
**Stationary source emissions —
Determination of mass concentration of
sulfur dioxide — Ion chromatography
method**

*Émission de sources fixes — Détermination de la concentration en masse
de dioxyde de soufre — Méthode par chromatographie ionique*

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ISO 11632:1998

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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. 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 11632 was prepared by Technical committee ISO/TC 146, *Air quality*, Subcommittee SC 1, *Stationary source emissions*.

Annex A of this International Standard is for information only.

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Introduction

For determining the mass concentration of sulfur dioxide in waste gas of stationary emissions sources, several integrated sampling and analysis methods exist. These include methods for independent manual sampling [ISO 7934] and for automated measuring systems [ISO 7935]. The latter have found a greater usage since a continuous measurement of sulfur dioxide, often specified by environmental authorities, can be obtained. The manual methods are required however, in calibrating automated measuring instruments.

This International Standard offers an alternative method to ISO 7934, replacing the Thorin method for analysing sulfate ions by a method based on ion chromatography. In addition, this International Standard is intended to apply to a lower emission range than ISO 7934.

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Stationary source emissions — Determination of mass concentration of sulfur dioxide — Ion chromatography method

1 Scope

This International Standard specifies a method for the determination of the mass concentration of sulfur dioxide emitted from combustion facilities and technical processes, and defines the most important performance characteristics.

The method described in this International Standard has been tested for a sulfur dioxide concentration range of 6 mg/m³ to 333 mg/m³ with sampling periods of 30 min. It is applicable to mass concentrations of sulfur dioxide exceeding this range by carrying out an appropriate dilution of the sample solutions prior to the analysis or by using larger volumes of absorption solution, and to sulfur dioxide concentrations below this range by extending the sampling period.

This International Standard is applicable to the analysis of samples containing negligible levels of sulfur trioxide and volatile sulfates (< 5 % of the expected sulfur dioxide concentration), and ammonia (< 5 mg/m³). All concentrations are based on dry gas at a temperature of 273,2 K and pressure of 101,3 kPa.

2 Normative references

ISO 11632:1998

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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 available valid International Standards.

ISO 6879:1995, *Air quality — Performance characteristics and related concepts for air quality measuring methods.*

ISO 7934:1989, *Stationary source emissions — Determination of the mass concentration of sulfur dioxide — Hydrogen peroxide/barium perchlorate/Thorin method.*

ISO 7935:1992, *Stationary source emissions — Determination of the mass concentration of sulfur dioxide — Performance characteristics of automated measuring methods.*

ISO 10396:1993, *Stationary source emissions — Sampling for the automated determination of gas concentrations.*

ISO Guide 25:1990, *General requirements for the competence of calibration and testing laboratories.*

3 Principle

A representative sample of waste gas is extracted via a temperature-controlled probe, filtered and drawn through a hydrogen peroxide solution for a specified time and flow rate. The sulfur dioxide in the waste gas sample is absorbed by the solution and sulfate anions are formed. The mass concentration of sulfate in the absorption solution is subsequently determined using ion chromatography.

4 Reagents

During the analysis, use only reagents of recognized analytical grade. The sulfate-free water shall have an electrical conductivity of $< 0,01$ mS/m and shall not contain particulate matter of a particle size $> 0,45$ μm . Normal, accepted laboratory safety practices should be followed during reagent preparation.

4.1 Absorption solution, 3 % H_2O_2

Pipette 100 cm^3 of a 27 % (mole fraction) to 30 % (mole fraction) solution of hydrogen peroxide (H_2O_2) into a 1000 cm^3 one-mark volumetric flask. Make up to the mark with water and mix well. Prepare this solution if possible on the day of use.

4.2 Eluent solution

The choice of eluent depends on the manufacturer's separator column and detector. For the exact composition of the eluent, refer to the instructions given by the manufacturer.

NOTE For an ion chromatograph using the suppressor technique, a typical eluent is a solution of $1,7 \times 10^{-3}$ mol/l NaHCO_3 and $1,8 \times 10^{-3}$ mol/l Na_2CO_3 .

4.3 Standard sulfate stock solution, $10,4 \times 10^{-3}$ mol/l SO_4^{2-}

Dissolve 1,8141 g of analytical grade potassium sulfate (K_2SO_4) in reagent water and dilute to 1 l using a 1000 cm^3 one-mark volumetric flask. 1 cm^3 of stock solution corresponds to 1 mg of SO_4^{2-} .

NOTE Standard sulfate stock solution is stable for at least 28 days when stored at 277 K. Calibration standards are prepared by diluting the standard stock solution with the absorption solution as specified in 7.4.2.

4.4 Regeneration solution for suppressor ISO 11632:1998

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For the exact composition of the suppressor regeneration solution, refer to the instructions given by the manufacturer of the suppressor.

NOTE An example is a solution of $12,5 \times 10^{-3}$ mol/l H_2SO_4 .

5 Apparatus

5.1 Sampling equipment

5.1.1 General

Alternative variations of the sampling equipment fulfilling the specified performance requirements for each component may be used. The performance characteristics set out in clause 9 refer, however, to the examples of sampling equipment described in 5.1.1 to 5.1.16. It is important that all parts of the sampling equipment upstream of the first absorber are heated and that the components shall not react with or absorb SO_2 .

NOTE In special cases an unheated gas connector line may be used between the heated filter and first absorber, but this must be thoroughly rinsed with absorption solution after sampling and the washings combined with the sample.

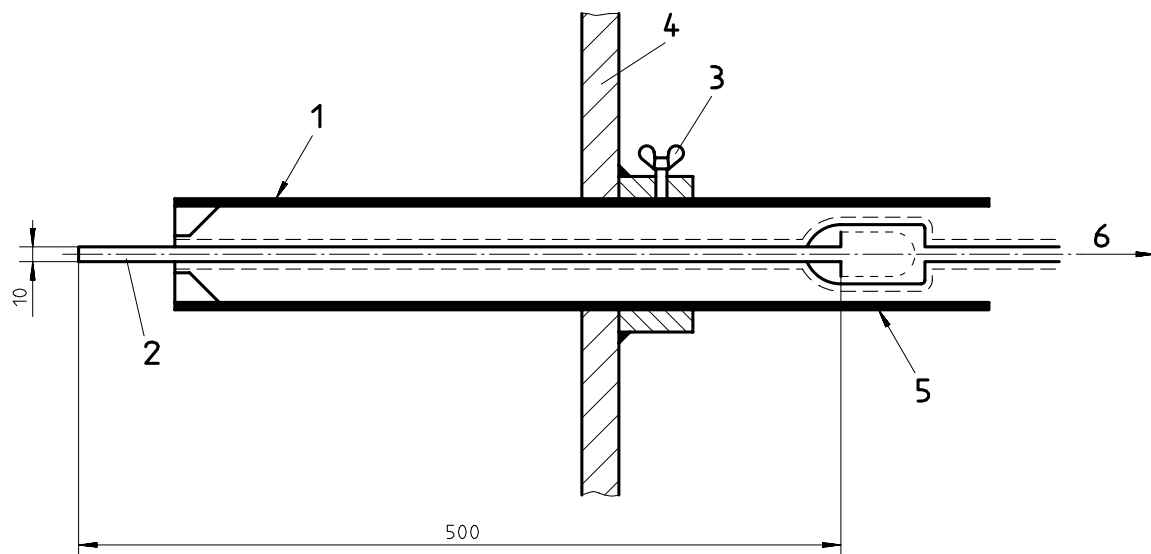
5.1.2 Sampling probe

The probe shall be of borosilicate glass or fused silica tube with a spherical ground joint at one end. Alternative probes of different lengths and inner diameters may be used, but the residence time of the sample gas in the probe shall be minimized. The sampling probe, which is surrounded by a heating jacket, shall be protected and positioned

using a metal outer tube. A clamp screw shall be used to adjust the probe length to reach the representative measurement point in the measurement plane of the waste gas duct.

NOTE An example of suitable probe dimensions is shown in figure 1.

Dimensions in millimetres



Key

- | | |
|---------------------------|------------------------------|
| 1 Protective metal tubing | 4 Waste gas duct wall |
| 2 Sampling probe | 5 Particle filter and holder |
| 3 Clamp screw | 6 To absorbers |

- - - indicates heated areas.

sampling probe length up to particle filter (A) 500 mm,

sampling probe diameter 10 mm,

protective metal tube inner diameter 80 mm to 100 mm

Figure 1 — Positioning of sampling probe, filter holder and protective tube

5.1.3 Filter holder

The filter holder shall be of borosilicate glass or fused silica glass with tube ends of spherical ground joints. The filter holder, which is encircled by the heating jacket, shall be connected to the sampling probe and housed within the protective metal tubing as shown in figure 1. The temperature after the filter holder is verified using a thermocouple.

NOTE 1 In special cases where the waste gas temperature is > 473 K, the heating jacket around the sampling probe, filter holder and connector line may be omitted. The temperature in the sampling line before the first absorber, however, should not fall below the acid dew point temperature of the waste gas.

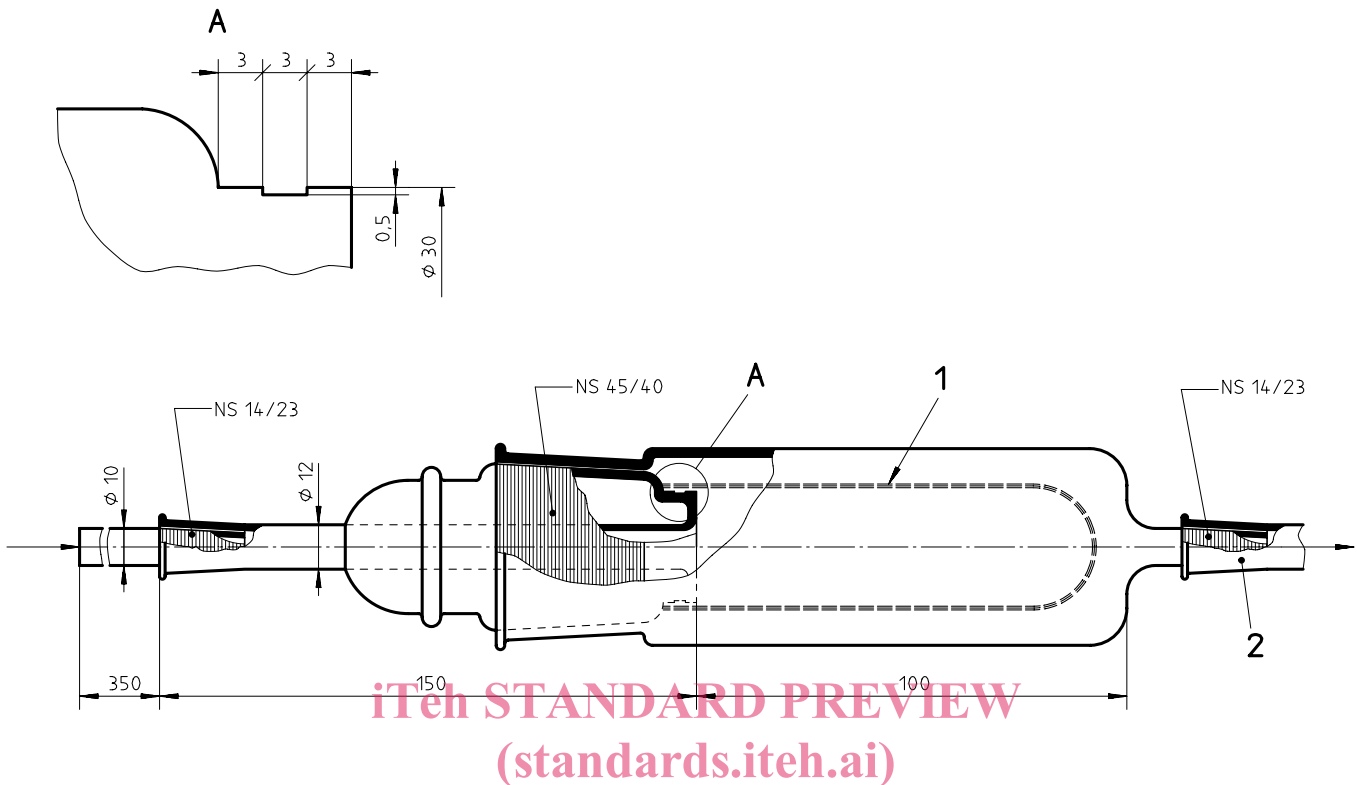
NOTE 2 An example of suitable filter holder dimensions is shown in figure 2.

NOTE 1 An example of a suitable quartz-fibre particle filter of the "thimble" variety is shown in figure 2. The filter is held in position in the filter holder by stainless steel wire.

NOTE 2 In certain cases, a check on the possibility of a reaction between SO_2 in the sampled gas and particles retained on the filter may be warranted. This can be performed by comparing sulfate analyses on the particle fractions obtained from (i) the

filter used when sampling according to this International Standard, and (ii) particles obtained from another source at the site, e.g. cyclone.

Dimensions in millimetres



- 1 Quartz fibre particle filter ('thimble variety') 90 mm × 30 mm inner diameter
- 2 Thermocouple

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Figure 2 — Example of a particle filter and filter holder

5.1.5 Absorbers

Two absorption bottles with spherical ground glass joints and equipped with an absorption bottle insert having a sintered filter. Alternative absorber sizes and configurations may be used, provided that the absorption efficiency criteria specified in 7.1 are met.

NOTE As an example, two 125 cm size absorption bottles of the Drechsel type with pore diameters in the sintered filter between 40 μm and 90 μm may be used (see figure 3).

5.1.6 Heating jacket

Heating jacket or bandage capable of producing a temperature of at least 473 K.

5.1.7 Temperature regulator

Temperature regulator suitable for use with the heating jacket or bandage.

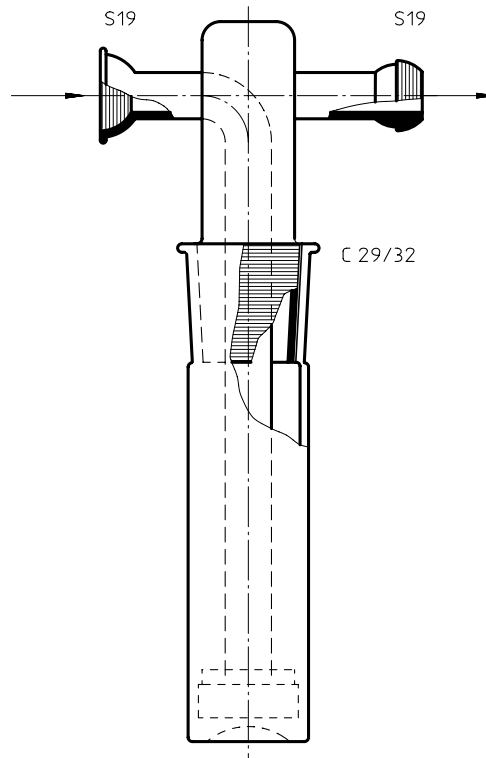


Figure 3 — Example of an absorption bottle
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5.1.8 Trap

Absorption bottle equipped with an absorption bottle insert not having a sintered filter. This bottle is connected after the second absorber and collects possible splashes of absorption solution.

5.1.9 Drying tube

Glass tube or absorption bottle packed with drying agent to dry the sample gas and protect the gas metering device and pump.

NOTE As an example, silica gel (1 mm to 3 mm particle size) previously dried at 448 K for at least 2 h may be used.

5.1.10 Sampling pump

Leak-free diaphragm pump capable of drawing sample gas at a flowrate within the range 0,02 m³/h to about 0,2 m³/h during the sampling period against a pressure of -10 kPa to -30 kPa. A small surge tank between the pump and rotameter may be used to eliminate the pulsation effect of the diaphragm pump on the rotameter.

5.1.11 Rotameter

Flowmeter or rotameter capable of measuring the selected sample gas flow with limits of error < ± 2 % of the upper limit of measurement.

5.1.12 Regulating valve

Needle valve capable of adjusting the sample gas flowrate within the range 0,02 m³/h to 0,2 m³/h.

5.1.13 Gas metering device

Dry-gas meter capable of use at a sample gas flowrate within the range 0,02 m³/h to about 0,2 m³/h, limits of error < ± 2 % of the measured volume, and equipped with a thermometer (5.1.14).

5.1.14 Connecting tubing

Connecting tubing in a range of lengths and internal diameters. All parts of the sampling train upstream of the first absorber shall be of a material which does not react with or absorb SO₂. The requirements are less stringent for parts of the sampling system downstream of the absorbers but corrosion resistant materials are recommended.

NOTE Examples of convenient materials are borosilicate glass, fused silica and polytetrafluoroethylene (upstream of the absorbers) and polyethylene and silicone rubber (downstream of the absorbers).

5.1.15 Thermometer

Thermometer with measuring range 268 K to 323 K, limits of error < ± 2 % of the upper limit of measurement.

5.1.16 Barometer

Barometer capable of measuring the atmospheric pressure (kPa) present at the sampling location, limits of error < ± 1 % of the upper limit of measurement.

5.1.17 Stopwatch

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5.2 Analysis equipment

5.2.1 Analytical balance

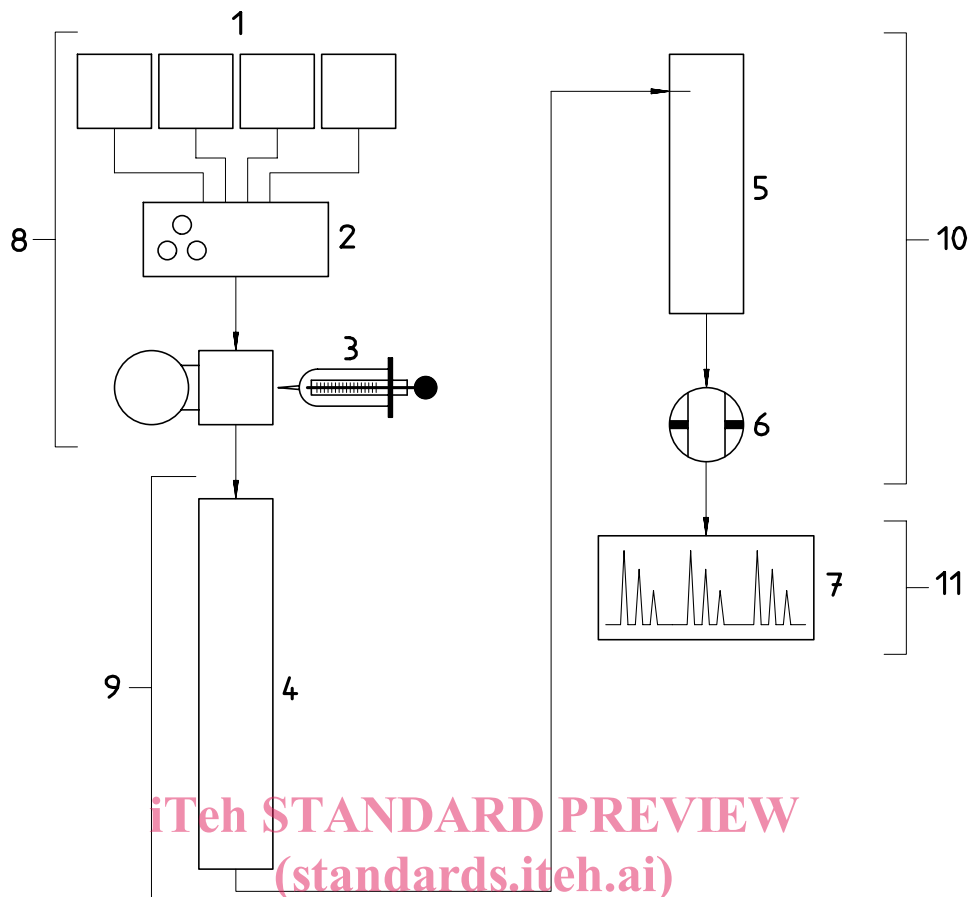
Analytical balance capable of weighing to the nearest 0,0001 g.

5.2.2 Ion chromatograph

Analytical system, complete with ion chromatograph and all required accessories including syringes, analytical columns, compressed gases, detector and recording device. The essential minimum requirements for an ion chromatograph system for the scope of this International Standard are as follows (see figure 4):

- Sample injection system.** An automated constant-volume injection system may be used.
- Anion separator column.** This column produced the separation of anions in the sample, enabling a clear measurement of the sulfate anion peak area and height. Other anions which may be present in the sample solution include F⁻, Cl⁻ and NO₃⁻. The resolution power of the separator column shall be sufficient so that the peak resolution (*R_s*) shall not fall below 1,3 where:

$$R_s = \frac{2(t_2 - t_1)}{(w_1 + w_2)}$$



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Key

1 Eluent reservoirs	7 Recorder, integrator, computer
2 Pump	8 Eluent/sample delivery
3 Sample injector	9 Analyte separation
4 Separator column	10 Analyte detection
5 Suppressor device	11 Data output
6 Detector cell	

Figure 4 — Example of an ion chromatograph

where

t_1 = retention time of the first peak, in seconds;

t_2 = retention time of the second peak, in seconds;

w_1 = peak width, in seconds on the time axis, of the first peak;

w_2 = peak width, in seconds on the time axis, of the second peak.

In general it is recommended to use a pre-column or anion guard column to protect the anion separator column. If this is omitted from the system, the retention times will be shorter. Two different types can be used: those containing the same substrate as the separator column, and those packed with a macroporous polymer.