
**Petroleum products — Determination
of carbon residue — Micro method**

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

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 10370:2014

<https://standards.iteh.ai/catalog/standards/sist/9ac3c542-ab2c-45bf-a358-6fac1f4fe2d2/iso-10370-2014>



iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 10370:2014

<https://standards.iteh.ai/catalog/standards/sist/9ac3c542-ab2c-45bf-a358-6fac1f4fe2d2/iso-10370-2014>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2014

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

	Page
Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
5 Reagents and materials	2
6 Apparatus	2
7 Sample preparation	4
7.1 General instructions	4
7.2 Regular procedure	5
7.3 Modified procedure	5
8 Sample transfer	5
9 Test procedure	6
10 Calculation	6
11 Expression of results	7
12 Precision	7
12.1 General	7
12.2 Repeatability, r	7
12.3 Reproducibility, R	7
13 Test report	8
Annex A (informative) Relationship of carbon residue (micro method) to carbon residue (Conradson method)	9
Bibliography	10

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword — Supplementary information](#).

The committee responsible for this document is ISO/TC 28, *Petroleum products and lubricants*.

This second edition cancels and replaces the first edition (ISO 10370:1993). Apart from updates regarding reference fuels and chemicals, the results of an interlaboratory study carried out by the Energy Institute in the UK on the 10 % volume distillation residue procedure for middle distillates using 4 ml vials and automatic distillation units, have been incorporated. It also incorporates ISO 10370:1993/Cor1:1996.

Petroleum products — Determination of carbon residue — Micro 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 the determination of the amount of carbon residue, in the range 0,10 % (m/m) to 30,0 % (m/m), left after evaporation and pyrolysis of petroleum products under specified conditions.

NOTE 1 The carbon residue value serves as an approximation of the tendency of petroleum products to form carbonaceous deposits under similar degradation conditions, and may be useful in the assessment of relative carbon-forming tendencies of products within the same class. In this case, care should be taken in the interpretation of results.

For products which yield a residue in excess of 0,10 % (m/m), the test results are equivalent to those obtained by the Conradson carbon residue test (see ISO 6615[1]) in the range of 0,10 (m/m) to 25,0 (m/m) (for details see [Annex A](#)).

This International Standard is also applicable to petroleum products which consist essentially of distillate material, and which may yield a carbon residue below 0,10 % (m/m). On such materials, a 10 % (V/V) distillation residue is prepared by the procedure described in [7.3.1](#) and [7.3.2](#) before analysis.

Both ash-forming constituents, as defined by ISO 6245[2] and non-volatile additives present in the sample add to the carbon residue value and are included in the total value reported.

NOTE 2 The presence of organic nitrates incorporated in certain distillate fuels will yield abnormally high values for the carbon residue. The presence of alkyl nitrate in the fuel may be detected by ISO 13759.[3]

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Terms and definitions

For the purposes of this document, the following term and definition apply

3.1

carbon residue

the whole residue produced of a sample from the specific conditions of evaporation and pyrolysis described in this International Standard

4 Principle

A weighed aliquot of the oil sample is placed in a glass vial and heated to 500 °C under an inert (nitrogen) gas stream in a controlled manner for a specific time. Volatiles formed during the reactions are swept away by the inert gas. The carbonaceous residue remaining is weighed.

5 Reagents and materials

5.1 Nitrogen, low oxygen content, i.e. 99,998 % (V/V) or better, with appropriate regulation to provide a delivery pressure of 0 kPa to 200 kPa.

NOTE The practical minimum delivery pressure is 140 kPa.

6 Apparatus

6.1 Glass sample vials, of 2 ml capacity, 12 mm outside diameter, approximately 35 mm high.

A vial of 4 ml capacity, 12 mm outside diameter, approximately 72 mm high may be used with samples of very low carbon residue content, i.e. below approximately 0,20 % (m/m), but no precision data has been obtained for them.

NOTE 1 An interlaboratory study^[4] has been carried out by the Energy Institute on the 10 % volume distillation residue procedure for middle distillates using 4 ml vials and automatic distillation units and the precision is given in [Clause 12](#).

NOTE 2 A glass sample vial of approximately 20 ml capacity (20,5 mm to 21 mm outside diameter by (70±1) mm high) for samples that are expected to yield residues < 0,10 % (m/m) may also be used, so that a more appreciable mass difference may be determined. In this case, a 5 g sample size is suggested. No precision has been determined for this technique, but it may be found suitable for very low 10 % residue samples.

6.2 Eyedropper or small rod, suitable for sample transfer.

6.3 Coking oven, comprising a circular heating chamber approximately 85 mm diameter by 100 mm deep for top-loading, capable of heating to 500 °C at a rate of between 10 °C and 40 °C per min, with exhaust port of 13 mm inside diameter for nitrogen purge of oven chamber (inlet near top, exhaust at bottom centre), with thermocouple sensor located in oven chamber next to but not touching sample vials, and with lid capable of sealing out air.

The condensate outlet leads into a short vertical section where most of the vapour condenses and falls into a removable trap located directly below the oven. A schematic diagram is given in [Figure 1](#).

6.4 Sample vial holder, comprising a cylindrical aluminium block approximately 76 mm diameter by 17 mm thick with 12 evenly spaced holes (for vials) each 13 mm diameter by 13 mm deep.

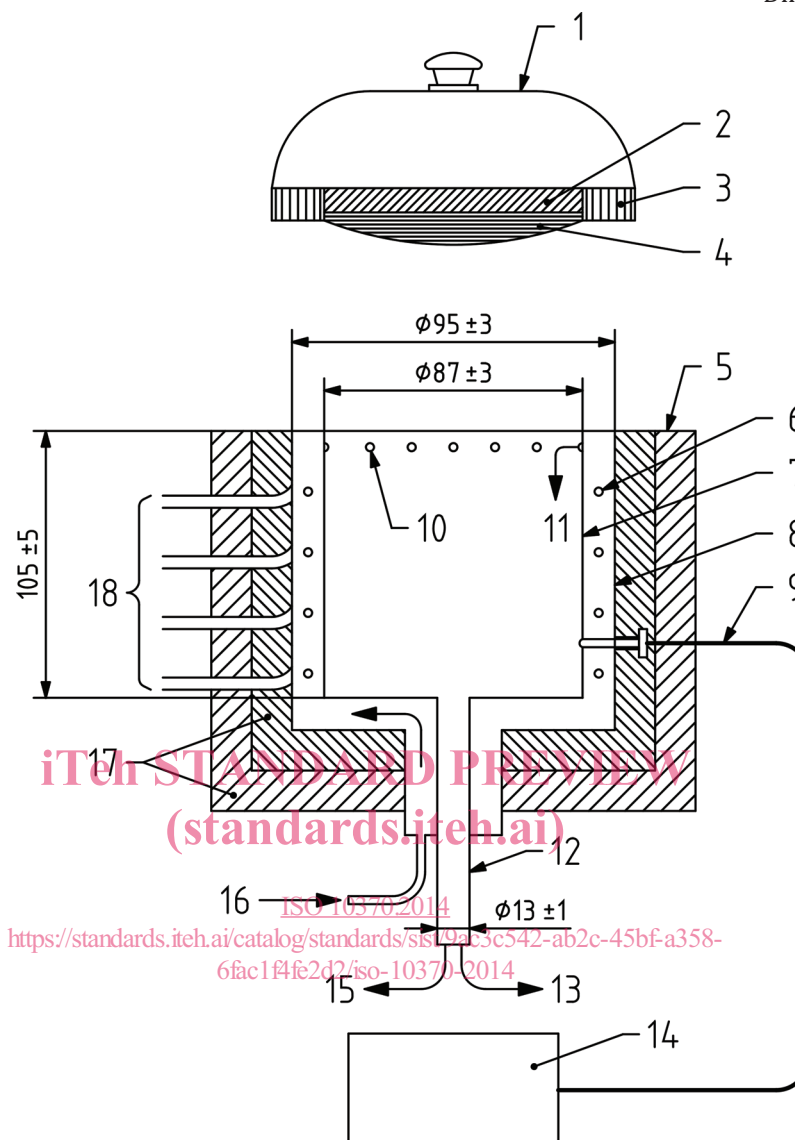
The holes shall be arranged in a circular pattern approximately 3 mm from the perimeter. The holder shall have legs 6 mm long with guides to centre in the oven chamber, and an index mark on the side to use as position reference. A typical holder is shown in [Figure 2](#).

6.5 Thermocouple, iron-constantan, with exterior read-out and a range including 450 °C to 550 °C.

6.6 Analytical balance, of 0,1 mg sensitivity.

6.7 Cooling vessel, desiccator or similar tightly closed vessel, without desiccant.

Dimensions in millimetres

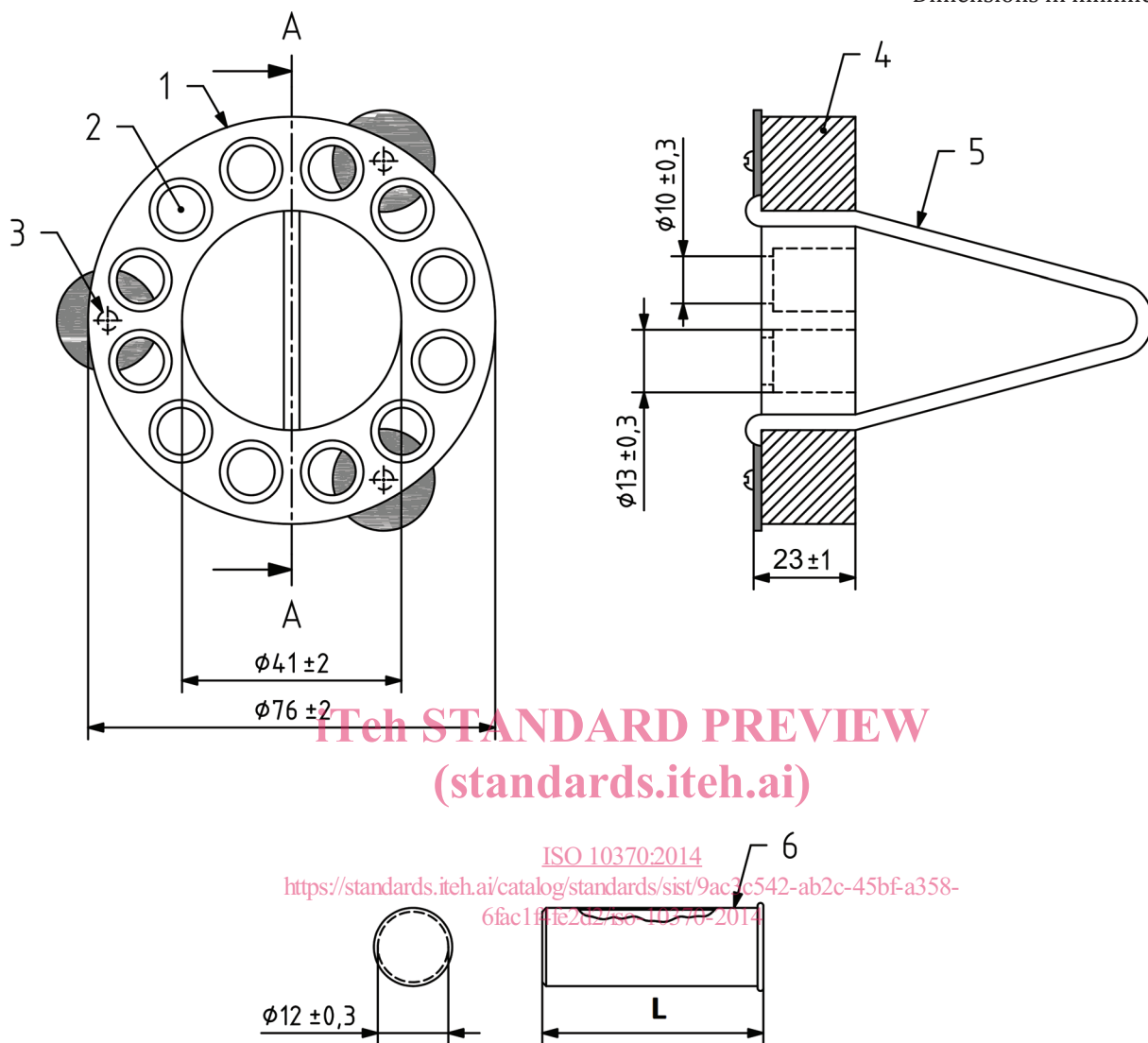


Key

- | | | | |
|---|--|----|---|
| 1 | lid | 10 | series of holes ($\varnothing 1$) |
| 2 | insulation | 11 | nitrogen in |
| 3 | ceramic ring | 12 | stainless steel exhaust tube |
| 4 | 304 stainless steel spherical top plug | 13 | condensate |
| 5 | oven | 14 | microprocessor control |
| 6 | cross-section heater coils | 15 | smoke |
| 7 | inner cylindrical shell, stainless steel | 16 | nitrogen supply inlet |
| 8 | outer cylindrical shell, stainless steel | 17 | insulation (two layers) |
| 9 | thermocouple leads | 18 | heater lead to 2 x 700 W tubular heater coils |

Figure 1 — Coking oven set-up

Dimensions in millimetres



Key

- 1 vial holder
- 2 12 identical holes uniformly spaced — vials fit loosely
- 3 small screws (three) for feet — steel basket centring washer (one on each foot)
- 4 ring — aluminium alloy material
- 5 handle — stainless steel alloy material
- 6 vial — soda lime glass
- L vial length — (37 ± 3) for 2 ml and (72 ± 3) for 4 ml

Figure 2 — Sample vial (soda lime glass, bottom) and vial holder (top)

7 Sample preparation

7.1 General instructions

For samples which consist essentially of distillate material, either follow the preparation as in 7.2 or prepare a distillation residue following a modified procedure of ISO 3405, given in 7.3. This latter procedure uses 4 ml vials (6.1) and shall be used for middle distillate samples which may yield a carbon residue below 0,30 % (*m/m*).

7.2 Regular procedure

Thoroughly stir the sample to be tested, first warming if necessary to reduce its viscosity. If the samples are in liquid form, transfer directly to the vials (6.1) using a rod or syringe. If the samples are solid materials, they shall either be heated, or frozen with liquid nitrogen and then shattered to provide manageable pieces.

7.3 Modified procedure

7.3.1 Ensure that the 4 ml vials (6.1) are clean and stored in a desiccator (without desiccant) before use. Set up the distillation as described in ISO 3405 using a clean 125 ml flask with no carbon deposits and add six glass or ceramic 'anti bumping' beads (1,5 mm to 2,5 mm diameter is recommended) to aid the mixing of residue as well as reducing bumping.

The thermometer may be omitted and replaced with a snug fitting, well rolled cork or silicone rubber stopper because it is the volume of distillate collected that is critical not the temperature of distillation. For an automatic unit, the temperature recording device may be necessary to allow the instrument to operate.

7.3.2 Discontinue heating when 88 ml of distillate has been recovered in the receiver. Allow the flask to cool for 5 min at the end of the distillation. Mix the residue in the flask by swirling. Immediately after mixing, transfer 2,5 ml to 3,0 ml to a pre-weighed vial of 4 ml (6.1) and reweigh. Take care to exclude any anti-bumping beads. This residue represents a 10 % (V/V) bottom portion of the original sample.

8 Sample transfer

8.1 During weighing and filling, handle the vials with forceps to minimize weighing errors. Discard the vials after use.

8.2 Weigh the clean sample vials, and record the mass to the nearest 0,1 mg.

8.3 Transfer an appropriate mass of the sample as indicated in Table 1 into the bottom of a weighed sample vial, taking care to avoid contact between the sample and the vial wall, reweigh to the nearest 0,1 mg and record. Place the loaded sample vials into the vial holder (up to 12), noting the position of each sample with respect to the index mark.

A control sample may be included in each batch of samples being tested. This control sample should be a typical sample which has been tested at least 20 times in the same equipment in order to define an average percent carbon residue and standard deviation.

Results for each batch are deemed acceptable when results for the control sample fall within the average percent carbon residue plus/minus three standard deviations. Control results which are outside these limits indicate problems with the procedure or the equipment.

Table 1 — Sample size

Sample description	Expected carbon residue % (m/m)	Sample size g
Black viscous or solid	> 5,0	0,15 ±0,05
Brown or black opaque and mobile	1,0 to 5,0	0,50 ±0,10
Transparent or translucent	0,2 to 1,0	1,50 ±0,50
Middle distillate, 10 % (V/V) residue	< 0,3	3,00 ±0,50 when used in conjunction with the 4 ml vial