INTERNATIONAL STANDARD ISO 3705

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Sulphur for industrial use — Determination of arsenic content — Silver diethyldithiocarbamate photometric method

Soufre à usage industriel — Dosage de l'arsenic — Méthode photométrique au diéthyldithiocarbamade d'argent

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FOREWORD

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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 3705 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the Member Bodies in January 1975.

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

Austria	Ireland	Switzerland
Belgium	Israel	Turkey
Brazil	Poland	United Kingdom
Bulgaria	Portugal	U.S.S.R.
France	Romania	Yugoslavia
Germany	South Africa, Rep. of	
India	Spain	

No Member Body expressed disapproval of the document.

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Sulphur for industrial use — Determination of arsenic content — Silver diethyldithiocarbamate photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a silver diethyldithiocarbamate photometric method for the determination of the arsenic content of sulphur for industrial use.

The method is applicable to products having arsenic contents equal to or greater than 0,5 mg/kg.

2 REFERENCE

ISO 2590, *General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method.*

3 PRINCIPLE

Dissolution of a test portion in carbon tetrachloride. Oxidation by bromine and nitric acid.

Determination of the arsenic content by the method specified in ISO 2590.

4 REAGENTS

See clause 4 of ISO 2590.

However, replace reagent 4.1 (hydrochloric acid) by

4.1 Sulphuric acid, approximately 15 N solution.

Also, add the following reagents :

4.9 Bromine solution containing 3 volumes of carbon tetrachloride and 2 volumes of bromine.

4.10 Nitric acid, ρ approximately 1,40 g/ml, about 68 % (*m*/*m*) solution.

5 APPARATUS

See clause 5 of ISO 2590.

6 SAMPLING AND PREPARATION OF TEST SAMPLE

Proceed in accordance with the appropriate International Standard.[1)]

Prepare a test sample from the laboratory sample by drying a sufficient quantity for 2 h in an oven maintained at about 80 °C. After cooling in a desiccator, grind until fine enough to pass through a sieve of nominal aperture 630 μm.

7 PROCEDURE

WARNING — Because of the toxicity and unpleasant odour of pyridine, it is recommended that it should be handled with care and in a well-ventilated fume cupboard.

7.1 Test portion and preparation of test solution

Weigh, to the nearest 0,1 g, about 5 g of the test sample (clause 6) and place in a beaker of suitable capacity (for example 400 ml).

In a well-ventilated fume cupboard, add 20 ml of the bromine solution (4.9). Allow to stand for 45 min and then add, drop by drop, while swirling gently, 25 ml of the nitric acid solution (4.10). During this operation, cool the beaker in an ice/water mixture to prevent a too rapid evolution of nitrous fumes. If the oxidation of sulphur is incomplete, the operation may be repeated using a few millilitres of the bromine solution (4.9) and of the nitric acid solution (4.10).

Heat in a boiling water bath to eliminate the excess bromine, carbon tetrachloride and nitric acid. If the solution is not clear, allow to cool, add a little of the nitric acid solution (4.10), and evaporate until no further nitrous fumes are evolved. Add a little water and evaporate again on a sand bath until white sulphuric acid fumes are evolved. Repeat this operation and fume for 10 min to remove the last traces of nitrous compounds. Allow to cool.

Dilute to about 80 ml with water and allow to cool.

1) Under study.