

International Standard



2192

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

Petroleum products — Determination of total sulfur content — Lamp method

Produits pétroliers — Dosage du soufre total — Méthode à la lampe

Second edition — 1984-11-15

UDC 665.7 : 543.845

Ref. No. ISO 2192-1984 (E)

Descriptors : petroleum products, chemical analysis, determination of content, sulphur, apparatus, gravimetric analysis, volumetric analysis.



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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.

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International Standard ISO 2192 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

ISO 2192 was first published in 1973. This second edition cancels and replaces the first edition, of which it constitutes a technical revision.

Petroleum products — Determination of total sulfur content — Lamp method

1 Scope and field of application

1.1 This International Standard specifies a method for the determination of the total sulfur content of liquid petroleum products in concentrations above 0,002 % (*m/m*).

A special sulfate analysis procedure is described in annex A that permits the determination of sulfur in concentrations as low as 5 mg/kg.

In the basic procedure, sulfate is determined acidimetrically by titration with sodium hydroxide; an alternative gravimetric procedure (precipitation as barium sulfate) is described in annex B.

1.2 The direct burning procedure (see clause 7) is applicable to the analysis of such materials as gasoline, kerosine, naphtha, and other liquids that can be burned completely in a wick lamp.

The blending procedure (see clause 8) is applicable to the analysis of gas oils and distillate fuel oils, high sulfur content petroleum products, and many other materials that cannot be burned satisfactorily by the direct burning procedure. It is not suitable for heavy residues such as residual fuel oils, bitumens and tars.

1.3 Phosphorus compounds normally present in commercial gasoline do not interfere in the determination. A correction is given for the small amount of acid resulting from the combustion of the lead anti-knock fluids in gasolines.

Appreciable concentrations of acid-forming or base-forming elements from other sources interfere when the titration procedure is employed since no correction is provided in these cases.

2 Principle

2.1 Burning of a test portion in a closed system, using a suitable lamp and an artificial atmosphere composed of 70 % (*V/V*) carbon dioxide and 30 % (*V/V*) oxygen to prevent the formation of oxides of nitrogen. Absorption of the oxides of sulfur and oxidation to sulfuric acid by means of hydrogen peroxide solution which is then flushed with air to remove dissolved carbon dioxide. Determination of the sulfur as sulfate in the absorbent, either acidimetrically by titration

with standard sodium hydroxide solution, or gravimetrically by precipitation as barium sulphate (see annex B).

2.2 Alternatively, burning of the test portion in air, and gravimetric determination of the sulfur in the absorbent as sulfate by precipitation as barium sulfate (see annex B).

NOTE — In the absence of acid-forming or base-forming elements, other than sulfur, results by the volumetric and gravimetric finishes described are equivalent within the limits of precision of the method.

2.3 For sulfur contents below 0,002 % (*m/m*), it is necessary to determine the sulfate content in the absorbent solution turbidimetrically as barium sulfate (see annex A).

3 Reagents

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

3.1 Carbon dioxide (CO_2) and **oxygen** (O_2), both at least 99,5 % (*V/V*) pure and meeting the requirements specified in 7.5.

3.2 Diluent, having a sulfur content less than 0,001 % (*m/m*), completely miscible with the sample to be analysed, and permitting burning at a moderate rate without smoking. *n*-Heptane, *iso*-octane and absolute ethanol have been found suitable.

3.3 Hydrogen peroxide, solution (1 + 19).

Mix 1 volume of concentrated hydrogen peroxide [H_2O_2 , 30 % (*m/m*)] with 19 volumes of water.

Store in a dark-coloured glass-stoppered bottle.

3.4 Sodium hydroxide, 100 g/l solution.

Dissolve 100 g of sodium hydroxide (NaOH) in water and dilute to 1 l.

3.5 Sodium hydroxide, standard volumetric solution, $c(\text{NaOH}) = 0,05 \text{ mol/l}$.

Dilute 2,8 ml of saturated sodium hydroxide solution to 1 l (see note 1), using the clear saturated solution decanted after standing long enough to permit any precipitate to settle out.

Standardize by titration against a standard volumetric solution of acid, using the methyl purple indicator (3.6).

Store in an alkali-resistant glass bottle (see note 2) and protect to minimize contamination by carbon dioxide from the air.

Use only pure gum rubber tubing for connections between the storage bottles and burettes.

NOTES

1 The calculation of results may be simplified by adjusting the concentration of the sodium hydroxide solution to $0,0624 \pm 0,0001 \text{ mol/l}$. Then 1 ml of the sodium hydroxide solution will be equivalent to 0,0010 g of sulfur. In this case, the value of 1,603c in the calculation (10.1) becomes 0,1.

2 A bottle of a suitable plastics materials is permitted as an alternative to glass.

3.6 Methyl purple indicator, aqueous solution containing approximately 0,1 % active constituent.

NOTE — Methyl violet indicator is not suitable as an alternative to methyl purple.

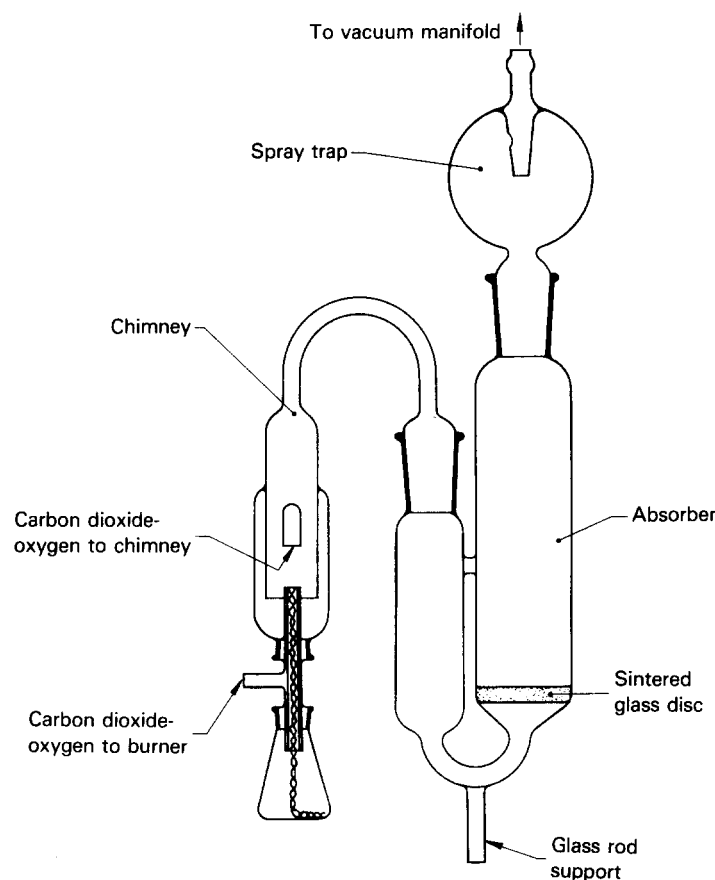


Figure 1 — Sketch of the assembled lamp unit

4 Apparatus

4.1 Lamps, chimneys, absorbers and spray traps (see figure 1), as described in detail in annex C.

The standard flask and burner (see figure 4), as shown, is not suitable for burning highly aromatic mixtures without blending as described in clause 8. The flask and burner for aromatic samples (see figure 4) permits burning these samples directly without blending and may also be used to burn non-aromatic samples; with this lamp, a second port with control valve in the burner manifold is required.

4.2 Cotton wicking. Clean, unused, uniform, twisted white cotton yarn of good quality.¹⁾ To enable the burner to burn aromatic samples use long staple, fine spun, commercial fine grade cotton.

4.3 Manifold system, consisting of a vacuum manifold with regulating device, valves, etc. (see figure 2) and a dual manifold (burner and chimney) supplying a gas mixture of approximately 70 % (V/V) carbon dioxide (CO_2) and 30 % (V/V) oxygen (O_2) at regulated pressures. The vacuum manifold shall be connected to a pump of sufficient capacity to permit a steady gas

flow of about 3 l/min through each absorber and to maintain a constant manifold pressure of approximately 4 kPa (40 mbar) below atmospheric. The gas mixture in the chimney manifold shall be maintained at a nearly constant pressure of 100 to 200 Pa (1 to 2 mbar), and the burner manifold at approximately 2 kPa (20 mbar). A suitable arrangement is shown in figure 2 and described in annex C, but any other similar system may be used. Modifications of the manifold and associated equipment for burning samples in air are described in annex B and shown in figure 3.

5 Preparation of apparatus

5.1 When the apparatus is first assembled, charge the absorber with $30 \pm 2 \text{ ml}$ of water. Adjust the individual valves between the vacuum manifold and spray traps so that approximately 3 l/min of air will be drawn through each absorber when the chimney outlets are open to the atmosphere, while maintaining the pressure in the vacuum manifold at approximately 4 kPa (40 mbar) below atmospheric. When all adjustments have been made, remove the water from the absorbers. The height of the liquids in the pressure and vacuum regulators shall be as indicated in figure 2, and during operation a slow leak of gas shall be maintained through them.

1) Rug yarn, white, 4-strand (2 or 3 mg/cm per strand), or equivalent material has been found satisfactory for this purpose.

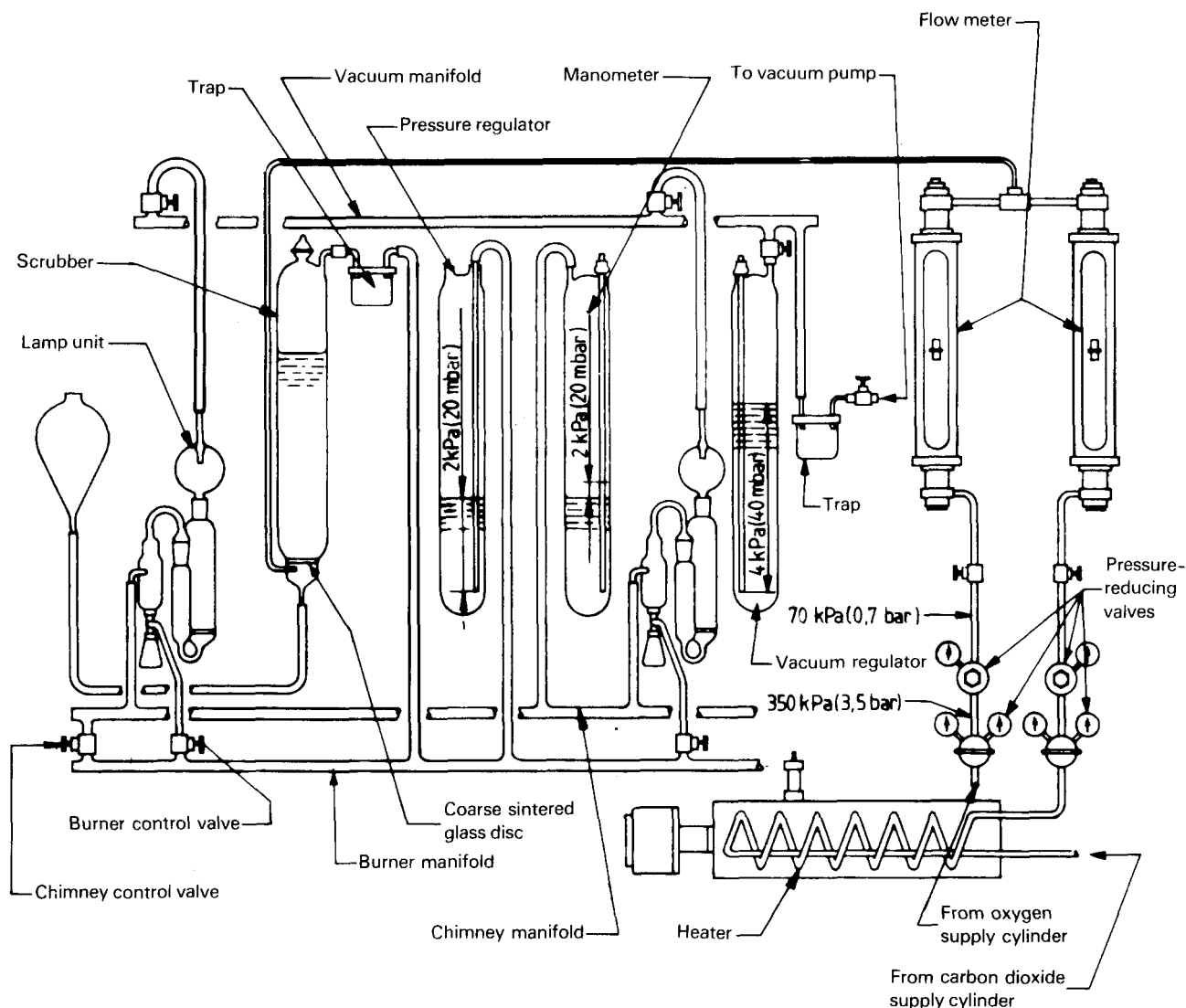


Figure 2 — Schematic diagram of carbon dioxide-oxygen supply manifold and lamp system

NOTE — In use, place 300 to 400 ml of hydrogen peroxide solution (3.3) in the scrubber. As the manifold manometer also serves as a scrubber at the end of the test to remove carbon dioxide from the absorbent, use the hydrogen peroxide solution (3.3) as the manometric liquid. Replace weekly or whenever the volume becomes appreciably less than the original.

5.2 Neutralize the hydrogen peroxide solution (3.3) immediately before use. As 30 ml of the solution is needed, transfer to a beaker a multiple of 30 ml sufficient for the number of absorbers to be used simultaneously. Add 1 drop of the methyl purple indicator solution (3.6) for each 100 ml of hydrogen peroxide solution and then add the sodium hydroxide standard volumetric solution (3.5) drop by drop until the colour changes from purple to light green.

5.3 Introduce 30 ± 2 ml of the freshly neutralized hydrogen peroxide solution (5.2) into the larger bulb of each absorber. In addition, for each set of test portions burned, prepare an extra

absorber for use as a control blank. Attach the spray traps and chimneys, and connect them to their respective manifolds by means of sulfur-free rubber tubing. Close the chimney openings by means of corks.

NOTE — Suitable sulfur-free rubber or plastics bungs are permitted as an alternative to corks.

5.4 With the burner control valves closed, the valve to the vacuum regulator fully open, and the pressure in the vacuum manifold adjusted to approximately 4 kPa (40 mbar) below atmospheric, turn on the carbon dioxide and oxygen supplies.

CAUTIONARY NOTE — A hazardous (explosive) condition may result if the carbon dioxide supply is interrupted and the oxygen flow is continued while test portions are being burned. The installation of suitable warning or control equipment is recommended.