SVENSK STANDARD
SS-ISO 7934:2020

Utsläpp från stationära källor – Bestämning av masskoncentrationen av svaveldioxid – Väteperoxid / bariumperklorat / Thorin (ISO 7934:1989, IDT)

Stationary source emissions – Determination of the mass concentration of sulfur dioxide – Hydrogen peroxide/barium perchlorate/Thorin (ISO 7934:1989, IDT)

Svenska Institutet för Standarder

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Standarden är framtagen av kommittén för Utsläpp, SIS/TK 423/AG 05.

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Den internationella standarden ISO 7934:1989 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av ISO 7934:1989.

The internationell standard ISO 7934:1989 has the status of a Swedish Standard. This document contains the official english version of ISO 7934:1989.

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 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 7934 was prepared by Technical Committee ISO/TC 146, *Air quality*.

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Stationary source emissions — Determination of the mass concentration of sulfur dioxide — Hydrogen peroxide/barium perchlorate/Thorin method

1 Scope

This International Standard specifies a hydrogen peroxide/barium perchlorate/Thorin [1] method for the determination of the mass concentration of sulfur dioxide emitted from combustion facilities and technical processes with negligible amounts of sulfur trioxide and sulfuric acid. It is applicable from a minimum of 30 mg/m^3 sulfur dioxide by reference to sampling periods of normally 30 min.

At mass concentrations of sulfur dioxide greater than 2 000 mg/m^3, the volume of the waste gas under investigation passed through the sampling train is 30 litres.

Substances, which, if contained in the waste gas under investigation and thus in the waste gas sample, are known to have an effect on the titration reading, are given in 7.4. Information on performance characteristics is given in 8.2.

At mass concentrations of sulfur dioxide less than 30 mg/m^3, a sampling period greater than that specified in this International Standard is used.

All concentrations are based on dry gas at a temperature of 273,1 K and a pressure of 101,3 kPa.

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 3696 : 1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 6879 : 1983, *Air quality — Performance characteristics and related concepts for air quality measuring methods.*

3 Principle

Absorption of the sulfur dioxide present in the waste gas sample passing through a hydrogen peroxide solution within a specified period, resulting in the formation of sulfuric acid solution.

Adjustment of the pH of the sample solution to pH 3,5 with sodium hydroxide solution or perchloric acid solution as required. Determination of the mass concentration of sulfate ions present in the treated sample solution by titration with a barium perchlorate solution using Thorin as indicator and calculation of the mass concentration of sulfur dioxide.

4 Reagents

During the analysis use only reagents of recognized analytical grade and only water of at least grade 3 purity according to ISO 3696.

WARNING — Use the reagents in accordance with the appropriate health and safety regulations.

4.1 Propan-2-ol [$CH_3CH(OH)CH_3$].

4.2 Absorption solution.

Place 100 ml of a 27 % (m/m) to 30 % (m/m) solution of hydrogen peroxide (H_2O_2) into a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix well.

Prepare this solution on the day of use.

4.3 Barium perchlorate, standard volumetric solution, $c[Ba(ClO_4)_2] = 0{,}005$ mol/l.

Use a commercially available barium perchlorate solution of defined concentration or, if this is not possible, prepare for example as follows.

1) Thorin is also known as Thoron or Thoronol, the sodium salt of 4-[(2-arsonophenyl)-azo]-3-hydroxy-2,7-naphthalene-disulfonic acid.

Dissolve 1,7 g of anhydrous barium perchlorate [$Ba(ClO_4)_2$] in about 200 ml of water in a 1 000 ml one-mark volumetric flask. Make up to the mark with propan-2-ol (4.1) and mix well.

Standardize this solution accurately by titration against a 0,005 mol/l standard volumetric sulfuric acid solution.

1 ml of exactly 0,005 mol/l barium perchlorate solution is equivalent to a mass of sulfur dioxide of 0,320 33 mg.

4.4 Sodium hydroxide, standard volumetric solution, $c(NaOH) = 0,1$ mol/l.

4.5 Perchloric acid, standard volumetric solution, $c(HClO_4) = 0,1$ mol/l.

4.6 Thorin, {4-[2-arsonophenyl)-azo]-3-hydroxy-2,7-naphthalene-disulfonic acid disodium salt} 2 g/l solution.

Dissolve 0,2 g of Thorin in water in a 100 ml one-mark volumetric flask. Make up to the mark with water and mix well.

Store this solution in a bottle made of fused silica or polyethylene.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Sampling equipment as specified in 5.1.1 to 5.1.13.

5.1.1 Sampling probe

Borosilicate glass or fused silica tube with a spherical ground joint at one end, of a suitable length to reach the representative measurement point(s) in the measurement plane of the waste gas flue and encircled by a heating jacket capable of producing a temperature of at least 200 °C.

NOTES

1 The heating jacket also serves as a protective tube to the gas sampling probe. The gas sampling probe should, therefore, always be used encircled by the heating jacket.

2 A stop valve before the first absorber is necessary to prevent loss of agents when sampling in flues under suction conditions.

5.1.2 Particle filter

Borosilicate glass or fused silica tube glass tube ends with spherical ground joints, packed in a progressive manner with chemically pure quartz wool. As an example, a particle filter that has proven suitable is shown in figure 1.

5.1.3 Absorbers

Absorption bottles of the Drechsel type, nominal capacity 100 ml or 250 ml as required (see table 1) equipped with an absorption bottle insert having a sintered filter (see figure 2), the porosity of which shall be fine enough to enable an absorption efficiency of at least 0,95 to be obtained. Sintered filters having pore diameters between 40 µm and 90 µm are suitable.

The absorption efficiency of each individual absorber should be tested twice a year as specified in 7.1.

NOTE — Impingers may be used if it is proven that absorption efficiency of at least 0,95 can be achieved.

5.1.4 Heating bandage, capable of producing a temperature of at least 200 °C.

5.1.5 Voltage regulator.

5.1.6 Trap

Absorption bottle of the Drechsel type, equipped with an absorption bottle insert not having a sintered filter.

5.1.7 Sampling pump, capable of drawing waste gas at a volume flow rate within the range 0,02 m^3/h to about 0,2 m^3/h during the sampling period against a pressure of − 10 kPa to − 30 kPa.

5.1.8 Regulating valve

Needle valve capable of adjusting a waste-gas flow rate within the range 0,02 m^3/h to about 0,2 m^3/h.

5.1.9 Gas metering device

Wet-gas meter (or dry gas meter with a drying tube upstream) capable of use at a waste-gas volume flow rate within the range 0,02 m^3/h to about 0,2 m^3/h, limits of error < 2 %, equipped with a thermometer (5.1.11).

Test the limits of error twice a year, for example by means of an appropriate soap bubble meter.

5.1.10 Connecting tubing of different lengths and of different internal diameters, made of polyethylene, silicone rubber or polytetrafluorethylene.

5.1.11 Thermometer, measuring range − 5 °C to + 50 °C, limits of error < ± 0,2 °C.

5.1.12 Barometer capable of measuring the atmospheric pressure present at the sampling location, limits of error approximately ± 1 % of the upper limit of measurement.

5.1.13 Stop watch.

5.2 Direct reading pH meter, preferably equipped with temperature compensation, measuring range 0 to 14, limits of error around pH 3,5 : < 0,2.

Calibrate the direct reading pH meter in accordance with the manufacturer's instructions using an appropriate buffer solution, the pH of which shall be accurately known at a given temperature. After calibration, rinse the electrodes thoroughly before use.