175

INTERNATIONAL STANDARD

ISO 4283

Second edition
1990-06-01

All grades of fluorspar — Determination of carbonate content — Titrimetric method

Tous les spaths fluor — Dosage des carbonates — Méthode titrimétrique

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Reference number
ISO 4283:1990(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.

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International Standard ISO 4283 was prepared by Technical Committee ISO/TC 175, *Fluorspar*.

This second edition cancels and replaces the first edition (ISO 4283:1978), of which it constitutes a minor revision.

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All grades of fluorspar — Determination of carbonate content — Titrimetric method

1 Scope

This International Standard specifies a titrimetric method for the determination of the carbonate content of all grades of fluorspar.

The method is applicable to products having carbonate contents, expressed as calcium carbonate ($CaCO_3$), equal to or greater than 0,04 % (*m*/*m*).

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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 565:1983, *Test sieves — Woven metal wire cloth, perforated plate and electroformed sheet — Nominal sizes of openings.*

ISO 4282:1977, *Acid-grade fluorspar — Determination of loss in mass at 105 °C.*

ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

3 Principle

Treatment of a test portion with hydrochloric acid solution, absorption of the evolved carbon dioxide in barium hydroxide solution, neutralization of excess alkali with hydrochloric acid solution, addition of an exactly measured excess of a standard volumetric hydrochloric acid solution to dissolve the precipitated barium carbonate and back-titration with a standard volumetric sodium hydroxide solution using methyl orange or screened methyl orange as indicator.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity, free from carbon dioxide.

4.1 Butan-1-ol.

4.2 Nitrogen, free from carbon dioxide.

4.3 Boric acid.

4.4 Barium chloride dihydrate ($BaCl_2.2H_2O$), 122 g/l solution.

4.5 Hydrochloric acid, ρ approximately 1,12 g/ml, about 25 % (*m*/*m*) solution.

Dilute 3 volumes of concentrated hydrochloric acid, ρ approximately 1,19 g/ml, with 2 volumes of water.

4.6 Pumice, impregnated with copper(II) sulfate.

Mix grains of pumice with a saturated copper(II) sulfate solution. Allow to stand for 1 h and filter off the pumice grains. Dry the impregnated grains in an oven (5.3) at 150 °C to 180 °C.

4.7 Potassium hydroxide, about 20 % (*m*/*m*) solution.

4.8 Hydrochloric acid, approximately 36,5 g/l solution.

4.9 Sodium hydroxide, approximately 40 g/l solution.

4.10 Hydrochloric acid, standard volumetric solution, $c(HCl)$ = 0,1 mol/l.

4.11 Sodium hydroxide, standard volumetric solution, $c(NaOH) = 0,1$ mol/l.

4.12 Methyl orange, 1 g/l solution, or

4.13 Screened methyl orange, solution.

Dissolve 1 g of methyl orange and 1,4 g of xylene cyanole FF in 500 ml of 50 % (V/V) ethanol.

4.14 Phenolphthalein, 0,25 g/l solution in 50 % (V/V) ethanol.

5 Apparatus

Ordinary laboratory apparatus, together with the following:

5.1 Gas evolution and absorption apparatus, as illustrated in figure 1), consisting of the following elements:

5.1.1 Washing bottle, equipped with a sintered-glass disc, porosity P 1,6 (see ISO 4793), as shown in figure 1, or similar type, and containing potassium hydroxide solution (4.7).

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5.1.2 Three-necked flask, of capacity 500 ml, fitted with a dropping funnel and a reflux water condenser.

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5.1.3 U-shaped absorption tube (not illustrated in figure 1), containing a suitable quantity of copper(II)-sulfate-impregnated pumice (4.6), normally about 8 g.

NOTE 1 Depletion of the absorption capacity of the impregnated pumice can be readily gauged by the change of colour of the pumice from greyish-blue to black. As long as a small zone of active absorption material is visible, the tube will be fully efficient.

5.1.4 Two washing bottles, Drechsel type.

5.2 Electric oven, capable of being maintained at 105 °C ± 2 °C.

5.3 Oven, capable of being maintained at 150 °C to 180 °C.

6 Test sample

Use the residue from the determination of the loss in mass at 105 °C (see ISO 4282) to prepare the test sample.

NOTE 2 ISO 4282, although specified for acid-grade fluorspar, is equally applicable to all grades of fluorspar.

7 Procedure

7.1 Test portion

Grind several grams of the test sample (see clause 6) in an agate mortar until it all passes through a 63 µm mesh sieve (see ISO 565). Dry the sieved material for 2 h in the electric oven (5.2), maintained at 105 °C ± 2 °C, allow to cool in a desiccator and weigh, to the nearest 1 mg, about 5 g of this sample.

It is essential that the total amount of carbon dioxide, expressed as calcium carbonate, in the test portion does not exceed 100 mg. For samples containing more than 2 % (m/m) of $CaCO_3$, the mass of the test portion shall therefore be reduced in proportion.

7.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same reagents as used for the determination but omitting the test portion.

7.3 Determination

Transfer the test portion (see 7.1) quantitatively to the flask (5.1.2), using about 100 ml of water. Add 4 g of boric acid (4.3). Close the necks of the flask and pass a stream of nitrogen (4.2) through the flask at a rate of 50 ml/min for 10 min.

Without interrupting the nitrogen stream, connect the absorption tube (5.1.3) and the washing bottles (5.1.4), each containing 10 ml of sodium hydroxide solution (4.9), 10 ml of barium chloride solution (4.4), 1 ml of phenolphthalein solution (4.14), 1 ml of butan-1-ol (4.1), and 20 ml of water. Introduce 30 ml of hydrochloric acid solution (4.5) into the flask through the dropping funnel, if necessary by using a rubber bulb. Close the stopcock of the dropping funnel.

Heat the flask slowly and boil gently for 45 min. Stop heating and allow to cool for 10 min without interrupting the nitrogen flow.

Disconnect the second washing bottle (see figure 1) from the apparatus (5.1), and remove and rinse the inlet tube, collecting the washings in the bottle. Titrate the contents of the bottle with hydrochloric acid solution (4.8) until near the end-point. To avoid absorption of atmospheric carbon dioxide during titration of the excess sodium hydroxide in the absorption solution, pass a stream of nitrogen (4.2) through the air space above the solution in the washing bottle.

Continue the titration, but with standard volumetric hydrochloric acid solution (4.10), until the

phenolphthalein is just colourless, taking care not to overshoot the end-point.

Add an exactly measured volume of the standard volumetric hydrochloric acid solution (4.10) until the precipitate dissolves completely. Dip the inlet tube into this solution so as to dissolve any adhering barium carbonate, remove and rinse again. Add a few drops of methyl orange solution (4.12) or screened methyl orange solution (4.13) and back-titrate the excess of hydrochloric acid with standard volumetric sodium hydroxide solution (4.11).

Neutralize and titrate the contents of the first washing bottle (see figure 1) in the same manner.

8 Expression of results

The carbonate content, expressed as a percentage by mass of calcium carbonate ($CaCO_3$), is given by the formula

$$\frac{[(V_1c_1 - V_2c_2) - (V_3c_1 - V_4c_2)] \times 0{,}05005 \times 100}{m}$$

where

V_1 is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (4.10) used to dissolve the barium carbonate in both washing bottles;

V_2 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (4.11) used for the back-titration of the excess hydrochloric acid in both washing bottles;

V_3 and V_4 are the corresponding volumes, in millilitres, of the standard volumetric hydrochloric acid solution (4.10) and the standard volumetric sodium hydroxide solution (4.11) used for the blank test (7.2);

c_1 is the actual concentration, in moles of HCl per litre, of the standard volumetric hydrochloric acid solution (4.10) used;

c_2 is the actual concentration, in moles of NaOH per litre, of the standard volumetric sodium hydroxide solution (4.11) used;

m is the mass, in grams, of the test portion (see 7.1);

0,050 05 is the mass, in grams, of calcium carbonate corresponding to 1,00 ml of hydrochloric acid solution, c(HCl) = 1,000 mol/l.

NOTE 3 Examples of calcium carbonate contents obtained on certified reference fluorspars are given in annex A.

9 Test report

The test report shall include the following particulars:

a) all details necessary for the identification of the sample;

b) a reference to this International Standard;

c) the results and the units in which they have been expressed;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard, or in the International Standards to which reference is made, as well as any operation regarded as optional.

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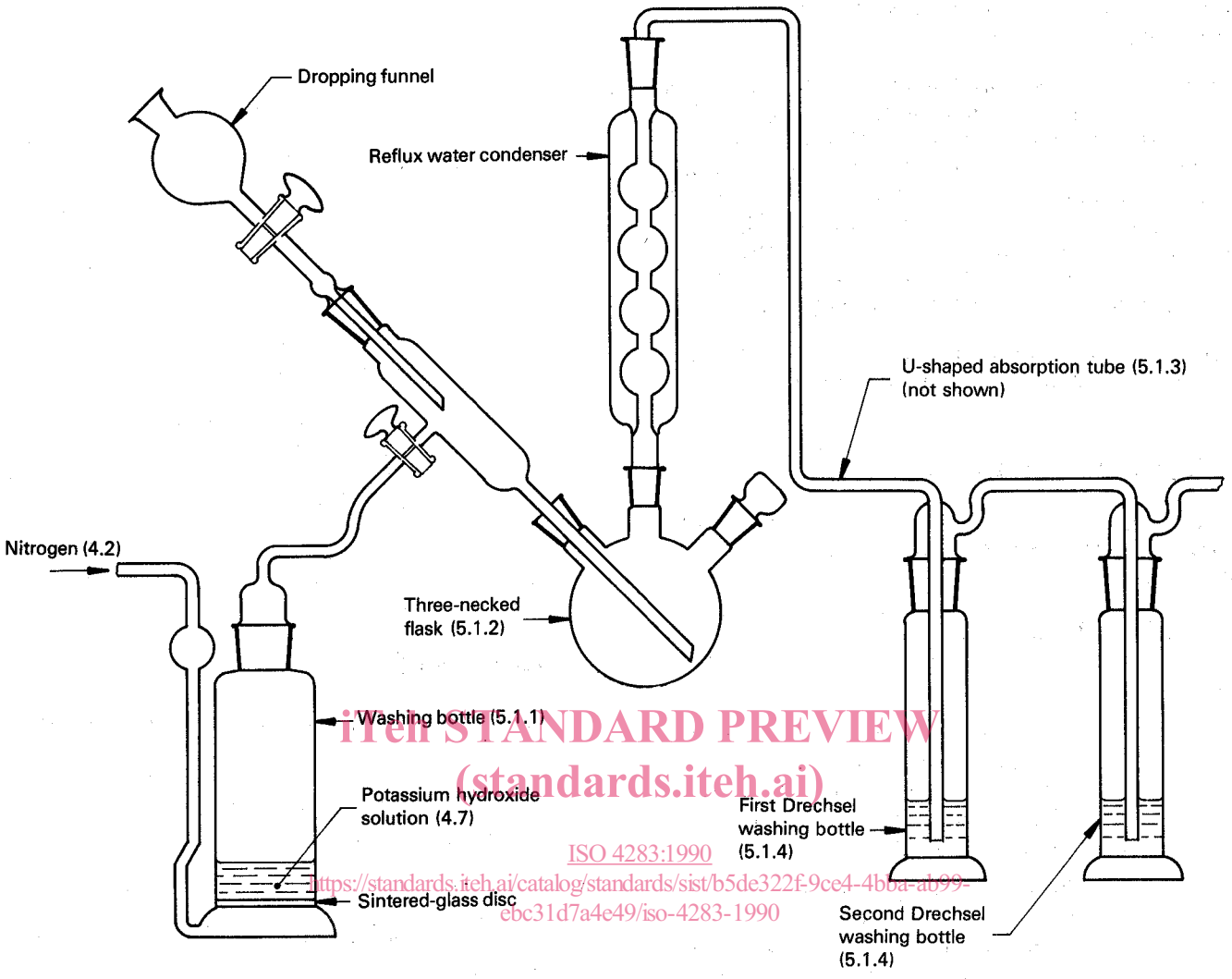

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Figure 1 — Typical gas evolution and absorption apparatus

Annex A
(informative)

Examples of test results

The calcium carbonate contents of acid-grade fluorspars, as obtained in tests on certified reference materials (SARM 14 and SARM 15), are given by way of example in table A.1.

Table A.1 — Results of tests on certified reference materials

$CaCO_3$ [% (m/m)]

Sample	Certified value	Results obtained using the method specified in this International Standard
SARM 14	0,34	0,32; 0,30
SARM 15	1,60	1,58; 1,51; 1,57

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