

Method Abstract

Scope This method is used to determine orthophosphate in estuarine and coastal waters (seawater) according to USEPA Method 365.5 and Standard Methods 4500– P F. This method can also be used to analyze low-turbidity limnological and fresh water samples. Additionally, this method enables orthophosphate analysis according to ISO Method 15681-2.

Summary Orthophosphate reacts with molybdenum(VI) and antimony(III) in an acidic solution to form an antimony-phosphomolybdate complex. Ascorbic acid subsequently reduces this complex to form a blue color, and the absorbance is measured at 880 nm.

Interferences Filter turbid samples prior to analysis. The presence of more than 40 mg/L of iron(III), more than 10 mg/L of copper, or more than 10 mg/L of silica may interfere. Samples with background absorbance at the analytical wavelength may interfere. Residual phosphate in the flow system components and from continuous analysis may interfere. Wash the system with dilute HCl to avoid interferences.

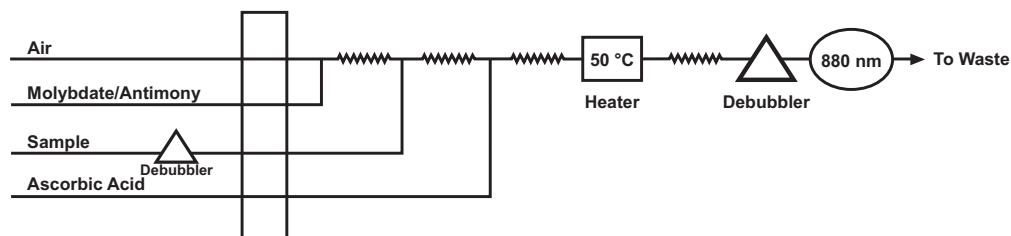
Performance Specifications

Range	1.0–400 $\mu\text{g/L}$ (0.03–13 $\mu\text{moles/L}$)
Throughput	30 samples/hour
Precision at:	
4 $\mu\text{g/L}$	<2% RSD
40 $\mu\text{g/L}$	<1% RSD
Method Detection Limit (MDL)	0.25- $\mu\text{g/L}$ (0.008 $\mu\text{moles/L}$)

Chemicals

Ammonium Molybdate Tetrahydrate, (NH_4) ₆ Mo ₇ O ₂₄ •4H ₂ O	Magnesium Sulfate Heptahydrate, MgSO ₄ •7H ₂ O
Sodium Hydroxide, NaOH	Potassium Phosphate Monobasic, KH ₂ PO ₄
Ascorbic Acid, C ₆ H ₈ O ₆	Sodium Bicarbonate, NaHCO ₃
Deionized Water (ASTM Type I or II)	Sodium Chloride, NaCl
DOWFAX® 2A1 (PN A000080)	Antimony Potassium Tartrate Hemihydrate, K(SbO)C ₄ H ₄ O ₆ •Q/wH ₂ O
Hydrochloric Acid, concentrated, HCl	Sulfuric Acid, concentrated, H ₂ SO ₄

Basic Flow Diagram



Selected References

Determination of Orthophosphate in Estuarine and Coastal Waters by Automated Colorimetric Analysis. *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, 1997; Method 365.5.

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

Water Quality–Determination of Orthophosphate and Total Phosphorus Contents by Flow Analysis (FIA and CFA)–Part 1: Method by Continuous Flow Analysis (CFA). International Standard; ISO 15621–2:2003 (E); 1st. ed: Geneva, Switzerland, 2003.

Figures

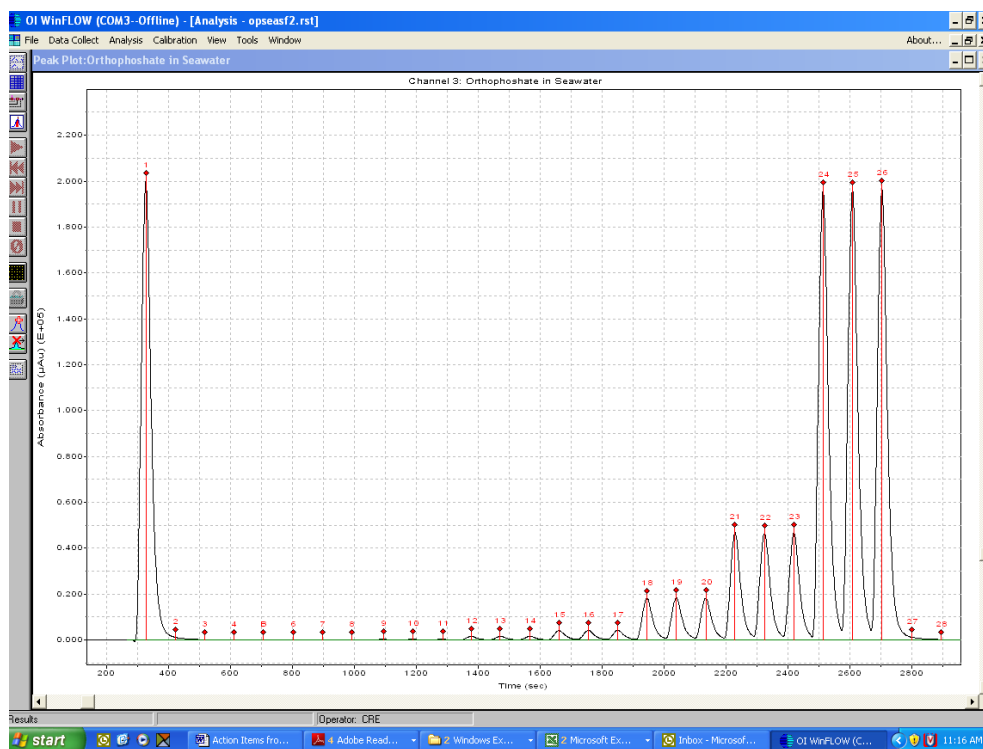


Figure 1. Calibration (1.0–400 $\mu\text{g/L}$)

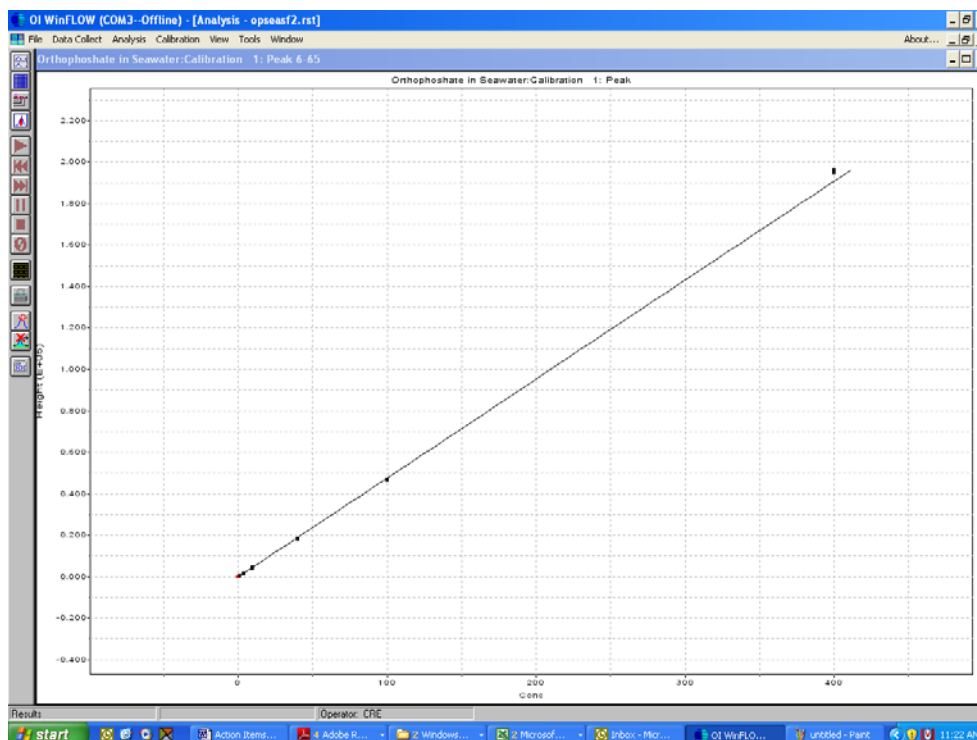


Figure 2. Calibration Curve (1.0–400 µg/L)

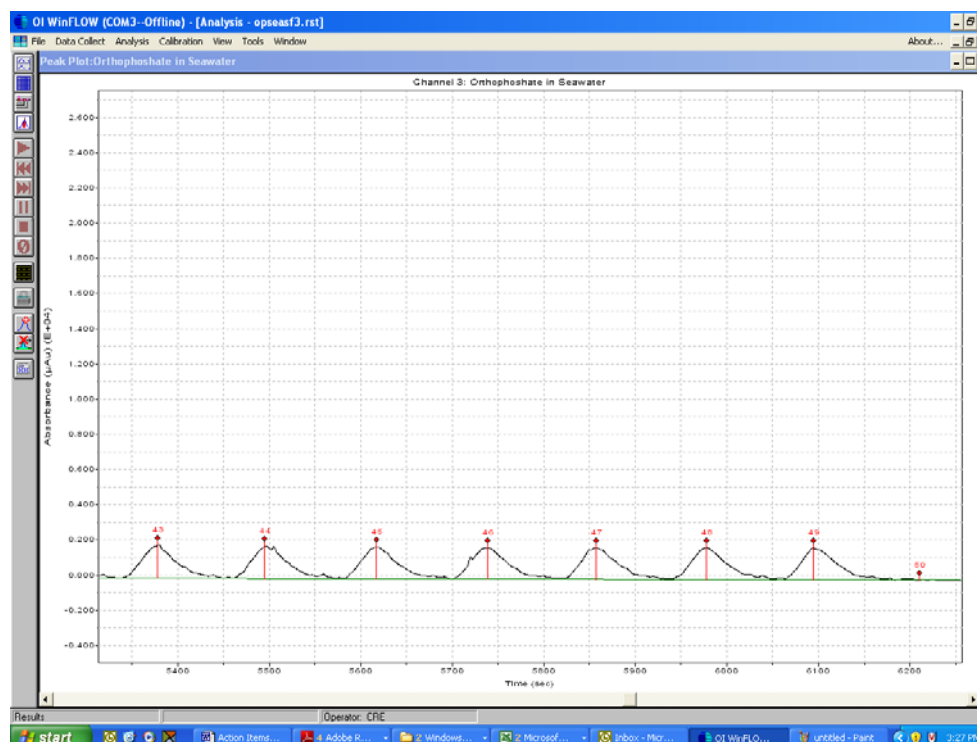


Figure 3. Method Detection Limit (at 4.0 ppb)

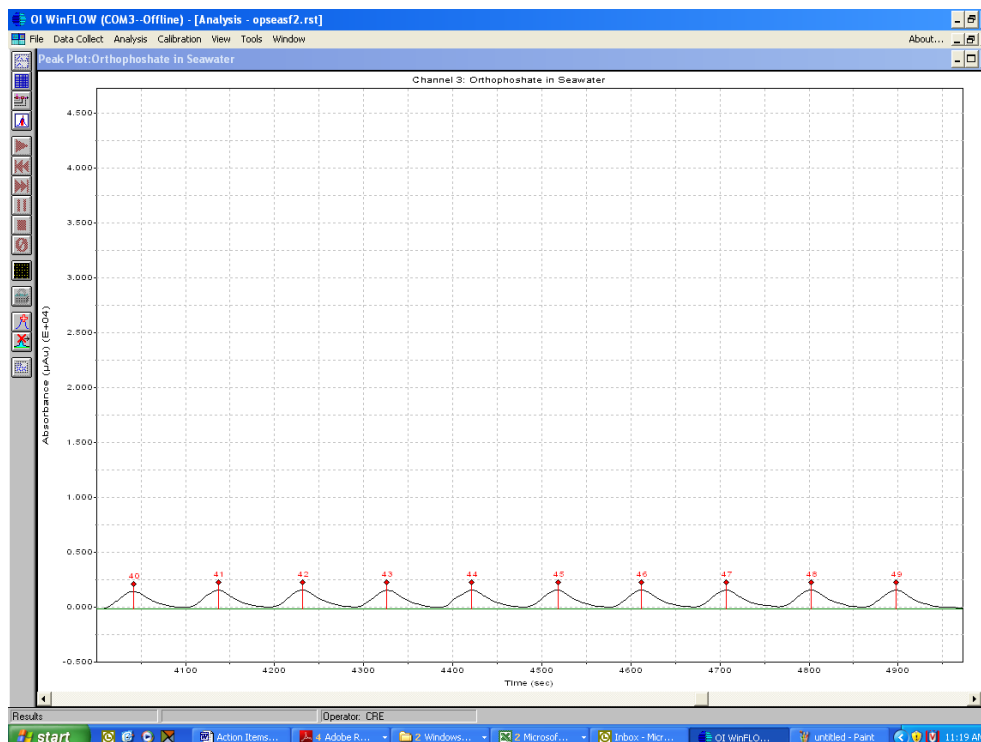


Figure 4. Precision at 4.0 µg/L (<2% RSD)

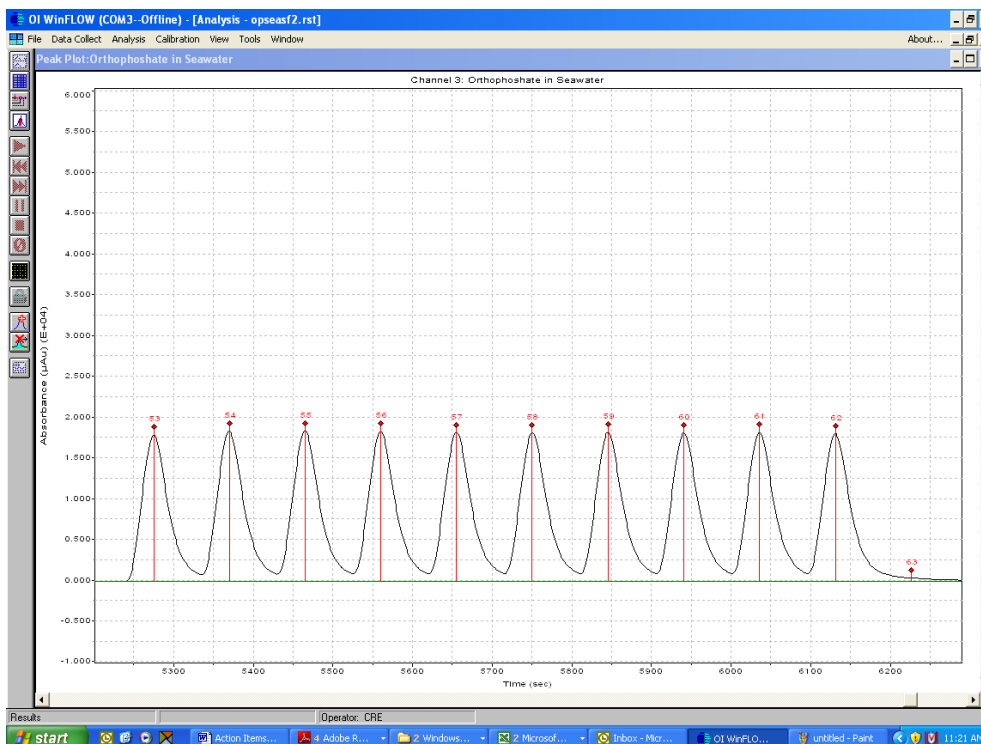


Figure 5. Precision at 40 µg/L (<1% RSD)

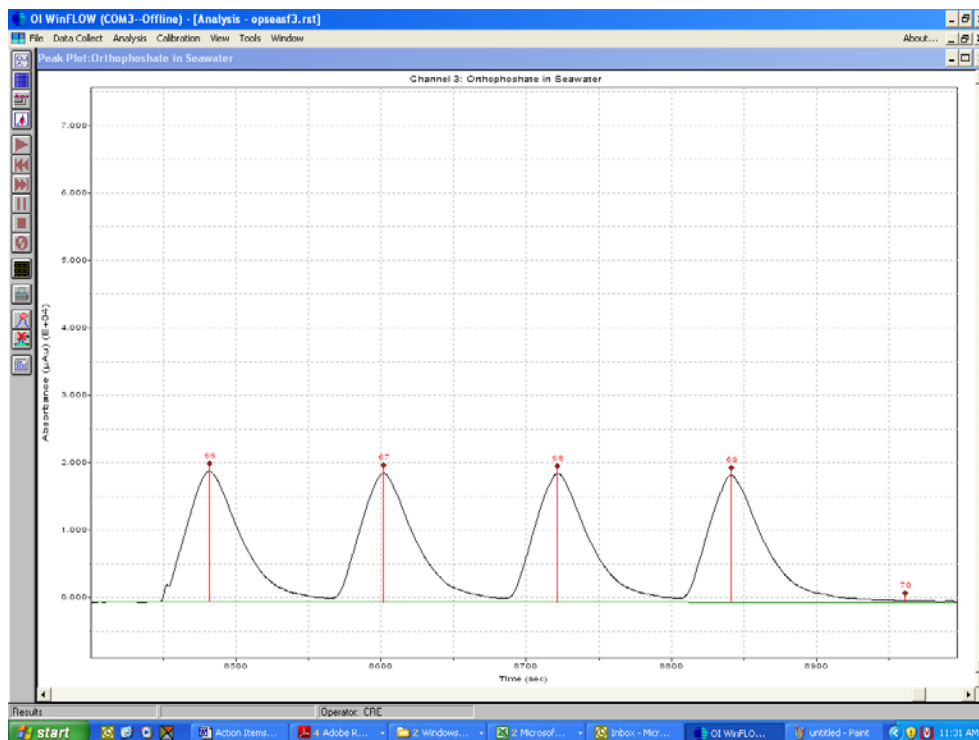


Figure 6. ERA QC (40 ppb)

OI WinFLOW (COM3-Offline) - [Analysis - opseasf3.rst]

Orthophosphate in Seawater: Calibration 1: Peak

Name	Conc	Height
* Cal 0.00 ppb	0.000000	-41.482294
* Cal 0.00 ppb	0.000000	-56.346401
* Cal 0.00 ppb	0.000000	-70.820602
* Cal 1.00 ppb	1.000000	374.036346
* Cal 1.00 ppb	1.000000	403.305542
* Cal 1.00 ppb	1.000000	412.484378
* Cal 4.00 ppb	4.000000	1589.75781
* Cal 4.00 ppb	4.000000	1622.56433
* Cal 4.00 ppb	4.000000	1624.00817
* Cal 10.0 ppb	10.000000	4108.68508
* Cal 10.0 ppb	10.000000	4222.60400
* Cal 10.0 ppb	10.000000	4199.63720
* Cal 40.0 ppb	40.000000	18211.7558
* Cal 40.0 ppb	40.000000	18350.0703
* Cal 40.0 ppb	40.000000	18156.6074
* Cal 100 ppb	100.000000	46870.0628
* Cal 100 ppb	100.000000	46243.8518
* Cal 100 ppb	100.000000	46610.6878
* Cal 400 ppb	400.000000	195959.421
* Cal 400 ppb	400.000000	194912.018
* Cal 400 ppb	400.000000	195533.687

Calib Coef:	
y=bx+a	
a: (intercept)	-1.4778e+02
b:	4.7755e+02
Corr Coef:	0.999926
Carryover:	0.5946
No Drift Peaks	

Figure 7. Calibration Results (1.0–400 µg/L)

Method Abstract

Table 1. Orthophosphate in Seawater Validation Results Table

Parameter	Calibrant 4.0 µg/L	Calibrant 4.0 µg/L	Calibrant 40 µg/L	ERA QC Standard 40 µg/L
Rep 1	4.1750	3.6898	38.0171	43.3424
Rep 2	4.0383	3.9047	38.6523	42.4701
Rep 3	3.9623	3.9027	38.7161	42.4450
Rep 4	3.9457	3.8743	38.5921	41.9483
Rep 5	3.9902	3.9280	38.3155	—
Rep 6	4.0411	3.9104	38.2750	—
Rep 7	3.9953	3.9304	38.3525	—
Rep 8	—	3.9050	38.2286	—
Rep 9	—	3.8935	38.4257	—
Rep 10	—	3.9085	38.0134	—
Average	4.0211296	3.8847261	38.358836	42.551442
Standard Deviation	0.0765285	0.0703237	0.2437438	0.5794373
% RSD	1.9031585	1.8102603	0.6354307	1.3617337
MDL	0.2402994	—	—	—
% Accuracy	—	—	—	105.83