

## References

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## Appendix A

### Corrosion of metals and alloys — Corrosivity of atmospheres — Determination of corrosion rate of standard specimens for the evaluation of corrosivity

**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

This International Standard specifies methods which can be used for the determination of corrosion rate with standard specimens.

The values obtained from the measurements (corrosion rates for the first year of exposure) are to be used as classification criteria for the evaluation of atmospheric corrosivity according to ISO 9223.

#### 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 8407:1991, *Corrosion of metals and alloys — Removal of corrosion products from corrosion test specimens*.

ISO 8565:1992, *Metals and alloys — Atmospheric corrosion testing — General requirements for field tests*.

ISO 9223:1992, *Corrosion of metals and alloys — Corrosivity of atmospheres — Classification*.

#### 3 Principle

The corrosivity of the exposure locations or of industrial installation sites is deduced from the corrosion rate, calculated from the loss of mass per unit area of standard specimens following the descaling of corrosion products from the specimens after exposure periods of one year.

In the case of alloys of iron, zinc and copper, mass loss is a proven measure of corrosion damage. In the case of aluminium alloys, mass loss is a valid measure of corrosion. This is the aim of this International Standard, however it does not measure the corrosion penetration.

#### 4 Standard specimens

Two types of standard specimens may be used.

Helix specimens often give results which are significantly different from those obtained with flat specimens; therefore, comparisons of results should be based on specimens of the same type.

The materials used to prepare the standard specimens are of current fabrication, i.e.:

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Steel:	unalloyed carbon steel (Cu 0,03 % to 0,10 %, P < 0,07 %)
Zinc:	98,5 % min.
Copper:	99,5 % min.
Aluminium:	99,5 % min.

Prior to exposure, all specimens shall be solvent degreased. Steel specimens with visible rust stains or corrosion products on their surfaces shall be polished with 120 grit abrasive paper prior to degreasing to remove these visible corrosion products. Copper, zinc and aluminium specimens shall not be used if visible corrosion products are present before exposure.

### 4.1 Flat plate specimens

The specimens are rectangular plates with dimensions of preferably 100 mm × 150 mm but at least 50 mm × 100 mm, and a thickness of approximately 1 mm.

### 4.2 Open helix specimens

The materials used to prepare the standard specimens are wires intended for thermal spraying.

Wires with a diameter,  $d$ , of 2 mm to 3 mm are cut to a length of approximately 1 000 mm. They are then rolled into a helix using a rod with a diameter of 24 mm.

## 5 Exposure of standard specimens

The preparation and the exposure of the weighed and marked standard specimens shall be done according to the specifications of ISO 8565.

Three specimens of each metal should be exposed for one year, starting at the beginning of the worst corrosive period of the year. Helix specimens must be exposed in upright position (see figure 1).

After exposure, the corrosion products formed on specimens shall be removed in accordance with the specifications of ISO 8407 and reweighed to the nearest 0,1 mg. Procedures suitable for chemical cleaning are given in annex A. The cleaning procedure should be repeated several times in equal cleaning cycles.

## 6 Expression of results

The corrosion rate,  $r_{corr}$ , for each metal, expressed in grams per square metre year [ $\text{g}/(\text{m}^2\cdot\text{a})$ ], is given by the equation

$$r_{corr} = \frac{\Delta m}{A \cdot t} \quad \dots (1)$$

where

$\Delta m$  is the mass loss, in grams;

$A$  is the surface area, in square metres;

$t$  is the exposure time, in years.

The corrosion rate,  $r_{corr}$ , can also be expressed in micrometres per year ( $\mu\text{m}/\text{a}$ ), and is given by the equation

$$r_{corr} = \frac{\Delta m}{A \cdot \rho \cdot t} \quad \dots (2)$$

where

$\rho$  is the density, in grams per cubic centimetre, of the metal:

$$\begin{aligned} \rho_{Fe} &= 7,86 \text{ g/cm}^3 \\ \rho_{Zn} &= 7,14 \text{ g/cm}^3 \\ \rho_{Cu} &= 8,96 \text{ g/cm}^3 \\ \rho_{Al} &= 2,70 \text{ g/cm}^3 \end{aligned}$$

$\Delta m$ ,  $A$  and  $t$  have the same meaning as the symbols in equation (1).

The corrosion rate for open helix specimens,  $r_{corr}$ , expressed in micrometres per year ( $\mu\text{m}/\text{a}$ ), is given by the equation

$$r_{corr} = 0,25 \times \frac{\Delta m \cdot d}{m \cdot t} \quad \dots (3)$$

where

$\Delta m$  is the mass loss, in milligrams;

$d$  is the wire diameter, in millimetres;

$m$  is the original mass, in grams;

$t$  is the exposure time, in years.

All single values and their mean values shall be represented in the test report.

## Appendix A

Material	Chemical	Time min	Temperature °C	Remarks
Steel	500 ml of hydrochloric acid (HCl, $\rho = 1,19$ g/ml) 3,5 g of hexamethylene tetramine Distilled water to make up to 1 000 ml	10	20 to 25	—
Zinc	200 g of chromium trioxide (CrO <sub>3</sub> ) Distilled water to make up to 1 000 ml	1	80	Chloride contamination of the chromic acid from corrosion products formed in salt environments should be avoided to prevent attack of the zinc base metal.
Copper	54 ml of sulfuric acid (H <sub>2</sub> SO <sub>4</sub> , $\rho = 1,84$ g/ml) Distilled water to make up to 1 000 ml	30 to 60	40 to 50	Deaerate solution with nitrogen. Brushing of test specimens to remove corrosion products followed by re-immersion for 3 s to 4 s is recommended.
Aluminium	50 ml of phosphoric acid (H <sub>3</sub> PO <sub>4</sub> , $\rho = 1,69$ g/ml) 20 g of chromium trioxide (CrO <sub>3</sub> ) Distilled water to make up to 1 000 ml	5 to 10	90 to 95	If corrosion product films remain, then follow with the nitric acid procedure below.
	Nitric acid (HNO <sub>3</sub> , $\rho = 1,42$ g/ml)	1 to 5	20 to 25	Remove extraneous deposits and bulky corrosion products to avoid reactions that may result in excessive removal of base metal.

## Appendix B

### 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 (SO<sub>2</sub>) (sulfur compounds) and airborne salinity.

It does not cover concentration measurements. The concentration of sulfur dioxide (SO<sub>2</sub>) 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 (SO<sub>2</sub>) deposition rate on lead dioxide (PbO<sub>2</sub>) sulfation plates

##### 2.1 Principle

Atmospheric sulfur dioxide (SO<sub>2</sub>) reacts with the lead dioxide to form lead sulfate. The plates are recovered 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/(m<sup>2</sup>·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.

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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

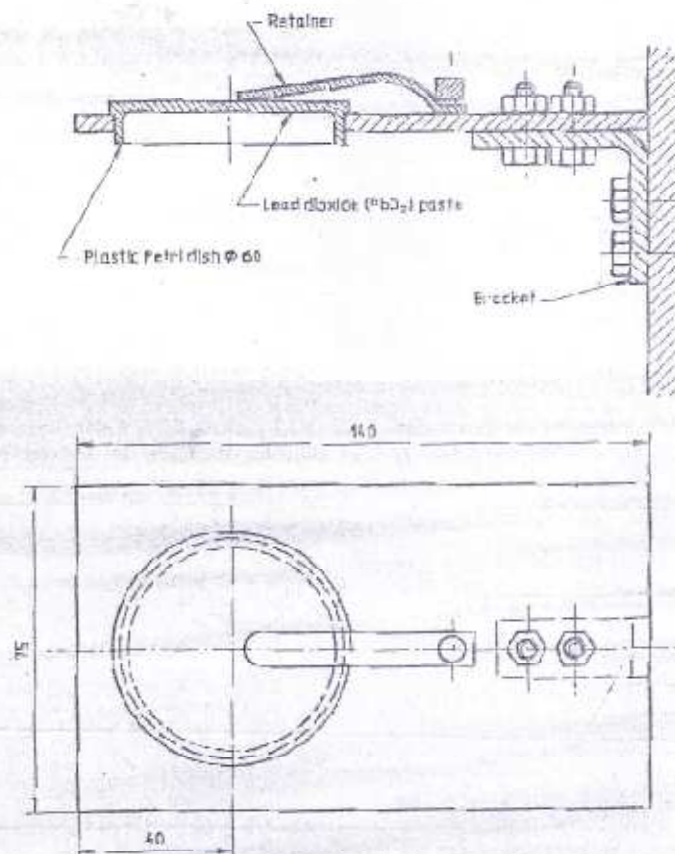


Figure 1 — Sulfation plate holder



## Appendix B

### 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 ( $\text{Na}_2\text{CO}_3$ ) 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 $\text{SO}_4^{2-}$ per litre.

Weigh, to the nearest 0,1 mg, 0,740 g of anhydrous sodium sulfate ( $\text{Na}_2\text{SO}_4$ ), 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  $\mu\text{g}$  of  $\text{SO}_4^{2-}$ .

#### 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  $\mu\text{g}$ , 100  $\mu\text{g}$ , 150  $\mu\text{g}$ , 200  $\mu\text{g}$ , 250  $\mu\text{g}$ , 500  $\mu\text{g}$  and 750  $\mu\text{g}$ .

## Appendix B

### 2.5 Expression of results

The sulfation rate is calculated in terms of sulfur dioxide ( $\text{SO}_2$ ) 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 ( $\text{SO}_2$ ), expressed in milligrams per square metre day [ $\text{mg}/(\text{m}^2 \text{d})$ ],  $R_{\text{SO}_2}$ , is given by the equation

$$R_{\text{SO}_2} = \frac{(m_1 - m_0) \times 16,67}{A \cdot t \times 1.000} \quad \dots (1)$$

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.

## Appendix C

### 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 [ $\text{mg}/(\text{m}^2 \cdot \text{d})$ ].

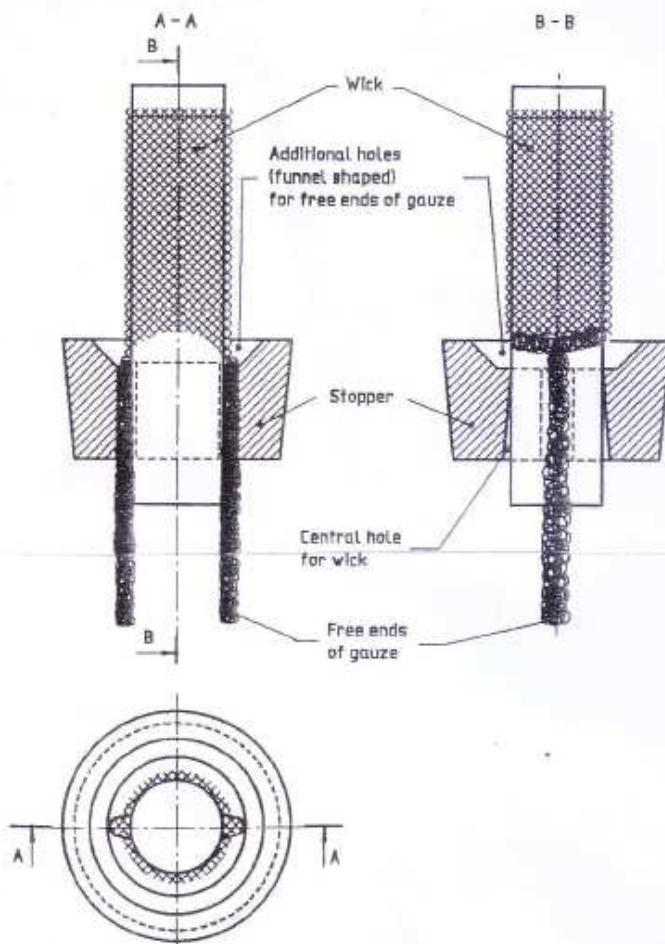


Figure 3 — Detail of rubber stopper

#### 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  $100 \text{ cm}^2$ , 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 [ $\text{CHOH}(\text{CH}_2\text{OH})_2$ ] with distilled water to a volume of 1 000 ml. To the solution is added 20 drops of octanoic acid ( $\text{C}_8\text{H}_{16}\text{O}_2$ ).

In situations of prolonged extreme temperatures, i.e. greater than  $25^\circ\text{C}$  or less than  $-25^\circ\text{C}$ , 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.

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### 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.

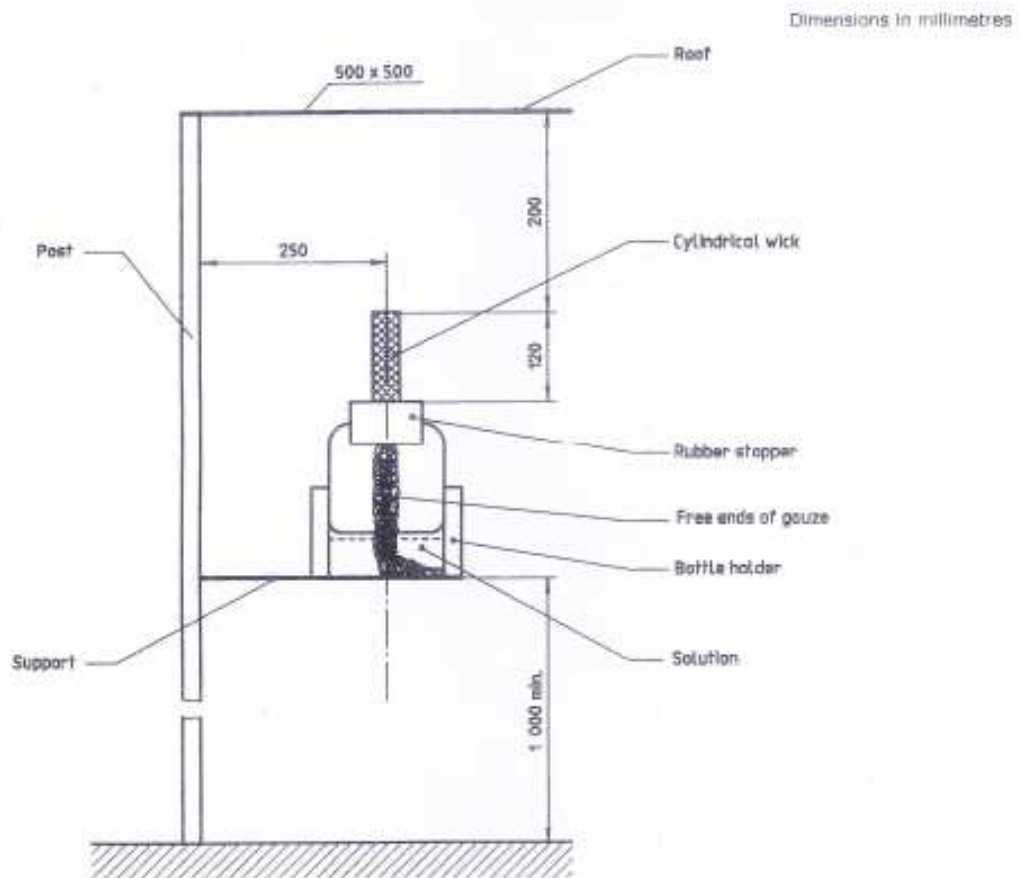


Figure 4 — Sampling apparatus assembly

### 4.3 Sampling

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

- adjust the length of the exposed part of the wick to the desired value;
- remove the stopper and wick from the bottle, wash the free ends of the gauze and the bottle with distilled water;
- 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;

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- remove the stopper and wick from the bottle and wash the free ends of the gauze as they are withdrawn from the bottle;
- place the stopper and wick into a new bottle containing 200 ml of fresh glycerol and water solution, ensuring that the free ends of the gauze dip well into the solution; replace the candle in its holder;
- screw on the lid of the removed bottle tightly, making sure that it does not leak.

Mark the bottle clearly with the test site name, location and dates of exposure and removal.

The solution in the bottle is prepared for analysis according to 4.4.3.

### 4.4 Chloride analysis

A suitable chloride analysis is described below. Any other analytical procedure which provides satisfactory precision can be used.

#### 4.4.1 Principle

The amount of chloride in a sample is determined by mercurimetric titration using diphenylcarbazone/bromophenol blue mixed indicator. The endpoint of the titration is indicated by the formation of a blue-violet complex of mercuric diphenylcarbazone in the pH interval 2,3 - 2,8.

#### 4.4.2 Reagents

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

##### 4.4.2.1 Ethanol, 95 % (V/V).

##### 4.4.2.2 Nitric acid, solution, $c(\text{HNO}_3) = 0,05 \text{ mol/l}$ .

Dilute 3 ml of nitric acid ( $\rho = 1,40 \text{ g/ml}$ ) to 1 000 ml with water.

##### 4.4.2.3 Sodium hydroxide, solution, $c(\text{NaOH}) = 0,25 \text{ mol/l}$ .

Dissolve 10 g of sodium hydroxide in water and dilute to 1 000 ml.

##### 4.4.2.4 Sodium chloride, standard reference solution, $c(\text{NaCl}) = 0,025 \text{ mol/l}$ .

Dry the sodium chloride for 1 h at 300 °C. Dissolve 1,4613 g in water and dilute to 1 000 ml in a volumetric flask.

##### 4.4.2.5 Mercury(II) nitrate, standard volumetric solution, $c[\text{Hg}(\text{NO}_3)_2] = 0,0125 \text{ mol/l}$ .

Dissolve 4,2830 g of mercury(II) nitrate monohydrate  $[\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}]$  in 50 ml of water acidified with 0,5 ml of nitric acid ( $\text{HNO}_3$ ) ( $\rho = 1,40 \text{ g/ml}$ ). Dilute to 1 000 ml with water in a volumetric flask. Filter if necessary and determine the exact concentration by titration using sodium chloride standard reference solution (4.4.2.4) as described in 4.4.3.

##### 4.4.2.6 Mercury(II) nitrate, standard volumetric solution, $c[\text{Hg}(\text{NO}_3)_2] = 0,00705 \text{ mol/l}$ .

Dissolve 2,4200 g of mercury(II) monohydrate  $[\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}]$  in 25 ml of water acidified with 0,25 ml of nitric acid ( $\rho = 1,40 \text{ g/ml}$ ). Dilute to 1 000 ml with water in a volumetric flask. Filter if necessary and determine the exact concentration by titration using sodium chloride standard volumetric solution (4.4.2.4) as described in 4.4.3.

#### 4.4.2.7 Mixed indicator solution.

Dissolve 0,5 g of diphenylcarbazone and 0,5 g of bromophenol blue in 75 ml of ethanol (4.4.2.1) and dilute to 100 ml with ethanol. Keep in a dark bottle in a refrigerator.

### 4.4.3 Analysis

Sample a known aliquot of the sample prepared according to 4.3 and containing not more than 20 mg of chloride ions. Determine the approximate content of chloride ions in the sample solution by preliminary titration using mercury(II) nitrate (4.4.2.5).

If the aliquot of the sample solution contains more than 2,5 mg of chloride ions, perform the titration using mercury(II) nitrate standard volumetric solution (4.4.2.5).

If the aliquot of the sample solution contains less than 2,5 mg of chloride ions, perform the titration using mercury(II) nitrate standard volumetric solution (4.4.2.6) and a microburette. In this case, perform a blank titration on 50 ml of water. If the aliquot contains less than 0,1 mg of chloride ions per litre, evaporate a suitable volume to 50 ml.

Add 5 to 10 drops of mixed indicator solution (4.4.2.7).

If a blue-violet or red colour develops add nitric acid solution (4.4.2.2) drop by drop until the colour changes to yellow and then add 1 ml more of the nitric acid solution. If a yellow or orange colour forms immediately when the indicator is added, develop the blue-violet colour by adding sodium hydroxide solution (4.4.2.3) drop by drop, then proceed with the acidification.

## Appendix D

# Corrosion of metals and alloys – Removal of corrosion products from corrosion test specimens

## 1 Scope

1.1 This International Standard specifies procedures for the removal of corrosion products formed on metal and alloy corrosion test specimens during their exposure in corrosive environments. For the purpose of this International Standard, the term "metals" refers to metals and alloys.

1.2 The procedures specified are designed to remove all corrosion products without significant removal of base metal. This allows an accurate determination of the mass loss of the metal which occurred during exposure to the corrosive environment.

1.3 These procedures may, in some cases, also be applied to metal coatings. However, possible effects from the substrate must be considered.

## 2 Procedures

### 2.1 General

2.1.1 A light mechanical cleaning treatment by brushing with a soft bristle brush under running water should first be applied to remove lightly adherent or bulky corrosion products. In some cases, this treatment will be sufficient to remove all corrosion products and no further treatment will be necessary.

2.1.2 If the treatment described in 2.1.1 does not remove all corrosion products it will be necessary to use other procedures. These are of three types:

- a) chemical;
- b) electrolytic;
- c) more vigorous mechanical treatments.

Whichever method is used, it may be necessary to repeat the cleaning treatment to ensure complete

removal of corrosion products. Removal shall be confirmed by visual examination. The use of a low-power microscope (i.e.  $\times 7$  to  $\times 30$ ) is particularly helpful with a pitted surface when corrosion products may accumulate in pits.

2.1.3 An ideal procedure should remove only corrosion products and not result in removal of any base metal. Two procedures can be used to confirm this point. One procedure uses a control specimen (2.1.3.1) and the other requires a certain number of cleaning cycles on the corroded specimen (2.1.3.2).

2.1.3.1 Uncorroded control specimens which should be similar chemically, metallurgically, and geometrically to the test specimens, should be cleaned by the same procedure as used for the test specimen. By weighing the control specimen before and after cleaning (weighing to the fifth significant figure is suggested, e.g. a 70 g specimen should be weighed to three decimal places), the metal loss resulting from the cleaning may be determined. The mass loss of the control specimen will reflect the mass loss of test specimens resulting from the cleaning procedure.

2.1.3.2 The cleaning procedure should be repeated several times with the corroded test specimen after removal of corrosion products is completed. The mass can be shown on a graph as a function of the number of equal cleaning cycles (see figure 1). In many cases the masses obtained in these cleaning cycles (applied after removal of corrosion products) will have a linear relation with cleaning cycles. Two lines, AB and BC, will be obtained. Line AB characterizes the removal of corrosion products and may not always be visible. Line BC characterizes the removal of substrate after the corrosion products are gone. By extrapolation of line BC to the ordinate axis, we obtain point D which characterizes the mass at zero number of cleaning cycles. In other cases the relation may not be linear and the most appropriate extrapolation shall then be made.

## Appendix D

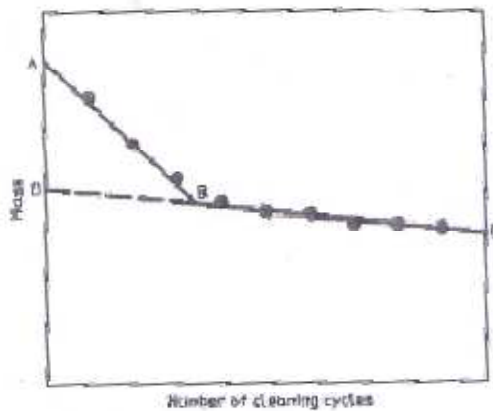


Figure 1 — Mass of corroded specimens after repetitive cleaning cycles

2.1.3.3 The true mass of the specimen after removal of the corrosion products will be between points B and D, depending on the degree of protection furnished by the corrosion products during the cleaning procedure.

2.1.4 The preferred cleaning method will be that which

- provides efficient removal of corrosion products;
- provides low or zero mass loss when applied to new uncorroded specimens (see 2.1.3.1);
- provides a curve of mass as a function of the number of cleaning cycles, which is close to horizontal when the latter is plotted as the abscissa (see 2.1.3.2).

2.1.5 When chemical or electrolytic procedures are used, solutions freshly prepared with distilled or deionized water and reagent grade chemicals shall be used.

2.1.6 After cleaning, the metal specimen should be thoroughly rinsed, first with tap water and finally with distilled water. Then the specimen shall be dried in an oven and allowed to cool in a desiccator to the balance room temperature before weighing. An alternative drying procedure is to immerse the metal specimen in an ethanol solution and then dry with an air hand blower.

### 2.2 Chemical procedure

Chemical procedures involve immersion of the corrosion test specimen in a chemical solution which is specifically designed to remove the corrosion pro-

ducts with minimal dissolution of any base metal. Several procedures are listed in annex A (see table A.1).

2.2.1 Chemical cleaning is often preceded by light brushing of the test specimen to remove lightly adherent, bulky corrosion products.

2.2.2 Intermittent removal of specimens from the chemical solution for light brushing can often facilitate the removal of lightly adherent corrosion products.

2.2.3 Chemical cleaning is often followed by light brushing to remove loose products.

### 2.3 Electrolytic procedures

Electrolytic cleaning also can be used to remove corrosion products. Several methods of electrolytic cleaning of corrosion test specimens are given in annex A (see table A.2).

Electrolytic cleaning should be preceded by light brushing of the test specimen to remove lightly adherent, bulky corrosion products. Brushing should also follow electrolytic cleaning to remove any loose slime or deposits. This will help to minimize any redeposition of metal from reducible corrosion products, which would reduce the mass loss.

### 2.4 Mechanical procedures

Mechanical procedures can include scraping, scrubbing, brushing, ultrasonic methods, mechanical shock and impact blasting (grit blasting, water-jet blasting, etc.). These methods are often used to remove heavily encrusted corrosion products. Scrubbing with a bristle brush and a mild abrasive/distilled water slurry can also be used to remove corrosion products.

Vigorous mechanical cleaning will result in the removal of some base metal, so care should be exercised. These methods should be used only when others fail to provide adequate removal of corrosion products. As with the other methods, correction for metal loss due to the cleaning method is recommended. The mechanical forces used in cleaning shall be maintained as nearly constant as possible.

## 3 Test report

The test report shall include the following information:

- reference to this International Standard;
- the procedure used to remove corrosion products;

## Appendix D

- c) for chemical procedures, the composition and concentration of chemicals employed, the solution temperature and the duration of cleaning;
- d) for electrolytic procedures, the composition and concentration of chemicals employed, the solution temperature, the anode material and current density, and the duration of cleaning;
- e) for mechanical procedures, the specific mechanical method employed (bristle brush scrubbing, wooden scraper, etc.), any abrasive compounds used, and the duration of cleaning;
- f) where multiple procedures are used, the appropriate details for each method and the sequence of methods;
- g) the results of control cleaning (see 2.1.3.1) or from repetitive cleaning steps (see 2.1.3.2) designed to assess mass loss from the removal of base metal during the cleaning process;
- h) the mass loss due to corrosion (see 2.1.3.3).





## Appendix E

### Macro Program Used to find out the best fit value of Constants

```
Sub looping()
Cells(1, 10) = 1
Cells(4, 10) = 1
Cells(1, 11) = .1
Cells(4, 11) = 0.1
Cells(1, 12) = 1
Cells(4, 12) = 1
Cells(1, 13) = 0.1
Cells(4, 13) = 0.1
Cells(4, 14) = 1
Cells(4, 14) = 1
Cells(4, 15) = .1
Cells(4, 15) = .1
Cells(4, 16) = 1
Cells(4, 16) = 1
Cells(4, 17) = .1
Cells(4, 17) = .1
Cells(1, 18) = 0.001
Cells(4, 18) = 0.001
Cells(1, 19) = 1
Cells(4, 19) = 1
Cells(1, 26) = 1
Cells(4, 26) = 1
Cells(1, 27) = .1
Cells(4, 27) = .1
Cells(3, 24) = 1000
For p = 1 To 1000 Step 1
For a = -0.1 To 0.1 Step 0.1
Cells(4, 10) = Cells(4, 10) + a
If Cells(5, 24) < Cells(3, 24) Then Cells(1, 10) = Cells(4, 10)
If Cells(5, 24) < Cells(3, 24) Then Cells(3, 24) = Cells(5, 24)
Cells(4, 10) = Cells(4, 10) - a
Next a
Cells(4, 10) = Cells(1, 10)

For b = -0.1 To 0.1 Step 0.1
Cells(4, 11) = Cells(4, 11) + b
If Cells(5, 24) < Cells(3, 24) Then Cells(1, 11) = Cells(4, 11)
If Cells(5, 24) < Cells(3, 24) Then Cells(3, 24) = Cells(5, 24)
Cells(4, 11) = Cells(4, 11) - b
Next b
Cells(4, 11) = Cells(1, 11)
```

## Appendix E

For c = -0.1 To 0.1 Step 0.1  
Cells(4, 12) = Cells(4, 12) + c  
If Cells(5, 24) < Cells(3, 24) Then Cells(1, 12) = Cells(4, 12)  
If Cells(5, 24) < Cells(3, 24) Then Cells(3, 24) = Cells(5, 24)  
Cells(4, 12) = Cells(4, 12) - c  
Next c  
Cells(4, 12) = Cells(1, 12)

For d = -0.1 To 0.1 Step 0.1  
Cells(4, 13) = Cells(4, 13) + d  
If Cells(5, 24) < Cells(3, 24) Then Cells(1, 13) = Cells(4, 13)  
If Cells(5, 24) < Cells(3, 24) Then Cells(3, 24) = Cells(5, 24)  
Cells(4, 13) = Cells(4, 13) - d  
Next d  
Cells(4, 13) = Cells(1, 13)

For e = -5 To 5 Step 5  
Cells(4, 14) = Cells(4, 14) + e  
If Cells(5, 24) < Cells(3, 24) Then Cells(1, 14) = Cells(4, 14)  
If Cells(5, 24) < Cells(3, 24) Then Cells(3, 24) = Cells(5, 24)  
Cells(4, 14) = Cells(4, 14) - e  
Next e  
Cells(4, 14) = Cells(1, 14)

For f = -0.1 To 0.1 Step 0.1  
Cells(4, 15) = Cells(4, 15) + f  
If Cells(5, 24) < Cells(3, 24) Then Cells(1, 15) = Cells(4, 15)  
If Cells(5, 24) < Cells(3, 24) Then Cells(3, 24) = Cells(5, 24)  
Cells(4, 15) = Cells(4, 15) - f  
Next f  
Cells(4, 15) = Cells(1, 15)

For g = -05 To 5 Step 5  
Cells(4, 16) = Cells(4, 16) + g  
If Cells(5, 24) < Cells(3, 24) Then Cells(1, 16) = Cells(4, 16)  
If Cells(5, 24) < Cells(3, 24) Then Cells(3, 24) = Cells(5, 24)  
Cells(4, 16) = Cells(4, 16) - g  
Next g  
Cells(4, 16) = Cells(1, 16)

## Appendix E

```
For h = -0.1 To 0.1 Step 0.1
Cells(4, 17) = Cells(4, 17) + h
If Cells(5, 24) < Cells(3, 24) Then Cells(1, 17) = Cells(4, 17)
If Cells(5, 24) < Cells(3, 24) Then Cells(3, 24) = Cells(5, 24)
Cells(4, 17) = Cells(4, 17) - h
Next h
Cells(4, 17) = Cells(1, 17)
```

```
For j = -0.1 To 0.1 Step 0.1
Cells(4, 18) = Cells(4, 18) + j
If Cells(5, 24) < Cells(3, 24) Then Cells(1, 18) = Cells(4, 18)
If Cells(5, 24) < Cells(3, 24) Then Cells(3, 24) = Cells(5, 24)
Cells(4, 18) = Cells(4, 18) - j
Next j
Cells(4, 18) = Cells(1, 18)
```

```
For t = -0.1 To 0.1 Step 0.1
Cells(4, 19) = Cells(4, 19) + t
If Cells(5, 24) < Cells(3, 24) Then Cells(1, 19) = Cells(4, 19)
If Cells(5, 24) < Cells(3, 24) Then Cells(3, 24) = Cells(5, 24)
Cells(4, 19) = Cells(4, 19) - t
Next t
Cells(4, 19) = Cells(1, 19)
```

```
For k = -5 To 5 Step 0.5
Cells(4, 26) = Cells(4, 26) + k
If Cells(5, 24) < Cells(3, 24) Then Cells(1, 26) = Cells(4, 26)
If Cells(5, 24) < Cells(3, 24) Then Cells(3, 24) = Cells(5, 24)
Cells(4, 26) = Cells(4, 26) - k
Next k
Cells(4, 26) = Cells(1, 26)
```

```
For l = -0.1 To 0.1 Step 0.1
Cells(4, 27) = Cells(4, 27) + l
If Cells(5, 24) < Cells(3, 24) Then Cells(1, 27) = Cells(4, 27)
If Cells(5, 24) < Cells(3, 24) Then Cells(3, 24) = Cells(5, 24)
Cells(4, 27) = Cells(4, 27) - l
Next l
Cells(4, 27) = Cells(1, 27)
```

```
Next p
End Sub
```