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Olsen Phosphate (Sodium Bicarb Phosphate)

FIAlab standard method for the Olsen phosphate (Sodium Bicarb phosphate) assay using the FIAlab-2500/2600/2700 system.

Assay	Typical Throughput	Concentration Range	Notes
Phosphate (Mid to High)	120 samples/hour	10 to 300 mg (P)/L	1 cm flow cell
Phosphate (Low)	100 samples/hour	2 to 60 mg (P)/L	10 cm flow cell

Principle:

Soluble phosphorus is typically found in three forms: **1.)** Orthophosphate (i.e. PO43-); **2.)** Organo-phosphates (R-P04H-R') and **3.)** Condensed phosphates (metaphosphates, pyrophospates, and polyphosphates). In this method, all three forms can be analyzed, but only *reactive phosphorus* can be directly determined without any pretreatment. *Reactive phosphorus* is simply hydrolyzed orthophosphates. Orthophosphates react directly with molybdate anions to form a yellow-colored phosphomolybdate complex. This complex is then reduced by ascorbic acid to create a *molybdenum blue* species that has a broad absorbance range from 700nm to 900nm, making this method suitable for a variety of light sources and detectors.

Comments:

Olsen (Sodium Bicarb) extracted samples contain high quantities of sodium bicarbonate which normally cause bubbles (foam) to be created when the sample comes in contact with the acidic reagents. This will cause significant problems with accurate absorbance measurements in the flow cell.

Solution: By putting a FIAlab supplied OP pressure restrictor on the flow cell output line, and running the pump at 65%, the resulting overpressure prevents gas from coming out of solution. In this fashion, Olsen phosphate can then be run like other phosphate extractions (e.g., Bray).

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Due to common "brown" coloring in samples, Olsen phosphate must usually be run using 880 nm for the primary and 925 nm for the reference wavelength. It is recommended that a NIR LED light source be used instead of the LS1 Tungsten lamp.

The sample loop should be 6 inches of .03" tubing.

Heat is set to 45 C.

For lower concentrations (commonly occurring in soil samples), the 10 cm flow cell is recommended.

It is critical that PEEK insert tube adaptors be used for the Carrier, R1, and R2 lines (between pump and LOV manifold) to prevent the pump tubing from popping off due to increase pressure.

Interferences:

The colorimetric *molybdenum blue* reaction is sensitive to changes in acidity, and works best in the pH 6-8 range. The sample should not have a strong matrix absorbance at the selected detection wavelength. Turbidity will cause high phosphate results and noisy data. This can be a problem with acid digested samples. High iron concentrations (>50 mg/L) will decrease the phosphate signal. While the presence of As3+ can cause high phosphorus results.

Reagents:

Carrier: DI Water
1-Liter Degassed DI Water.

Reagent 1: 6 mM Ammonium Molybdate.
10.0 grams Ammonium molybdate tetra-hydrate [1235.81 FW] 0.2 grams Antimony Potassium Tartrate half-hydrate [333.94 FW] *catalyst*
40 mL conc. H2SO4 acid (36N) ACS grade (Sigma-Aldrich 38,337-6)

1-Liter Degassed DI Water. Place acid into 800mL of DI water, mix and let cool to room temperature. Add molybdate and antimony potassium tartrate and mix until dissolved. Fill flask to the mark. Transfer solution into a dark and airtight glass bottle for maximum longevity. This solution is stable for several weeks.

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Reagent 2: Reagent Carrier Stream of 300mM Ascorbic Acid.
30 grams Ascorbic acid [176.12 FW] 1.0 grams Sodium dodecyl sulfate [288.38 FW] surfactant (Sigma-Aldrich 436143-25G)

1-Liter Degassed DI Water. Place the ascorbic acid into a 1-liter volumetric flask and mix with 600 cc of DI water until dissolved. Add the sodium dodecyl sulfate and mix slowly (prevent foaming) until dissolved. Fill flask to the mark. Transfer solution into an airtight light sensitive glass bottle for maximum longevity. Minimize exposure to air and prepare fresh weekly since this solution is unstable.

Standards:

100ml ICPHO-100 (Phosphate standard)
Source: 727-524-7732 - www.exaxol.com

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