



# Standard Practice for Monitoring Atmospheric SO<sub>2</sub> Using the Sulfation Plate Technique<sup>1</sup>

This standard is issued under the fixed designation G 91; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice provides a weighted average effective SO<sub>2</sub> level for a 30-day interval through the use of the sulfation plate method, a technique for estimating the effective SO<sub>2</sub> content of the atmosphere, and especially with regard to the atmospheric corrosion of stationary structures or panels. This practice is aimed at determining SO<sub>2</sub> levels rather than sulfuric acid aerosol or acid precipitation.

1.2 The results of this practice correlate approximately with volumetric SO<sub>2</sub> concentrations, although the presence of dew or condensed moisture tends to enhance the capture of SO<sub>2</sub> into the plate.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 516 Test Methods for Sulfate Ion in Water<sup>2</sup>

D 2010/D2010 M Test Method for Evaluation of Total Sulfation Activity in the Atmosphere by the Lead Dioxide Technique<sup>3</sup>

G 16 Guide for Applying Statistics to Analysis of Corrosion Data<sup>4</sup>

## 3. Summary of Practice

3.1 Sulfation plates consisting of a lead peroxide reagent in an inverted dish are exposed for 30-day intervals. The plates are recovered and sulfate analyses performed on the contents to determine the extent of sulfur capture. The results are reported in terms of milligrams of SO<sub>2</sub> per square metre per day.

## 4. Significance and Use

4.1 Atmospheric corrosion of metallic materials is a func-

tion of many weather and atmospheric variables. The effect of specific corrodants, such as sulfur dioxide, can accelerate the atmospheric corrosion of metals significantly. The sulfation plate method provides a simple technique to independently monitor the level of SO<sub>2</sub> in the atmosphere to yield a weighted average result.

4.2 Sulfation plate results may be used to characterize atmospheric corrosion test sites regarding the effective average level of SO<sub>2</sub> in the atmosphere at these locations.

4.3 Sulfation plate testing is useful in determining microclimate, seasonal, and long term variations in the effective average level of SO<sub>2</sub>.

4.4 The results of sulfation plate tests may be used in correlations of atmospheric corrosion rates with atmospheric data to determine the sensitivity of the corrosion rate to SO<sub>2</sub> level.

4.5 The sulfation plate method may also be used with other methods to characterize the atmosphere at sites where buildings or other construction is planned in order to determine the extent of protective measures required for metallic materials.

## 5. Interferences

5.1 The lead peroxide reagent used in this practice may convert other compounds such as mercaptans, hydrogen sulfide, and carbonyl sulfide into sulfate.

NOTE 1—Hydrogen sulfide and mercaptans, at concentrations which affect the corrosion of structural metals significantly, are relatively rare in most atmospheric environments, but their effects regarding the corrosion of metals are not equivalent to sulfur dioxide. Therefore, if H<sub>2</sub>S, COS, or mercaptans are present in the atmosphere, the lead peroxide method must not be used to assess atmospheric corrosivity. It should also be noted that no actual measurements have been made which would establish the correlation between atmospheric H<sub>2</sub>S, COS, or mercaptan level and sulfation as measured by this practice.

5.2 The inverted exposure position of the sulfation plate is intended to minimize capture of sulfuric acid aerosols and sulfur bearing species from precipitation.

## 6. Sulfation Plate Preparation and Exposure

6.1 Sulfation plates can be prepared according to the method of Huey.<sup>5</sup> The plate preparation method is given in

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.04 on Atmospheric Corrosion.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.03.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 03.02.

<sup>5</sup> Huey, N. A., "The Lead Dioxide Estimation of Sulfur Dioxide Pollution," *Journal of the Air Pollution Control Association*, Vol 18, No. 9, 1968, pp. 610–611.

Appendix X1. Laboratory prepared plates should be exposed within 120 days of preparation.

6.2 In general, the level of atmospheric sulfur dioxide varies seasonally during the year so that a minimal exposure program requires four 30-day exposures each year at roughly equal intervals. In order to establish the atmospheric SO<sub>2</sub> level at an atmospheric corrosion test site which has not been monitored previously, a program in which six 30-day exposures per year for a period of 3 years is recommended. More extensive testing may be desirable if large variability is encountered in the results. Thereafter, the location should be monitored with at least four tests in a 1-year period every 3 years. If the subsequent tests are not consistent with the initial testing, then another 3-year program of six tests per year is required. Also, if a major change in the general area occurs in terms of industrial or urban development, then six tests per year for 3 years should again be carried out.

6.3 In monitoring exposure sites, a minimum of four plates shall be used for each exposure period.

6.3.1 Sites which have a grade or elevation variation should be monitored with at least two plates at the highest elevation and two plates at the lowest elevation.

6.3.2 Plates should be exposed, if possible, at both the highest and lowest level above the ground at which corrosion test specimens are exposed.

6.3.3 Sites larger than 10 000 m<sup>2</sup> shall have at least eight plates exposed for each period. In rectangular sites on level ground, it is desirable to expose two plates at each corner.

NOTE 2—Some investigators have reported significantly higher sulfation results at locations closest to the ground.

6.4 Brackets shall be used to hold the plates securely in an inverted position so that the lead peroxide mixture faces downward. The plate shall be horizontal and shall be placed so that it is not protected from normal winds and air currents. The bracket design should include a retaining clip or other provision to hold the plate in the event of strong winds. The retainer clip may be made from stainless steel, spring bronze, hard aluminum alloy (3003H19), or other alloys with sufficient strength and atmospheric corrosion resistance. A typical bracket design is shown in Fig. 1.

6.5 A 30 ± 2-day exposure period is recommended. At the conclusion of this 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. The plate identification, exposure location, and exposure initiation date should be recorded when the plate exposure is initiated. At the termination of exposure, the completion date should be added to the exposure records.

NOTE 3—The 30 day exposure is not very discriminating in areas of low SO<sub>2</sub> concentrations. Experience has shown that 60 to 90 days exposure may be necessary to develop a measurable SO<sub>2</sub> capture on the plate.

6.6 The sulfation plates shall be analyzed for sulfate content using any established quantitative analysis technique.

NOTE 4—In conducting the sulfate analysis, it is necessary to remove the contents of the sulfation plate and solubilize the sulfate, for example, using a solution of sodium carbonate. It has been found that 20 mL of 50

g/L Na<sub>2</sub>CO<sub>3</sub> (ACS reagent grade) is sufficient to solubilize the sulfate in this test method in a 3-hour period. Thereafter, conventional sulfate analysis can be employed, for example, by barium precipitation and either gravimetric or turbidimetric analysis (see Test Methods D 516).

## 7. Calculation

7.1 The sulfate analysis provides the quantity of sulfate on each disc analyzed. This should be converted to an SO<sub>2</sub> capture rate, *R*, by the following equation.

$$R = (m - m_0) \times MWSO_2 / MWSO_4 \times A \times T \quad (1)$$

where:

- m* = mass of sulfate found in the plate, mg,
- m*<sub>0</sub> = mass of sulfate found in a blank (unexposed) plate, mg,
- MWSO*<sub>2</sub> = 64,
- MWSO*<sub>4</sub> = 96,
- A* = area of the plate, m<sup>2</sup>, and
- T* = exposure time of the plate, days.

$$R = SO_2 \text{ capture rate, mg SO}_2/\text{m}^2 \text{ day} \quad (2)$$

7.2 The SO<sub>2</sub> capture rate may be converted to equivalent SO<sub>3</sub> or SO<sub>4</sub> values if desired, but for comparison purposes, SO<sub>2</sub> rates shall be used.

7.3 The average value and standard deviation of the values should be calculated according to Guide G 16.

## 8. Report

8.1 The report shall include the following information:

8.1.1 A description of the exposure site and the locations where the plates were exposed, including the bracket identity number or designation and the location on the exposure stand,

8.1.2 The exposure initiation and termination dates,

8.1.3 The identification numbers and sources of the sulfation plates,

8.1.4 The calculated SO<sub>2</sub> capture rates for each plate and the average and standard deviations for each site and exposure interval,

8.1.5 The sulfate analysis method, and

8.1.6 Any deviations from this practice.

8.2 Comparison should be made to previously determined values in ongoing monitoring programs.

## 9. Precision and Bias

9.1 Repeatability for a group of plates prepared in one batch and exposed for 30 days under essentially identical conditions, the standard deviation<sup>6</sup> has been found to be related to the average sulfation level by the equation given below:

$$\sigma = 0.0790 \bar{m} \quad (3)$$

where:

$\sigma$  = standard deviation of the plate SO<sub>2</sub> capture in mg SO<sub>2</sub>/m<sup>2</sup>day,

*m* = average net SO<sub>2</sub> capture in mg SO<sub>2</sub>/m<sup>2</sup> day.

This relationship was determined in 10 runs with 6 or more plates per run. The standard error of estimate of the regression

<sup>6</sup> Levadie, B., "Sampling and Analysis of Atmospheric Sulfur Dioxide with the Lead Dioxide Plate (Huey Plate)," *Journal of Testing and Evaluation*, Vol 7, No. 2, March 1979, pp. 61-67.

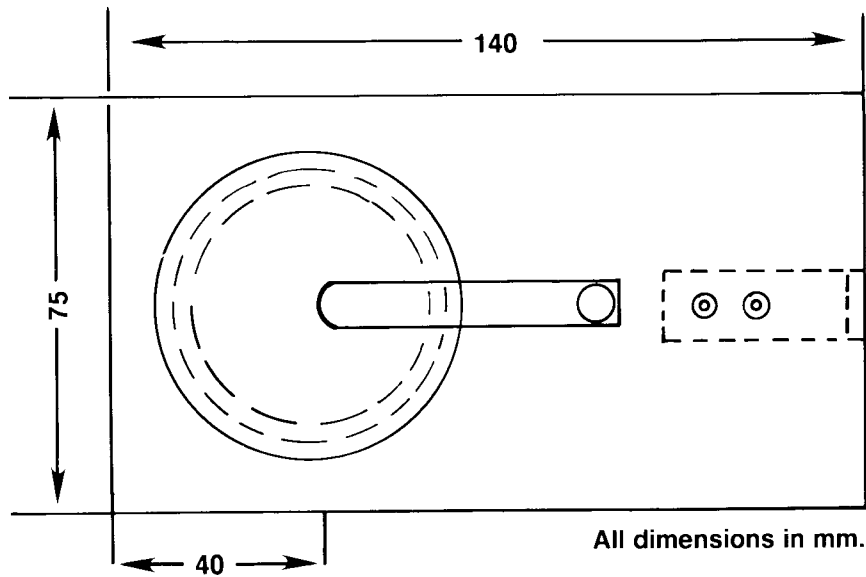
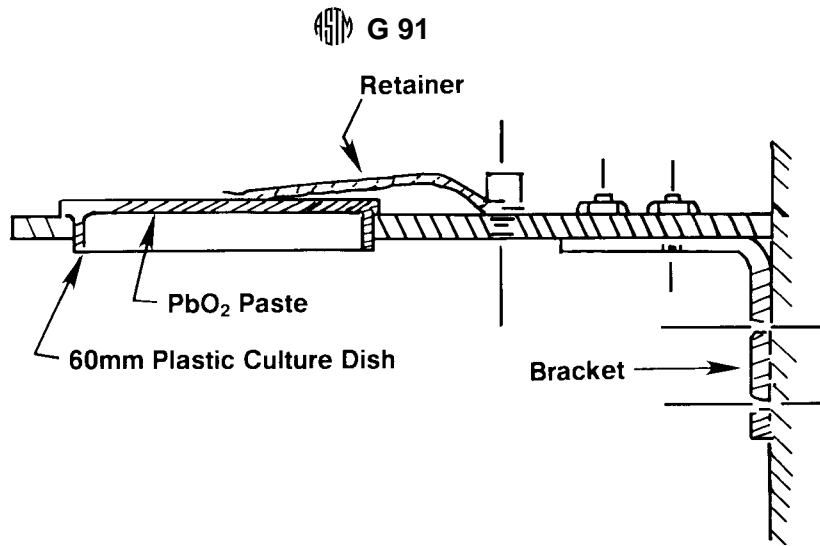


FIG. 1 Sulfation Plate Holder

equation was 0.69 based on 8 degrees of freedom. This error is therefore the lower limit for  $\sigma$ , that is, the value of  $\sigma$  becomes a constant value of  $0.69 \text{ mg SO}_2/\text{m}^2 \text{ day}$  when  $\bar{m}$  is less than  $8.8 \text{ mg SO}_2/\text{m}^2 \text{ day}$ .

9.2 *Reproducibility*—No statement can be made at this time for results from plates produced, or analyzed, or both, by different laboratories exposed under identical conditions.

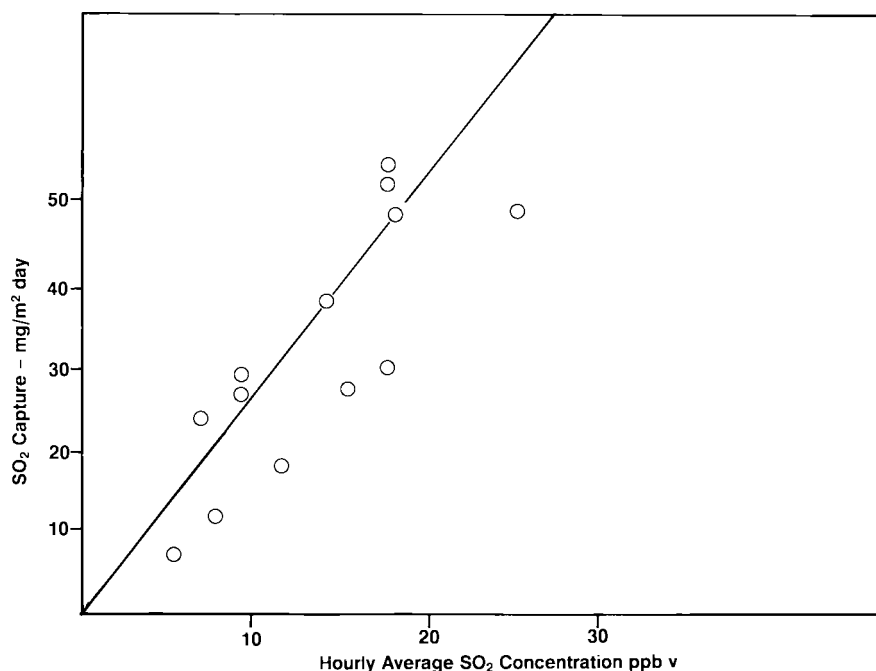
9.3 *Bias*—Although the dry deposition of  $\text{SO}_2$  from the atmosphere is related to the gaseous  $\text{SO}_2$  concentration in the ambient air, (see Fig. 2), the deposition rate is also controlled by other factors such as wind velocity and temperature. The lead peroxide sulfation plate is considered to be a reliable

measure of  $\text{SO}_2$  deposition within the limitation discussed in this section. Consequently, this procedure for measuring atmospheric  $\text{SO}_2$  dry depositions is defined only in terms of this practice.

9.4 Other methods of measuring  $\text{SO}_2$  dry deposition include a wet candle technique (Test Method D 2010/D 2010M).

## 10. Keywords

10.1 atmospheric corrosion; exposures; measurement; plate preparation; sulfate analysis; sulfation plates; sulfur dioxide



NOTE 1—A regression analysis on these results yielded the following least squares fit of the data.

$$R = (2.216 \pm 0.016)V$$

where:

$R$  = the SO<sub>2</sub> capture rate in mg SO<sub>2</sub>/m<sup>2</sup> day, and

$V$  = the average hourly volumetric SO<sub>2</sub> concentration in parts per billion.

The correlation coefficient for this data set was 0.917 and the standard error of estimate was 7.5 with 13 df.

**FIG. 2 Correlation Between Sulfation Plate Results and Mean Volumetric SO<sub>2</sub> Concentration**

## APPENDIX

### (Nonmandatory Information)

#### X1. SULFATION PLATE PREPARATION

The following practice may be used to prepare sulfation plates:

X1.1 Bond filter paper circles to the bottom of polystyrene culture (petri) dishes. Either a 50-mm to 60-mm dish size is convenient. The bonding process is carried out by placing a filter paper circle, rough side up (S & S grade 30 is acceptable) in the bottom of the dish. Paper is bonded to the plate by adding reagent grade acetone from a wash bottle until the filter just becomes saturated. Avoid splashing acetone on the walls or outside the dish. Press the paper firmly with a glass rod so that all parts of the filter are pressed into the dish. Allow acetone to evaporate. One 900-mL batch of lead peroxide will cover about eighty 50-mm plates or fifty-five 60-mm plates. The bonding may be carried out well in advance of the plate preparation procedure.

X1.2 Place a batch of bonded plates, eighty 50-mm or fifty-five 60-mm plates, in a rack and rinse with distilled or deionized water. Then fill plates with water again and allow to stand for one hour. Pour water out and refill one-quarter to one-half with distilled or deionized water.

X1.3 Add 3.5 g of gum tragacanth and 900 mL distilled or deionized water to a high speed blender container. Set at low speed and blend for two hours.

X1.4 Pour the contents of the blender into a 1 L beaker and return 350 mL of the solution to the blender container. Pulp 3.5 g of filter paper in the 350 mL of gum solution with the blender set at a moderate speed until the mixture appears smooth and uniform.

X1.5 Return 400 mL of the gum solution previously removed from the blender and blend at moderate speed for 1 min.

X1.6 Turn the blender to high speed and add 112 g of lead peroxide. Blend for 2 min and turn the blender back to low speed.

X1.7 Carefully pipet 10 mL of the mixture into each 50-mm plate or 15 mL into each 60-mm plate. Make sure mixture spreads uniformly through the water layer in the plate to the edge of each plate.

X1.8 Place the rack of plates in an oven set at 40 to 50°C for 20 h.

X1.9 Remove plate from oven and allow to cool. Seal plates with tight fitting covers to preserve until the exposure begins.

X1.10 Plates shall be numbered and placed on exposure within 120 days of preparation. Commercially obtained plates may be retained for up to one year before exposure. Retain at least one plate from each batch as a blank.

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