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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1017

BENZENE EXTRACT FROM BROWN COALS AND LIGNITES

DETERMINATION OF ACETONE-SOLUBLE MATERIAL

("RESINOUS SUBSTANCES")

1st EDITION
March 1969

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 1017, *Benzene extract from brown coals and lignites – Determination of acetone-soluble material* ("*Resinous substances*"), 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 1966, to the adoption of a Draft ISO Recommendation.

In August 1967, this Draft ISO Recommendation (No. 1284) 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	Italy	Switzerland
Austria	Korea, Rep. of	Turkey
Canada	Netherlands	U.A.R.
Czechoslovakia	New Zealand	United Kingdom
Denmark	Portugal	U.S.S.R.
France	Romania	Yugoslavia
India	South Africa, Rep. of	
Iran	Spain	

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 March 1969, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 1017 March 1969

BENZENE EXTRACT FROM BROWN COALS AND LIGNITES

DETERMINATION OF ACETONE-SOLUBLE MATERIAL

("RESINOUS SUBSTANCES")

1. SCOPE

This ISO Recommendation describes a method of determining the amount of acetone-soluble material ("resinous substances") in the benzene extract from brown coals and lignites.

NOTE. – The acetone extract will also contain a percentage of wax dissolved simultaneously with the "resinous substances".

2. PRINCIPLE

The sample of benzene extract from brown coal or lignite obtained by the procedure described in ISO Recommendation R 975, *Determination of the yield of benzene-soluble extract in brown coals and lignites*. is extracted with acetone at a temperature of 18 to 22 °C. The soluble fraction is filtered or centrifuged off and after evaporation of the solvent dried to constant mass. The percentage of acetone-soluble material is calculated from the mass of residue after drying.

NOTE. – The high selectivity of acetone requires a strict temperature control during the determination. The temperature of the solvent, the room temperature at the beginning of the determination and the room temperature at the end of the determination should not differ from each other by more than 0.5 °C and should be within the range 18 to 22 °C.

3. SAMPLE

The residue obtained from the benzene extract is crushed and passed through a sieve of 0.1 mm aperture.

If the residue is a viscous liquid it is cooled in solid carbon dioxide to –80 °C and then crushed.

4. REAGENT

Acetone, of analytical reagent quality.

5. APPARATUS

5.1 *Centrifuge*, capable of being spun at 1600 rev/min.

5.2 *Glass vessels*, either cylindrical or conical, of 15 ml capacity and fitted with rubber stoppers, for use in the centrifuge.

5.3 *Evaporating dish*, of glass or silica, about 20 mm high by 50 mm diameter.

5.4 *Vacuum drying oven*, electrically heated.

5.5 *Air-oven*, electrically heated to a temperature of 100 to 110 °C.

5.6 *Infra-red drying lamp.*

6. PROCEDURE

Weigh to the nearest 0.001 g, about 0.5 g of the sample into the glass vessel, Add 7 ml of the acetone (4) and shake for exactly 2 minutes (see Note 1, below). Allow the acetone-soluble fraction to clear and decant it into the weighed, dry evaporating dish. If the fraction does not clear it may be centrifuged for 1 minute and then decanted, or filtered if necessary (see Note 2, below), into the evaporating dish (see Note 3, below).

Add a further 7 ml of the acetone to the glass vessel and repeat the above extraction (see Note 4, below). If a filter has been used, rinse it with a few millilitres of the acetone and add the rinsings to the evaporating dish.

Place the evaporating dish in the vacuum drying oven and evaporate off the acetone at 80 ± 20 °C and about 0.5 bar absolute. Alternatively, the evaporation may be carried out using the infra-red drying lamp. Transfer the dish to the air oven and dry to constant mass.

NOTES

1. Warming of the solvent may be minimized by holding the glass vessel at the upper end between the index and middle fingers, while the thumb secures the rubber stopper. Rubber finger shields should be worn.

2. Since the acetone solution will creep up the filter paper, the smallest convenient size of paper should be used.

3. Any particles of benzene extract adhering to the upper end of the glass vessel after shaking should be washed back by cautious tilting and the fraction again left so settle, or centrifuged.

4. If the second acetone extract is strongly coloured, a third extraction should be carried out.