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TOLERANCE LIMITS FOR EMISSIONS FROM SULFURIC ACID PLANTS

SRI LANKA STANDARDS INSTITUTION

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SLS 854:1989

Gr. 6

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FOREWORD

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Sri Lanka Standards Institution on 1989-07-25, after the draft, finalized by the Drafting Committee on Air Quality had been approved by the Chemicals Divisional Committee.

The production of sulfuric acid involves processing of sulfur or sulfur bearing materials to sulfuric acid or oleum.

In the manufacture of sulfuric acid, sulfur dioxide, sulfur trioxide and acid mist are considered significant pollutants. As sulfur trioxide is readily converted to sulfuric acid on exposure to atmosphere, limits have been set only for sulfur dioxide and acid mist.

At the time this standard was formulated, only the Double Conversion Double Absorption (DCDA) process was in use. Therefore, limits have been specified only for emissions from DCDA plants.

The emission limits prescribed in this standard are applicable under normal operating conditions of the plant with a minimum stack height of 30 m. Start up conditions may have higher sulfur dioxide emissions. The limits specified in this standard are not applicable in the first 12 hours after the start up.

Automatic monitoring equipment should be installed within the industrial premises for continuous monitoring of emissions and these records should be made available to authorities whenever necessary .

All standard values given in this specification are in SI units.

For the purpose of deciding whether a particular requirement of this specification is complied with, the final value, observed or calculated, expressing the result of a test or an analysis, shall be rounded off in accordance with CS 102. The number of significant places retained in the rounded off value shall be the same as that of the specified value in this specification.

In the preparation of this standard, assistance derived from the relevant publications of the Bureau of Indian standards, HM Air Pollution Inspectorate, U.K, Environmental Protection Agency, U.S.A, and Central Board for the Prevention and Control of Water Pollution, New Delhi, is gratefully acknowledged.

1 SCOPE

This specification prescribes the limits, methods of test and sampling for gaseous emissions from sulfuric acid plants.

2 REFERENCES

IS 11255 Methods for measurement of emissions from stationary sources
Part 3 : Flow Rate
CS 102 Presentation of numerical values.

3 DEFINITIONS

For the purpose of this standard the following definitions shall apply:

3.1 **emissions** : The sum total of substances discharged into air from a stack, vent or any other discrete source. It is generally applicable to harmful and injurious substances.

3.2 **acid mist** : Low concentration dispersion of liquid particles of large size.

4 EMISSION LIMITS

4.1 Sulfur dioxide

Sulfur dioxide emissions shall not exceed 5 kilograms per metric tonne of 100 per cent sulfuric acid produced.

4.2 Acid mist

Acid mist emissions shall not exceed the concentration of 50 mg/m^3.

5 SAMPLING

5.1 Stack sampling and the measurement of the flow rate of the flue gas shall be carried out as prescribed in Appendix A and IS 11255 : Part 3.

5.2 Samples shall be drawn four times a day. Each sampling shall be over a period of one hour.

6 METHODS OF TEST

6.1 Sulfur dioxide

The test shall be carried out as prescribed in Appendix A.

6.2 Acid mist

The test shall be carried out as prescribed in Appendix A.

7 CONFORMITY TO LIMITS

The mean of the four values obtained when tested as in 5.2 shall not exceed the relevant limits specified in 4.

APPENDIX A

DETERMINATION OF SULFURIC ACID MIST AND SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

(SOURCE : EPA METHOD)

A.1 PRINCIPLE

A gas sample is extracted from the sampling point in the stack. The sulfuric acid mist (including sulfur trioxide) and the sulfur dioxide are separated, and both fractions are measured separately by the barium-thorin titration method.

A.2 APPLICABILITY

This method is applicable for the determination of sulfuric acid mist (including sulfur trioxide in the absence of other particulate matter) and sulfur dioxide from stationary sources.

A.3 APPARATUS

A.3.1 *Sampling train,* as shown in Fig 1 and as described in A.3.1.1 to A.3.1.9.

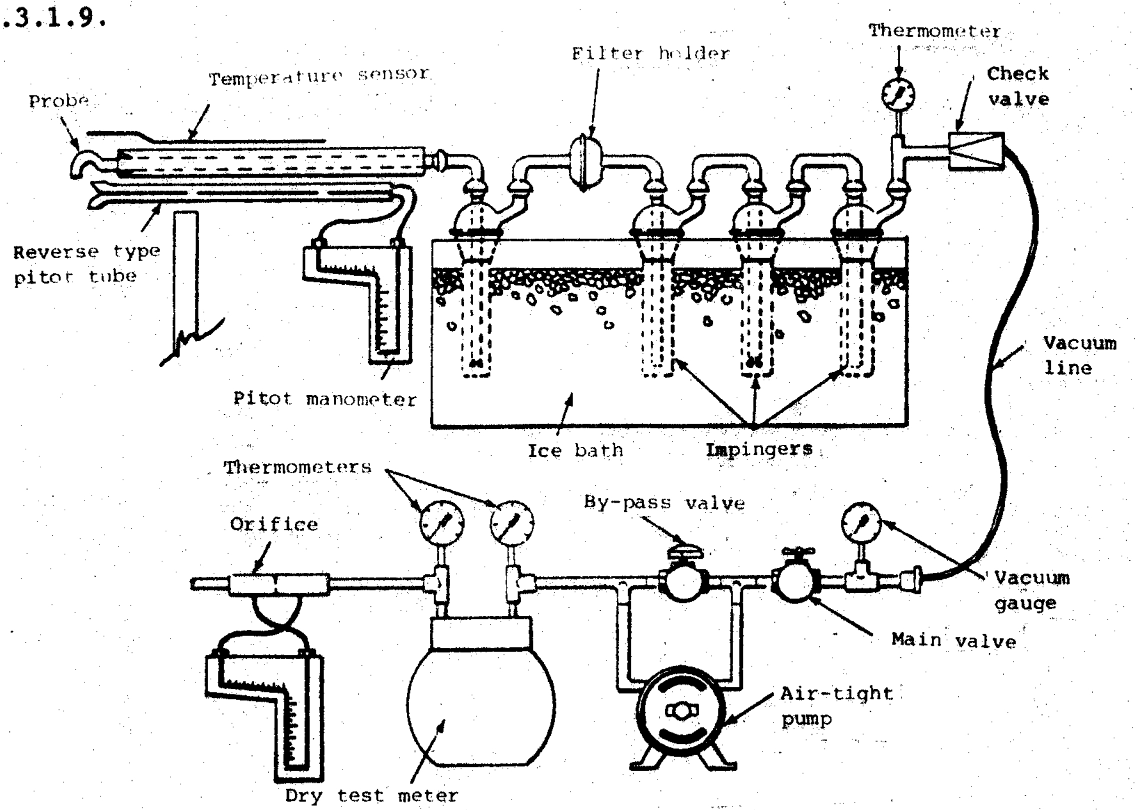

FIGURE 1

A.3.1.1 *Probe,* borosilicate glass, approximately 6 mm inside diameter with a heating system to prevent water condensation.

A.3.1.2 *Pitot tube,* as described in IS 11255 : Part 3.

A.3.1.3 *Filter holder,* borosilicate glass, with a glass frit filter support and a silicone rubber gasket. The holder design shall provide a positive seal against leakage from the outside or around the filter. The filter holder shall be placed between the first and second impingers.

NOTE

Do not heat the filter holder.

A.3.1.4 *Impingers,* four numbers as shown in Fig 1. The first and third shall be of the standard design with standard tips. The second and fourth shall be of the standard design modified by replacing the insert with an approximately 13 mm (0.5 inches) glass tube, having an unconstricted tip located 13 mm from the bottom of the flask.

Similar collection systems may be used in place of these impingers.

A.3.1.5 *Temperature gauge,* dial thermometer or equivalent, to measure temperature of gas leaving impinger train to within 1 °C.

A.3.1.6 *Main valve,* to regulate sample gas flow rate.

A.3.1.7 *Pump,* leak free diaphragm pump, or equivalent to pull gas through the train.

A.3.1.8 *Dry test meter,* dry gas meter, sufficiently accurate to measure the sample volume within 2 per cent, calibrated at the selected flow rate and conditions actually encountered during sampling and equipped with a manometer for measuring static pressure and a temperature gauge (dial thermometer or equivalent capable of measuring temperature within 0.5 °C).

A.3.1.9 *Ratemeter,* rotameter, or equivalent capable of measuring flow rate to within 2 per cent of the selected flow rate of about 1 litre/min.

A.3.2 *Storage bottles,* leak free polyethylene bottles, 1 litre size (two for each sampling run)

A.4 REAGENTS

Only analytical grade reagents should be used for sampling and analysis. Other specifications for sampling materials are as given below.

A.4.1 *Specifications for analytical grade materials*

A.4.1.1 *Water,* deionized.

A.4.1.2 *Isopropanol,* 100 per cent V/V.

A.4.1.3 *Thorin indicator (Thoranol, Thoron, Napthanon, APANS),*
1 - (o-arsonophenylazo)-2- napthol-3, 6-disulphonic acid, disodium salt, or equivalent. Dissolve 0.20 g in 100 ml of deionized, distilled water.

A.4.1.4 *Barium perchlorate solution ;* $c\,[Ba\,(ClO_4)_2] = 0.0050$ *mol/l.*
Dissolve 1.95 g of barium perchlorate trihydrate. $Ba(ClO_4)_2.3H_2O$ in 200 ml distilled water and dilute to 1 litre with isopropanol. Alternatively, 1.22 g of $BaCl_2.2H_2O$ may be used instead of the perchlorate.

A.4.1.5 *Sulfuric acid standard;* $c(H_2SO_4) = 0.0050$ *mol/l,*
standardized to ± 0.0002 mol/l against 0.0100 mol/l NaOH which has been previously standardized against potassium acid phthalate.

A.4.2 *Other specifications for sampling materials*

A.4.2.1 *Isopropanol, 80 per cent (V/V),* mix 80 ml of isopropanol with 20 ml of deionized, distilled water.

A.4.2.2 *Hydrogen peroxide, 3 per cent (V/V)* dilute 100 ml of 30 per cent hydrogen peroxide to 1 litre with deionized distilled water. Prepare fresh daily.

A.4.2.3 *Crushed ice*

A.5 PROCEDURE

A.5.1 *Preparation of collection train*

Place 100 ml of 80 per cent isopropanol in the first impinger, 100 ml of 3 per cent hydrogen peroxide in both the second and third impingers. Retain a portion of each reagent to be used as a blank solution. Place about 200 g of silica gel in the fourth impinger. Assemble the train as shown in Fig. 1. Adjust probe heater to a temperature sufficient to prevent water condensation. Place crushed ice and water around the impingers.

A.5.2 *Leak check procedure*

A leak check prior to the sampling run is optional, however, a leak check after the sampling run is mandatory.

The leak check procedure is as follows:

Temporarily attach a suitable rotameter to the outlet of the dry gas meter and place a vaccum gauge at or near the probe inlet. Plug the probe inlet, pull a vacuum of at least 250 mm Hg and note the flow rate as indicated by the rotameter. A leakage rate not in excess of 2 per cent of the average sampling rate is acceptable.

NOTE

Carefully release the probe inlet plug before turning off the pump.

A.5.3 *Sample collection*

Record the initial dry gas meter reading and barometric pressure. Position the tip of the probe at the sampling point. Adjust the sampling flow rate to a constant rate of approximately 1 litre/min, as indicated by the rotameter. Maintain this constant rate (± 10 per cent) during the entire sampling run. Take readings (dry gas meter, temperature at dry gas meter and at impinger outlet and rotameter) at least every five minutes. At the conclusion of each run, turn off the pump, remove probe from the stack, and record the final readings. Conduct a leak check as in A.5.2. Drain the ice bath and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes at the sampling rate.

A.5.4 *Sample recovery*

A.5.4.1 Container No. 1

Transfer the contents of the first impinger to a 250-ml graduated cylinder. Rinse the probe, first impinger, all connected glassware before the filter, and the front half of the filter holder with 80 per cent isopropanol. Add the rinsed solution to the cylinder. Dilute to 250 ml with 80 per cent isopropanol. Add the filter to the solution, mix and transfer to the storage container. Protect the solution against evaporation. Mark the level of liquid on the container and identify the sample container.

A.5.4.2 Container No. 2

Transfer the solutions from the second and third impingers to a 1 litre graduated cylinder. Rinse all connecting glassware (including back half of the filter holder) between the filter and silica gel impinger with deionized distilled water, and add this rinsed water to the cylinder. Dilute to a volume of 1000 ml with deionized, distilled water. Transfer the solution to a storage container. Mark the level of liquid on the container. Seal and identify the sample container.

A.5.5 *Sample analysis*

A.5.5.1 Container No. 1

Shake the container holding the isopropanol solution and the filter. If the filter breaks up, allow the fragments to settle for few minutes before removing the sample. Pipette 100 ml of this solution to 250 ml Erlenmeyer flask, add 2 to 4 drops of thorin indicator and titrate to a pink end point using 0.0050 mol/l barium perchlorate. Repeat the titration and average the values.

Replicate titrations must agree within 1 per cent or 0.2 ml whichever is greater.

A.5.5.2 Container No. 2

Thoroughly mix the solution in the container holding the contents of the second and third impingers. Pipette a 10 ml sample into a 250-ml Erlenmeyer flask. Add 40 ml of isopropanol, 2 to 4 drops of thorin indicator, and titrate to a pink end point using 0.0050 mol/l barium perchlorate. Repeat the titration and average the values.

Replicate titrations must agree within 1 per cent or 0.2 ml whichever is greater.

A.5.5.3 Blanks

Prepare blanks by adding 2 to 4 drops of thorin indicator to 100 ml of 80 per cent isopropanol. Titrate the blanks in the same manner as samples.

A.6 CALCULATIONS

A.6.1 *Dry gas volume*

Correct the sample measured by the dry gas meter to normal conditions (298°K and 760 mm Hg) by using the following equation.

$$V_{m(std)} = V_m \frac{T_{std}}{T_m} \times \frac{P_{bar}}{P_{std}}$$

where,

$V_{m(std)}$	is the dry gas volume measured by the dry gas meter, corrected to standard conditions, m^3;
V_m	is the dry gas volume as measured by the dry gas meter, m^3;
T_{std}	is the standard temperature 298 K;
T_m	is the average dry gas meter temperature K;
P_{bar}	is the barometric pressure at the exit orifice of the dry gas meter, mm Hg; and
P_{std}	is the standard absolute pressure, 760 mm Hg.

A.6.2 *Sulfur dioxide concentration*

Calculate the concentration of sulfur dioxide using the following equation.

$$C(SO_2) = \frac{0.064\ c\ (V_t - V_{tb})\ \frac{V_{soln}}{Va}}{V_{m(std)}}$$

A.6.3 *Sulfuric acid mist concentration*

Calculate the concentration of sulfuric acid mist using the following equation.

$$C(H_2SO_4) = \frac{0.098\ c\ (V_t - V_{td})\ \frac{V_{soln}}{Va}}{V_{m(std)}}$$

where,

c	is the concentration of barium perchlorate titrant, mol/l;
V_t	is the volume of barium perchlorate titrant used for the sample, ml (average of replicate titrations);
V_{tb}	is the volume of barium perchlorate titrant used for the blank, ml;
V_{soln}	is the total volume of solution in which the sulfuric acid or sulfur dioxide sample is contained;
V_a	is the volume of samples aliquot titrated, ml;
$V_{m(std)}$	is the dry gas volume measured by the dry gas meter, corrected to normal conditions, m^3;

A.6.4 *Sulfur dioxide emissions*

Calculate the emissions of sulfur dioxide (E_{SO_2}) as follows:

E_{SO_2} (g/h) = $C(SO_2) \times Q_n$

where,

$C(SO_2)$ is the concentration of sulfur dioxide g/m^3 ;
Q_n is the flue gas flow rate, wet conditions, m^3/h.

A.6.5 *Sulfuric acid mist emissions*

Calculate the emission of sulfuric acid mist ($E_{H_2SO_4}$) as follows:

$E_{H_2SO_4}$ (g/h) = $C(H_2SO_4) \times Qn$

where,

$C(H_2SO_4)$ is the concentration of sulfuric acid mist, g/m^3

NOTE

Q_n is determined in accordance with IS 11255 : Part 3.

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SRI LANKA STANDARDS INSTITUTION

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