SLOVENSKI STANDARD
SIST ISO 351:1998

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Trdna fosilna goriva - Določ evanje celotnega ǵvepla - Metoda zgorevanja pri visoki temperaturi

Solid mineral fuels -- Determination of total sulfur -- High temperature combustion method

Combustibles minéraux solides -- Dosage du soufre total -- Méthode par combustion à haute température

Ta slovenski standard je istoveten z: **ISO 351:1996**

ICS:

73.040	Premogi	Coals
75.160.10	Trda goriva	Solid fuels

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INTERNATIONAL STANDARD

ISO 351

Third edition
1996-02-15

Solid mineral fuels — Determination of total sulfur — High temperature combustion method

Combustibles minéraux solides — Dosage du soufre total — Méthode par combustion à haute température

Reference number
ISO 351:1996(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 351 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 351:1984), which has been technically revised.

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Introduction

An alternative method to that specified in this International Standard is given in ISO 334:1992, *Solid mineral fuels — Determination of total sulfur — Eschka method*.

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Solid mineral fuels — Determination of total sulfur — High temperature combustion method

1 Scope

This International Standard specifies a method of determining the total sulfur content of hard coal, brown coal and lignite, and coke by high temperature combustion.

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 587:1981, *Solid mineral fuels — Determination of chlorine using Eschka mixture.*

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.*

3 Principle

A known mass of a sample of coal or coke is burnt in a stream of oxygen, in a tube furnace at a temperature of 1 350 °C. The acid gases formed (chlorine and oxides of sulfur) are absorbed in hydrogen peroxide and subsequently determined titrimetrically. A correction is made to take account of any chlorine liberated and a suitable combustion additive prevents the retention of sulfur in the ash.

4 Reactions

4.1 $SO_2 + H_2O_2 \rightarrow H_2SO_4$

4.2 $Cl_2 + H_2O_2 \rightarrow 2HCl + O_2$

4.3 $Na_2B_4O_7 + 2HCl + 5H_2O \rightarrow 2NaCl + 4H_3BO_3$

4.4 $Na_2B_4O_7 + H_2SO_4 + 5H_2O \rightarrow Na_2SO_4 + 4H_3BO_3$

4.5 $NaCl + Hg(OH)CN \rightarrow NaOH + HgClCN$

4.6 $2NaOH + H_2SO_4 \rightarrow Na_2SO_4 + 2H_2O$

5 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.

5.1 Aluminium oxide (alumina), finely divided.

5.2 Hydrogen peroxide, approximately 3 % (*m/m*) neutral solution.

Dilute 100 ml of 30 % (*m/m*) hydrogen peroxide solution to 1 litre with water and neutralize with the sodium tetraborate solution (5.4) using the mixed indicator (5.6).

5.3 Mercury(II) oxycyanide, saturated solution at 20 °C, approximately 45 g/l.

Saturate a suitable volume of water with mercury(II) oxycyanide by prolonged agitation, filter and neutralize the filtrate with the sulfuric acid (5.5), using bromothymol blue as an external indicator. Store in a dark glass bottle; do not keep for more than 4 d.

5.4 Sodium tetraborate, standard volumetric solution, $c(Na_2B_4O_7) = 0{,}025$ mol/l.

Dissolve 9,534 2 g of sodium tetraborate decahydrate in water and dilute to 1 litre. Mix thoroughly.

5.5 Sulfuric acid, standard volumetric solution, $c(H_2SO_4) = 0{,}012\ 5$ mol/l.

5.6 Mixed indicator solution.

5.6.1 Solution A

Dissolve 0,125 g of 2-(4-dimethylaminophenylazo)benzoic acid, sodium salt (methyl red) in 100 ml of water.

5.6.2 Solution B

Dissolve 0,083 g of 3,7-bis(dimethylamino)phenothiazine-5-ylium chloride (methylene blue) in 100 ml of water. Store in a dark bottle.

5.6.3 Mixed solution

Mix equal volumes of solution A and solution B. Store in a dark bottle. Discard the mixed solution after 1 week.

5.7 Oxygen, laboratory grade, of purity at least 99,6 % (*m/m*).

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

6 Apparatus

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

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

6.3 Furnace, capable of heating a tube, of approximately 28 mm external diameter over a length of 125 mm to 150 mm, to a temperature of 1 350 °C.

The furnace may be heated electrically, using either silicon carbide resistance rods (controlled by a variable transformer) or a resistance wire (controlled by a variable resistance).

6.4 Combustion tube, of approximately 28 mm external diameter, 3 mm wall thickness and 650 mm length, made of aluminous porcelain which is impervious to gases up to the working temperature (see figure 1).

A straight tube is most convenient and may be used in conjunction with an adapter of fused silica having a bell-shaped end, which gives a narrow clearance from the inner wall of the heated tube, and a heat-resistant stopper (acrylonitrile or chloroprene is suitable). Alternatively, the tube may have, a beak end, at the exit, with a tubulure to enable condensation products to be washed out after a test; or a straight tube of aluminous porcelain may be used in conjunction with a borosilicate glass adapter, having a cap-shaped end which fits onto the outer wall of the tube. In this case it will be necessary to lag the end of the combustion tube with a suitable heat-resistant mineral fibre to prevent condensation in the tube.

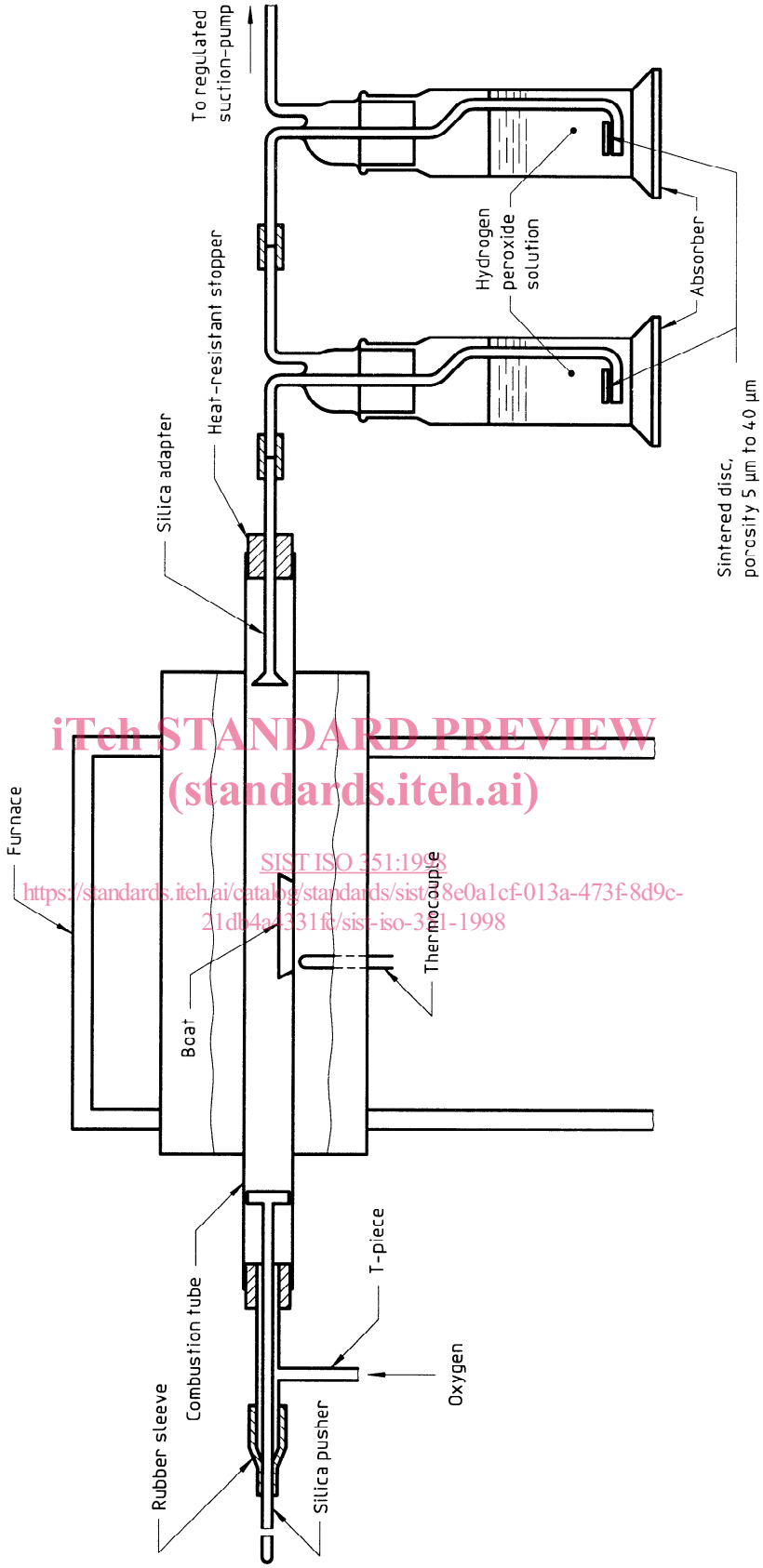

Figure 1 — Arrangement of furnace and absorption train