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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

**ISO RECOMMENDATION
R 975**

**DETERMINATION OF THE YIELD OF BENZENE-SOLUBLE EXTRACT
IN BROWN COALS AND LIGNITES**

**1st EDITION
February 1969**

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BRIEF HISTORY

The ISO Recommendation R 975, *Determination of the yield of benzene-soluble extract in brown coals and lignites*, was drawn up by Technical Committee ISO/TC 27, *Solid mineral fuels*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question led, in 1965, to the adoption of a Draft ISO Recommendation.

In November 1966, this Draft ISO Recommendation (No. 1126) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in February 1969, to accept it as an ISO RECOMMENDATION.

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DETERMINATION OF THE YIELD OF BENZENE-SOLUBLE EXTRACT IN BROWN COALS AND LIGNITES

1. SCOPE

This ISO Recommendation describes a method of determining the yield of benzene-soluble extract in brown coals and lignites.

2. PRINCIPLE OF METHOD

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.

3. REAGENT

Benzene, of analytical reagent quality, $\rho_{20} = 0.876$, distillation range 80 to 81 °C. At least 95 % should distil within this range.

4. APPARATUS

- 4.1 *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 × 90 mm, supported in a wire gauze framework.
- 4.2 *Distillation head*. A condenser with a 40/38 ground glass cone to fit the socket of the conical flask.
- 4.3 *Vacuum oven*. An electrically heated oven in which a temperature of 80 ± 2 °C and a pressure of about 0.5 bar can be maintained.
- 4.4 *Evaporating dish*. A glass or porcelain dish about 30 mm high and 80 mm in diameter.
- 4.5 *Balance*, accurate to 1 mg.

5. 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.

6. PROCEDURE

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

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 hours 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 0.5 bar*. Dry the residual extract to constant mass.

NOTE. – Constancy in mass is 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 Recommendation R ...**, *Determination of moisture in brown coals and lignites by the direct volumetric method.*

* 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.

** At present Draft ISO Recommendation No. 1282.