

Determination of Calcium in Natural Water by Atomic Absorption Spectrophotometry

Eugene M. Bentley and G. Fred Lee

Water Chemistry Laboratory, University of Wisconsin, Madison, Wis. 53706

■ The determination of calcium in natural waters by atomic absorption spectrophotometry (AAS) is highly pH dependent. The response of the AAS for a constant calcium concentration in natural water increased with decreasing pH in the range of pH 7.0 to 3.8. It was constant in the pH range of 3.8 to 1.8 and decreased with decreasing pH below 1.8. Correct analyses were obtained when samples were adjusted to pH 1.8 to 3.8. The pH effect on calcium response above pH 3.8 is due to bicarbonate. Calcium carbonate precipitation occurs in the burner and causes the low response. In addition to pH adjustment, the addition of 50% of the EDTA required to titrate the hardness of the water or the addition of 1% lanthanum will eliminate the pH dependence of calcium response.

Since water samples are routinely analyzed for calcium, a quick, accurate, and precise analytical procedure is desirable. Analytical procedures currently employed for the determination of calcium are EDTA titration, precipitation, and weighing of calcium oxalate, redissolving precipitated calcium oxalate and titrating with permanganate, flame photometry, and atomic absorption spectrophotometry. The atomic absorption spectrophotometric method of analysis for calcium compares favorably with other methods (West and Herring, 1965). Some of the problems encountered with other methods of analysis, such as difficulty in determining colorimetric end points, the large sample volumes required, and interferences from other elements, are not encountered in the atomic absorption spectrophotometric procedure (Butler and Drink, 1963). The literature on the use of atomic absorption spectrophotometry for water analysis has been reviewed (Bentley, 1967).

An atomic absorption spectrophotometer is currently utilized for calcium analysis of lake, stream, and river water samples in this laboratory. The procedure is quick, accurate, and precise; however, some problems have been encountered. The apparent calcium concentration obtained by this procedure is pH dependent—i.e., as the pH is varied, the apparent

calcium concentration varies. This paper presents the results of a study to determine why the apparent calcium concentration obtained by this procedure is pH dependent.

Experimental

Calcium analyses were performed using a Perkin-Elmer atomic absorption spectrophotometer, Model 303. Measurements were made at a wavelength of 4227 Å, and a slit setting of 4. The visible range was used and the source current was set at 14 ma. A scale setting of 1 and an air flow rate of 5 flow-rater units was employed. Before the start of each series of analyses, the gas (acetylene) flow rate was adjusted to give maximum absorbance while aspirating a standard solution. This value was usually 9.5 flow-rater units. The aspiration rate was checked by using a stopwatch and graduated cylinder. Plugging of the aspirator was not excessive and, when it occurred, was rectified by aspirating 1 to 1 hydrochloric acid for 1 minute.

A Beckman pH meter, Model GS, was used to make pH measurements on each sample before determination of calcium. Each sample was kept in a covered volumetric flask between the time of preparation and the time of analysis.

Sodium, potassium, and magnesium were determined with the atomic absorption spectrophotometer utilizing the manufacturer's recommended instrument settings (Perkin-Elmer Corp., 1966).

Reagent grade chemicals were used to prepare all solutions utilized in the experimental work.

Unless otherwise specified, sample refers to Lake Mendota water samples which were obtained from the Water Chemistry Laboratory lake water tap. This system provides untreated lake water from approximately 600 feet offshore from a depth of about 10 feet below the surface. Samples from other lakes were obtained on field trips and stored in polyethylene bottles at 5° C. All samples were filtered with a Whatman No. 1 paper, and glass-distilled water was used to prepare dilutions, as well as to zero the atomic absorption spectrophotometer.

Synthetic lake water was prepared by weighing and diluting with glass-distilled water, chemicals in sufficient quantities to give values of elements comparable to those found in Lake Mendota (Lee, 1966). This solution was used in the deter-

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