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 $\pm$0.2 inches of mercury.

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

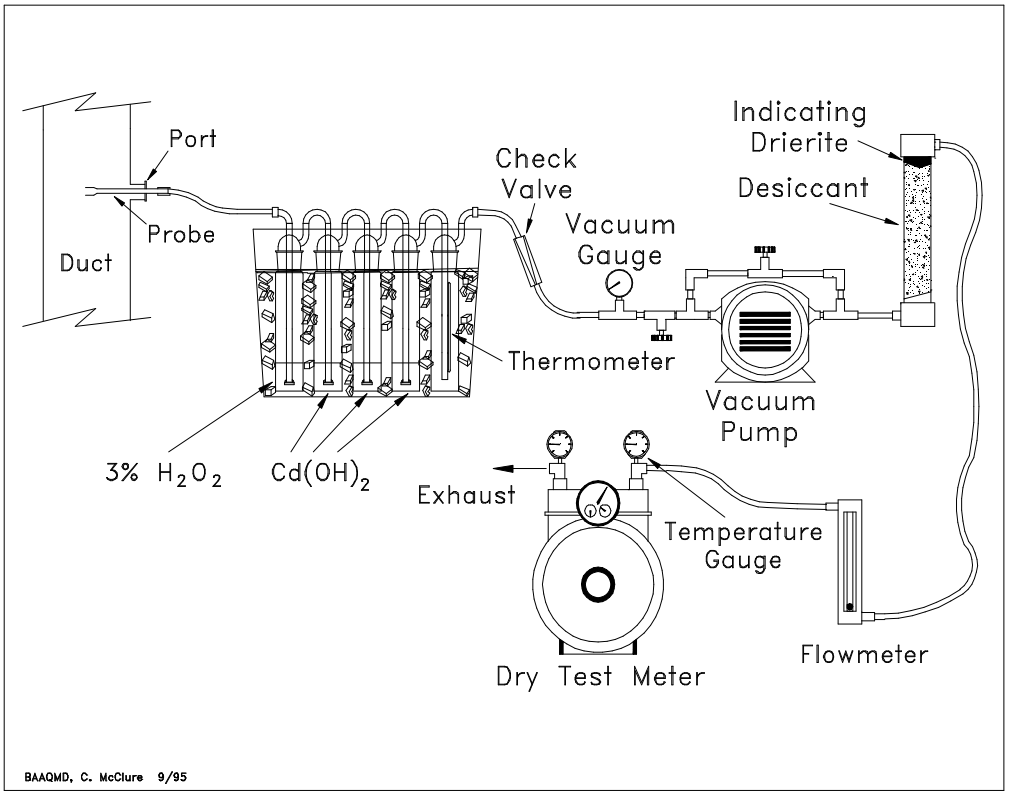
6. REAGENTS

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 ($Cd(OH)_2$). Mix 4.3 g cadmium sulfate hydrate ($3CdSO_4\ 8H_2O$) 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 $Cd(OH)_2$ 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 ^{o}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 H_2S 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:

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

Where:

V_o = corrected sample volume, SDCF at 70 ^{o}F and 29.92 in Hg
T_m = average meter temperature, ^{o}R,
V_m = uncorrected meter volume, ft^3
P_b = barometric pressure, inches Hg
17.71 = constant correcting to standard conditions

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

10.2 Hydrogen Sulfide concentration:

$$C_{H_2S} = \frac{W \times 15.43}{V_o}$$

Where:

C_{H_2S} = 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 %H_20 _____

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. ^0F	ΔP "H_20	Angle of Flow	Traverse Point I.D.	ΔP "H_20	Duct Temp. ^0F	Vs FPS	Time (minutes)	Meter Rate CFH	Meter Temp. ^0F	Meter Volume Ft^3	Train Vacuum "Hg	Sat'd Gas Temp. ^0F

Post Run Impinger Catch (ml) = _____

Assumed O_2 = _____

Assumed CO_2 = _____

Post Run Calculated %H_2O = _____

Source Test Team _____

Comments: _____

Form 28-2

Distribution: Firm Permit Services Enforcement Services Technical Services Planning Requester DAPCO	**BAY AREA** **AIR QUALITY MANAGEMENT DISTRICT** *939 Ellis Street* *San Francisco, California 94109* *(415) 771-6000* **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:

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

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