International Standard 6615

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Petroleum products — Determination of carbon residue — Conradson method

Produits pétroliers — Détermination du résidu de carbone — Méthode Conradson

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

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Petroleum products — Determination of carbon residue — Conradson method

1 Scope and field of application

1.1 This International Standard specifies a method for determining the amount of carbon residue left after evaporation and pyrolysis of an oil, and is intended to provide some indication of relative coke-forming propensities. The method is generally applicable to relatively non-volatile petroleum products which partially decompose on distillation at atmospheric pressure. Petroleum products containing ash-forming constituents as determined by ISO 6245 will have an erroneously high carbon residue, depending upon the amount of ash formed.

NOTES

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

2 Values obtained by this method are not numerically the same as those obtained by ISO 4262, nor have satisfactory correlations been found between the results by the two methods for all materials which may be tested, because the carbon residue test is applied to a wide variety of petroleum products. The Conradson carbon residue is finding use to characterize heavy residue fuel coker feed stocks, etc., which cannot readily be loaded into a Ramsbottom coker bulb, and when it is desirable to examine or further test the residue.

1.2 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 amyl nitrate is absent (or if it is present, provided the test is performed on the base fuel without additive) the carbon residue of diesel fuel correlates approximately with combustion chamber deposits.

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

1.4 The following anomalous cases should be noted:

a) Motor oils. 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.

b) Diesel fuels. The carbon residue value of diesel oils containing amyl nitrate is erroneously high. If, however, the test is carried out on diesel fuels not containing amyl nitrate or on the base fuel to be blended with amyl nitrate, the carbon residue value correlates approximately with combustion chamber deposits.

c) Petroleum products containing ash-forming additives may give carbon residue values which may not correlate with the tendency to form deposits and may be higher than the corresponding tendency to form deposits.

2 References

ISO 3405, *Petroleum products — Determination of distillation characteristics.*

ISO 4262, *Petroleum products — Determination of carbon residue — Ramsbottom method.*

ISO 6245, *Petroleum products — Determination of ash.*

3 Principle

A weighed test portion 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 test portion and reported as Conradson carbon residue.

4 Apparatus

4.1 Porcelain crucible, wide form, glazed throughout, or a **silica crucible,** 29 to 31 ml capacity, 46 to 49 mm in rim diameter. Porcelain crucible size $^{1}/_{45}$ specified in ISO 1772.

4.2 Skidmore iron crucible, flanged and ringed, 65 to 82 ml capacity, 53 to 57 mm inside and 60 to 67 mm outside diameter of flange, 37 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 to 32 mm.

4.3 Spun sheet-iron crucible with cover, 78 to 82 mm in outside diameter at the top, 58 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.

4.4 Wire support-triangle of bare Nichrome wire of approximately 2,0 to 2,3 mm, having an opening small enough to support the bottom of the sheet-iron crucible at the same level as the bottom of the insulator block or hollow sheet-metal box (4.6).

4.5 Circular sheet-iron hood, 120 to 130 mm in diameter, the height of the lower perpendicular side being from 50 to 53 mm, provided at the top with a chimney 50 to 60 mm in height and 50 to 56 mm in the inside diameter, which is attached to the lower part having the perpendicular side by a cone-shaped member, bringing the total height of the complete hood to 125 to 130 mm. The hood may be made from a single piece of metal, provided that 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.

4.6 Insulator, ceramic heat-resistant block, refractive ring, or hollow sheet-metal box, 150 to 175 mm in diameter if round, or on a side if square, 32 to 38 mm in thickness, provided with a metal-lined, inverted cone-shaped opening through the centre, 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, provided that the ring is of hard, heat-resistant material.

4.7 Burner, Meker type or equivalent, having an orifice approximately 25 mm in diameter.

Dimensions in millimetres

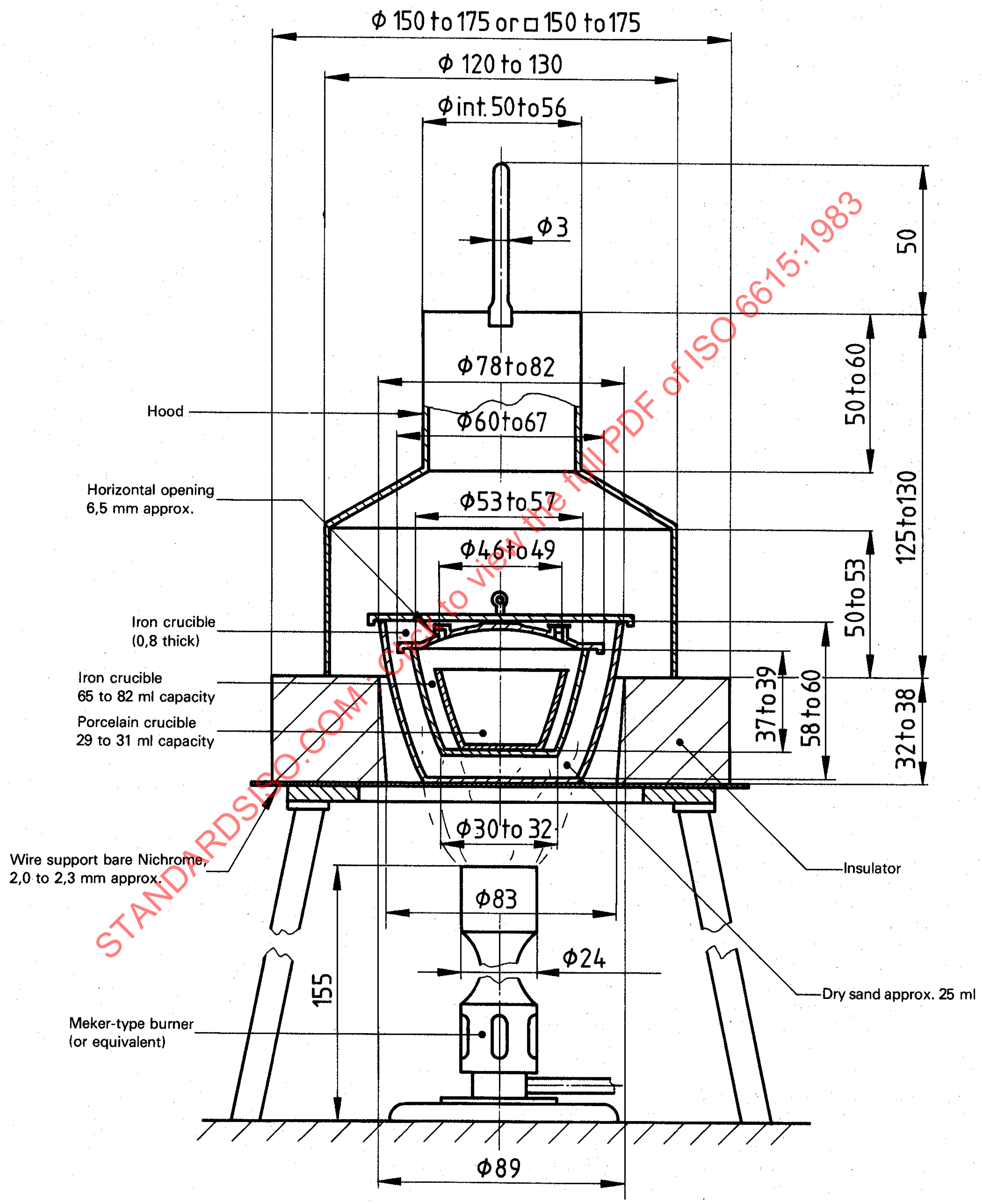

Figure 1 — Apparatus for determining Conradson carbon residue

5 Sample

Take a representative sample of the product to be tested according to ISO 3170 or ISO 3171 (or other relevant method).

6 Procedure

6.1 Weigh, to the nearest 5 mg, a 10 ± 0,5 g test portion of the oil, free of moisture and other suspended matter, into a tared porcelain or silica crucible (4.1) containing two glass beads about 2,5 mm in diameter. Place this crucible in the centre of the Skidmore crucible (4.2). Level the sand in the large sheet-iron crucible (4.3) and set the Skidmore crucible on it in the exact centre of the iron crucible. Apply covers to both the Skidmore and the iron crucible, the one on the latter fitting loosely to allow free exit to the vapours as formed.

NOTE — The mass of the test portion may be varied in relation to the expected carbon residue after coking according to the following table.

Expected carbon residue, %	Mass of test portion, g
less than 0,1	10 ± 0,005[1]
0,1 to less than 5	10 ± 0,005
5 to 15	5 ± 0,005
more than 15	3 ± 0,005

[1] The test portion should be sampled from the 10 % distillation residue.

6.2 On a suitable stand or ring, place the wire support triangle (4.4) and on it the insulator (4.6). Next centre 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 (4.5) in order to distribute the heat uniformly during the process (see figure 1).

6.3 Apply heat with a high, strong flame from the Meker-type gas burner (4.7) so that the pre-ignition period will be 10 ± 1,5 min (a shorter time may 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 vapours. Then remove the heat temporarily, and before replacing adjust the rate of heating so that the ignited vapours burn uniformly with the flame above the chimney, but not above the wire bridge. Heat may be increased, if necessary, if the flame does not show above the chimney. The period of burning the vapours shall be 13 ± 1 min. If it is found impossible to meet the requirements for both flame and burning time, the requirement for burning time is more important.

NOTE — If the test portion boils over, reduce the test portion size first to 5 g and then to 3 g as necessary to avoid the difficulty (see also 7.2).

6.4 When the vapours 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 ± 2 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 (about 20 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.

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

7 Modified procedure for residues exceeding 5 %

7.1 Field of application

This procedure is applicable to such materials as heavy crude oils, residuums, heavy fuel oils, and heavy gas oils. When the carbon residue as obtained by the procedure specified in clause 6 (using a 10 g test portion) is in excess of 5 %, difficulties may be experienced due to boiling over of the test portion. Trouble also may be encountered with test portions of heavy products which are difficult to dehydrate.

7.2 Procedure using 5 ± 0,5 g test portion

7.2.1 For the test samples showing more than 5,0 and less than 15,0 % carbon residue by the procedure specified in 6.1, repeat the test using a 5 ± 0,5 g test portion, weighed to the nearest 5 mg. In event that a result greater than 15,0 % is obtained, repeat the test, reducing the test portion to 3 ± 0,1 g, weighed to the nearest 5 mg.

7.2.2 When the 5 or 3 g test portion is used, it may not be possible to control the pre-ignition and vapour burning times within the limits specified in 6.3; in such cases, the results are nevertheless valid.

8 Modified procedure for determination of a carbon residue on a 10 % distillation residue

8.1 Field of application

This procedure is applicable to light distillates having a Conradson carbon residue less than 0,1 % before distillation.

8.2 Procedure using a 200 ml sample

8.2.1 Assemble the distillation apparatus described in ISO 3405, except that a 250 ml distillation flask and a 200 ml measuring cylinder shall be used.

A thermometer is not essential but the use of the high temperature range thermometer specified in ISO 3405 is recommended.

8.2.2 Place a volume of sample equivalent to 200 ml at 13 to 18 °C in the flask. Maintain the condenser bath at 0 to 4 °C; for some oils it may be necessary to maintain the temperature between 38 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.

8.2.3 Apply heat to the flask at a uniform rate so regulated that the first drop of condensate falls from the condenser 10 to 15 min after initial application of heat. After the first drop falls, move the measuring 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 to 10 ml per min. Continue the distillation until 178 ± 1 ml of distillate has been collected, then discontinue heating and allow the condenser to drain until 180 ml (90 % of the charge in the flask) has been collected in the cylinder.

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

8.2.5 While the distillation residue is warm enough to flow freely, pour approximately 10 ± 0,5 g of it into the previously weighed crucible to be used in the carbon residue test. After cooling, determine the mass of the test portion to the nearest 5 mg and carry out the carbon residue test in accordance with the procedure specified in clause 6.

9 Expression of results

9.1 Method of calculation

Calculate the Conradson carbon residue, CR, of the sample or of the 10 % distillation residue, as a percentage by mass as follows:

$$\mathrm{CR} = \frac{m_1}{m_0} \times 100$$

where

m_0 is the mass of the test portion, in grams;

m_1 is the mass of the carbon residue, in grams.

Express the result as "Conradson carbon residue, %" or as "Conradson carbon residue on 10 % distillation residue, %".

9.2 Precision

The precision of the method, as obtained by statistical examination of interlaboratory test results, is as follows:

9.2.1 Repeatability

The difference between successive 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 figure 2 only in one case in twenty.

9.2.2 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 figure 2 only in one case in twenty.