SLOVENSKI STANDARD
SIST EN 24260:1998

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Nadomešča:
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Naftni proizvodi in ogljikovodiki - Določevanje žvepla - Metoda s sežigom po Wickboldu (ISO 4260:1987)

Petroleum products and hydrocarbons - Determination of sulfur content - Wickbold combustion method (ISO 4260:1987)

Mineralölerzeugnisse und Kohlenwasserstoffe - Bestimmung des Schwefelgehaltes - Verbrennung nach Wickbold (ISO 4260:1987)

Produits pétroliers et hydrocarbures - Dosage du soufre - Méthode de combustion Wickbold (ISO 4260:1987)

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ICS:

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English version

Petroleum products and hydrocarbons - Determination of sulfur content - Wickbold combustion method (ISO 4260:1987)

Produits pétroliers et hydrocarbures - Dosage du soufre - Méthode de combustion Wickbold (ISO 4260:1987)

Mineralölerzeugnisse und Kohlenwasserstoffe - Bestimmung des Schwefelgehaltes - Verbrennung nach Wickbold (ISO 4260:1987)

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Foreword

This European Standard has been taken over by the Technical Committee CEN/TC 19 "Methods of test and specifications for petroleum products" from the work of ISO/TC 28 "Petroleum products and lubricants" of the International Organization for Standardization (ISO).

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 1994, and conflicting national standards shall be withdrawn at the latest by September 1994.

In accordance with the CEN/CENELEC Internal Regulations, following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

Endorsement notice

The text of the International Standard ISO 4260:1987 has been approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

ISO 4260

First edition
1987-04-01

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

Petroleum products and hydrocarbons — Determination of sulfur content — Wickbold combustion method

Produits pétroliers et hydrocarbures — Dosage du soufre — Méthode de combustion Wickbold

Reference number
ISO 4260 : 1987 (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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4260 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants.*

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Printed in Switzerland

Petroleum products and hydrocarbons — Determination of sulfur content — Wickbold combustion method

CAUTION — The procedure specified in this International Standard includes the combustion of hydrogen in glass apparatus or stainless steel apparatus (in the case of olefins), which is potentially hazardous, and all precautions should be carefully observed.

1 Scope and field of application

This International Standard specifies a method for the determination of total sulfur in petroleum products, natural gas and olefins. The method may be applied to products having sulfur contents in the range 1 to 10 000 mg/kg and is particularly suitable for distillates with total sulfur contents of less than 300 mg/kg. Test samples which are viscous, highly aromatic, or of high sulfur content may be first diluted with a sulfur-free solvent.

The method can be used for the determination of the total sulfur content of natural and refinery gases, also for substances supplied to the burner in the liquid state and for the determination of volatile sulfur in substances supplied to the burner in the gaseous state after vaporization from the liquid phase. It is not suitable for the determination of sulfur in heavy-duty engine oils. For the determination of sulfur in light olefins, see clause 13, special case.

NOTES

1 If required, total chlorine content of petroleum products can be determined by the usual volumetric, gravimetric or potentiometric methods for determination of the chloride ions present in the absorption solution after combustion by this method.

The inorganic bound chlorine has to be removed by water extraction prior to the burning procedure, otherwise interference will occur.

2 When viscous or solid materials, such as bitumen or heavy fuel oils, are burnt in a combustion boat, some of the sulfur may be bound to the ash retained in the boat. If this is the case, the sulfur bound in the ash has to be determined in the residue.

2 References

ISO 641, *Laboratory glassware — Interchangeable spherical ground joints*.

ISO 3170, *Petroleum products — Liquid hydrocarbons — Manual sampling*.

ISO 3171, *Petroleum products — Liquid hydrocarbons — Automatic pipeline sampling*.

ISO 4850, *Personal eye-protectors for welding and related techniques — Filters — Utilisation and transmittance requirements*.

3 Principle

Gaseous or liquid test portions are passed to the oxy-hydrogen flame of a suction burner where they are burnt with considerable excess of oxygen. Viscous or solid test samples are preferably dissolved in light petroleum/toluene blend and treated as liquid test samples or may be burnt in a stream of oxygen in a combustion boat.

The resulting sulfur oxides are converted into sulfuric acid by absorption in hydrogen peroxide solution. Depending on the sulfur content of the test portion, the sulfate ions in the absorption solution are determined using the method of analysis shown in table 1 and set out in clause 9.

Table 1 — Relation between expected sulfur content, mass of test portion, and method of analysis recommended

Expected sulfur content mg/kg	Mass of test portion[1] g	Sulfur in absorption solution µg	Aliquot portion of absorption solution	Sulfur in the aliquot portion µg	Method of analysis recommended for different levels of sulfur content		
	100	100	1/2	50	Conductimetric titration (9.4)	Nephelometric titration (9.2)	Turbidimetric titration (9.3)
	50	50	1/1	50			
1	20	20	1/1	20			
5	20	100	1/2	50			
	50	250	1/5	50			
	50	250	1/1	250			
10	5	50	1/1	50			
	10	100	1/2	50	Visual titration (9.1)		
	20	200	1/2	100			
	50	500	1/2	250			
30	5	150	1/2	75			
	10	300	1/2	150			
	20	600	1/2	300			
	50	1 500	1/5	300			
50	5	250	1/2	125			
	10	500	1/2	250			
	30	1 500	1/5	300			
100	2	200	1/2	100			
	5	500	1/2	250			
	10	1 000	1/5	200			
1 000	1	1 000	1/5	200			
	2	2 000	1/5	400			
10 000	1	10 000	1/10	1 000			

1) The volume of a gas test sample required may be calculated with sufficient accuracy from the mass and density of the gas. The precision data in clause 12 do not apply for gas test samples.

4 Reagents and materials

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

Volumetric solutions and other reagents as specified in clause 9 under the individual method of analysis and

4.1 Hydrogen peroxide, 3 % (*m*/*m*) solution, free from sulfur.

4.2 Ethanol, 96 % (*V*/*V*), free from sulfur.

4.3 Blend containing 4 volumes of light petroleum, (boiling range 60 to 80 °C) and 1 volume of toluene, free from sulfur (in the following text referred to as "light petroleum/toluene blend").

4.4 Oxygen, compressed gas, commercial grade, free from sulfur.

4.5 Hydrogen, compressed gas, commercial grade, free from sulfur.

4.6 Hydrochloric acid, concentrated, ϱ_{20} 1,19 g/ml.

4.7 Mercury.

4.8 Dihexyldisulfide or dibenzothiophene reference blend.

Dissolve a known mass of dihexyldisulfide or dibenzothiophene, weighed to the nearest 0,1 mg, in the light petroleum/toluene blend (4.3). The concentration of the product should be chosen from the sulfur concentration in the range shown in column 1 of table 1 according to the finish which will be used.

Dihexyldisulfide contains 27,36 % (*m*/*m*) of sulfur, and dibenzothiophene contains 14,7 % (*m*/*m*) of sulfur.

CAUTION — It is recommended that high-pressure gas cylinders are not stored in the laboratory.

5 Apparatus

NOTE — In order to provide a detailed description of the mode of operation, this International Standard has been based on two types of burners and a single type of absorption train.

Other types of burner using the principle of combustion of a test portion in an oxy-hydrogen flame with excess oxygen may be used (see clause 13). These should be operated as described in the manufacturers' instructions and checked by the combustion of test portions of the standard sulfur-containing reference blends (see clause 10).

Apparatus specified in clause 9 under individual method of analysis and

5.1 Combustion apparatus

5.1 Combustion apparatus (see figure 1 for the schematic layout of the apparatus for the combustion of gaseous or liquid test samples and figure 2 for that for the combustion of viscous or solid test samples), consisting essentially of the following components:

5.1.1 Reducing valve (1), with gauge, range 0 to 5 bar (0 to 500 kPa)[1)], for oxygen.

5.1.2 Reducing valve (2), with gauge, range 0 to 2 bar (0 to 200 kPa), for hydrogen.

5.1.3 Flowmeter (3), range 20 to 300 l/h, operating on the floating element principle, with precision control valve, for the secondary oxygen line.

5.1.4 Flowmeter (4), range 20 to 200 l/h, operating on the floating element principle, with precision control valve, for the hydrogen line.

5.1.5 Flowmeter (5), range 200 to 2 000 l/h, operating on the floating element principle, with precision control valve, for the primary oxygen line.

5.1.6 Excess-pressure vessels (6), for example wash bottles, containing mercury (4.7) and white oil (see 8.2), or metal pressure safety valves. Three are required. The inlet valves must be of the non-return type.

5.1.7 Flame trap (7), with metal connections.

5.1.8 Flow indicator (8), glass.

5.1.9 Vacuum gauge (11), range from approximately 0,6 to 1,1 bar (60 to 110 kPa) absolute.

5.1.10 Vacuum line (12), with vacuum valve (9), and branch line to vacuum gauge (11), and a branch line with stopcock (10) to the flow indicator (8).

5.1.11 Narrow-necked one-mark volumetric flask (13), 100 or 250 ml capacity, with spherical ground glass joint, ISO 641-S29/15.

5.1.12 Combustion chamber[2)] (20), of transparent fused quartz, **cooler (19), absorber tower (17), frit-type filter, pore size index 1,6 mm (16), drip chamber (15), three-way stopcock (14), and ground glass spherical joint,** ISO 641-S29/15, assembled into a single unit. The combustion chamber, cooler and absorber tower are enclosed in a water-cooled jacket.

5.1.13 Burner[2)] (21), of stainless steel or transparent fused quartz. The burner should be of the suction type (see figure 1) for the combustion of gaseous or liquid products. A stainless steel burner may be used as an alternative and such a burner shall be used for light olefins (see figure 3).

5.1.14 Stopcock (18), fitted in the line connecting the absorption solution flask and absorber tower (17).

5.1.15 Test portion container (22). The assembly of the combustion equipment shown in figure 1 uses a test portion container, for example a conical flask, approximately 100 ml capacity. (See also 8.6.1 to 8.6.4.) When testing highly volatile test samples, the conical flask should be enclosed by a vacuum jacketed vessel or other similar device. The flask is held in place by means of an adjustable support.

The assembly of the combustion equipment shown in figure 2 uses a combustion boat as the test portion container.

5.1.16 Gas sample meter. A dry gas meter connected to a precision type valve for measuring the quantity of test portion for gas samples shall be used, unless this is to be determined gravimetrically. The range of the meter shall be appropriate for the quantity of sample to be burned and the meter shall be recently calibrated.

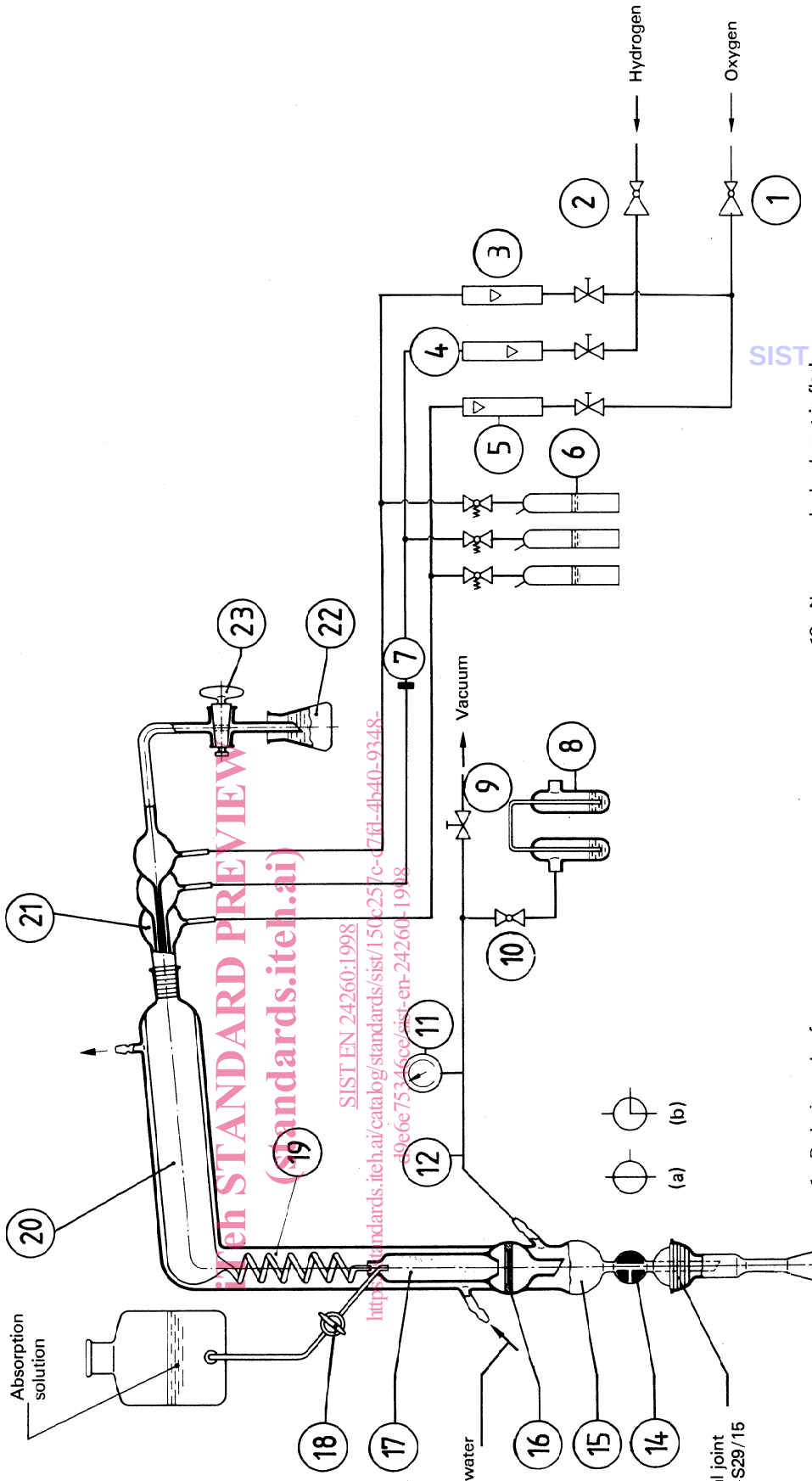
5.1.17 Connectors. For connecting the hydrogen and oxygen cylinders with flowmeters (3), (4), and (5), use high-pressure metal piping. The remaining piping in the system may be made of elastomers, such as silicone rubber.

5.1.18 Bunsen burner.

5.1.19 Analytical balance.

1) 1 Pa = 1 N/m^2 = 10^{-5} bar

2) The burner and combustion chamber as shown are claimed to be the subject of patents in some countries. Information on the patent position should be sought from local suppliers. However, most of these patents are unlikely to be still current.



1 Reducing valve for oxygen
2 Reducing valve for hydrogen
3 Flowmeter for secondary oxygen line
4 Flowmeter for hydrogen line
5 Flowmeter for primary oxygen line
6 Excess-pressure vessels
7 Flame trap
8 Flow indicator
9 Vacuum valve
10 Stopcock
11 Vacuum gauge
12 Vacuum line
13 Narrow-necked volumetric flask
14 Three-way stopcock for positions (a) and (b)
15 Drip chamber
16 Sintered glass filter
17 Absorber tower
18 Stopcock
19 Cooler
20 Combustion chamber
21 Suction-type burner
22 Test portion container
23 Stopcock

Figure 1 — Schematic layout of apparatus for combustion of gaseous or liquid test samples

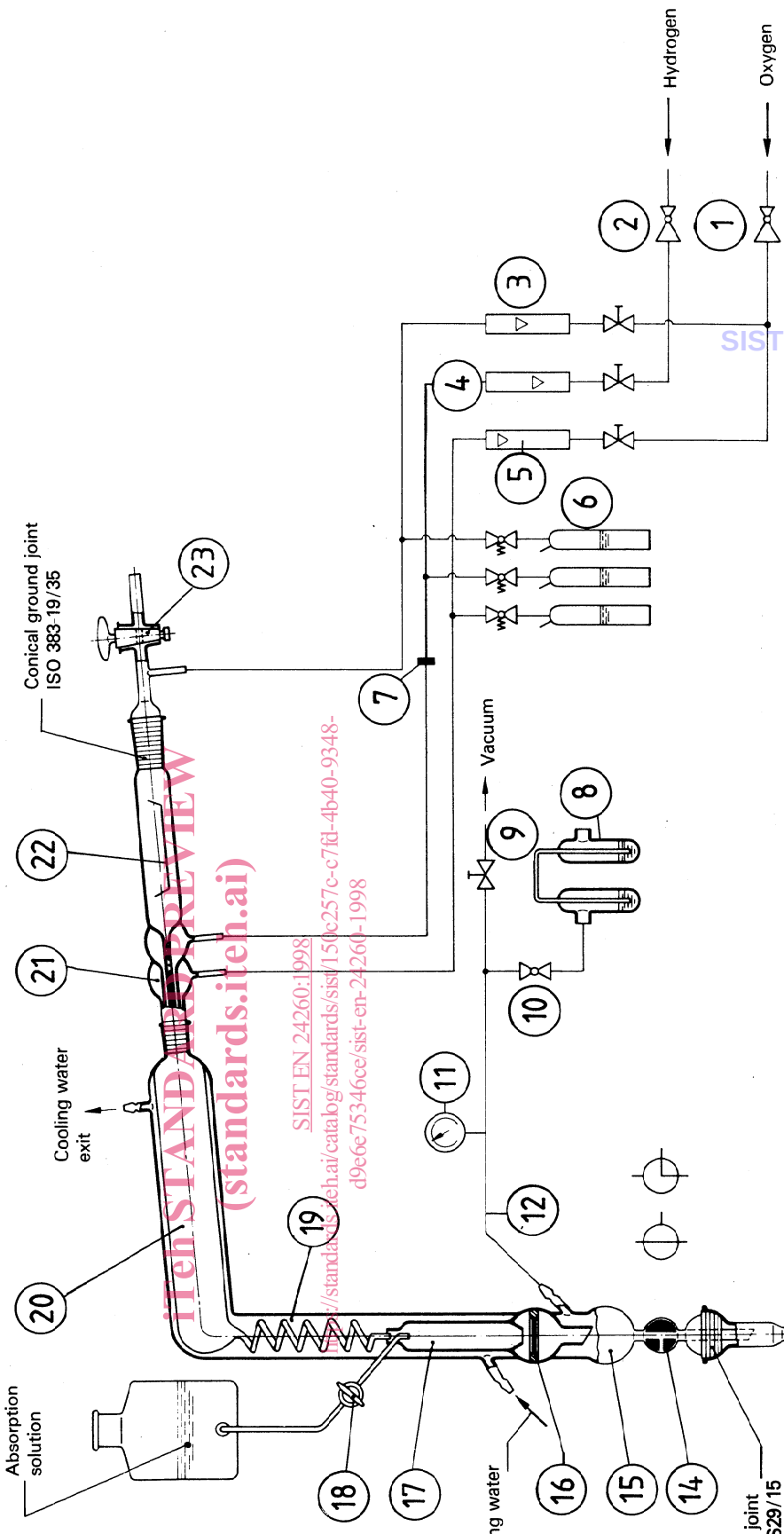


Figure 2 – Schematic layout of apparatus for combustion of viscous or solid test samples