

METHOD #: 286.1 Approved for NPDES (Issued 1974)
TITLE: Vanadium (AA, Direct Aspiration)
ANALYTE: CAS # V Vanadium 7440-62-2
INSTRUMENTATION: AA
STORET No. Total 01087
Dissolved 01085
Suspended 01086
Optimum Concentration Range: 2-100 mg/L using a wavelength of 318.4 nm
Sensitivity: 0.8 mg/ L
Detection Limit: 0.2 mg/L

1.0 Preparation of Standard Solution

- 1.1 Stock Solution: Dissolve 1.7854 g of vanadium pentoxide, V_2O_5 (analytical reagent grade) in 10 mL of conc. nitric acid and dilute to 1 liter with deionized distilled water. 1 mL = 1 mg V (1000 mg/L).
- 1.2 Aluminum nitrate solution: Dissolve 139 g aluminum nitrate, $Al(NO_3)_3 \cdot 9H_2O$, in 150 mL of deionized distilled water; heat to effect solution. Allow to cool and make up to 200 mL.
- 1.3 Prepare dilutions of the stock vanadium 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 the aluminum nitrate 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 4.1.4 of the Atomic Absorption Methods section of this manual have been found to be satisfactory.

4.0 Instrumental Parameters (General)

- 4.1 Vanadium hollow cathode lamp
- 4.2 Wavelength: 318.4 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 It has been reported that high concentrations of aluminum and titanium increase the sensitivity of vanadium. This interference can be controlled by adding excess aluminum (1000 ppm) to both samples and standards. [Talanta 15, 871(1968)].
- 7.0 Notes
- 7.1 For concentrations of vanadium below 0.5 mg/L, the furnace procedure, Method 286.2, is recommended.
- 7.2 The gallic acid colorimetric method may also be used (Standard Methods, 14th Edition, p 260).
- 7.3 Data to be entered into STORET must be reported as $\mu\text{g/L}$.
- 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 V/L, the standard deviations were ± 0.10 , ± 0.1 and ± 0.2 , respectively. Recoveries at these levels were 100%, 95% and 97%, respectively.