
International Standard



975

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Brown coals and lignites — Determination of yield of toluene-soluble extract

Charbons bruns et lignites — Détermination du rendement en extrait de toluène soluble

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Foreword

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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 975 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*.

This second edition cancels and replaces the first edition (ISO 975:1975), 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.

Brown coals and lignites — Determination of yield of toluene-soluble extract

1 Scope and field of application

This International Standard specifies a method of determining the yield of toluene-soluble extract in brown coals and lignites.

2 Reference

ISO 1015, *Brown coals and lignites — Determination of moisture content — Direct volumetric method.*

3 Principle

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

4 Reagent

Toluene, of analytical reagent grade, $\rho_{20} = 0,867$ g/ml, distillation range 109 to 111 °C. At least 95 % shall distil within this range.

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

5 Apparatus

5.1 Extraction apparatus. A suitable apparatus (see the figure) consists of the following items:

5.1.1 Flat-bottomed conical flask, of 500 ml capacity, fitted with a 40/38 ground glass socket.

5.1.2 Reflux condenser, fitted with a 40/38 ground glass cone and having a minimum length of water jacket of 400 mm.

5.1.3 Filter-paper extraction thimble, ϕ 30 mm $\times$ 90 mm, supported in a gauze framework.

5.2 Distillation head: A condenser with a 40/38 ground glass cone to fit the socket of the conical flask (5.1.1).

5.3 Sand bath, or other suitable means of heating.

5.4 Vacuum oven, electrically heated, in which a temperature of 80 ± 2 °C and a pressure of about 50 kPa can be maintained.¹⁾

5.5 Evaporating dish, glass or porcelain, about 30 mm high and 80 mm in diameter.

5.6 Balance, accurate to 1 mg.

5.7 Wire cloth test sieve, of nominal aperture size 1 mm $\times$ 1 mm.

6 Preparation of sample

Spread the laboratory sample on a tray and allow it to attain approximate moisture equilibrium with the atmosphere. Carefully crush the sample to pass the test sieve (5.7). The crushed sample may be stored in a stoppered container filled to more than 80 % of its capacity.

Before commencing the determination, mix the crushed sample thoroughly for at least 1 min, preferably by mechanical means.

7 Procedure

Weigh, to the nearest 1 mg, about 10 g of the crushed sample, transfer to the extraction thimble (5.1.3) and cover with clean cotton wool.

Place the extraction thimble in the wire gauze framework and attach the framework to the end of the reflux condenser (5.1.2) so that the condensed toluene (clause 4) will drip into the extraction thimble. Add 150 ml of the toluene to the flask (5.1.1) and connect the flask to the reflux condenser. Heat the

1) A ventilated air oven maintained at 110 to 115 °C or other means of drying may be employed, provided that care is taken to prevent ignition of the toluene-soluble extract.

flask on the sand bath or other suitable means (5.3) so as to maintain a steady flow of refluxed toluene through the sample. Continue heating for 4 h or until the toluene leaving the extraction thimble is nearly clear, whichever is the longer.

Replace the reflux condenser by the distillation head (5.2) and distil off the toluene until about 20 ml of liquid is left in the flask; transfer this quantitatively to the tared evaporating dish (5.5), by washing with toluene from a wash bottle. Evaporate the remaining toluene in the vacuum oven (5.4), maintained at 80 ± 2 °C and about 50 kPa. Dry the residual extract to constant mass.

NOTE — Constancy in mass is considered to have been achieved when the difference between successive weighings does not exceed 1 % of the mass of the extract.

Carry out a moisture determination on a separate test portion by the method specified in ISO 1015.

8 Expression of results

The toluene-soluble extract, E_T , in the sample, expressed as a percentage by mass, is given by the equation

$$E_T = \frac{m_2 \times 100}{m_1}$$

where

m_1 is the mass, in grams, of the test portion;

m_2 is the mass, in grams, of toluene-soluble extract.

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

$$E_T \times \frac{100}{100 - M}$$

where M is the moisture content of the sample for analysis, expressed as a percentage by mass.

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

9 Precision of the method

Yield of toluene soluble extract, % (m/m) (dry basis)	Maximum acceptable differences between results	
	Repeatability	Reproducibility
Less than 5	0,3 % absolute	0,5 % absolute
5 to 10 inclusive	0,5 % absolute	0,7 % absolute
Greater than 10	5 % of the mean result	7 % 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 two representative test portions taken from the same crushed sample for analysis, shall not differ by more than the above value.

9.2 Reproducibility

The mean of the results of duplicate determinations, carried out in two different laboratories on representative test portions taken from the same crushed sample for analysis, shall not differ by more than the above value.

NOTE — If the samples are circulated at a size greater than 1 mm, these tolerances may be exceeded.

10 Test report

The test report shall include the following particulars:

- identification of the product tested;
- the reference of the method used;
- the results and the method of expression used;
- any unusual features noted during the determination;
- any operation not included in this International Standard or in the International Standard to which reference is made, or regarded as optional.

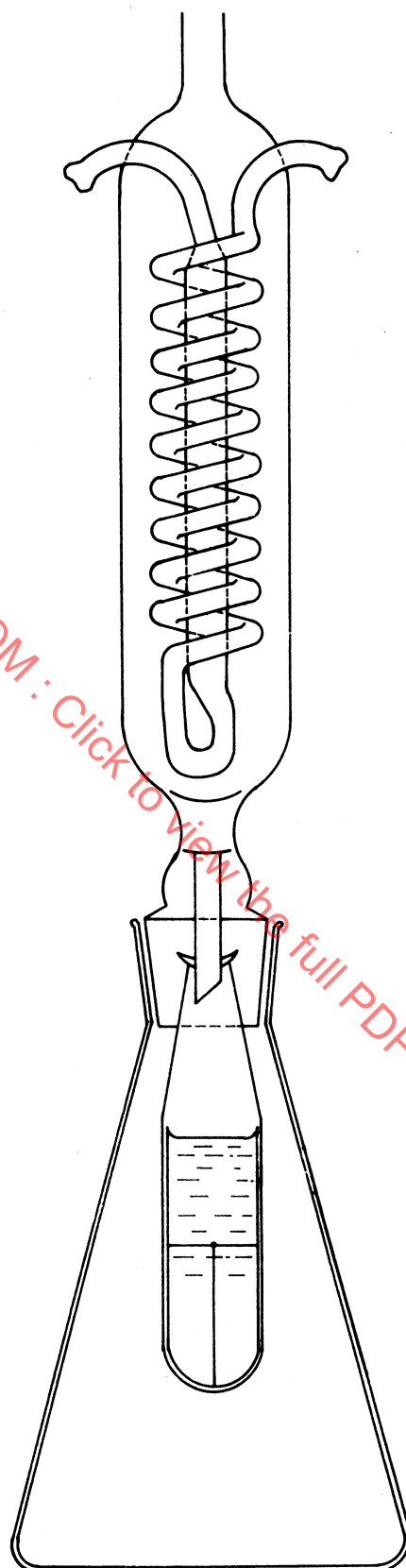


Figure — Apparatus assembly for determination of yield of toluene-soluble extract

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