



Parameter and Sample Type

Orthophosphate in Surface and Wastewater by Colorimetry

Introduction

New Thermo Scientific Orion* AQ3700 AQUAfast* Colorimeter offers preprogrammed methods for testing nutrients. This application note covers testing orthophosphate in surface and wastewater using the Orion AC2095 Tablets. Ortho-Phosphate ions react with two reagent tablets to form an intense blue color, which is proportional to the phosphorous concentration. The Thermo Orion AC2095 Method is accepted by U.S. EPA for drinking water and wastewater compliance monitoring.

References

- Orion AC2095 Orthophosphate Test Kit Method, rev 6, June 4, 2003; EPA accepted on March 26, 2004 (drinking water) and June 7, 2004 (wastewater).
 www.thermoscientific.com/water
- 2. AQ3700 Colorimeter User Guide, Method #320. www.thermoscientific.com/water
- Method 4500-P E, Ascorbic Acid Method. Standard Methods for the Examination of Water and Wastewater. APHA, AWWA, & WEF, Washington, D.C. <u>www.standardmethods.org</u>

Recommended Equipment

Orion AQ3700 Colorimeter; Orion AC2095 Phosphorous LR tablets (Tablets 1 and 2); Orion AC2V24 24mm vials; Glassware rinsed with warm Hydrochloric Acid / Water (1:1)

Required Solutions

Stock orthophosphate standard, 250 mg PO₄/L; intermediate orthophosphate standard, 25 mg PO₄/L; calibration verification orthophosphate standard, 2.0 mg PO₄/L; deionized (DI) water, free of orthophosphate.

Solutions Preparation

- 1. Prepare 250 mg/L orthophosphate stock by dissolving 359.25mg of dried reagent grade potassium phosphate with DI water in a 1000 mL volumetric.
- 2. Prepare 25 mg/L orthophosphate intermediate standard by transferring 10 mL of the 250 mg/L stock standard to a 100 mL volumetric flask and diluting to the mark with DI water.
- 3. Prepare 2.0 mg/L orthophosphate calibration verification standard by transferring 8 mL of the 25 mg/L intermediate standard to a 100 mL volumetric flask and diluting to the mark with DI water.

Meter Setup

Turn on the meter, press the shift key and number pads 3-2-0 to select the "Phosphate, ortho LR with Tablet" method. Fill a clean vial to the line with DI water. Cap and wipe w/ lint-free wiper until dry and free of smears. Handling the vial by the

cap, insert the vial into the meter aligning the mark on the vial with the mark on the meter. Zero the meter on DI water.

Sample Storage and Preparation

Store samples in sealed air tight containers refrigerated (<6°C) Test samples within 48 hours.

Sample Vial Storage and Cleaning

Clean and store vials per instructions in the user guide. Do not allow reacted samples to remain in the vials overnight.

Testing Procedure

Fill a cleaned vial to the line with 10 mL of sample, standard, or blank water for testing. Use a pipet for best accuracy. (Note: If the sample is colored or turbid, zero the meter with sample to compensate for that. Before testing another sample, re-zero the meter with DI or the new sample). Tear open the foil pouch containing the AC2095 LR 1 Tablet and add the tablet to the vial without touching the tablet. Crush the tablet with the tamping rod. Tear open the foil pouch containing the AC2095 LR 2 Tablet and add that tablet to the vial. Crush it with the tamping rod. Cap the vial and shake for 15 seconds. It is not necessary for all of the crushed tablets to be dissolved. Wipe the vial until clean and dry. Insert into the meter aligning marks. Press the test key. After 10 minutes, the meter will automatically read the sample and display the phosphate reading.

Meter Performance Check/Calibration Verification

Check meter accuracy by reading a phosphate standard at 2 mg/L and a reagent blank. The reagent blank should read <0.05 mg/L ("underrange" will be displayed) and the 2 mg/L standard should read within +/- 10%, e.g., 1.80 - 2.20 mg/L.

If the meter performance check fails, take corrective actions as follows: 1) wipe the vial carefully with a lint-free wipe to remove all fingerprints and liquid drips from the exterior, handle the vial by the cap only, and remeasure; 2) if the tablets are not white and do not hold their form, use a fresh tablet or another lot of tablets. 3) using a clean vial, rezero the meter with DI water. Using the same vial, fill with 2.0 mg/L standard or DI, add reagent tablets, and retest; 4) prepare the standards again and retest. If these corrective actions fail, perform manual calibration according meter user guide.

Calibration

The meter is shipped precalibrated. The meter performance is very stable and does not require frequent calibration. If a standard reading is not within criteria, see corrective actions above.

Quality Control (QC)

Recommended QC procedures include: calibration verification, reagent blank analysis, QC samples, sample duplicates & spikes.





Testing Results:

Calibration Range Verification						
	Results (mg/L PO ₄)					
Sample	Criteria	Meter 1	Performance	Meter 2	Performance	
Blank*		< 0.05		< 0.05		
0.06 mg/L PO ₄	90-110% R	0.06	100.0% R	0.06	100.0% R	
0.18 mg/L PO ₄	90-110% R	0.18	100.0% R	0.18	100.0% R	
2.0 mg/L PO ₄	90-110% R	2.04	102.0% R	2.06	103.0% R	
3.9 mg/L PO ₄	90-110% R	3.93	100.8% R	3.96	101.5% R	

Note: These orthophosphate standards were analyzed to confirm the accuracy of the meter across the range of concentrations it is capable of testing. The blank sample reading was "underrange" which indicated a concentration less than the lower limit of 0.05 mg/L orthophosphate. *The calculate MDL was 0.04 mg/L PO_4 based on testing seven replicates of 0.18 mg/L PO_4

Water Samples

	Results (mg/L PO ₄)					
Sample	Criteria	Meter 1	Performance	Meter 2	Performance	
Pond Water - 1		0.07		0.08		
Pond Water - 2		0.07		0.08		
Pond Water - 3		0.08		0.07		
Pond Water - 4		0.07		0.07		
Pond Water - 5		0.08		0.08		
Pond Water Mean		0.07		0.08		
Pond Water Std Dev			0.01 SD		0.01 SD	
Pond Water % CV			7.4% RSD		7.2% RSD	
Pond 1 mg/L PO ₄ Spike	89-111% R	1.11	103.6% R	1.12	104.4% R	
Pond 1 mg/L PO ₄ Spike (Dupl)	89-111% R	1.10	102.6% R	1.12	104.4% R	
Pond Water Spike RPD	11%		0.9% RPD		0.0% RPD	
Effluent Water - 1		5.60		5.64		
Effluent Water - 2		5.58		5.62		
Effluent Water - 3		5.58		5.62		
Effluent Water - 4		5.60		5.66		
Effluent Water - 5		5.60		5.62		
Effluent Water Mean		5.59		5.63		
Effluent Water Std Dev			0.01 SD		0.02 SD	
Effluent Water % CV			0.2% RSD		0.3% RSD	
Effluent 1 mg/L PO ₄ Spike	87-115% R	6.56	96.8% R	6.58	94.8% R	
Effluent 1 mg/L PO ₄ Spike (Dupl)	87-115% R	6.56	96.8% R	6.58	94.8% R	
Effluent Water Spike RPD	14%		0.0% RPD		0.0% RPD	

Note: Effluent was diluted 1:1 with deionized water to be within range of the meter. Results were calculated by multiplying meter readout by 2 to account for the dilution factor.



Note: The performance evaluation sample (ERA Cat. No. 4023) has a certified orthophosphate concentration value of 2.99 mg/L (as diluted) and acceptance limits of 2.64 - 3.33 mg/L. The results of four replicates were very reproducible (STDEV = 0.02mg/L) and accurate (well within the acceptable limits).