SLOVENSKI STANDARD
SIST ISO 975:2002

01-junij-2002

Rjavi premogi in ligniti - Določ evanje izkoristka ekstrakta, topnega v benzenu - Polavtomatska metoda

Brown coals and lignites -- Determination of yield of benzene-soluble extract -- Semi-automatic method

Charbons bruns et lignites -- Détermination du rendement en extrait de benzène soluble -- Méthode semi-automatique

Ta slovenski standard je istoveten z: **ISO 975:2000**

ICS:

73.040	Premogi	Coals

SIST ISO 975:2002 **en**

INTERNATIONAL STANDARD

ISO 975

Third edition
2000-10-01

Brown coals and lignites — Determination of yield of benzene-soluble extract — Semi-automatic method

Charbons bruns et lignites — Détermination du rendement en extrait de benzène soluble — Méthode semi-automatique

Reference number
ISO 975:2000(E)

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Printed in Switzerland

Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 975 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 975:1985), which has been technically revised.

Introduction

The determination of yield of benzene-soluble extract is carried out using a semi-automatic instrument; a system combining extraction, rinsing and evaporation. As long as the sample is put in the extraction chamber of the instrument, the test can be done automatically.

Brown coals and lignites — Determination of yield of benzene-soluble extract — Semi-automatic method

1 Scope

This International Standard specifies a semi-automatic method for determination of the yield of benzene-soluble extract in brown coals and lignites.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For a dated reference, subsequent amendments to, or revisions of, the publication do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For an undated reference, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 5068:1983, *Brown coals and lignites — Determination of moisture content — Indirect gravimetric method.*

3 Principle

A test portion of the brown coal or lignite is extracted with benzene in a semi-automatic extraction instrument. The solvent is then removed by evaporation and the soluble residue dried to constant mass. The percentage of benzene-soluble extract is calculated from the mass of residue after drying and is reported on the dry basis.

4 Reagent

Benzene, of analytical reagent grade, ρ_{20} = 0,876 g/ml, distillation range 80 °C to 81 °C. At least 95 % shall distil within this range.

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

The test must be carried out in a hood and the benzene must be recovered as completely as possible.

5 Apparatus

5.1 Semi-automatic extraction instrument, containing mainly two units: the continuous extraction-evaporation device and the controller. The continuous extraction-evaporation device consists of a 100 ml conical flask, an extraction chamber and a condenser. The extraction chamber is 180 mm long and 30 mm in internal diameter and is provided with a water jacket through which the bath water is circulated in order to maintain the extraction temperature around the extraction chamber.

5.2 Extraction thimble, 25 mm × 80 mm. Cellulose or other thimbles are purchased or made as follows.

Cut filter paper into pieces of 75 mm × 75 mm and 25 mm × 25 mm. Moisten one large piece of the filter paper with distilled water and roll it snugly onto the external wall of a test tube of 25 mm diameter with a small hole pierced at the bottom. A small piece of filter paper is next moistened and rolled onto the bottom. Three large pieces and two small pieces are then rolled alternately onto the test tube. Remove the formed moist thimble by blowing at the mouth of the test tube and dry it in air or in an oven at 100 °C.

5.3 Air oven, capable of maintaining a temperature between 105 °C and 110 °C, or **vacuum oven**, electrically heated, in which a temperature of 80 °C ± 2 °C and a pressure of about 5 kPa can be maintained.

5.4 Analytical balance, accurate to 0,1 mg.

6 Preliminary adjustment of instrument

6.1 Adjustment of the extraction temperature

Place an extraction thimble with about 2 g of coal sample in the extraction chamber. Add 60 ml to 70 ml of benzene to the conical flask. Connect the flask to the extraction chamber. Switch on the electrical power and push down the programme button. The extraction-evaporation device will then be automatically lowered until the flask is immersed in the water bath and the water bath starts heating. As the first drop of condensed benzene drips from the condenser, adjust the temperature of water bath so that the dripping rate of benzene is about 4 ml/min to 5 ml/min and the sample is completely immersed in benzene in the thimble. Record the temperature and fix the position of the temperature controller. This temperature should be readjusted for changed ambient temperature.

6.2 Selection of periods of extraction, rinsing and evaporation

In general, the suitable periods of the above three steps are respectively 180 min, 10 min and 50 min. They can be readily adjusted with the corresponding timers. In the case of a high content of benzene-soluble constituents in the sample or excessively low barometric pressure, etc., readjustment of the extraction period may be necessitated in order to ensure the correct end-point of extraction, which is to be judged by the colourlessness of the last drops of extract solution.

6.3 Procedure

Weigh, to the nearest 0,2 mg, about 2 g of general analysis test sample, transfer to the extraction thimble (5.2) and cover with a pad of absorbent cotton, which is fitted snugly on the wall of the thimble.

Place the extraction thimble with sample in the extraction chamber (see 5.1).

Add 60 ml to 70 ml of benzene to a previously dried and accurately weighed flask.

Assemble the instrument.

Switch on the electrical power. Push down the programme button. The instrument will automatically perform the experiment in accordance with the following sequence:

The extraction-evaporation device is lowered until the flask is immersed in the water bath as in the preliminary adjustment and the condenser is in an inclined position permitting refluxing. The heating of the water bath is started simultaneously.

When the extraction temperature previously set is reached, the pump begins to circulate the hot water between the bath and the jacket of the extraction chamber. The benzene vapour from the flask passes through the extraction chamber and reaches the condenser, where it is condensed and drips onto the thimble. The extraction stage is thus in progress.

After 180 min, or an otherwise set time, the pump stops and extraction is finished. Hot water flows back to the bath. The temperature of the extraction chamber drops to a temperature at which the benzene vapour can only reach the extraction chamber and condenses there. Thus a rinsing action is achieved, by means of which the benzene-

soluble extract adhered to the wall is washed down into the flask by the condensed benzene. This is the rinsing stage.

After 10 min, rinsing is finished. The condenser is automatically changed to an inverted position permitting distillation. The pump works again so as to resume hot-water circulation. The benzene vapour condenses in the condenser and flows to the receiver. The stage of evaporation starts.

After 50 min of evaporation is finished, the extraction-evaporation system is elevated to the original position, permitting the flask to be detached from the extraction chamber. The programme is thus terminated.

Detach the flask with soluble residue. Dry it in the air oven (5.3) maintained at 105 °C to 110 °C or in the vacuum oven maintained at 80 °C ± 2 °C and about 50 kPa to constant mass.

NOTE Constancy in mass is considered to have been achieved when the difference between successive dryings does not exceed 0,001 g.

Carry out a moisture determination on a separate test portion by the method specified in ISO 5068.

7 Expression of results

The yield of benzene-soluble extract, $w_{E,ad}$, in the general analysis test sample, expressed as a percentage by mass, is given by the formula

$$w_{E,ad} = \frac{m_1 \times 100}{m_2}$$

where

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

m_2 is the mass, in grams, of the test portion.

The yield, expressed on the dry basis, is given by the formula

$$w_{E,d} = \frac{100}{100 - w_M} \times w_{E,ad}$$

Where w_M is the mass fraction of moisture, in percent, of the general analysis test sample.

The result (the mean of duplicate determinations, see 8.1) shall be reported on the dry basis to the nearest 0,1 %.

8 Precision of the method

8.1 Repeatability limit

The results of duplicate determinations, carried out at different times within a short interval, in the same laboratory, by the same operator, with the same apparatus, on two representative test portions taken from the same analysis sample, should not differ by more than the values shown in Table 1.