



Standard Test Method for Conradson Carbon Residue of Petroleum Products¹

This standard is issued under the fixed designation D189; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the determination of the amount of carbon residue (Note 1) left after evaporation and pyrolysis of an oil, and is intended to provide some indication of relative coke-forming propensities. This test method is generally applicable to relatively nonvolatile petroleum products which partially decompose on distillation at atmospheric pressure. Petroleum products containing ash-forming constituents as determined by Test Method D482 or IP Method 4 will have an erroneously high carbon residue, depending upon the amount of ash formed (Note 2 and Note 4).

NOTE 1—The term *carbon residue* is used throughout this test method to designate the carbonaceous residue formed after evaporation and pyrolysis of a petroleum product under the conditions specified in this test method. The residue is not composed entirely of carbon, but is a coke which can be further changed by pyrolysis. The term *carbon residue* is continued in this test method only in deference to its wide common usage.

NOTE 2—Values obtained by this test method are not numerically the same as those obtained by Test Method D524. Approximate correlations have been derived (see Fig. X1.1), but need not apply to all materials which can be tested because the carbon residue test is applied to a wide variety of petroleum products.

NOTE 3—The test results are equivalent to Test Method D4530, (see Fig. X1.2).

NOTE 4—In diesel fuel, the presence of alkyl nitrates such as amyl nitrate, hexyl nitrate, or octyl nitrate causes a higher residue value than observed in untreated fuel, which can lead to erroneous conclusions as to the coke forming propensity of the fuel. The presence of alkyl nitrate in the fuel can be detected by Test Method D4046.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Liquid Fuels and Lubricants.

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In the IP, this test method is under the jurisdiction of the Standardization Committee and is issued under the fixed designation IP 13. The final number indicates the year of last revision. This test method was adopted as a joint ASTM–IP standard in 1964.

This procedure is a modification of the original Conradson method and apparatus for Carbon Test and Ash Residue in Petroleum Lubricating Oils. See *Proceedings, Eighth International Congress of Applied Chemistry*, New York, Vol 1, p. 131, September 1912; also *Journal of Industrial and Engineering Chemistry*, IECHA, Vol 4, No. 11, December 1912.

In 1965, a new Fig. 2 on reproducibility and repeatability combining ASTM and IP precision data replaced old Fig. 2 and Note 4.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—<http://www.epa.gov/mercury/faq.htm>—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

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

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

- D482 Test Method for Ash from Petroleum Products
- D524 Test Method for Ramsbottom Carbon Residue of Petroleum Products
- D4046 Test Method for Alkyl Nitrate in Diesel Fuels by Spectrophotometry (Withdrawn 2019)³
- D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ The last approved version of this historical standard is referenced on www.astm.org.

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D4530 Test Method for Determination of Carbon Residue (Micro Method)

E1 Specification for ASTM Liquid-in-Glass Thermometers

E133 Specification for Distillation Equipment

3. Terminology

3.1 Definitions:

3.1.1 *carbon residue, n*—the residue formed by evaporation and thermal degradation of a carbon containing material.

3.1.1.1 *Discussion*—The residue is not composed entirely of carbon but is a coke that can be further changed by carbon pyrolysis. The term carbon residue is retained in deference to its wide common usage.

D4175

4. Summary of Test Method

4.1 A weighed quantity of sample is placed in a crucible and subjected to destructive distillation. The residue undergoes cracking and coking reactions during a fixed period of severe heating. At the end of the specified heating period, the test crucible containing the carbonaceous residue is cooled in a desiccator and weighed. The residue remaining is calculated as a percentage of the original sample, and reported as Conradson carbon residue.

5. Significance and Use

5.1 The carbon residue value of burner fuel serves as a rough approximation of the tendency of the fuel to form deposits in vaporizing pot-type and sleeve-type burners. Similarly, provided alkyl nitrates are absent (or if present, provided the test is performed on the base fuel without additive) the carbon residue of diesel fuel correlates approximately with combustion chamber deposits.

5.2 The carbon residue value of motor oil, while at one time regarded as indicative of the amount of carbonaceous deposits a motor oil would form in the combustion chamber of an engine, is now considered to be of doubtful significance due to the presence of additives in many oils. For example, an ash-forming detergent additive may increase the carbon residue value of an oil yet will generally reduce its tendency to form deposits.

5.3 The carbon residue value of gas oil is useful as a guide in the manufacture of gas from gas oil, while carbon residue values of crude oil residua, cylinder and bright stocks, are useful in the manufacture of lubricants.

6. Apparatus (see Fig. 1)

6.1 *Porcelain Crucible*, wide form, glazed throughout, or a silica crucible; 29 mL to 31 mL capacity, 46 mm to 49 mm in rim diameter.

6.2 *Iron Crucible*—Skidmore iron crucible, flanged and ringed, 65 mL to 82 mL capacity, 53 mm to 57 mm inside and 60 mm to 67 mm outside diameter of flange, 37 mm to 39 mm in height supplied with a cover without delivery tubes and

having the vertical opening closed. The horizontal opening of about 6.5 mm shall be kept clean. The outside diameter of the flat bottom shall be 30 mm to 32 mm.

6.3 *Iron Crucible*—Spun sheet-iron crucible with cover; 78 mm to 82 mm in outside diameter at the top, 58 mm to 60 mm in height, and approximately 0.8 mm in thickness. Place at the bottom of this crucible, and level before each test, a layer of about 25 mL of dry sand, or enough to bring the Skidmore crucible, with cover on, nearly to the top of the sheet-iron crucible.

6.4 *Wire Support*—Triangle of bare Nichrome wire of approximately No. 13 B & S gage having an opening small enough to support the bottom of the sheet-iron crucible at the same level as the bottom of the heat-resistant block or hollow sheet-metal box (6.6).

6.5 *Hood*—Circular sheet-iron hood from 120 mm to 130 mm in diameter the height of the lower perpendicular side to be from 50 mm to 53 mm; provided at the top with a chimney 50 mm to 60 mm in height and 50 mm to 56 mm in inside diameter, which is attached to the lower part having the perpendicular sides by a cone-shaped member, bringing the total height of the complete hood to 125 mm to 130 mm. The hood can be made from a single piece of metal, provided it conforms to the foregoing dimensions. As a guide for the height of the flame above the chimney, a bridge made of approximately 3 mm iron or Nichrome wire, and having a height of 50 mm above the top of the chimney, shall be attached.

6.6 *Insulator*—Heat-resistant block, refractory ring, or hollow sheet-metal box, 150 mm to 175 mm in diameter if round, or on a side if square, 32 mm to 38 mm in thickness, provided with a metal-lined, inverted cone-shaped opening through the center; 83 mm in diameter at the bottom, and 89 mm in diameter at the top. In the case of the refractory ring no metal lining is necessary, providing the ring is of hard, heat-resistant material.

NOTE 5—It is not known what type of insulators were used in the round robin conducted for obtaining the precision given in Section 13.

6.7 *Burner*, Meker type, having an orifice approximately 24 mm in diameter.

7. Sampling

7.1 For sampling techniques see Practices **D4057** and **D4177**.

8. Procedure

8.1 Shake thoroughly the sample to be tested, first heating to $50^{\circ}\text{C} \pm 10^{\circ}\text{C}$ for 0.5 h when necessary to reduce its viscosity. Immediately following the heating and shaking, filter test portion through a 100 mesh screen. Weigh to the nearest 5 mg a 10 g sample of the oil to be tested, free of moisture and other suspended matter, into a tared porcelain or silica crucible containing two glass beads about 2.5 mm in diameter. Place this crucible in the center of the Skidmore crucible. Level the sand in the large sheet-iron crucible and set the Skidmore crucible on it in the exact center of the iron crucible. Apply

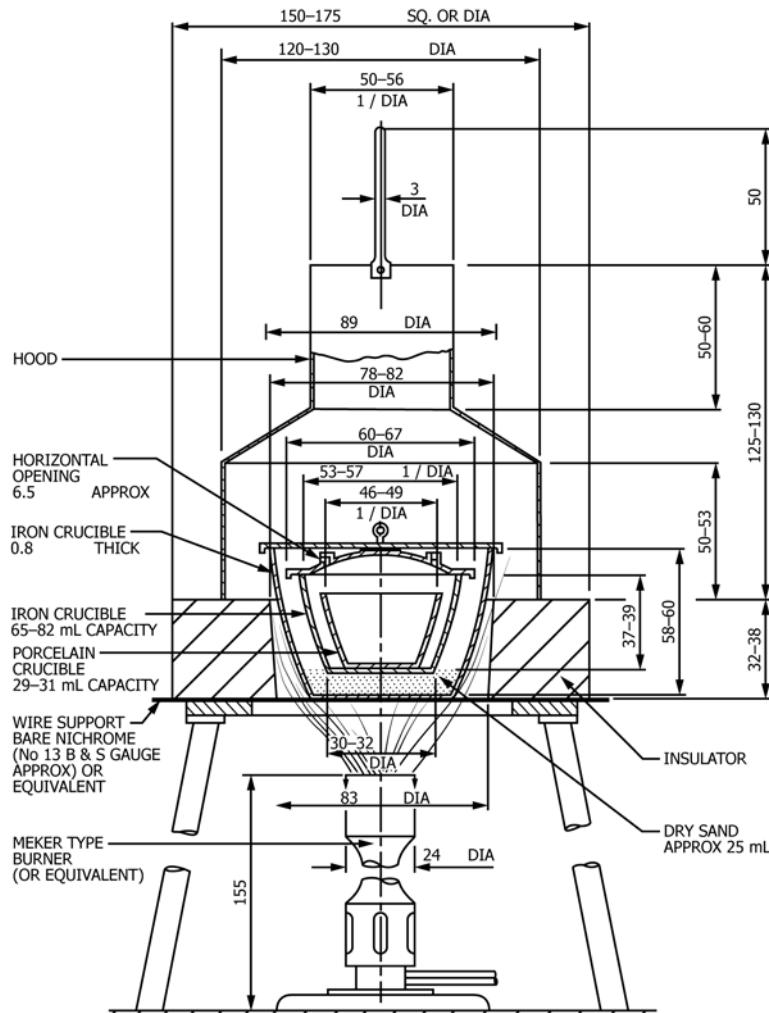


FIG. 1 Apparatus for Determining Conradson Carbon Residue

covers to both the Skidmore and the iron crucible, the one on the latter fitting loosely to allow free exit to the vapors as formed.

8.2 On a suitable stand or ring, place the bare Nichrome wire triangle and on it the insulator. Next center the sheet-iron crucible in the insulator with its bottom resting on top of the triangle, and cover the whole with the sheet-iron hood in order to distribute the heat uniformly during the process (see Fig. 1).

8.3 Apply heat with a high, strong flame from the Meker-type gas burner, so that the pre-ignition period will be $10 \text{ min} \pm 1.5 \text{ min}$ (a shorter time can start the distillation so rapidly as to cause foaming or too high a flame). When smoke appears above the chimney, immediately move or tilt the burner so that the gas flame plays on the sides of the crucible for the purpose of igniting the vapors. Then remove the heat temporarily, and before replacing adjust by screwing down the pinch-cock on the gas tubing so that the ignited vapors burn uniformly with the flame above the chimney but not above the wire bridge. Heat can be increased, if necessary, when the flame does not show above the chimney. The period of burning the vapors shall be $13 \text{ min} \pm 1 \text{ min}$. If it is found impossible to meet the

requirements for both flame and burning time, the requirement for burning time is the more important.

8.4 When the vapors cease to burn and no further blue smoke can be observed, readjust the burner and hold the heat as at the beginning so as to make the bottom and lower part of the sheet-iron crucible a cherry red, and maintain for exactly 7 min. The total period of heating shall be $30 \text{ min} \pm 2 \text{ min}$, which constitutes an additional limitation on the tolerances for the pre-ignition and burning periods. There should be no difficulty in carrying out the test exactly as directed with the gas burner of the type named, using city gas (20 MJ/m^3 to 40 MJ/m^3), with the top of the burner about 50 mm below the bottom of the crucible. The time periods shall be observed with whatever burner and gas is used.

8.5 Remove the burner and allow the apparatus to cool until no smoke appears, and then remove the cover of the Skidmore crucible (about 15 min). Remove the porcelain or silica crucible with heated tongs, place in the desiccator, cool, and weigh. Calculate the percentage of carbon residue on the original sample.

9. Procedure for Residues Exceeding 5 %

9.1 This procedure is applicable to such materials as heavy crude oils, residuums, heavy fuel oils, and heavy gas oils.

9.2 When the carbon residue as obtained by the procedure described in Section 8 (using a 10 g sample) is in excess of 5 %, difficulties can be experienced due to boiling over of the sample. Trouble also can be encountered with samples of heavy products which are difficult to dehydrate.

9.3 For samples showing more than 5.0 % and less than 15.0 % carbon residue by the procedure described in Section 8, repeat the test using a $5\text{ g} \pm 0.5\text{ g}$ sample weighed to the nearest 5 mg. In event that a result greater than 15.0 % is obtained, repeat the test, reducing the sample size to $3\text{ g} \pm 0.1\text{ g}$, weighed to the nearest 5 mg.

9.4 If the sample boils over, reduce the sample size first to 5 g and then to 3 g as necessary to avoid the difficulty.

9.5 When the 3 g sample is used, it can be impossible to control the preignition and vapor burning times within the limits specified in 8.3. However, in such cases, the results shall be considered as valid.

10. Procedure for Carbon Residue on 10 % Distillation Residue

10.1 This procedure is applicable to light distillate oils, such as ASTM No. 1 and No. 2 fuel oils.

10.2 Assemble the distillation apparatus described in Specification E133 using flask D (250 mL bulb volume), flask support board with 50 mm diameter opening, and graduated cylinder C (200 mL capacity). A thermometer is not required but the use of the ASTM High Distillation Thermometer 8F or 8C as prescribed in Specification E1 or the IP High Distillation Thermometer 6C, as prescribed in the IP Thermometer Specifications is recommended.

10.3 Place a volume of sample equivalent to 200 mL at 13°C to 18°C in the flask. Maintain the condenser bath at 0°C to 4°C (for some oils it may be necessary to hold the temperature between 38°C and 60°C to avoid solidification of waxy material in the condenser tube). Use, without cleaning, the cylinder from which the sample was measured as the receiver and place it so that the tip of the condenser does not touch the wall of the cylinder.

10.4 Apply the heat to the flask at a uniform rate so regulated that the first drop of condensate exits from the condenser between 10 min and 15 min after initial application of heat. After the first drop falls, move the receiving cylinder so that the tip of the condenser tube touches the wall of the cylinder. Then regulate the heat so that the distillation proceeds at a uniform rate of 8 mL/min to 10 mL/min. Continue the distillation until 178 mL of distillate has been collected, then discontinue heating and allow the condenser to drain until 180 mL (90 % of the charge to the flask) has been collected in the cylinder.

10.5 Immediately replace the cylinder with a small Erlenmeyer flask and catch any final drainage in the flask. Add to this flask, while still warm, the distillation residue left in the distilling flask, and mix well. The contents of the flask then represents a 10 % distillation residue from the original product.

10.6 While the distillation residue is warm enough to flow freely, pour approximately $10\text{ g} \pm 0.5\text{ g}$ of it in the previously weighed crucible to be used in the carbon residue test. After cooling, determine the weight of the sample to the nearest 5 mg and carry out the carbon residue test in accordance with the procedure described in Section 8.

11. Calculation

11.1 Calculate the carbon residue of the sample or of the 10 % distillation residue as follows:

$$\text{Carbon residue} = (A \times 100)/W$$

where:

A = mass of carbon residue, g, and
 W = mass of sample, g.

12. Report

12.1 Report the value obtained as *Conradson Carbon Residue, percent* or as *Conradson Carbon Residue on 10 % distillation residue, percent*, Test Method D189.

13. Precision and Bias⁴

13.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

13.1.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values shown in Fig. 2 only in one case in twenty.

13.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values shown in Fig. 2 only in one case in twenty.

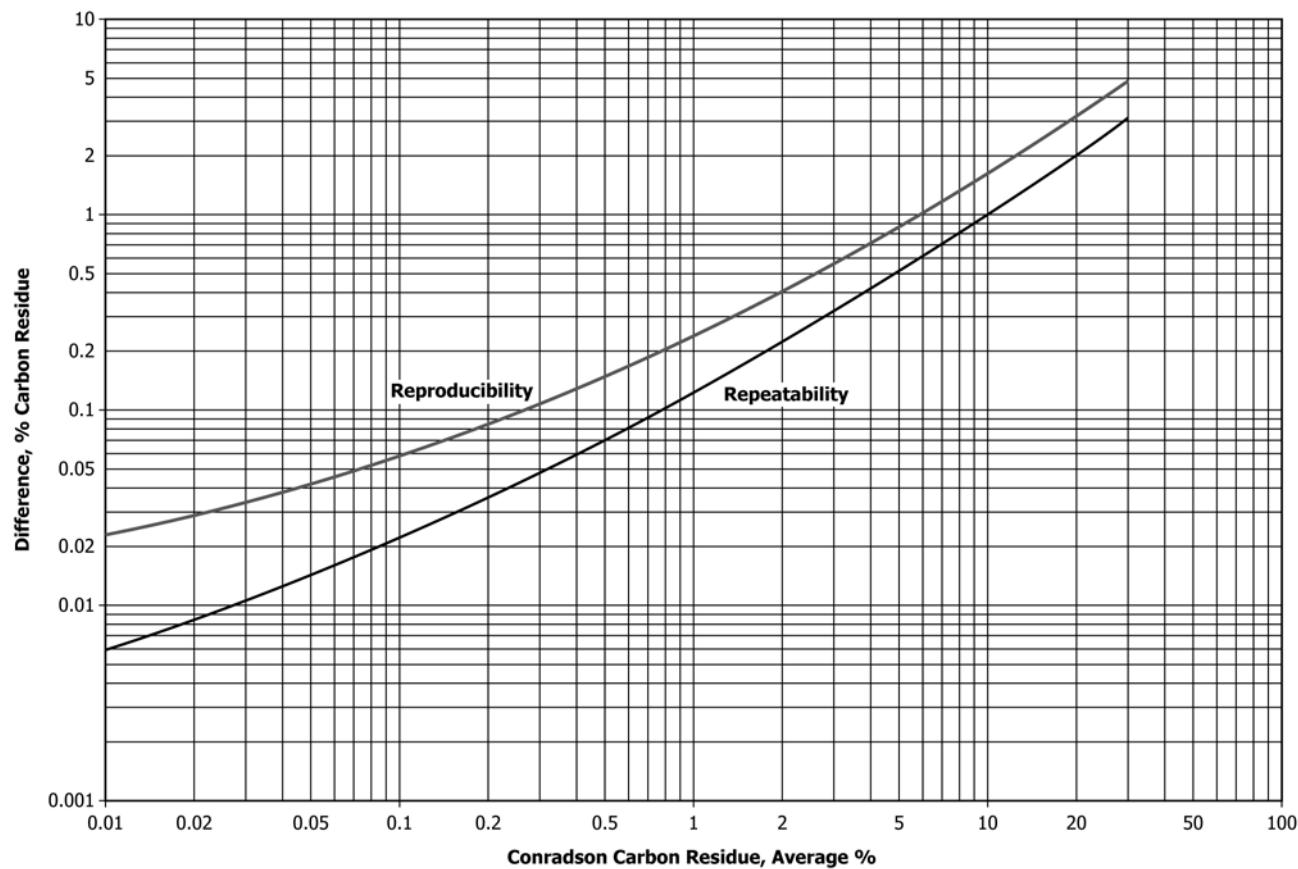
NOTE 6—Precision is based on data developed using inch-pound units. See Test Method D189 – 76.

13.2 *Bias*—This test method is based on empirical results and no statement of bias can be made.

14. Keywords

14.1 Conradson carbon residue; lubricants; petroleum products

⁴ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1227. Additional data used for the precision statement were obtained from the NRC, pending permission to reprint.



$\text{Log } r = -0.91666 + 0.82504 \text{ Log } x + 0.08239 (\text{Log } x)^2$
 $\text{Log } R = -0.62668 + 0.72403 \text{ Log } x + 0.10730 (\text{Log } x)^2$
 $x = \text{average of results being compared}$

FIG. 2 Precision

APPENDIX

(Nonmandatory Information)

X1. INFORMATION CONCERNING CORRELATION OF CARBON RESIDUE RESULTS DETERMINED BY TEST METHODS D189, D524, AND D4530

X1.1 No exact correlation of the results obtained by Test Methods D189 and D524 exists because of the empirical nature of the two tests. However, an approximate correlation (Fig. X1.1) has been derived by ASTM Committee D02 from the cooperative testing of 18 representative petroleum products and confirmed by further data on about 150 samples which were not tested cooperatively. Test results by both methods on unusual types of petroleum products need not fall near the correlation line of Fig. X1.1.

Caution should be exercised in the application of this approximate relation to samples of low carbon residues.

X1.2 A direct correlation of the results obtained by Test Methods D189 and D4530 has been derived by ASTM Committee D02 as shown in Fig. X1.2. Supporting data have been filed at ASTM Headquarters.⁵

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1192.

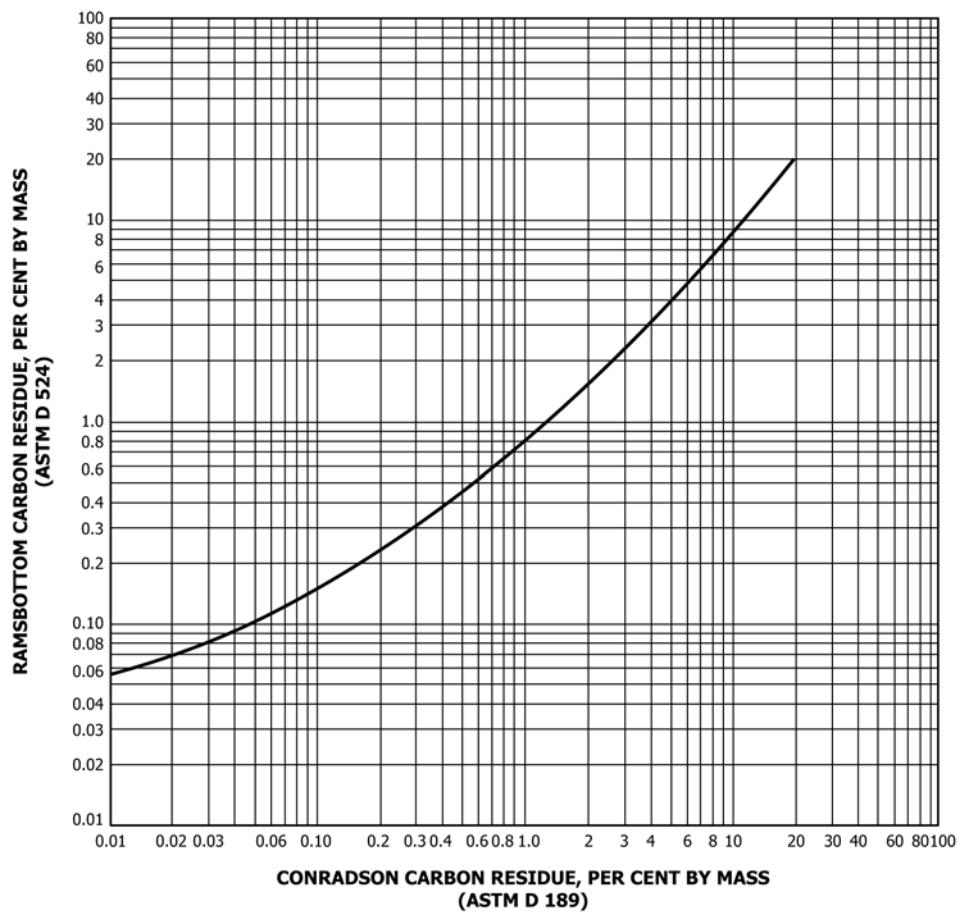


FIG. X1.1 Correlation Data

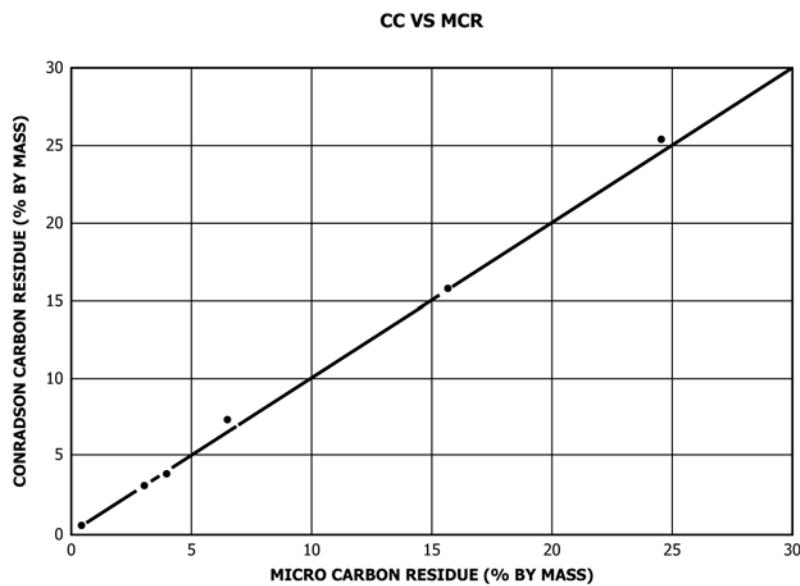


FIG. X1.2 Correlation of Conradson and Micro Carbon Residue Tests

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