

## Total Phosphorus

FIALab standard method for total phosphorus assay using the FIALab 2500/2600/2700 system. Variations of this method for lower/higher concentration samples exist, please contact FIALab for additional details.

Assay	Typical Throughput	Concentration Range	Notes
Total Phosphorus (mid)	80 samples/hour	0.1 to 25 mg (P/L)	1 cm flow cell
Total Phosphorus (low)	80 samples/hour	0.01 to 2.5 mg (P/L)	10 cm flow cell

### Principle:

Samples are first digested in a block digester prior to FIA assay, converting total phosphorus to reactive phosphorus. This can be then measured through the standard reactive phosphorus assay. Soluble phosphorus is typically found in three forms:

- 1.) Orthophosphate (i.e. PO<sub>4</sub><sup>3-</sup>);
- 2.) Organo-phosphates (R-P<sub>0</sub>4H-R') and
- 3.) Condensed phosphates (metaphosphates, pyrophosphates, 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:

A heater set to 45 C is recommended for concentrations < 100 ppm. The flow rate of the pump should be set to 50. The FIA LOV connections B should be bridged by a simple tubing (no Cadmium Column). Recommended wavelengths: 860 nm for the primary and 490 reference.

The sample loop from three inches of green tubing (.03" ID).

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**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. Very high Silica concentrations (> 3000 ppm) can result in a positive error.

**Reagents:**

**Carrier: DI Water and H<sub>2</sub>SO<sub>4</sub> Acid**

**1-Liter Degassed DI Water.** Add [approx.] 60 mL conc. H<sub>2</sub>SO<sub>4</sub> acid (36N) ACS grade (Sigma-Aldrich 38,337-6). The acid is to match the total phosphate matrix, so the actual quantity may vary with the specific digestion process. This becomes more critical at low sample concentrations.

**Reagent 1: Sample Carrier Stream of 6mM Ammonium Molybdate**

**10.0 grams Ammonium molybdate tetra-hydrate [1235.81 FW] (Sigma-Aldrich 22, 123-6).** **0.2 grams** Antimony Potassium Tartrate half-hydrate [333.94 FW] catalyst (Sigma-Aldrich 38,337-6).

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

**Reagent 2: Reagent Carrier Stream of 300 mM 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

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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)

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