

<b>METHOD #:</b> 253.1	Approved for NPDES (Technical Revision 1978)
<b>TITLE:</b>	Palladium (AA, Direct Aspiration)
<b>ANALYTE:</b>	CAS # Pd Palladium 7440-05-3
<b>INSTRUMENTATION:</b>	AA
<b>STORET No.</b>	Total 01210
<b>Optimum Concentration Range:</b>	0.5-15 mg/L using a wavelength of 247.6 nm
<b>Sensitivity:</b>	0.25 mg/L
<b>Detection Limit:</b>	0.1 mg/L

### 1.0 Preparation of Standard Solution

- 1.1 Stock Solution: Dissolve 0.1000 g of palladium wire in a minimum volume of aqua regia and evaporate just to dryness. Add 5 mL conc. HCl and 25 mL deionized water and warm until dissolution is complete. Dilute to 100 mL with deionized water. (1 mL = 1 mg Pd).
- 1.2 A standard AAS solution of palladous chloride, PdCl<sub>2</sub>, 1000 mg/L in aqueous matrix is available from Alfa Products, Beverly, Massachusetts 01915. Cat. #88085.
- 1.3 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The calibration standards should be prepared to contain 0.5% (v/v) HNO<sub>3</sub>.

### 2.0 Sample Preservation

- 2.1 For sample handling and preservation, see part 4.1 of the Atomic Absorption Methods section of this manual.

### 3.0 Sample Preparation

- 3.1 Transfer a representative aliquot of the well mixed sample to a Griffin beaker and add 3 mL of conc. distilled HNO<sub>3</sub>. Place the beaker on a steam bath and evaporate to near dryness. Cool the beaker and cautiously add a 5 mL portion of aqua regia. (See below for preparation of aqua regia.\* ) Cover the beaker with a watch glass and return to the steam bath. Continue heating the covered beaker for 30 minutes. Remove cover and evaporate to near dryness. Cool and add 1:1 redistilled HNO<sub>3</sub> (1 mL per 100 mL dilution). Wash down the beaker walls and watch glass with distilled water and filter the sample to remove silicates and other insoluble material that could clog the atomizer. Adjust the

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\*Aqua regia-prepare immediately before use by carefully adding three volume of conc. HCl to one volume of conc. HNO<sub>3</sub>.

volume to some predetermined value based on the expected metal concentration. The sample is now ready for analysis.

#### 4.0 Instrumental Parameters (General)

4.1 Palladium hollow cathode lamp

4.2 Wavelength: 247.6 nm

4.3 Fuel: Acetylene

4.4 Oxidant: Air

4.5 Type of flame: Oxidizing

#### 5.0 Analysis Procedure

5.1 For analysis procedure and calculation, see "Direct Aspiration", part 9.1 of the Atomic Absorption Methods section of this manual.

#### 6.0 Notes

6.1 For concentrations of palladium below 0.25 mg/L, the furnace procedure, Method 253.2, is recommended.

#### 7.0 Precision and Accuracy

7.1 Precision and accuracy data are not available at this time.