Method Abstract

Scope

This method is used for the determination of total phosphorus in surface water, drinking water, and domestic and industrial wastes according to USEPA method 365.1 and Standard Methods 4500–P B/F. Additionally, this method enables total phosphorus analysis according to ISO Method 15681-2. This method can also be used to determine total phosphorus in potassium chloride (KCl) extracts of soils and plants.

Summary

Prior to analysis, samples are digested via persulfate digestion to hydrolyze phosphorus to orthophosphate. Orthophosphate reacts with molybdenum(VI) and antimony(III) in an acidic solution to form an antimony-phosphomolybdate complex. This complex is subsequently reduced with ascorbic acid to form a blue color, and the absorbance is measured at 880 nm .

The quality of the analysis is assured through reproducible calibration and testing of the Segmented Flow Analysis (SFA) system.

Interferences

Filter turbid samples prior to analysis..

The presence of less than 50 mg/L of iron(III), less than 10 mg/L of copper, or less than 10 mg/L of silicate does not interfere with this assay.

Samples with background absorbance at the analytical wavelength may interfere.

Performance Specifications

Range:	0.001–1.0 mg/L P
Throughput:	33 samples/hour
Precision (at 0.005 mg/L):	~1% RSD
Precision (at 0.05 mg/L):	<1% RSD
Precision (at 0.5 mg/L):	<0.5% RSD
Method Detection Limit (MDL):	0.0007 mg/L
Accuracy*:	106%

*ERA (Environmental Resources Associates) WasteWatR Minerals Quality Control Sample

Chemicals

Ammonium Molybdate Tetrahydrate, $(NH_4)_6Mo_7O_{24}\bullet 4H_2O$

Ammonium Persulfate, $(NH_4)_2S_2O_8$

Antimony Potassium Tartrate Hemihydrate, $K(SbO)C_4H_4O_6\bullet ½H_2O$

Ascorbic Acid, $C_6H_8O_6$

Deionized Water (ASTM Type I or II)

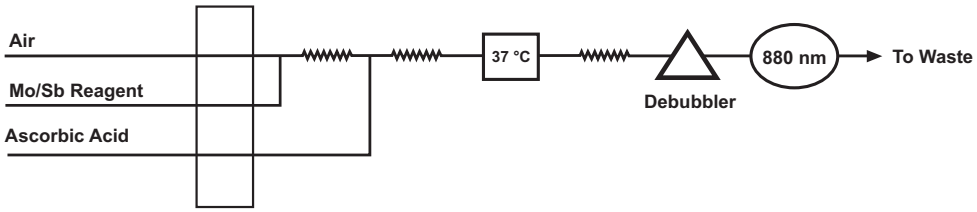
DOWFAX® 2A1 (Part #A000080)

Hydrochloric Acid, concentrated, HCl

Potassium Phosphate Monobasic, KH_2PO_4

Sulfuric Acid, concentrated, H_2SO_4

Basic Flow Diagram



Selected Reference

Phosphorus, All Forms. *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, 1993; Method 365.1

Figures

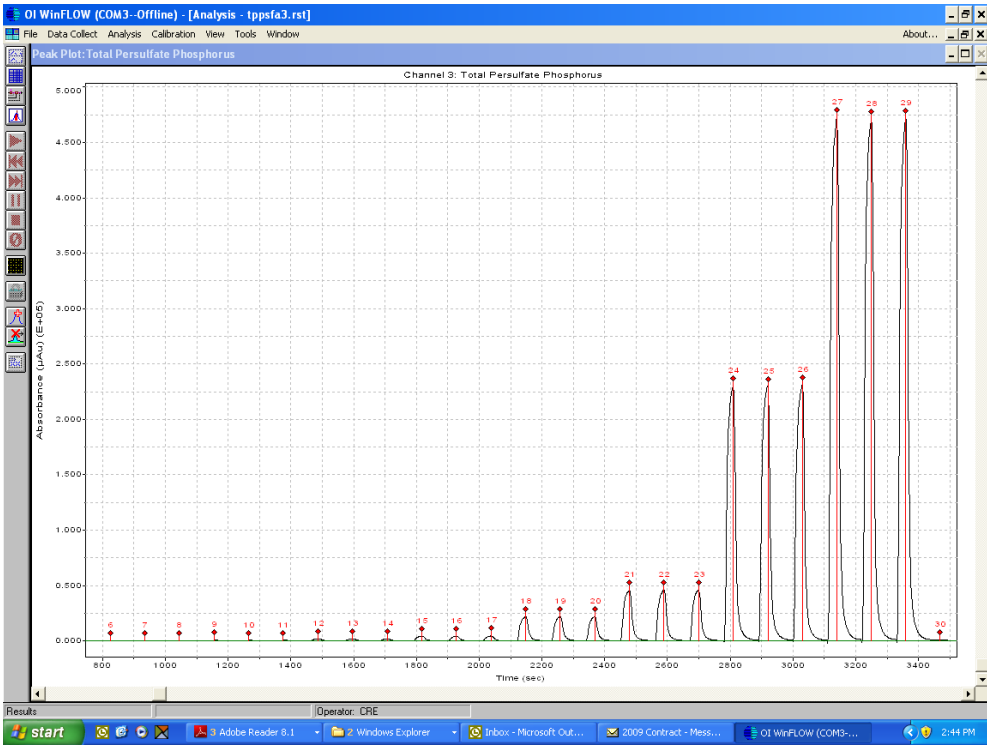


Figure 1. Total Phosphorus Calibration (0.001–0.1 mg P/L)

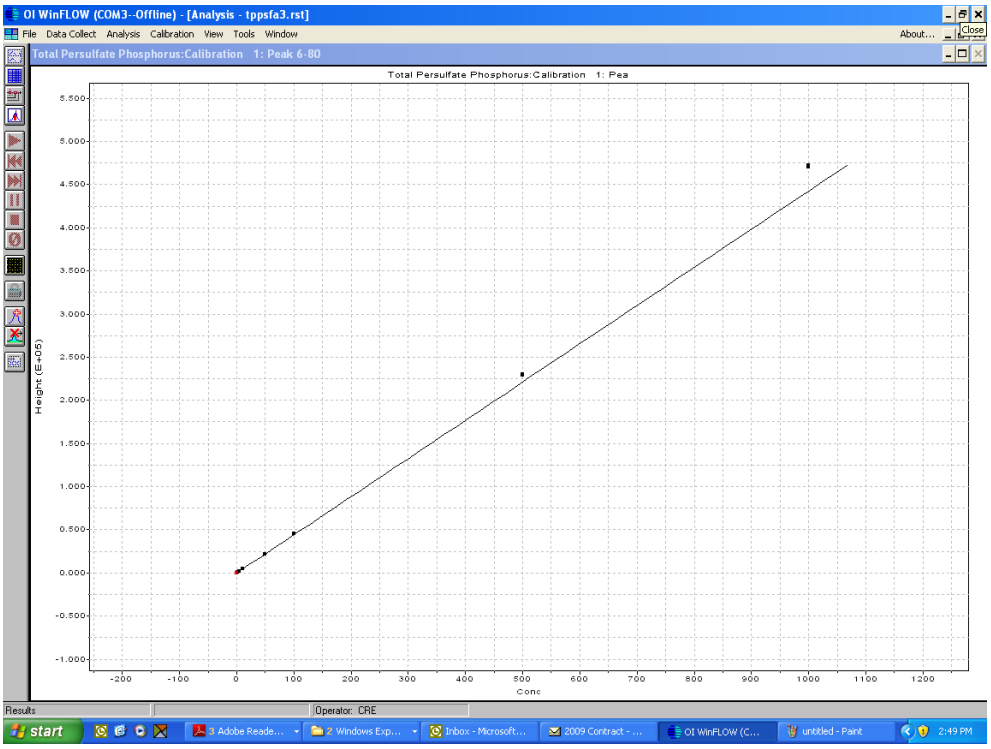


Figure 2. Total Phosphorus Calibration Curve (0.001–1.0 mg P/L)

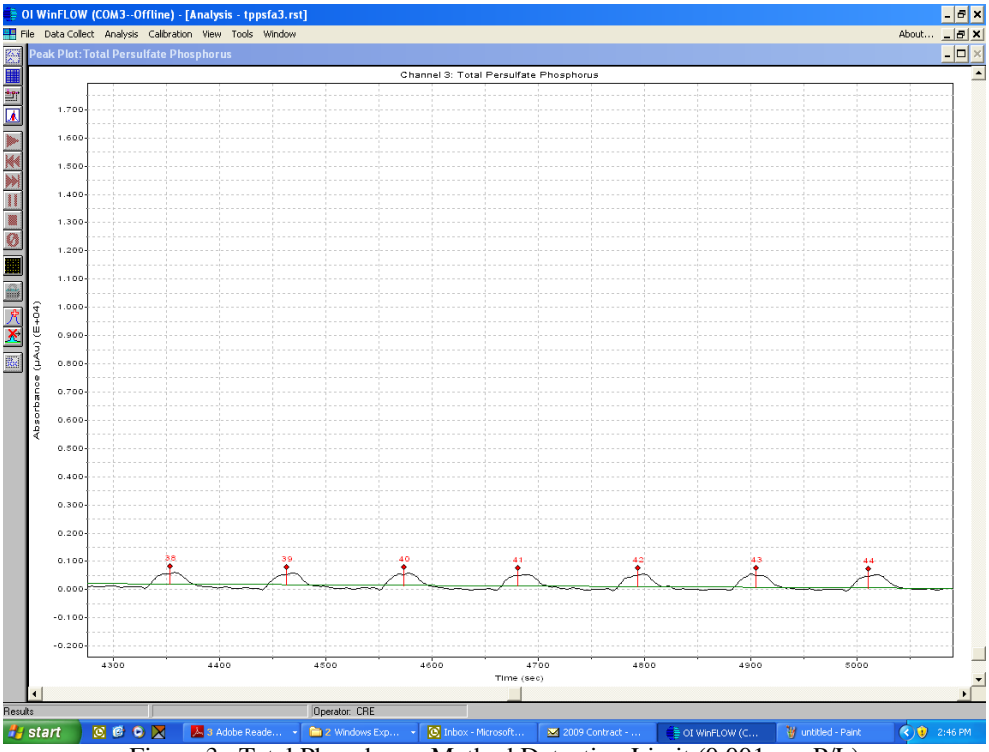


Figure 3. Total Phosphorus Method Detection Limit (0.001 mg P/L)

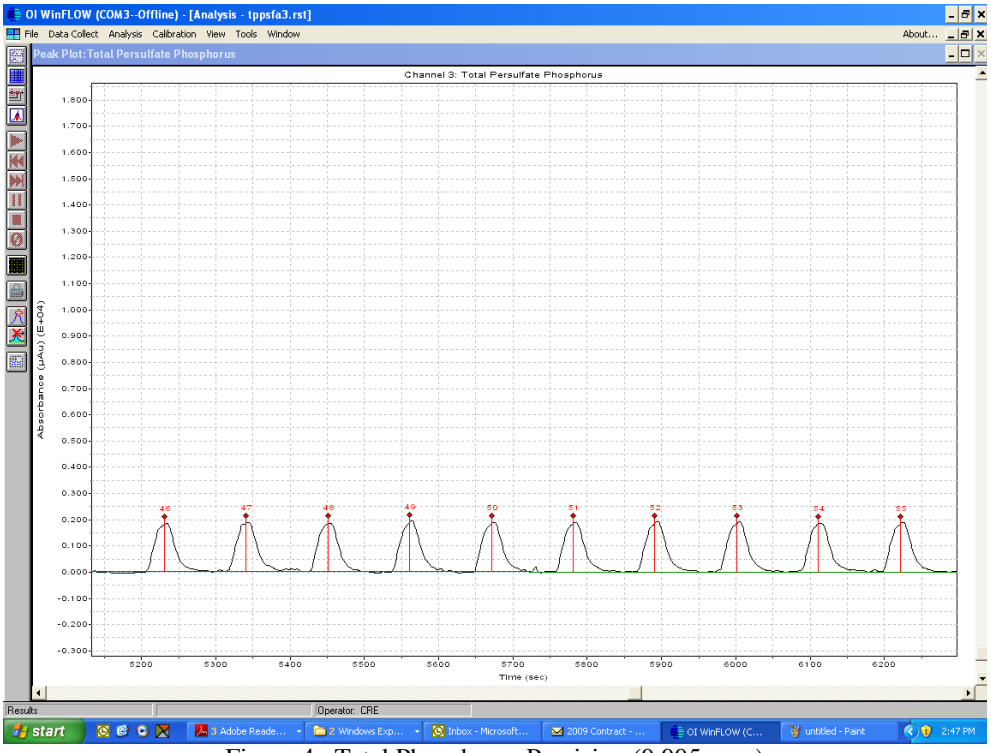


Figure 4. Total Phosphorus Precision (0.005 ppm)

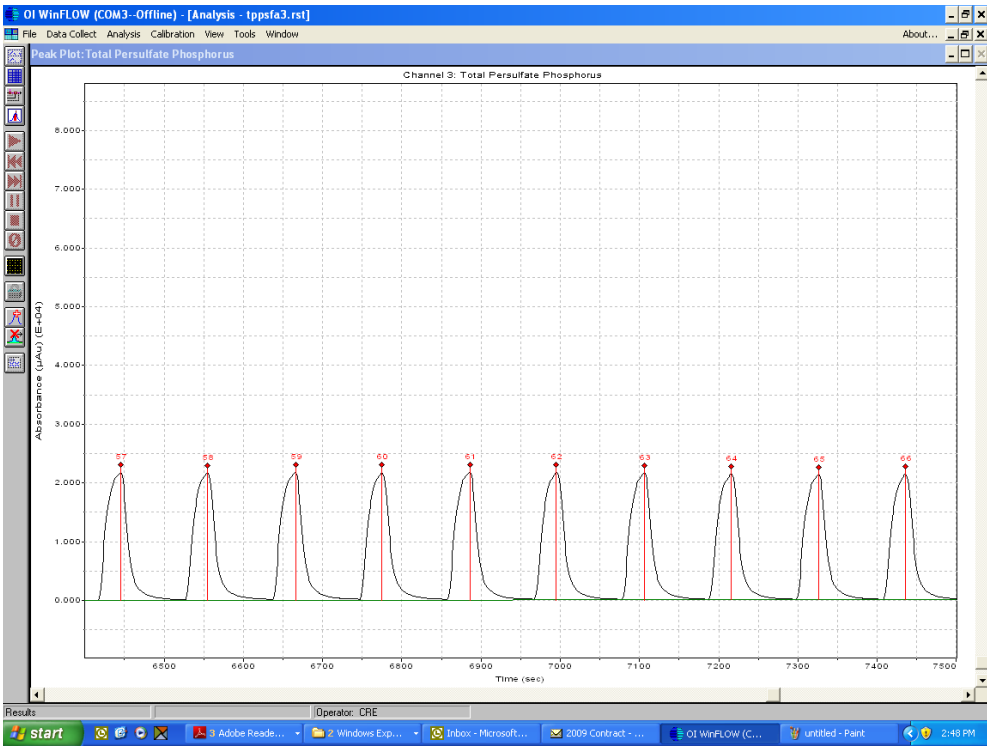


Figure 5. Total Phosphorus Precision (at 0.05 ppm)

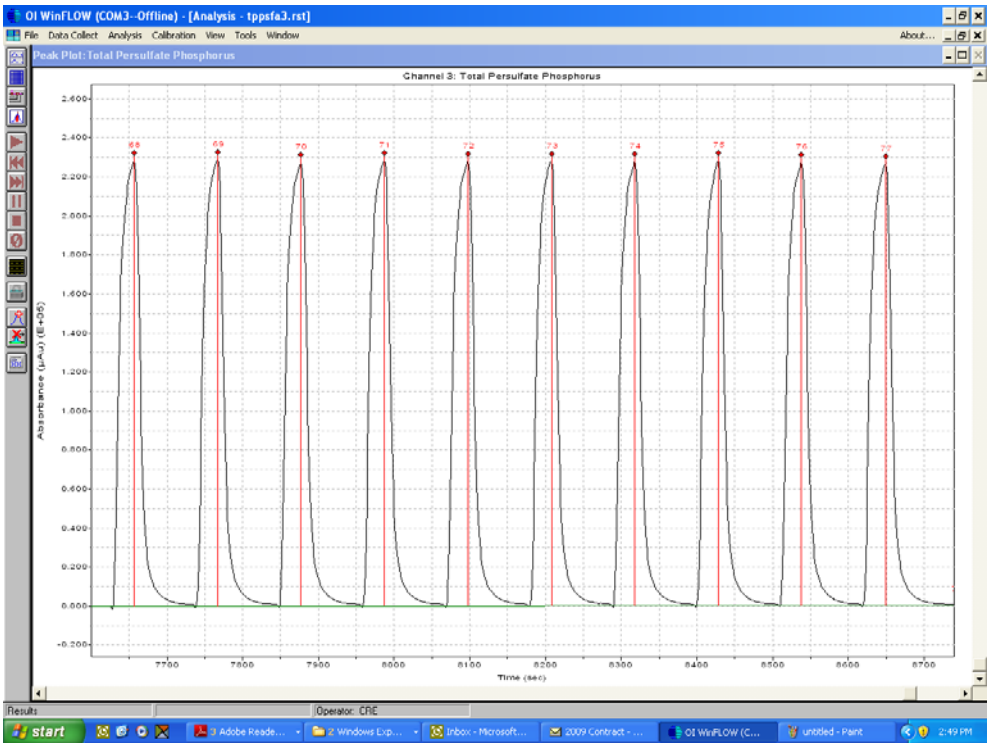


Figure 6. Total Phosphorus Precision (at 0.5 ppm)

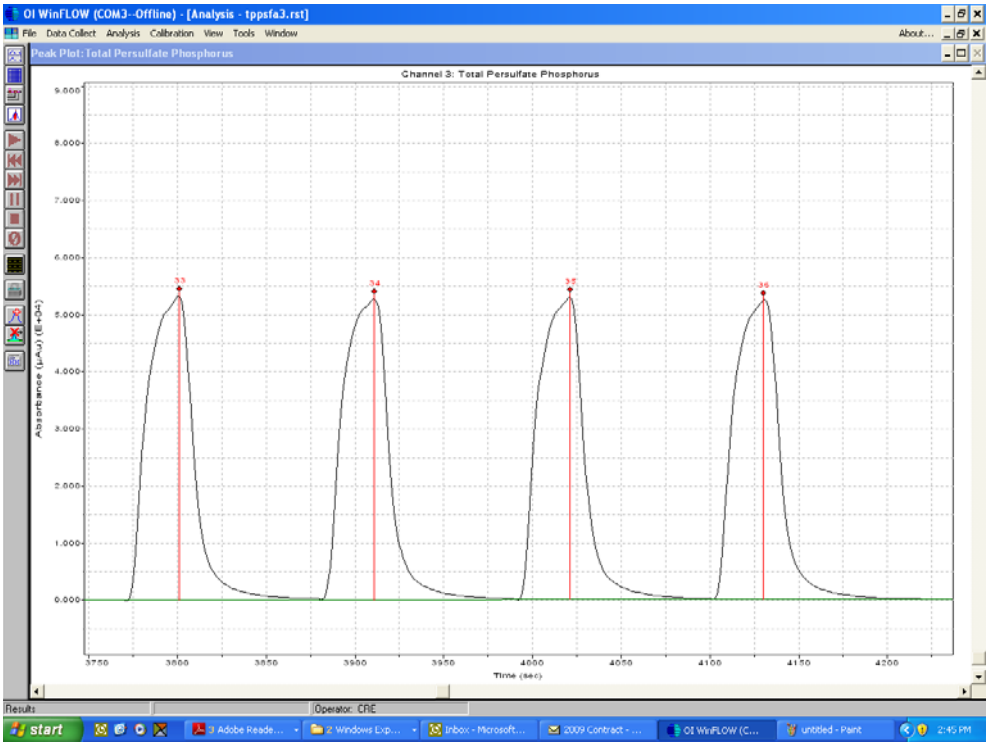
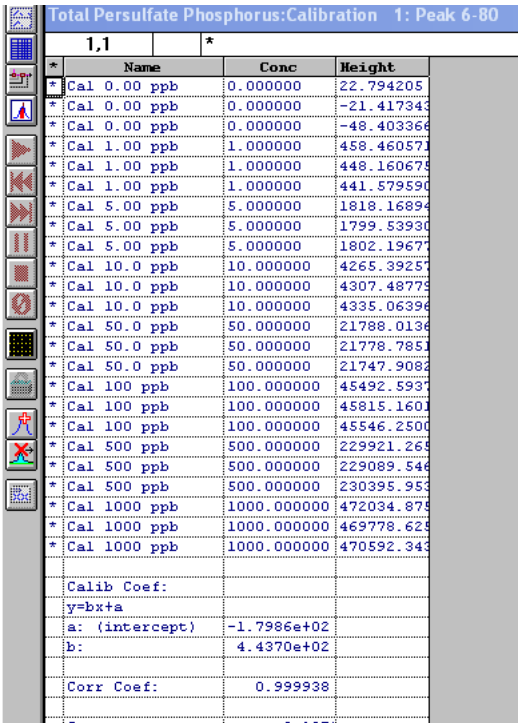


Figure 7. Total Phosphorus QC (0.11 ppm)



Total Persulfate Phosphorus:Calibration 1: Peak 6-80

1,1 *

*	Name	Conc	Height
*	Cal 0.00 ppb	0.000000	22.794205
*	Cal 0.00 ppb	0.000000	-21.417343
*	Cal 0.00 ppb	0.000000	-48.403366
*	Cal 1.00 ppb	1.000000	458.460571
*	Cal 1.00 ppb	1.000000	448.160675
*	Cal 1.00 ppb	1.000000	441.579590
*	Cal 5.00 ppb	5.000000	1818.16894
*	Cal 5.00 ppb	5.000000	1799.53930
*	Cal 5.00 ppb	5.000000	1802.19677
*	Cal 10.0 ppb	10.000000	4265.39257
*	Cal 10.0 ppb	10.000000	4307.48775
*	Cal 10.0 ppb	10.000000	4335.06396
*	Cal 50.0 ppb	50.000000	21788.0136
*	Cal 50.0 ppb	50.000000	21778.7851
*	Cal 50.0 ppb	50.000000	21747.9082
*	Cal 100 ppb	100.000000	45492.5937
*	Cal 100 ppb	100.000000	45815.1601
*	Cal 100 ppb	100.000000	45546.2500
*	Cal 500 ppb	500.000000	229921.265
*	Cal 500 ppb	500.000000	229089.546
*	Cal 500 ppb	500.000000	230395.953
*	Cal 1000 ppb	1000.000000	472034.875
*	Cal 1000 ppb	1000.000000	469778.625
*	Cal 1000 ppb	1000.000000	470592.343
	Calib Coef:		
	y=bx+a		
	a: (intercept)	-1.7986e+02	
	b:	4.4370e+02	
	Corr Coef:	0.999938	

Figure 8. Total Phosphorus Calibration Results (0.001–1.0 mg P/L)

Table 1. Total Phosphorus Method Data (0.001–1.0 mg/L)

Parameter	Calibrant 0.001 mg/L	Calibrant 0.005 mg/L	Calibrant 0.05 mg/L	Calibrant 0.5 mg/L	ERA QC Standard 0.11 mg/L
Rep 1	0.0012	0.0045	0.0493	0.5148	0.1185
Rep 2	0.0012	0.0046	0.0492	0.5149	0.1164
Rep 3	0.0013	0.0045	0.0493	0.5123	0.1178
Rep 4	0.0013	0.0046	0.0493	0.5139	0.1153
Rep 5	0.0013	0.0045	0.0493	0.5133	—
Rep 6	0.0014	0.0046	0.0494	0.5131	—
Rep 7	0.0014	0.0046	0.0491	0.5127	—
Rep 8	—	0.0045	0.0488	0.5135	—
Rep 9	—	0.0045	0.0483	0.5114	—
Rep 10	—	0.0045	0.0485	0.5095	—
Average	0.0013004	0.004547	0.0490465	0.5129378	0.1169992
Standard Deviation	.0000509311	.000048	0.0003872	0.0016146	0.0013911
% RSD	3.92	1.06	0.79	0.31	1.19
MDL	0.000159924	—	—	—	—
Accuracy	—	—	—	—	105.59

(800) 653-1711
(979) 690-1711
www.oico.com
oimail@oico.com