METHOD 6 – DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

Applicability

This method is applicable to the determination of sulfur dioxide emissions from stationary sources. This template will not be used to propose combining particulate sampling with sulfur dioxide sampling. The Method 8 template would be used in that situation.

Principle

A stack sample is withdrawn from a sampling point in the stack and is collected in three midget impingers. The sulfuric acid mist (including sulfur trioxide) is separated and the sulfur dioxide is measured by barium-thorin titration.

Interferences

Possible interferents are free ammonia and fine metallic fumes. Where fine metallic fumes are present, high efficiency glass fiber filters must be used in place of the standard glass wool called for in the method. If free ammonia is present, alternate procedures must be used (see Special Situations Section). The preferred option to avoid interference would be to use Method 6C instead. If Method 6C cannot be used, an explanation will be included below.

Based on the above we (appropriate box checked):

_____ Do not expect any interference.

_____ Do expect interference. The description and discussion of the anticipated interference follows.

In–Stack Detection Limits & Sample Times

The minimum detection limit of the method has been determined to be 3.4 mg/m^3 (2.12E-07 lb/ft^3) for a standard 60 minute (liter) sample. Actual in-stack method detection limits (ISDL) are based on actual source sampling parameters and analytical results. Actual detection limits can be improved through increased stack gas sampled (sample time). For this source, the in-stack concentration is:

Allowable = _____ lbs/hr
Stack flow = _____ dscfm

lb/ft^3 = (lb / hr) / (dscfm x 60) = _____ lb/ft^3

Therefore, the sample time will be _____ minutes.

Sample Train & Recovery Components & Supplies

A schematic of the sampling train is shown in Figure 6-1 of the method. Specifically, the sampling train will be constructed with components specified under EPA Method 6, Section 6.0, with the following highlights.

Sample Train

1) **Probe liner** will be constructed of Borosilicate glass or stainless steel and be heated.
2) A **filter**, either in-stack or heated out-of-stack. In most cases, a glass wool plug is sufficient. If fine metallic fumes are present (identified above) a high efficiency glass fiber filter will be used.
3) The **impinger train** will consist of three to four midget impingers connected in series with leak-free ground glass fittings or other leak-free, non-contaminating fittings. Silicone grease may be used if necessary. The first impinger or bubbler contains 15 ml 80% isopropanol (IPA) packed with glass wool prevents sulfuric acid mist carryover. The second and third impingers contain 15 ml of 3% H_2O_2. The final impinger is dry. A drying tube packed with silica gel is placed at the exit of the last impinger.
4) A Method 8 sampling train may be substituted for the Method 6 sampling train (see Special Situations Section).
5) **Pump -** leak-free diaphragm pump, or equivalent, with a small surge tank between the pump and rate meter.
6) **Rate Meter -** Rotameter, or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate of about 1 liter/min (0.035 cfm).
7) **Dry Gas Meter** (DGM) - sufficiently accurate to measure the sample volume to within 2 percent, calibrated at the selected flow rate and conditions actually encountered during sampling, and equipped with a temperature sensor (dial thermometer, or equivalent) capable of measuring temperature accurately to within 3 °C (5.4 °F). A critical orifice may be used in place of the DGM specified in this section provided that it is selected, calibrated, and used as specified in Section 16.0.

Sample Recovery

1) **Wash bottles** (glass or polyethylene); two 500 ml.
2) **Storage bottles** (polyethylene) one 100 ml per sample.

<u>Sampling</u>

Pre-test leak checks and post-test leak checks will be conducted as follows: temporarily attach a suitable rotameter to the outlet of the dry gas meter and place a vacuum gauge at or near the probe inlet. Plug the probe inlet and pull a vacuum of least 10 in. Hg. A leak rate as indicated by the rotameter shall not exceed 2% of the sampling rate. Pump leak checks are recommended.

The sampling train will be assembled as indicated above. Crushed ice and water will be placed around the impingers. The initial DGM reading and barometric pressure will be recorded. The sampling rate will be adjusted to approximately 1.0 liter/min as indicated by rate meter. This flow rate will be maintained ($\pm$10%) throughout the run. Readings as required by the method (DGM volume, temperatures at DGM and at impinger outlet, and rate meter flow rate) will be recorded at least every 5 minutes.

Post Test Purge

At the conclusion of an acceptable post leak check, the sampling train will be purged with clean ambient air (optionally passed through either charcoal or a midget impinger containing 15 ml 3% H_2O_2 if necessary) for 15 minutes at the sampling rate.

Sample Recovery

After disconnecting the impingers from purging, the contents of the midget bubbler/impinger will be discarded. The contents of the midget impingers will be poured into a leak free polyethylene bottle for shipment. The three midget impingers and connecting tubes will be rinsed with water and the washings added to the same container. The fluid level will be marked.

30 ml of 3% H_2O_2 will be collected as a blank in a separate container.

Sample Preparation & Analysis

The level of the fluid in the container will be noted. If significant leakage occurred, either the run will be voided or methods will be used to correct the results, subject to BTS approval. The contents of the storage container will be transferred to a 100 ml volumetric flask and diluted to exactly 100 ml with water. A 20 ml aliquot will be pipetted into a 250 ml Erlenmeyer flask and 80 ml of 100% isopropanol and two to four drops of thorin indicator will be added and titrated to a pink endpoint with 0.0100 N barium perchlorate. Titration volumes will be repeated and averaged. A blank will be run with each series of samples. The blank is prepared for analysis in the same manner as the samples, as indicated above. Replicate titrations will agree within 1 % or 0.2 ml, whichever is larger.

Calculations

All calculations will be performed as per Method 6, including blank corrections.
Detailed sample calculations will be included in the final report.

Emissions will be presented in the following units:

_____

Audit Samples

If provided, audit samples will be analyzed consistent with Section 11.3 of the method and the results will be provided in the final test report. It is recognized that failure to achieve method acceptance criteria for the audit could result in the requirement to repeat the stack test program.

Proposed Deviations from this BTS Template or the Method

(Insert any proposed deviations here)

Special Situations Section – If either of these two situations is relevant, they will be checked in the checkbox prior to the procedure.

(_) 1. **Alternative Procedures when Ammonia is Present**:

Sampling and Recovery Procedures

The probe will be maintained at 275°C (527°F) and equipped with a high-efficiency in-stack filter (glass fiber) to remove particulate matter. The filter material will be unreactive to SO_2. Whatman 934AH (formerly Reeve Angel 934AH) filters treated as described in Reference 10 in Section 17.0 of Method 5 is an example of a filter that has been shown to work. Where alkaline particulate matter and condensed moisture are present in the gas stream, the filter will be heated above the moisture dew point but below 225°C (437°F).

Sample recovery will be the same as above, except the contents of the midget IPA bubbler/impinger, along with water rinses of the bubbler/impinger, will be transferred to a separate polyethylene bottle.

Analysis

Analysis will be as described in Section 16.3.3 of the method.

(_) 2. **Substituting Method 8 sampling train for midget impingers:**

The sampling equipment described in Method 8 may be substituted for the midget impinger train described herein; however, the train must be modified to include a heated filter between the probe and isopropanol impinger and the operation of the sampling train and sample analysis must be at flow rates and solution volumes defined in Method 8.