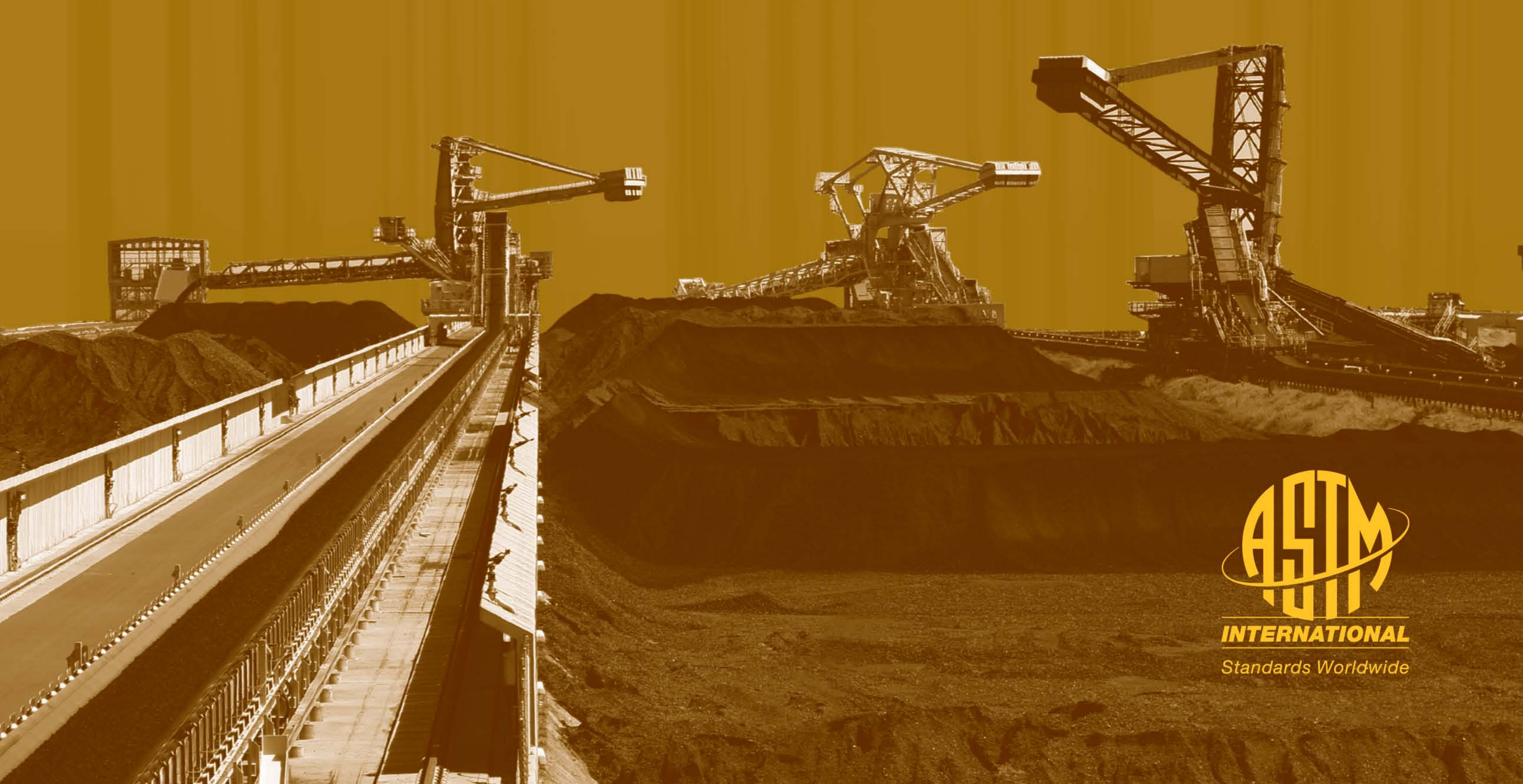
Guide to ASTM Test Methods for the Analysis of Coal and Coke

R.A. Kishore Nadkarni



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Guide to ASTM Test Methods for the Analysis of Coal and Coke

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HOW TO USE THIS MANUAL

Table I—lists the ASTM test methods alphanumerically by ASTM designation. If you know the ASTM designation, this is the easiest way to find what you need.

Table of Contents—lists the ASTM test methods by their principle subject matter. In many cases, the citation may be replicated in this table because of multiple key words involved. If you do not know the ASTM test method designation, then look in this table to find the test method by subject matter.

FOREWORD

This publication, *Guide to ASTM Test Methods for the Analysis of Coal and Coke*, was sponsored by ASTM Committee D05 on Coal and Coke, and edited by R. A. Kishore Nadkarni, Millennium Analytics, Inc., East Brunswick, NJ. This publication is Manual 61 of ASTM's manual series.

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INTRODUCTION

Coal and coke are probably the earliest fuels used by mankind even before the dawn of civilization. They truly represent the "fossil" fuels. In spite of their antiquity, their use continues to grow throughout the world to provide heat and electricity to an energy hungry world. For example, in the United States, considered as the "Saudi Arabia" of coal, although the number of operating mines decreased dramatically from over 11,000 in the 1920s to under 1500 today, the coal production continues to rise thanks to the efficient and effective technological means used in the coal mining industry.

In an excellent article published in 2004 at the time of the centennial celebration of the establishment of ASTM's Committee D05 on Coal and Coke, Janke, Luppens, and Graham provide a comprehensive review of the origin, development, and current status of both the coal industry and Committee D05 (1). It is illuminating to cite some information excerpted from this article.

It is estimated that the United States possesses about 25 percent of the world's known accessible and minable coal reserves. Production through later decades of the 20th century on into the new millennium rose to nearly 1.1 billion tons a year. Coal is a critical component for manufacturing steel and cement. Coal is used to generate over 50 percent of all electricity produced in the United States. In 1917, 33 million tons of coal was burned annually in the U.S.; today that figure is over 830 million tons.

Over the last century, ASTM Committee D05 on Coal and Coke has led the efforts to standardize the methods for characterization of coal and coke properties. These efforts span a worldwide effort with ties to many other national consensus standard writing organizations, including the International Organization for Standardization (ISO) Technical Committee 27 on Solid Fuels. In virtually all instances, the ASTM D05 standards are considered as the final arbitrator for the quality of a coal or a coke related product. Many other national and international standards are based on original ASTM D05 standards.

There are about 55 analytical test method standards under D05 Committee's jurisdiction. These standards involve a variety of physical, chemical, and spectroscopic analytical techniques to qualitatively and quantitatively identify over 40 chemical and physical properties of coal, coke, and their products and by-products. These test methods are annually published in the *Annual Book of ASTM Standards* (2). Additional methods continue to be developed in many energy company laboratories and elsewhere either to improve on the existing methods or to enable determination of other properties.

With the certainty that coal resources will continue to be a dominant contributor to the energy industry in the United States and elsewhere in the world, ASTM Committee D05 continues to provide standards that ensure significant technological, economic, productivity, and environmental benefits from effective utilization of coal resources.

It is certainly not the intention of this author to replace the *Annual Book of ASTM Standards* with the current book, but rather to develop a complementary manual for the user of ASTM coal and coke test methods. This manual makes available in one handy volume the essential elements of all analytical tests used to characterize coal, coke and their products. It is critical for testing laboratory personnel to be fully familiar with all the details of the tests they are performing. But it is also important for non-laboratory personnel to understand at least the significance, advantages and limitations of particular tests used to characterize coal and coke quality. Buyers and suppliers of coal and coke need to agree on the appropriate product quality specifications, and this can be done only with a thorough understanding of the limitations of these tests. Product specifications that are not based on realistic testing capabilities can lead to quality complaints and dissatisfaction on the part of both buyers and sellers. As such, we expect that this book will prove useful not only to laboratory personnel, but also to the product specification writers, process engineers, researchers, and marketing staff in understanding the importance and limitations of these tests, so that sound judgements can be made regarding the quality and performance of a company's products.

All technical information in this manual is based on the year 2007 edition of the *Annual Book of ASTM Standards* (2). We plan to update the manual at some frequency depending on how many new or significantly revised standards are issued by the Committee D05. If you notice any errors or omissions, please let us know. We will correct them in future editions.

References

(1) L.C.G Janke, J.A. Luppens, and R. D. Graham—*ASTM Standardization News*, ASTM International, West Conshohocken, PA, July 2004, pp. 22–27.

(2) Annual Book of ASTM Standards, Vol. 05.06, ASTM International, West Conshohocken, PA, 2006.

(3) Committee on Standards, "Precision and Bias," *ASTM Standardization News*, ASTM International, West Conshohocken, PA, January 1985, pp. 45.

EXPLANATION OF TERMS

Accuracy—The accuracy of a test is a measure of how close the test result will be to the true value of the property being measured. As such the accuracy can be expressed as the bias between the test result and the true value. However, the absolute accuracy can be established if the true value is known.

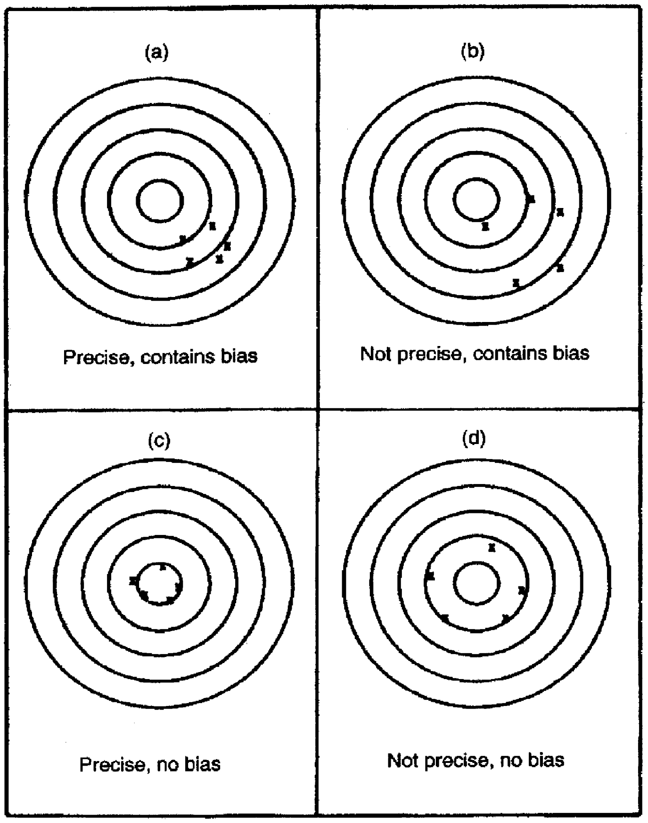
ASTM International—West Conshohocken, PA.

ISO—International Organization for Standardization (Switzerland).

NIST—National Institute of Standards and Technology, Gaithersburg, MD.

Precision—The precision of a test method is defined in terms of the variability between test results obtained on the same material, using a specific test method. The precision of a test is usually unrelated to its accuracy. The results may be precise, but not necessarily accurate. Figures 1 to 4 which are found in reference 3 depict in a bull's eye analogy the relationship between precision and accuracy. The ideal condition would be obtaining the one with most precise and most accurate results. Precision can be expressed as repeatability and reproducibility.

Repeatability Limits—ASTM Standard Terminology D121 defines repeatability limit as the value below which the absolute difference between two results from separate and consecutive test determinations, carried out on the same sample in the same laboratory by the same operator using the same apparatus on the samples taken at random from a single quantity of homogenous material, may be expected to occur with a probability of approximately 95 %. The repeatability limit is 2.8 times the repeatability standard deviation. The multiplier is independent of the size of the interlaboratory study. The approximation to 0.95 is reasonably good (say, 0.90 to 0.98) when many laboratories (30 or more) are involved, but it is likely to be poor when fewer than eight laboratories are studied.



Reproducibility Limits—ASTM Standard Terminology D121 defines reproducibility limit as the value below which the absolute difference between two test results obtained under reproducibility conditions between different laboratories may be expected to occur with a probability of approximately 0.95 (95 %). The reproducibility limit is 2.8 times the reproducibility standard deviation. The multiplier is independent of the size of the interlaboratory study (i.e., of the number of laboratories participating). The approximation to 0.95 is reasonably good (say 0.90 to 0.98) when many laboratories (30 or more) are involved but is likely to be poor when fewer than 8 laboratories are studied.

The reproducibility limit is generally (but not always) higher than the repeatability limit by a factor of 2 to 4.

The repeatability and reproducibility values, have very important implications in the market place. As the demand for unambiguous product specifications, and for the control of product consistency grows, it is meaningless to establish product specifications that are more restrictive than the reproducibility/repeatability values of the specified test methods.

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D4326	Major and Minor Elements in Coal and Coke Ash by X-Ray Fluorescence	23
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D4749	Sieve Analysis of Coal	31

ARSENIC AND SELENIUM IN COAL BY HYDRIDE GENERATION/ATOMIC ABSORPTION METHOD

D4606

EXPLANATION

The presence of arsenic and selenium in coal can be of concern when coal is combusted. When coal samples are prepared for analysis in accordance with this test method, the arsenic and selenium are both quantitatively retained and representative of their total amount in coal.

TEST METHOD SUMMARY

Arsenic and selenium are determined by mixing a weighed coal sample with Eschka mixture and igniting at 750°C. The mixture is dissolved in hydrochloric acid and the gaseous hydride of each element is generated from the appropriate oxidation state, and determined by atomic absorption spectrometry.

TEST METHOD PRECISION AND BIAS

Element	Concentration Range, ppm	Repeatability Limit, ppm	Reproducibility Limit, ppm
Arsenic	0.7 – 12.2	0.3 + 0.07 X	0.6 + 0.1 X
Selenium	0.6 – 5.0	0.31 + 0.2 X	0.1 + 0.3 X

Where X is the average of two results.

Based on the analysis of a NIST SRM 1632a coal there is no bias between the results obtained by this method and values certified by NIST for SRM 1632a.

ASH IN THE ANALYSIS SAMPLE OF COAL AND COKE FROM COAL

D3174

EXPLANATION

Ash, as determined by this test method, is the residue remaining after burning the coal and coke under specified conditions. The ash obtained by this test method differs in composition from the inorganic constituents present in the original coal. Incineration causes an expulsion of all water, the loss of carbon dioxide from carbonates, the conversion of iron pyrites into ferric oxide, and other chemical reactions. Ash, as determined by this test method, may differ in amount from ash produced in furnace operations and other firing systems because incineration conditions influence the chemistry and amount of the ash.

The results obtained by this test method can be applied as the ash in the proximate analysis standard Practice D3172, and in ultimate analysis standard Practice D3176. The constituents in the ash can be determined by Test Methods D2795 (withdrawn), D3682, D4326 and D6349.

TEST METHOD SUMMARY

Ash is determined by weighing the residue remaining after burning the coal or coke sample under rigidly controlled conditions of sample weight, temperature, time, atmosphere, and equipment specifications.

TEST METHOD PRECISION AND BIAS

Range	Repeatability, %	Reproducibility, %
Coal: 2.689 to 17.86 % (250 μm topsize)	0.22	0.32
Bituminous: 5.0 to 15.0 % (2.36 mm topsize)	0.30	0.49
Subbituminous -Lignite: 4.5 to 30.0 % (2.36 mm topsize)	0.33	0.47

Since this is an empirical test method, the degree of absolute bias cannot be determined.

GROSS CALORIFIC VALUE OF COAL AND COKE D5865

EXPLANATION

The gross calorific value can be used to compute the total calorific content of the quantity of coal or coke represented by the sample for payment purposes for computing the calorific value versus the sulfur content to determine whether the coal meets regulatory requirements for industrial fuels and for evaluating the effectiveness of beneficiation processes. The gross calorific value can be required to classify coals according to standard Classification D388.

TEST METHOD SUMMARY

This test method determines the gross calorific value of coal or coke by either isoperibol or adiabatic bomb calorimetry. The heat capacity of the calorimeter is determined by burning a specified mass of benzoic acid in oxygen. A comparable mass of the analysis sample is burned under the same conditions in the calorimeter such that the mass burned produces approximately the same temperature rise as during calibration. The calorific value of the analysis sample is computed by multiplying the corrected temperature rise, by the heat capacity adjusted for extraneous heat effects and dividing by the mass of the sample.

Unnecessary exposure of the prepared samples to air from the time of sampling or unnecessary delay in analysis should be avoided since oxidation of coals after sampling can result in reduced calorific value. This effect is more pronounced for lignites and sub-bituminous rank coals than for samples of higher rank coals.

TEST METHOD PRECISION AND BIAS

Instrument	Coal Type	Calorific Value, J/g	Repeatability, J/g	Reproducibility, J/g
Manual-Adiabatic Calorimeter	Bituminous	29535–33721	160	249
Manual-Adiabatic Calorimeter	Subbituminous and Lignites	20442–29651	140	326
Microprocessor controlled Auto	Bituminous	26279–34186	149	256
Microprocessor controlled Auto	Subbituminous and Lignites	21860–30000	193	381
MicroProcessor controlled Auto	Coke	30028	479	928

Calorific values have not been determined for anthracite coals or for No. 8 sieve size coal. Bias in the determination of the gross calorific value is eliminated provided the samples are treated identically to the benzoic acid used in the determination of the calorimeter heat capacity.

TOTAL, COMBUSTIBLE, AND CARBONATE CARBON IN SOLID RESIDUES FROM COAL AND COKE
D6316

EXPLANATION

The combustible carbon content of solid residues is used to calculate the efficiency of fuel combustion in boiler furnaces and similar combustors. Combustible carbon values are also used to determine the residual fuel value of incompletely combusted coal and coke in fluidized bed furnaces and other reactors which consume carbonaceous fuels. The combustible carbon content of flyash is an important parameter in the use of flyash as a cement additive. This test method covers the determination of total, combustible, and carbonate carbon remaining in the solid byproducts of combustion from boiler furnaces and similar reactors, including ash, flyash, char, slag, and similar materials. This test method is intended for the use in industry to determine the performance of boiler furnaces and similar combustion reactors and aid in determining the quality of the solid residue from the combustion.

TEST METHOD SUMMARY

This test method comprises the use of any of several methods to determine total carbon content combined with any of several methods to determine carbonate carbon, and the calculation, by difference, of the combustible carbon remaining in a sample.

Alternatively, this test method applies to the determination of total carbon remaining in a material after acidification with strong acid to evolve carbon dioxide from carbonate carbon. In this case, the combustible carbon is the total carbon measured in the sample after acidification.

Total Carbon is determined by oxidative thermal decomposition of a weighed quantity of sample in a closed system, and after complete oxidation and purification of the resulting gaseous products, measurement of the carbon dioxide produced by one of several methods. In D3178, the evolved carbon dioxide is fixed on an absorption train and is measured quantitatively by weighing the absorbent. In D5373, the carbon dioxide is measured quantitatively by an electronic detection system calibrated against an appropriate reference standard.

Carbonate Carbon is determined by decomposing a weighed quantity of the sample with a dilute mineral acid, and after the purification of the evolved gases, the evolved carbon dioxide is measured quantitatively. In Test Method D1756, the evolved gases are purified and carbon dioxide is fixed as sodium carbonate on a pre-weighed absorption unit and is gravimetrically measured. In Test Method D513 part B, the evolved gas is swept through a scrubber and into an absorption cell where it is coulometrically titrated. In another method an instrumental technique similar to one used for total carbon is used, with the provision for sample combustion replaced by a provision for acidification. In practice, this test method uses an adaptation to instruments designed to measure total carbon content.

Combustible Carbon is determined directly by first acidifying and heating to dryness a weighed sample. All carbonate carbon is evolved as carbon dioxide. The dried residue is then analyzed for total carbon content as above.

TEST METHOD PRECISION AND BIAS

Parameter	Range, %	Repeatability Limit, %	Reproducibility Limit, %
Total Carbon	0.34–15.12	0.14 + 0.04 X	0.07 + 0.12 X
Combustible Carbon	0.14–14.00	0.25 + 0.06 X	0.82 + 0.14 X
Carbonate Carbon	0.01–2.75	0.01 + 0.08 X	0.04 + 0.13 X

Where X is the average of two single test results.

Bias could not be determined since certified standard reference materials are not available.

CARBON AND HYDROGEN IN COAL AND COKE
D3178

EXPLANATION

Carbon and hydrogen values are used to calculate the amount of oxygen (air) required in combustion processes and in the calculation of efficiency of combustion processes. Carbon and hydrogen determinations are used in material balances in coal conversion processes; also, one or the other is frequently used in correlations of chemical and physical properties, such as yields of products in liquefaction, reactivity in gasification, and the density and porosity of coal.

TEST METHOD SUMMARY

These methods are used to determine carbon and hydrogen in samples of coal or coke in one operation. The results indicate total carbon and hydrogen, and include not only the carbon and hydrogen in the organic matter, but also the carbon present as mineral carbonates, and the hydrogen present in the free moisture present in the sample and as water of hydration of silicates.

When the data are converted and reported on a "dry" basis, the hydrogen value is corrected for the hydrogen present in the free moisture accompanying the sample. A weighed quantity of the sample is burned in a closed system and combustion products are fixed in an absorption train after complete oxidation and purification from interfering substances.

Interferences—Formation of nitrogen oxides during the combustion procedure may lead to slightly higher results for carbon. However, this effect has been found to be insignificant in commercial applications.

TEST METHOD PRECISION AND BIAS

	Repeatability Limit, %	Reproducibility Limit, %
Carbon	0.3	—
Hydrogen	0.07	—

The bias of this test method has not been determined at present.

Note—The Test method D3178 was withdrawn in June 2007 because of the lack of a reproducibility limit statement.

INSTRUMENTAL DETERMINATION OF CARBON, HYDROGEN, AND NITROGEN IN LABORATORY SAMPLES OF COAL AND COKE

D5373

EXPLANATION

Carbon and hydrogen values are used to determine the amount of oxygen (air) required in combustion processes and for the calculation of efficiency of combustion processes. Carbon and hydrogen determinations are used in material balance calculations on coal conversion processes; also, one or the other is frequently used in correlations of chemical and physical properties, such as yield of products in liquefaction, reactivity in gasification, and the density and porosity of coal.

Nitrogen data are used for comparing coals and in research. Nitrogen data is required as part of the ultimate analysis, particularly if oxygen is estimated by difference. Also, the data can be used to evaluate the potential formation of nitrogen oxides as a source of atmospheric pollution.

TEST METHOD SUMMARY

These test methods cover the instrumental determination of carbon, hydrogen, and nitrogen in coal and coke samples. The test methods are applicable to either the air-dry or moisture-free laboratory samples, or both. These test methods can be used to provide for the requirements specified in standard Practice D3176 for the ultimate analysis.

Because of a variety of instruments used in these analyses, the procedures can vary substantially from each other. In all cases, the sample material is introduced in an oxygen stream to convert the constituents to carbon dioxide, water vapor, nitrogen oxides, sulfur oxides and ash. Subsequently, the gases are quantitatively determined in an appropriate reference gas stream. In some configurations a series of traps selectively absorb and detect individual gas components. Ultimately, the components may be determined using thermal conductivity or infra-red detection. A modified gas chromatographic system has also been used utilizing thermal conductivity detection.

TEST METHOD PRECISION AND BIAS

Element	Concentration range, %	Repeatability Limit, %	Reproducibility Limit, %
Carbon	48.6 to 90.6	0.64	2.51
Hydrogen	0.14 to 5.16	0.16	0.30
Nitrogen	0.69 to 1.57	0.11	0.17

Bias is eliminated when the instrument is properly calibrated against certified reference standards.

COKE REACTIVITY INDEX (CRI) AND COKE STRENGTH AFTER REACTION (CSR)
D5341

EXPLANATION

When coke lumps descend into the blast furnace, they are subjected to reaction with countercurrent carbon dioxide and to abrasion as they rub together and against the walls of the furnace. These concurrent processes physically weaken and chemically react with the coke lumps, producing an excess of fines that can decrease burden permeability and result in increased coke rates and lost hot metal production. This test method is designed to measure indirectly this behavior of coke in a blast furnace. The method, patterned after the Nippon Steel test procedure, describes the equipment and techniques used for determining CRI and CSR by tumbling in a cylindrical chamber referred to as an I-tester.

TEST METHOD SUMMARY

A sample of dried coke of designated origin and size is reacted with carbon dioxide gas in a retort at a specified elevated temperature for a specified length of time. Two indices, coke reactivity index (CRI) and coke strength after reaction (CSR), are determined using the reacted coke residue. The weight loss after the reaction determines the CRI. The weight retained after sieving the tumbled reacted coke in a designated number of revolutions over a designated turning rate determines the CSR.

TEST METHOD PRECISION AND BIAS

Parameter	Repeatability Limit	Reproducibility Limit
CRI (% db)	2.8	3.21 + 0.12 X
CSR (% db)	5.4	14.4 − 0.13 X

Where X is the average of two single between-laboratory results.

Certified reference materials for this test are not available, hence bias cannot be determined.

CARBONATE CARBON IN COAL
D1756

EXPLANATION

Carbonate minerals occur in many coals ranging from small to large amounts. The determination of these carbonates is the purpose of this test method. The value found for carbon dioxide is used to estimate the mineral content, particularly calcium carbonate and magnesium carbonate of high-carbonate coals.

TEST METHOD SUMMARY

The determination of carbon dioxide is made by decomposing with a mineral acid a weighed quantity of the sample in a closed system and absorbing the carbon dioxide in an absorbent. The increase in weight of the absorbent is a measure of the carbon dioxide present in the sample used.

TEST METHOD PRECISION AND BIAS

Range, %	Repeatability Limit, %	Reproducibility Limit, %
<1	0.05	0.10
>1	0.10	0.20

The bias of this test method has not been determined.

CHLORINE IN COAL

EXPLANATION

The chlorine content of coal may be useful in the evaluation of slagging problems, corrosion in engineering processes, and in the total analysis of coal and coke.

There are three methods available for the determination of total chlorine in coal.

D2361

TEST METHOD SUMMARY

The coal specimen is oxidized by combustion and the reacted chlorine is absorbed in alkaline reagents using one of two procedures. In one a sample is burned in a combustion bomb containing oxygen under pressure and a small amount of ammonium carbonate solution. In a second procedure, a weighed sample is mixed with Eschka mixture and heated at a specified temperature in an oxidizing atmosphere. Finally, the chloride contained in the ammonium carbonate solution in the first procedure or extracted from the incinerated Eschka mixture in the second procedure is determined by potentiometric titration with standard silver nitrate solution.

D4208

TEST METHOD SUMMARY

Total chlorine is determined by combustion of a weighed sample in an oxygen bomb with dilute base adsorbing the chlorine vapors. The bomb is rinsed into a beaker with water and after adding an ionic strength adjuster, the chloride is determined using an ion-selective electrode.

D6721

TEST METHOD SUMMARY

A sample of coal is combusted with tungsten accelerator in a humidified oxygen gas flow at 900°C. Halogens are oxidized and converted to hydrogenated halides, which are flushed into a titration cell where they accumulate. Chlorine is converted to hydrochloric acid. Once the chloride is captured in the electrolyte of the titration cell, it can be quantitatively determined by microcoulometry, where chloride ions react with silver ions present in the electrolyte. The silver ion thus consumed is coulometrically replaced and the total electrical work needed to replace it is proportional to the chloride in the test sample.

Bromide and iodide if present, are calculated as chloride. However, fluorides are not detected by this test method.

TEST METHODS PRECISION AND BIAS

Test Method	Chlorine Concentration, ppm	Repeatability Limit, ppm	Reproducibility Limit, ppm	Bias
D2361	—	300	600	Not Known
D4208	220–2100	48.4 + 0.13 X	200 + 0.23 X	Not Determined
D6721	22–1136	1.92 + 0.06 X	6.13 + 0.07 X	No Bias

Where X is the average of two single test results.

CONTOURS OR CROSS SECTIONS METHOD FOR DETERMINATION OF VOLUME OF BULK MATERIALS

D6172

EXPLANATION

This test method is used to audit the volume of material in a stockpile and is used with a density value to calculate a tonnage value used to compare the book value to the physical inventory results.

TEST METHOD SUMMARY

This test method covers procedures concerning site preparation, technical procedures, quality control, and equipment to direct the efforts for determining volumes of bulk material. These procedures include practical and accepted methods of volumetric determination.

This test method allows for only two volume computation methods: contour test method and cross-section test method. The method requires direct operator compilation for both contours and cross-section development. The use of Digital Terrain Model software and procedures to create contours or cross sections for volume calculation is not encompassed in this test method.

In the contour test method, a horizontal slice is taken to determine volume. After creating a new contour map of the pile, the cubic volume is computed by averaging the areas of adjacent contours and multiplying by the vertical distance between them.

In the cross-section test method, a vertical slice is taken for determining the volume. Using elevations obtained in parallel lines across the surface and base of the pile, the cubic volume is computed by averaging the areas of adjacent cross sections and multiplying by the horizontal distance between them.

TEST METHOD PRECISION AND BIAS

Confidence interval @ 2 SD = 2SE where SD is the standard deviation and SE is standard error. The industry accepted error is ±2 %. Since there is no accepted reference material for this test, no bias statement can be made.

CUBIC FOOT WEIGHT OF CRUSHED BITUMINOUS COAL

D291

EXPLANATION

This test method concerns the compaction of crushed coal to determine either its compacted or uncompacted weight, for purposes such as charging of coke ovens. This test method is not applicable to the testing of powdered coal as used in boiler plants, nor to the determination of weights per cubic foot of coal in storage piles.

TEST METHOD SUMMARY

This test method covers two procedures for determining the cubic foot weight of crushed coal less than 1.5 in (37 mm) in size, such as is charged into coke ovens.

In procedure A, the cone procedure is used for determining an uncompacted weight per cubic foot (cubic meter).

In procedure B, the dropped-coal procedure is used for determining a compacted weight per cubic foot (cubic meter), comparable to actual bulk densities attained in coke ovens.

TEST METHOD PRECISION AND BIAS

Repeatability Limit	1.0 lb/cubic foot	1.6 kg/cubic meter
Reproducibility Limit	Not Known	Not Known

This test method is empirical, and no statement of bias is possible.

APPARENT AND TRUE SPECIFIC GRAVITY AND POROSITY OF LUMP COKE

D167

EXPLANATION

Apparent and true specific gravity, as determined by this test method, are influenced by the types of coals carbonized and the operating and preparation conditions of that carbonization, (i.e., charge bulk density, heating rate, and pulverization level). In turn, these properties directly influence the performance in processes using coke.

TEST METHOD SUMMARY

This test method describes the determination of apparent specific gravity and true specific gravity of lump coke larger than 25-mm (1-in.) size and calculating porosity from the specific gravity data.

TEST METHOD PRECISION AND BIAS

For both specific gravity methods, repeatability and reproducibility are 0.01 each. Since these test methods are empirical standards, the degrees of their absolute bias cannot be determined.

BULK DENSITY OF COAL

D6347/D6347M

EXPLANATION

The measured mean bulk density is used with a measurement of the stockpile volume per Test Method D6172 to measure the quantity of stockpiled coal. This measure of quantity is often used as a reference value for adjusting inventory records. This test method covers procedures using instrumentation that measures the relative backscatter of nuclear gamma radiation throughout the depth of the stockpile under test. This procedure is applicable to all ranks of coal. Proper density determination involves an understanding of the physical characteristics of the coal types and the stockpiling facilities.

TEST METHOD SUMMARY

The area of the stockpile accessible for stockpile penetration is first identified. An access (sampling) plan for the accessible area is then determined, and the depth of the stockpile is accessed at locations within the area according to plan. The probe is used to obtain test count readings at vertical intervals within each access hole. Test count readings of compacted coal in field calibration vessels are taken using coal from the stockpile so as to convert stockpile test count readings to bulk density using a calibration curve. The mean bulk density of the stockpile is estimated using the test count reading and calibration data.

TEST METHOD PRECISION AND BIAS

The conventional way of expressing repeatability or reproducibility is not used in this test method. A special method is described in the Annex A5 of the test method. There is no accepted reference method for determining the bulk density of coal in a stockpile, thus bias has not been determined.

DROP SHATTER TEST FOR COAL AND COKE

D440

EXPLANATION

The values determined in this test method, when evaluated in terms of pertinent experience with other coals, may be used as indications of the extent to which the coal will break in conventional transit and handling beyond the point of sampling. The test method is serviceable for ascertaining the similarity of coal in respect to its size stability and friability rather than for determining the values within narrow limits in order to emphasize their dissimilarity. This test method is considered applicable for testing a selected single size of different coals, for testing different single sizes of the same coal, and for mixed sizes of the same or different coals. The test method appears best suited for measuring the relative resistance to breakage of the larger sizes of coal when handled in thin layers such as from loader to mine car, from loading boom to railroad car, from shovel to chute, etc. While it may not be so well adapted for measuring the liability to breakage of coal when handled in mass, as in unloading open-bottom cars, emptying bins, etc., it is believed that the test method will also serve to indicate the relative size stability of composite sizes of coal where, in commercial handling, the smaller size pieces have a cushioning effect which tends to lessen the breakage of the larger pieces of coal.

TEST METHOD SUMMARY

This test method of drop shatter test covers the determination of the relative size stability and its complement, the friability, of sized coal. This test method uses the same shatter test machine as in the Test Method D3038.

TEST METHOD PRECISION AND BIAS

Data for repeatability and reproducibility is not available. This is an empirical test method and no statement of bias is possible.

D3038

TEST METHOD SUMMARY

Two procedures are described here. Procedure A is used when the coke sample contains 50 percent or less of 100 mm (4 in.) after removal of coke smaller than 50 mm (2 in.). Procedure B is used when the coke sample contains more than 50 percent 100 mm (4 in.) after removal of coke smaller then 50 mm (2 in.). These procedures use the same shatter test machine as used in the Test Method D440.

Similar to Test method D440, no data on precision or bias is available for this test method.

EQUILIBRIUM MOISTURE OF COAL AT 96-97 % RELATIVE HUMIDITY AND 30°C

D1412

EXPLANATION

This test method affords a means of estimating the bed moisture of either coal that is wet and shows visible surface moisture or coal that may have lost some moisture. It may be used for estimating the surface, or extraneous moisture of wet coal, such moisture being the difference between the total moisture as determined by the Test Method D3302 and the equilibrium moisture.

When samples are collected in conformity with standard Classification D388, the equilibrium moisture is considered to be equal to bed moisture with the exception of some low rank coals that yield equilibrium moisture values below bed moisture.

TEST METHOD SUMMARY

A sample of coal is crushed and treated with boiled distilled water for 30 minutes of shaking and 3 hours of equilibration. At the end, the mixture is filtered and a portion of wet coal is placed in a weighing bottle kept in a vacuum desiccator containing a saturated solution of potassium sulfate for maintaining a relative humidity of 96 to 97 %. The closed and evacuated desiccator is placed in a constant temperature water bath maintained at 30° ± 0.2°C for 48 h for coal of higher ranks and 72 h for lignites. After this time period, the vacuum is released and dry air is admitted in the desiccator. After weighing the sample bottle, it is placed in an oven at 105°C for 90 min. Finally, the bottle is removed from the oven, cooled to room temperature and re-weighed to determine the weight loss.

TEST METHOD PRECISION AND BIAS

Range, %	Repeatability Limit, %	Reproducibility Limit, %
Under 5	0.3	0.5
5 to 15	0.5	1.0
Over 15	1.0	1.5

Bias of this test method cannot be determined since certified standards or absolute methods are not available for this analysis.

GRINDABILITY OF COAL BY HARDGROVE MACHINE METHOD
D409

EXPLANATION

This test method gives a measure of grinding or pulverizing characteristics that can be used to evaluate the yield, and/or energy input, required in a grinding or pulverizing process. Some coals such as some high-volatile bituminous, subbituminous, and lignite coals, can undergo physical change as the natural or seam moisture is released during handling and preparation. This change is often sufficient to alter the grindability characteristics that will be reported when tested in the laboratory and could produce different indices dependent on the conditions of drying and the moisture level of the materials used in the test. Therefore, the repeatability and reproducibility cited in this test method may not apply for the above mentioned coal types.

TEST METHOD SUMMARY

A prepared and sized coal sample receives a definite amount of grinding energy in a miniature pulverizer, and the size consist of the pulverized product is determined by sieving. The resultant size consist is used to produce an index relative to the ease of grinding, and is called Hardgrove Grindability Index (HGI).

TEST METHOD PRECISION AND BIAS

Repeatability Limit	2 HGI
Reproducibility Limit	3 HGI

This is an empirical standard and therefore the degree of absolute bias cannot be determined.

SAMPLING AND FINENESS TEST OF PULVERIZED COAL
D197

EXPLANATION

This test provides a means for assisting in the evaluation of pulverizers and pulverizer systems in terms of fineness specifications. It may also be used to confirm the influence of coal fineness on combustion performance and to evaluate carbon loss. By consent among interested parties, it may be used for evaluation of coal fineness in preparation, pneumatic transfer systems, etc.

This test method covers the determination of fineness by sieve analysis of coal samples from a dry pulverizing operation. It is not applicable to products of wet milling or to fines that have clustered into an agglomerated mass.

TEST METHOD SUMMARY

The test sample is air dried in an oven at 18 to 27°F (10 to 15°C) above the room temperature until the weight loss is not more than 0.1 % per hour. The air-dried sample is divided into 50 to 100 g amounts. They are subjected to successive sieve analysis. All fines collected are combined and weighed.

TEST METHOD PRECISION AND BIAS

Repeatability Limit	2 %
Reproducibility Limit	4 %

There is no reference sample available to estimate the bias of this method.

LABORATORY FROTH FLOTATION OF COAL IN A MECHANICAL CELL

D5114

EXPLANATION

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 μm 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 uses specific starting point conditions for the froth flotation response to (a) assess response of one or more coals or blends of coal, and (b) evaluate and determine froth flotation circuit performance. This method uses fine coal of nominal top size 600 μm. This test does not completely cover specific procedures for the investigation of flotation kinetics. Such a test is specialized and highly dependent upon the objectives of the data. The 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.

TEST METHOD SUMMARY

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. Commercially available flotation systems are used in this test.

TEST METHOD PRECISION AND BIAS

The precision at the starting point conditions is being investigated. Other operating conditions are too numerous to establish precision statements. Pending an evaluation of this test procedure and the absence of a reference material precludes a bias statement.

TOTAL FLUORINE IN COAL AND COKE

EXPLANATION

The knowledge of the amount of fluorine in coal is necessary for the evaluation of potential fluorine emission from coal combustion or conversion processes. When coal samples are combusted in accordance with these test methods, the fluorine is quantitatively retained. There are two test methods for this analysis.

D3761

TEST METHOD SUMMARY

A weighed sample of coal is combusted in an oxygen bomb with a dilute base to absorb the fluorine vapors. The bomb is rinsed into a beaker with water. A citrate buffer is added and the fluoride is determined by an ion selective electrode.

This test method has been successfully used on coals containing 25 percent ash or less.

TEST METHOD PRECISION AND BIAS

The precision quoted in an earlier version of this test method quotes both repeatability and reproducibility limits as 15 ppm each. This is being re-examined in a new study since technical aspects of the method have been altered.

D5987

TEST METHOD SUMMARY

A weighed test portion of coal sample is subjected to pyrohydrolytic conditions in oxygen which separate fluorine from the coal/coke matrix. The pyrohydrolysate is then gravimetrically processed and final determinations are made by using either ion selective electrode or ion chromatographic techniques.

This test method has been successfully tested on coal containing 37 percent ash or less.

TEST METHOD PRECISION AND BIAS

The relative precision of this test method is being determined. An earlier Australian study with coals in the fluorine range from 20 to 120 ppm showed a repeatability of 10 ppm and a reproducibility of 20 ppm. The bias of this test method cannot be determined at this time.

FUSIBILITY OF COAL AND COKE ASH
D1857

EXPLANATION

The design of most coal combustion and coal conversion equipment anticipates that the ash either remains solid or assumes some degree of fluidity, depending on the particular design. Ash fusibility temperatures help predict whether the ash will perform properly in the process for which the coal was chosen.

TEST METHOD SUMMARY

This test method covers the observation of the temperatures at which triangular pyramids (cones) prepared from coal and coke ash attain and pass through certain defined stages of fusing and flow when heated at a specified rate in controlled, mildly reducing and where desired, oxidizing atmospheres.

The test method is empirical, and strict observance of the requirements and conditions is necessary to obtain reproducible temperatures and enable different laboratories to obtain concordant results.

Originally, a gas fired furnace was used for creating a reducing (hydrogen, hydrocarbons, or carbon monoxide), an oxidizing (oxygen, carbon dioxide, water vapor), or an inert (nitrogen) atmosphere. Today electric furnaces are more commonly used in which the atmospheres can be more easily controlled. Ash is prepared from coal or coke in a muffle furnace at 800 to 900°C. After homogenizing the ash, it is mixed with a binder and formed into pyramidl shaped cones by using cone molds. These cones are mounted on a refractory base and placed in a furnace heated up to 800°C for gas fired furnaces and not over 400°C for electric furnaces. A reducing or oxidizing condition is maintained throughout the test duration.

TEST METHOD PRECISION AND BIAS

Repeatability Limit		50°F (30°C)
Reproducibility Limit		
Reducing Atmosphere—	Initial deformation temperature (IT)	125°F (70°C)
	Softening temperature (ST)	100°F (55°C)
	Hemispherical temperature (HT)	100°F (55°C)
	Fluid temperature (FT)	150°F (85°C)
Oxidizing Atmosphere—	All temperatures	100°F (55°C)

LOSS ON IGNITION OF SOLID COMBUSTION RESIDUES
D7348

EXPLANATION

These test methods cover the determination of mass loss from solid combustion residues upon heating in an air or oxygen atmosphere to 950°C. The mass loss can be due to the loss of moisture, carbon, sulfur, etc. from the decomposition or combustion of the residue. LOI values from these test methods can be used by industries that utilize combustion residues in various processes and products.

In the cement industry and to combustion engineers, use of the term LOI normally refers to a mass loss in a sample heated to 950°C.

If the solid combustion residue is heated to estimate the combustible or unburned carbon in the sample, it has been shown that LOI and estimation of unburned carbon do not necessarily agree well with each other and that LOI should not be used as an estimate of unburned carbon in all combustion residues. Direct determination of unburned (combustible) carbon can be carried out by using Test Method D6316.

TEST METHOD SUMMARY

Loss on Ignition (LOI) is determined by measuring the loss in mass of the test specimen when heated under controlled conditions of temperature, time, atmosphere, specimen mass, and equipment specifications. The LOI can be determined by measuring the mass loss in a single procedure or in a two step procedure in which mass losses, equivalent to the moisture and ash values of the test specimen, are determined.

There are no known interferences for these test methods.

TEST METHOD PRECISION AND BIAS

TABLE 1 Concentration Range and Limits of Repeatability and Reproducibility for LOI (950°C) Test Methods

	Concentration Range, %	Repeatability Limit, *r*	Reproducibility Limit, *R*
Single-step Procedure			
Macro-TGA	1.1–11.7	0.21	0.28
Muffle Furnace	1.1–11.8	0.47	0.75
Two-Step Procedure (dry basis)			
Macro-TGA	1.0–11.4	0.17	0.22
Muffle Furnace	0.9–11.5	0.36	0.71

Bias—A bias statement is not available because at present a suitable certified Reference Material is not available. Based on the analysis of NIST SRM 1880a Portland Cement standard there appears to be a bias of −0.236 % in the muffle furnace two step procedure but none in the macro TGA two step procedure.

ELEMENTAL ANALYSIS OF COAL, COKE AND COMBUSTION RESIDUES BY SPECTROSCOPIC METHODS

EXPLANATION

A compositional analysis of ash from the coal is often useful in the total description of the coal. Many trace elements occur in coal, primarily as a part of the mineral matter but may also be associated with the organic matrix. Concern over release of certain trace elements into the environment as a result of coal utilization has made the determination of these elements an increasingly important aspect of coal analysis. Knowledge of ash composition is also useful in predicting the behavior of ashes and slags in combustion chambers. Utilization of ash by-products of coal combustion sometimes depends on the chemical composition of the ash. The chemical composition of the laboratory prepared coal ash may not exactly represent the composition of mineral matter in the coal or the composition of the fly ash and slag resulting from commercial scale burning of the coal.

There are several test methods available for the determination of major, minor, and trace elements in coal, coke and ashes thereof. All are based on sample decomposition followed by atomic spectroscopic determination of the metals in them. A summary of these tests is described in Table 1.

TABLE 1 Spectroscopic Methods for Elemental Analysis.

Test Method	Matrix Analyzed	Sample Preparation	Measurement Technique	Elements Determined
D3682	Coal combustion residues	Li-tetraborate fusion and acid dissolution	AAS	Al, Ca, Fe, K, Mg, Na, Si, Ti
D3683	Ash from coal or coke	Acid dissolution	AAS	Be, Cr, Cu, Mn, Ni, Pb, V, Zn
D4326	Ash from coal or coke	Formation of a glass bead by ash fusion with Li-tetraborate	XRF	Al, Ba, Ca, Fe, K, Mg, Mn, Na, P, Si, Sr, Ti
D6349	Coal, coke, and combustion residues	Ash fused and dissolved in acid; or ash digested in mixed acid	ICP-AES	Al, Ba, Ca, Fe, K, Mg, Mn, Na, P, S, Si, Sr, Ti

TABLE 1 Spectroscopic Methods for Elemental Analysis.

Test Method	Matrix Analyzed	Sample Preparation	Measurement Technique	Elements Determined
D6357	Coal, coke, and combustion residues	Ash dissolved in acid	ICP-AES, ICP-MS, or GFAAS	As, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, V, Zn

MAJOR AND MINOR ELEMENTS IN COMBUSTION RESIDUES FROM COAL UTILIZATION PROCESSES

D3682

TEST METHOD SUMMARY

Combustion residue is ignited in air at 750°C to a constant weight. The ash is fused with lithium tetraborate followed by a final dissolution of the melt in either dilute hydrochloric or nitric acid. The solution is analyzed by atomic absorption /emission spectrometry for applicable elements.

TEST METHOD PRECISION AND BIAS

TABLE 1 Concentrations Ranges and Limits for Repeatability and Reproducibility for Major and Minor Elemental Oxides in Ash from Combustion Residues

Elemental Oxide	Concentration Range, %	Repeatability Limit r	Reproducibility Limit R
SiO_2	33.14–57.73	$0.33+0.04\ \bar{x}^A$	$-1.21+0.14\ \bar{x}$
Al_2O_3	11.32–31.83	0.80	$-0.24+0.09\ \bar{x}$
Fe_2O_3	2.93–41.11	$0.01+0.06\ \bar{x}$	$0.36+0.06\ \bar{x}$
MgO	0.45–7.12	$0.04+0.03\ \bar{x}$	$0.04+0.09\ \bar{x}$
CaO	1.46–22.19	$0.21+0.04\ \bar{x}$	$0.15+0.11\ \bar{x}$
TiO_2	0.57–1.44	0.13	0.19
K_2O	0.45–2.99	$0.03+0.03\ \bar{x}$	$0.05+0.05\ \bar{x}$
Na_2O	0.14–7.15	$0.06+0.03\ \bar{x}$	$0.04+0.18\ \bar{x}$

Based on the analysis of a NIST standard coal ash SRM by this test method significant difference at 95 % confidence was found for potassium. For other applicable elements determined, there was no bias.

TRACE ELEMENTS IN COAL AND COKE ASH BY ATOMIC ABSORPTION SPECTROMETRY

D3683

TEST METHOD SUMMARY

A sample of coal or coke is ashed under controlled conditions, and the ash is dissolved in mineral acids, and the individual elements are determined using atomic absorption spectrometry.

TEST METHOD PRECISION AND BIAS

Element	Range, ppm	Repeatability Limit, ppm	Reproducibility Limit, ppm
Beryllium	0.5–5	0.2	0.5
Chromium	5–50	3	5
Copper	5–50	2	3
Manganese	<50		6

Element	Range, ppm	Repeatability Limit, ppm	Reproducibility Limit, ppm
Manganese	10–300	3	
Manganese	50–500		20
Nickel	<5	1	3
Nickel	5–30	3	
Nickel	9–30		9
Lead	10–100	2	9
Vanadium	10–50		9
Vanadium	50–100		20
Vanadium	10–100	5	
Zinc	<50	3	4
Zinc	50–100	8	17

All values are in μg/g (ppm) of air-dried coal.

Based on the analysis of NIST coal SRM 1632, no bias was found for the analysis of Be, Cr, Cu, Ni, Pb, and Zn. However, there was some bias for the determination of vanadium.

MAJOR AND MINOR ELEMENTS IN COAL AND COKE ASH BY X-RAY FLUORESCENCE
D4326

TEST METHOD SUMMARY

A sample of coal or coke is ashed under standard conditions and ignited to constant weight. Previously ashed materials are also ignited to constant weight under standard conditions prior to analysis. The ash is fused with lithium tetraborate or other suitable flux and either ground and pressed into a pellet or cast into a glass disk The pellet or disk is then analyzed by wavelength dispersive X-ray fluorescence spectrometer at selected wavelengths of elements of interest using appropriate K spectral lines. The measured intensities are compared to calibration data for the determination of elemental composition. Twelve elements are commonly measured (Si, Al, Fe, Ca, Mg, Na, K, P, Ti, Mn, Sr, and Ba), and test results are generally reported on an oxide basis.

TEST METHOD PRECISION AND BIAS

Concentration Ranges and Intervals or Limits for Repeatability and Reproducibility for Major and Minor Elemental Oxides in Coal and Coke Ash

	Percent, by Weight, of Moisture-Free Ash		
Elemental Oxide	Concentration Range	Repeatability, $I(r)$	Reproducibility, $I(R)$
SiO_2	33.2–57.5	1.26	4.39
Al_2O_3	11.6–33.1	0.83	$0.51+0.10(x)$
Fe_2O_3	3.1–41.8	$0.21+0.005(x)$	$0.99+0.035(x)$
CaO	1.5–25.2	$0.16+0.01(x)$	$0.22+0.07(x)$
MgO	0.4–4.5	$0.09+0.04(x)$	$0.14+0.09(x)$
Na_2O	0.2–7.41	0.20	0.42
K_2O	0.3–3.1	0.14	0.16
P_2O_5	0.1–3.4	$0.05+0.04(x)$	0.23

No statement on bias can be made because analysis of ash by XRF is empirical method and its accuracy is dependent upon the accuracy of the reference materials to be used for calibration and compliance to this test method.

MAJOR AND MINOR ELEMENTS IN COAL, COKE, AND SOLID RESIDUES FROM COMBUSTION OF COAL AND COKE BY ICP-AES

D6349

TEST METHOD SUMMARY

This test method covers a procedure for the analysis of the commonly determined major and minor elements in coal, coke, and solid residues from combustion of coal and coke. These residues may be laboratory ash, bottom ash, fly ash, flue gas desulfurization sludge, and other combustion process residues. The sample is ashed under standard conditions and ignited to a constant weight. The ash is fused with a fluxing agent followed by dissolution of the melt in dilute acid solution. Alternatively, the ash is digested in a mixture of hydrofluoric, nitric, and hydrochloric acids, diluted with water, and analyzed by inductively coupled plasma atomic emission spectrometry for the elements of interest.

TEST METHOD PRECISION AND BIAS

Concentration Ranges and Limits for Repeatability and Reproducibility for Major and Minor Elemental Oxides in Ash from Coal, Coke, and Solid Combustion Residues

Elemental Oxide	Concentration Range	Repeatability Limit (*r*)	Reproducibility Limit (*R*)
SiO_2	2.04–73.73 %	$-0.13+0.09\,\bar{x}$	$2.00+0.10\,\bar{x}$
Al_2O_3	1.04–29.54 %	$0.17+0.06\,\bar{x}$	$0.86+0.07\,\bar{x}$
Fe_2O_3	0.39–47.94 %	$0.13\,\bar{x}$	$0.23\,\bar{x}$
MgO	0.40–7.29 %	$0.02+0.08\,\bar{x}$	$0.11+0.11\,\bar{x}$
CaO	1.04–44.03 %	$0.11\,\bar{x}$	$0.25\,\bar{x}$
TiO_2	0.06–1.47 %	$0.02+0.07\,\bar{x}$	$0.05+0.12\,\bar{x}$

Based on the analysis of a NIST coal fly ash SRM, a very small positive bias for iron determination was observed. There was no bias found for other elements determined: Si, Al, Mg, Ca, Ti, K, P, Na, Mn, Ba, and Sr.

TRACE ELEMENTS IN COAL AND COMBUSTION RESIDUES FROM COAL UTILIZATION PROCESSES BY ICP-AES, ICP-MS, AND GF-AAS

D6357

TEST METHOD SUMMARY

These test methods pertain to the determination of the trace elements Sb, As, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, V, and Zn in coal, coke, and residues from coal combustion processes. The methods may be applicable to the determination of other trace elements.

The coal or coke sample is ashed under controlled conditions, digested in a mixture of aqua regia and hydrofluoric acid, and finally dissolved in 1 percent nitric acid. Combustion residues are digested on an as-received basis. The trace elements in this solution are determined using either inductively coupled plasma—atomic emission spectrometry or inductively coupled plasma—mass spectrometry. Selected trace elements that occur at concentrations below the detection limits of ICP-AES can be quantitatively analyzed by graphite furnace atomic absorption spectrometry.

TEST METHOD PRECISION AND BIAS

Concentration Range and Limits for Repeatability and Reproducibility for Priority Trace Elements in Coal, Coke, and Solid Combustion Residues

Priority Element	Concentration Range, $\mu g/g$	Repeatability Limit, r	Reproducibility Limit, R
Sb	0.17–5.71	$-0.06+0.29\,\bar{x}$	$0.08+0.44\,\bar{x}$
As	0.56–138.79	$0.42+0.13\,\bar{x}$	$1.73+0.23\,\bar{x}$
Be	0.42–13.11	$0.08+0.08\,\bar{x}$	$0.14+0.30\,\bar{x}$
Cd	0.02–0.84	$0.03+0.16\,\bar{x}$	$0.04+0.43\,\bar{x}$
Co	0.76–47.18	$0.28+0.11\,\bar{x}$	$1.26+0.18\,\bar{x}$
Cr	2.37–221	$1.03+0.09\,\bar{x}$	$1.50+0.18\,\bar{x}$
Cu	3.43–107.06	$0.62+0.10\,\bar{x}$	$-0.31+0.28\,\bar{x}$
Mn	11.69–419.61	$0.98+0.10\,\bar{x}$	$8.12+0.15\,\bar{x}$
Mo	0.40–20.52	$0.23+0.11\,\bar{x}$	$0.80+0.18\,\bar{x}$
Ni	2.00–113.32	$0.35+0.13\,\bar{x}$	$1.26+0.19\,\bar{x}$
Pb	1.57–66.99	$0.26+0.16\,\bar{x}$	$0.13+0.30\,\bar{x}$
Zn	3.76–202.31	$0.70+0.10\,\bar{x}$	$2.98+0.18\,\bar{x}$
V	4.50–293.17	$0.75+0.13\,\bar{x}$	$2.02+0.21\,\bar{x}$

Based on the analysis of three NIST coal and fly ash SRMs, significant bias was found at 95 % confidence level for cadmium in SRM 1632 b, lead and vanadium in SRM 1635, and chromium, copper and nickel in SRM 1633b.

TOTAL MERCURY IN COAL AND COAL COMBUSTION RESIDUES

The possible emission of mercury during coal combustion is a serious environmental concern. When test portions are burned according to these procedures, the total mercury is quantitatively retained and is representative of concentrations in whole coal.

There are three methods available for the determination of total mercury, all based on the measurement of mercury vapor. Mercury and mercury salts can be volatilized at low temperatures. Precautions against such mercury loss should be taken when using these methods. A summary of relevant points is given in the table below.

TABLE 2 Determination of Total Mercury in Coal Products.

Parameter	D3684	D6414	D6722
Matrix	Coal	Coal and coal combustion residues	Coal and coal combustion residues
Sample Preparation	Oxygen bomb combustion	Acid extraction or wet oxidation	Combustion and gold amalgamation
Measurement	Cold vapor atomic absorption spectrometry	Cold vapor atomic absorption spectrometry	Atomic absorption spectrometry
Concentration Range, ppm	0.042–0.192	0.032–0.585	0.017–0.586
Repeatability Limit, ppm	0.036	$0.012+0.11\,X$	$0.008+0.06\,X$
Reproducibilty Limit, ppm	0.054	$0.003+0.25\,X$	$0.007+0.13\,X$
Bias	Yes	Yes	No

Where X is the average of two single measurements.

Note—Bias statements in this table may be misleading, in that statistically significant bias were not seen in every case for Test Methods D6414 and D6722. Also, even when statistical significant biases may not have been of practical importance, the user would need to interpret this by reference to the bias data listed in the original test methods.

BY OXYGEN BOMB COMBUSTION AND ATOMIC ABSORPTION
D3684

TEST METHOD SUMMARY

A weighed sample of coal is combusted in an oxygen bomb containing dilute nitric acid which absorbs the mercury vapors. The bomb is rinsed into a reduction vessel with dilute nitric acid, and the mercury is measured by the flameless cold vapor atomic absorption technique.

BY ACID EXTRACTION OR WET OXIDATION AND COLD VAPOR—ATOMIC ABSORPTION SPECTROMETRY
D6414

TEST METHOD SUMMARY

In Procedure A, mercury is solubilized by heating the sample at a specified temperature in a mixture of nitric and hydrochloric acids. The acid solutions produced are transferred into a vessel in which the mercury is reduced to elemental mercury. The mercury vapor thus generated is determined by flameless cold-vapor atomic absorption spectroscopy.

In procedure B, mercury is solubilized by heating the sample in a mixture of nitric and sulfuric acids with vanadium pentoxide. The acid solutions produced are transferred into a vessel in which the mercury is reduced to elemental mercury. The mercury vapor thus generated is determined by flameless cold-vapor atomic absorption spectroscopy.

BY DIRECT COMBUSTION ANALYSIS
D6722

TEST METHOD SUMMARY

Controlled heating of the analysis sample in oxygen is used to liberate mercury. The sample is heated to dryness in the instrument and then thermally and chemically decomposed. The decomposition products are carried by flowing oxygen to the catalytic section of the furnace, where oxidation is completed, and halogens as well as nitrogen and sulfur oxides are trapped. The remaining decomposition products are carried to a gold amalgamator that selectively traps mercury. After the system is flushed with oxygen to remove any remaining decomposition products, the amalgamator is rapidly heated, releasing mercury vapor. Flowing oxygen carries the mercury vapor through absorption cells positioned in the light path of a single wavelength atomic absorption spectrophotometer. Absorbance peak height or peak area, as a function of mercury concentration is measured at 253.7 nm.

MICROSCOPICAL DETERMINATION OF TEXTURAL COMPONENTS OF METALLURGICAL COKE
D5061

EXPLANATION

The determination of the volume percent of the textural components in coke is useful to characterize the optical properties of coke as it relates to utilization. Specifically, the technique has been used as an aid in determining coal blend proportions (after correcting for coke yield), and recognition of features present in the coke that can be responsible for coke quality or production problems such as reduced coke strength or difficulty in removing coke from commercial ovens, or both. The study of coke texture is also useful in promoting a better understanding of coke reactivity, and the relationship between coal petrography and conversion of coal to coke.

This test is used in scientific and industrial research, but not for compliance or referee tests. This test method covers the equipment and procedures used for determining the types and amounts of coke carbon forms and associated recognizable coal- and process-derived textural components in metallurgical coke in terms of volume percent. This test method does not include coke structural components such as coke pores, coke wall dimensions or other structural associations.

TEST METHOD SUMMARY

The textural components of coke (coke carbon forms and associated coal- and process-related components) in a representative crushed particulate coke sample, prepared in the form of a briquetted, polished specimen as described in standard Practice D3997, are identified under a microscope according to their degree of anisotropism, carbon form domain sizes, their morphology, relative reflectance, and other optical properties. The proportions of these textural components in a sample are determined by observing a statistically adequate number of points, and summing those representative of each component. Only area proportions of components are observed on the briquette surface. However, the area and volume proportions are the same when the components are randomly distributed throughout the sample.

TEST METHOD PRECISION AND BIAS

The precision of this method is being determined. Since there is no acceptable reference material suitable for use in this test, bias cannot be determined.

MICROSCOPICAL DETERMINATION OF MACERAL COMPONENTS OF COAL

D2799

EXPLANATION

The volume percent of physical components of coal is used as an aid in coal seam correlation and in the characterization of coals for their use in carbonization, gasification, liquefaction, and combustion processes. This test is for use in scientific and industrial research, and not for compliance or referee tests.

TEST METHOD SUMMARY

This test method covers the equipment and techniques used for determining the physical composition of a coal sample in terms of volume percent of the organic components and of mineral matter, if desired. The components in a representative crushed coal sample prepared as described in Practice D2797 are identified under a microscope according to their reflectance, other optical properties, and morphology. The proportions of these components in a sample are determined by observing a statistically adequate number of points, and summing those representative of each component. Only area proportions of components are determined on a surface section of a sample. However, the area and volume proportions are the same when the components are randomly distributed throughout the sample.

TEST METHOD PRECISION AND BIAS

The precision of this procedure is being determined. Since there is no certified reference material available for such analyses, bias has not been determined.

SINGLE-STAGE TOTAL MOISTURE LESS THAN 15 % IN COAL REDUCED TO 2.36-mm (NO. 8 SIEVE) TOPSIZE

D2961

EXPLANATION

The measurement of total moisture is required to determine whether coal meets commercial or environmental specifications, or both. Within the limitations prescribed in the scope, this test method describes a procedure for the determination of total moisture that requires less time than the procedure described in D3302.

TEST METHOD SUMMARY

This test method covers a single-stage procedure for the determination of total moisture less than 15 % in coal reduced to 2.36 mm (No. 8 sieve) topsize. In this test method moisture is determined by establishing the weight loss of the coal sample by drying in an oven with forced air circulation. Materials subjected to this test shall not be used in the determination of other test parameters. It is recognized that the conditions of this test can increase the potential for significant oxidation effects on some coals. If the oxidation potential is of concern, the use of this single-stage method shall involve prior agreement between the parties involved. This test method shall not be construed as the referee standard test method for total moisture.

Based on several statistical studies, there is no bias between the results obtained by this test method and those by D3302, at the 95 % confidence level for moisture levels between 1.4 and 15.8 % . Some ranks of coal including lignite and anthracite were not included in that study.

TEST METHOD PRECISION AND BIAS

Repeatability Limit	0.30 %
Reproducibility Limit	0.50 %

Bias of this test method has not been determined.

MOISTURE IN THE ANALYSIS SAMPLE OF COAL AND COKE
D3173

EXPLANATION

Moisture as determined by this test method is used for calculating other analytical results to a moisture free basis using procedures in Practice D3180. When used in conjunction with the air drying loss as determined by Test Method D2013 or standard Practice D346, each analytical result can be calculated to an as-received basis.

TEST METHOD SUMMARY

Moisture is determined by establishing the loss in weight of the sample when heated under rigidly controlled conditions of temperature, time, atmosphere, sample weight, and equipment specifications.

TEST METHOD PRECISION AND BIAS

In the moisture range of 1.0 to 21.9 %, the following precision was found.

Repeatability Limit	$0.09 + 0.01\ X$
Reproducibility Limit	$0.23 + 0.02\ X$

Where X is the average of two single test results.

Certified reference materials are not available for the determination of bias by this test method.

TOTAL MOISTURE IN COAL
D3302

EXPLANATION

This test method covers the measurement of the total moisture in coal as it exists at the site, at the time, and under the conditions it is sampled. It is applicable to coals as mined, processed, shipped or used in normal commercial pursuits. It is not applicable to coal-water slurries, sludges or pulverized products under 0.5-mm diameter sieve size. It is applicable to coals of all ranks within the recognized limitations imposed by oxidation and decomposition characteristic of lower rank coals. Because of its empirical nature, strict adherence to basic principles and permissive procedures are required for valid results.

Since coals can vary from extremely wet (water saturated) to completely dry, special emphasis must be placed on the sampling, sample preparation, and the moisture determination itself to ensure total reliability of measurement.

The complete standard entailing collection of the gross sample, sample preparation, and the method of determination, is referred to as the referee method. Embodied in the standard is the commercial method starting with the crushed and divided sample when the gross sample is not too wet to crush and divide. The most commonly used commercial method is D2961.

TEST METHOD SUMMARY

This test method is based on the loss in weight of a coal sample in an air atmosphere under rigidly controlled conditions of temperature, time, and airflow.

The Referee Method may be used in cases of dispute or arbitration. The gross sample is air dried to equilibrate it with the atmosphere at each stage of division and reduction. No air drying is necessary if the sample is already at equilibrium with the atmosphere as indicated by a stable weight.

The Commercial Method may be used in routine commercial practice or when the concerned parties agree upon this method. The crushed and divided sample is air dried to equilibrate it with the atmosphere in which further division and reduction are to occur.

Residual moisture determination is made in a heated forced-air circulation oven under rigidly defined conditions. Total moisture is calculated by formula from loss (or gains) in air drying and the residual moisture. Sometimes users of moisture data simply add the moisture values without compensating for changes in basis.

TEST METHOD PRECISION AND BIAS

Coal Rank	Moisture Range, %	Repeatability, Limit, %	Reproducibility, Limit, %
Bituminous	1.6–7.9	0.14	0.62
Sub-bituminous and lignite	12.4–31.2	0.42	0.70

Since there is no acceptable reference material for determining the bias for this test method, no statement on bias is being made.

NITROGEN IN THE ANALYTICAL SAMPLE OF COAL AND COKE

D3179

EXPLANATION

Nitrogen results obtained by these test methods are required to fulfill the requirement of ultimate analysis, standard Practice D3176. Also, the results obtained may be used to evaluate the potential formation of nitrogen oxide as a source of atmospheric pollution. Nitrogen data are used in comparing coals and in research. When the oxygen content of coal is determined by difference, it is necessary to make a nitrogen determination.

TEST METHOD SUMMARY

Two test methods are included in this standard.

* Kjeldahl-Gunning macro analysis
* Kjeldahl-Gunning semi-micro analysis

In these procedures, nitrogen is converted into ammonium salts by destructive digestion of the sample with a hot, catalyzed mixture of concentrated sulfuric acid and potassium sulfate. These salts are subsequently decomposed in a hot alkaline solution from which the ammonia is recovered by distillation and finally determined by alkalimetric or acidimetric titration.

No significant interferences have been determined using these procedures. However, strict adherence is necessary when using these procedures to obtain good reproducible results.

TEST METHOD PRECISION AND BIAS

Nitrogen Range, wt. %	Repeatability Limit	Reproducibility Limit
0.85 to 1.68	$-0.24+0.31$ X	$-0.26+0.41$ X

Where X is the average of two single test results.

The bias of this test method has not been determined at present.

PLASTIC PROPERTIES OF COAL BY CONSTANT-TORQUE GIESELER PLASTOMETER

D2639

EXPLANATION

Reliable values of the plastic properties of coals are used to predict or explain the behavior of a coal or blends during carbonization or in other processes such as gasification, liquefaction, and combustion.

TEST METHOD SUMMARY

This test method covers a relative measure of the plastic behavior of coal when heated under prescribed conditions. The Gieseler plastometer measures the plastic properties of coals by the use of a constantly applied torque on a stirrer placed in a crucible into which the coal is placed and compared. The crucible is immersed in a bath and the temperature increased uniformly. The rates of movement of the stirrer are recorded in relation to the increase in temperature.

TEST METHOD PRECISION AND BIAS

Repeatability Limit—Temperature points should agree within 5 °C; maximum rates of drum dial movement should agree within ±10 %.

Reproducibility Limit—Numerous round robins have demonstrated the inherent variance of test equipment. This test method should, therefore, be used only as an indicator of the plastic properties of coal.

Bias—This is an empirical test method; hence, the degree of absolute bias cannot be determined.

PROXIMATE ANALYSIS OF THE ANALYSIS SAMPLE OF COAL AND COKE

D3172

EXPLANATION

The test methods as described here can be used to establish the rank of coals, show the ratio of combustible to noncombustible constituents, provide the basis for buying and selling, and evaluate for beneficiation or for other purposes.

TEST METHOD SUMMARY

This practice covers the determination of moisture, ash, and volatile matter, and the calculation of fixed carbon on coals and cokes sampled and prepared by prescribed methods and analyzed according to ASTM established procedures.

* Moisture	Test Method D3173 and D5142
* Ash	Test Method D3174 and D5142
* Volatile Matter	Test Method D3175 and D5142
* Fixed Carbon	Calculation

(Fixed Carbon, $\% = 100 - (\text{moisture, wt. } \% + \text{ash, wt. } \%$ $+ \text{volatile matter, wt. } \%)$

PROXIMATE ANALYSIS OF THE ANALYSIS SAMPLE OF COAL AND COKE BY INSTRUMENTAL PROCEDURES

D5142

EXPLANATION

See explanation in the Test Method D3172.

TEST METHOD SUMMARY

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 Test Method D2013 and standard Practice D346. The test results obtained have been shown sometimes to differ from those obtained with Test Methods D3173, D3174, and D3175 for coal and coke. Where a relative bias between this test method and the others mentioned above are shown to exist, the instrumental results shall be corrected or the instrument calibrated using samples of known proximate analysis. The latter methods shall be considered as the referee test methods for proximate analysis.

In these test methods, moisture, volatile matter, and ash may be determined sequentially in a single instrumental procedure. In some systems, the procedure consists of simply placing a specimen in the microprocessor-controlled instrument and initiating the automatic process of analysis. In other systems, the analytical process is manually controlled, although the instruments may have microprocessors to assist in controlling instrument conditions and for calculations.

TEST METHOD PRECISION AND BIAS

Parameter	Concentration, %	Repeatability Limit, %	Reproducibility Limit, %
Moisture	0.2–27.9	Auto: 0.2 + 0.012 X Man: 0.19 + 0.030 X	Auto: 0.24 + 0.034 X Man.: 0.49 + 0.036 X
Volatile Matter	1.0–50.8	Auto: 0.29 + 0.014 X Man.: 0.72 + 0.023 X	Auto: 0.62 + 0.047 X Man.: 1.95 + 0.028 X
Ash	6.0–19.6	Auto: 0.07 + 0.020 X Man.: 0.38	Auto: 0.14 + 0.023 X Man.: 1.23

+Where X is the average of two single test results.
+Where Auto are automated instruments and where Man. are semi-automated instruments.
These test methods are empirical and no suitable reference materials are available for the determination of the bias for these methods.

SIEVE ANALYSIS OF COKE D293

EXPLANATION

This test method determines the size distribution of coke for conformance to specifications of percentages retained on designated screen sizes. The method describes the separation of a coke sample into defined size fractions and expressing said fractions as a weight percent of the gross sample.

TEST METHOD SUMMARY

Square hole sieves conforming to standard Specification E11 are utilized. For complete characterization of the size range of a coke sample, the number and size of the selected sieves should be such that no more than 25 percent of the gross sample weight will be retained on any given sieve. Starting with the sieve having the largest opening, the coke sample should be sieved in quantities small enough to prevent plugging and clogging of the sieve.

TEST METHOD PRECISION AND BIAS

No statement regarding precision or bias can be made for this test method at present time.

SIEVE ANALYSIS OF COAL D4749

EXPLANATION

This test method concerns the sieving of coal into designated size fractions for the purpose of characterizing the material as to its particle size distribution for further processing or for commercial purposes. Raw as well as prepared (i.e., crushed, cleaned or screened) coal can be tested by this test method. This test method is applicable to all types of coals, except for pulverized coals such as those fed into steam boilers. Low rank coals, that is, lignites, sub-bituminous, and high volatile bituminous C, must be dried with caution and handled with care to minimize the deterioration or size degradation during sieving. This test method is applicable to wet or dry sieving of coal at sizes from 200 mm (8 in.) to 38 μm (No. 400 USA Standard). Methods for sizing materials below 38 μm are outside the scope of this method. This test method also concerns the designation of a coal sample as to its upper (nominal top-size) and lower (nominal bottom-size) limiting sizes for the purpose of characterizing the material for further processing or for commercial purposes.

This test method explains how to designate coal sizes from the results of sieve analysis data in order to represent the condition of the coal as sold. In the case of special mixtures or coals with non-continuous ranges of sizes, a sufficiently complete sieve analysis must be made to properly describe the size distribution. Enough material may not be collected by this test method to meet subsequent test procedures, such as washability analyses (Test Method D4371).

TEST METHOD SUMMARY

Standard test sieves that conform to standard Specification E11 are always used for wire cloth sieves. Perforated plate sieves are made to conform to standard Specification E323. Mechanical sieve shakers are used. Of these, batch types are acceptable; continuous types are not. A gross sample of coal is collected per Test Method D2234. Its weight also must conform to Test Method D2234. When necessary, the gross sample is divided and air dried per Test Method D2013. The gross sample is divided into test samples according to Test Methods D2013 or D2234. If necessary, the sample is dried per the Test Method D2013. The test-ready samples may be hand sieved, by using a mechanical dry sieving technique, by using wet sieving, or by using combined wet and dry sieving. The sum of the size fractions masses are calculated as the sum of the combined mass.

Sieve analyses are often presented graphically for comparison with specification requirements, or for general evaluation. Among the several methods of graphical representation of test results that have a wide acceptance are the Rosin-Rammler and the Gaudin-Schuhmann plots.

Part B of this test method uses the sieve data to convert to coal size designation.

TEST METHOD PRECISION AND BIAS

No precision statement has been developed for this test method because of the impracticality of obtaining, transporting, and handling representative splits of the materials in the quantities that would be needed to establish the precision statement. The lack of a reference material precludes a bias statement.

SULFATE SULFUR IN ASH FROM COAL AND COKE
D1757

EXPLANATION

Laboratory furnace temperature and furnace ventilation have an influence on SO_3 retention in the laboratory ash. Consequently, sulfur in ash as determined in the laboratory cannot be assumed to be equivalent to sulfur present in the mineral matter in coal or to the retention of sulfur in ash produced under the conditions of commercial utilization.

The sulfate sulfur determined by these test methods can be used to calculate the SO_3 portion of ash so that the ash content or ash composition can be reported on a SO_3 free basis.

TEST METHOD SUMMARY

Test method A is a modified British method in which a specified quantity of ash is digested in boiling dilute HCl solution to which bromine water is added to convert sulfite that may be present to the sulfate form. After neutralization and precipitation of iron with ammonium hydroxide, the mixture is filtered, and the sulfate in the filtrate is gravimetrically determined as barium sulfate.

In test method B, a specified quantity of the ash is ignited in air with Eschka mixture. The sulfates are dissolved in hot water and separated from undissolved ash residue and MgO by filtration. The sulfate in the filtrate is gravimetrically determined as barium sulfate.

Interferences—Barium in coal ashes can result in incomplete recovery of sulfate sulfur. High iron content can be encountered in coal and coke ash and can introduce error if, during the sulfate precipitation, the iron is partially absorbed by the barium sulfate precipitate or coprecipitated as iron sulfate.

TEST METHOD PRECISION AND BIAS

SO_3 Range, %	British Method: Repeatability Limit	British Method: Reproducibility Limit	Eschka Method: Repeatability Limit	Eschka Method: Reproducibility Limit
<1	0.1	0.2	0.1	0.2
$>1-<5$	0.2	0.3	0.3	0.4
$>5-<10$	0.4	0.4	0.4	0.8
$>10-<20$	0.6	1.0	0.5	1.0

Bias of this test method has not been determined.

FORMS OF SULFUR IN COAL
D2492

EXPLANATION

This test method provides for a separation of coal-associated sulfur into two commonly recognized forms: pyritic and sulfate. Organic sulfur is calculated by difference. Results obtained by this test method are used to serve a number of interests, including the evaluation of coal preparation and processing operations designed to reduce coal sulfur levels. The test method is not applicable to coke or other carbonaceous materials. Monosulfides of iron and elements such as Cd, Pb, V, and Zn can be present in coal. In the concentration up to 100 ppm, these monosulfides do not contribute significantly to the total inorganic sulfide content.

TEST METHOD SUMMARY

Sulfate Sulfur is extracted from the analysis sample with dilute HCl. The sulfate sulfur in the extract is gravimetrically determined as barium sulfate. Sulfates are soluble in hydrochloric acid, but pyritic and organic sulfur are not.

Pyritic Sulfur is calculated as a stoichiometric combination with iron. Using two different methods:

The Referee Method can be used in cases of dispute or arbitration. The iron combined in the pyritic state is extracted with dilute nitric acid from the coal residue remaining after sulfate extraction. The iron is determined with atomic absorption spectrometry.

The Alternative Method can be used in routine practice or when the concerned parties agree on this test method. The iron originally combined in the pyritic state can be extracted with dilute HCl from the ash obtained by incinerating the coal residue remaining after sulfate extraction. The iron is determined by atomic absorption spectrometry.

TEST METHOD PRECISION AND BIAS

Sulfur Form	Range, wt. %	Repeatability Limit, %	Reproducibility Limit, %
Sulfate Sulfur		0.02	0.04
Pyritic Sulfur	0.1 to 12		
-Referee Method		0.08+0.09 X	0.15+0.27 X
-Alternative Method		0.06+0.11 X	0.07+0.19 X

Where X is the average of two single test results.

Since no suitable certified reference materials for these analyses are available, absolute bias cannot be determined for this test method. However, based on matched pair comparison, the alternative method for pyritic sulfur was found to be biased high with respect to the referee method, with minimum detectable bias being 0.06 %.

TOTAL SULFUR IN COAL AND COKE

Determination of total sulfur is, by definition, part of the ultimate analysis of coal. Sulfur results obtained by these methods are used to serve a number of interests: evaluation of coal preparation, evaluation of potential sulfur emissions from coal combustion or conversion processes, evaluation of the coal quality in relation to contract specification, and other purposes of commercial or scientific interest.

There are three test methods available for the determination of total sulfur in coal and coke.

SULFUR IN COAL AND COKE
D3177

TEST METHOD SUMMARY

These test methods cover two alternative procedures.

(a) In the Eschka method, a weighed sample and the Eschka mixture are intimately mixed and ignited together. The sulfur is dissolved in hot water and then precipitated from the resulting solution as barium sulfate. The precipitate is filtered, washed, ashed, and weighed.

(b) In the bomb washing method, sulfur is precipitated as barium sulfate from oxygen-bomb calorimeter washings and the precipitate is filtered, washed, ashed, and weighed.

TEST METHOD PRECISION AND BIAS

Range, %	Repeatability Limit, %	Reproducibility Limit, %
Under 2	0.05	0.10
Over 2	0.10	0.20
Coke	0.03	0.05

There is no bias known for this method.

SULFUR IN COAL AND COKE
D4239

TEST METHOD SUMMARY

These test methods cover two alternative procedures using high temperature tube furnace combustion methods for the rapid determination of total sulfur in coal and coke. When automatic equipment is used to perform either of these two methods, the procedures can be classified as instrumental methods. There are several manufacturers that offer such instruments. The instrumental methods are especially applicable when results must be obtained rapidly for the successful completion of industrial, beneficiation, trade, or other evaluations.

Method A—A weighed sample is burned in a tube furnace at 1350°C in a stream of oxygen. During combustion, all sulfur present in the sample is oxidized to gaseous oxides of sulfur, and the chlorine in the sample released as gaseous hydrogen chloride. These products are then absorbed into a solution of hydrogen peroxide where they dissolve to form dilute solutions of sulfuric and hydrochloric acids. The quantities of both acid produced are directly dependent upon the amounts of sulfur and chlorine present in the original sample. Once the amounts of each acid present have been determined the percentage of sulfur contained in the coal may be calculated.

Method B—The sample is combusted in a tube furnace at 1350°C in a stream of oxygen to oxidize the sulfur present in the sample. Moisture and particulates are removed from the gas by traps filled with anhydrous magnesium perchlorate. The gas stream is passed through a cell in which sulfur dioxide is measured by an infrared absorption detector. One cell is used as both a reference and a measurement chamber. Total sulfur as sulfur dioxide is determined on a continuous basis. This method is empirical; therefore, the apparatus must be calibrated by using certified reference materials.

The high temperature combustion method with an iodimetric detection procedure, formerly Method B of this standard and published up to the 2000 version is still a viable method that may be used.

TEST METHOD PRECISION AND BIAS

Method	Coal Type	Range, %	Repeatability Limit, %	Reproducibility Limit, %
Acid-base detection	250 μm (No. 60) samples of coal and coke	0.5–6.0	0.06+0.03 X	0.03+0.11 X
IR Detection	Coal and coke	0.28–5.61	0.02+0.03 X	0.02+0.09 X
IR Detection	Bituminous 2.36 mm (No. 8) sample	0.2–3.0	0.05 %	0.09 %
IR Detection	Sub-Bituminous Lignite	0.3–1.6	0.8 %	0.13 %

Where X is the average of two single test results.

Bias of this method is eliminated when the instrument is properly calibrated against Certified Reference Materials.

D5016

TEST METHOD SUMMARY

This test method describes a procedure using a high temperature tube furnace and infrared detection for the rapid determination of sulfur in ash from coal and coke. This test method is an alternative for test methods described in Test Method D1757.

A weighed test portion is mixed with a promoting agent and ignited in a tube furnace at 1350°C in a stream of oxygen. The combustible sulfur present in the sample is oxidized to gaseous oxides of sulfur. Moisture and particulates are removed by traps filled with anhydrous magnesium perchlorate. The gas stream is passed through a cell in which sulfur dioxide is measured by an infrared absorption detector. One cell is used both as a reference and a measurement chamber. Total sulfur as sulfur dioxide is detected on a continuous basis. This test method is applicable for use with commercially available sulfur analyzers equipped to carry out the above operations automatically, and must be calibrated using certified reference materials covering the range of sulfur in the ash samples being analyzed.

TEST METHOD PRECISION AND BIAS

Sulfur Range, wt. %	Repeatability Limit, %	Reproducibility Limit, %
0.7 to 3.8	0.06 X+0.06	0.2 X+0.2

where X is the average of two single test results.

Compared against NIST SRM 1633b this test method shows a −8.1 % bias.

FREE SWELLING INDEX OF COAL
D720

EXPLANATION

This test method, in addition to indicating the caking properties of a coal when burned as a fuel, can be used to give a broad indication of the degree of oxidation of a coal. This test method is a small-scale for obtaining information regarding the free-swelling properties of a coal. This test is not recommended as a method for the determination of expansion of coals in coke ovens.

TEST METHOD SUMMARY

The coal sample is ground to pass a 250 μm (No. 60) sieve. The test should be performed within 24 h of the time that a sample is pulverized to 60 mesh. For some coals, size consist of the sample is a variable that can affect the size of the coke button produced. The coke buttons are prepared either using an electric furnace or gas flame method. A run is completed in 2.5 min. Three buttons are prepared in each case. The prepared coke buttons are viewed and compared with a series of standard profiles. Mechanical or electronic devices may be used to determine the standard profiles if they give results equivalent to those obtained with manual measurement technique.

TEST METHOD PRECISION AND BIAS

Relative precision of this test method for the determination of free swelling index covers the index range from 1 to 9. Repeatability limit is one standard profile, and the reproducibility limit is two units. Since there is no accepted reference material suitable for determining bias of this test method, bias has not been determined.

SWELLING PROPERTIES OF BITUMINOUS COAL USING A DILATOMETER

D5515

EXPLANATION

Values of the dilatation properties of coal may be used to predict or explain the behavior of a coal or blends during carbonization or in other processes such as gasification, liquefaction, and combustion. This test method is limited in applicability to those coals which have a free swelling index equal to or greater than 1 as determined with Test Method D720.

It is also of note that the Ruhr and Audibert-Arnu methods while still being used in the international community have been officially withdrawn from ISO for a number of years. The ISO dilatation method is currently undergoing thorough ruggedness testing in Australia (ACARP-sponsored project) to evaluate the numerous dilatation methods used in metallurgical coal evaluation laboratories (Ruhr, Audibert-Arnu, ASTM, Australian standards and the new ISO draft) for developing the most suitable international dilatation standard.

TEST METHOD SUMMARY

The principle of the ASTM test method is that the final volume of char obtained at the conclusion of a standard dilatation test is dependent on the mass of the coal in the coal pencil and on the radius of the retort tube. This method incorporates a procedure which determines the mass of air-dried coal in the coal pencil, provides a means to measure the average retort tube radii, and employ a means to report coal expansion on an air-dried coal weight basis. The test involves preparing a coal pencil and determining the changes of the coal pencil height in a retort tube during a prescribed heating cycle.

TEST METHOD PRECISION AND BIAS

Measured Parameter	Range	Repeatability Limit	Reproducibility Limit
T1	360–440°C	6°C	20°C
T2	415–475°C	6°C	20°C
T3	465–495°C	6°C	20°C
% $D_{2.5}$	<100 %	7 %	17 %
% $D_{2.5}$	100–200 %	16	35

Since no suitable reference materials are currently available for this analysis, no statement on bias for this test method can be made.

TUMBLER TEST FOR COAL

D441

EXPLANATION

This test method is a measure of the resistance of the coal to breakage upon impact and abrasion as coal rubs together and against the walls of the transportation vehicle. The test affords a means of measuring the liability of coal to break into smaller pieces when subjected to repeated handling at the mine or subsequently, by the distributor or by the consumer. This test method is serviceable for ascertaining the similarity of coals in respect to friability rather than for determining values within narrow limits to emphasize their dissimilarity. This test method may also serve to indicate the relative extent to which sized coals will suffer size degradation in certain mechanical feed devices. This test method may be used for differentiating between certain ranks and grades of coal, and therefore the test method is of service for coal classification purposes.

TEST METHOD SUMMARY

About 1 kg of coal prepared according to Test Method D410 is placed in a jar and rotated for 1 h in the tumbler test machine at 40±1 rpm. After tumbling, the coal is thoroughly graded as to size on the sieves either by hand or mechanically, though the former method is preferable. At least four single-jar tests are made, if sufficient sample is available. The weight percent distribution is determined.

TEST METHOD PRECISION AND BIAS

Data for test method precision do not exist but are being determined. This is an empirical test method and no statement of bias is possible.

TUMBLER TEST FOR COKE
D3402

EXPLANATION

Coke undergoes various degrees of degradation during removal from coke ovens, transportation, and its descent within a blast furnace to the combustion zone. These processes subject the coke to impact and abrasion. This test method is a relative measure of the resistance of coke to breakage when subjected to these degradation processes.

TEST METHOD SUMMARY

In the standard procedure, a coke sample sized to −75 mm (−3 in.) and +50 mm (+2 in.) is used. In the alternative procedure, 50 percent of coke sized 63 × 50 mm (2.5 × 2 in.) and 50 percent of 50 × 37.5 mm (2 × 1.5 in.) is used. A 10 ± 0.25 kg portion of the −3 in +2 in sized sample or a 5 ± 0.25 kg portion of the alternatively sized sample is placed in the drum of the tumbling machine and rotated at 24 ± 1 rpm for a total of 1400 revolutions. Coke is removed from the tumbler and is sieved using a 25 mm (1 in.) square mesh sieve and a 6.3 mm (0.25 in.) sieve. The coke remaining on each sieve and the material passing through the 6.3 mm sieve are weighed.

TEST METHOD PRECISION AND BIAS

See above under Test Method D441.

ULTIMATE ANALYSIS OF COAL AND COKE
D3176

EXPLANATION

Summarizing the ash content and the content of the organic constituents in a specific format under the heading, *Ultimate Analysis*, provides a convenient and uniform system for comparing coals or cokes. This tabulation used with that of *Proximate Analysis* (standard Practice D3172) permits cursory valuation of coals for use as fuel or in other carbonaceous processes and of cokes for metallurgical purpose.

TEST METHOD SUMMARY

Following Test Methods and Practices are used for collecting data for ultimate analysis.

Coal Sample Collection	D2234
Coal Sample Preparation	D2013
Coke Sampling and Preparation	D346
Carbon and Hydrogen	D3178 or D5373
Sulfur	D3177 or D4239
Nitrogen	D3179 or D5373
Ash	D3174 or D5142
Oxygen	By difference
Moisture	D3173 or D5142

The data in the ultimate analysis may be reported on as determined basis, moisture free basis, or dry basis.

TEST METHOD PRECISION AND BIAS

The permissible difference between two or more determinations shall not exceed the values listed in the precision sections of the specific test methods for the parameters determined.

VOLATILE MATTER IN THE ANALYSIS SAMPLE OF COAL AND COKE
D3175

EXPLANATION

Volatile matter, when determined as described in this test method, may be used to establish the rank of coals, to indicate coke yield on carbonization process, to provide the basis for purchasing or selling, or to establish the burning characteristics. In this test method, the percentage of gaseous volatiles in the sample, exclusive of moisture vapor, which are released under the specific conditions of the test is determined. This test method is empirical in nature; hence, strict adherence to basic principles and permissible procedures is required to obtain valid results. D5142 is more commonly used for volatile matter measurements than D3175 test method.

TEST METHOD SUMMARY

Volatile matter is determined by establishing the loss in weight resulting from heating a coal or a coke sample under rigidly controlled conditions. The use of platinum crucibles is considered the standard reference method for volatile matter. Volatile matter determinations using alternate nickel-chromium alloy crucibles having the same physical dimensions have been shown to differ from those obtained using platinum crucibles. A laboratory using a nickel-chromium crucible should first establish if there is a bias between their results and those obtained using platinum crucibles. If a relative bias is shown to exist, the volatile matter results determined using nickel-chromium crucibles shall be corrected for the bias.

TEST METHOD PRECISION AND BIAS

Rank	Repeatability Limit, %	Reproducibility Limit, %
High-temperature coke	0.2	0.4
Anthracite	0.3	0.6
Semi-anthracite, bituminous, low temperature coke, & chars	0.5	1.0
Subbituminous	0.7	1.4
Lignite & peat	1.0	2.0

Since this is an empirical test method, the degree of absolute bias cannot be determined. Bias between the use of platinum and nickel-chromium crucibles (mentioned above) shall be determined in the laboratory on the coal being tested.

WASHABILITY CHARACTERISTICS OF COAL
D4371

EXPLANATION

This test method standardizes procedures utilized for performing washability analyses, the data from which can be used for interpreting preparation plant efficiency, for determining preparation plant design, and for determining the potential recovery and quality of coal reserves. This test method is also used for assisting in process control and to determine the efficiency of operating preparation plants.

Each sample being analyzed can have more than one coarse-coal size fraction and more than one fine-coal size fraction. While in general this test method has useful application in determining the washability characteristics of low rank coals, in the strictest sense, it is not necessarily the most technically correct test method for such analysis because of problems relative to the loss of moisture through drying during sample preparation and analysis. Methods that are directly applicable to low rank coals are to be developed.

The usefulness of this test and results generated from using it are directly related to the care exercised in developing and following the sampling regime while taking into consideration the desired degree of precision. For most exacting work, it would be necessary to know the general washability characteristics of the coal being analyzed.

TEST METHOD SUMMARY

In Procedure A, each coarse-coal size fraction is independently separated into two distinct specific gravity fractions by placing the sample in a large tank containing a screen basket insert and a solution of organic liquids to the required specific gravity. The material that floats on the solution is first removed, followed by the portion of the sample that sinks.

In Procedure B, similar to Procedure A a sample of fine-coal size fraction is placed in a float/sink flask containing a solution of organic solution of required specific gravity. This suspension is then stirred. The material that floats is first carefully removed by filtration. The sink material is also then similarly removed.

In all cases above, the material collected is dried and weighed.

TEST METHOD PRECISION AND BIAS

Generally, the precision and bias statement for samples of a topsize larger than 6.3 mm cannot be easily developed because of the impracticality of obtaining, transporting, and handling representative sample in quantities that would be necessary. The data below is given for samples of size fractions of 6.3 mm × 1.18 mm.

Parameter	Specific Gravity Fraction	Repeatability Limit	Reproducibility Limit
Recovery	<1.40	273 g	490 g
	1.40–1.65	(0.247 (W) + 59) g	(0.296 (W) + 17) g
	>1.65	(0.168 (W) − 49) g	(0.181 (W) − 16) g
Moisture	<1.40	0.285 (M, %) − 0.03	0.559 (M, %) + 0.14
	1.40–1.65	0.533 (M, %) − 0.39	0.626 (M, %) + 0.09
	>1.65	0.824 (M, %) − 0.81	0.760 (M, %) − 0.12
Ash, dry basis	<1.40	0.38 %	0.66 %
	1.40–1.65	1.07 %	2.15 %
	>1.65	2.08 %	4.29 %
Sulfur, dry basis	<1.40	0.047 (S, %) + 0.028	0.128 (S,%)
	1.40–1.65	0.110 (S, %) − 0.072	0.190 (S, %) − 0.083
	>1.65	0.125 (S, %) − 0.020	0.199 (S, %) + 0.063

Where W is the weight of the sample recovered at that specific gravity fraction, g.

Where M is the moisture content of the sample recovered at that specific gravity fraction.

Where S is the sulfur content of the sample recovered at that specific gravity fraction.

Since there is no accepted reference material suitable for determining the bias for this test method, bias has not been determined.

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