INTERNATIONAL STANDARD 3425

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

Sulphur for industrial use — Determination of ash at 850-900 °C and of residue at 200 °C

Soufre à usage industriel — Détermination des cendres à 850-900 °C et du résidu à 200 °C

First edition — 1975-10-15

UDC 661.21 : 543.822

Ref. No. ISO 3425-1975 (E)

Descriptors : sulphur, chemical analysis, determination of content, ashes, residues.

Price based on 2 pages

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 3425 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in March 1974.

It has been approved by the Member Bodies of the following countries :

Austria	Hungary	South Africa, Rep. of
Belgium	India	Spain
Bulgaria	Ireland	Switzerland
Chile	Israel	Thailand
Czechoslovakia	Italy	Turkey
Egypt, Arab Rep. of	New Zealand	United Kingdom*
France	Portugal	U.S.S.R.
Germany	Romania	Yugoslavia

* with the exception of clause 3.

No Member Body expressed disapproval of the document.

Printed in Switzerland

Sulphur for industrial use — Determination of ash at 850-900 °C and of residue at 200 °C

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the ash at 850-900 °C and a method for the determination of residue at 200 °C of sulphur for industrial use.

The methods specified are applicable to sulphur for industrial use containing at least 98 % of elemental sulphur S expressed as a percentage by mass of the dry product, i.e. of the product dried at 80 °C for 2 h.

NOTE — The difference between the ash at 850-900 °C and the residue at 200 °C, expressed as percentages by mass of the dry product, corresponds to the "non-volatile bituminous matter" content at 200 °C.

2 DETERMINATION OF ASH AT 850-900 °C

2.1 Principle

Slow combustion in air of a test portion of dry sulphur. Heating in a furnace at 850-900 °C followed by weighing.

2.2 Apparatus

Ordinary laboratory apparatus and

2.2.1 Silica dish, tall-form.

2.2.2 Hot-plate (or, failing this, a Bunsen burner and refractory triangle).

2.2.3 Electric furnace, capable of being controlled at 850-900 °C.

2.3 Sampling and preparation of test sample

Follow the procedure specified in ISO . . .[1)]

Prepare a test sample from the laboratory sample by reducing the particle size, if necessary, to less than 2 mm and then drying in an oven at 80 °C for 2 h.

2.4 Procedure

2.4.1 Test portion

Weigh, to the nearest 0,001 g, about 50 g of the test sample (2.3) into the dish (2.2.1), previously heated at 850-900 °C, cooled in a desiccator and weighed to the nearest 0,001 g.

2.4.2 Determination

Place the dish containing the test portion (2.4.1) on the hot-plate (2.2.2) (or on a refractory triangle over a Bunsen burner) and gently melt the sulphur. When melting is complete, set fire to the sulphur with a small gas flame (for example, from a Bunsen burner). Adjust the heating rate so that the sulphur burns slowly (temperature just sufficient to maintain combustion) and completely (combustion period : 3 to 4 h).

Heat the dish and the residue from the combustion of the sulphur in the furnace (2.2.3), controlled at 850-900 °C. Allow to cool in a desiccator and weigh to the nearest 0,001 g. Replace the dish in the furnace and repeat the previous operations until two successive weighings do not differ by more than 0,005 g.

2.5 Expression of results

The ash, expressed as a percentage by mass of the dry product, is given by the formula

$$\frac{m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the residue after heating.

Express the result to three decimal places.

2.6 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.

1) In preparation.

3 DETERMINATION OF RESIDUE AT 200 °C

3.1 Principle

Slow evaporation, at 200 °C in a nitrogen atmosphere, of the volatile matter and the sulphur present in a test portion, followed by weighing of the residue.

3.2 Apparatus

Ordinary laboratory apparatus and

3.2.1 Shallow vessel, surface area about 50 cm^2 (for example, porcelain combustion boat, 80 mm × 60 mm).

3.2.2 Electric oven, capable of being controlled at 200 ± 10 °C.

3.2.3 Nitrogen cylinder, fitted with a reduction valve.

3.2.4 Gas-washing bottle, 200 ml capacity, three-quarters filled with sulphuric acid (ρ approximately 1,84 g/ml).

3.3 Sampling and preparation of test sample

See 2.3.

3.4 Procedure

3.4.1 Test portion

Weigh, to the nearest 0,000 2 g, about 2 g of the test sample (3.3) into the shallow vessel (3.2.1), previously dried at 200 °C, cooled in a desiccator and weighed to the nearest 0,000 2 g.

3.4.2 Determination

Pass a current of nitrogen into the oven (3.2.2), controlled at 200 ± 10 °C. For this purpose, a glass tube may be fitted to the air intake at the bottom of the oven and connected by rubber tubing to the gas-washing bottle (3.2.4), and then to the reduction valve of the nitrogen cylinder (3.2.3). Adjust the nitrogen flow rate to 2 to 3 bubbles per second.

Place the vessel containing the test portion (3.4.1) in the oven (3.2.2), controlled at 200 ± 10 °C, and leave until constant mass is reached, which will require about 10 h. Remove the vessel from the oven, allow it to cool in a desiccator and weigh it to the nearest 0,000 2 g. Replace the vessel in the oven and repeat the previous operations until two successive weighings do not differ by more than 0,001 g.

3.5 Expression of results

The residue at 200 °C, expressed as a percentage by mass of the dry product, is given by the formula

$$\frac{m_3}{m_2} \times 100$$

where

m_2 is the mass, in grams, of the test portion;

m_3 is the mass, in grams, of the residue at 200 °C.

3.6 Test report

See 2.6.

ANNEX

ISO PUBLICATIONS RELATING TO SULPHUR FOR INDUSTRIAL USE

ISO 2866 – Determination of total carbon content – Titrimetric method.

ISO 3425 – Determination of ash at 850-900 °C and of residue at 200 °C.

ISO 3426 – Determination of loss in mass at 80 °C.

ISO 3704 – Determination of acidity – Titrimetric method.

ISO 3705 – Determination of arsenic content – Silver diethyldithiocarbamate photometric method.