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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

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 27, *Coal and coke*, Subcommittee SC 5, *Methods of analysis*.

This fifth edition cancels and replaces the fourth edition (ISO 975:2013), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— referenced documents have been updated;

— terms and definitions have been added;

— sample has been added;

— calculation and expression of results have been amended;

— precision has been amended;

— test report has been amended.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 5068-2, *Brown coals and lignites — Determination of moisture content — Part 2: Indirect gravimetric method for moisture in the analysis sample*

ISO 13909-4, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*

ISO 18283, *Coal and coke — Manual sampling*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 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.

5 Reagent

5.1 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.

6 Apparatus

6.1 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.

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

Cut filter paper into pieces of 75 mm × 75 mm (large) and 25 mm × 25 mm (small). 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.

6.3 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 50 kPa can be maintained.

6.4 Analytical balance, with a resolution of 0,1 mg or better.

7 Preparation of sample

The sample shall be the general analysis test sample, prepared to a nominal top size of 212 µm by the preparation procedures specified in ISO 13909-4 or ISO 18283.

The sample should be brought in moisture equilibrium with the laboratory atmosphere by exposure in a thin layer on a tray. Exposure time shall be kept to a minimum.

The sample shall be thoroughly mixed immediately before analysis, preferably by mechanical means.

Duplicate determinations of moisture from the same test sample shall be conducted concurrently with the determination of the yield of benzene-soluble extract by the method specified in ISO 5068-2.

8 Preliminary adjustment of instrument

8.1 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 the 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.

8.2 Selection of periods of extraction, rinsing and evaporation

In general, the suitable periods of extraction, rinsing and evaporation 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.

8.3 Procedure

Transfer a mass of about 2 g (determined to the nearest 0,1 mg) of general analysis test sample to the extraction thimble (6.2) 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 (6.1).

Add 60 ml to 70 ml of benzene to a previously dried flask of known mass (determined to the nearest 0,1 mg).

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 (6.3) maintained at 105 °C to 110 °C or in the vacuum oven (6.3) maintained at 80 °C ± 2 °C and about 50 kPa to constant mass after allowing it to cool to ambient temperature.

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

9 Calculation and expression of results

9.1 The yield of benzene-soluble extract, $w_{E,ad}$, in the general analysis test sample, expressed as a percent, is given by Formula (1):

$$w_{E,ad} = \frac{m_1 \times 100}{m_2} \tag{1}$$

where

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

m_2 is the mass, in grams, of the test sample;

100 is conversion factor from dimensionless mass fraction to percent, in %.

9.2 The yield, expressed on the dry basis, $w_{E,d}$, is given by Formula (2):

$$w_{E,d} = \frac{100}{100 - w_{M,ad}} \times w_{E,ad} \quad (2)$$

where $w_{M,ad}$ is the mass fraction of moisture, in percent, of the general analysis test sample.

9.3 The result (the mean of duplicate determinations, see 10.1) shall be reported on the dry basis to the nearest 0,1 %.

10 Precision

10.1 Repeatability limit

The results of duplicate determinations, carried out in the same laboratory by the same operator with the same apparatus within a short interval of time on representative portions taken from the same analysis sample, shall not differ by more than the values of the repeatability limit, *r*, shown in Table 1.

10.2 Reproducibility limit

The means of the results of duplicate determinations, carried out in each of two different laboratories, on representative test portions taken from the same sample at the last stage of sample preparation, shall not differ by more than the values of the reproducibility limit, *R*, shown in Table 1.

Table 1 — Repeatability limit and reproducibility limit

Test parameter $w_{E,d}$ %	Maximum acceptable differences between results	
	Repeatability limit r (air-dried basis)	Reproducibility limit R (dry basis)
less than 5	0,3 % absolute	0,5 % absolute
5 to 10 inclusive	0,5 % absolute	0,7 % absolute
more than 10	5 % of the mean result	7 % of the mean result

11 Test report

The test report shall include the following information:

a) identification of the sample tested;

b) the method used by reference to this document, i.e. ISO 975:2021;

c) the date of the determination;

d) the results and the method of expression used.