A New Method for the Determination of Phosphorus by Atomic Absorption Spectrophotometry Using Molybdenum as a Light Source*[1]

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(Received November 22, 1966)

In atomic absorption spectrophotometry, a hollow cathode lamp which emits the bright spectral lines belonging to the element to be determined is generally used as a light source. Because of this restriction, it has been impossible to determine most non-metals by the atomic absorption method.

In the cource of our studies on application of atomic absorption spectrophotometry for determination of anions,(1,2) it was found that a small amount of phosphorus could be determined under the following principle.

It has been well known in the conventional absorption spectrophotometric methods for the determination of phosphorus that molybdophosphoric acid formed by the reaction between phosphate and molybdate ions in the slightly acidic solution can be extracted into various organic solvents such as *n*-butyl acetate,(3,4) ethyl acetate,(4,5) methyl isobutyl ketone,(6) *n*-butanol in chloroform,(7,8) *n*-butanol(9) or isobutanol.(10) It appears possible to determine phosphorus by spraying the extracted organic solution into the flame of an atomic absorption spectrophotometer having a molybdenum hollow cathode lamp. To find a suitable solvent for atomic absorption spectrophotometry, various solvents were tested: *n*-butyl acetate proved to be the most effective extractant, not only because of its higher selectivity and good extractability for molybdophosphoric acid but also its better combustibility in the flame.

To establish the optimum conditions for the determination of phosphorus, various factors on the intensity were studied: kind of fuel, fuel pressure, lamp current, burner height and so on. The recommended procedure for the calibration curve is as follows.

Five milliliters of hydrochloric acid (2.5 N), 2.5 m*l* of sodium molybdate solution (5%), and varying amounts of the standard ammonium dihydrogen phosphate solution (10.0 ppm as P_2O_5, 0—2.0 m*l*) are mixed. After the solution is diluted with water to 25 m*l* and allowed to stand for 5 min, it is shaken for 2 min with 10.0 m*l* of *n*-butyl acetate. The organic phase is separated and dried with 1 g of anhydrous sodium sulfate. The atomic absorption is then measured using a Nippon Jarrell-Ash Model AA-1 atomic absorption spectrophotometer equipped with a molybdenum hollow cathode lamp as a light source at the 3133 Å molybdenum line. An air - nitrous oxide - acetylene flame through a 5 cm slit burner is used with the flow rate of 6.0 *l*/min, 3.4 *l*/min, and 2.1 *l*/min, respectively.

Under the procedure described above, in which the conditions for the solvent extraction were followed to those in the previous paper,(3) the analytical curve was found to be linear for the tested range (0—0.35 ppm) of concentration of phosphorus as phosphate ion in the aqueous solution. The sensitivity of the proposed method was found to be comparable and effects of diverse ions appeared to be the same as those for the absorption spectrophotometric methods. For example, silicon, arsenic and germanium do not interfere with the determination of phosphorus because of their unextractability into the solvent. The proposed method may be applied to the determination of phosphorus in the samples such as iron, steel, biological materials or industrial water, if the phosphorus could be converted into the phosphate by a suitable oxidation.

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*[1] Added in proof: After the submisson of this article, W. S. Zaugg and R. J. Knox have reported in a brief note that they proposed a method of indirect determination of inorganic phosphate by atomic absorption spectrophotometric determination of molybdenum using 2-octanol as the solvent [*Anal. Chem.*, **38**, 1759 (1966)].

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