INTERNATIONAL STANDARD 1017

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Brown coals and lignites — Determination of acetone-soluble material ("resinous substances") in the benzene extract

Charbons bruns et lignites — Détermination des matières de l'extrait au benzène solubles dans l'acétone («substances résineuses»)

First edition — 1975-11-15

UDC 662.642 : 543.832

Ref. No. ISO 1017-1975 (E)

Descriptors : coal, lignite, chemical analysis, determination of content, dissolved matter, acetone.

Price based on 2 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 27 has reviewed ISO Recommendation R 1017 and found it technically suitable for transformation. International Standard ISO 1017 therefore replaces ISO Recommendation R 1017-1969 to which it is technically identical.

ISO Recommendation R 1017 was approved by the Member Bodies of the following countries :

Australia	Iran	Spain
Austria	Italy	Switzerland
Canada	Korea, Rep. of	Turkey
Czechoslovakia	Netherlands	United Kingdom
Denmark	New Zealand	U.S.S.R.
Egypt, Arab Rep. of	Portugal	Yugoslavia
France	Romania	
India	South Africa, Rep. of	

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1017 into an International Standard.

Printed in Switzerland

Brown coals and lignites — Determination of acetone-soluble material ("resinous substances") in the benzene extract

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method of determining the amount of acetone-soluble material ("resinous substances") in the benzene-soluble extract from brown coals and lignites.

NOTE — The acetone extract will also contain a percentage of wax dissolved simultaneously with the "resinous substances".

2 REFERENCE

ISO 975, *Brown coals and lignites — Determination of yield of benzene-soluble extract.*

3 PRINCIPLE

The sample of benzene extract from brown coal or lignite obtained by the procedure described in ISO 975 is extracted with acetone at a temperature of 18 to 22 °C. The soluble fraction is filtered or centrifuged off and, after evaporation of the solvent, dried to constant mass. The percentage of acetone-soluble material is calculated from the mass of residue after drying.

NOTE — The high selectivity of acetone requires a strict temperature control during the determination. The temperature of the solvent, the room temperature at the beginning of the determination and the room temperature at the end of the determination should not differ from each other by more than 0,5 °C and should be within the range 18 to 22 °C.

4 REAGENT

Acetone, of analytical reagent quality.

5 APPARATUS

5.1 Centrifuge, capable of operating at 1 600 rev/min.

5.2 Glass vessels, either cylindrical or conical, of 15 ml capacity and fitted with rubber stoppers, for use in the centrifuge.

5.3 Evaporating dish, of glass or silica, about 20 mm high by 50 mm diameter.

5.4 Vacuum drying oven, electrically heated.

5.5 Air oven, electrically heated, capable of maintaining a temperature of 100 to 110 °C.

5.6 Infra-red drying lamp.

6 PREPARATION OF SAMPLE

The residue obtained from the benzene extract shall be crushed to pass a sieve of 0,1 mm aperture.

If the residue is a viscous liquid, it shall be cooled in solid carbon dioxide to − 80 °C and then crushed.

7 PROCEDURE

Weigh, to the nearest 0,001 g, about 0,5 g of the sample into the glass vessel. Add 7 ml of the acetone and shake for exactly 2 min (see note 1). Allow the acetone-soluble fraction to clear and decant it into the weighed, dry evaporating dish. If the fraction does not clear, it may be centrifuged for 1 min and then decanted, or filtered if necessary (see note 2), into the evaporating dish (see note 3).

Add a further 7 ml of the acetone to the glass vessel and repeat the above extraction (see note 4). If a filter has been used, rinse it with a few millilitres of the acetone and add the rinsings to the evaporating dish.

Place the evaporating dish in the vacuum drying oven and evaporate off the acetone at 80 ± 20 °C and about 50 kPa absolute. Alternatively, the evaporation may be carried out using the infra-red drying lamp. Transfer the dish to the air oven and dry to constant mass.

NOTES

1 Warming of the solvent may be minimized by holding the glass vessel at the upper end between the index and middle fingers, while the thumb secures the rubber stopper. Rubber finger-shields should be worn.

2 Since the acetone solution will creep up the filter paper, the smallest convenient size of paper should be used.

3 Any particles of benzene extract adhering to the upper end of the glass vessel after shaking should be washed back by cautious tilting and the fraction again left to settle, or centrifuged.

4 If the second acetone extract is strongly coloured, a third extraction should be carried out.

8 EXPRESSION OF RESULTS

The acetone-soluble material, Ac_{20}, in the sample as analysed, expressed as a percentage, is given by the formula

$$Ac_{20} = \frac{Km_2}{m_1}$$

where

m_1 is the mass, in grams, of benzene extract taken;

m_2 is the mass, in grams, of acetone-soluble material recovered;

$$K = 100 + 2{,}5\,(20 - t)$$

$$\text{where } t = \frac{t_1 + t_2 + t_3}{3}$$

t_1 being the temperature, in degrees Celsius, of the acetone used for the extraction;

t_2 being the ambient temperature, in degrees Celsius, at the beginning of the determination;

t_3 being the ambient temperature, in degrees Celsius, at the end of the determination.

The result, preferably the mean of duplicate determinations (see clause 9), shall be reported to the nearest 0,1 %.

9 PRECISION OF THE METHOD

Amount of acetone-soluble material	Maximum acceptable difference between results	
	Repeatability	Reproducibility
Less than 20 %	0,3 % absolute	0,5 % absolute
20 to 30 %	0,4 % absolute	0,7 % absolute
30 to 50 %	0,5 % absolute	0,9 % absolute
over 50 %	1,0 % of result	1,8 % of result

9.1 Repeatability

The results of duplicate determinations, carried out at different times in the same laboratory by the same operator with the same apparatus on the same benzene-soluble fraction, shall not differ by more than the above value.

9.2 Reproducibility

The means of the results of duplicate determinations, carried out in two different laboratories on representative test portions taken from the same benzene-soluble fraction, shall not differ by more than the above value.

10 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.