
International Standard



601

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

ANSI Interior Div Sect

Solid mineral fuels — Determination of arsenic content using the standard silver diethyldithiocarbamate photometric method of ISO 2590

Combustibles minéraux solides — Détermination de la teneur en arsenic utilisant la méthode photométrique au diéthylthiocarbamate d'argent normalisée dans l'ISO 2590

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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 601 was developed by Technical Committee ISO/TC 27, *Solid mineral fuels*, and was circulated to the member bodies in November 1979.

It has been approved by the member bodies of the following countries :

Australia	Germany, F. R.	South Africa, Rep. of
Austria	India	United Kingdom
Belgium	Korea, Rep. of	USA
Bulgaria	Netherlands	USSR
Canada	Poland	Yugoslavia
Denmark	Romania	

The member body of the following country expressed disapproval of the document on technical grounds :

Czechoslovakia

This International Standard cancels and replaces ISO Recommendation R 601-1967, of which it constitutes a technical revision.

Solid mineral fuels — Determination of arsenic content using the standard silver diethyldithiocarbamate photometric method of ISO 2590

1 Scope and field of application

This International Standard specifies a method for the photometric determination of amounts of arsenic in hard coal, brown coal and lignite and coke using the silver diethyldithiocarbamate method in accordance with ISO 2590.

The method is applicable to the determination of arsenic (As) contained either in all the test solution or in the aliquot portion taken for the determination of between 1 and 20 µg corresponding to not less than 1 ppm in the test portion.

2 References

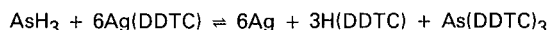
ISO 383, *Laboratory glassware — Interchangeable conical ground joints*.

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

3 Principle

Oxidation of the sample by nitric and sulphuric acids or by the use of Eschka mixture. Then, using the method specified in ISO 2590, reduction of the arsenic by zinc in sulphuric acid or hydrochloric acid medium with the formation of arsine and absorption of the arsine in a solution of silver diethyldithiocarbamate in pyridine, followed by photometric measurement of the purplish-red colour produced by the colloiddally dispersed silver at the maximum of the absorption curve (wavelength approximately 540 nm).

NOTE — The reaction of the formation of the colloidal silver is :



4 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent quality. All the reagents and the zinc in particular shall be free from arsenic or have a very low arsenic content. In addition to the reagents specified in ISO 2590, clause 4, the following are required.

4.1 Sulphuric acid, concentrated, ρ 1,84 g/ml, (for 7.2).

4.2 Nitric acid, concentrated, ρ 1,42 g/ml, (for 7.2).

4.3 Hydrochloric acid, ρ 1,18 g/ml, (for 7.1).

4.4 Eschka mixture (for 7.1).

Mix two parts by mass of light calcined magnesium oxide with one part of anhydrous sodium (or potassium) carbonate. The mixture shall entirely pass a test sieve of 212 µm nominal aperture.

5 Apparatus

All glass apparatus shall be constructed from borosilicate glass. Ground glass joints, when used, shall comply with the requirements specified in ISO 383. The balance used shall be accurate to 0,1 mg.

In addition to the apparatus specified in ISO 2590, clause 5, the following is required :

5.1 Digestion apparatus (see the figure) (for 7.2), consisting of the following :

5.1.1 Kjeldahl flask, of capacity 300 ml, fitted with a 24/29 ground glass socket complying with the requirements of ISO 383.

5.1.2 Fume ducts, of outside diameter approximately 28 mm, fitted with a dropping funnel of capacity at least 15 ml and a 24/29 ground glass cone, complying with the requirements of ISO 383. The fume ducts may be of one piece or assembled from separate units by means of ground glass joints.

5.1.3 Fume extractor, consisting of a glass tube, of diameter approximately 40 mm, sealed at one end and drawn out at the other end to form a connection to the water pump. The tube is fitted with a drain stopcock and a series of lipped holes to accommodate a number of fume ducts.

5.1.4 Glass water pump.

5.1.5 Digestion rack, fitted with several positions, each of which will accommodate a Kjeldahl flask held at an angle of 45°, with a holder for the fume extractor.

5.2 Muffle furnace (for 7.1), electrically heated, with a zone of substantially uniform temperature at 800 ± 25 °C. The ventilation through the muffle furnace shall be such as to give about five air changes per minute.