SLOVENSKI STANDARD
SIST ISO 925:1998

01-maj-1998

Trda fosilna goriva - Doloļ evanje karbonatnega ogljika - Gravimetrijska metoda

Solid mineral fuels -- Determination of carbonate carbon content -- Gravimetric method

Combustibles minéraux solides -- Dosage du carbone sous forme de carbonate -- Méthode gravimétrique

Ta slovenski standard je istoveten z: **ISO 925:1997**

ICS:

75.160.10	Trda goriva	Solid fuels

SIST ISO 925:1998 **en**

INTERNATIONAL STANDARD

ISO
925

Third edition
1997-05-01

Solid mineral fuels — Determination of carbonate carbon content — Gravimetric method

Combustibles minéraux solides — Dosage du carbone sous forme de carbonate — Méthode gravimétrique

Reference number
ISO 925:1997(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 925 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This third edition cancels and replaces the second edition (ISO 925:1980), which has been technically revised.

Annex A of this International Standard is for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

Printed in Switzerland

INTERNATIONAL STANDARD © ISO **ISO 925:1997(E)**

Solid mineral fuels — Determination of carbonate carbon content — Gravimetric method

1 Scope

This International Standard specifies a gravimetric method of determining the carbon in the mineral carbonates associated with solid mineral fuels.

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

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 331:1983, *Coal — Determination of moisture in the analysis sample — Direct gravimetric method.*

ISO 687:1974, *Coke — Determination of moisture in the analysis sample.*

ISO 1015:1992, *Brown coals and lignites — Determination of moisture content — Direct volumetric method.*

ISO 1170:1977, *Coal and coke — Calculation of analyses to different bases.*

ISO 1988:1975, *Hard coal — Sampling.*

ISO 2309:1980, *Coke — Sampling.*

ISO 5068:1983, *Brown coals and lignites — Determination of moisture content — Indirect gravimetric method.*

ISO 5069-2:1983, *Brown coals and lignites — Principles of sampling — Part 2: Sample preparation for determination of moisture content and for general analysis.*

ISO 9411-1:1994, *Solid mineral fuels — Mechanical sampling from moving streams — Part 1: Coal.*

ISO 9411-2:1993, *Solid mineral fuels — Mechanical sampling from moving streams — Part 2: Coke.*

3 Principle

A known mass of 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 carbonates is absorbed and weighed.

4 Reagents

WARNING — Care should be exercised when handling reagents, many of which are toxic and corrosive.

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water, or water of equivalent purity.

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

4.1 Hydrochloric acid, approximately 3 mol/l.

4.2 Hydrogen sulfide absorbent

Any of the following:

a) copper(II) phosphate, granular, particle size 1,2 mm to 0,7 mm;

NOTE 1 Copper(II) phosphate granules may be prepared as follows:

Mix copper(II) phosphate powdered reagent to a stiff paste with 1 % starch solution. Press through a sheet of metal, perforated with apertures of approximately 1 mm diameter. Dry the extruded material at 110 °C. Sieve to recover the desired size fraction.

b) copper(II) sulfate, deposited on a supporting base of ground pumice;

NOTE 2 A suitable absorbent, based on copper(II) sulfate, may be prepared as follows:

Prepare pumice by crushing and sieving to obtain the 2,8 mm to 0,7 mm fraction. Transfer approximately 60 g of the prepared pumice to an evaporating basin, covering with a saturated solution of copper(II) sulfate, evaporate to dryness with constant stirring, and heat at 150 °C to 160 °C for 3 h to 4 h. Cool in a desiccator and store in a glass-stoppered bottle.

c) silver sulfate, granular.

4.3 Magnesium perchlorate, anhydrous, particle size 1,2 mm to 0,7 mm.

WARNING — Due regard must be taken of local regulations when disposing of exhausted magnesium perchlorate. It is essential that regeneration of magnesium perchlorate is not attempted, owing to the risk of explosion.

4.4 Sodium hydroxide, on an inert base, preferably of coarse grading, for example 1,7 mm to 1,2 mm, and preferably of the self-indicating type.

4.5 Wetting agent, suitable for use in acid solution.

NOTE — A liquid wetting agent at a concentration of 100 ml/l or ethanol [95 % (*V*/*V*)] are suitable.

4.6 Check test reagent

Either of the following:

a) anhydrous sodium carbonate;

b) anhydrous calcium carbonate.

5 Apparatus

5.1 Analytical balance, capable of weighing to the nearest 0,1 mg.

5.2 Graduated glassware, conforming to the requirements for Grade A in the International Standards prepared by ISO/TC 48, *Laboratory glassware and related apparatus.*

5.3 Purification tube, consisting of an absorption tube[1] containing sodium hydroxide on an inert base (4.4).

5.4 Reaction flask assembly, comprising a 300 ml round-bottomed flask fitted with a tap funnel, a double-surface condenser and a bulbed tube.

5.5 Absorption train, consisting of three absorption tubes a), b) and c)[1] packed respectively as follows:

a) magnesium perchlorate (4.3) to dry the gas;

b) hydrogen sulfide absorbent (4.2) followed by a protective layer of magnesium perchlorate (4.3). The connection from this tube to tube c) shall be fitted at its outlet end with a stopcock or other means of closure;

c) sodium hydroxide on an inert base (4.4), to absorb carbon dioxide generated in the reaction flask, followed by a protective layer of magnesium perchlorate (4.3) to absorb water produced in the reaction between carbon dioxide and sodium hydroxide. This tube shall be fitted with stopcocks or other means of closure at the inlet and outlet ends.

5.6 Air circulation equipment

A suction pump, capable of drawing air at a rate of 50 ml/min through the apparatus, connected through a flowmeter to a tee-piece fitted with a stopcock (the air vent).

5.7 Heating source, for the reaction flask.

Either an electric heating mantle to accommodate a 300 ml flask or a small gas burner.

An example of a suitable assembly of the apparatus is illustrated in figure 1.

6 Preparation of the test sample

The test sample is the general analysis test sample prepared in accordance with ISO 1988, ISO 2309, ISO 5069-2, ISO 9411-1 or ISO 9411-2 as appropriate. Ensure that the moisture content of the sample is in equilibrium with the laboratory atmosphere, exposing it, if necessary, in a thin layer for the minimum time required to achieve equilibrium.

Before commencing the determination, thoroughly mix the equilibrated test sample for at least one minute, preferably by mechanical means.

If the results are to be calculated other than on an "air-dried" basis (see clause 8), then, after weighing the test portion (see 7.2), determine the moisture content using a further portion of the test sample by the method described in ISO 331, ISO 687, ISO 1015 or ISO 5068, as appropriate.

---

1) Absorption tubes may be U-tubes or Midvale tubes (which reduce back-pressure and hence, risk of leakage). The tops of the reagent columns should be covered with a layer of glass wool to guard against entrainment of any fine particles in the circulating air.

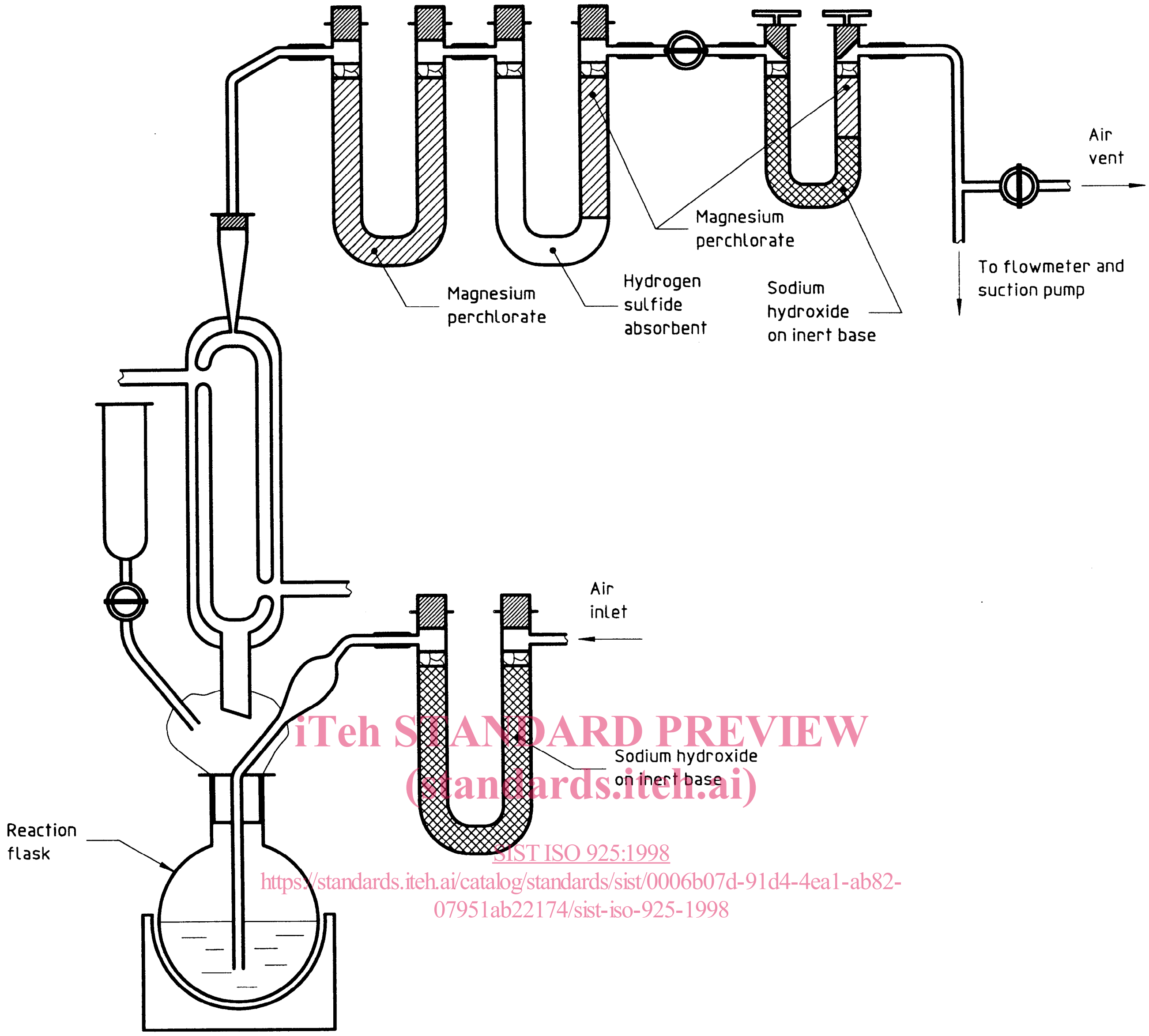

Figure 1 — A suitable assembly of the apparatus

7 Procedure

7.1 Check test

Check the air-tightness of the apparatus and condition of the reagents by running a test according to the procedure described in 7.2, but using approximately 60 mg of the check test reagent (4.6), weighed to the nearest 0,1 mg, instead of the test sample.

Calculate the theoretical mass of carbon dioxide (see A.2), expected to be liberated from the check test portion, and compare it with the measured value. The test is satisfactory if the measured value is within 10 % of the theoretical value; otherwise check the apparatus carefully to seal any leaks and/or replace the reagents. Re-test until a satisfactory result is obtained.

Carry out check tests after initially setting up the apparatus, after having made any changes to it or to the reagents, and before making a determination or series of determinations.

7.2 Determination

7.2.1 Preparation

Weigh accurately, to the nearest 0,01 g, 5 g of the sample into the reaction flask and add 5 drops of the wetting agent (4.5) 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 figure 1, with the air vent closed.

NOTE — For fuels containing more than 0,5 % carbonate carbon, the sample mass may be reduced pro-rata, to a minimum of 0,5 g.

7.2.2 Conditioning

Draw air through the apparatus at a rate of 50 ml/min for 10 min. Stop the circulation of air and close the ends of the carbon dioxide absorption tube and the outlet end of the hydrogen sulfide absorption tube connected to it. Remove the carbon dioxide absorption tube and wipe it with a clean, dry, lint-free cloth.

NOTE 1 Under certain conditions of humidity, wiping the absorbers with a cloth may induce a static charge, which, if significant, could affect the weighing. Consideration should therefore be given to employing static eliminators.

Allow the tube to cool to the balance room temperature and weigh it at 10 min intervals until constant weight is obtained, i.e. two successive weighings do not differ by more than 1 mg.

NOTE 2 Tubes used for the absorption of carbon dioxide, particularly Midvale tubes, cool slowly and up to 60 min should be allowed before weighing. When not connected to the apparatus, the tubes should be protected from atmospheric contamination by closing the taps and fitting guard seals of plugged rubber tubing to the open limbs. It is usual to weigh the absorption tubes without guard seals, after wiping.

7.2.3 Reaction and completion

Reconnect the absorption tube to the apparatus and open the stopcocks or other closures in the absorption train. Place 25 ml of the hydrochloric acid (4.1) in the tap funnel, open the air vent and admit the acid to the reaction flask. Close the air vent and draw air through the system at a rate of about 50 ml/min. 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. Turn off the heating source, stop the circulation of air, remove the absorption tube, wipe clean and weigh as before (see 7.2.2).

8 Expression of results

Results may be expressed in terms of carbonate carbon content or carbon dioxide content, as appropriate, as follows.

The carbonate carbon content of the sample W_C, as analyzed, expressed as a percentage by mass, is given by the formula

$$W_C = \frac{27{,}29 \times m_2}{m_1}$$

and the carbon dioxide content of the sample, W_{CO_2}, as analyzed, expressed as a percentage by mass, is given by the formula

$$W_{CO_2} = \frac{100 \times m_2}{m_1}$$

where

m_1 is the mass, in grams, of the test portion;

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