

EPA New England Region 1

Standard Operating Procedure for  
Total Phosphate and Orthophosphate Analysis  
By Lachat Analyzer

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1. **Scope and Application**

The SOP covers the determination of dissolved reactive phosphorus (mostly a measure of orthophosphate) and total phosphate in drinking, surface, waste and saline waters as well as soils.

2. **Summary**

Ammonium molybdate and antimony potassium tartrate react at acidic pH with Orthophosphate ion ( $\text{PO}_4^{3-}$ ) to form an antimony-phospho-molybdate complex. Ascorbic acid reduces this complex to a complex with an intensely blue color. The intensity of the color is proportional to the concentration of orthophosphate in the sample. Orthophosphate is the only form of phosphate that will form the blue complex. All organic forms of phosphate present in the sample may be converted to Orthophosphate form by persulfate digestion.

3. **Health and Safety Warnings**

Sulfuric acid used in this method can cause severe burns and should be handled by a analyst trained to work with this chemical. Gloves and protective clothing must be worn and chemicals should be kept under a fume hood. The reagents used are toxic and similar precautions should be taken when handling them. Safety information is available in the form of MSDS sheets and can be obtained in the library.

4. **Personnel qualifications**

The analyst should have at least 4 year degree in physical science. The analyst must have a satisfactory IDC/MDL in place before analyzing samples. All personnel shall be responsible for complying with all QA/QC requirements that pertain to their organizational/technical function.

5. **Interferences**

- 5.1. Silica forms a pale blue complex that absorbs at 880nm. This interference is generally insignificant as silicate concentration of approximately 50 mgSiO<sub>2</sub>/L would be required to produce a 0.0008mgP/L positive error in orthophosphate.
- 5.2. Phosphorus contamination is a common problem. For that reason, commercial detergents should never be used to clean glassware. All glassware is cleaned with 10% HCl, rinsed with distilled deionized water. The glassware must be dedicated to the phosphorus methods and after use should be rinsed with distilled water.
- 5.3. Sample turbidity must be removed by filtration prior to analysis for

orthophosphate. Samples for total phosphate should be filtered only after digestion if needed.

**6. Equipment and Supplies**

- 6.1 Balance- analytical, capable of accurately weighing to the nearest 0.0001g.
- 6.2 Glassware- Class volumetric flask and pipettes or plastic containers as required.
- 6.3 Automated Ion Analyzer- Lachat QuikChem AE
  - 6.3.1 Autosampler
  - 6.3.2 Multichannel proportioning pump.
  - 6.3.3 Manifold (reaction unit) with heated module.
  - 6.3.4 Colorimetric Detector
  - 6.3.5 Data System

**7. Sample collection and preservation**

Samples containers may be plastic or Pyrex glass.

- For TP: if the analysis cannot be performed the day of collection the sample should be preserved by addition of 1 mL concentrated  $\text{H}_2\text{SO}_4$  per liter preferably in the field and refrigerated at  $4^\circ\text{C}$ . Sample analysis should be performed within 28 days of sample collection.
- For Orthophosphate: Samples need to be refrigerated at  $4^\circ\text{C}$  and analyzed within 48 hours. Samples cannot be preserved.

**8. Reagents and Standards**

All reagents and standards should be stored in the appropriate bottles and labeled with the following information:

- Manufacturer.
- Lot number.
- Date of preparation.
- Date of expiration.
- Concentration.
- Initials of Preparer

Use DI water for all solution. To prevent bubble formation, degas all solutions except standards with helium. Use Heat 140kPa through a helium degassing tube. Bubble He through the solution for 2 minutes.

**8.1.Reagent 1. Molybdate Color Reagent**

To a 0.2 liter volumetric flask add about 50 mL of DI water, then add 0.043g Antimony Potassium Tartrate (potassium antimony tartrate hemihydrate) or 0.046g Antimony Potassium Tartrate (antimony potassium tartrate trihydrate) and 1.7 g Ammonium Molybdate. Add 4.2 mL concentrated sulfuric acid for Total Phosphate or 7.0 mL concentrated sulfuric acid for Orthophosphate.(CAUTION: The solution will get hot during preparation). Dilute to the mark and invert to mix. Prepare fresh before every run.

**8.2.Reagent 2.Ascorbic Acid Reducing Solution, 0.33M**

In a 200 mL volumetric flask, add 50 ml of DI water and dissolve 12.0g ascorbic acid and add 0.20g of dodecyl sulfate( $\text{CH}_3(\text{CH}_2)_{11}\text{OSO}_3\text{Na}$ ).Stir until dissolved, dilute to the mark with DI water. The solution is stable for about a week if prepared with water containing no more than trace amounts of heavy metals and stored at 4°C. Discard if the solution becomes yellow.

**8.3.Reagent 3. Sulfuric Acid, 0.13M** ( Carrier for total phosphate only)

In a 1 liter volumetric flask add 500 mL water and 7.2 mL concentrated sulfuric acid. Dilute to the mark and shake to mix. Prepare fresh as needed. Degas before run.

**8.4.DI Water** - Carrier for ortho-phosphate only

**8.5.Ammonium persulfate  $(\text{NH}_4)_2\text{S}_2\text{O}_8$**

**8.6.Reagent 4. Sulfuric Acid, 5.6M** (For persulfate digestion)

In a 1 liter volumetric flask add 500 mL DI water and 310 mL concentrated sulfuric acid. Dilute to the mark and shake to mix. Prepare as needed.

**8.7.Cleaning solution:** Preparation of 0.5 M EDTA rinsing solution (~pH 8.0):  
93.05g of EDTA (FW 372.2) plus 10.14g of NaOH (FW 40) to 500 ml with DI water

**8.8.Preparation of standards.**

Three different stock standards (lot or vendor) solutions (1000ppm orthophosphate solution), commercially prepared and purchased. Certificate of analysis required. One used for calibration standards, second for ICV and the third one for LFB/MS/MSD samples.

**8.8.1 Working standard 1-** 10ppm as  $(\text{PO}_4)^{3-}$ . To 100 mL volumetric flask add 1.0ml of stock standard and dilute to the mark with DI water. Six calibration standards and a blank need to be prepared for calibration curve. Prepare fresh before every run.

#### 8.8.1.1 Calibration standards

Cal. Standard	1000 ppb	600ppb	300ppb	150ppb	60 ppb	15ppb	0 ppb
Concentration ug (PO <sub>4</sub> ) <sup>3-</sup> /L	1000	600	300	150	60	15	0
Volume(mL) of std 8.8.1 Diluted to the 50ml with DI water	5.0	3.0	1.5	0.75	0.3	0.075	0.0

8.8.2 Working standard 2 -10ppm as (PO<sub>4</sub>)<sup>3-</sup>. To 100 mL volumetric flask add 1.0 mL of standard and dilute to the mark with DI water. Used for ICV quality control sample (section 11). Prepare fresh before every run.

8.8.3 Working standard 3 10ppm as (PO<sub>4</sub>)<sup>3-</sup>. To 100 mL volumetric flask add 1.0 mL of standard and dilute to the mark with DI water. Used for LFB and MS/MSD quality control samples (section 11). Prepare fresh before every run

### 9. Sample Preparation

Sample analysis should be completed within 28 days of sample collection for Total Phosphorous soils and liquids (if preserved) and 48 hours for reactive phosphorus (o-phosphate).

9.1 For aqueous Total Phosphorous: Add 1.0 mL 5.6 M sulfuric acid to 50 mls of sample or standard in DigiTube , add 0.4g ammonium persulfate (8.5). Boil gently until volume is about 20 mls. Cool and dilute to the mark (50mL) with DI water. The sample or standard is now prepared for determination of total phosphorus. DigiTubes need to be acid washed prior to digestion.

9.2 For reactive phosphorus (o-phosphate) samples do not need any preparation.

9.3 or soils Total Phosphorous: Add 1.0 mL 5.6 M sulfuric acid to approximately 0.5 g of sample in DigiTube , add 0.4g ammonium persulfate (8.5) and bring to the final 50 ml volume with DI water. Boil gently until volume is about 10 mls. Cool and dilute to the mark (50mL) with DI water. The sample is now prepared for determination of total phosphorus. DigiTubes need to be acid washed prior to digestion.

## **10.0 Analysis**

1. Allow at least 15 minutes for the heating manifold to warm up to 37°C.
2. For orthophosphate samples should be brought to the room temperature prior to analysis.
3. Set up manifold.
4. Input data system parameters as in Lachat manual. Orthophosphate can be integrated directly, while T.P. uses bracket integration. See Appendix 6 for timing parameters for T.P. and Orthophosphate.
5. Pump DI water through all reagent lines and check for leaks and smooth flow.
6. Place samples and standard in the autosampler.
7. Calibrate the instrument by injecting standards. The data system will then associate the concentration with the instrument responses for each standard.
8. Lachat Analytical Sequence

Instrument Calibration standards

ICV

CCV\*

Blank\*\*

LFB

Samples

\*CCV should be run after ICV, after every 10 injection and at the end of the run.

\*\* For orthophosphate - Blank( DI water) should be run after ICV , after every 10 injection and at the end of the run.

For total phosphorous eluent blank need to be run after ICV, after every 10 injection and at the end of the run.

At the end of the run, place all lines into the NaOH-EDTA solution and pump through lines for approximately five minutes. Follow with a thorough water rinse and air rinse.

## **11.0 Quality Control**

- Lab Reagent Blank (or Preparation Blank) -50mls distilled deionized water treated in the same manner as sample; one per each batch of 20 samples. DI water is used for water and soil samples due to false positive results from silica based sand forming a complex that absorbs at the same 880nm as phosphate.
- Lab Fortified Blank; one per 20 samples. Add 1.5 ml of 10 ppm

orthophosphate standard (8.7.3) to 50 ml volumetric flask and dilute to the mark with DI water. Total concentration-300 ppb as orthophosphate. The same for waters and soils.

- Matrix Spike and Matrix Spike Duplicate: one of each per 10 samples. Add 1.5 ml of 10 ppm (8.7.3) orthophosphate standard to 50 ml volumetric flask and dilute with sample solution to the mark. Total concentration-300 ppb as orthophosphate. For soils: to approximately 0.5 grams of sample add 1.5 mL's of 10ppm orthophosphate standard , add DI water to the final volume 50 ml.
- Initial Calibration: An initial calibration is performed by analysis of a blank and six standards. The correlation coefficient must be  $\geq 0.990$  for the calibration to be valid. Investigate and apply corrective action if criterion is not met.
- Initial Calibration Verification Standard: Middle level of initial calibration standards. To a 50 mL's volumetric flask add 1.5 mL's of 10 ppm (8.7.2) standard and dilute to the mark with DI water. Total concentration-300 ppb as orthophosphate.
- Duplicate and spikes sample analysis: one per ten samples.

## 12.0 Calculation

The computer yields result directly in ug/L as  $(\text{PO}_4)^{3-}$ . Divide phosphate results by 3.066 to convert result to phosphorus (P) ug/L .

## 13.0 Reporting limits

For aqueous samples, the RL is equal the lowest calibration standard.

For soil samples: using 50 mls reflux tube:

$$\text{RL (mg/Kg)} = \frac{0.05\text{L} \times \text{low cal. Std. (ug/L)}}{\text{wt. (g)}}$$

## 14.0 Data Management and Data Package Documentation

Each data package must include a copy of the following:

- 14.1. Sample preparation sheet (Appendix 4)
- 14.2. Standards preparation sheet (Appendix 3)
- 14.3. Reagent preparation sheet (Appendix 2)
- 14.4. Raw instrument data and analytical sequence



All standards must be traceable to the original vendor stock. Standards must be identified in detail (vendor, lot number, and certificate).

The sequence logbook shall be kept near the instrument (all current and past sequence logbooks are kept next to the instrument).

Electronically archived data are kept on a CD near the instrument.

#### **15.0 Project review**

Upon completion of a project a project review form should be filled out and accompany the final report in the report folder. The first section (requested analysis and data folder completeness check) should be completed by the analyst. The last two sections (data evaluation and final report) should be completed by two different chemists that have knowledge of the method (See Appendix 5 for the project review forms).

#### **16.0 Pollution Prevention and Waste Management**

NERL encourages all chemist and biologists to investigate micro analytical techniques, innovative technologies, and chemical substitution in laboratory processes to reduce waste and prevent pollution. As analytical SOPs are reviewed, on an annual basis, the responsible chemist or biologist will incorporate waste minimization practices where practicable and where these practices have been demonstrated to return data of equivalent quality.

Chemists and biologists must refer to the Waste Management Program SOP for proper disposal of laboratory waste. Personnel should contact the Environmental, Safety and Health Department if changes in the analytical SOP will generate new waste streams. Questions regarding the proper disposal of laboratory waste and purchase of new reagents should be directed to the Environmental, Safety and Health Department in advance of actually initiating a change in the analytical method.

Effluent from the channels as well as the sample effluent are acidic. Need to be disposed of in a labeled waste satellite containers.

#### **17.0 Preventive maintenance**

Required maintenance as described in the Lachat manual.

#### **18.0 Troubleshooting**

If base line drifts and cleaning the system in prescribed manner does not help, the heating coil tubing may need to be changed. An unusually noisy base line may due to insufficient purging of air from the reagent. Tiny bubbles tend to develop in the heated tubing and became trapped in the flow cell causing base line problems.

**19.0 Method References**

Method 365.1, Determination of Phosphorus by Semi-Automated Colorimetry, revision 2.0 August 1993.

Lachat Instruments, Method 10-115-01-1-B, Orthophosphate

Lachat Instruments Method 10-115-01-1-F, Determination of Total Phosphorus by Flow Injection Analysis Colorimetry, Revision 13 August 1998

Soils digestion- method developed in NERL

**Appendix I: Acceptance Criteria**

QA/QC Sample	Frequency	Acceptance Criteria	Corrective Action	
			o-Phosphate	Total Phosphate
Blanks: Distilled for T.P. Not distilled for OP	1 per batch (up to 20 samples) After calibration, continuing calibration, every 10 injections and at the end of the run	< RL	-	If results for samples below RL report all data. If results for samples less than 10 times of the blank value, re digest entire batch. If not enough sample qualify all data with B and write an explanation.
Initial Cal. Verification (ICV)	After calibration	$\pm 10\%$ of True value	Prepare new calibration curve, Prepare new ICV if failed terminate analysis, correct problems, re analyze all samples	Re prep entire batch
Continuing Calibration Verification (CCV)	after every 10 injections and at the end of the run	$\pm 10\%$ of True value	Prepare new calibration curve. Prepare new CCV if failed terminate analysis, correct problems, re analyze all samples	Re prep entire batch
Laboratory Fortified Blank (LFB)	1 per batch (up to 20 samples)	$\pm 10\%$ of True value	Prepare new calibration curve. Prepare new LFB if failed terminate analysis; correct problems, re analyze all samples	Re prep entire batch If not enough sample volume qualifies all data.
MS/MSD	1 per 10 samples	$\pm 10\%$ of true value for drinking water Control limits for soils and waste water	Qualify data for the sample if LFB is within spec.	

Laboratory Duplicate	1 per 10 samples	$\pm 10\%$ RPD for drinking water $\pm 20\%$ RPD or control charts for soils and waste water <sup>2</sup>	Qualify data (J) for sample with explanation	
Samples Samples Holding Time	Section 7	Samples must be analyzed within holding Times	If re-sampling is not available, results are estimated (J) for all samples and project notice submitted with the report.	
IDC/LOQ	One a year per analyst	4 replicates at concentration 1 to 2 times reporting limit 80-120% recovery <20% RSD <sup>3</sup>	Correct problems; reanalyze all samples.	Correct problems; re prep and re analyze all samples
MDL	Once per new instrument or there is the change in the method.	Run 7 replicates at concentration of 1 to 3 times reporting limits	Correct problems; reanalyze all samples.	Correct problems; re prep and re analyze all samples
QCS	Run as PT studies.	-	-	

1= Acceptance criteria from Reference Method or Reference QC documentation

2= Acceptance criteria calculated from in-house historical data (control charts)

**Appendix 2: Total Phosphorus/Orthophosphate Reagent Preparation**

Date:

PN:

Analyst:

Survey:

**1. Preparation of Color reagent (SOP Section 8.1, Reagent 1)**

Antimony Potassium Tartrate:

Barcode: \_\_\_\_\_

Mfg: \_\_\_\_\_

Lot#: \_\_\_\_\_

Date Rec'd: \_\_\_\_\_

Date Opened: \_\_\_\_\_

\_\_\_\_\_ grams antimony potassium tartrate +

\_\_\_\_\_ grams ammonium molybdate +

\_\_\_\_\_ mL conc. H<sub>2</sub>SO<sub>4</sub> diluted

to \_\_\_\_\_ L with DI water

Ammonium Molybdate:

Barcode: \_\_\_\_\_

Mfg: \_\_\_\_\_

Lot#: \_\_\_\_\_

Date Rec'd: \_\_\_\_\_

Date Opened: \_\_\_\_\_

Sulfuric acid:

Barcode: \_\_\_\_\_

Mfg: \_\_\_\_\_

Lot#: \_\_\_\_\_

Date Rec'd: \_\_\_\_\_

Date Opened: \_\_\_\_\_

**2. Preparation of Ascorbic Acid Reducing Solution, 0.33M (SOP Section 8.2, Reagent 2)**

Ascorbic acid:

Barcode: \_\_\_\_\_

Mfg: \_\_\_\_\_

Lot#: \_\_\_\_\_

Date Rec'd: \_\_\_\_\_

Date Opened: \_\_\_\_\_

\_\_\_\_\_ grams ascorbic acid +

\_\_\_\_\_ grams dodecyl sulfate diluted

to \_\_\_\_\_ L with DI water

Dodecyl Sulfate:

Barcode: \_\_\_\_\_

Mfg: \_\_\_\_\_

Lot#: \_\_\_\_\_

Date Rec'd: \_\_\_\_\_

Date Opened: \_\_\_\_\_

**3. Preparation of Sulfuric acid, 0.13M (SOP Section 8.3, Reagent 3)**

Barcode: \_\_\_\_\_

Mfg: \_\_\_\_\_

Lot#: \_\_\_\_\_

Date Opened: \_\_\_\_\_

Date Rec'd: \_\_\_\_\_

\_\_\_\_\_ mL conc. H<sub>2</sub>SO<sub>4</sub> diluted

to \_\_\_\_\_ L with DI water

**4. Preparation of Sulfuric acid, 5.6M (SOP Section 8.5, Reagent 4) - for persulfate digestion**

Barcode: \_\_\_\_\_

Mfg: \_\_\_\_\_

Lot#: \_\_\_\_\_

Date Opened: \_\_\_\_\_

Date Rec'd: \_\_\_\_\_

\_\_\_\_\_ mL conc. H<sub>2</sub>SO<sub>4</sub> diluted

to \_\_\_\_\_ L with DI water

### Appendix 3: Total Phosphorus/Orthophosphate Standard Preparation

Date:

Analyst:

PN:

Survey:

Stock Orthophosphate Solutions (1000ppm):

#1

Mfg: \_\_\_\_\_

Lot#: \_\_\_\_\_

Date Rec'd: \_\_\_\_\_

Date Opened: \_\_\_\_\_

Date Expires: \_\_\_\_\_

Used for: \_\_\_\_\_

Barcode: \_\_\_\_\_

#2

Mfg: \_\_\_\_\_

Lot#: \_\_\_\_\_

Date Rec'd: \_\_\_\_\_

Date Opened: \_\_\_\_\_

Date Expires: \_\_\_\_\_

Used for: \_\_\_\_\_

Barcode: \_\_\_\_\_

#3

Mfg: \_\_\_\_\_

Lot#: \_\_\_\_\_

Date Rec'd: \_\_\_\_\_

Date Opened: \_\_\_\_\_

Date Expires: \_\_\_\_\_

Used for: \_\_\_\_\_

Barcode: \_\_\_\_\_

10ppm as  $(\text{PO}_4)^{3-}$  working standards: \_\_\_\_\_ mL of stock to 100mL DI water = 10ppm

Calibration Standard Table

Cal. Std.	1000 ppb	600 ppb	300 ppb	150 ppb	60 ppb	15 ppb	0 ppb
Concentration ug $(\text{PO}_4)^{3-}$ /L	1000	600	300	150	60	15	0
Volume (mL) of 10ppm working std. (Sec. 8.6.1) diluted to 50 mL with DI Water	5.0	3.0	1.5	0.75	0.3	0.075	0

ICV = \_\_\_\_\_ mL of 10ppm working std. \_\_\_\_\_ to 50mL DI water = 300 ug  $(\text{PO}_4)^{3-}$ /L

LFB = \_\_\_\_\_ mL of 10ppm working std. \_\_\_\_\_ to 50mL DI water = 300 ug  $(\text{PO}_4)^{3-}$ /L

MS/MSD = \_\_\_\_\_ mL of 10ppm working std. \_\_\_\_\_ to 25mL with sample = 300 ug  $(\text{PO}_4)^{3-}$ /L

## Appendix 4: Total Phosphorus Sample Preparation

Date:  
PN:

Analyst:  
Survey:

[illegible]

Ammonium persulfate: Mfg:

Lot#:

Barcode:

Date Received:

Date Opened:

Expires:

**Appendix 5: Timing parameters for total phosphate and ortho-phosphate:**

Parameters	Total phosphate	Ortho-phosphate	Comments
Sample throughput	60 samples/h	60 samples/h	
Pump speed	35	35	
Cycle period	60	60	
Chemistry	Brackish	Direct/Brackish	
Calibration fit type	2 <sup>nd</sup> Order Polynomial	2 <sup>nd</sup> Order Polynomial	
Load time	0	0	
Load period	18	22	
Injection period	42	38	
Sample reaches first valve	24	20	
Probe in samp. Period	23	30	
Min. probe wash	10	10	



**Appendix 6: Method Comparison**

Method	EPA Series Method 365.1	ELASOP-INGTP10
Parameter		
Applicability	Drinking, surface, domestic and industrial wastes.	Drinking, surface, domestic and industrial wastes
Number of Analytes	Phosphorus	Total Phosphate and Orthophosphate
Method Validation	<p>Initial demonstration of performance:</p> <ol style="list-style-type: none"> <li>1. The Linear Calibration Range (LCR) must be determined initially and verified every 6 months. The verification of linearity must use a minimum of a blank and 3 standards.</li> <li>2. A Quality Control sample (QCS), an independent standard, is prepared and analyzed at least quarterly to verify the calibration standards and instrument performance. If not within <math>\pm 10\%</math> of stated value, determine source of problem and correct before continuing with analyses.</li> <li>3. Determine MDLs by analyzing seven replicates of Laboratory fortified blanks at concentration of 2 to 3 times the estimated detection limit. MDLs must be determined every six months.</li> </ol>	<p>Initial demonstration of performance:</p> <ol style="list-style-type: none"> <li>1. The Calibration Range (CR) must be determined initially and verified every run. A blank and 6 standards used for calibration</li> <li>2. An independent standard (ICV) analyzed with every batch to verify the calibration standards and instrument performance. Acceptance criteria <math>\pm 10\%</math> of the true value.</li> <li>3. Determine MDLs by analyzing seven replicates of Laboratory fortified blanks at concentration of 2 to 4 times the reporting limit. MDLs need to be run one per new instrument or if major changes were done to the method.</li> </ol>
QC Check Standards/ Samples	<p>After calibration is completed, verify by analyzing QCS. If not within <math>\pm 10\%</math> of stated value, terminate analysis and re calibrate instrument.</p> <p>Prepare and analyze a Laboratory</p>	<p>After calibration is completed, verify by analyzing ICV. If not within <math>\pm 10\%</math> of stated value, terminate analysis and re calibrate instrument.</p> <p>Prepare and analyze a Laboratory</p>

Method	EPA Series Method 365.1	ELASOP-INGTP10
Parameter		
<b>QC Check Standards/Samples</b>	Fortified Blank (LFB) with each batch of samples by fortifying laboratory reagent water with the QCS. If the recovery of the analyte is not within 90-110%, the analyte is judged out of control. Determine source of problem and correct before continuing with analyses.	Fortified Blank (LFB) with each batch of samples by fortifying laboratory reagent water with the standard. If the recovery of the analyte is not within 90-110%, the analyte is judged out of control. Determine source of problem and correct before continuing with analyses.
<b>Standard Solution Expiration</b>	Stock standard: Not specified.	Stock standard used by expiration date. Working standard solution: Prepare before run
<b>Initial Calibration</b>	Minimum of 3 levels and a blank. Range: 0.01-1.0 mgP/L	Up to 6 levels and a blank. Range: 5-300 ugP/L
<b>Continuing Calibration</b>	<p>Analyze Instrument performance check (IPC) solution (mid-range check standard) immediately following calibration, after every 10 samples and at the end of the run.</p> <ol style="list-style-type: none"> <li>1. If not within <math>\pm 10\%</math> of stated value, reanalyze IPC.</li> <li>2. If second analysis of IPC is not within <math>\pm 10\%</math> of stated value, discontinue analysis, determine the cause and/or in the case of drift recalibrate instrument. Reanalyze all samples since last compliant IPC.</li> </ol>	<ol style="list-style-type: none"> <li>1. Analyze CCV solution (mid-range check standard) immediately following calibration, after every 10 injection and at the end of the run.</li> <li>3. If not within <math>\pm 10\%</math> of stated value, reanalyze CCV</li> <li>4. If second analysis of CCV is not within <math>\pm 10\%</math> of stated value, discontinue analysis, determine the cause and/or in the case of drift recalibrate instrument. Reanalyze all samples since last compliant CCV</li> </ol>

Method	EPA Series Method 365.1	EIASOP-INGTP10
Parameter		
<b>Accuracy/ Precision</b>	<p>Spike and analyze one sample out of every 10 (Laboratory Fortified Matrix: LFM).</p> <p>The added analyte concentration should be the same as that used in the LFB. %R = 90-110</p>	<p>Spike and analyze one sample out of every 10 (Laboratory Fortified Matrix: LFM).</p> <p>The added analyte concentration should be the same as that used in the LFB. %R = 90-110 for drinking water and %R = 85-115 for soil and waste water</p>
<b>Blanks</b>	<p>A Laboratory reagent blank (LRB) is carried through the entire sample preparation and analysis scheme with each batch of samples.</p> <p>Values that exceed the MDL indicate contamination should be suspected and corrective actions must be taken before continuing analysis.</p> <p>A Calibration blank (CCB) is to be analyzed after each IPC solution.</p>	<p>A Laboratory reagent blank (LRB) is carried through the entire sample preparation and analysis with each batch of samples.</p> <p>Values that exceed the RL indicate contamination should be suspected and corrective actions must be taken before continuing analysis.</p> <p>Lab blank (CCB) is to be analyzed after each CCV solution.</p>
<b>Preservation/ Storage Conditions</b>	pH <2 with H <sub>2</sub> SO <sub>4</sub> for TP and not preserved for OP 4°C storage required	pH <2 with H <sub>2</sub> SO <sub>4</sub> for TP not preserved for OP 4°C storage required
<b>Holding Time</b>	28 days for TP. As soon as possible for OP	28 days for TP. As soon as possible for OP and not exceed 48 hours
<b>Field Sample Amount Required</b>	Glass or plastic container.	Glass or plastic container.
<b>Amount for Digestion</b>	50 ml for TP	50 ml for TP

<b>Method</b>	<b>EPA Series Method 365.1</b>	EIASOP-INGTP10
<b>Parameter</b>		
<b>Reagent Preparation</b>	Combine color reagent: mix 50 ml of 5N H <sub>2</sub> SO <sub>4</sub> , 5 mL of antimony potassium tartrate solution, 15 ml of ammonium molybdate solution and 30 mL of ascorbic acid solution in 100 ml volume Prepare freshly for each run	Reagent1: 0.043g Antimony potassium tartrate, 1,7g Ammonium molybdate , 50 mL of water and 4.2 mL of concentrated H <sub>2</sub> SO <sub>4</sub> for TP or 7 mL of concentrated H <sub>2</sub> SO <sub>4</sub> for OP Prepare freshly for each run