METHOD #: 283.1 Approved for NPDES (Issued 1974)

TITLE: Titanium (AA, Direct Aspiration)

ANALYTE: CAS # Ti Titanium 7440-32-6

INSTRUMENTATION: AA

STORET No. Total 01152

Dissolved 01150 Suspended 01151

Optimum Concentration Range: 5-100 mg/L using a wavelength of 365.3 nm

Sensitivity: 2 mg/L **Detection Limit:** 0.4 mg/L

1.0 Preparation of Standard Solution

- 1.1 Stock solution: Dissolve 1.000 g of titanium metal (powder or small pieces) in 200 mL 6N HCl. Heat to near 100° C to effect dissolution. Cool and dilute to 1 liter with deionized water. 1 mL = 1 mg Ti (1000 mg/L)
- 1.2 Potassium chloride solution: Dissolve 95 g potassium chloride, KCl, in distilled water and make up to 1 liter.
- 1.3 Prepare dilutions of the stock titanium solution to be used as calibration standards at the time of analysis. The calibration standards should be prepared using the same type of acid and at the same concentration as will result in the sample to be analyzed either directly or after processing. To each 100 mL of standard and sample alike, add 2 mL of potassium chloride solution.

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 The procedures for preparation of the sample as given in parts 4.1.1 thru 4.1.3 of the Atomic Absorption Methods section of this manual have been found to be satisfactory with the following modification. For processing total and suspended titanium, concentrated H_2SO_4 (2 mL per 100 mL of final dilution) must be added in addition to the nitric acid. Reflux the sample adding additional nitric acid as needed. When solubilization is complete, heat until the appearance of SO_3 fumes. Cool and add sufficient distilled nitric acid so that the final dilution contains 0.5% (v/v) HNO_3 .

4.0 Instrumental Parameters (General)

- 4.1 Titanium hollow cathode lamp
- 4.2 Wavelength: 365.3 nm
- 4.3 Fuel: Acetylene
- 4.4 Oxidant: Nitrous Oxide
- 4.5 Type of flame: Fuel rich

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 Interferences

6.1 A number of elements increase the sensitivity of titanium. To control this problem, potassium (1000 mg/L) must be added to standards and samples alike. [Atomic Absorption Newsletter 6, p 86 (1967)]

7.0 Notes

- 7.1 For concentrations of titanium below 1.0 mg/L, the furnace procedure, Method 283.2, is recommended.
- 7.2 Data to be entered into STORET must be reported as μ g/L.

8.0 Precision and Accuracy

8.1 In a single laboratory (EMSL), using a mixed industrial-domestic waste effluent spiked at concentrations of 2.0, 10 and 50 mg Ti/L, the standard deviations were ± 0.07 , ± 0.1 and ± 0.4 , respectively. Recoveries at these levels were 97%, 91% and 88%, respectively.