

Flow Solution™ FS 3700 Automated Chemistry Analyzer

Low-Level Phosphorus – All Forms
USEPA 365.1 by Flow Injection Analysis (FIA)
Cartridge Part Number 330095CT

Scope and Application

This method is used for the determination of orthophosphate in surface, and domestic and industrial wastes according to **USEPA Method 365.1**.

The methods are based on reactions that are specific for the orthophosphate ion. Thus, depending on the prescribed pretreatment of the sample, the various forms of phosphorus that may be determined are defined in **Appendix B**.

Except for in-depth and detailed studies, the most commonly measured forms are total and dissolved phosphorus and orthophosphate. Hydrolyzable phosphorus is normally found only in sewage-type samples. Insoluble forms of phosphorus are determined by calculation.

Method Performance

Range	1.0–100 µg/L
Rate	45 samples/hour
Precision	~10% RSD
Method Detection Limit (MDL)	0.3 µg/L

A general flow diagram of the Flow Injection Analysis (FIA) system is shown below.

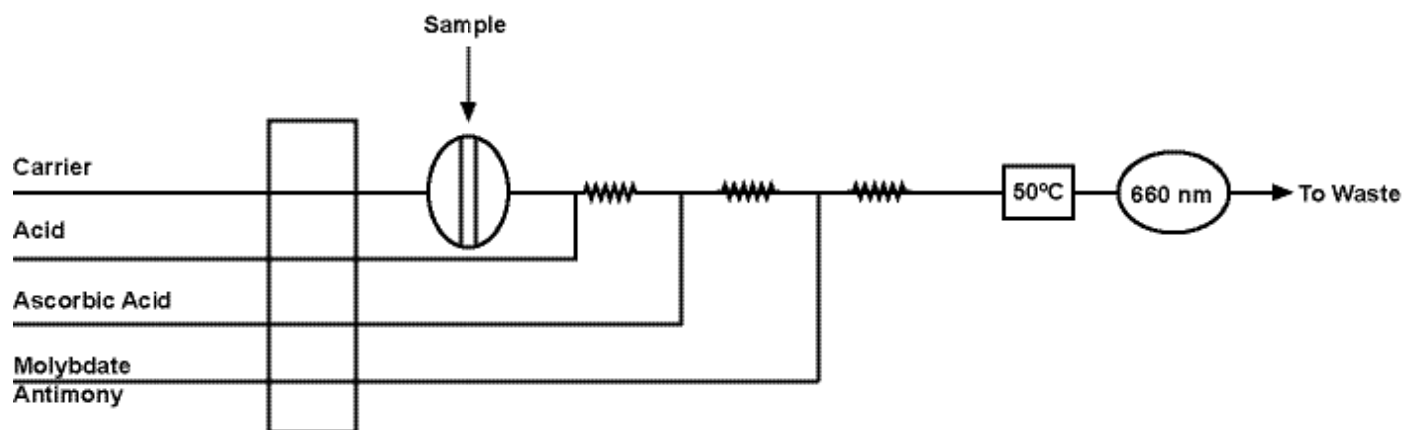


Figure 1. General Flow Diagram for Low-Level Phosphorus by USEPA 365.1

Reagents and Calibrants

Chemical Name	CAS #	Chemical Formula	Part Number
Ammonium molybdate	12054-85-2	$(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$	
Antimony potassium tartrate	28300-74-5	$\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6 \cdot 1/2 \text{H}_2\text{O}$	
Ascorbic acid	50-81-7	$\text{C}_6\text{H}_8\text{O}_6$	
DOWFAX 2A1	12626-49-2		A000080
Potassium dihydrogen phosphate	7778-77-0	KH_2PO_4	
Sulfuric acid, concentrated	7664-93-9	H_2SO_4	
Water, deionized		H_2O	

Summary of US EPA Method 365.1

US EPA Method 365.1 summarizes the method and interferences as follows below.

Method

1. Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.
2. Only orthophosphate forms a blue color in this test. Polyphosphates (and some organic phosphorus compounds) may be converted to the orthophosphate form by manual sulfuric acid hydrolysis. Organic phosphorus compounds may be converted to the orthophosphate form by manual persulfate digestion. The developed color is measured automatically.

Interferences

1. Ferric iron up to 50 mg/L, copper up to 10 mg/L, and silica up to 10 mg/L do not interfere (Reference 8). However, high iron concentrations can cause precipitation of, and subsequent loss, of phosphorus. Ferrous iron 1 mg/L and higher interferes.
2. The salt error for samples ranging from 5 - 20% salt content was found to be less than 1%.
3. Arsenate is determined similarly to phosphorus and should be considered when present in concentrations higher than phosphorus. However, at concentrations found in sea water, it does not interfere.
4. Sample turbidity must be removed by filtration prior to analysis for orthophosphate. Samples for total or total hydrolyzable phosphorus should be filtered only after digestion. Sample color that absorbs in the photometric range used for analysis will also interfere.
5. Method interference may be caused by contamination of the reagent water, reagents, glassware and/or other sample processing apparatus.

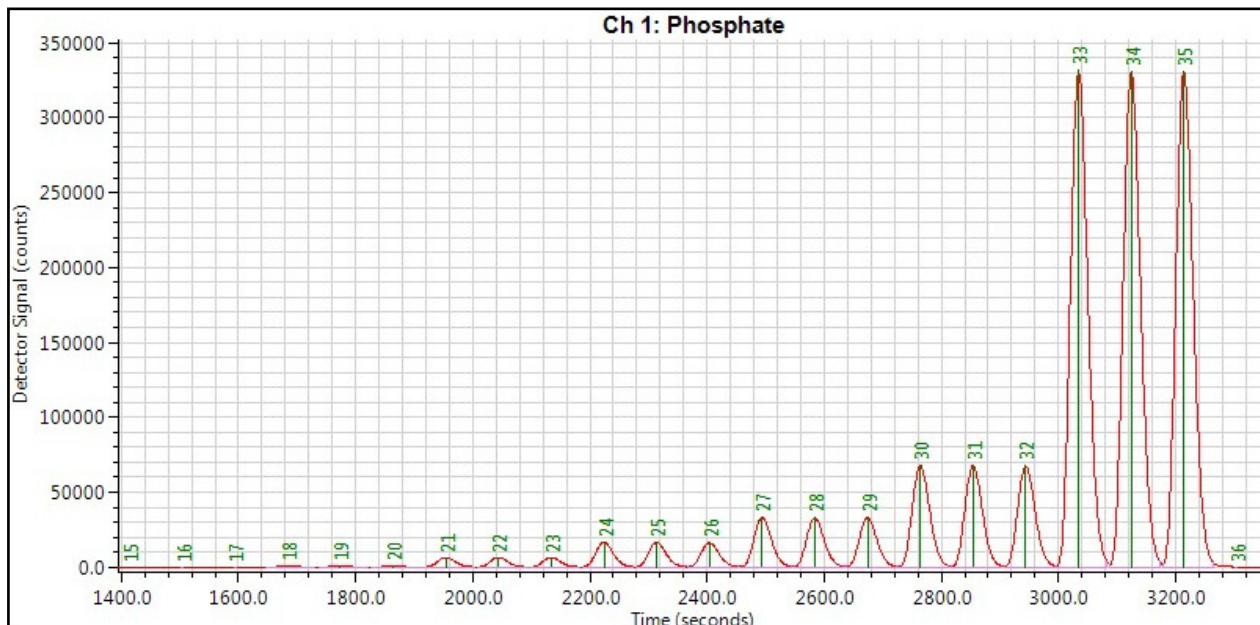


Figure 2. Low-Level Phosphorus Calibration Series

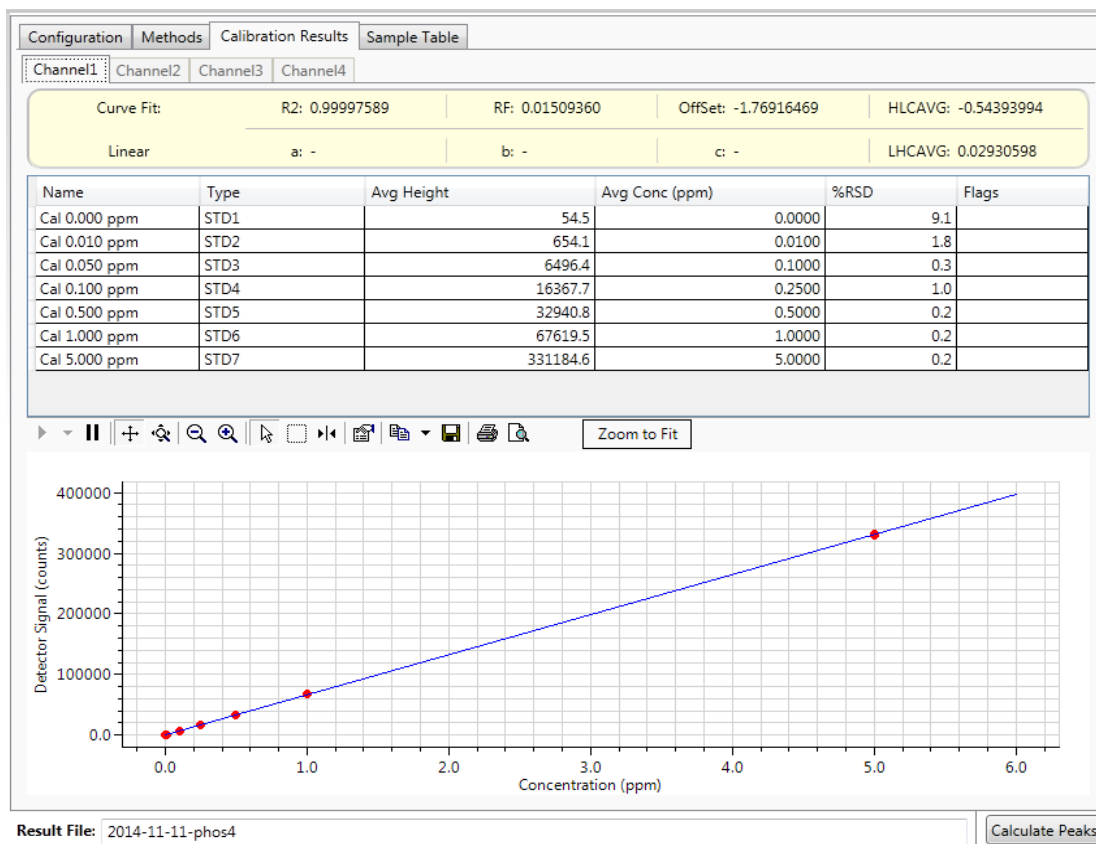


Figure 3. Calibration Curve and Statistics



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