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**Determination of pollution for
evaluation of corrosivity of
atmospheres**

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sulphur dioxide, salinity, determination of content, chemical analysis
and testing, salts

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Foreword

This translation has been based on the original Japanese Industrial Standard established by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law.

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Determination of pollution for evaluation of corrosivity of atmospheres

Introduction This Japanese Industrial Standard was made based on the form and the contents of the original International Standard **ISO 9225** *Corrosion of metals and alloys — Corrosivity of atmospheres — Measurement of pollution* published in 1992 as the first edition. **ISO 9225** says “the ability of an atmosphere to cause corrosion of metals and alloys is controlled by the following factors. 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 (sea salt particle, Cl⁻)”.

In this Standard, the title “Determination of pollution for evaluation of corrosivity of atmospheres” is given and part of the specified contents is altered in accordance with the actual condition in our country.

The parts underlined (with dots) in this Standard are the matters modified from the contents specified in the original International Standard or not included therein.

1 Scope This Japanese Industrial Standard specifies the methods for the measurement of deposition rates of sulfur dioxide (sulfur oxide) in the air and airborne salinity (sea salt particle).

Remarks : The measurement methods included in this Standard apply to characterization of corrosivity of the test site.

WARNING : Some of the procedures included in this Japanese Industrial Standard entail the use of potentially hazardous chemicals. It is emphasized that all appropriate safety precautions should be taken.

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. The most recent editions of the standards indicated below shall be applied.

JIS K 0101 *Testing methods for industrial water*

JIS K 8101 *Ethanol (99.5)*

JIS K 8155 *Barium chloride dihydrate*

JIS K 8625 *Sodium carbonate*

JIS P 3801 *Filter paper (for chemical analysis)*

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

3.1 Principle Atmospheric sulfur dioxide (SO₂) reacts with the lead dioxide to form lead sulfate and is captured. The lead dioxide plate exposed in the air for a specific time is recovered and sulfate analysis is performed on this lead dioxide plate to determine the extent of sulfur dioxide capture. The deposition rate of sulfur dioxide is expressed in milligrams per square meter 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 lead dioxide plate is intended to minimize sulfur capture from acid precipitation or sulfuric acid aerosol.

3.2 Measuring apparatus

3.2.1 Lead dioxide plate Lead dioxide plates shall be prepared as follows :

- a) Bond filter paper circles (50 mm or 60 mm in diameter) to the bottom of polystyrene Petri dishes. 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 firmly with a glass rod so that it adheres completely to the dish. Allow the acetone to evaporate.
- b) Place the Petri dish to which a batch of filter papers are bonded (hereafter referred to as "plate") on a rack and rinse with distilled or deionized water. Fill the plates with distilled or deionized 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.
- c) 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.
- d) Pour the contents of the blender into a 1 liter beaker and pour 350 ml of the solution back into the blender. Pulp 3.5 g of filter paper, add it to the 350 mm of gum solution and set the blender at a moderate speed until the mixture appears smooth and uniform.
- e) Pour 400 ml of the gum solution prepared previously in c) and blend at a moderate speed for 1 min. Next, set the blender at a high speed and add 112 g of lead dioxide ⁽¹⁾. Blend for 2 min and then turn the blender back to a low speed.
- f) Carefully pipette 10 ml of the mixture into each 50 mm plate or 15 ml into each 60 mm plate in b). Make sure that the mixture spreads uniformly to the edge of each plate.
- g) Place the rack of plates in an oven set at 40 °C to 50 °C for 20 h to dry. Remove the plates from the oven, allow to cool and seal them with covers to protect them until exposure begins and store them.
- h) Number the plates and expose them within 120 days of preparation. Retain at least three plates from each batch for obtaining blank values.

Note ⁽¹⁾ As the activity of lead dioxide powder differs according to quality, it is recommended to use the standard products of U.K. – D.S.I.R. (Department of Scientific Industrial Research).

3.2.2 Exposure rack The exposure rack shall be as follows :

- a) The rack shall be able to hold the plates securely horizontally in an inverted position (facing the ground) (Figure 1 shows an example of rack) and shall be constructed from a material which has adequate resistance to atmospheric corrosion.

- b) The rack shall not be an obstruction to normal winds and air circulation currents to the plates.
- c) The rack shall be equipped with a retaining clip or other provision to hold the plate in the event of strong winds.

Unit : mm

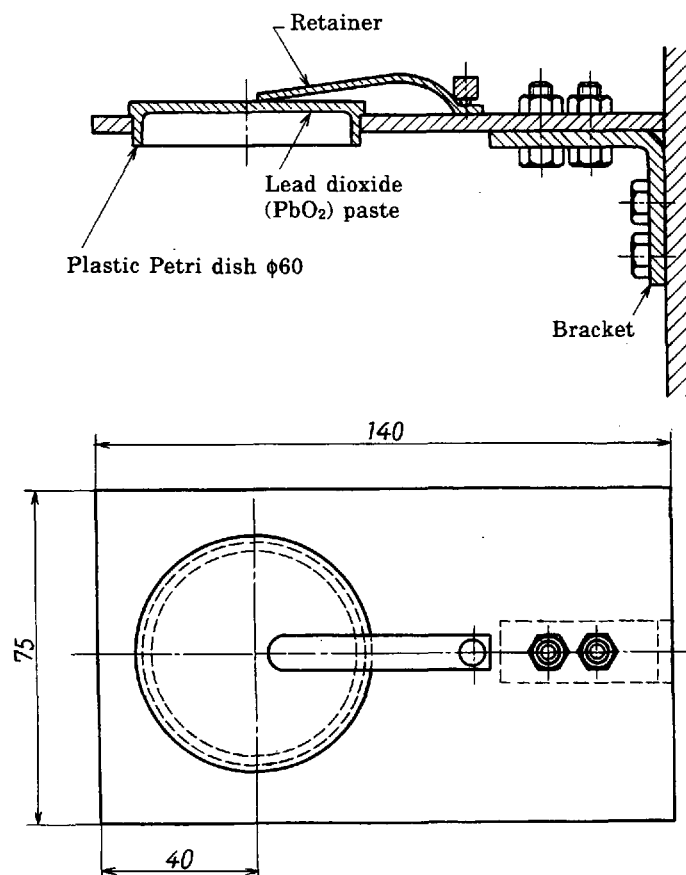


Fig. 1 Examples of sulfur dioxide plate and exposure rack

3.3 Exposure of plates The exposure of plates shall be as follows.

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) Fix the plates on the exposure rack in the direction facing the ground to exposure.
- b) The exposure period shall be one month starting usually on the first day of each month and removing the plate on the first day of the next month.
- c) After the end of the exposure period, the plates shall be removed from the rack and covered tightly to store until the time of analysis. At this time, the exposure location and the dates of exposure initiation and completion shall be recorded on the plates.

- d) Analysis of the plates shall be completed within 60 days of the completion of the exposure.

3.4 Analysis of sulfur dioxide (SO₂) and expression of deposition rate Sulfur dioxide (SO₂) analysis and the expression of deposition rate of the plates are usually as shown in the following, but the method specified in 4.4 is allowed.

3.4.1 Principle The contents of the 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.

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

- a) **Sodium carbonate** Approximately 50 g/l solution Dissolve (50 + 0.5) g of anhydrous sodium carbonate (Na₂CO₃) in 1 000 ml of water.
- b) **Hydrochloric acid (HCl) solution** 0.7 mol/l Dilute 60 ml of concentrated hydrochloric acid (ρ = 1.19 g/ml) to 1 000 ml of water.
- c) **Barium chloride dihydrate 5 g/l solution** Dissolve 5 g of barium chloride dihydrate (BaCl₂ · 2H₂O) in 1 000 ml of water.
- d) **Sodium sulfate standard solution corresponding to 500 mg of SO₄²⁻/l** Weigh, to the nearest 0.1 mg, 0.740 g of anhydrous sodium sulfate (Na₂SO₄), 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 SO₄²⁻.

3.4.3 Plate analysis The plate analysis shall be as follows :

- a) 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 [3.4.2 a)] and stir. Allow to stand for 3 h with occasional stirring.
- b) Place in a 100 °C water bath for 30 min and then cool and dilute to the mark with water.
- c) Filter at least 15 ml through a quantitative fine-grade filter paper into a clean, dry test tube.
- d) Pipette 10.0 ml of filtrated 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 [3.4.2 b)]. Swirl to mix and check the pH with indicator paper. The pH must be between 2.5 and 4.0. Take it as the sample solution. If not, discard and repeat this step.
- e) Pipette 5.0 ml aliquots of the sample into two test tubes of 25 mm diameter and add 15 ml of water to each.

- f) Add 1 ml of barium chloride solution [3.4.2 c)], mix vigorously and allow to stand for 5 min before measuring turbidity.
- g) 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 3.4.4.
- h) Unexposed plates taken from the same batch as of the exposed plates should be analyzed at the same time to obtain the blank value.

3.4.4 Preparation of the calibration curve The calibration curve shall be prepared as follows :

- a) Dilute 10.0 ml of the sodium sulfate solution [3.4.2 d)] to 100 ml using a volumetric flask.
- b) 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.
- c) Dilute each volume to 20 ml with water.
- d) Then follow the procedure specified in 3.4.3 to obtain each absorbance (turbidity).
- e) On 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, and prepare the calibration curve.

3.4.5 Expression of deposition rate Calculate by the following formula and express with $R_{(\text{SO}_2)}$ [mg/(m² · d)]

$$R_{(\text{SO}_2)} = \frac{(m_1 - m_0) \times 16.67}{A \cdot t \times 1000}$$

where, $R_{(\text{SO}_2)}$: deposition rate of sulfur dioxide [mg/(m² · d)]
 m_0 : mass of sulfates contained in an unexposed plate (µg)
 m_1 : mass of sulfates contained in an exposed plate (µg)
 A : area of a plate (m²)
 t : exposure period (d)

4 Determination of sulfur dioxide (SO₂) by lead dioxide (PbO₂) sulfation cylinder

4.1 Principle Atmospheric sulfur dioxide (SO₂) reacting with the lead dioxide to form sulfate is captured. The lead dioxide cylinder exposed in the air for a specific time is recovered and sulfate analysis is performed on this lead dioxide cylinder to determine the extent of sulfur dioxide capture. The deposition rate of sulfur dioxide is expressed in milligrams per square meter 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, into sulfate.