SLOVENSKI STANDARD
SIST ISO 1017:1998

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Rjavi premogi in ligniti - Določ evanje v acetonu topnih snovi iz toluenskega ekstrakta

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

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

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73.040 Premogi Coals

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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 toluene-soluble extract

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

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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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 1017 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*.

This second edition cancels and replaces the first edition (ISO 1017-1975), of which it constitutes a technical revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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

3 Principle

The sample of toluene-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

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 vessels, either cylindrical or conical, of 15 ml capacity 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 Infra-red drying lamp.

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

6 Preparation of sample

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

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

7 Procedure

7.1 Test conditions

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

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 (see note 1). Allow the acetone-soluble fraction to clear and decant it into the 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 (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 until three extractions have been carried out or the extractant is clear, whichever is achieved first. 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 (5.4) and evaporate off the acetone at 80 ± 2 °C and about 50 kPa. Alternatively, the evaporation may be carried out using the infra-red drying lamp (5.6). Transfer the dish to the air oven (5.5) and dry to constant mass at 105 ± 3 °C.

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 ground glass 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 toluene-soluble 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.

8 Expression of results

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

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

where

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

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

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

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

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 (the mean of duplicate determinations, see 9.1) shall be reported to the nearest 0,1 % (m/m).

9 Precision of the method

Amount of acetone-soluble material % (m/m)	Maximum acceptable differences 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 the mean result	1,8 % of the mean 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 toluene-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 toluene-soluble fraction, shall not differ by more than the above value.

10 Test report

The test report shall include the following particulars:

a) identification of the product tested;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or in the International Standard to which reference is made, or regarded as optional.