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Ambient air — Determination of the mass concentration of sulfur dioxide — Tetrachloromercurate (TCM)/pararosaniline method

Air ambiant — Détermination de la concentration en masse du dioxyde de soufre — Méthode au tétrachloromercurate (TCM) et à la pararosaniline

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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 6767 was prepared by Technical Committee ISO/TC 146, *Air quality*.

Annexes A and B form an integral part of this International Standard. Annex C is for information only.

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Introduction

This International Standard is a guideline, based on the West-Gaeke method, for the determination of the mass concentration of sulfur dioxide in ambient air. In annex A, a test is given for the purity and purification of pararosaniline hydrochloride; in annex B, a determination is given for the mass concentration of sulfur dioxide present in the sodium disulfite solution used for routine checks; in annex C, a method is given for the recovery of mercury from fresh and used solutions.

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Ambient air — Determination of the mass concentration of sulfur dioxide — Tetrachloromercurate (TCM)/pararosaniline method

1 Scope

This International Standard specifies a spectrophotometric method, known as the tetrachloromercurate (TCM)/pararosaniline method, for the determination of the mass concentration of sulfur dioxide in ambient air within the range of 20 µg/m³ to about 500 µg/m³.

The sampling period is 30 min to 60 min.

If a longer sampling period than 60 min is used, or higher concentrations of sulfur dioxide (up to about 2 000 µg/m³) are expected, care is necessary to ensure that the concentrations of sulfur dioxide in the absorption solution given in clause 6, paragraph 2 are not exceeded. This can be achieved by a reduction of the volume flow rate during sampling. Sample solutions obtained by this procedure may be stored for up to 24 h before making measurements, provided that they are kept in a refrigerator at about 5 °C.

Substances which are known to interfere and which might be present in the air being sampled are listed in 7.5

Indications of the precision and accuracy of the method, and of the lower detection limit are given in 8.2.

Detection limit, standard deviations and interferences qualify the TCM-method for orientating field measurements in the higher concentration range. When more accurate measurements are necessary, instruments which are specially tested and calibrated should be used.

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 4219:1979, *Air quality — Determination of gaseous sulphur compounds in ambient air — Sampling equipment.*

ISO 6349:1979, *Gas analysis — Preparation of calibration gas mixtures — Permeation method.*

3 Principle

Absorption of sulfur dioxide present in the air sample by passage through a sodium tetrachloromercurate (TCM) solution within a specified period resulting in the formation of a dichlorosulfitomercurate complex.

Destruction of any nitrite ions formed in the sodium tetrachloromercurate solution, by nitrogen oxides present in the air sample, by adding sulfamic acid solution. Conversion of the dichlorosulfitomercurate complex into intensely violet coloured pararosaniline methyl sulfonic acid, by adding a formaldehyde solution and an acidified pararosaniline hydrochloride solution to the resultant solution.

Determination of the absorbance of the sample solution at a wavelength of about 550 nm using an appropriate spectrophotometer (or colorimeter) and calculation of the mass concentration of sulfur dioxide by means of a calibration graph prepared using calibration gas mixtures.

Depending on the the equipment available in the laboratory, it may be convenient to use sodium disulfite solutions for routine checks. However, the

solutions are only used after proper calibration using a permeation device.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water, preferably double-distilled water, or water of equivalent purity, free from oxidants.

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

4.1 Sodium tetrachloromercurate (TCM), absorption solution, $c(Na_2[HgCl_4]) = 0{,}04$ mol/l.

Dissolve 10,9 g of mercury(II) chloride ($HgCl_2$), 4,7 g of sodium chloride (NaCl) and 0,07 g of ethylenedinitrilo tetraacetic acid, disodium salt dihydrate $[(HOCOCH_2)_2N(CH_2)_2N(CH_2COONa)_2.2H_2O]$ EDTA in water in a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix well.

Store the solution in a well stoppered bottle.

The solution is stable for several months, but discard it if a precipitate is formed.

NOTE 1 A procedure for recovering mercury from fresh and used solutions is given in annex C.

4.2 Pararosaniline hydrochloride (PRA), 0,16 g/l solution.

4.2.1 Transfer 86 ml of approximately 36 % (*m*/*m*) hydrochloric acid (HCl) ($\rho \approx 1{,}19$ g/ml) into a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix well.

4.2.2 Transfer 205 ml of approximately 85 % (*m*/*m*) phosphoric acid H_3PO_4 ($\rho \approx 1{,}69$ g/ml) into a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix well.

4.2.3 Dissolve 0,2 g of pararosaniline hydrochloride ($C_{19}H_{17}N_3.HCl$), the purity of which has been tested for each new PRA batch according to annex A, in 100 ml of the hydrochloric acid solution prepared in 4.2.1 .

4.2.4 Pipette 20 ml of the solution prepared in 4.2.3 into a 250 ml one-mark volumetric flask. Add 25 ml of the phosphoric acid solution prepared in 4.2.2. Mix well, and make up to the mark with water.

The solution is stable for several months if stored in the dark.

If the PRA solution is prepared from a PRA stock solution obtained by the purification of PRA in accordance with clause A.2, for each 1 % of difference between a degree of purity of 100 % and the degree of purity obtained, add an additional 0,2 ml of PRA stock solution before making up to the mark with water.

4.3 Formaldehyde, approximately 2 g/l solution.

Pipette 5 ml of a commercially available 36 % (*m*/*m*) to 38 % (*m*/*m*) formaldehyde (HCHO) solution 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.

4.4 Sulfamic acid, 6 g/l solution.

Dissolve 0,6 g of sulfamic acid (NH_2SO_3H) in 100 ml of water.

This solution is stable for a few days, if protected from air.

4.5 Sodium disulfite, 0,512 g/l solution.

4.5.1 Dissolve 0,3 g of sodium disulfite ($Na_2S_2O_5$) in 500 ml of freshly distilled water which has been deaerated, for example by boiling and cooling to room temperature.

This solution is not stable.

This solution contains the mass equivalent of 320 µg to 400 µg of sulfur dioxide per millilitre. Determine, as specified in annex B, the actual mass concentration of sulfur dioxide present in the solution.

4.5.2 Immediately after the determination of the actual mass concentration of sulfur dioxide present in the solution as specified in 4.5.1, pipette 2,0 ml of this solution into a 100 ml one-mark volumetric flask. Make up to the mark with absorption solution (4.1) and mix well.

This solution is stable for 30 days, if stored at about 5 °C, or for one day only, if stored at room temperature.

4.6 Calibration gas mixtures.

Immediately before use, prepare zero gas and gas mixtures of sulfur dioxide and air, using the permeation technique specified in ISO 6349. The latter shall be available in at least four different mass concentration levels of sulfur dioxide covering the desired working range.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Sampling equipment, as specified in ISO 4219 and in 5.1.1 to 5.1.5.

5.1.1 Air intake (see ISO 4219), made of polytetrafluoroethylene or borosilicate glass washed with perchloric acid then with distilled water and dried.

5.1.2 Filter for particulate matter (see ISO 4219).

Whenever possible, avoid the use of a filter for particulate matter. However, if a filter for particulate matter is used it shall be made of a material which meets the requirements specified in ISO 4219.

5.1.3 Absorber (see ISO 4219).

The absorption efficiency of the absorption bottle for sulfur dioxide shall be determined before use and shall be at least 0,95. Examples of suitable absorption bottles are given in figure 1.

The absorption efficiency varies with the geometry of the bottle, the size of the gas bubbles and their contact time with the solution. It can be determined by inserting into the sampling train a second absorber in series with the first, and relating the amount of sulfur dioxide found in the first bottle to the total amount of sulfur dioxide found in the two absorbers. When working with midget impingers under the conditions described in clause 6, the absorption efficiency was found to be better than 0,9

The use of mixtures of sulfur dioxide and air for calibration, as described in 7.1, automatically takes into account the absorption efficiency of the system.

5.1.4 Gas meter or **air-flow regulator** (see ISO 4219).

As an alternative to the gas meter, a critical orifice in a temperature controlled box can be used. In this case, the pump shall be able to reach $p_d/p_u \leqslant 0,5$, where p_d and p_u are the pressures downstream and upstream of the needle, respectively (see 5.1.5).

5.1.5 Two manometers, accurate to 1 kPa, for measurement of pressure when using a critical orifice instead of a gas meter.

5.2 Spectrophotometer or **colorimeter**, suitable for measuring the absorbance at about 550 nm. If a spectrophotometer is used, carry out the measurements with the instrument set at a wavelength of 548 nm. If a colorimeter is used, use a filter that has its transmittance maximum at a wavelength between 540 nm and 550 nm. Reagent blank problems may occur with apparatus having a spectral bandwith greater than 20 nm.

5.3 Optical cells, plane matched pairs, with an optical path length of 1,0 cm to 5,0 cm.

5.4 Polyethylene bottle, of capacity 100 ml, for transferring the exposed absorption solutions to the laboratory.

6 Sampling

Assemble a sampling train in accordance with the examples shown in figure 2 and any special requirements for the air mass under investigation.

Transfer 10 ml of the absorption solution (4.1) to an absorption bottle (5.1.3) and insert it into the sampling train. Choose a sampling period of either 30 min or 60 min and a volume flow rate between 0,5 l/min and 1 l/min.

The best results are obtained if 0,25 µg to 2,5 µg (0,1 µl to 0,95 µl at 25 °C and 101,3 kPa) of sulfur dioxide per millilitre of absorption solution is trapped.

After sampling, determine the volume of air sampled and note the atmospheric pressure (see 8.1, note 3). If the laboratory sample has to be stored before analysis, it may be kept at 5 °C for not longer than 24 h.

If the laboratory sample shows a precipitate, it is probably due to the reaction of mercury(II) with a reducing sulfur compound. Remove the precipitate by filtration or centrifugation before the analysis.

7 Procedure

7.1 Calibration

7.1.1 Preparation of a set of calibration solutions

Mixtures of sulfur dioxide and air are prepared according to ISO 6349.

In order to prepare the calibration graph, which is a plot of absorbance versus sulfur dioxide concentration, at least four different concentration levels of sulfur dioxide within the range specified in clause 2 are needed.

Sample each gas mixture under the same conditions as are used for the unknown air sample, in particular for the same sampling period and at the same flow rate. Treat the obtained sample solutions as described in 7.3 (see 4.6).

7.1.2 Zero member solution

Prepare a zero member solution from zero gas (see 4.6). This preparation is carried out according to 7.1.1.

7.1.3 Plotting the calibration graph

Correct the absorbance values to allow for the absorbance of the blank solution (7.4). Plot the net absorbance of each solution against the mass, in micrograms, of sulfur dioxide in the gas sample from which it was derived. Calculate the calibration factor f (reciprocal of the slope of the line) for use in the calculation of results (8.1).

7.2 Preparation of routine check graph

Pipette 0 ml, 1,0 ml, 2,0 ml, 3,0 ml and 4,0 ml of sodium disulfite solution (4.5.2) into a series of 25 ml one-mark volumetric flasks. Add sufficient absorption solution (4.1) to each flask to bring the volume to approximately 10 ml. Then carry out the analysis as described in 7.3.2.

Plot the absorbances of the solutions as ordinates against the mass, in micrograms, of sulfur dioxide calculated according to annex B. A linear relationship is obtained. The intercept of the line best fitting the points with the vertical axis is usually within 0,02 absorbance units of the zero member solution reading if 1 cm cells are used. Calculate the calibration factor f' (reciprocal of the slope of the line). This calibration factor can be used for routine purposes; the value obtained when dividing f' by the volume, in cubic metres, of air sampled during the calibration procedure according to 7.1, shall differ by less than 10 % from the calibration factor f obtained with calibration gas mixtures.

7.3 Determination

7.3.1 Leave the sample (clause 6) for at least 20 min after sampling to allow trapped ozone to decompose. Then transfer the sample solution quantitatively to a 25 ml one-mark volumetric flask, using about 5 ml of water for rinsing.

7.3.2 Add 1 ml of the sulfamic acid solution (4.4) to each flask and allow to react for 10 min to destroy the nitrites from oxides of nitrogen. Then pipette 2,0 ml of the formaldehyde solution (4.3) followed by 5 ml of the pararosaniline reagent (4.2.4) into the flasks. Make up to the mark with freshly boiled and cooled distilled water and store at 20 °C ± 1 °C. Using the spectrophotometer or colorimeter (5.2) measure the absorbance of the sample solution and the zero member solution, against distilled water in the reference cell, between 30 min and 60 min after the addition of the reagents and immediately after filling the cells.

Do not allow the coloured solutions to remain in the cell as a coloured film will be deposited on the inside walls.

NOTE 2 Fixed time intervals between the addition of each reagent, for example 1 min, ensure a better reproducibility of the formation of the absorbing compound.

7.4 Preparation of the blank solution

Prepare a blank by pipetting 10 ml of unexposed absorption solution (4.1) into a 25 ml one-mark volumetric flask, add the reagents as before, treat the solution as described in 7.3.2 and read the absorbance against distilled water using 1 cm cells. Compare this value with the one recorded for the zero member solution obtained when preparing the calibration graph (7.1). Differences of more than 10 % between the two values indicate contamination of the distilled water or the reagents or decomposition of the latter, in which case fresh reagents shall be prepared.

7.5 Interference

The amount of EDTA added eliminates interference from heavy metals up to 30 µg of manganese(II), 10 µg of chromium(III), 10 µg of copper(II) and 22 µg of vanadium(V) in 10 ml of the absorption solution.

If sulfamic acid (4.4) is used with the described procedure up to 50 µg of nitrogen dioxide per 10 ml of absorption solution can be tolerated.

Interference from oxides of nitrogen, ozone and reducing sulfur compounds (for example hydrogen sulfide and mercaptans) are eliminated or minimized. Sulfuric acid and sulfates do not interfere. No interference by sulfur trioxide has been proved experimentally, since this presumably becomes hydrolysed to sulfuric acid in the absorption solution.

8 Expression of results

8.1 Calculation

Calculate the mass concentration of sulfur dioxide, $\rho(SO_2)$, expressed in micrograms per cubic metre, by using the equations

$$\rho(SO_2) = \frac{f(A_s - A_b)}{V_1}$$

$$\rho(SO_2) = \frac{f'(A_s - A_b)}{V_1}$$

where

f and f' are calibration factors (see 7.1 and 7.2);

A_s is the absorbance of the sample solution;

A_b is the absorbance of the blank;

V_1 is the volume, in cubic metres, of the air sample.

NOTE 3 If the sulfur dioxide concentration at reference conditions (298 K, 101,3 kPa ≈ 10^2 kPa) is needed, replace the volume V_1 of air sampled by the corresponding value V_2 of the volume under the reference conditions:

$$V_2 = \frac{298 V_1 p}{(273 + t)10^2}$$

where

p is the barometric pressure, in kilopascals;

t is the temperature, in degrees Celsius, of the air sample.

8.2 Performance characteristics

8.2.1 Precision and accuracy

The method is not known to have any inherent bias or inaccuracy. The accuracy achieved in practice will depend on the care used in performing the various calibrations and measurements.

As an indication of performance, a relative standard deviation of the order of ± 10 % should be achievable for a measured concentration between 80 µg SO_2/m^3 and 200 µg SO_2/m^3 in ambient air.

8.2.2 Lower detection limit

The lower detection limit of sulfur dioxide in 10 ml of TCM sample solution is between 0,2 µg and 1,0 µg, based on twice the standard deviation of the zero member solution, (7.1.2). This corresponds to a concentration between 7 µg SO_2/m^3 and 33 µg SO_2/m^3 [0,002 ppm (V/V) to 0,011 ppm (V/V) in an air sample of 30 litres (for example 1 h sampling at 0,5 l/min)].

9 Test report

The test report shall include the following information:

a) a complete identification of the air sample;

b) a reference to this International Standard;

c) the results obtained;

d) any unusual features noted during the determination;

e) any operation not specified in this International Standard or in the International Standards to which reference is made, or regarded as optional.

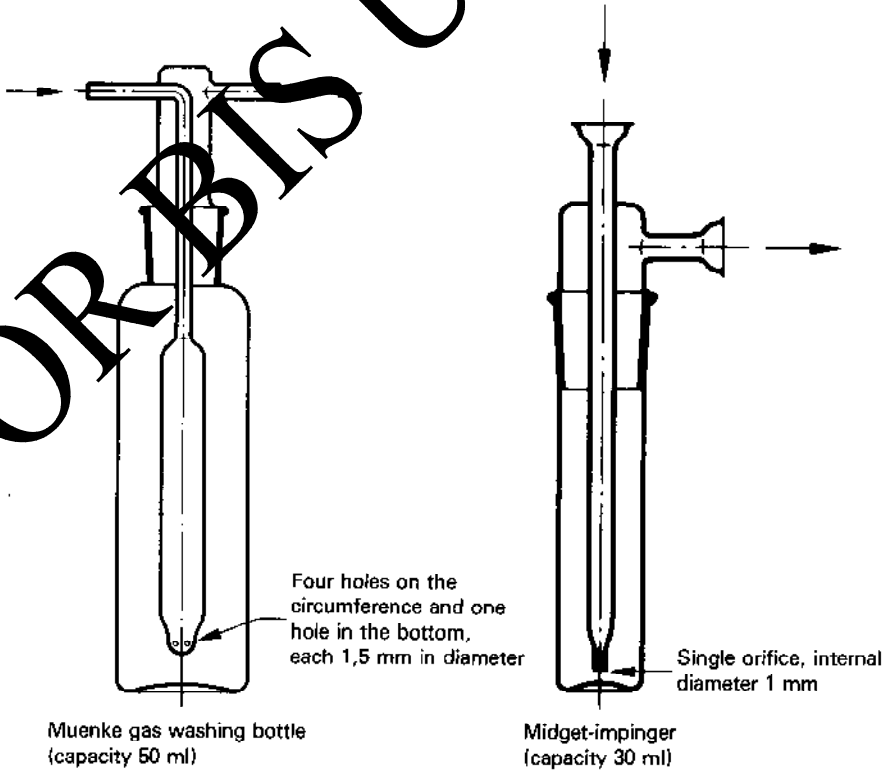

Figure 1 — Examples of absorption bottles suitable for sampling sulfur dioxide in air

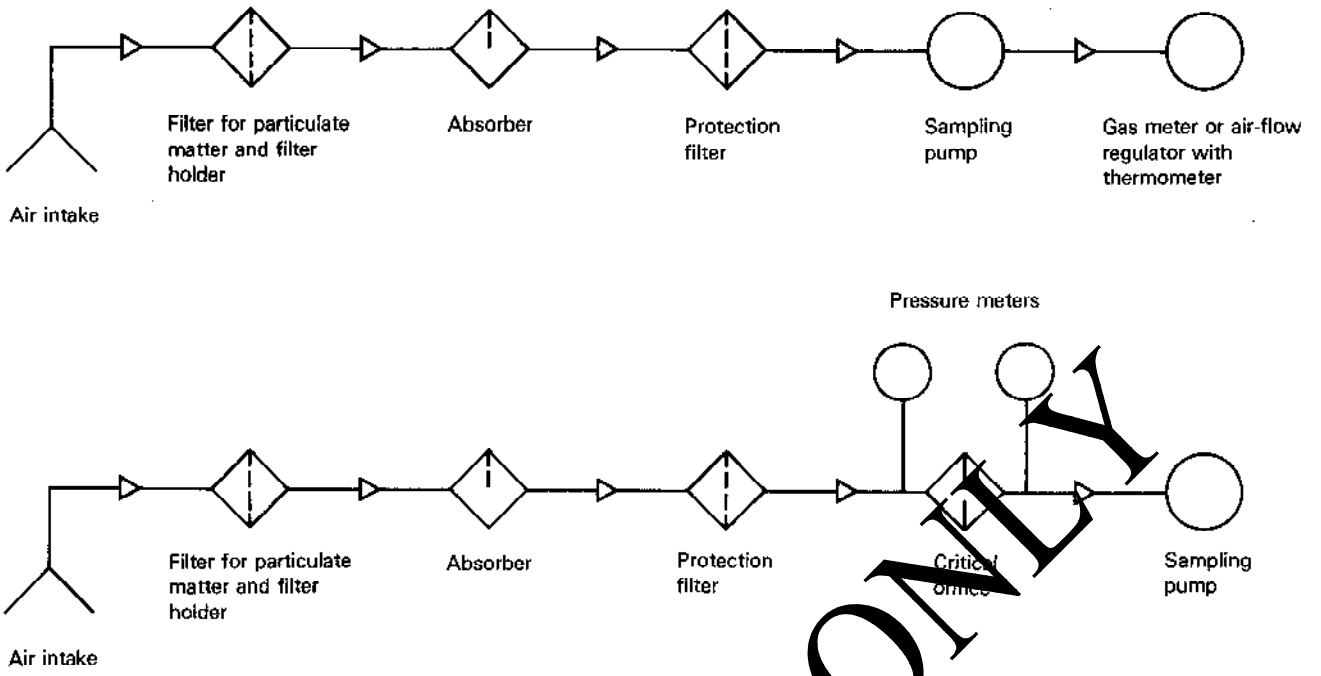

Figure 2 — Examples of sampling trains for the determination of the mass concentration of sulfur dioxide in ambient air

Annex A
(normative)

Test for the purity and method of purification of pararosaniline hydrochloride (PRA)

A.1 Test for the purity of PRA

Carry out the following two tests to check the purity of the pararosaniline hydrochloride (PRA). If one of the tests is negative, either reject the PRA batch or purify it by the procedure described in clause A.2 .

A.1.1 Checking of the blank value

Prepare a reagent with the pararosaniline solution (4.2) as described in 7.3.

The absorbance of the reagent blank shall not exceed 0,17.

A.1.2 Purity check

Dilute 1 ml of the pararosaniline solution (4.2.3) to 100 ml with distilled water. Transfer 5 ml to a 50 ml one-mark volumetric flask and add 5 ml of a 0,1 mol/l acetic acid/sodium acetate buffer solution. Make up to the mark with water and mix. Allow the absorbing compound to form for 1 h. The solution, which has a maximum absorption at 540 nm, shall have an absorbance of at least 0,45 at this wavelength when measured in a 1 cm cell.

Use the absorbance at 540 nm to calculate the purity of the PRA, expressed as a percentage by mass, by means of the formula

$$\frac{A \times K}{m_1}$$

where

A is the absorbance of the solution;

m_1 is the mass, in milligrams, of the dye in 50 ml of the PRA solution (4.2.3);

K = 21 300, when using a spectrophotometer with a bandwith less than 10 nm.

The purity of the reagent shall be at least 95 %.

A.2 Purification of PRA

In a 250 ml separating funnel, equilibrate 100 ml each of 1-butanol and 1 mol/l hydrochloric acid. Weigh 0,1 g of pararosaniline hydrochloride (PRA) into a beaker. Add 50 ml of the butanol-saturated acid and allow to stand for several minutes. Add 50 ml of the HCl-saturated 1-butanol to a 125 ml separating funnel. Transfer the acid solution containing the PRA to the funnel and extract. Any violet impurity will transfer to the organic phase. Transfer the lower (aqueous) phase into another separating funnel and add a 20 ml portion of HCl-saturated 1-butanol. This is usually sufficient to remove almost all the violet impurity which contributes to the reagent blank. If the violet impurity still appears in the 1-butanol phase after five extractions, discard this batch of dye.

After the final extraction, filter the aqueous phase through a cotton wool plug into a 50 ml one-mark volumetric flask and make up to the mark with hydrochloric acid [c(HCl) = 1 mol/l]. This purified solution will be yellowish red and serves as a PRA stock solution (see 4.2.3).

NOTE 4 Certain batches of 1-butanol contain oxidants that create a sulfur dioxide demand. Check by shaking 20 ml of 1-butanol with 5 ml of a 15 % (m/m) potassium iodide solution. If a yellow colour appears in the alcohol phase, redistil the 1-butanol from silver oxide.

Annex B
(normative)

Determination of the mass concentration of sulfur dioxide present in the sodium disulfite solution for routine checks

B.1 Reagents

B.1.1 Iodine, stock solution, $c(I_2) \approx 0,05$ mol/l.

Weigh 12,7 g of iodine (I_2) into a 250 ml beaker, add 40 g of potassium iodide (KI) and 25 ml of water. Stir until total dissolution and transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask. Make up to the mark with water.

B.1.2 Iodine, solution, $c(I_2) \approx 0,005$ mol/l.

Dilute 50 ml of the iodine stock solution (B.1.1) to 500 ml with water.

B.1.3 Starch indicator, 2 g/l solution.

Triturate 0,4 g of soluble starch and 0,002 g of mercury(II) iodide (HgI_2) preservative, with a little water, and add the paste slowly to 200 ml of boiling water. Continue boiling until the solution is clear, cool and transfer to a glass-stoppered bottle.

B.1.4 Potassium iodate (KIO_3), 3,0 g/l solution.

Dry a few grams of potassium iodate overnight at 180 °C. Weigh, to the nearest 0,1 mg, about 1,5 g into a 500 ml one-mark volumetric flask and dilute to the mark with water.

B.1.5 Hydrochloric acid, 44 g/l.

Dilute 100 ml of concentrated hydrochloric acid ($\rho \approx 1,19$ g/ml) to 1 litre.

B.1.6 Sodium thiosulfate, stock volumetric solution, $c(Na_2S_2O_3.5H_2O) \approx 0,1$ mol/l.

Dissolve 25 g of sodium thiosulfate pentahydrate in 1 litre of freshly boiled distilled water and add 0,1 g of sodium carbonate to the solution. Allow the solution to stand for 1 day before standardizing as follows.

Pipette 25 ml of the potassium iodate solution (B.1.4) into a 500 ml iodine flask. Add 1 g of potassium iodide (KI) and 10 ml of hydrochloric acid (B.1.5). Stopper the flask. After 5 min, titrate with sodium thiosulfate solution to a pale yellow colour. Add 2 ml of the starch indicator (B.1.3) and titrate until the blue colour just disappears.

Calculate the amount-of-substance concentration, c_1, expressed in moles per litre, of the sodium thiosulfate solution by using the equation

$$c_1 = \frac{m_2 \times 10^3 \times 0,1}{V_3 \times 35,67} = \frac{100 m_2}{35,67 V_3}$$

where

m_2 is the mass, in grams, of the potassium iodate in the 25 ml portion of solution (B.1.4);

V_3 is the volume, in millilitres, of sodium thiosulfate solution used for the titration;

35,67 is the relative molecular mass of $(KIO_3)/6$.

B.1.7 Sodium thiosulfate, standard volumetric solution, $c(Na_2S_2O_3.5H_2O) \approx 0,01$ mol/l.

Dilute 50,0 ml of the sodium thiosulfate stock solution (B.1.6) to 500 ml with water and mix.

The solution is not stable and shall be prepared on the day of use.

B.2 Procedure

To prepare a blank, introduce 25 ml of water into a 500 ml conical flask and then pipette 50 ml of the iodine solution (B.1.2) into the flask. Pipette 25 ml of the sodium disulfite solution (4.5.1) into a second conical flask and then pipette 50 ml of the iodine solution (B.1.2) into this flask. Stopper the conical flasks and allow to react for 5 min.

Titrate the contents of each flask in turn with sodium thiosulfate solution (B.1.7) to a pale yellow colour. Then add 5 ml of the starch indicator (B.1.3) and continue the titration until the blue colour disappears.

B.3 Expression of results

Calculate the mass concentration of sulfur dioxide, $\rho(SO_2)$, expressed in micrograms per millilitre, in the sodium disulfate solution (4.5.1) by using the equation

$$\rho(SO_2) = \frac{(V_4 - V_5)32{,}02c_2}{V_6}$$

where

V_4 is the volume, in millilitres, of the sodium thiosulfate solution (B.1.7) used for the titration of the blank;

V_5 is the volume, in millilitres, of the sodium thiosulfate solution used for the titration of the sample;

V_6 is the volume, in millilitres, of the sodium disulfite solution (4.5.1) used;

c_2 is the amount-of-substance concentration, in moles per litre, of sodium thiosulfate in the sodium thiosulfate solution (B.1.7);

32,02 is the relative molecular mass of $(SO_2)/2$.

The concentration of sulfur dioxide in the sodium disulfite solution (4.5.2) is found by dividing the result by 50.

Annex C
(informative)

Recovery of mercury from fresh and used solutions

C.1 Scope

This annex describes a method for the removal of mercury from the residual solutions obtained when using the absorption solution (4.1).

C.2 Reagents

C.2.1 Sodium hydroxide (NaOH), approximately 400 g/l solution

C.2.2 Hydrogen peroxide (H_2O_2), approximately 30 % (*m/m*)

C.2.3 Sodium sulfide nonahydrate ($Na_2S.9H_2O$).

C.3 Procedure

Collect the residual solutions whose mercury content is too high to allow them to be discarded down the sink in a polyethylene container of volume 50 litres. When the collected volume reaches approximately 40 litres, add, in the following order, while stirring by means of air bubbling through the solution, a volume of sodium hydroxide solution (C.2.1) sufficient for neutralisation, an additional 400 ml of sodium hydroxide solution, 100 g of sodium sulfide nonahydrate (C.2.3) and, after 10 min, slowly add 400 ml of hydrogen peroxide solution (C.2.2).

Leave the mixture for 24 h and then draw off and discard the clear liquid.

Discard the residue according to the national regulations.

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