

Source Test Procedure **ST-10**

MERCURY

Adopted (January 20, 1982)

REF: Regulation 11-5-301

1. APPLICABILITY

1.1 This method is used to quantify emissions of gaseous and particulate mercury. It determines compliance with Regulation 11-5-301.

2. PRINCIPLE

2.1 The sample stream is drawn through a solution of iodine monochloride which collects the mercury in the gas stream. The mercury compounds in the iodine monochloride (ICl) are reduced to elemental mercury and analyzed according to Analytical Procedure Lab 6. If high concentrations of particulate are expected, use particulate filters as prescribed in ST-15.

3. RANGE

3.1 The minimum measurable emission of mercury is 2×10^{-5} grains/SDCF.

3.2 Elevated concentrations of mercury may be determined by increasing the concentration of iodine monochloride in the absorbing reagent. The concentration of reagent to be used may be determined by stoichiometry, allowing a 50% excess.

4. INTERFERENCES

None Known.

5. APPARATUS

5.1 Probe Nozzle. The probe nozzle shall be equivalent to the nozzle shown in Figure 10-1. The probe nozzle shall be constructed of borosilicate glass, quartz, or stainless steel.

5.2 Filter Holder. Use filter tubes equivalent to the one shown in Figure 10-1 and constructed of borosilicate glass or quartz.

5.3 Filter Tube Holder. Use a filter tube holder which will contain three filter tubes and the probe nozzle in a leak-free manner. Figure 10-2 shows an acceptable filter tube holder configuration.

5.4 Filter Medium. Use borosilicate glass wool, or equivalent. Corning Glass Works, Catalogue #3950 is acceptable.

5.5 Connections. The connection between the filters and the first impinger must be able to withstand stack temperatures. Vinyl tubing is acceptable in making all other connections.

- 5.6 Pitot Tube. Use a Stauscheibe (Type-S), or equivalent, with a known coefficient which is constant within $\pm 5\%$ over the entire working range. The pitot type coefficient is determined by placing both the S-type and the standard pitot tube in a gas stream and measuring the pressure head with both over the entire Type-S pitot tube as follows:

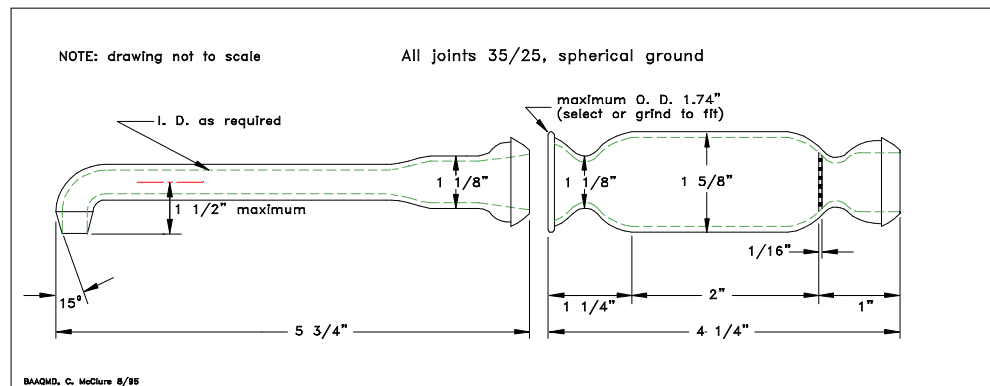
$$Cp_s = Cp_{std} \left[\frac{\Delta P_{std}}{\Delta P_s} \right]^{\frac{1}{2}}$$

where:

- Cp_s = Type-S pitot tube coefficient
 ΔP_s = Pressure head, Type-S pitot tube
 ΔP_{std} = Pressure head, standard pitot tube
 Cp_{std} = Standard pitot tube coefficient

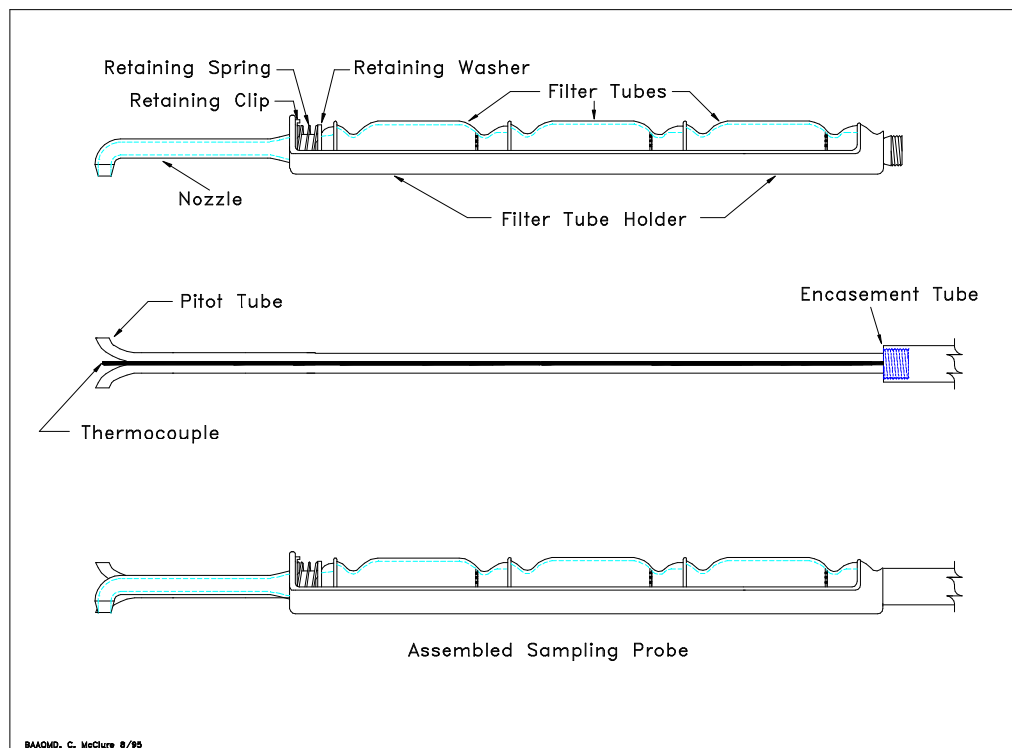
Figure 10-1

Nozzle and Filter Tube



- 5.7 Temperature Measuring Device. Use a Chromel-Alumel thermocouple accurate to $\pm 15^{\circ}\text{F}$, connected to a temperature compensated null type potentiometer, or equivalent, to measure stack temperatures.
- 5.8 Encasement Tube. Assemble the items in 5.3, 5.6 and 5.7 in a pipe as shown in Figure 10-2 to facilitate handling. To prevent the pitot tube from interfering with the gas flow or stream-lines approaching the sample nozzle, position the pitot tube and sample nozzle not closer together than 0.75 inches.
- 5.9 Absorber. Use four Greenberg-Smith impingers. The fourth impinger shall be modified by removing the impaction plate and attaching a thermometer to the inlet stem.
- 5.10 Cooling System. Use an ice bath to contain the impingers.
- 5.11 Sample Pump. Use a leak-free vacuum pump capable of maintaining a 1.0 CFM flow rate at 15 inches of mercury. The pump must have a sample rate control valve and a vacuum gauge attached to the inlet.

- 5.12 Silica Gel Tube. Use approximately 500cc of silica gel, followed by a Drierite indicator, to insure that the gas entering the dry test meter is free of H₂O.
- 5.13 Dry Test Meter. Use a 175 CFH dry test meter accurate within $\pm 2\%$ of the true volume and equipped with a thermometer to measure the outlet temperature. The working pressure across the meter shall not exceed one inch water column.
- 5.14 Rotameter. Use a calibrated rotameter to measure the sampling rate.
- 5.15 Pressure Gauge. Use a Magnehelic differential pressure gauge, or equivalent, in the same range as the velocity and static pressures being measured in the stack.
- 5.16 Analytical Balance. An analytical balance capable of measuring condensate weights to the nearest 0.1 gram is acceptable.
- 5.17 Barometer. Use a barometer that is accurate to within ± 0.2 inches of mercury.

Figure 10-2**Filter Tube Assembly****6. REAGENTS**

- 6.1 0.1M Iodine Monochloride. Refer to Analytical Procedure Lab 6.

7. PRE-TEST PROCEDURES

- 7.1 Impinger Preparation. 100 ml of the 0.1 M ICI shall be placed in the first three impingers which shall then be weighed. Record the data on the data sheet shown in Form 10-1.
- 7.2 Nozzle Size Determination. Do the preliminary test outlined below to determine the correct nozzle size to aid in isokinetic sampling.
- 7.2.1 Determine the number and location of the stack traverse points in accordance with ST-18.
- 7.2.2 Conduct a velocity traverse in accordance with ST-17 and measure the stack gas temperature.
- 7.2.3 Determine the moisture content of the stack gases in accordance with ST-23.
- 7.2.4 Nozzle Diameter:

$$D_n = 13.7 \left[\frac{T_s}{V_s(100 - \%H_2O)} \right]^{\frac{1}{2}}$$

Where:

- D_n = Nozzle Diameter, mm
- T_s = Stack Gas Temperature, °R
- H_2O = Stack Gas Moisture Concentration, %
- 13.7 = A constant based on an assumed meter rate of 0.5 CFM, meter temperature of 70°F, and a molecular weight of 28.8
- V_s = Stack Gas Velocity fps as calculated in Section 8.2 of ST-17

- 7.3 Assemble the sampling train as shown in Figure 10-3.
- 7.4 The entire sampling train must be leak-checked before each test run. Plug the sampling probe, start the pump, and adjust the pump vacuum to 380mm Hg (15" Hg). A leak rate through the meter which exceeds 0.57 1pm (0.02 CFM) is unacceptable.

8. SAMPLING

- 8.1 Each test run shall be of 50 minute duration when testing emissions from continuous operations. Each test run at batch process operations shall be for 90% of the batch time or 50 minutes, whichever is less.
- 8.2 Sample at the traverse points determined in accordance with ST-18.
- 8.3 The sampling rate at each traverse point must be isokinetic. Measure the stack velocity and stack temperature at each sample point and adjust the meter flow rate according to the following equation:

$$Q_m = \frac{2.638 \times 10^{-3} (100 - \%H_2O) D_n^2 V_s}{T_s}$$

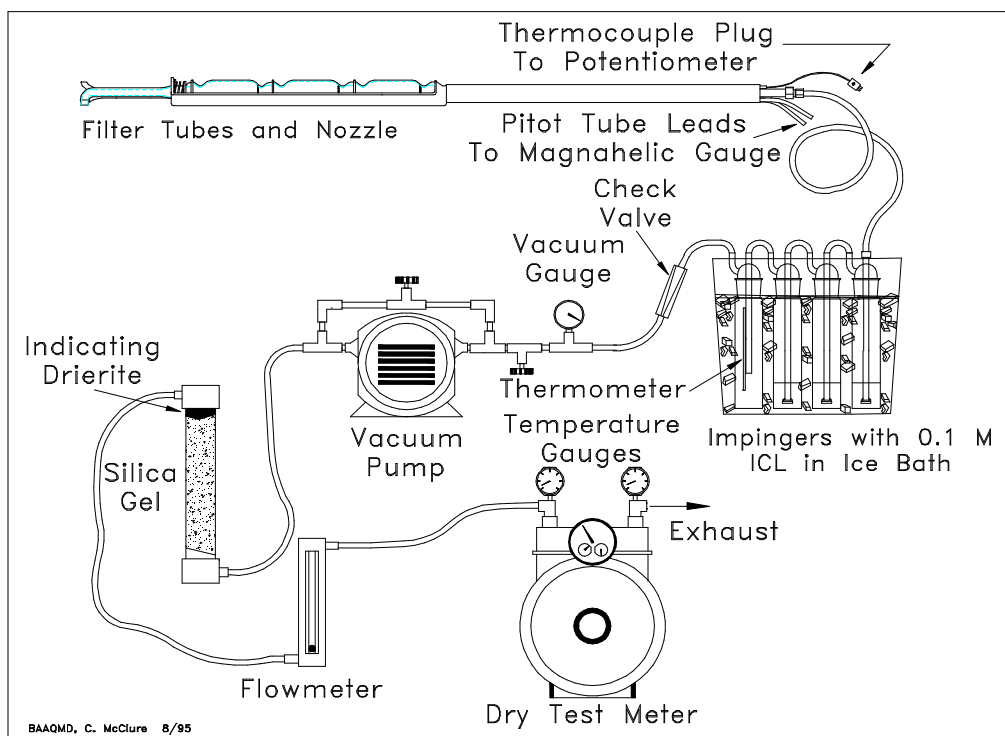
Where:

- Q = Isokinetic Sampling Rate, ACFM
 T_m = Meter Temperature, $^{\circ}R$
 H_2O = Stack Gas Moisture Content (from 7.2.3)
 2.638×10^{-3} = Constant derived from 60 sec/min, $70^{\circ}F$, 29.92 inches Hg
 and the molecular weight

- 8.4 When inserting the probe into the stack rotate the nozzle so it points down stream to avoid particulate collection prior to sampling. Immediately before sampling rotate the probe so that the nozzle points upstream.

Figure 10-3

Mercury Sampling Train



- 8.5 Record the following information at five-minute intervals or whenever changing sampling locations on a field data form as shown in Form 10-2.

Stack Velocity Head
 Sample Time
 Sample Rate
 Cumulative Sample volume
 Impinger Saturation Temperature
 Stack Gas Temperature
 Impinger Vacuum
 Dry Test Meter Temperature

- 8.6 Add ice as necessary to maintain impinger temperature at 7 °C (45°F) or less.
- 8.7 At the conclusion of each run, stop the pump, remove the probe from the stack, record the final meter reading. Point the probe upward and purge the sample train with ambient air.
- 8.8 Take three consecutive samples.

9. POST-TEST PROCEDURES

- 9.1 Stopper the impingers to minimize sample losses.
- 9.2 Weigh each impinger to the nearest 0.1 g.

10. AUXILIARY TESTS

- 10.1 Determine the CO₂, O₂, and CO concentrations simultaneously with each particulate run in accordance with ST-5, ST-14, and ST-6. An Orsat analysis (ST-24) is also acceptable.

11. CALCULATIONS

- 11.1 Standard Dry Sample Volume

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

where:

- V_o = Standard dry sample volume, SDCF @ 70 °F and 29.92 inches Hg.
- V_m = Actual Metered Volume, ft³
- P_b = Barometric Pressure, inches Hg.
- T_m = Average Meter Temperature, °R
- 17.71 = Constant correcting to 70 °F and 29.92 inches Hg.

- 11.2 Water Vapor Content

$$H_2O = \frac{(0.0474 W_c) + \frac{V_o P_{sat}}{P_b - P_i - P_{sat}}}{V_o + (0.0474 W_c) + \frac{V_o P_{sat}}{P_b - P_i - P_{sat}}} \times 100$$

Where:

- W_c = Total condensate weight, all impingers, grams
- P_{sat} = Water saturation pressure, inches Hg
- P_b = Barometric pressure, inches Hg
- P_i = Pump inlet vacuum, inches Hg
- H_2O = Percent water vapor
- 0.0474 = Cubic feet of vapor resulting from 1 cubic centimeter of liquid H₂O.

11.3 Stack Gas Molecular Weight

$$MW = 0.44 (\%CO_2) + 0.32 (\%O_2) + 0.28 (\%N_2 + \%CO) + 0.18 (\%H_2O)$$

Where:

- MW = Molecular Weight
 %CO₂ = Percent Carbon Dioxide by volume (dry basis)
 %O₂ = Percent Oxygen by volume (dry basis)
 %CO = Percent Carbon Monoxide by volume (dry basis)
 %H₂O = Percent Moisture by volume
 %N₂ = Percent Nitrogen by volume (dry basis - determine by difference)

11.4 Stack Gas Flow Rate - Determine in accordance with ST-17.

11.5 Total mercury emissions. The emission rate of mercury shall be calculated as:

$$M = \frac{W \times 10^6 \times Q_o \times 60 \times T}{V_o}$$

Where:

- M = Mass emission rate, g/day
 W = Total weight of Mercury collected, μg (micrograms)
 Q_o = Stack gas flowrate, SDCFM
 V_o = Sample Volume, SDCF
 T = Plant operation, hr/day
 60 = minutes/hour

11.6 Isokinetic Ratio. Calculate for each traverse point as:

$$R_i = \frac{T_{si} Q_{mi}}{60(100 - H_2O) A V_{si} t_i T_m} \times 100\%$$

Where:

- R_i = Isokinetic ratio at given point, i
 t_i = Time, at point i, minutes
 A = Nozzle area, ft²
 V_{si} = Stack velocity, point i, FPS
 T_{mi} = Meter temperature, point i, °R
 T_{si} = Stack temperature, point i, °R
 Q_{mi} = Metered volume, point i
 60 = Minutes/hr.

Overall isokinetic ratio for each run:

$$R = \frac{Q_{mi}}{60(100 - H_2O) A T_m V_{si} t_i T_{si}} \times 100\%$$

12. REPORTING

12.1 The data and information indicated in Form 10-3 shall be reported.

Bay Area Air Quality Management District

Form 10-1

Source Test Laboratory Data Sheet

Impinger Weightings

Plant Name: _____	Plant Number: _____
Source Operation: _____	Test Date: _____
Source Test #: _____	Page: <u>1</u> of _____
Impinger Solution: _____	Initial: _____

Impinger I. D. #	(A) Tare Weight (g)	(B) Filled Weight (g)	(C) Final Weight (g)

Impinger I. D. #	(C-A) Sample Weight (g)	(C-B) Condensate Wt. (g)	Condensate Weight / Run (g)
			Run A
			Run B
			Run C
			Run D

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

Form 10-2
Source Test Data Sheet

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

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

Run # _____ Date: _____
 Temp Meter # _____ Box ΔH@ _____
 Mag. Gauge # _____ Meter (Y) _____

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 10-3

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-17	Stack Volume Flowrate, SDCFM					
	Stack Gas Temperature, °F					
ST-23	Water Content, Volume %					
ST-14	Oxygen, Volume %					
ST-5	Carbon Dioxide, Volume %					
ST-6	Carbon Monoxide, ppmv					
	Carbon Monoxide, lb/hr					
ST-10	Mercury Emissions, g/day					
	Isokinetic Ratio, act./theo.					

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