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Corrosion of metals and alloys — Corrosivity of atmospheres — Measurement of pollution

*Corrosion des métaux et alliages — Corrosivité des atmosphères —
Mesurage de la pollution*

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Foreword

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International Standard ISO 9225 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

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Introduction

The ability of an atmosphere to cause corrosion of metals and alloys is controlled by the following factors: temperature-humidity complex and pollution. A basic requirement for the determination of the corrosivity of atmospheres is the standardized measurement of concentration and deposition rates of environmental pollution. The concentration of sulphur dioxide (SO₂) in the air should be measured in accordance with special standards. This International Standard represents methods for the measurement of deposition rates of sulphur dioxide (SO₂) and airborne salinity.

The methods included in this International Standard have been chosen for their easy applicability and good comparability of results. It is important to stress that the classification of sulphur dioxide (SO₂) and airborne chloride (Cl⁻) levels given in ISO 9223:1992, *Corrosion of metals and alloys — Corrosivity of atmospheres — Classification*, is based on measurement methods described in this International Standard.

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Corrosion of metals and alloys — Corrosivity of atmospheres — Measurement of pollution

WARNING — Some of the procedures included in this International Standard entail the use of potentially hazardous chemicals. It is emphasised that all appropriate safety precautions should be taken.

1 Scope

1.1 This International Standard specifies three methods for measuring the deposition rates of sulfur dioxide (SO₂) (sulfur compounds) and airborne salinity.

It does not cover concentration measurements. The concentration of sulfur dioxide (SO₂) and other pollutants in the air should be measured in accordance with standard methods, e.g. ISO 4221:1980, *Air quality — Determination of mass concentration of sulphur dioxide in ambient air — Thorin spectrophotometric method*.

The measurement methods included in this International Standard apply for characterization of corrosivity of the test site. The pollution data obtained by these methods are generally used for the determination of corrosivity categories.

2 Determination of sulfur dioxide (SO₂) deposition rate on lead dioxide (PbO₂) sulfation plates

2.1 Principle

Atmospheric sulfur dioxide (SO₂) reacts with the lead dioxide to form lead sulfate. The plates are re-covered and sulfate analysis is performed on the contents to determine the extent of sulfur dioxide capture. The deposition rate of sulfur dioxide is expressed in milligrams per square metre day [mg/(m²·d)].

The lead dioxide reagent used in this method may also convert other sulfur bearing compounds, such as hydrogen sulfide and mercaptans, to sulfate.

The inverted position of the disc is intended to minimize sulfur capture from acid precipitation or sulfuric acid aerosol.

2.2 Sampling apparatus

2.2.1 Sulfation plate

Sulfation plates may be purchased ready for exposure or may be prepared. The following method is recommended for the preparation of sulfation plates.

Bond filter paper circles to the bottom of polystyrene Petri dishes. The circle diameters may be 50 mm or 60 mm. Bonding is carried out by placing a filter paper rough side up, in the bottom of the dish. The filter paper should fit inside the dish without wrinkling. Carefully squirt acetone into the dish so that the filter becomes just saturated. Press the filter paper firmly with a glass rod so that it adheres completely to the dish. Allow the acetone to evaporate.

Place a batch of bonded plates (80 50 mm or 55 60 mm plates) in a rack and rinse with distilled or deionized water. Fill the plates with water again and allow to stand for 1 h. Pour the water out of the plates and refill to between one quarter and one half with distilled or deionized water.

Add 3,5 g of tragacanth gum and 900 ml distilled or deionized water to a high speed blender. Set at a low speed and blend for 2 h.

Pour the contents of the blender into a 1 litre beaker and pour 350 ml of the solution back into the blender. Pulp 3,5 g of filter paper, add it to the 350 ml of gum solution and set the blender at a moderate speed until the mixture appears smooth and uniform.

Pour 400 ml of the previously prepared gum solution into the blender and blend at a moderate speed for 1 min.

Set the blender at a high speed and add 112 g of lead dioxide. Blend for 2 min and then turn the blender back to a low speed.

Carefully pipette 10 ml of the mixture into each 50 mm plate or 15 ml into each 60 mm plate. Make sure that the mixture spreads uniformly to the edge of each plate.

Place the rack of plates in an oven set at 40 °C to 50 °C for 20 h.

Remove the plates from the oven, allow to cool and seal them with tight-fitting covers to protect them until exposure begins.

Number the plates and expose them within 120 days of preparation. Retain at least three plates from each batch for reference.

2.2.2 Exposure rack

Brackets shall be used to hold the plates securely in an inverted position, so that the lead dioxide mixture faces downwards. The plates shall be horizontal and shall not be obstructed from normal winds and air circulation currents. The brackets shall be constructed from a material which has adequate resistance to atmospheric corrosion. They shall include a retaining clip or other provision to hold the plate in the event of strong winds. A typical bracket design is shown in figure 1.

Dimensions in millimetres

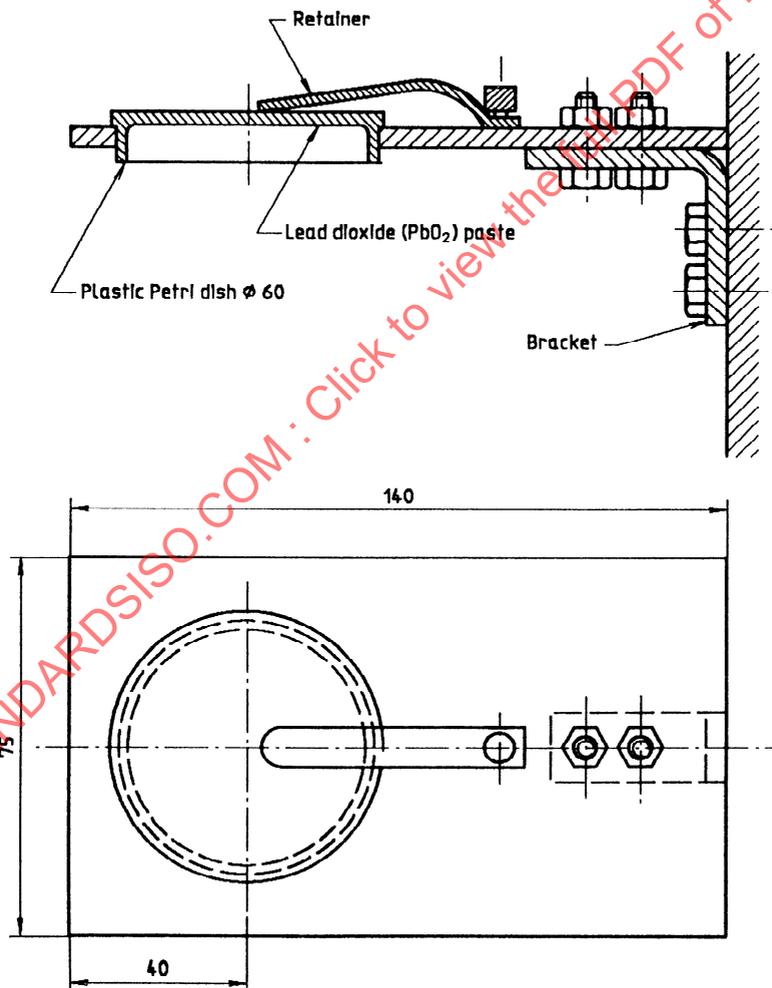


Figure 1 — Sulfation plate holder

2.3 Sampling

When monitoring exposure sites, a minimum of three plates shall be used for each exposure period.

The plates should be placed, if possible, at the highest and lowest levels of exposure of corrosion test specimens.

A 30 day \pm 2 day exposure period is recommended. At the end of the exposure period, the plates should be removed from the bracket and covered tightly to prevent additional sulfation. Analysis of the plates should be completed within 60 days of the completion of the exposure. When the exposure is finished, the plate identification, exposure location and the dates of exposure initiation and completion shall be recorded.

2.4 Sulfate analysis

The determination of the sulfate content of the sulfation plates shall be conducted using any established quantitative analysis technique. A turbidimetric method is described below.

2.4.1 Principle

The contents of the sulfation plate are removed and dissolved, for example using a solution of sodium carbonate. The sulfate is then precipitated with the barium ion and measured turbidimetrically.

2.4.2 Reagents

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

2.4.2.1 Sodium carbonate, approximately 50 g/l.

Dissolve 50 g \pm 0,5 g of anhydrous sodium carbonate (Na_2CO_3) in 1 litre of water.

2.4.2.2 Hydrochloric acid, solution, $c(\text{HCl}) = 0,7 \text{ mol/l}$.

Dilute 60 ml of concentrated hydrochloric acid ($\rho = 1,19 \text{ g/ml}$) to 1 litre with water.

2.4.2.3 Barium chloride dihydrate, 5 g/l solution.

Dissolve 5 g of barium chloride dihydrate ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in 1 litre of water.

2.4.2.4 Sodium sulfate, standard solution corresponding to 500 mg of SO_4^{2-} per litre.

Weigh, to the nearest 0,1 mg, 0,740 g of anhydrous sodium sulfate (Na_2SO_4), place it in a 1 000 ml one-

mark volumetric flask, dissolve in water, dilute to the mark and mix well.

1 ml of this standard solution contains 500 μg of SO_4^{2-} .

2.4.3 Plate analysis

Quantitatively remove the lead dioxide and as much of the fibrous material as possible from the plate and transfer it to a 50 ml one-mark volumetric flask.

Add 20 ml of sodium carbonate solution (2.4.2.1) and stir.

Allow to stand for 3 h with occasional stirring.

Place in a 100 °C water bath for 30 min.

Cool and dilute to the mark with water.

Filter at least 15 ml through a quantitative fine-grade filter paper into a clean, dry test tube. Do not wash.

Pipette 10,0 ml of filtered sample into a 150 mm long test tube of diameter 25 mm and add 10,0 ml of water and 5,0 ml of the hydrochloric acid solution (2.4.2.2). Swirl to mix and check the pH with indicator paper. The pH must be between 2,5 and 4,0. If not, discard and repeat this step.

Pipette 5,0 ml aliquots of the sample into two test tubes of 25 mm diameter and add 15 ml of water to each.

Add 1 ml of barium chloride solution (2.4.2.3), mix vigorously and allow to stand for 5 min before measuring turbidity.

Measure the turbidity of the sample solution at 500 nm against a solution containing no barium. Convert the absorbance (turbidity) reading to micrograms of sulfate using the calibration curve obtained as specified in 2.4.4.

Blank samples consisting of unexposed sulfation plates taken from each batch of prepared and exposed plates should be analyzed at the same time.

2.4.4 Preparation of the calibration curve

Dilute 10,0 ml of the sodium sulfate standard solution (2.4.2.4) to 100 ml using a volumetric flask.

Pipette the following volumes of the diluted sodium sulfate solution into test tubes: 1 ml, 2 ml, 3 ml, 4 ml, 5 ml, 10 ml and 15 ml. Dilute each volume to 20 ml with water and then follow the procedure specified in 2.4.3.

On linear graph paper plot the absorbance readings of each of the above solutions against the respective mass of sulfate which they contain, i.e., 50 μg , 100 μg , 150 μg , 200 μg , 250 μg , 500 μg and 750 μg .

2.5 Expression of results

The sulfation rate is calculated in terms of sulfur dioxide (SO₂) capture by the plate. The mass of sulfate obtained from the plate analysis procedure is converted to net sulfation mass by subtracting the blank value obtained from the batch of plates in question.

The deposition rate of sulfur dioxide (SO₂), expressed in milligrams per square metre day [mg/(m²·d)], $R_{(SO_2)}$, is given by the equation

$$R_{(SO_2)} = \frac{(m_1 - m_0) \times 16,67}{A \cdot t \times 1\,000} \quad \dots (1)$$

where

- m_0 is the mass, in micrograms, of sulfates contained in the blank test;
- m_1 is the mass, in micrograms, of sulfates contained in a plate;
- A is the area, in square metres, of a plate;
- t is the exposure time, in days

3 Determination of sulfur dioxide (SO₂) deposition rate on alkaline surfaces

3.1 Principle

Sulfur oxides (SO_x) and other sulfur compounds of an acid character are collected on the alkaline surface of porous filter plates saturated by a solution of sodium or potassium carbonate. Sulfur compounds which are converted into sulfates are determined. The deposition rate of sulfur dioxide is expressed in milligrams per square metre day [mg/(m²·d)].

3.2 Sampling apparatus

3.2.1 Sulfation plate

Sulfation plates shall be prepared by the following method.

Immerse paper filter plates of 150 mm × 100 mm × 3 mm, for 2 min in a 70 g/l sodium or potassium carbonate solution. (It is admissible to use chromatographic paper filter plates or other materials of similar properties.)

Remove the plates from the solution, drain and dry for either 1 h or 8 h at a temperature of 105 °C ± 2 °C or 90 °C ± 2 °C, respectively. After drying, seal the plates in plastic containers (polyethylene, polypropylene). Take five plates from every processed series in which the sulfate content is determined as a blank.

3.2.2 Exposure rack

The test plates are exposed on a rack (see figure 2) in a vertical position so that their surfaces are parallel to the prevailing direction of wind. The bottom edges of the plates shall be 1,8 m to 2 m above the ground. A roof over the rack prevents washing out by rain but enables free access of air to the plates.

3.3 Sampling

At the test location (test site) install three test plates with the fasteners on the rack. The exposure time of the test plates is 30 days ± 2 days, unless the character of the test or the pollution level necessitate a different time (60 days or 90 days). After the exposure is complete, remove the test plates without damaging the surface layer and seal them separately in plastic containers. Mark the plastic containers with the test site name and dates of exposure and removal.

3.4 Sulfate analysis

The sulfate analysis shall be completed within 60 days of the completion of exposure.

The volumetric determination of sulfates in the test plates is described below. Any other analytical procedure which gives a satisfactory precision, for example gravimetric or spectrophotometric methods, can be used.

3.4.1 Principle

The amount of sulfates is determined by the titration of a water/ethanol sample solution with a barium perchlorate water/ethanol titrant in the presence of thorin indicator. The end-point of the titration is indicated by the yellow-pink colour transition in the pH interval 2-3.

3.4.2 Reagents

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

Dimensions in millimetres

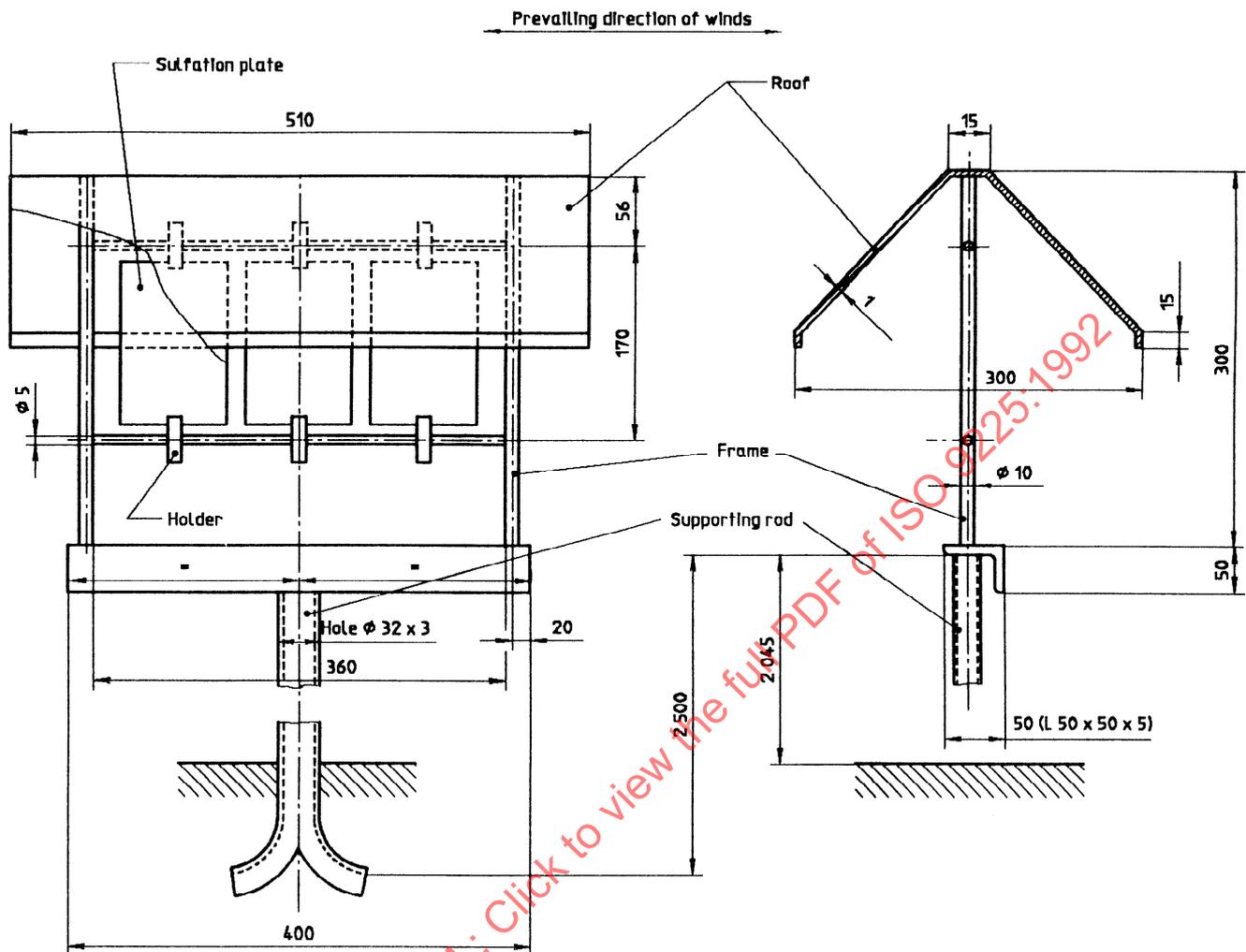


Figure 2 — Exposure rack for alkaline sulfation plates

3.4.2.1 **Hydrogen peroxide**, 150 g/l solution.

3.4.2.2 **Ethanol**, 95 % (V/V).

3.4.2.3 **Perchloric acid**, diluted 1 + 5.

3.4.2.4 **Sulfuric acid**, standard reference solution, $c(\text{H}_2\text{SO}_4) = 0,005 \text{ mol/l}$.

3.4.2.5 **Barium perchlorate**, standard volumetric solution $c[\text{Ba}(\text{ClO}_4)_2] = 0,005 \text{ mol/l}$.

Dissolve 2,0 g of barium perchlorate, trihydrate $[\text{Ba}(\text{ClO}_4)_2 \cdot 3\text{H}_2\text{O}]$ in 200 ml of water, and adjust the pH of the solution to a value of 2 to 3 by the addition of perchloric acid (3.4.2.3). Dilute the solution to 1 000 ml with ethanol (3.4.2.2). The exact concentration is determined by the titration of 10 ml of sulfuric acid standard reference solution (3.4.2.4) as described in 3.4.3.2.

3.4.2.6 **Thorin indicator**, 2 g/l solution.

Dissolve 0,2 g of thorin indicator in 100 ml of water.

3.4.3 Plate analysis

3.4.3.1 Preparation of the sample solution

Remove the test plate from its plastic container and transfer it to an 800 ml beaker. After wetting, split it into pieces by means of a glass rod. Adjust the volume of the suspension to 400 ml with water.

Add 1 ml of hydrogen peroxide solution (3.4.2.1) and stir the suspension thoroughly.

Boil the mixture for a short time and leave it to rest overnight.

Vacuum filter the suspension through a quantitative coarse grade filter paper and wash the residue on the filter twice with water. Adjust the volume of the

filtrate to 500 ml in a volumetric flask (sample solution).

3.4.3.2 Analysis

Pipette 10 ml of the sample solution into a titration flask. If the sulfate concentration in the sample solution is high, a lower volume may be pipetted and the volume made up to 10 ml with water. If the sulfate concentration in the sample solution is low, 5 ml of sulfuric acid standard reference solution (3.4.2.4) may be added.

Adjust the pH to the value of 2 to 3 by the addition of perchloric acid (3.4.2.3).

Add 40 ml of ethanol (3.4.2.2) and 3 drops of thorin indicator solution (3.4.2.6).

Titrate the solution using barium perchlorate standard solution (3.4.2.5) to the yellow-pink colour transition.

3.5 Expression of results

The results shall be expressed as sulfur dioxide deposition rate, in milligrams per square metre day [$\text{mg}/(\text{m}^2 \cdot \text{d})$], $R_{(\text{SO}_2)}$, and are calculated according to the equation

$$R_{(\text{SO}_2)} = \frac{m}{A \cdot t} \quad \dots (2)$$

where

- m is the total mass of sulfur dioxide (SO_2), in milligrams, on the test plate;
- A is the area, in square metres, of the exposed part of the test plate;¹⁾
- t is the exposure time, in days.

For the titration of sulfates by barium perchlorate standard volumetric solution, the results are calculated according to the equation

$$R_{(\text{SO}_2)} = \frac{(V_1 - V_0)c_1 \times 64,06 V_T}{V_A \cdot A \cdot t} \quad \dots (3)$$

where

- $R_{(\text{SO}_2)}$ is the sulfur dioxide deposition rate, in milligrams per square metre day;
- V_0 is the volume, in millilitres, of barium perchlorate standard volumetric solution (3.4.2.5) used for titration of the blank;
- V_1 is the volume, in millilitres, of barium perchlorate standard volumetric solution (3.4.2.5) used for titration of the aliquot of sample;
- V_A is the volume, in millilitres, of pipetted aliquot of sample solution;
- V_T is the total volume, in millilitres, of sample solution;
- c_1 is the exact concentration, in moles of $\text{Ba}(\text{ClO}_4)_2$ per litre, of barium perchlorate standard volumetric solution (3.4.2.5);
- A is the area, in square metres, of the exposed part of the test plate;¹⁾
- t is the exposure time, in days.

4 Determination of chloride deposition rate by the wet candle method

4.1 Principle

A rain-protected wet textile surface, with a known area, is exposed during a specified time. The amount of chloride deposited is determined by chemical analysis. From the results of this analysis the chloride deposition rate is calculated, expressed in milligrams per square metre day [$\text{mg}/(\text{m}^2 \cdot \text{d})$].

1) $A = 0,03 \text{ m}^2$ for a test plate of 150 mm \times 100 mm.

4.2 Sampling apparatus

4.2.1 Wet candle

The wet candle is formed of a wick inserted into a bottle. The wick consists of a central core of about 25 mm in diameter made of inert material (polyethylene) over which is stretched or wound a double layer of tubular surgical gauze or of a band of surgical gauze. The surface of the wick exposed to the atmosphere shall be about 100 cm², which corresponds to a wick length of about 120 mm. The exposed area shall be accurately known. One end of the wick is inserted into a rubber stopper. The stopper has two additional holes through which the free ends of the gauze pass (if tubular gauze is used, the lower end is cut along the length of the gauze until about 120 mm is left). The edges of the three holes are shaped into a funnel so that liquid

running down the gauze drains through the stopper (see figure 3). The free ends of the gauze must be long enough to reach the bottom of the bottle.

The stopper is inserted into the neck of a bottle of polyethylene or another inert material, with a volume of about 500 ml. The bottle contains 200 ml of a glycerol and water solution [20 % (V/V)] with octanoic acid added to prevent the growth of fungi such as *Aspergillus niger*. The solution is made up by mixing 200 ml of glycerol [CHOH(CH₂OH)₂] with distilled water to a volume of 1 000 ml. To the solution is added 20 drops of octanoic acid (C₈H₁₆O₂).

In situations of prolonged extreme temperatures, i.e. greater than 25 °C or less than -25 °C, it may be necessary to increase the glycerol content to 40 % (V/V) to prevent freezing or excessive evaporation, or to replace the glycerol solution by ethylene-glycol at 20 % (V/V) strength.

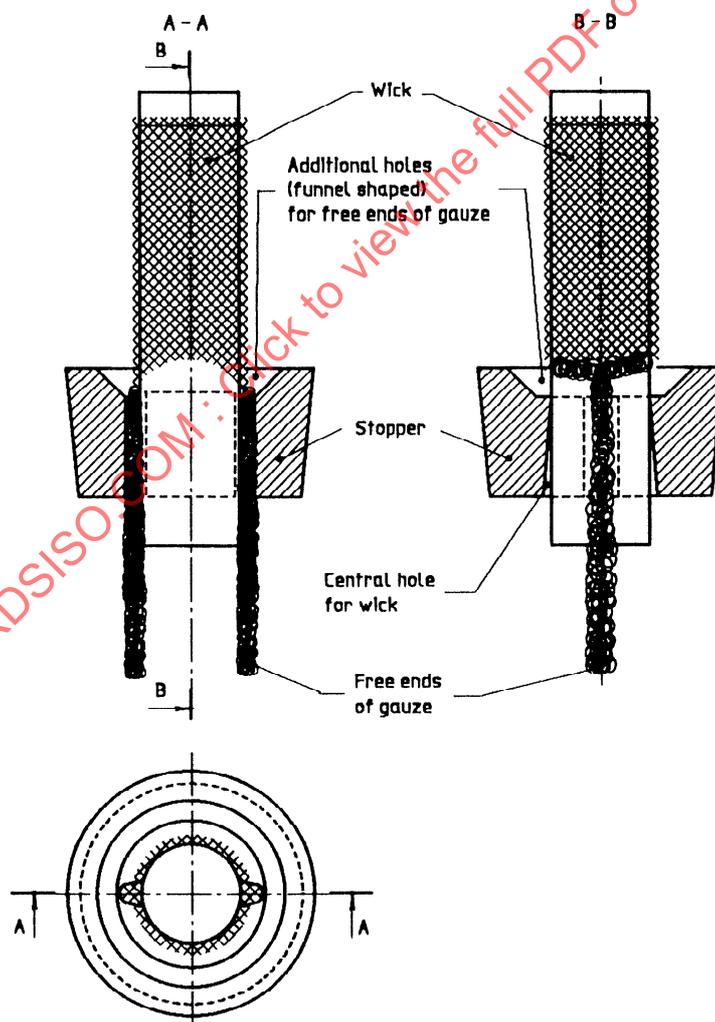


Figure 3 — Detail of rubber stopper

4.2.2 Exposure rack

The wet candle is exposed on a rack under the centre of a roof as shown in figure 4. The roof should be a square of 500 mm side, inert and opaque. The candle should be attached so that the distance from

the roof to the top of the wick is 200 mm and so that it is centred on the roof. The distance between the bottle and the ground level should be at least 1 m. The candle should be exposed towards the sea or other chloride source.

Dimensions in millimetres

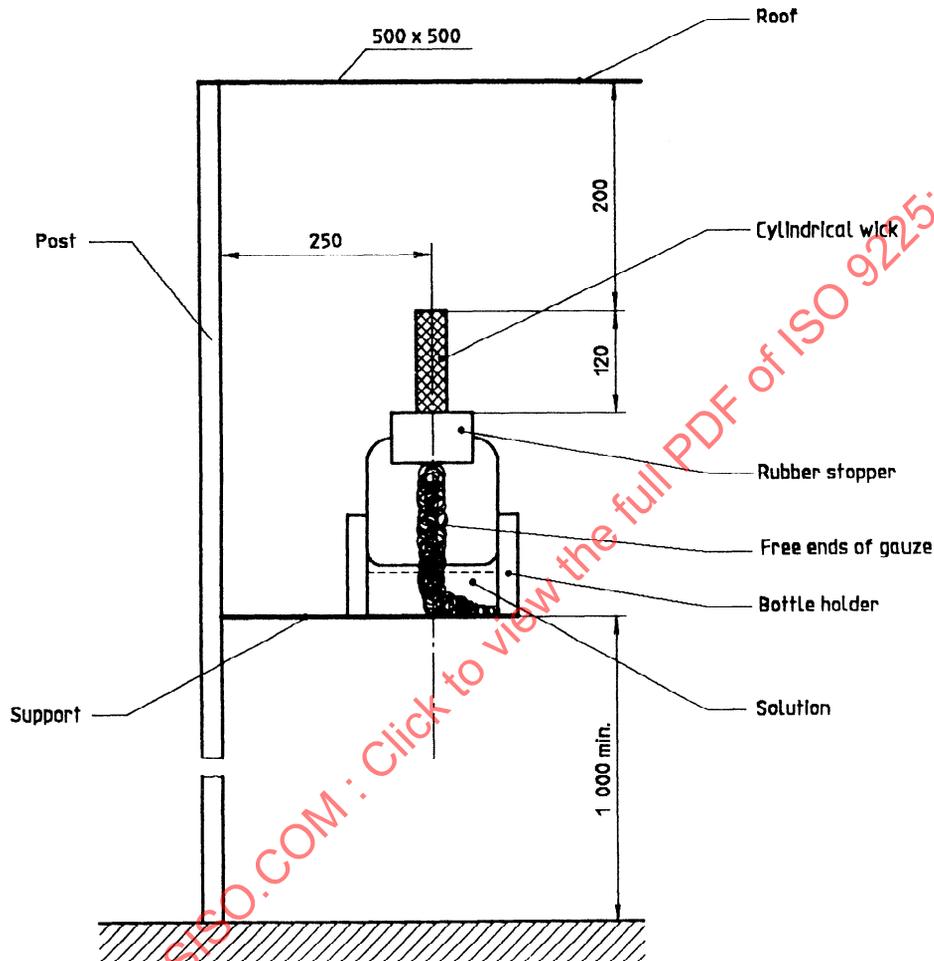


Figure 4 — Sampling apparatus assembly

4.3 Sampling

Install the prefabricated candle at the test location and carry out the following steps:

- a) adjust the length of the exposed part of the wick to the desired value;
- b) remove the stopper and wick from the bottle, wash the free ends of the gauze and the bottle with distilled water;
- c) place 200 ml of the glycerol and water solution (see 4.2.1) in the bottle;

- d) reassemble the wick and bottle;
- e) place the candle in the exposed position according to figure 4.

The glycerol and water solution should be changed at monthly intervals as follows:

- loosen the stopper in the bottle;
- carefully wash down the wick with at least 200 ml of distilled water, ensuring that the washings run through the drain holes in the stopper and thence into the bottle;