

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 647

DETERMINATION OF THE YIELDS OF TAR, WATER, GAS
AND COKE RESIDUE BY LOW TEMPERATURE DISTILLATION
OF BROWN COAL AND LIGNITE

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 647, *Determination of the yields of tar, water, gas and coke residue by low temperature distillation of brown coal and lignite*, was drawn up by Technical Committee ISO/TC 27, *Solid mineral fuels*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question by the Technical Committee began in 1955 and led, in 1964, to the adoption of a Draft ISO Recommendation.

In November 1965, this Draft ISO Recommendation (No. 865) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

Argentina	Germany	Romania
Australia	India	Spain
Austria	Ireland	Sweden
Belgium	Italy	Switzerland
Brazil	Japan	Turkey
Canada	Korea, Rep of	U.A.R.
Chile	Netherlands	United Kingdom
Czechoslovakia	New Zealand	U.S.S.R.
Denmark	Poland	Yugoslavia
France	Portugal	

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in February 1968, to accept it as an ISO RECOMMENDATION.

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DETERMINATION OF THE YIELDS OF TAR, WATER, GAS AND COKE RESIDUE BY LOW TEMPERATURE DISTILLATION OF BROWN COAL AND LIGNITE

INTRODUCTION

The yield of the 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.

1. SCOPE

This ISO Recommendation describes 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. PRINCIPLE

The sample is heated in an aluminium retort to a temperature of 520 °C during a period of 80 minutes. 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, also by entrainment with toluene or xylene, 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, of tar and of decomposition water. The results are reported on the "as analysed" and on the "dry" basis.

3. REAGENTS

- 3.1 **Graphite paste.** Ground dry and made into a suitable paste with water or thick lubricating oil.
- 3.2 **Xylene**, boiling point 135 to 140 °C *or*
Toluene, boiling point 110 °C.

4. APPARATUS

- 4.1 **Retort**, of aluminium, with the dimensions shown in Figure 1; with the cover fitted, its capacity with the outlet tube is 170 ± 10 cm³; the outlet tube is made of brass and its internal wall is clean and polished. A new assembly should be heated at 520 °C for 20 minutes before use.
- 4.2 **Furnace**, heated either electrically or by gas. For electrical heating, a resistance wire furnace or a silicon carbide rod furnace may be used.
- 4.3 **Thermocouple** and millivoltmeter, or a nitrogen-filled mercury thermometer, calibrated and capable of indicating the temperatures up to 550 °C.

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

- 4.4 Receiver.** Round bottomed glass flask of 750 ml capacity, with conical ground joint and with either long or short neck depending on the method of connection to the retort (see Fig. 2), provided with a rubber or glass stopper.
- 4.5 Cooling bath.** Water bath such that the distance between the receiver and the walls of the bath is not less than 2 cm. The water flow is adjusted to maintain a temperature of between 10 and 15 °C in the bath.
- 4.6 Distillation apparatus.** Suitable distillation apparatus for the determination of moisture in brown coal or lignite, as specified in ISO Recommendation R ..., * *Determination of moisture in brown coal and lignite.*

5. 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 whilst 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 an hermetically sealed container. Alternatively, the sample may be kept for a period not longer than one week in a stoppered container filled to more than 80 % of its capacity.

NOTE. — When samples are kept for longer than one week in containers which are not hermetically sealed or are not entirely filled, the loss of tar yield can be up to 0.5 % In certain cases the loss may be considerably greater.

6. 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 Recommendation R *

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 Fig. 2(a)) or a glass adapter tube (see Fig. 2 (b)). In the latter case the brass outlet tube should be inserted about 8 mm into the glass adapter tube and sealed to it by means of a short length of rubber tubing. The joint should be wound with cotton, asbestos, linen, filter paper or similar material and cooled by a stream of water whilst the retort is being heated. Place the retort in the furnace (see Note 1 below) and the receiver in the cooling bath (see Note 2 below) and ensure that the apparatus is gastight. Start the flow of water through the cooling bath and heat the retort according to the following schedule:

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

* At present at the stage of draft proposal.

The rate of heating should be maintained within the limits shown in Figure 3.

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 minutes 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 (see Note 3 below). 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 tared 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 the method given in ISO Recommendation R*

NOTES

1. It is necessary to pre-heat certain types of furnaces in order to reach 220 °C within 10 minutes of inserting the retort.
2. The receiver should be immersed in the cooling bath as far as possible, but the rubber stopper or the ground joint should not touch the water.
3. Only a very small residue of tar will be found in a clean smooth brass tube.

7. CALCULATION AND REPORTING OF RESULTS

If

m = mass of sample, expressed in grammes,

m_1 = mass of empty receiver and stopper, expressed in grammes.

m_2 = mass of receiver and stopper, plus tar plus total water, expressed in grammes,

m_3 = mass of total water determined by entrainment, expressed in grammes,

m_4 = mass of coke residue, expressed in grammes,

M = moisture content of the sample, expressed as a percentage,

then the yields on the "as analysed" basis are:

$$\text{Coke residue, \%} = \frac{m_4 \times 100}{m}$$

$$\text{Tar, \%} = \frac{(m_2 - m_1 - m_3) \times 100}{m}$$

$$\text{Water (decomposition), \%} = \frac{m_3 \times 100}{m} - M$$

$$\begin{aligned} \text{Gas (plus errors), \%} &= 100 - (\text{coke} + \text{tar} + \text{total water}) \\ &= \frac{(m + m_1 - m_2 - m_4) \times 100}{m} \end{aligned}$$

The yields on the "dry" basis are obtained by multiplying the above results by $\frac{100}{100 - M}$

The result, preferably the mean of duplicate determinations (see section 8 below), should be reported to the nearest 0.1 %. Values for tar, coke residue, decomposition water and gas should be reported both on the "as analysed" basis and on the "dry" basis. The tar content may also be calculated on the "dry, ash free" basis.

* At present at the stage of draft proposal.

8. PRECISION OF DETERMINATION

Test parameter (dry basis)	Maximum acceptable differences between results obtained	
	in the same laboratory	in different laboratories
Tar	0.5% absolute	0.7% absolute
Water	0.4% absolute	0.8% absolute
Coke residue	0.7% absolute	1.0% absolute

8.1 In the same laboratory

The results of duplicate determinations, carried out at different times in the same laboratory, by the same operator, with the same apparatus, on representative portions taken from the same test sample, should not differ by more than the above values.

8.2 In different laboratories

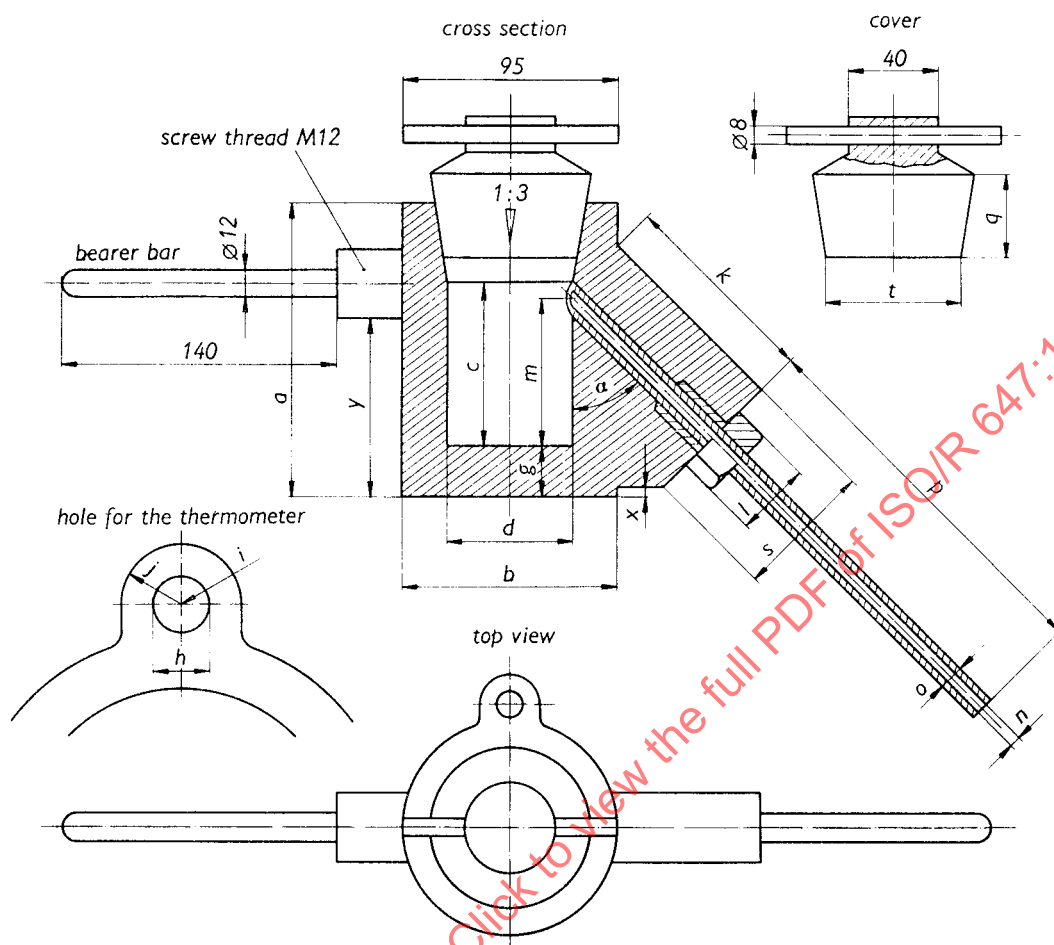
The means of the results of duplicate determinations, carried out in each of two different laboratories on test portions taken from the same laboratory sample, should not differ by more than the above values.

Key to Figure 1, page 9

Retort	millimetres
<i>a</i> = height without cover	125 ± 1
<i>b</i> = outside diameter	95 ± 2
<i>c</i> = height of cylindrical section	64 ± 1
<i>d</i> = inside diameter of cylindrical section	57 ± 0.5
<i>g</i> = base thickness	25 ± 1
<i>h</i> = diameter of the hole for thermometer	10
<i>i</i> = depth of the hole for thermometer	85 ± 1
<i>j</i> = radius of the boss for thermometer	14 ± 1
<i>k</i> = length of "nose" section of the retort	82 ± 2
<i>s</i> = diameter of "nose" section of the retort	60 ± 2
<i>α</i> = angle between outlet tube and retort	45°
<i>x</i> = height of lower point of "nose" section above the base of the retort	10
<i>y</i> = height of the handle above the base of the retort	75
Outlet tube	
<i>l</i> = outside diameter of stop nut	32 ± 2
<i>m</i> = height above base of cylindrical section	57 + 0 - 1
<i>n</i> = inside diameter	9
<i>o</i> = outside diameter	11
<i>p</i> = length of exposed section:	
(i) as in Figure 2 (a)	195 ± 5
(ii) as in Figure 2 (b)	110 ± 5
Cover	
<i>q</i> = height of the conical part	36 ± 0.5
<i>t</i> = lower diameter	60 + 0 - 0.5

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 cm³ and a new cover is required. The new oversize cover is 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 cm³.

Dimensions in millimetres



MATERIALS: Aluminium retort; aluminium content $> 99\%$
 volume of retort: $170 \pm 10 \text{ cm}^3$
 Outlet tube: brass.

FIG. 1 — Retort