Method Abstract

Scope

This method is used for determining total phosphorus in drinking, surface, and domestic and industrial waste according to U.S. EPA Method 365.4 and Standard Methods 4500–P G. This method can also be used for the determination of total phosphorus in potassium chloride (KCl) extracts of soils and plants.

Summary

Prior to analysis, digest samples using Kjeldahl digestion to hydrolyze phosphorus to orthophosphate. Orthophosphate reacts with molybdenum (VI) and antimony (III) in an acidic media to form an antimony-phosphomolybdate complex. Reduce this complex with ascorbic acid to form a blue color, and measure the absorbance at 880 nm.

Interferences

Ferric iron up to 50 mg/L, copper up to 10 mg/L, and silica up to 10 mg/L do not interfere. Filter turbid samples prior to analysis. Samples with background absorbance at the analytical wavelength may interfere.

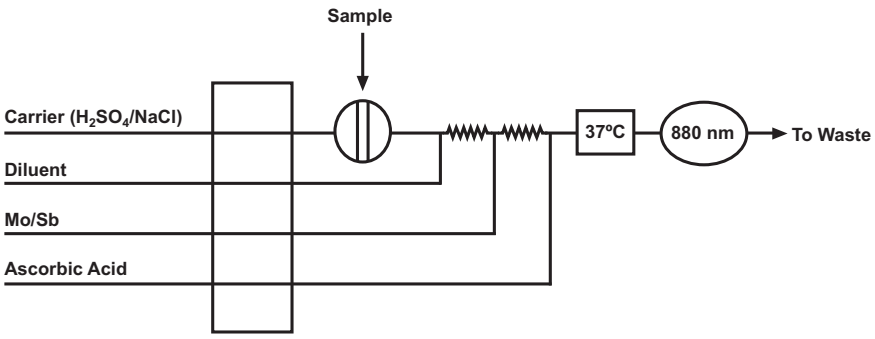
Performance Specifications

Range:	0.02–2.00 mg/L
Throughput:	60 samples/hour
Precision (at 0.1 mg/L):	<1% RSD
Precision (at 0.5 mg/L):	<1% RSD
Method Detection Limit (MDL):	0.005 mg/L
Accuracy:	104%

Chemicals

Acetone, C_3H_6O	Deionized (DI) Water, ASTM Type I or II
Ammonium Molybdate Tetrahydrate, $(NH_4)_6Mo_7O_{24}\bullet 4H_2O$	DOWFAX® 2A1, part number A000080
	Potassium Dihydrogen Phosphate, KH_2PO_4
Antimony Potassium Tartrate Hemihydrate $K(SbO)C_4H_4O_6\bullet ½H_2O$	Potassium Sulfate, K_2SO_4
	Red Mercuric Oxide, HgO
Ascorbic Acid, $C_6H_8O_6$	Sodium Chloride, NaCl
Chloroform, $CHCl_3$	Sulfuric Acid, concentrated, H_2SO_4

Basic Flow Diagram



Note

This method complies with USEPA Method 365.4 and Standard Methods 4500–PH.

Selected References

Methods for Chemical Analysis of Water and Wastewater; EPA-600/4-79-020; U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory: Cincinnati, OH, 1984; Method 365.1.

Standard Methods for the Examination of Water and Wastewater, 21st ed.; American Public Health Association: Washington, D.C., 2005.

Figures

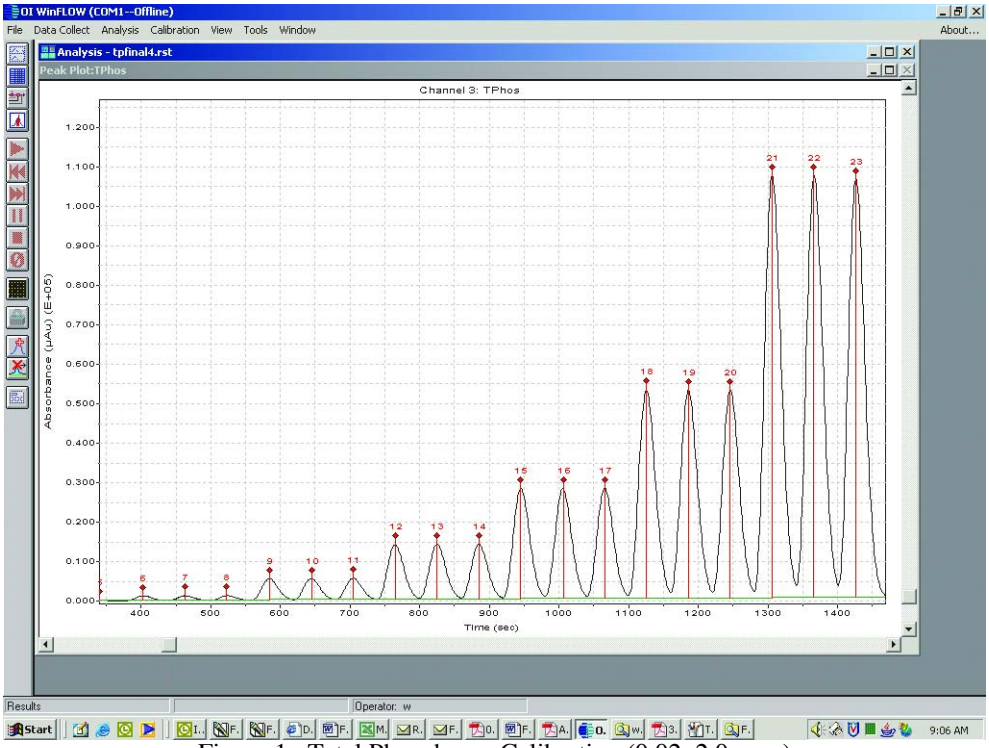


Figure 1. Total Phosphorus Calibration (0.02–2.0 ppm)

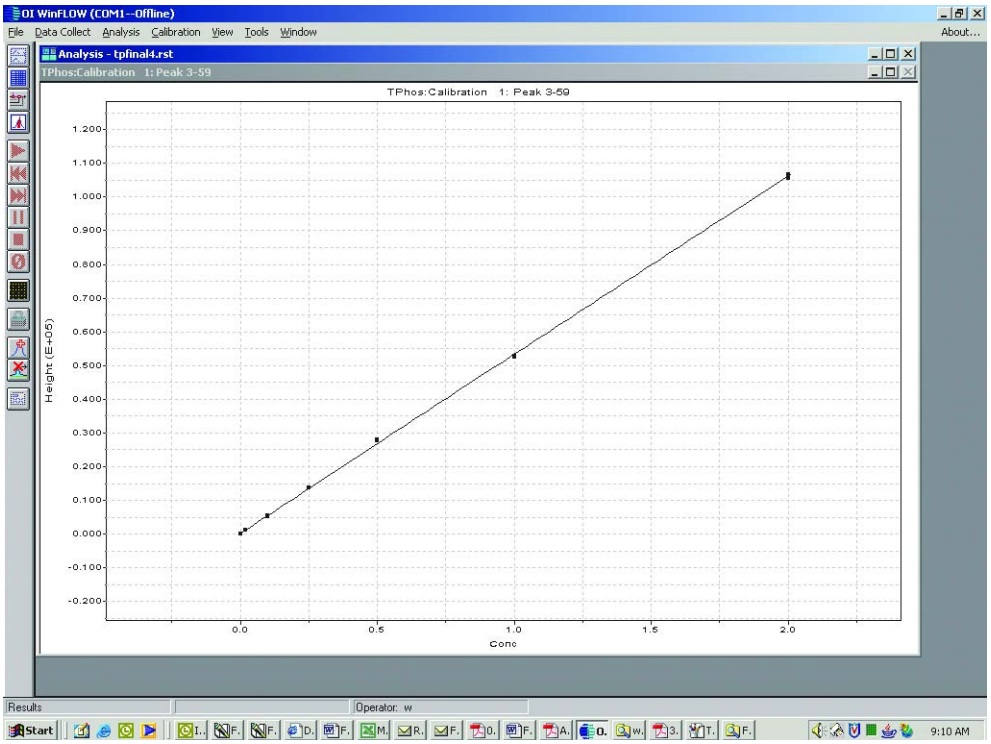


Figure 2. Total Phosphorus Calibration Curve (0.02–2.0 ppm)

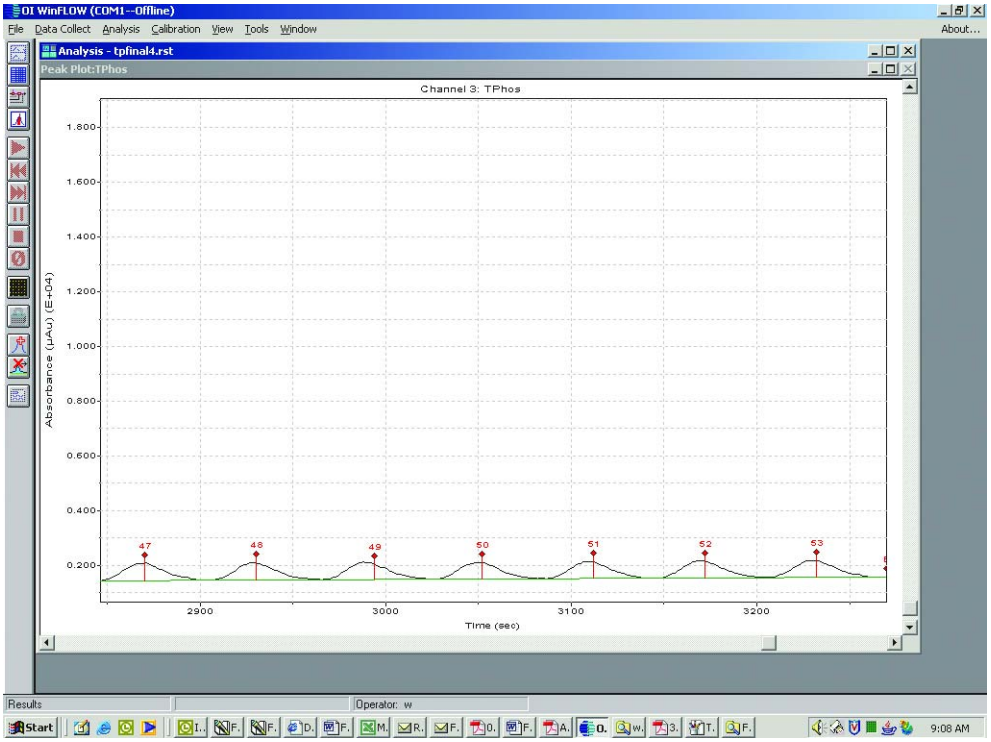


Figure 3. Total Phosphorus Method Detection Limit (at 0.05 ppm)

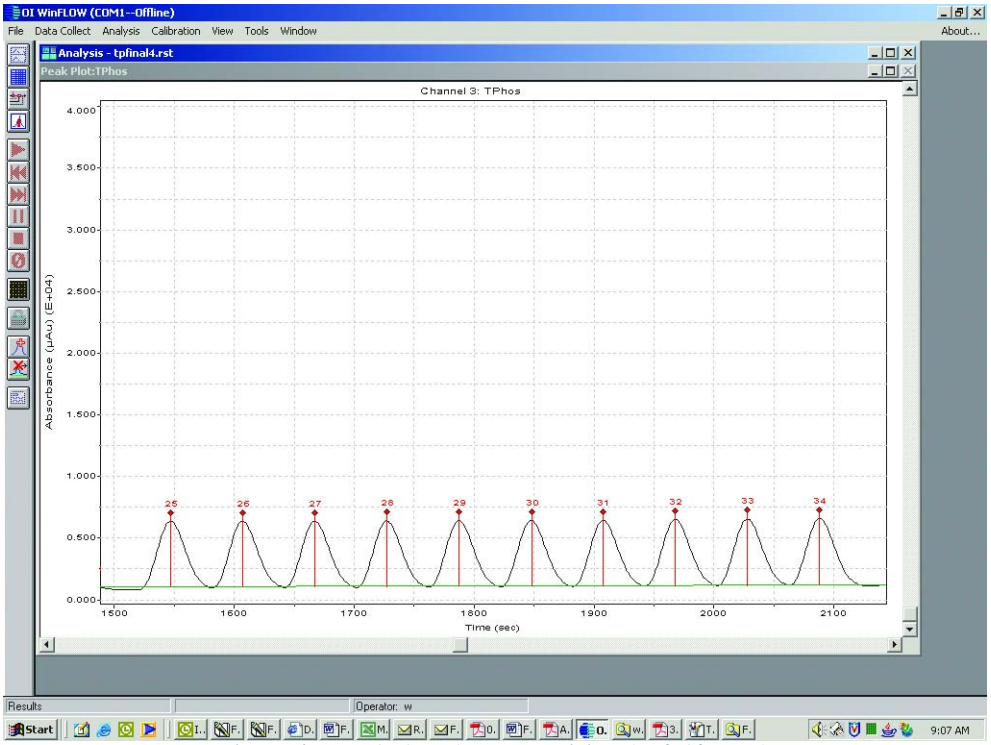


Figure 4. Total Phosphorus Precision (at 0.10 ppm)

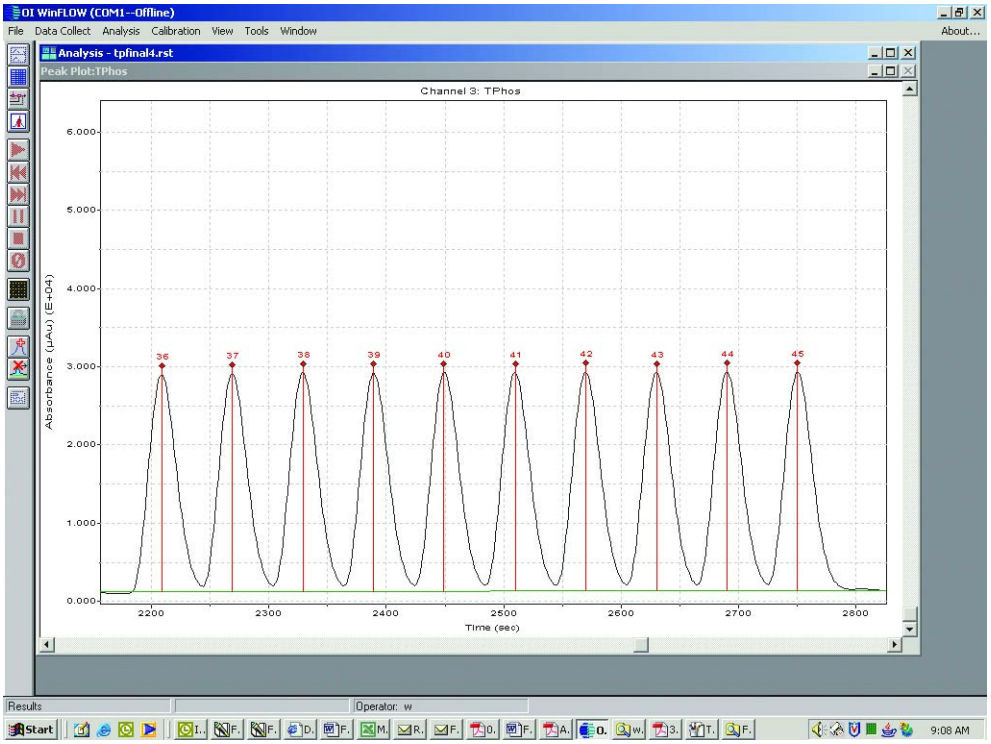


Figure 5. Total Phosphorus Precision (at 0.50 ppm)

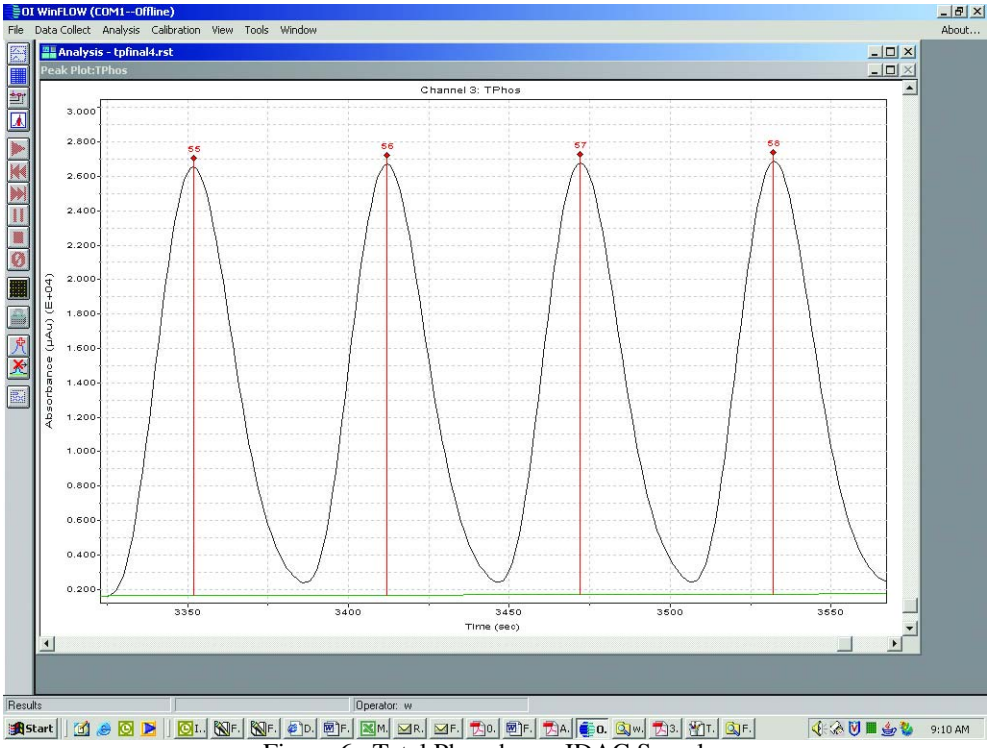
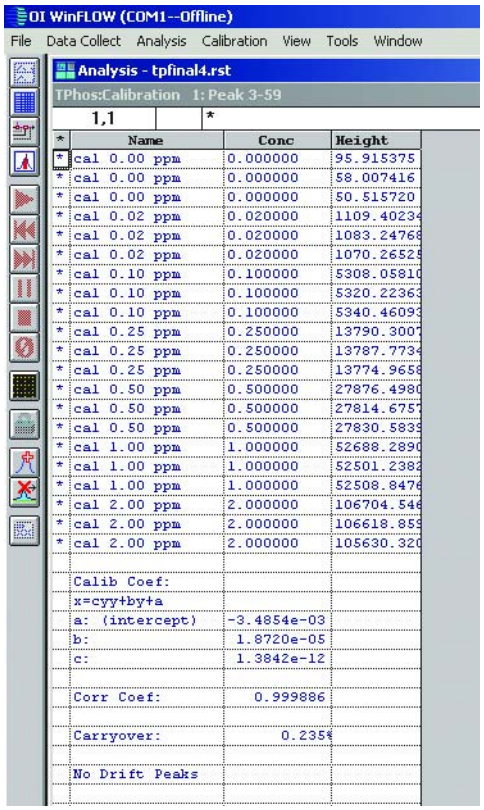


Figure 6. Total Phosphorus IDAC Samples



OI WinFLOW (COM1--Offline)

File Data Collect Analysis Calibration View Tools Window

Analysis - tpfinal4.rst

TPhos:Calibration 1: Peak 3-59

1,1 *

*	Name	Conc	Height
*	cal 0.00 ppm	0.000000	95.915375
*	cal 0.00 ppm	0.000000	58.007416
*	cal 0.00 ppm	0.000000	50.515720
*	cal 0.02 ppm	0.020000	1109.40234
*	cal 0.02 ppm	0.020000	1083.24768
*	cal 0.02 ppm	0.020000	1070.26525
*	cal 0.10 ppm	0.100000	5308.05810
*	cal 0.10 ppm	0.100000	5320.22363
*	cal 0.10 ppm	0.100000	5340.46093
*	cal 0.25 ppm	0.250000	13790.3007
*	cal 0.25 ppm	0.250000	13787.7734
*	cal 0.25 ppm	0.250000	13774.9658
*	cal 0.50 ppm	0.500000	27876.4980
*	cal 0.50 ppm	0.500000	27814.6757
*	cal 0.50 ppm	0.500000	27830.5839
*	cal 1.00 ppm	1.000000	52688.2890
*	cal 1.00 ppm	1.000000	52501.2382
*	cal 1.00 ppm	1.000000	52508.8476
*	cal 2.00 ppm	2.000000	106704.546
*	cal 2.00 ppm	2.000000	106618.859
*	cal 2.00 ppm	2.000000	105630.320
	Calib Coef:		
	x=cyy+by+a		
	a: (intercept)	-3.4854e-03	
	b:	1.8720e-05	
	c:	1.3842e-12	
	Corr Coef:	0.999886	
	Carryover:	0.235%	
	No Drift Peaks		

Figure 7. Total Phosphorus Calibration Results

Table 1. Total Kjeldahl Phosphorus Precision Calculations

	0.10 mg P/L	0.50 mg P/L	0.01 mg P/L	ERA P127-739B
Replicate 1	0.100	0.517	0.008	4.78
Replicate 2	0.100	0.517	0.008	4.77
Replicate 3	0.100	0.518	0.007	4.76
Replicate 4	0.099	0.520	0.011	4.67
Replicate 5	0.099	0.520	0.010	—
Replicate 6	0.099	0.520	0.010	—
Replicate 7	0.098	0.521	0.009	—
Replicate 8	0.099	0.522	—	—
Replicate 9	0.099	0.522	—	—
Replicate 10	0.099	0.521	—	—
Mean	0.099	0.520	0.009	4.74
Standard Deviation	0.000632	0.001874	0.001414	0.05079
% RSD	0.64	0.36	15.7	1.07
% Recovery	99	104	90	104
MDL	—	—	0.004	—
ERA sample recovery on non-digested, simple nutrient Ready-to-Use Quality Control Sample.				