

INTERNATIONAL STANDARD



975

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Brown coals and lignites — Determination of yield of ~~benzene~~-soluble extract

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

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Toluene

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 27 has reviewed ISO Recommendation R 975 and found it technically suitable for transformation. International Standard ISO 975 therefore replaces ISO Recommendation R 975-1969 to which it is technically identical.

ISO Recommendation R 975 was approved by the Member Bodies of the following countries :

Australia	India	Romania
Austria	Ireland	South Africa, Rep. of
Belgium	Israel	Spain
Bulgaria	Italy	Sweden
Chile	Japan	Switzerland
Czechoslovakia	Korea, Rep. of	Turkey
Denmark	Netherlands	United Kingdom
Egypt, Arab Rep. of	New Zealand	U.S.A.
France	Poland	U.S.S.R.
Germany	Portugal	Yugoslavia

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 975 into an International Standard.

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

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method of determining the yield of benzene-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 known mass of the brown coal or lignite is extracted with benzene in an extraction apparatus. 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 quality, ρ_{20} 0,876 g/ml, distillation range 80 to 81 °C. At least 95 % shall distil within this range.

5 APPARATUS

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

- a flat-bottomed conical flask, 500 ml, fitted with a 40/38 ground glass socket;
- a reflux condenser, fitted with a 40/38 ground glass cone and having a minimum length of water jacket of 400 mm;
- a filter-paper extraction thimble, 30 mm X 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.

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

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

5.5 Balance, accurate to 1 mg.

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 a 1 mm square mesh sieve. 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 accurately about 10 g of the crushed sample, transfer to the extraction thimble 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 so that the condensed benzene will drip into the extraction thimble. Add 150 ml of benzene to the flask and connect the flask to the reflux condenser. Heat the flask on a sand bath, or by other suitable means, so as to maintain a steady flow of refluxed benzene through the sample. Continue heating for 4 h or until the benzene leaving the extraction thimble is nearly clear, whichever is the longer.

Replace the reflux condenser by the distillation head and distil off the benzene until about 20 ml of liquid is left in the flask; transfer this quantitatively to a weighed evaporating dish, by washing with benzene from a wash bottle. Evaporate the remaining benzene in the vacuum oven at 80 °C and about 50 kPa.¹⁾ 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 0,001 g.

Carry out a moisture determination on a separate portion of the sample by the method given in ISO 1015.

8 EXPRESSION OF RESULTS

The benzene-soluble extract, E_B , in the sample, as a percentage by mass, is given by the formula

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

where

- m_1 is the mass, in grams, of the test portion;
- m_2 is the mass, in grams, of benzene-soluble extract.

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

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

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

The result, preferably the mean of duplicate determinations (see clause 9), shall be reported on the dry basis to the nearest 0,1 %.

9 PRECISION OF THE METHOD

Yield of benzene-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 :

- a) the reference of the method used;
- b) the results and the method of expression used;
- c) any unusual features noted during the determination;
- d) any operation not included in this International Standard or regarded as optional.

1) A ventilated air oven maintained at 100 to 105 °C or other means of drying may be employed, provided that care is taken to prevent ignition of the benzene vapour or decomposition of the residue.

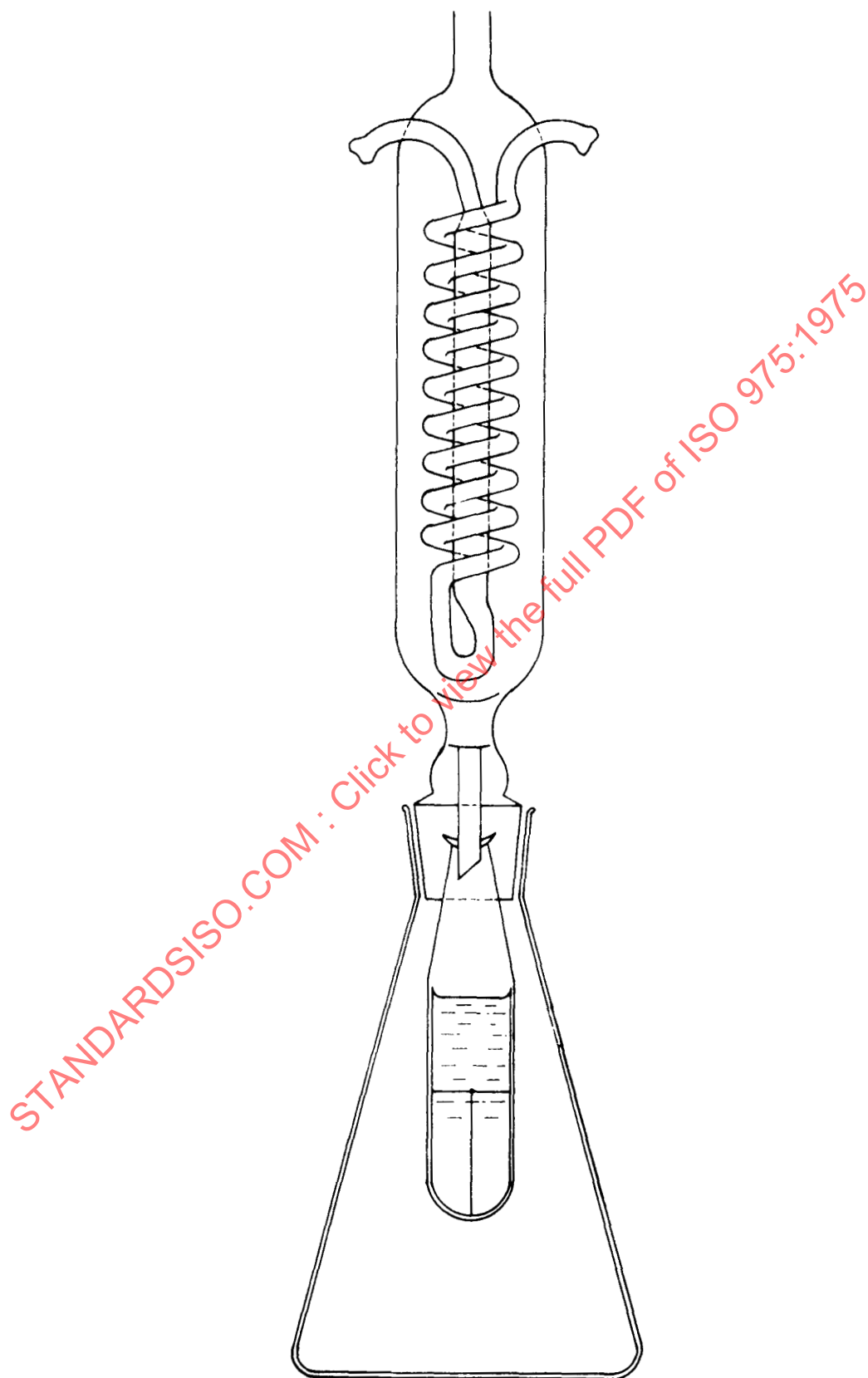


FIGURE — Apparatus assembly for determination of benzene-soluble extract

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