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INTERNATIONAL STANDARD ISO 925

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION •МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ •ORGANISATION INTERNATIONALE DE NORMALISATION

Hard coal — Determination of carbon dioxide content — Gravimetric method

Houille — Dosage du dioxyde de carbone — Méthode gravimétrique

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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 925 and found it technically suitable for transformation. International Standard ISO 925 therefore replaces ISO Recommendation R 925-1969 to which it is technically identical.

ISO Recommendation R 925 was approved by the Member Bodies of the following countries :

Australia
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Romania
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Switzerland
Turkey
United Kingdom
U.S.A.
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Yugoslavia

No Member Body expressed disapproval of the Recommandation.

No Member Body disapproved the transformation of ISO/R 925 into an International Standard.

Printed in Switzerland

INTERNATIONAL STANDARD

ISO 925-1975 (E)

Hard coal – Determination of carbon dioxide content – Gravimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method for the determination of carbon dioxide in the mineral carbonates associated with hard coal.

NOTE – The result obtained will include any carbon dioxide absorbed by the coal.

2 PRINCIPLE

The sample of coal is treated with hydrochloric acid, which reacts with the carbonates present to liberate carbon dioxide. The carbon dioxide resulting from the decomposition of the carbonate is absorbed and weighed.

3 REAGENTS

All reagents, unless otherwise specified, shall be of analytical reagent quality, and water free from carbon dioxide shall be used throughout.

NOTE – Distilled water may be freed from carbon dioxide by boiling gently for 15 min.

3.1 Hydrochloric acid, approximately 3 N.

3.2 Copper phosphate, granular, particle size 0,7 to 1,2 mm.

3.3 Magnesium perchlorate, dried, particle size 0,7 to 1,2 mm.

NOTE – Regeneration of magnesium perchlorate should not be attempted, owing to the risk of explosion. When exhausted, the magnesium perchlorate should be washed down the sink with a stream of water.

3.4 Soda-asbestos, particle size 1,2 to 1,7 mm, preferably self-indicating.

4 APPARATUS

All graduated apparatus shall be of the best analytical quality available.

The system comprises a purification train, a reaction flask assembly and an absorption train. A suitable apparatus is shown in the figure, and consists of :

4.1 Purification train. A bubbler containing sulphuric acid (ρ 1,84 g/ml) and an absorption tube containing soda-asbestos.

4.2 Reaction flask assembly. A 300 ml flat-bottomed flask fitted with a tap funnel, a double-surface condenser and a bulbed tube.

4.3 Absorption train. Three absorption tubes packed respectively as follows :

a) magnesium perchlorate to dry the gas;

b) granular copper phosphate to absorb hydrogen sulphide, followed by a protective layer of magnesium perchlorate;

c) soda-asbestos to absorb carbon dioxide, followed by a protective layer of magnesium perchlorate to absorb water produced in the reaction between carbon dioxide and soda-asbestos.

4.4 Balance, accurate to 0,1 mg.

5 PREPARATION OF SAMPLE

The coal used for the determination is the analysis sample, ground to pass a sieve of 0,2 mm aperture. Before commencing the determination, mix the sample thoroughly for at least 1 min, preferably by mechanical means.

If necessary, expose the sample in a thin layer for the minimum time required to reach approximate equilibrium with the laboratory atmosphere.

6 PROCEDURE

Weigh accurately, to the nearest 0,01 g, about 5 g of the coal (see note 1 below) into the reaction flask and add 100 ml of water. Close the flask by means of a rubber stopper and shake vigorously to wet the coal. Remove the stopper and wash any coal adhering to it back into the flask. Assemble the apparatus as shown in the figure, and draw air through it at a rate of about 50 ml/min for about 10 min. Stop the circulation of air, remove the absorption tube and close the open end of the tube connected to it. Wipe the absorption tube with a clean dry cloth free from loose fibres, allow to cool to the balance room temperature and weigh after 15 min. Reconnect the absorption tube to the apparatus and recirculate air as before. Place 25 ml of the hydrochloric acid (3.1) in the tap funnel. Vent the system to air and admit the acid to the reaction flask. Raise the temperature of the liquid in the reaction flask slowly so that it boils after about 15 min (see note 2 below). Continue boiling for a further 30 min, the rate of boiling being adjusted so that the condenser is not overloaded. Stop the circulation of air, remove the absorption tube, condition and weigh as before.

NOTES

1 For coals containing more than 2 % of carbon dioxide, the sample mass may be reduced.

2 If undue frothing occurs, repeat the determination, adding a few drops of industrial spirit to the mixture in the reaction flask.

7 EXPRESSION OF RESULTS

The carbon dioxide (CO_2) content in the sample as analysed, expressed as a percentage by mass, is given by the formula

$$\frac{100\, m_2}{m_1}$$

where

m_1 is the mass, in grams, of coal taken;

m_2 is the increase in mass, in grams, of the absorption tube.

The result (preferably the mean of duplicate determinations; see clause 8) shall be reported to the nearest 0,01 %.

8 PRECISION OF THE METHOD

Carbon dioxide	Maximum acceptable differences between results	
	Repeatability	Reproducibility
Up to 1 %	0,05 % absolute	0,10 % absolute
1 % and over	5 % of the mean result	10 % of the mean result

8.1 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 portions taken from the same analysis sample (see note below), shall not differ by more than the above value.

8.2 Reproducibility

The means of the results of duplicate determinations, carried out in two different laboratories on representative portions taken from the same analysis sample at the last stage of sample preparation (see note below), shall not differ by more than the above value.

NOTE — If the analysis sample has not been ground to pass a 2 mm sieve, the differences between duplicate determinations may be proportionately larger.

9 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.

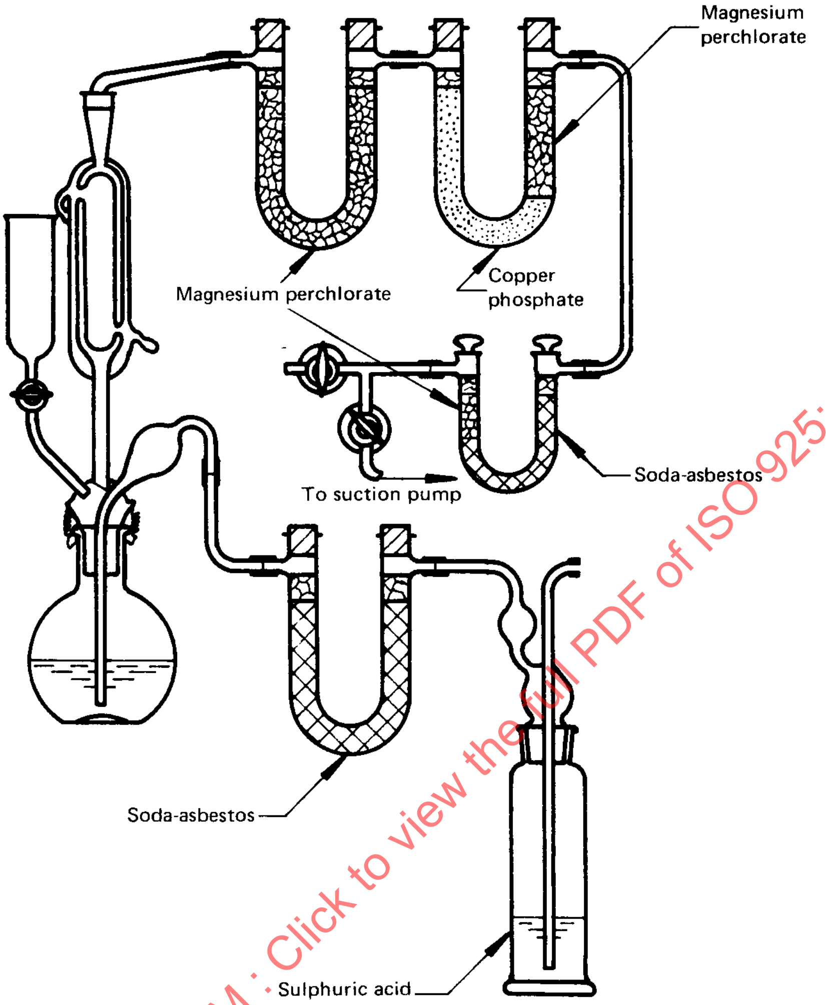

FIGURE – **Apparatus assembly**