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# International Standard



# 925

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

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## Solid mineral fuels — Determination of carbon dioxide content — Gravimetric method

*Combustibles minéraux solides — Dosage du dioxyde de carbone — Méthode gravimétrique*

Second edition — 1980-08-01

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UDC 662.66 : 546.234-31 : 543.21

Ref. No. ISO 925-1980 (E)

Descriptors : Coal, chemical analysis, carbon dioxide, determination of content, gravimetric analysis.

## 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.

International Standard ISO 925 was developed by Technical Committee ISO/TC 27, *Solid mineral fuels*.

This second edition was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 925-1975), which had been approved by the member bodies of the following countries :

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

No member body had expressed disapproval of the document.

# Solid mineral fuels — 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 solid mineral fuels.

NOTE — The result obtained will include any carbon dioxide absorbed by the fuel.

## 2 Principle

The sample 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 mol/l.

**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.

**3.5 Wetting agent**, e.g. ethanol 95 % (V/V).

## 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 ( $\rho = 1,84$  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 sample used for the determination is the analysis sample, ground to pass a sieve of 200  $\mu\text{m}$  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 sample (see note) into the reaction flask and add 5 drops of the wetting agent (3.5) and 100 ml of water. Close the flask by means of a rubber stopper and shake vigorously to wet the sample. Remove the stopper and wash any sample 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 it to cool to the balance room temperature and weigh it 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. 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 it as before.

NOTE — For fuels containing more than 2 % of carbon dioxide, the sample mass may be reduced.

## 7 Expression of results

The carbon dioxide (CO<sub>2</sub>) 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 sample 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.

## 9 Test report

The test report shall include the following particulars :

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

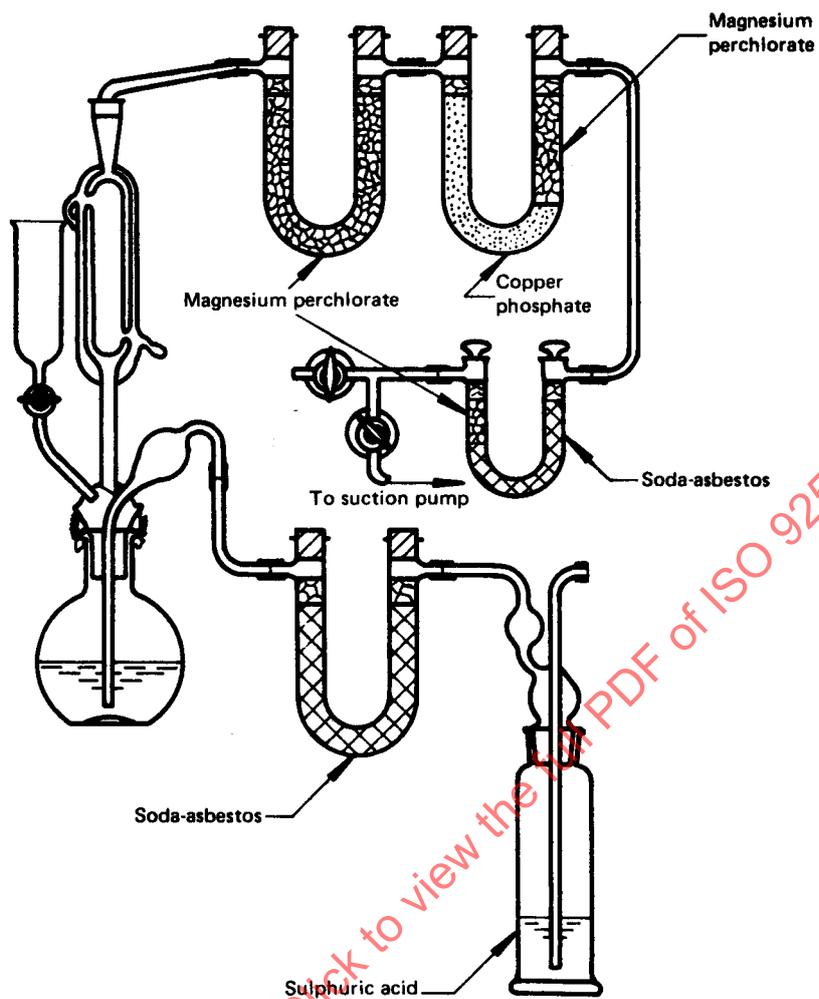


Figure – Apparatus assembly

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