Source Test Procedure **ST-19B**

TOTAL SULFUR OXIDES, INTEGRATED SAMPLE

REF: Regulations 9-1-302, 9-1-304 through 310, 10-1-301

1. APPLICABILITY

1.1 This method is used to quantify emissions of total sulfur oxides. It determines compliance with Regulations 9-1-302, 9-1-304 through 310, 10-1-301 and is an acceptable alternate method to ST-19A.

2. PRINCIPLE

2.1 Sample gas is drawn through 3% hydrogen peroxide (H_2O_2) solution which absorbs the sulfur oxides. The peroxide solution is then analyzed according to Analytical Procedure Lab-11.

3. RANGE

3.1 The minimum measurable concentrations of sulfur oxides is 7 ppm at the sample volume specified in this procedure.

3.2 The maximum measurable concentration of sulfur oxides is 2.5%.

4. INTERFERENCE

4.1 The presence of strong acids, such as hydrochloric acid or sodium bisulfite give erroneously high results. The presence of alkaline gases or reactive basic solids, such as ammonia or calcium oxide give erroneously low results.

5. APPARATUS

5.1 Probe. The probe is constructed of borosilicate glass tubing.

5.2 Absorbers. Use three Greenberg-Smith impingers. The final impinger has a thermometer attached to the inlet stem.

5.3 Filter. Use a glass filter tube of the same type as is used in ST-15.

5.4 Cooling System. An ice bath is used to contain the impingers.

5.5 Sample Pump. Use a leak-free vacuum pump capable of maintain a 0.5 CFM flow rate at 15 inches of mercury-vacuum. The pump must have a flow control valve and vacuum gauge attached to the inlet.

5.6 Silica Gel Tube. Use approximately 500 cc of silica gel (with Drierite indicator) to insure that the gas entering the dry gas meter is dry.

5.7 Dry Test Meter. Use a dry gas test meter accurate with $\pm$ 2% of the true volume and equipped with a thermometer to measure the outlet temperature.

5.8 Connections. Use Teflon tubing in making all connections that come in contact with the sample. Vinyl tubing is acceptable for all other connections.

5.9 Barometer. Use a barometer that is accurate to within $\pm$ 0.2 inches of mercury.

5.10 Rotameter. Use a calibrated Rotameter to measure the sampling rate.

6. REAGENTS

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

7. PRE-TEST PROCEDURES

7.1 Add 100 ml of 3% H_2O_2 to each of two impingers.

7.2 Stopper the impingers.

7.3 Retain 100 ml of the hydrogen peroxide solution to analyze as a blank.

7.4 Assemble the sampling train as shown in Figure 19B-1.

7.5 Leak-test the sampling train by turning on the 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 dry test meter. Before stopping the pump, carefully release the plug in the sample probe to avoid back flow of the impinger solution.

7.6 Record the initial dry test meter reading and barometric pressure as shown in Figure 19B-2.

Figure 19B-1

Sulfur Oxides Sampling Train

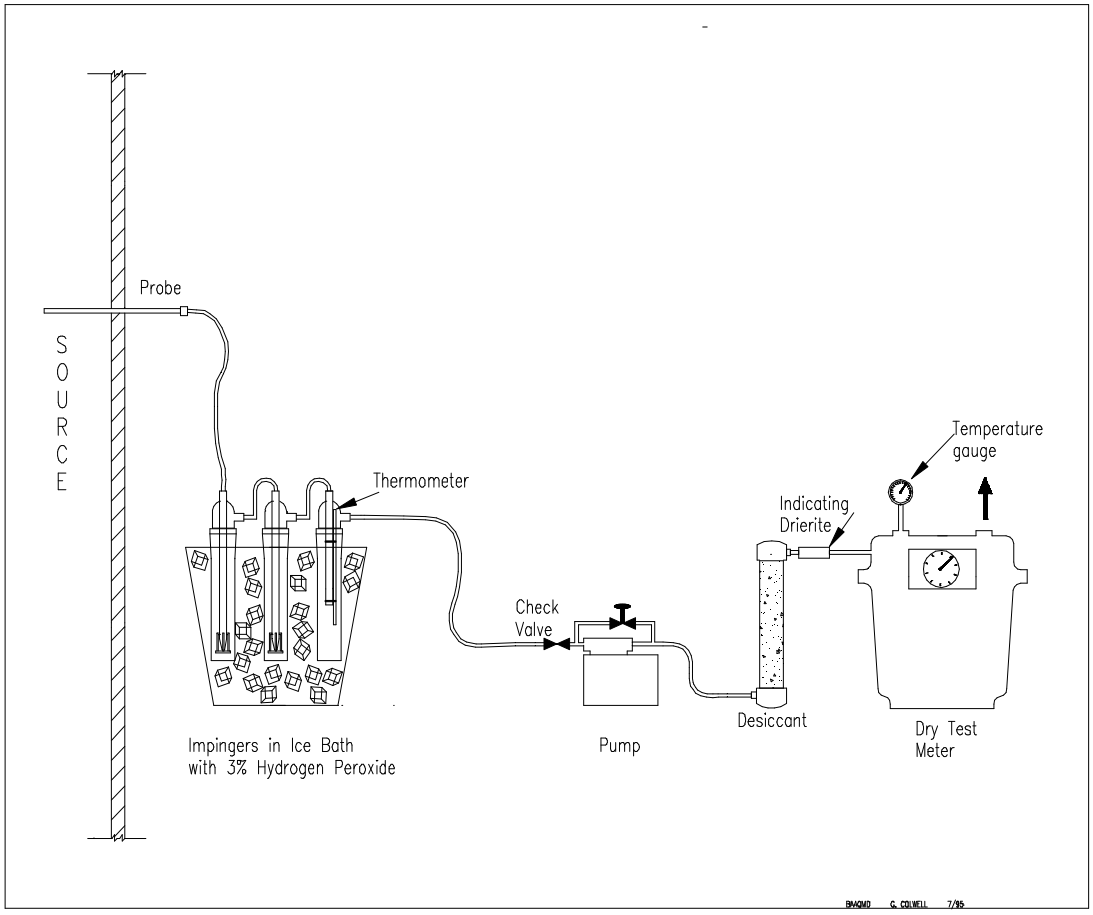
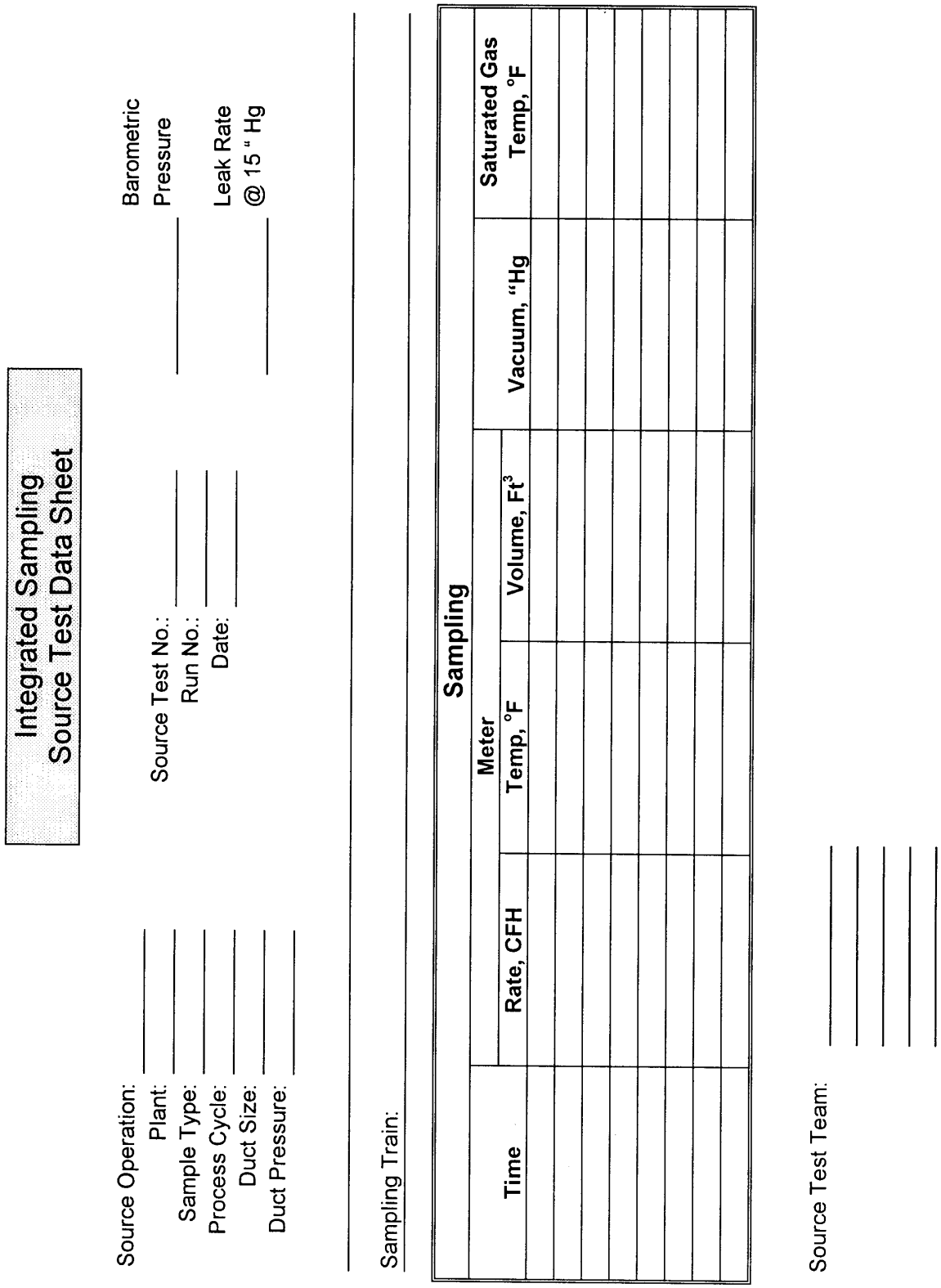


Figure 19B-2
Integrated Sampling Data Sheet



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.

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 CFH) 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-minute intervals:

Dry Test Meter Temperature
Impinger Outlet Temperature
Dry Test Meter Volume

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 t he stack, and record the final meter reading.

8.7 Take three consecutive samples.

9. POST-TEST PROCEDURES

9.1 Immediately after each test run, stopper the impingers to minimize sample losses.

9.2 Individually analyze the hydrogen peroxide solutions for total sulfur oxide content according to Analytical Procedure Lab-11.

10. AUXILIARY TESTS

10.1 Stack Flowrate. Determine the stack gas flowrate after each run in accordance with ST-17.

10.2 Moisture. Determine the stack gas moisture content in accordanc e with ST-23.

10.3 Production Rate. Determine the sulfuric acid or sulfur production from plant data based on a 24 hour, daily production rate in tons per day, if the test is being conducted on a sulfur recovery unit or a sulfuric acid plant.

10.4 Oxygen concentration. Use ST-14 to determine the oxygen concentration of the stack gas.

11. CALCULATIONS

11.1 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 inches Hg.

V_m = Uncorrected meter volume, ft^3

T_m = Average Run Meter Temperature, ^oR

P_b = Barometric pressure, inches Hg

17.71 = A constant correcting to 70 ^{o}F and 29.92 inches Hg

11.2 Sulfur oxide concentration:

$$C_{SO} = \frac{1.33 \times 10^4 W}{V_o}$$

Where:

C_{SO} = Sulfur oxides concentration, as SO_2 ppm by volume on a dry basis

W = Total Weight of sulfur oxides in the impinger catch, for each run, grams.

1.33 = Constant derived from molar volume, 454 grams/lb, and molecular weight of SO_2.

11.3 Mass emission rate of SO_X:

$$M_{SO_x} = KD_{SO_x}Q_o$$

Where:

M_{SO_x} = Mass emission rate of SO_X (as SO_2) kg/hr (lb/hr)

Q_o = Stack gas flow rate $\frac{SDM^3}{\min}\left(\frac{SDFT^3}{\min}\right)$

K = 9.93 x 10 $^{-6}$ $\frac{lb}{ppm - ft^3 - hr}$ for English units

= 1.59 x 10^{-4} $\frac{kg}{ppm - m^3 - hr}$ for SL units.

11.4 Mass emissions based on production rate:

$$M_{tp} = \frac{M_{SO_2}}{M_d}$$

Where:

M_{tp} = Mass emissions per ton of product, $\frac{Kg}{metricTon}\left(\frac{lbs}{shortTon}\right)$

M_{SO_2} = Mass emission rate of SO_2, $\frac{Kg}{hr}\left(\frac{lbs}{hr}\right)$

M_d = Production rate per hour (based on a 48 hour a verage).

11.5 Oxygen Correction factor:

$$F = \frac{21.95 \times X_{O_2}}{21.95 \times P_{O_2}}$$

Where:

F = Oxygen correction factor

X_{O_2} = Oxygen concentration to be corrected to 0% for Sulfur Recovery plants and 12% for Sulfuric Acid plants

P_{O_2} = Oxygen content present in stack gas

11.6 SO_2 concentration corrected for oxygen content.

$$C_{SO_2}F = F \times C_{SO_2}$$

Where:

$C_{SO_2}F$ = SO_2 concentration corrected for oxygen content

12. REPORTING

Report the data indicated on Figure 19B-3.

Figure 19B-3

Report No.: _____ **Test Date:** _____	**BAY AREA** **AIR QUALITY MANAGEMENT DISTRICT** **Summary of** **Source Test Results**	**Test Times:** **Run A:** _____ **Run B:** _____ **Run C:** _____

Source Information		**Test Representatives**
Firm Name and Address	Firm Representative and Title Phone No. ( )	
Permit Conditions:	Source: Plant No. Permit No. Operates Hr/Day & Day/Yr.	Operating Parameters
Applicable Regulations:		

Source Test Results and Comments:

METHOD	TEST	RUN A	RUN B	RUN C	AVERAGE	LIMIT
	Run time, minutes					
	Stack gas temperature, F^o					
ST-17	Stack flow rate, SDCFM					
ST-19B	Sulfur Oxides, ppm					
	Sulfur Oxides, lb/hr					
	SO_2, lb/ton product					

Test Team Leader Date	Reviewed by Date	Approved By Date