, principally by reducing the surface tension of water.

3.2.9 *grade/recovery*—the relationship between quality and quantity of the clean coal product. The quality can be defined in terms of ash, sulfur, or Btu content. The quantity can be designated as yield or heating value recovery (Btu or combustibles).

3.2.10 *mechanical cell*—a type of flotation cell that employs mechanical agitation of a pulp by means of an immersed impeller (rotor) and stator stirring mechanism. Aeration to the cell can be from an external pressurized air source or self-induced air.

3.2.11 *natural pH*—the measured pH of the pulp prior to the addition of collector, frother, or any conditioning agents.

3.2.12 *pulp*—a fluid mixture of solids and water, also known as slurry.

3.2.13 *recovery*—the percent of the valuable component (that is, Btu or combustible) from the feed that reports to the froth concentrate product.

3.2.14 *solids concentration*—the ratio, expressed as a percent, of the weight (mass) of solids to the sum of the weight of solids plus water.

3.2.15 *tailings*—the underflow product from coal froth flotation.

3.2.16 *yield*—the weight percent of the feed that reports to the concentrate.

### 4. Significance and Use

4.1 This test method uses specific starting point conditions for the froth flotation response to accomplish the following:

4.1.1 Assess responses of one or more coals or blends of coal, and

4.1.2 Evaluate and determine froth flotation circuit performance.

### 5. Apparatus

5.1 *Laboratory Flotation Machine*, with a minimum volume of 2 L and a maximum volume of 6 L. Fig. 1 schematically depicts a batch mechanical flotation cell<sup>3</sup> which can be used in conjunction with this test method. The major criterion is that the unit must be able to provide for constant mechanical removal of froth from the cell. In addition, the laboratory unit must have some means of automatic liquid level control.

5.1.1 An example of a mechanical paddle laboratory froth flotation apparatus is shown in Fig. 1. The froth paddles are rotated at approximately 30 r/min, thus avoiding variation caused by manual removal of froth. The froth paddle shall not rotate below the pulp surface and not more than 6 mm (¼ in.) above the pulp level. The distance between the overflow lip and the edge of the froth paddle shall be at least 3 mm (⅛ in.) but not more than 6 mm (¼ in.).

5.1.2 The pulp in the cell is maintained at a constant level by a small tank with an overflow at precisely the desired level to be maintained in the flotation cell.

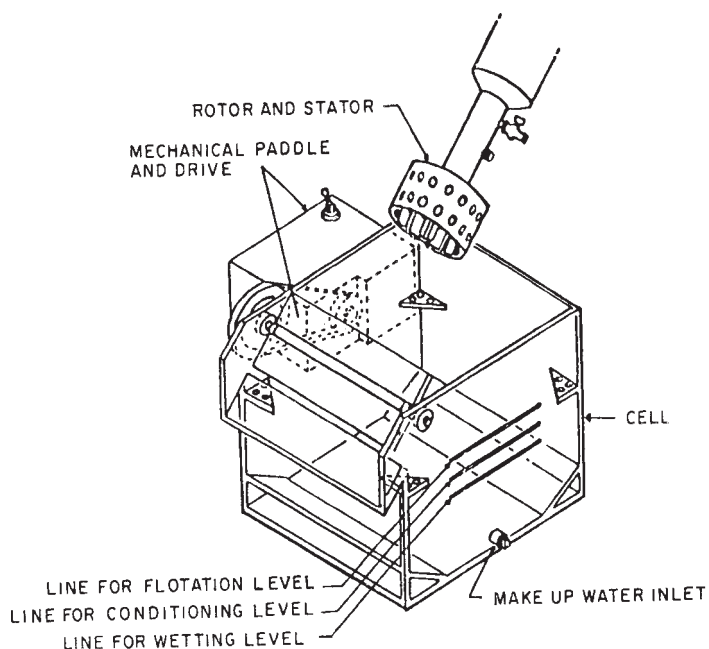


FIG. 1 5.5-L Mechanical Paddle Laboratory Froth Flotation Cell

<sup>3</sup> A suitable cell, available from WEMCO, 1796 Tribute Rd., Sacramento, CA 95815, or equivalent can be used.

NOTE 1—Another suitable slurry level control system consists of a resistance type level probe, a resistance sensor relay, a solenoid valve, and associated connecting wires.<sup>4</sup> The level probe is mounted inside the cell and is connected to the resistance relay which operates the solenoid valve. When the slurry level drops below the tip of the probe, the relay energizes the solenoid valve. Then, makeup water flows into the cell. When the level rises up to the probe, the solenoid valve is de-energized, which stops the makeup water flow.

5.2 *pH Meter*, sensitive to 0.1 units.

5.3 *Timing Device* that displays cumulative minutes and seconds.

5.4 *Air Flow Meter*.

5.5 *Microsyringes or Pipets*.

5.6 *Balances*, with a readability of at least 0.5 % of the total weight.

5.7 *Vacuum or Pressure Filter*, or a filter funnel for gravity filtration.

5.8 *Drying Oven* with forced air, capable of maintaining a maximum temperature of 40°C (104°F) and meeting the requirements of Method D 2013.

5.9 *Rinse Bottle*.

## 6. Sample Preparation

6.1 The sample history, moisture content, alteration of the inherent moisture, or alteration of the surface properties have considerable effect on the flotation characteristics of the coal. It is important that all samples used in flotation testing are stored and handled so as to minimize alteration of the surface properties. The origin and history of the sample should be recorded. It is imperative that all samples be prepared in a similar manner. Since the generation of grade/recovery curves will involve several individual tests, sample subdivision and preparation must be carefully performed to ensure that each subsample is representative of the original whole sample.

## 7. Flotation Conditions

7.1 The conditions under which a test program is conducted will be systematically varied to generate grade/recovery curves (Appendix X1). Table 1 outlines recommended starting point conditions for a single laboratory-scale test. These conditions are for laboratory testing parameters and are not designed to simulate in-plant operating conditions that can be highly variable, such as water temperature and chemistry.

7.2 *Slurry Temperature*—The operating temperature shall be  $22 \pm 5^\circ\text{C}$  ( $72 \pm 9^\circ\text{F}$ ).

7.3 *Water*—Plant, tap, or distilled water may be used, whichever is consistent with the object of the test. The source of water must be recorded.

7.4 *Solids Content*—The solids content corresponds with that of the industrial preparation plant slurry, if the object of the test is to simulate plant conditions. Otherwise, an 8 % solids concentration shall be used.

7.5 *Pulp Level*—Maintain between 12.7 and 15.9 mm (0.50 and 0.62 in.) below the lip of the cell as measured with the air on and stirrer operating.

7.6 *Wetting of Coal*—Before the addition of reagents and subsequent flotation, it is important to ensure that the proper air bubble attachment can take place at the coal-water interface. Wetting is accomplished in the cell by running the impeller at the r/min specified for the flotation step with the air off. Perform this step for 5 to 10 min before reagent addition. If the sample is in slurry form this wetting step is not necessary.

7.7 *Reagent Addition*—Collector, frother, conditioning agent, or any combination thereof shall be governed by the requirements of the test. Add reagents to the coal slurry and condition to ensure proper distribution of reagents. Conduct the conditioning step at the same impeller speed as the flotation step with the air flow off.

7.7.1 Add the reagents using either a calibrated microsyringe or a pipet.

7.8 *Air Flow*—Rate shall be measured and recorded.

7.9 *Impeller Speed*—The starting speed shall be 1200 r/min.

NOTE 2— Impeller speed is an important variable and should be investigated during optimization, depending on the object of the test.

**TABLE 1 Starting Point Conditions for Laboratory Froth Flotation of Coal**

NOTE 1— Additional time can be required for a slowly responsive coal; record any extra time.

Solids concentration	8 % solids
Total volume	2 to 6 L
Wetting time	5 min
pH	natural
Impeller speed	1200 r/min
Reagent additions and conditioning times:	
1. Add collector	
2. Condition for 90 s	
3. Add frother	
4. Condition for 30 s	
Air flow rate	3 L/min per litre of pulp
Skimmer rotation	30 r/min
Collection increments	15, 30, 60, 90, 120, 240 (cumulative time in seconds)

## 8. Procedure

8.1 Calculate the total mass of coal required for the number of flotation tests based on the measured cell volume and the test solids content.

8.2 Divide the total mass into representative portions by riffing, in accordance with Method D 2013. A few small increments, totalling no more than 15 % of the total mass, may be either taken from the subsample or added to the subsample in order to obtain the exact weight.

8.3 Determine the particle size distribution of one of the portions from 8.2 in accordance with Test Method D 4749.

8.4 Rinse the cell thoroughly with water. Add from one half to two thirds of the total required water to the cell. Confirm that the air is turned off. Turn the impeller on and adjust to the desired speed. Transfer a sample into the cell. Be careful to remove all of the coal from the sides of the transfer container. Continue this wetting step for approximately 5 min. Add most of the additional water but reserve a sufficient quantity for rinsing (see 8.8).

8.5 Determine the pH and temperature of the slurry with the air turned off.

<sup>4</sup> A suitable slurry level control system, available from C&R Technology, Inc., P.O. Box 114, Fall Branch, TN 37656, or equivalent can be used.

8.6 Start the timing device. Add the collector to the slurry and condition for 90 s. After this first conditioning step, use a small quantity of rinse water to wash down any coal that is clinging to the sides of the cell.

8.7 Again start the timer. Add the frother to the slurry and condition for 30 s.

8.8 After this second conditioning step, wash down any coal that is clinging to the sides of the cell. At this time, the pulp level shall be the operating level specified in 7.5.

8.9 Confirm that the water valve is open to the constant level control system.

8.10 Turn on the froth paddles and start the air flow.

8.11 Start the froth collection timer when the air is turned on.

8.12 Collect the froth in a series of pans. Continue collecting the incremental froth produced for each of the predetermined time periods or until the froth is no longer coal laden (lack of black color to the froth), recording the time,  $T_f$ , at which this occurs (see Table 2).

8.13 Continually rinse the froth clinging to the sides of the flotation cell into the pulp.

**TABLE 2 Report**

Test No.: _____						Sample identity: _____				
	Process Variables:					Setting/Dosage:				
Frother						_____				
Collector						_____				
Conditioning reagent						_____				
Pulp solids concentration						_____				
Pulp pH						_____				
Slurry temperature						_____				
Air flow rate						_____				
Weight of charge						_____				
Impeller r/min						_____				
Water type						_____				
Froth Collection Fraction <sup>AB</sup>							Tailing			
Cumulative Time										
	15 s <i>n</i> = 1	30 s <i>n</i> = 2	60 s <i>n</i> = 3	90 s <i>n</i> = 4	120 s <i>n</i> = 5	240 s <i>n</i> = 6				
Dry weight of solids							$w_t$			
Weight recovery	$w_c(n)$	_____	_____	_____	_____	_____				
Ash	$Y(n)$	_____	_____	_____	_____	_____	$Y_t$			
Sulfur	$A_c(n)$	_____	_____	_____	_____	_____	$A_t$			
Btu	$S_c(n)$	_____	_____	_____	_____	_____	$S_t$			
Btu recovery	$B_c(n)$	_____	_____	_____	_____	_____	$B_t$			
Ash reduction	$R(n)$	_____	_____	_____	_____	_____	$R_t$			
Sulfur reduction	$Z(n)$	_____	_____	_____	_____	_____	$Z_t$			
Last coal laden froth time, $T_f$	$V(n)$	_____	_____	_____	_____	_____	$V_t$			
							Feed Head Sample		Recombined Head Sample	
Weight, gram							_____		_____	
Ash, dry weight percent							_____		_____	
Sulfur, dry weight percent							_____		_____	
Btu, dry Btu/lb							_____		_____	

A

Where:

*f* = feed,

*n* = nth timed froth increment sampled, and

*t* = tailing.

$$W_t = W_c(1) + W_c(2) + \dots + W_c(n) + W_t$$

$$A_t = Y(1) * A_c(1) + Y(2) * A_c(2) + \dots + Y_c(n) * A_c(n) + Y_t * A_t$$

$$S_t = Y(1) * S_c(1) + Y(2) * S_c(2) + \dots + Y_c(n) * S_c(n) + Y_t * S_t$$

$$B_t = Y(1) * B_c(1) + Y(2) * B_c(2) + \dots + Y_c(n) * B_c(n) + Y_t * B_t$$

<sup>B</sup> The mass of the reconstituted feed,  $W_f$ , is the sum of the masses of the concentrated samples,  $W_c(n)$ , and the tailing,  $W_t$ . If  $W_f$  differs greater than weight 3 % from the mass of the initial feed, then the data should be questioned.

8.14 At the end of the flotation period, close the valves to the constant level control tank and air supply. Rinse all material adhering to the sides of the cell and stand pipe into the cell. Wash all material remaining on the cell lip and scraper paddles into the concentrate.

8.15 Separately dewater (usually by filtration), air dry, and weigh each concentrate and tailing. Refer to Method D 2013. Determine the residual moisture, ash content, and any other parameters required for each sample.

## 9. Calculation

9.1 Calculate all parameters on a dry basis.

9.2 Calculate yield,  $Y$ , in weight percent as follows:

$$Y = \frac{100 \times W_c}{W_c + W_t}$$

where:

$W_c$  = weight of froth concentrate and

$W_t$  = weight of tailing.

9.3 Calculate the percent recovery,  $A$ , of any analytical parameter using the following formula, which uses the feed value reconstituted from the froth concentrate and tailing.

$$A = \frac{Y \times P_c}{P_f}$$

where:

$P_c$  = is one of the following:

$A_c$  = percent ash in the froth concentrate fraction,

$S_c$  = percent sulfur in the froth concentrate fraction,

$B_c$  = Btu/lb in the froth concentrate fraction, and

$C_c$  = percent combustible in the froth concentrate fraction, and

$P_f$  = is one of the following reconstituted feed parameters calculated from the froth concentrate fractions and the tailing (see Table 2):

$A_f$  = percent ash in the feed,

$S_f$  = percent sulfur in the feed,

$B_f$  = Btu/lb in the feed, and

$C_f$  = percent combustible in the feed.

9.4 Calculate the percent impurity reduction,  $A$ , for any analytical parameter as follows:

$$A = \frac{P_f - P_c}{P_f}$$

9.5 Calculate the weight percent of parameter removal,  $A$ , for any analytical parameter as follows:

$$A = \frac{(100 - Y) \times (P_t)}{P_f}$$

where:

$Y$  and  $P_f$  are as defined above,

$P_t$  is one of the following:

$A_t$  = percent ash in the tailing fraction,

$S_t$  = percent sulfur in the tailing fraction,

$B_t$  = Btu/lb in the tailing fraction, and

$C_t$  = percent combustible in the tailing fraction.

9.6 Calculate the efficiency index,  $E$ , as follows:

$$E = \frac{Y \times A_t}{A_c}$$

## 10. Report

10.1 A test report shall be issued containing the following information:

10.1.1 Sample identity and history,

10.1.2 Feed size distribution,

10.1.3 Reagent concentration of frother, collector, and conditioning agent,

10.1.4 Wetting time,

10.1.5 Conditioning times,

10.1.6 Solids concentration,

10.1.7 Pulp pH,

10.1.8 Slurry temperature,

10.1.9 Air flow rate,

10.1.10 Weight of charge,

10.1.11 Impeller r/min,

10.1.12 Weight of concentrate fractions and tailings,

10.1.13 Collection time period(s), and

10.1.14 Source of water.

10.2 Report ash, sulfur, Btu/lb, or combustible recovery and yield data on the form shown in Table 2.

## 11. Precision and Bias

11.1 *Precision*—The precision at the starting point conditions is being investigated by a task group. Other operating conditions are too numerous to establish precision statements at this time.

11.2 *Bias*—Pending an evaluation of this test procedure, the absence of a reference material precludes a bias statement.

## 12. Keywords

12.1 collector; flotation; flotation cell; froth flotation; frother; froth paddles; mechanical cell; pulp level; slurry level; starting point conditions

APPENDIX

(Nonmandatory Information)

**X1. OPTIMIZATION CONSIDERATIONS**

X1.1 The procedure outlined in this Appendix provides a means of evaluating the flotation characteristics of a coal through the manipulation of process variables to achieve a grade/recovery relationship indicative of the separation that may be expected from the froth flotation process for a given coal at a given particle size distribution.

X1.2 Laboratory froth flotation testing need not be representative of flotation response of a particular coal in a full-scale situation. However, the grade/recovery curves generated from laboratory procedures can be used to provide information regarding how a coal reacts to changes in various operating parameters. The flotation of coal involves a complex interaction of several factors, including:

- X1.2.1 Flotation conditions,
- X1.2.2 Surface characteristics, and
- X1.2.3 Particle size distribution.

X1.3 All of these factors can be varied to affect the flotation characteristics of a given coal. Among the flotation conditions most often varied to understand and control flotation are the following:

- X1.3.1 Conditioning time,
- X1.3.2 Flotation time,
- X1.3.3 Reagent type,
- X1.3.4 Reagent dosages,
- X1.3.5 Air flow rates,
- X1.3.6 pH of pulp, and
- X1.3.7 Solids loading.

X1.4 Individual flotation tests are required for reviews to identify the effect of varying each of these parameters in an effort to determine the best flotation conditions for a particular coal. This appendix contains procedures by which the conditions can be systematically altered and subsequent flotation results presented for data evaluation.

X1.5 Among the most common flotation tests performed in the laboratory are those in which flotation reagents are altered incrementally to evaluate the ideal dosages to be run in a preparation plant. To produce data for evaluation in a grade/recovery curve, a test plan can be formulated to conduct several individual flotation tests on equivalent splits of a coal sample. Each, of these flotation tests would be run using this test method but with reagent dosages for each test corresponding to the test plan.

X1.5.1 *Example*—It can be desirable to determine the effect of the amount of fuel oil used to condition the surface of the coal on the ash reduction and thermal recovery of the coal. To accomplish this, a test plan might be formulated in which a number of individual flotation tests would be run. For instance, fuel oil dosages from 0.25 to 2.5 g/kg (0.5 to 5.0 lb/ton) varied in 0.25-g/kg (0.5-lb/ton) increments can be used. Each flotation test would be run according to this flotation standard and the results would be presented in the standard format shown in Table 2. Flotation results generated by varying one or more parameters may be graphically depicted as presented in Fig. X1.1 and Fig. X1.2.<sup>5</sup> Fig. X1.1 shows where optimum pine oil addition occurs in the flotation of Mary Lee Seam coal.

X1.5.2 Such data is not definitive with respect to actual plant operation. However, it does provide information about the ease and efficiency of processing any particular coal sample. Evaluation of grade/recovery curves will assist in the determination of the course for further test work. It should not be viewed in and of itself as an indication of an intrinsic floatability of a coal, but as a reflection of the response of the coal to the specific levels of all process variables as presented by this test procedure.

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<sup>5</sup> See Chernosky, F. J., "Evaluation of Coal Flotation Frothers on a Yield-Selectivity Cost Basis," *Transactions of AIME*, Vol 226, March 1963, pp. 24–25.

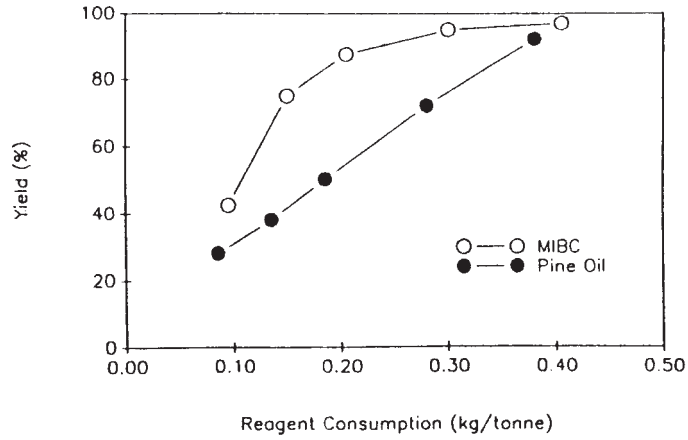


FIG. X1.1 Example of Consumption Curve for Mary Lee Coal

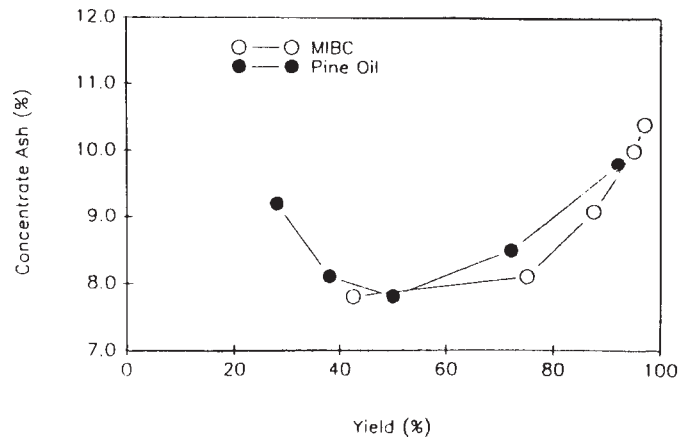



FIG. X1.2 Example of Grade Versus Yield Curves for Mary Lee Coal

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