

METHOD #: 215.1 Approved for NPDES (Editorial Revision 1974)

TITLE: Calcium (AA, Direct Aspiration)

ANALYTE: CAS # Ca Calcium 7440-70-2

INSTRUMENTATION: AA

STORET No. Total 00916
Dissolved 00915

Optimum Concentration Range: 0.2-7 mg/L using a wavelength of 422.7 nm

Sensitivity: 0.08 mg/L

Detection Limit: 0.01 mg/L

1.0 Preparation of Standard Solution

- 1.1 Stock Solution: Suspend 1.250 g of CaCO_3 (analytical reagent grade), dried at 180°C for 1 hour before weighing, in deionized distilled water and dissolve cautiously with a minimum of dilute HCl. Dilute to 1000 mL with deionized distilled water. 1 mL = 0.5 mg Ca (500 mg/L).
- 1.2 Lanthanum chloride solution: Dissolve 29 g of La_2O_3 , slowly and in small portions, in 250 mL conc. HCl (Caution: Reaction is violent) and dilute to 500 mL with deionized distilled water.
- 1.3 Prepare dilutions of the stock calcium solutions to be used as calibration standards at the time of analysis. To each 10 mL volume of calibration standard and sample alike add 1.0 mL of the lanthanum chloride solution, i.e., 20 mL of standard or sample + 2 mL LaCl_3 = 22 mL.

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 For the analysis of total calcium in domestic and industrial effluents, the procedures for the determination of total metals as given in parts 4.1.3 and 4.1.4 of the Atomic Absorption Methods section of this manual have been found to be satisfactory.
- 3.2 For ambient waters, a representative aliquot of a well-mixed sample may be used directly for analysis. If suspended solids are present in sufficient amounts to clog the nebulizer, the sample may be allowed to settle and the supernatant liquid analyzed directly.

4.0 Instrumental Parameters (General)

- 4.1 Calcium hollow cathode lamp

- 4.2 Wavelength: 422.7 nm
- 4.3 Fuel: Acetylene
- 4.4 Oxidant: Air
- 4.5 Type of flame: Reducing

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 Phosphate, sulfate and aluminum interfere but are masked by the addition of lanthanum. Since low calcium values result if the pH of the sample is above 7, both standards and samples are prepared in dilute hydrochloric acid solution. Concentrations of magnesium greater than 1000 mg/L also cause low calcium values. Concentrations of up to 500 mg/L each of sodium, potassium and nitrate cause no interference.
- 6.2 Anionic chemical interferences can be expected if lanthanum is not used in samples and standards.
- 6.3 The nitrous oxide-acetylene flame will provide two to five times greater sensitivity and freedom from chemical interferences. Ionization interferences should be controlled by adding a large amount of alkali to the sample and standards. The analysis appears to be free from chemical suppressions in the nitrous oxide-acetylene flame. (Atomic Absorption Newsletter 14, 29 [1975]).
- 6.4 The 239.9 nm line may also be used. This line has a relative sensitivity of 120.
- 6.5 Data to be entered into STORET must be reported as mg/L.
- 6.6 The EDTA titrimetric method may also be used (Standard Methods, 14th Edition, p 189).

7.0 Precision and Accuracy

- 7.1 In a single laboratory (EMSL), using distilled water spiked at concentrations of 9.0 and 36 mgCa/L, the standard deviations were ± 0.3 and ± 0.6 , respectively. Recoveries at both these levels were 99%.