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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 925

DETERMINATION OF CARBON DIOXIDE IN COAL BY THE GRAVIMETRIC METHOD

1st EDITION
January 1969

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BRIEF HISTORY

The ISO Recommendation R 925, *Determination of carbon dioxide in coal by the gravimetric method,* 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 by the Technical Committee led, in 1966, to the adoption of a Draft ISO Recommendation.

In November 1966, this Draft ISO Recommendation (No. 1125) 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
Austria
Belgium
Bulgaria
Canada
Chile
Czechoslovakia
Denmark
France
Germany
India
Ireland
Italy
Japan
Korea, Rep. of
Netherlands
New Zealand
Poland
Portugal
Romania
Spain
South Africa, Rep. of
Sweden
Switzerland
Turkey
U.A.R.
United Kingdom
U.S.S.R.
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 January 1969, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 925 January 1969

DETERMINATION OF CARBON DIOXIDE IN COAL BY THE GRAVIMETRIC METHOD

1. SCOPE

This ISO Recommendation describes the 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 OF THE METHOD

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, should be of analytical reagent quality, and water free from carbon dioxide should be used throughout (see Note 1 below).

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 (see Note 2 below).

3.4 *Soda-asbestos,* particle size 1.2 to 1.7 mm, preferably self-indicating.

NOTES

1. Distilled water may be freed from carbon dioxide by boiling gently for 15 minutes.
2. 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.

4. APPARATUS

All graduated apparatus should 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 ($d = 1.84$) 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 guard of magnesium perchlorate,

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

4.4 *Balance,* sensitive to 0.1 mg.

5. 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, the sample should be mixed thoroughly for at least 1 minute, preferably by mechanical means.

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 minutes. 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 minutes. 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 minutes (see Note 2 below). Continue boiling for a further 30 minutes, 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 weight 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 calculated from the following formula :

$$\text{Content of } CO_2 = \frac{100\, M_2}{M_1}$$

where

M_1 is the mass of coal taken, in grammes,

M_2 is the increase in mass of the absorption tube, in grammes.

The result (preferably the mean of duplicate determinations; see section 8 below) should be reported to the nearest 0.01 %.

8. PRECISION OF METHOD

Carbon dioxide	**Maximum acceptable differences between results obtained** **in the same laboratory (repeatability)**	**in different laboratories (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), should 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), should 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 determination may be proportionately larger.

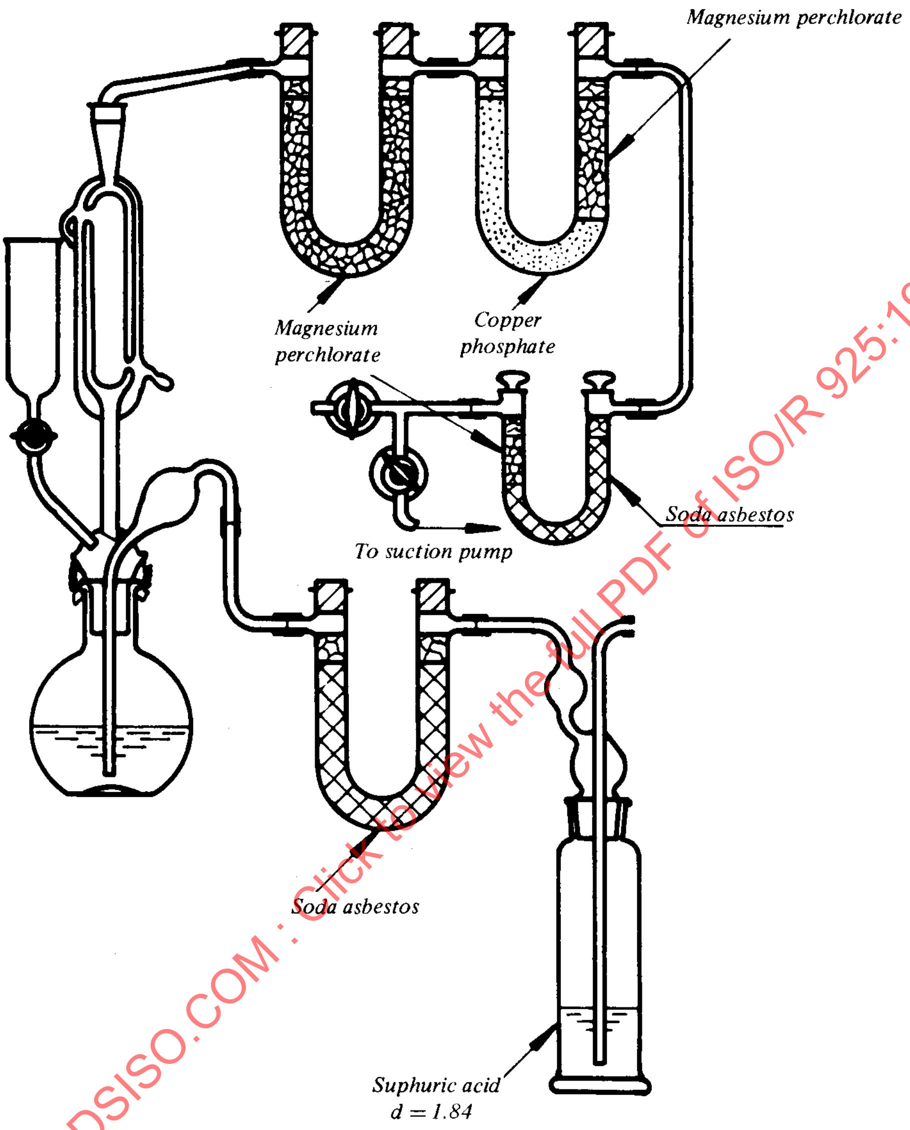

FIGURE – **Apparatus assembly**