INTERNATIONAL STANDARD

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

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

Reference number
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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

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

ISO 1017 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This third edition cancels and replaces the second edition (ISO 1017:1985), which has been technically revised.

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

1 Scope

This International Standard specifies a method of determining the mass fraction of acetone-soluble material ("resinous substance") 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 substance".

2 Normative references

The following referenced documents are indispensable for the application 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 975, *Brown coals and lignites — Determination of yield of benzene-soluble extract — Semi-automatic method*

3 Principle

The sample of benzene-soluble 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.

4 Reagent

4.1 Acetone, of analytical reagent grade.

WARNING — Acetone is flammable, and toxic by inhalation, ingestion or skin absorption

5 Apparatus

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

The rotational frequency of the centrifuge shall be sufficient to ensure separation of the soluble fraction from the parent coal.

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

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

5.4 Vacuum drying oven, electrically heated, in which a temperature of 80 ± 2 °C and a pressure of about 50 kPa can be maintained.

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

5.6 Infrared drying lamp.

5.7 Wire-cloth test sieve, of nominal aperture size 100 µm.

6 Preparation of sample

The residue obtained from the benzene-soluble extract obtained by the method specified in ISO 975 shall be crushed to pass the sieve (5.7).

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

7 Procedure

7.1 Test condition

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 shall not differ from each other by more than 0,5 °C and shall be within the range 18 to 22 °C.

7.2 Determination

Record the room temperature at the beginning of the extraction (t_2) and the temperature of the acetone (t_1). Weigh, to the nearest 1 mg, about 0,5 g of the sample into a glass vessel (5.2). Add 7 ml of the acetone (Clause 4) and shake for exactly 2 min. Minimise warming of the solvent by holding the glass vessel at the upper end between the index and middle fingers, while the thumb secures the ground-glass stopper. Rubber finger-shields should be worn.

Allow the acetone-soluble fraction to clear and decant it into a tared, dry evaporating dish (5.3). If the fraction does not clear, it may be centrifuged for 1 min and then decanted, or filtered if necessary using the smallest convenient size of filter paper, into the evaporating dish.

Wash back any particles of benzene-soluble extract adhering to the upper end of the glass vessel, after shaking by cautiously tilting the vessel. Let any washed fraction settle or centrifuge it.

Add a further 7 ml of the acetone to the glass vessel and repeat the above extraction (but not more than 4 times) until the extract fraction is colourless.

Record the room temperature at the end of the extraction (t_3) to the nearest 0,1 °C.

If a filter has been used, rinse it with a few millilitres of acetone and add the rinsings to the evaporating dish.

Place the evaporating dish in the vacuum drying oven (5.4) and evaporate off the acetone at 80 ± 2 °C and about 50 kPa. Alternatively, the evaporation may be carried out using the infrared drying lamp (5.6). Transfer the dish to the air oven (5.5) and dry to constant mass at 105 ± 3 °C.

8 Expression of results

The mass fraction of acetone-soluble material, $w_{Ac,20}$, in the sample analysed, expressed as a percentage, is given by the equation

$$w_{Ac,20} = \frac{Km_2}{m_1}$$

where

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

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

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

in which $t = \frac{t_1 + t_2 + t_3}{3}$

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

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

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

The result (the mean of duplicate determinations, see 9.1) should be reported to the nearest 0,1 % (mass fraction).

9 Precision of the method

Mass fraction of acetone-soluble material results %	Maximum acceptable difference between	
	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 the mean result	1,8 % of the mean result

9.1 Repeatability

The results of duplicate determinations, carried out in the same laboratory, by the same operator, using the same apparatus, on the same benzene-soluble extract, within short intervals of time, should 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 extract, should not differ by more than the above value.

10 Test report

The test report should include the following information:

a) identification of the product tested;

b) a reference to this International Standard;

c) the results and the method of expression used.