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Brown coals and lignites — Determination of the yields of tar, water, gas and coke residue by low temperature distillation

Charbons bruns et lignites — Détermination des rendements en goudron, en eau, en gaz et en résidu de coke par distillation à basse température

Reference number
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Foreword

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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).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 27, *Solid minerals fuels*, Subcommittee SC 5, *Methods of analysis*.

This second edition cancels and replaces the first edition (ISO 647:1974), of which it constitutes a minor revision. The changes compared to previous edition are as follows: dated references and other minor items have been changed.

Introduction

The yield of distillation products by low temperature distillation, especially the yield of tar, forms the basis for the classification of brown coal and lignite for use in low temperature carbonization.

Brown coals and lignites — Determination of the yields of tar, water, gas and coke residue by low temperature distillation

1 Scope

This document specifies a method for the determination of the yields of tar, water, gas and coke residue obtained from brown coal and lignite by distillation to a final temperature of 520 °C.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5068-2, *Brown coals and lignites — Determination of moisture content — Part 2: Indirect gravimetric method for moisture in the analysis sample*

ISO 1170, *Coal and coke — Calculation of analyses to different bases*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Principle

The sample is heated in an aluminium retort to a temperature of 520 °C during a period of 80 min. The products of decomposition pass into a water-cooled receiver. The tar and water are condensed while gaseous products pass to atmosphere. The coke residue remaining in the retort is weighed. The receiver and its contents are also weighed and the mass of the water in it determined by entrainment with toluene or xylene. The mass of tar is obtained by difference.

The total water in the receiver includes the moisture in the coal as well as that from the decomposition of the coal. A separate determination of moisture in the coal is made so that the decomposition water can be calculated.

The percentage of gas (plus errors) is obtained by subtracting from 100 the sum of the percentages of coke residue, tar and total water. The results are reported on the "as analysed" basis and on the "dry" basis.

5 Reagents

5.1 Graphite paste.

Ground dry and made into suitable paste with water or thick lubricating oil.

5.2 Xylene.

Boiling point 135 °C to 140 °C.

5.3 Toluene.

Boiling point 110 °C.

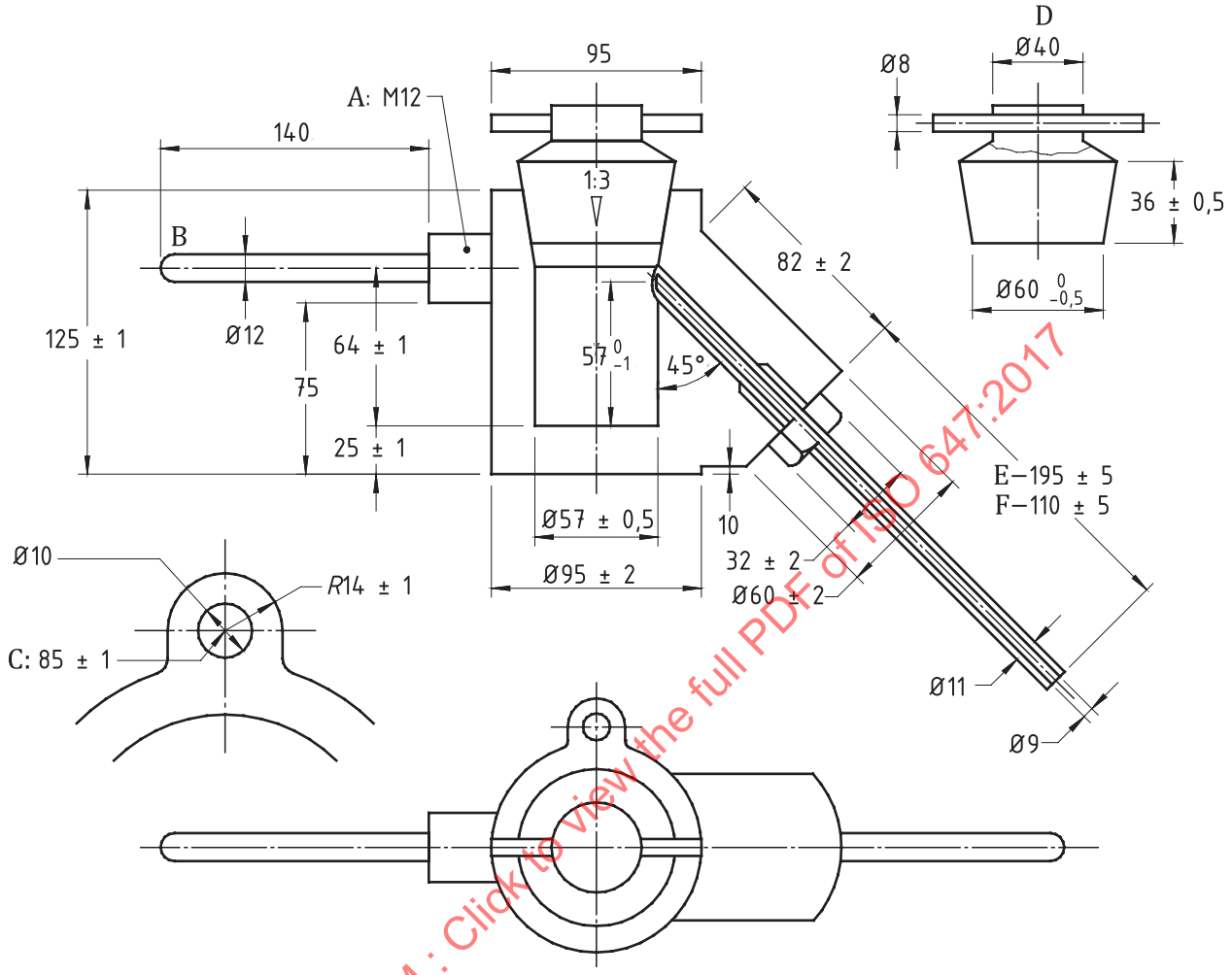
6 Apparatus

6.1 Retort.

Made of aluminium, with the dimensions shown in Figure 1. With the cover fitted, its capacity with the outlet tube shall be 170 ml ± 10 ml. The outlet tube shall be made of brass and its internal wall shall be clean and polished. A new assembly shall be heated at 520 °C for 20 min before use.

If, through wear, the upper edge of the conical portion of the cover is below the top surface of the retort, its free volume will be less than 160 ml and a new cover is required. The new oversize cover shall be ground so that when fitted, the upper edge of the round portion is less than 7 mm above the top surface of the retort. This will ensure that the free volume of the retort does not exceed 180 ml.

Dimensions in millimetres



Materials: Aluminium retort, aluminium content > 99 %

Volume of retort: 170 ml ± 10 ml

Outlet tube: Brass

Key

A screw thread
B bearer bar
C depth of hole for the thermometer
D cover
E as in Figure 2 a)
F as in Figure 2 b)

Figure 1 — Retort

6.2 Furnace.

Heated either electrically or by gas. For electrical heating, a resistance wire furnace or a silicon carbide rod furnace may be used.

6.3 Thermocouple and millivoltmeter or nitrogen-filled mercury thermometer.

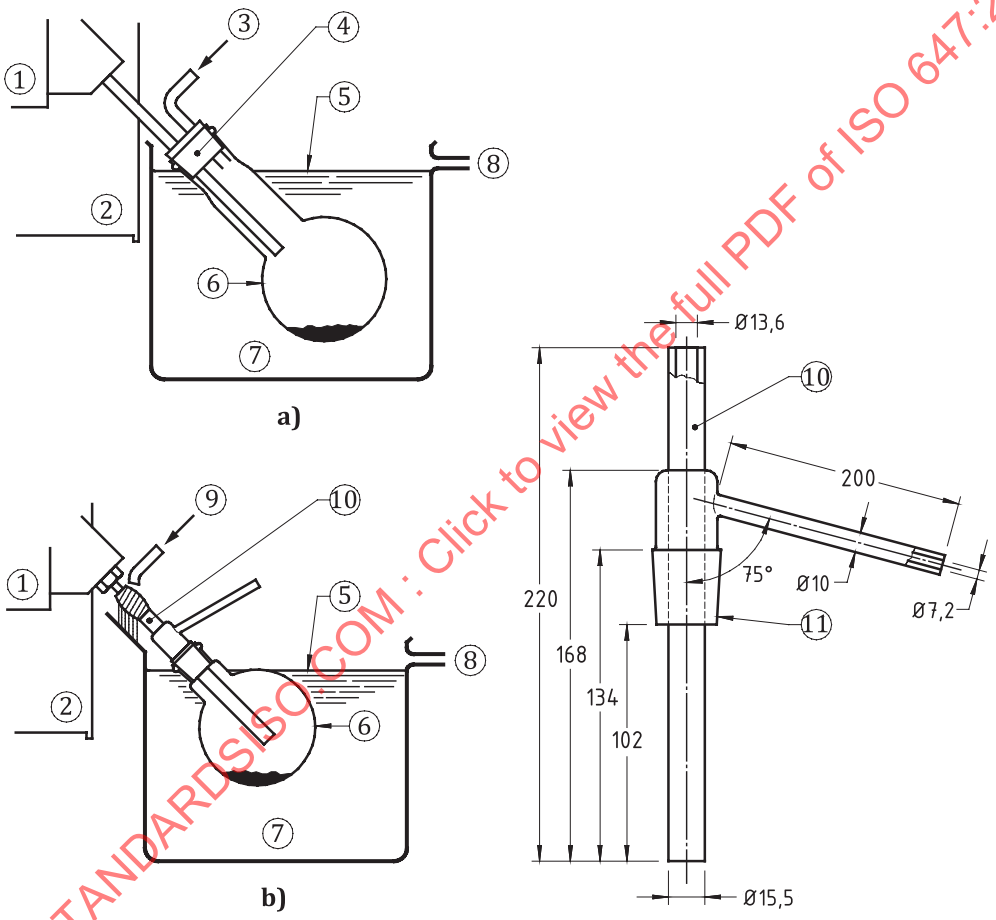
Calibrated and capable of indicating temperatures up to 550 °C.

A new thermometer shall be aged and then calibrated before use and shall be rechecked at intervals of one month by comparing it with a standard thermometer in a manner approved by a national testing authority.

6.4 Receiver.

Round-bottomed glass flask, capacity 750 ml, with conical ground joint and with either long or short neck depending on the method of connection to the retort (see Figure 2), provided with a rubber or glass stopper.

Dimensions in millimetres



Key

1 retort with gas outlet tube
2 extent of heating furnace
3 gas outlet tube
4 heat resistant stopper
5 level of cooling water
6 receiver
7 cooling bath
8 outlet
9 cold water
10 glass adapter tube
11 ground joint

Figure 2 — Arrangement of the receiver in the cooling bath

6.5 Cooling bath.

The distance between the receiver and the walls of the bath is not less than 20 mm. The water flow shall be adjusted to maintain a temperature of between 10 °C and 15 °C in the bath.

6.6 Distillation apparatus.

Composed of condenser, graduated tube for measurement of water and distillation flask. All parts are connected by means of ground glass joints.

7 Preparation of test sample

Spread the laboratory sample on a tray and allow it to attain approximate moisture equilibrium with the atmosphere. Carefully crush the sample so that at least 90 % passes through a sieve of 1 mm aperture while not more than 50 % passes through a sieve of 0,2 mm aperture. If the moisture content of the crushed sample is still greater than 20 %, further air-drying should be carried out to reduce the moisture content to between 10 % and 20 %. The test sample may be stored in a hermetically sealed container. Alternatively, the sample may be kept for a period not longer than 1 week in a stoppered container filled to more than 80 % of its capacity.

NOTE When samples are kept for longer than 1 week in containers which are not hermetically sealed or are not entirely filled, the loss of tar yield can be up to 0,5 % and in certain cases, the loss can be considerably greater.

8 Procedure

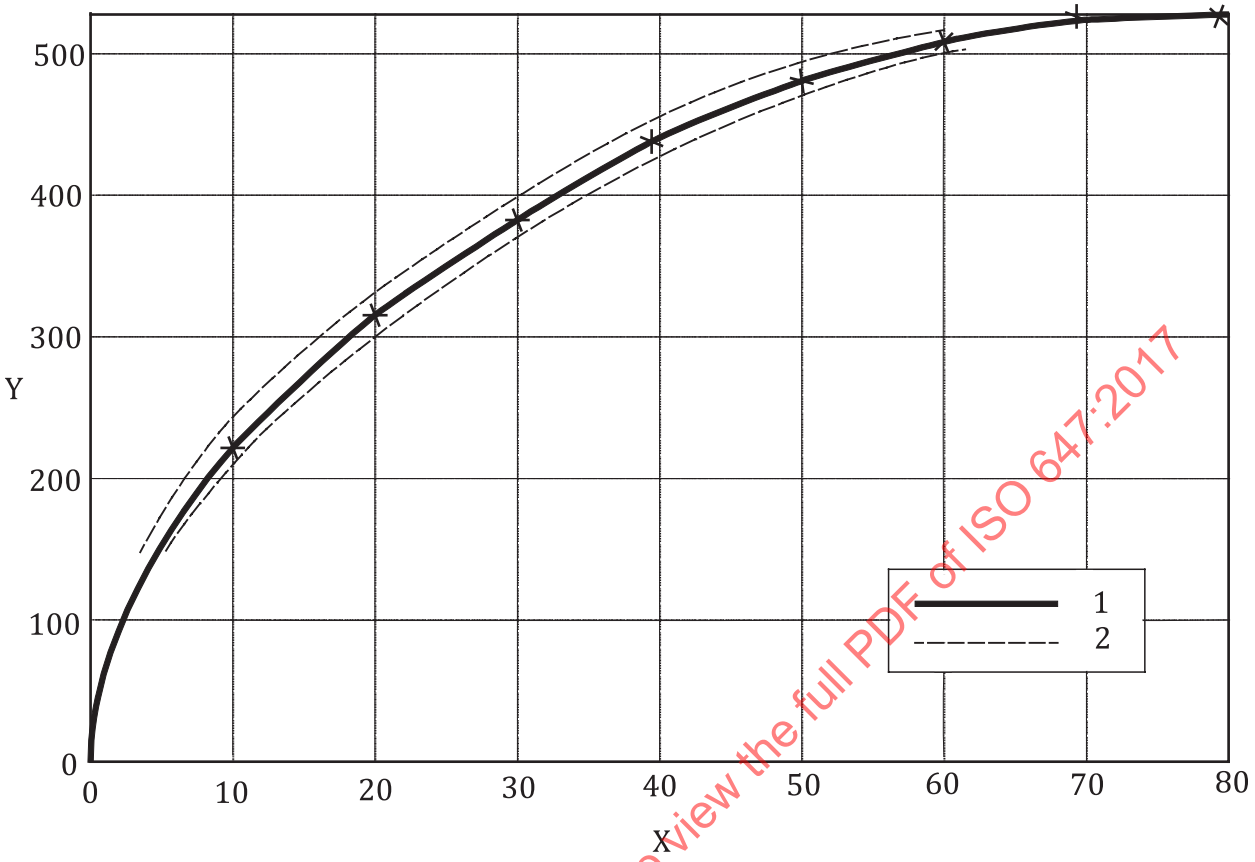
Weigh, to the nearest 0,05 g, about 50 g of the test sample and transfer it completely to the retort. Lightly smear the conical portion of the cover with the graphite paste, replace the cover and seal by rotating it. Determine the moisture content of the test sample at the same time by the method given in ISO 5068-2.

Weigh the receiver and stopper to the nearest 0,05 g and connect the receiver to the outlet tube of the retort by means of either a heat resistant stopper [see Figure 2 a)] or a glass adapter tube [see Figure 2 b)]. In the latter case, insert the brass outlet tube about 8 mm into the glass adapter tube and seal it to it by means of a short length of rubber tubing. Wind the joint with cotton, asbestos, linen, filter paper or similar material and cool by a stream of water while the retort is being heated. Place the retort in the furnace and the receiver in the cooling bath and ensure that the apparatus is gastight. It is necessary to pre-heat certain types of furnaces in order to reach 220 °C within 10 min of inserting the retort, and the receiver shall be immersed in the cooling bath as far as possible, but the rubber stopper or the ground joint shall not touch the water. Start the flow of water through the cooling bath and heat the retort according to the specification of Table 1.

Table 1 — Specification of heating the retort

Time from start min	Temperature °C
10	220
20	310
30	380
40	440
50	480
60	505
70	520
80	520

Maintain the rate of heating within the limits shown in Figure 3.



Key

X time, minutes
Y temperature, °C
1 temperature graph against time
2 limits

NOTE 1 Total time for low temperature distillation between 20 °C and 520 °C: 80 min.

NOTE 2 Effective time for low temperature distillation between 310 °C and 520 °C: 60 min.

Figure 3 — Schedule of heating

At the end of the above period, stop the heating and remove the retort from the furnace with the receiver still connected. Allow to stand for 10 min to enable the tar collected in the outlet tube to trickle down into the receiver. Disconnect the receiver from the retort and, if necessary, transfer the remaining tar from the outlet tube into the receiver with a small spatula. Only a very small residue of tar will be found in a clean smooth brass tube. Close the receiver and the outlet tube of the retort with stoppers and cool the retort to room temperature. Remove the coke residue carefully and weigh it to the nearest 0,05 g in a previously counterpoised weighing bottle.

Wipe off adhering water from the outside of the receiver and re-weigh to obtain the mass of the tar plus total water. Add 200 ml of toluene or xylene to the receiver and determine the total water content by entrainment using the distillation apparatus (6.6).