

**METHOD 102 - DETERMINATION OF PARTICULATE AND GASEOUS
MERCURY EMISSIONS FROM CHLOR-ALKALI PLANTS (HYDROGEN
STREAMS)**

NOTE: This method does not include all of the specifications (e.g., equipment and supplies) and procedures (e.g., sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part and in Appendix A to 40 CFR Part 60. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, Method 5, and Method 101.

1.0 Scope and Application.

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Mercury (Hg)	7439-97-6	Dependent upon recorder and spectrophotometer.

1.2 Applicability. This method is applicable for the determination of Hg emissions, including both particulate and gaseous Hg, from chlor-alkali plants and other sources (as specified in the regulations) where the carrier-gas stream in the duct or stack is principally hydrogen.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 *Summary of Method.*

2.1 Particulate and gaseous Hg emissions are withdrawn isokinetically from the source and collected in acidic iodine monochloride (ICl) solution. The Hg collected (in the mercuric form) is reduced to elemental Hg, which is then aerated from the solution into an optical cell and measured by atomic absorption spectrophotometry.

3.0 *Definitions.* [Reserved]

4.0 *Interferences.*

Same as Method 101, Section 4.2.

5.0 *Safety.*

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 Corrosive Reagents. Same as Method 101, Section 5.2.

5.3 Explosive Mixtures. The sampler must conduct the source test under conditions of utmost safety because hydrogen and air mixtures are explosive. Since the sampling train essentially is leakless, attention to safe operation

can be concentrated at the inlet and outlet. If a leak does occur, however, remove the meter box cover to avoid a possible explosive mixture. The following specific precautions are recommended:

5.3.1 Operate only the vacuum pump during the test. The other electrical equipment, e.g., heaters, fans, and timers, normally are not essential to the success of a hydrogen stream test.

5.3.2 Seal the sample port to minimize leakage of hydrogen from the stack.

5.3.3 Vent sampled hydrogen at least 3 m (10 ft) away from the train. This can be accomplished by attaching a 13-mm (0.50-in.) ID Tygon tube to the exhaust from the orifice meter.

NOTE: A smaller ID tubing may cause the orifice meter calibration to be erroneous. Take care to ensure that the exhaust line is not bent or pinched.

6.0 Equipment and Supplies.

Same as Method 101, Section 6.0, with the exception of the following:

6.1 Probe Heating System. Do not use, unless otherwise specified.

6.2 Glass Fiber Filter. Do not use, unless otherwise specified.

7.0 *Reagents and Standards.*

Same as Method 101, Section 7.0.

8.0 *Sample Collection, Preservation, Transport, and Storage.*

Same as Method 101, Section 8.0, with the exception of the following:

8.1 *Setting of Isokinetic Rates.*

8.1.1 If a nomograph is used, take special care in the calculation of the molecular weight of the stack gas and in the setting of the nomograph to maintain isokinetic conditions during sampling (Sections 8.1.1.1 through 8.1.1.3 below).

8.1.1.1 Calibrate the meter box orifice. Use the techniques described in APTD-0576 (see Reference 9 in Section 17.0 of Method 5). Calibration of the orifice meter at flow conditions that simulate the conditions at the source is suggested. Calibration should either be done with hydrogen or with some other gas having similar Reynolds Number so that there is similarity between the Reynolds Numbers during calibration and during sampling.

8.1.1.2 The nomograph described in APTD-0576 cannot be used to calculate the C factor because the nomograph is designed for use when the stack gas dry molecular weight is 29 ± 4 . Instead, the following calculation should be made

to determine the proper C factor:

$$C = 0.00154) H_{\text{e}} C_p^2 T_m (P_s / P_m) \frac{(1 - B_{ws})^2}{(1 - B_{ws}) + 18 B_{ws} / M_d} \quad \text{Eq. 102-1}$$

where:

B_{ws} = Fraction by volume of water vapor in the stack gas.

C_p = Pitot tube calibration coefficient, dimensionless.

M_d = Dry molecular weight of stack gas, lb/lb-mole.

P_s = Absolute pressure of stack gas, in. Hg.

P_m = Absolute pressure of gas at the meter, in. Hg.

T_m = Absolute temperature of gas at the orifice, °R.

$)H_{\text{e}}$ = Meter box calibration factor obtained in Section 8.1.1.1, in. H₂O.

0.00154 = (in. H₂O/°R).

NOTE: This calculation is left in English units, and is not converted to metric units because nomographs are based on English units.

8.1.1.3 Set the calculated C factor on the operating nomograph, and select the proper nozzle diameter and K factor as specified in APTD-0576. If the C factor obtained in Section 8.1.1.2 exceeds the values specified on the existing operating nomograph, expand the C scale

logarithmically so that the values can be properly located.

8.1.2 If a calculator is used to set isokinetic rates, it is suggested that the isokinetic equation presented in Reference 13 in Section 17.0 of Method 101 be consulted.

8.2 Sampling in Small (<12-in. Diameter) Stacks. When the stack diameter (or equivalent diameter) is less than 12 inches, conventional pitot tube-probe assemblies should not be used. For sampling guidelines, see Reference 14 in Section 17.0 of Method 101.

9.0 Quality Control.

Same as Method 101, Section 9.0.

10. Calibration and Standardizations.

Same as Method 101, Section 10.0.

11.0 Analytical Procedure.

Same as Method 101, Section 11.0.

12.0 Data Analysis and Calculations.

Same as Method 101, Section 12.0.

13.0 Method Performance.

Same as Method 101, Section 13.0.

13.1 Analytical Range. After initial dilution, the range of this method is 0.5 to 120 $\mu\text{g Hg/ml}$. The upper limit can be extended by further dilution of the sample.

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 *References.*

Same as Method 101, Section 16.0.

17.0 *Tables, Diagrams, Flowcharts, and Validation Data.*

[Reserved]