

Source Test Procedure **ST-28**

HYDROGEN SULFIDE, INTEGRATED SAMPLING

(Adopted January 20, 1982)

REF. Regulation 10-8-303

1. APPLICABILITY

- 1.1 This method is applicable for the determination of hydrogen sulfide (H_2S) emissions from stationary sources for compliance with Regulation 10-8-303.

2. PRINCIPLE

- 2.1 Hydrogen sulfide is collected in a series of impingers and reacted with alkaline cadmium hydroxide ($Cd(OH)_2$) to form cadmium sulfide (CdS). The precipitated CdS is dissolved in hydrochloric acid and absorbed in an iodine solution. The iodine consumed is related to the H_2S content of the gas. The sample train is preceded by an impinger containing hydrogen peroxide to remove SO_2 which interferes with the determination.

3. RANGE AND SENSITIVITY

- 3.1 The minimum measurable concentration of hydrogen sulfide is 0.0002 grains/SDCF.

4. INTERFERENCES

- 4.1 High concentrations of SO_2 , or other acid gases, could saturate the peroxide impinger and interfere.

5. APPARATUS

- 5.1 Probe. The probe is constructed of borosilicate glass tubing.
- 5.2 Absorbers. Use five Greenberg-Smith impingers. The final impinger has a thermometer attached to the inlet stem.
- 5.3 Cooling System. An ice bath is used to contain the impingers.
- 5.4 Sample pump. Use a leak-free vacuum pump capable of maintaining a 0.5 CFM flow rate at 15 inches of mercury. The pump has a flow control and vacuum gauge attached to the inlet.
- 5.5 Silica Gel Tube. Use approximately 500 cc of silica gel with a drierite indicator to insure dry gas entering the dry gas meter.
- 5.6 Dry Test Meter. Use a test meter accurate to within $\pm 2\%$ of the true volume and equipped with a thermometer to measure the outlet temperature.
- 5.7 Connections. Use Teflon tubing in making all connections that come in contact with the sample. Vinyl tubing is acceptable for all other connections.
- 5.8 Barometer. Use a barometer that is accurate within ± 0.2 inches of mercury.

5.9 Rotameter. Use a calibrated rotameter to measure the sampling rate.

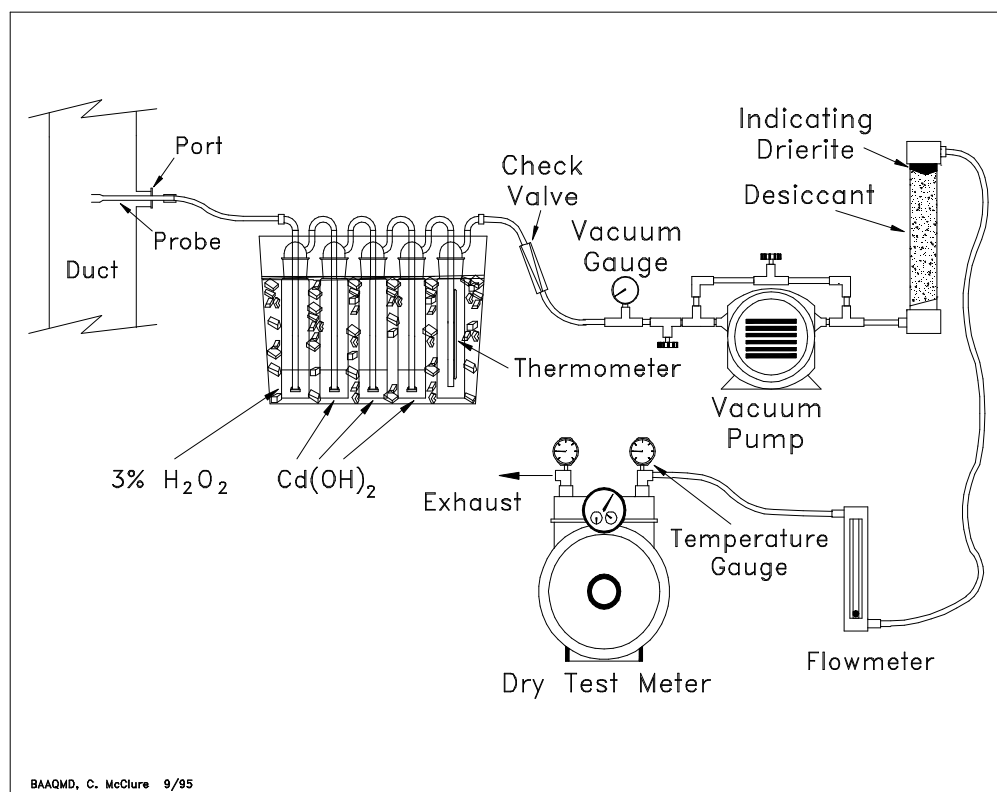
6. REAGENTS

6.1 Hydrogen peroxide (H_2O_2) solution, 3%. Prepare a solution of 3% by volume hydrogen peroxide in distilled water.

6.2 Cadmium hydroxide ($\text{Cd}(\text{OH})_2$). Mix 4.3 g cadmium sulfate hydrate ($3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$) and 0.3 g of sodium hydroxide (NaOH) in 1 liter of distilled water.

Figure 28-1

Hydrogen Sulfide Sampling Train



7. PRE-TEST PROCEDURES

7.1 Add 100 ml of 3% H_2O_2 to one impinger.

7.2 Add 100 ml of $\text{Cd}(\text{OH})_2$ to each of three impingers.

7.3 Assemble the sampling train as shown in Figure 28-1.

7.4 Leak-test the sampling train by starting the sample pump, plugging the probe, and adjusting the pump inlet vacuum to 10 inches Hg. The leak rate must not exceed 0.02 CFM through the meter. Before stopping the pump, carefully release the plug in the sample probe to avoid backflow of the impinger solution.

7.5 Record the initial dry test meter reading and barometric pressure as shown in Form 28-1.

8. SAMPLING

- 8.1 Each test run shall be of thirty minute duration when testing emissions from continuous operations. Each test run at batch process operations shall be for 90% of the batch time or thirty minutes, whichever is less. If the yellow color of cadmium sulfide is visible in the third cadmium hydroxide impinger discontinue sampling.
- 8.2 Position the probe at the sampling point and start the pump.
- 8.3 Sample at a constant rate of 14.3 liter/min (0.5 CFM) during the test as determined by the rotameter. Use the rotameter only to establish the initial sampling rate. Then remove it from the system.
- 8.4 Record the following information at five-minutes intervals:
- dry test meter temperature
impinger outlet temperature
dry test meter reading
- 8.5 Add ice as necessary to maintain impinger temperatures at 7 °C or less.
- 8.6 At the conclusion of each run, stop the pump, remove the probe from the stack, and record the final meter reading.
- 8.7 Take three consecutive samples.

9. POST-TEST PROCEDURES

- 9.1 Disconnect the impinger train from the probe and purge the train with clean ambient air for 15 minutes to ensure all H₂S is removed from the hydrogen peroxide.
- 9.2 Stopper the impingers to minimize sample losses.
- 9.3 Individually analyze the cadmium hydroxide solutions for hydrogen sulfide according to Analytical Procedure, Lab-18.

10. CALCULATIONS

- 10.1 Standard Sample Volume:

$$V_o = \frac{17.71V_m P_b}{T_m}$$

Where:

- V_o = corrected sample volume, SDCF at 70 °F and 29.92 in Hg
 T_m = average meter temperature, °R,
 V_m = uncorrected meter volume, ft³
 P_b = barometric pressure, inches Hg
 17.71 = constant correcting to standard conditions

$$17.71 = \frac{(530^\circ\text{R})}{(29.92 \text{ inches Hg})}$$

10.2 Hydrogen Sulfide concentration:

$$C_{\text{H}_2\text{S}} = \frac{W \times 15.43}{V_o}$$

Where:

$C_{\text{H}_2\text{S}}$ = Hydrogen sulfide concentration, grains/SDCF.

W = Total weight of hydrogen sulfide in the impinger catch for each run, grams.

15.43 = Factor converting grams to grains

11. REPORTING

11.1 Report the data indicated on Form 28-2.

Bay Area Air Quality Management District
 939 Ellis Street, San Francisco, CA 94109

Form 28-1
Source Test Data Sheet

Plant # _____
 Source I.D. _____
 Sample Type _____
 Process Cycle _____
 Duct Size _____
 Duct Shape _____
 Duct Pressure _____
 Assumed %H₂O _____

Run # _____
 Temp Meter # _____
 Mag. Gauge # _____

Date: _____
 Box ΔH@ _____
 Meter (Y) _____

_____ Nozzle Diameter
 _____ Pitot Tube I.D., Cp
 _____ Gas System
 _____ Pbar, Barometer
 _____ Leak Test Rate
 _____ Time @ Point
 _____ # of Points
 _____ Time/Run (Min.)

Sampling Train: Probe # _____ Filter # _____ Imp. # _____ Imp. # _____ Pump/Box # _____

Initial Traverse Data					Sampling Data									
Trav. Point I.D.	Dist. from Wall	Duct Temp. °F	ΔP "H ₂ O	Angle of Flow	Traverse Point I.D.	ΔP "H ₂ O	Duct Temp. °F	Vs FPS	Time (minutes)	Meter Rate CFH	Meter Temp. °F	Meter Volume Ft ³	Train Vacuum "Hg	Sat'd Gas Temp. °F

Post Run Impinger Catch (ml) = _____
 Assumed O₂ = _____
 Assumed CO₂ = _____
 Post Run Calculated %H₂O = _____

Source Test Team _____

Comments: _____

Form 28-2

Distribution: Firm Permit Services Enforcement Services Technical Services Planning Requester DAPCO	BAY AREA AIR QUALITY MANAGEMENT DISTRICT <i>939 Ellis Street San Francisco, California 94109 (415) 771-6000</i> Summary of Source Test Results	Report No.: _____ Test Date: _____ Test Times: Run A: _____ Run B: _____ Run C: _____
Source Information		BAAQMD Representatives
Firm Name and Address	Firm Representative and Title Phone No. ()	Source Test Engineers
Permit Conditions:	Source: Plant No. Permit No. Operates	Permit Services Division/Enforcement Division Test Requested By:
Operating Parameters:		
Applicable Regulations:		VN Recommended:

Source Test Results and Comments:

<u>METHOD</u> <u>TEST</u>	<u>RUN A</u>	<u>RUN B</u>	<u>RUN C</u>	<u>AVERAGE</u>	<u>LIMIT</u>
ST-28 Hydrogen Sulfide, gr/SDCF					

Air Quality Engineer II	Date	Supervising Air Quality Engineer Date	Approved by Air Quality Engineering Manager
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