Phosphorous

For water, wastewater and seawater

Amino Acid, Ascorbic Acid and Molybdovanadate Methods

Introduction

Phosphorus occurs in natural water and wastewaters almost solely as phosphates. Phosphates may enter water from agricultural run-off and as biological and industrial wastes. They may be added to water in municipal and industrial water treatment processes to control corrosion. A certain amount of phosphate is essential for most plants and animals, but too much phosphate in water can contribute to eutrophication, especially when large amounts of nitrogen are also present.

Phosphorus can be classified as orthophosphate, condensed phosphate or organically bound phosphate. Condensed phosphates are formed by dehydrating the orthophosphate radical; they include metaphosphate, pyrophosphate and polyphosphate. The only form of phosphate determined directly is orthophosphate; other forms require pretreatment for conversion to orthophosphate for analysis. When no pretreatment is used, phosphate analyses determine Reactive Phosphorus. Reactive phosphorus is a measure of orthophosphate, plus a small fraction of condensed phosphate that may have been hydrolyzed during the test.

Hach offers high and low range tests for reactive phosphorus. High range tests can be completed with the Amino Acid Method or the Molybdovanadate Method. The Molybdovanadate Method uses a single reagent and has a faster reaction than the Amino Acid Method. Both methods have a broad range and are free from most interferences. Low range tests use the Ascorbic Acid Method.

Condensed phosphates plus orthophosphate can be determined by acid hydrolysis using sulfuric acid, followed by the reactive phosphorus test for the appropriate range. A small amount of organically bound phosphorus will be included in this measurement. The results of the test are reported as acid-hydrolyzable phosphorus. Total phosphorus (orthophosphate, condensed and organically bound) can be determined by acid oxidation with persulfate, followed by the reactive phosphorus test. Organically bound phosphate can then be determined by subtracting the acid-hydrolyzable phosphorus.

Chemical reactions

Pretreatment steps
Reactions for pretreatment to determine acid-hydrolyzable and total phosphorus are illustrated below:

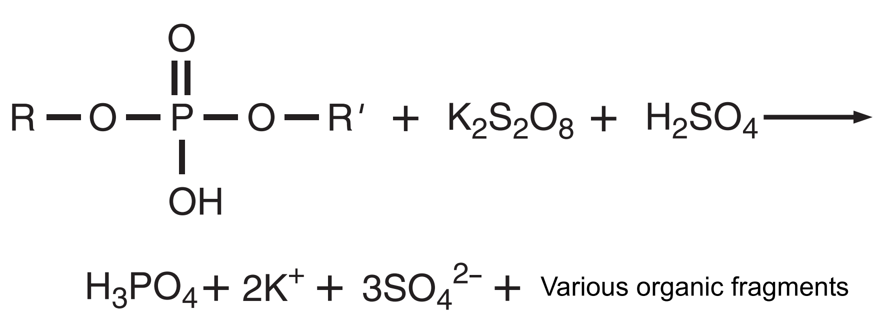

Figure 1 Example of potassium persulfate oxidation of organically bound phosphorus[1]

[1] R and R’ represent various organic groups

Amino acid and ascorbic acid methods
Reactive phosphorus is determined in essentially two steps for either the Ascorbic Acid Method (low range) or the Amino Acid Method (high range). The first step involves reaction of orthophosphate with molybdate in acid solution, which forms a yellow-colored phosphomolybdate complex:

$12MoO_3 + H_2PO_4^-$ ç $(H_2PMo_{12}O_{40})^-$

The phosphomolybdate complex is then reduced by either an amino acid or ascorbic acid, causing a characteristic molybdenum blue species. Various structures for the molybdenum blue species have been suggested in the literature. For example, see Killeffer, D. H., *Molybdenum Compounds-Their Chemistry and Technology*, Interscience Publishers, 1952.

All reagents for the Ascorbic Acid Method are contained in PhosVer™3 Reagent Powder Pillows. Reagents for the Amino Acid Method are contained in Amino Acid Reagent Solution and Molybdate Reagent Solution.

Molybdovanadate method
Reactive phosphorus combines with molybdate in an acid medium to form a phosphomolybdate complex. Vanadium, contained in Molybdovanadate Reagent, reacts with the complex to form vanadomolybdophosphoric acid. Intensity of the resulting yellow color is proportional to the concentration of reactive phosphorus. One possible formula for the complex is suggested below. The exact structure is not known.

$[PO_4$ ñ VO_3 ñ $16MoO_3]^{4-}$