

INTERNATIONAL STANDARD

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Petroleum products — Determination of carbon residue — Conradson method

*Produits pétroliers — Détermination du résidu de carbone — Méthode
Conradson*



Reference number
ISO 6615:1993(E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 6615 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This second edition cancels and replaces the first edition (ISO 6615:1983), which has been technically revised.

Annex A forms an integral part of this International Standard. Annex B is for information only.

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Petroleum products — Determination of carbon residue — Conradson method

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for determining the amount of carbon residue, in the range of 0,01 % (m/m) to 30,0 % (m/m), left after evaporation and pyrolysis of an oil, and is intended to provide some indication of relative coke-forming tendency. 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 exhibit 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 retained in this method only in deference to its widespread use.

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 obtained 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. ISO 10370 (micro method) gives results similar to this method on a wide range of petroleum products, and may in time replace both this method and ISO 4262.

3 The carbon residue of distillate and residual fuel oils gives an approximate ranking of such fuels in terms of their propensity to form deposits in specific applications.

4 The presence of alkyl nitrates in distillate fuels, or ash-forming additives in either distillate or residue fuels, will give carbon residue results that are higher than the corresponding values on the fuel without additives. These values may not correlate with the propensity of a fuel to form deposits.

5 The carbon residue of base lubricating oils may give an indication of the propensity of the oil to lay down deposits in combustion chambers, and/or of the relative chemical constitution of oils of similar viscosity. Most finished lubricating oils contain ash-forming additives, and thus the carbon residue of finished lubricants cannot be used in this manner.

6 The carbon residue of a gas oil is a useful guide in the manufacture of gas.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1772:1975, *Laboratory crucibles in porcelain and silica*.

ISO 3170:1988, *Petroleum liquids — Manual sampling*.

ISO 3171:1988, *Petroleum liquids — Automatic pipe-line sampling*.

ISO 3405:1988, *Petroleum products — Determination of distillation characteristics*.

3 Principle

3.1 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 mass percentage of the original test portion.

3.2 For light products where over 90 % (V/V) distils at below 370 °C, the procedure may be carried out on the residue remaining after 90 % (V/V) has been distilled. The procedure for obtaining this residue is given in annex A.

4 Apparatus

The assembled apparatus is shown in figure 1. The constituent parts are described in 4.1 to 4.6.

4.1 Crucible, either a wide-form porcelain crucible that is glazed throughout, size 1/45 specified in ISO 1772, or a silica crucible, 29 ml to 31 ml capacity, 46 mm to 49 mm in rim diameter.

4.2 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 diameters 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 approximately 6,5 mm shall be kept clean. The outside diameter of the flat bottom shall be 30 mm to 32 mm.

4.3 Spun sheet-iron crucible with cover, 78 mm to 82 mm outside diameter at the top, 58 mm to 60 mm in height, and approximately 0,8 mm thick.

Place at the bottom of this crucible, and level before each test, a layer of approximately 25 ml of dry sand, or enough to bring the Skidmore crucible, when placed inside with cover on, nearly to the top of the sheet-iron crucible.

4.4 Wire support triangle, of bare Nichrome wire of diameter approximately 2,0 mm 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 mm to 130 mm diameter, the height of the lower perpendicular side being from 50 mm to 53 mm, provided at the top with a chimney 50 mm to 60 mm in height, and of 50 mm to 56 mm 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 mm to 130 mm.

As a guide for the height of the flame above the chimney, a bridge made of approximately 3 mm diameter iron or Nichrome wire, and having a height of 50 mm above the top of the chimney, shall be attached.

NOTE 7 The hood may be made from a single piece of metal, provided that it conforms to the dimensions given in 4.5.

4.6 Insulator, consisting of a ceramic heat-resistant block, refractive 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 thick, provided with a metal-lined, inverted cone-shaped opening through the centre, of diameter 83 mm at the bottom and 89 mm at the top. In the case of the refractory ring, no metal lining is necessary provided the ring is of hard, heat-resistant material.

4.7 Burner, Meker type or equivalent, having an orifice approximately 24 mm in diameter.

4.8 Cooling container, dessicator, or suitable vessel, not containing a desiccating agent.

5 Samples and sampling

5.1 Samples should be obtained in accordance with ISO 3170, ISO 3171 or an equivalent national standard.

5.2 Products examined by this International Standard are not always completely homogeneous, and therefore appropriate precautions shall be taken during the procurement of both bulk and laboratory test samples.

Dimensions in millimetres

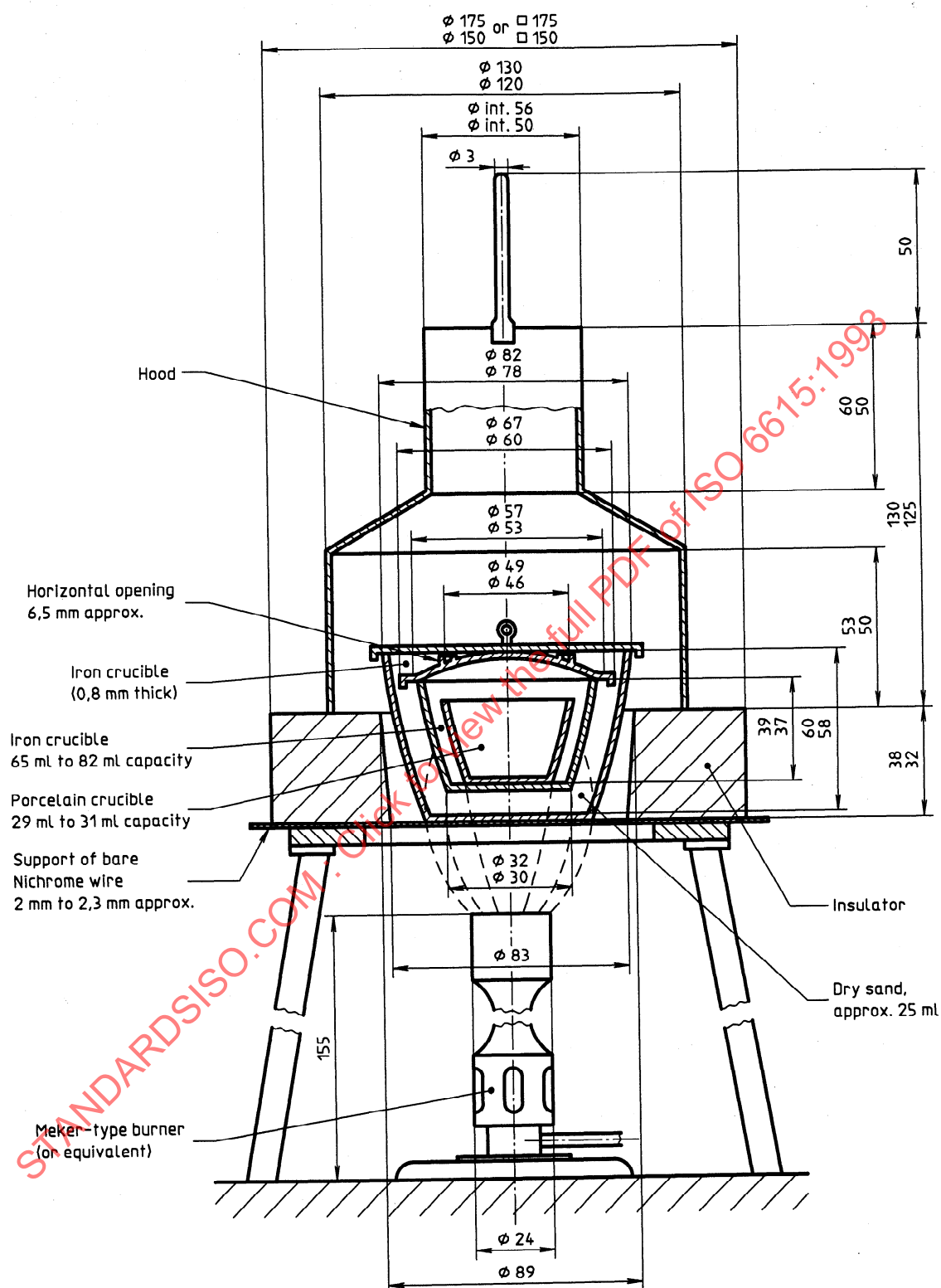


Figure 1 — Apparatus for determining Conradson carbon residue

6 Test procedure

6.1 Shake thoroughly the oil sample to be tested, first warming if necessary to reduce its viscosity. Place two glass beads approximately 2,5 mm diameter into the tared porcelain or silica crucible (4.1). Heat to 110 °C minimum, cool in the cooling container (4.8) and weigh to the nearest 0,1 mg until successive weighings differ by less than 0,5 mg. Weigh, to the nearest 5 mg, a test portion of oil of mass as shown in table 1 into this tared crucible. 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 crucibles, the one on the latter fitting loosely to allow free exit to the vapours as formed.

Table 1 — Test portion mass

Conradson carbon residue % (m/m)	Test portion mass g
≤ 0,10	10 ± 0,5 ¹⁾
0,11 to 5,00	10 ± 0,5
5,01 to 15,0	5 ± 0,5
15,1 to 30,0	3 ± 0,1
NOTE — When the 5 g 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.	
1) This test portion should be sampled from the 10 % (V/V) distillation residue where applicable (see annex A).	

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 burner (4.7) so that the pre-ignition period will be 10 min ± 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. Increase the heat if necessary, if the flame does not show above the chimney. The time period for burning the vapours shall be 13 min ± 1 min. If it is found impossible to meet the requirements for both flame height and specified burning time, the requirement for burning time shall take priority.

6.4 When the vapours cease to burn and no further blue smoke is observed, readjust the burner and hold the heat as at the start of test 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 min ± 2 min, which constitutes an additional limitation on the tolerances for the pre-ignition and burning periods. The time periods shall be observed with whatever burner and gas are used.

6.5 Remove the burner and allow the apparatus to cool until no smoke appears (approximately 15 min) and then remove the cover of the Skidmore crucible. Remove the porcelain or silica crucible with heated tongs, place in the cooling container (4.8), cool, examine (see note 8) and weigh to the nearest 0,1 mg.

NOTE 8 If the carbon residue is of unusual appearance or constitution, the test should be repeated. Examples of such a situation would include peeling (overheating), sticky (underheating), or residue outside the rim of the crucible (bubbling).

7 Calculation

Calculate the carbon residue of the sample, or of the 10 % (V/V) distillation residue (10 % b), from equation (1):

$$\text{Carbon residue, \% (m/m)} = \frac{m_3 - m_1}{m_2 - m_1} \times 100 \dots (1)$$

where

m_1 is the mass of empty crucible, in grams;

m_2 is the mass of crucible + test portion, in grams;

m_3 is the mass of crucible + residue, in grams.

8 Expression of results

8.1 Report results obtained from equation (1) as "Carbon residue — Conradson", or "Carbon residue — Conradson (10 % b)" with a reference to this International Standard.

8.2 Express the results to the nearest 0,01 % (m/m) up to 9,99 % (m/m), and to the nearest 0,1 % (m/m) from 10,0 % (m/m) to 30,0 % (m/m).

9 Precision

9.1 The precision is shown graphically in figure 2.

9.2 The repeatability (r), or 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 value below only in one case in 20:

$$\lg r = -0,892\ 05 + 0,847\ 23 \lg x + 0,086\ 88 (\lg x)^2$$

where x is the average of the results being compared, in % (m/m).

9.3 The reproducibility (R), or the difference between two single and independent results, obtained by different operators working in different laboratories on nominally identical test material, would in the long run, in the normal and correct operation of the test

method, exceed the value below only in one case in 20:

$$\lg R = -0,515\ 71 + 0,676\ 32 \lg x + 0,056\ 28 (\lg x)^2$$

where x is the average of the results being compared, in % (m/m).

10 Test report

The test report shall contain at least the following information:

- a) the type and complete identification of the product tested;
- b) a reference to this International Standard;
- c) the results of the test (see clause 8);
- d) any deviation, by agreement or otherwise, from the standard procedures specified;
- e) the date of the test.

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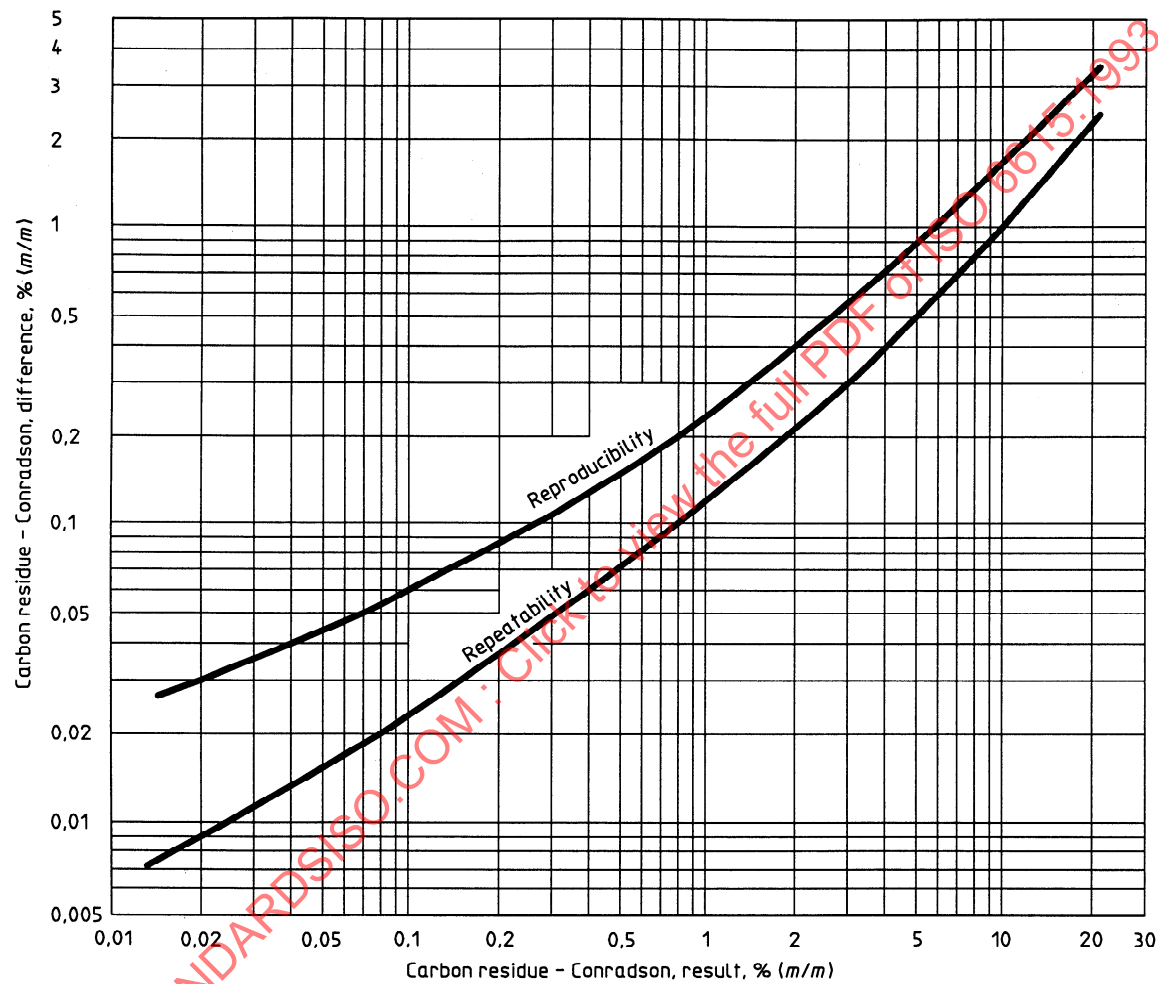


Figure 2 — Precision data

Annex A (normative)

Procedure for the preparation of a 10 % (V/V) distillation residue

A.1 Apparatus

A.1.1 Distillation flask, 250 ml, of borosilicate glass, conforming to the dimensions shown in figure A.1.

A.1.2 Cylinder, 200 ml, graduated, and conforming to the dimensions shown in figure A.2.

A.1.3 Distillation apparatus, conforming to that described in ISO 3405 with the exceptions of A.1.1 and A.1.2.

NOTES

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

10 If it is known that sufficient distillation residue to yield a minimum 5 mg of carbon residue will be obtained, the 125 ml distillation flask, with 100 ml of sample, as described in ISO 3405, may be used. The conditions of distillation should be those described in ISO 3405, discontinuing the heat when 89 ml of distillate has been collected.

A.2 Test procedure

A.2.1 Place a volume of sample equivalent to $200 \text{ ml} \pm 1 \text{ ml}$ at 13°C to 18°C in the flask. Either maintain the temperature of the cooling bath at 0°C to 4°C or, if the distillate is known to be waxy (and therefore may solidify in the condenser tube), raise the cooling bath temperature during the distillation to a temperature that maintains condensation but allows the distillate to flow. The maximum temperature of

the cooling bath shall be 60°C . 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. Insert a snug-fitting cork, with or without a thermometer (see A.1.3), into the neck of the flask.

A.2.2 Apply heat to the flask at a uniform rate so regulated that the first drop of condensate falls from the condenser 10 min 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 ml to 10 ml per min. Continue the distillation until $178 \text{ ml} \pm 1 \text{ ml}$ 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.

A.2.3 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 represents a 10 % distillation residue from the original product (assuming no volatile loss).

A.2.4 While the distillation residue is warm enough to flow freely, pour approximately $10 \text{ g} \pm 0,5 \text{ g}$ 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 of this International Standard.

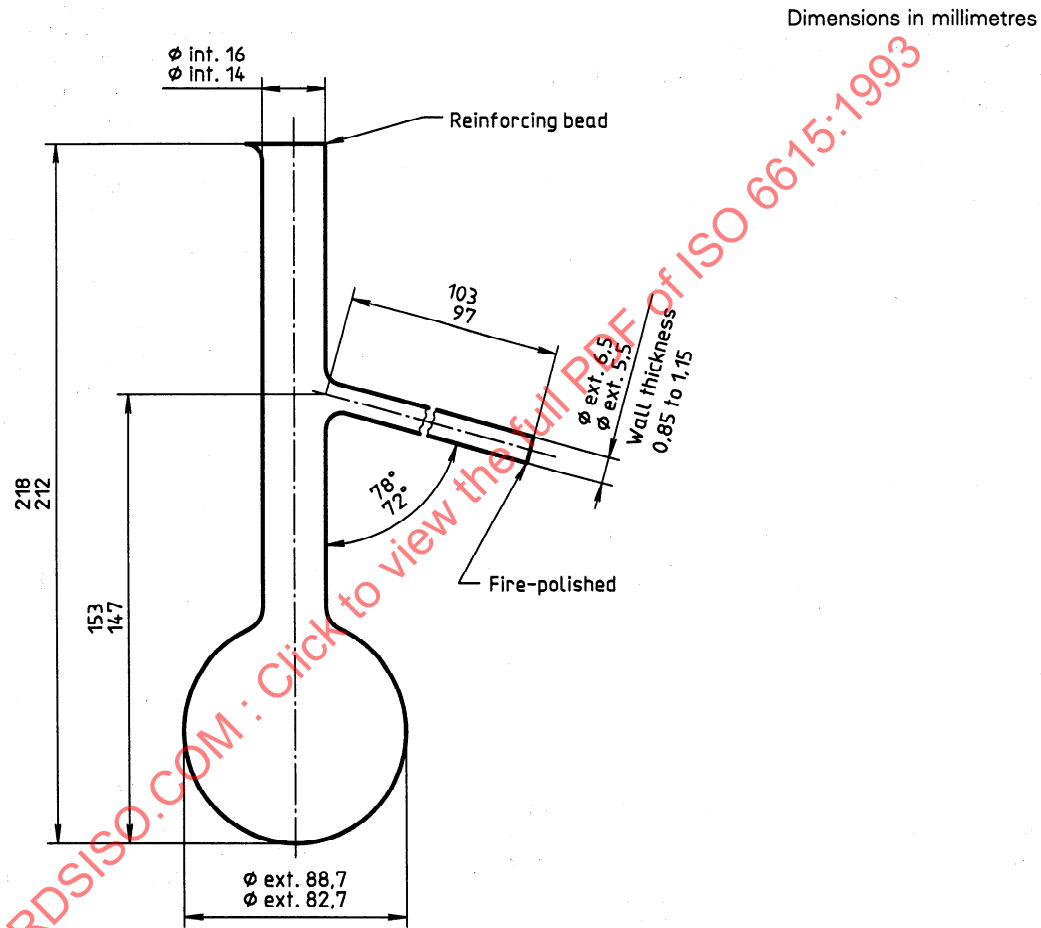
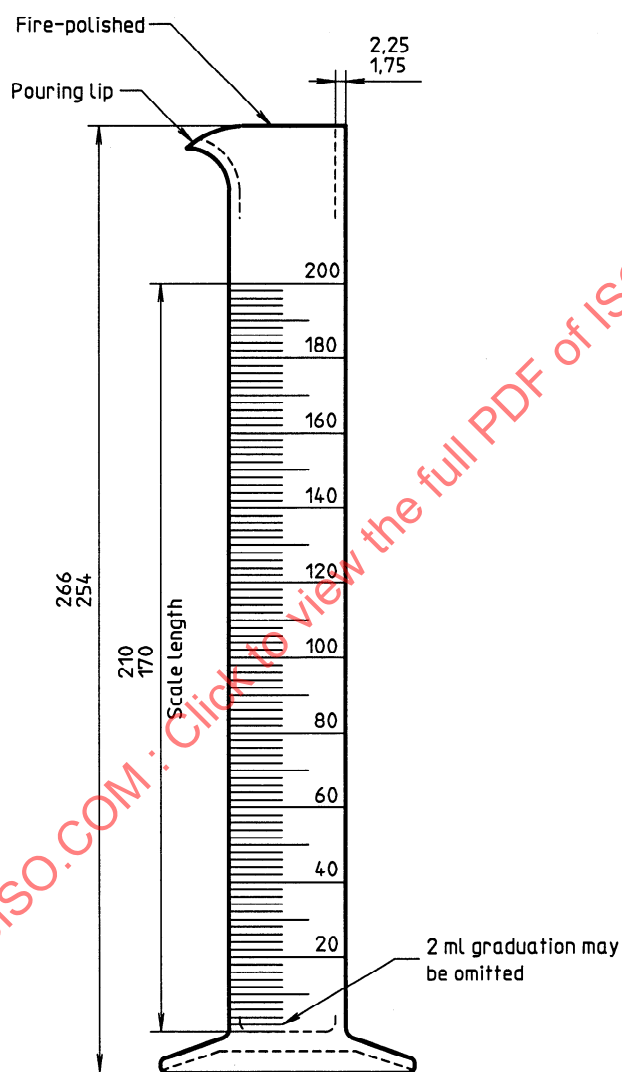


Figure A.1 — 250 ml distillation flask

Dimensions in millimetres



Shape of foot optional;
cylinder must stand on 15°
slope without tipping

Figure A.2 — 200 ml measuring cylinder

Annex B
(informative)

Bibliography

- [1] ISO 4262:1993, *Petroleum products — Determination of carbon residue — Ramsbottom method*.
- [2] ISO 6245:1993, *Petroleum products — Determination of ash*.
- [3] ISO 10370:1993, *Petroleum products — Determination of carbon residue — Micro method*.

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