



Designation: D 5142 – 90 (Reapproved 1998)

## Standard Test Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures<sup>1</sup>

This standard is issued under the fixed designation D 5142; 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.

### 1. Scope

1.1 These instrumental test methods cover the determination of moisture, volatile matter, and ash, and the calculation of fixed carbon in the analysis of coal and coke samples prepared in accordance with Method D 2013 and Practice D 346. Results obtained through the use of the instrumental tests have been shown to differ from those obtained with Test Methods D 3173, D 3174, and D 3175 on some coals and cokes. Where a relative bias between the instrumental methods and Test Methods D 3173, D 3174, and D 3175 for proximate analysis of coal and coke are shown to exist, the instrumental results shall be corrected or the instrument calibrated using samples of known proximate analysis. Test Methods D 3173, D 3174, and D 3175 shall be considered the referee test methods. The instrumental test methods are not applicable to thermogravimetric analyzers using microgram size samples.

1.2 The moisture value determined is used for calculating other analytical results to a dry basis. When used in conjunction with the air dry loss moisture, as determined in accordance with Method D 2013, Test Method D 3302, or Practice D 346, the calculated total moisture can be used to convert dry basis analytical results to the as-received basis.

1.3 The result of the ash determinations can be applied in the ultimate analysis, Practice D 3176.

1.4 *This standard does not purport to address all of 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.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 121 Terminology of Coal and Coke<sup>2</sup>
- D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis<sup>2</sup>
- D 1757 Test Method for Sulfate Sulfur in Ash from Coal and Coke<sup>2</sup>
- D 2013 Method of Preparing Coal Samples for Analysis<sup>2</sup>

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D-5 on Coal and Coke and are the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

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

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 3175 Test Method for Volatile Matter in the Analysis Sample of Coal and Coke<sup>2</sup>

D 3176 Practice for Ultimate Analysis of Coal and Coke<sup>2</sup>

D 3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases<sup>2</sup>

D 3302 Test Method for Total Moisture in Coal<sup>2</sup>

### 3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology D 121.

### 4. Summary of Test Methods

4.1 Moisture is determined by establishing the loss in mass of the analysis specimen of coal or coke when heated under rigidly controlled conditions of temperature, time, atmosphere, specimen mass, and equipment specifications.

4.2 Volatile matter is determined by measuring the loss in mass of the moist or dried analysis specimen of coal or coke when heated under rigidly controlled conditions. If appropriate, the measured mass loss establishes the volatile matter after correcting for the moisture content.

4.3 Ash is determined by measuring the mass of the residue remaining after burning the coal or coke specimen under rigidly controlled conditions of specimen mass, temperature, time, atmosphere, and equipment specifications.

4.4 In these test methods, moisture, volatile matter, and ash may be determined sequentially in a single instrumental procedure. With some systems, the procedure consists of simply placing a specimen in the microprocessor-controlled instrument and initiating the automatic process of analyzing the specimen.

4.5 In other systems, the analytical process is manually controlled, although the instruments may have microprocessors to assist in controlling conditions of the instrument and for calculations. The actual analytical process can vary from instrument to instrument, but the overall process shall provide for the determination of moisture, volatile matter, and ash as specified in 4.1, 4.2, and 4.3.

4.6 Different procedures are described to permit conformity



with differences in instrument design and for differences in sample behavior.

4.7 Where a relative bias between these test methods and other ASTM test methods for the proximate analysis of coal and coke has been shown to exist (12.2.1), the instrument used in these test methods must be calibrated using specimens of known composition covering the range of parameters being determined.

## 5. Significance and Use

5.1 Moisture, as determined by the instrumental test method, is used for calculating other analytical results to a moisture-free basis using procedures in Practice D 3180. Moisture percent determined by this test method may be used in conjunction with the air-dry moisture loss determined in Method D 2013 and Test Method D 3302 to determine total moisture in coal. Total moisture is used for calculating other analytical results to an *as-received* basis using Practice D 3180.

5.2 Ash yield, as determined by this test method, is the residue remaining after burning the coal and coke samples.

5.3 Volatile matter yield, when determined as herein described, may be used to (1) establish the rank of coals, (2) indicate coke yield on carbonization, (3) provide the basis for purchasing and selling, or (4) establish burning characteristics.

5.4 Fixed carbon is a calculated value. It is the difference between 100 and the sum of the percent moisture, ash, and volatile matter. All percents shall be on the moisture reference base.

5.5 Moisture, ash, volatile matter, and fixed carbon percents constitute the proximate analysis of coal and coke.

NOTE 1—The ash obtained differs in composition and amount from the mineral constituents present in the original coal. Incineration causes an expulsion of all water, the loss of carbon dioxide from carbonates, the conversion of iron pyrite into iron oxides and sulfur oxides, and other chemical reactions. Ash yield, as determined by this test method, can differ from the amount of ash produced in furnace operations or other combustion systems because incineration conditions influence the chemistry and amount of ash.

## 6. Apparatus

6.1 *Furnace or Oven*—The primary apparatus shall consist of a furnace or oven with a cavity large enough to accept crucibles containing the specimens. The furnace or oven shall be constructed so the cavity is surrounded by a suitable refractory and insulated so as to develop a uniform temperature in all parts of the cavity but with a minimum free space. The furnace shall be capable of being heated rapidly at a rate of 50°C/min from ambient to 950°C.

6.1.1 The temperatures within the cavity shall be monitored and maintained at values that are specific to each of the determinations. Temperatures, as measured in crucibles located throughout the cavity shall be maintained within the following limits:

Determination of	Temperature, °C	
	min	max
Moisture	104	110
Ash	700	750
Volatile matter	930	970

6.1.2 Provisions shall be made to introduce a sweep gas or

a reactant gas, and to remove products of drying, devolatilization, or combustion. Gases and acceptable flow rates for the different determinations are as follows:

Determination of	Gas	Flow Rate, Furnace Volume per Minute
Moisture	nitrogen or air	2–4
Ash	oxygen	0.4–0.8 <sup>A</sup>
	air	2–4
Volatile matter	nitrogen	2–4

<sup>A</sup>This oxygen flow is chemically equivalent to 2 to 4 volume changes of air per minute.

6.2 *Crucibles*, with covers, of a convenient form that allows the moisture and volatile matter, or moisture and ash, or all three determinations to be made on the same specimen, shall be used. The crucibles may be made of fused silica with self-sealing covers that have ground glass seals or of porcelain with self-sealing covers. The covers shall allow the rapid escape of gases during the determination of volatile matter. All crucibles used shall have the dimensions specified by the instrument manufacturer.

6.3 *Balance*, sensitive to 0.1 mg. The balance may be an internal balance and an integral part of the instrument used for determining the moisture, volatile matter, and ash, or it may be a separate piece of apparatus that may not be interfaced with the instrument.

6.4 The instrumental system in its entirety shall be operated in strict accordance with the manufacturer's operating instructions.

6.5 *Venting Equipment*—Combustion and volatile gases evolved during the test procedures shall be vented from the laboratory and suitable venting equipment shall be installed in the vicinity of the apparatus.

## 7. Analysis Sample

7.1 Obtain the sample for coal in accordance with Method D 2013 and the sample for coke in accordance with Practice D 346. Obtain the analysis sample by pulverizing the material to a fineness to pass the 250-µm (No. 60) sieve.

## 8. Reagents and Materials

8.1 *Dry Gas*—Nitrogen (>99.5 % purity) or air, dried to a moisture content of 1.9 mg/L or less (dew point –10°C or less) to be used to purge the drying oven. Any desiccant or drying method capable of achieving this degree of dryness is suitable. (See Reagent section of Test Method D 3173.)

## 9. Procedure

9.1 Some instruments allow for the determination of moisture, volatile matter, and ash in sequence using the same specimen. Alternatively, some instruments, or components of the instruments, may be used to carry out separate determinations of moisture, volatile matter, or ash using separate specimens of coal or coke.

9.2 Heat new crucibles and covers (see 9.2.1) for use in these test methods under the conditions of the test and cool before use. For each test, weigh a prepared crucible (Note 2). Add approximately 1 g of coal or coke to the crucible and then weigh the crucible and specimen. Make all weighings to the nearest 0.1 mg. Transfer the specimen from the sample bottle to the crucible quickly to minimize the exposure of the