

**METHOD #:** 265.1 Approved for NPDES (Technical Revision 1978)  
**TITLE:** Rhodium (AA, Direct Aspiration)  
**ANALYTE:** CAS # Rh Rhodium 7440-16-6  
**INSTRUMENTATION:** AA  
**STORET No.** Total Not Assigned  
**Optimum Concentration Range:** 1-30 mg/L using a wavelength of 343.5 nm  
**Sensitivity:** 0.3 mg/L  
**Detection Limit:** 0.05 mg/L

#### 1.0 Preparation of Standard Solution

- 1.1 Stock Solution: Dissolve 0.3768 g of ammonium chlororhodite,  $(\text{NH}_4)_3\text{RhCl}_6 \cdot \text{H}_2\text{O}$ , in a minimum volume of 10% (v/v) HCl and dilute to 100 mL with 10% HCl. (1 mL = 1mg Rh).
- 1.2 A standard AAS solution of rhodium trichloride,  $\text{RhCl}_3$ , 1000 mg/L in aqueous matrix is available from Alfa Products, Beverly, Massachusetts 01915. Cat. #88090
- 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)  $\text{HNO}_3$ .

#### 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  $\text{HNO}_3$ . 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 distilled  $\text{HNO}_3$  (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 volume to some predetermined value based on the expected metal concentration. The sample is now ready for analysis.

#### 4.0 Instrumental Parameters (General)

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\*Aqua regia-prepare immediately before use by carefully adding three volumes of conc. HCl to one volume of conc.  $\text{HNO}_3$ .

- 4.1 Rhodium hollow cathode lamp
- 4.2 Wavelength: 343.5 nm
- 4.3 Fuel: Acetylene
- 4.4 Oxidant: Air
- 4.5 Type of flame: Oxidizing

## 5.0 Analysis Procedure

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

## 6.0 Notes

- 6.1 For concentrations of rhodium below 0.2 mg/L, the furnace procedure, Method 265.2, is recommended.

## 7.0 Precision and Accuracy

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