

Total Phosphorous Dissolved Phosphorus Colorimetric, Combined Reagent

References: **SM 4500P-E**, Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

SM4500P-B, Section 5 (Persulfate Digestion), Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

AQ2 method: **EPA-119-A** Rev. 7, equivalent to EPA 365.1, version 2(1993) **SM4500-P-B, F(18-20)**

1. Scope and Application

Matrices: Water and wastewater samples and soils.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Phosphorus occurs in natural waters and in wastewaters almost solely as phosphates. These are classified as orthophosphates, condensed phosphates (pyro-, meta-, and other polyphosphates), and organically bound phosphates. They occur in solution, in particles or detritus, or in the bodies of aquatic organisms.

These forms of phosphate arise from a variety of sources. Small amounts of certain condensed phosphates are added to some water supplies during treatment. Larger quantities of the same compounds may be added when the water is used for laundering or other cleaning, because these materials are major constituents of many commercial cleaning preparations. Phosphates are used extensively in the treatment of boiler waters. Orthophosphates applied to agricultural or residential cultivated land as fertilizers are carried into surface waters with storm run-off and to a lesser extent with melting snow. Organic phosphates are formed primarily by biological processes. They are contributed to sewage by body wastes and food residues, and also may be formed from orthophosphates in biological treatment processes or by receiving water biota.

Phosphorus is essential to the growth of organisms and can be the nutrient that limits the primary productivity of a body of water. In instances where phosphate is a growth-limiting nutrient, the discharge of raw or treated wastewater, agricultural drainage, or certain industrial wastes to that water may stimulate the growth of photosynthetic aquatic micro- and macro-organisms in nuisance quantities.

Phosphates also occur in bottom sediments and in biological sludges, both as precipitated inorganic forms and incorporated into organic compounds.

Phosphorus analyses embody two general procedural steps: (a) conversion of the phosphorus form of interest to dissolved orthophosphate, and (b) colorimetric determination of dissolved orthophosphate. The separation of phosphorus into its various forms is defined analytically but the analytical differentiations have been selected so that they may be used for interpretive purposes.

Filtration through a 0.45- μ m-pore-diameter membrane filter separates dissolved from suspended forms of phosphorus. No claim is made that filtration through 0.45- μ m filters is a true separation of suspended and dissolved forms of phosphorus; it is merely a convenient and replicable analytical technique designed to make a gross separation.

Phosphates that respond to colorimetric tests without preliminary hydrolysis or oxidative digestion of the sample are termed "reactive phosphorus." While reactive phosphorus is largely a measure of orthophosphate, a small fraction of any condensed phosphate present usually is hydrolyzed unavoidably in the procedure. Reactive phosphorus occurs in both dissolved and suspended forms.

Acid hydrolysis at boiling-water temperature converts dissolved and particulate condensed phosphates to dissolved orthophosphates. The hydrolysis unavoidably releases some phosphate from organic compounds, but this may be reduced to a minimum by judicious selection of acid strength and hydrolysis time and temperature. The term "acid-hydrolyzable phosphorus" is preferred over "condensed phosphate" for this fraction.

The phosphate fractions that are converted to orthophosphate only by oxidation destruction of the organic matter present are considered "organic" or "organically bound" phosphorous. The severity of the oxidation required for this conversion depends on the form of, and to some extent on the amount of, the organic phosphorus present. Like reactive phosphorus and acid hydrolyzable phosphorus, organic phosphorus occurs both in the dissolved and suspended fractions.

The total phosphorus as well as the dissolved and suspended phosphorus fractions each may be divided analytically into the three chemical types that have been described: reactive, acid hydrolyzable, and organic phosphorus. Determinations usually are conducted only on the unfiltered and filtered samples. Suspended fractions generally are determined by difference.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Digestion Method: Because phosphorus may occur in combination with organic matter, a digestion method to determine total phosphorus must be able to oxidize organic matter effectively to release phosphorus as orthophosphate. This digestion is performed by using the persulfate oxidation technique.

Colorimetric Method: The ascorbic acid method is used for the determination of orthophosphate in environmental samples. An extraction step is recommended for the lower levels and when interferences must be overcome. Ammonium molybdate and potassium antimonyl tartrates react in acid medium with orthophosphate to form a heteropoly acid-phosphomolybdic acid that is reduced to intensely colored molybdenum blue by ascorbic acid. The absorbance of this complex is measured photometrically at 880nm.

2.1 Method Modifications from Reference

Glassware is acid rinsed with room temperature 1:1 HCl, instead of hot dilute HCl.

Initial testing of samples with phenolphthalein has been eliminated since samples are received already preserved with H₂SO₄ and are pH checked by the Login Department upon receipt.

Soil samples are analyzed using the same digestive procedure.

3. Reporting Limits

The Reported Detection Limit is 0.01mg/L for waters and 5.0mg/kg for soils

4. Interferences

Correction for Turbidity or Interfering Color: The natural color of water generally does not interfere at the high wavelength used. For highly colored or turbid waters, prepare a blank by adding all reagents except ascorbic acid and potassium antimonyl tartrate to the digested sample aliquot. Subtract the blank absorbance from the absorbance of each sample.

Arsenates react with the molybdate reagent to produce a blue color similar to that formed with phosphate. Concentrations as low as 0.1mg As/L interfere with the phosphate determination.

Hexavalent chromium and NO₂ interfere to give results about 3% low at concentrations of 1mg/L and 10 to 15% low at 10mg/L.

Sulfide (Na₂S) and silicate do not interfere at concentrations of 1.0 and 10mg/L.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Water samples are collected in 500mL plastic bottles, soil samples may be collected in plastic or glass jars.

6.2 Sample Preservation

If samples are for Dissolved Phosphorus analysis, filtration must take place prior to preservation with H₂SO₄ to a pH < 2.

All samples are preserved with H₂SO₄.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are stored under refrigeration at 4 ± 2 °C. Analysis must be performed within 28 days of collection. All samples should be analyzed as soon as possible after digestion. If a prolonged period passes in between, sample extracts are refrigerated at 4 ± 2 °C.

7. Equipment and Supplies

- 7.1 Spectrophotometer**, with infrared phototube for use at 880nm, providing a light path of 2.5cm.
- 7.2 Acid-washed Glassware:** Use acid-washed glassware for determining low concentration of phosphorus. Phosphate contamination is common because of its absorption on glass surfaces. Avoid using commercial detergents containing phosphate. Clean all glassware with 1:1 HCl two times followed by two DI water rinses. Preferably, reserve the glassware only for phosphate determination. Only disposable syringes and filters are to be used for filtering samples for Dissolved Phosphorus analysis.
- 7.3 Centrifuge Tubes:** 50mL volume. (Must be new and disposable.)
- 7.4 Hot Plate:** A 30cm x 50cm heating surface is adequate.
- 7.5 Scoop, 0.5gm** To hold required amounts of persulfate crystals.
- 7.6 Erlenmeyer Flasks:** 125mL volume.
- 7.7 0.45µm membrane filters:** For Dissolved Phosphorus sample preparation.
- 7.8 Borosilicate Glass beads**
- 7.9 SEAL AQ2 Discrete Analyzer**, with all associated reagent wedges, sample tubes, and reaction segments. The SEAL has a light and filter capable of maintaining a 880nm wavelength.
- 7.10 Boiling Chips** ultra-pure, non-reactive.
- 7.11 Syringes** to use with membrane filters.
- 7.12 Pipettes** Class A glass or automated.

8. Reagents and Standards

- 8.1 Calibration Curve and Spike, Stock Complex Phosphate Standard: 1000 mgP/L** This stock solution is certified and purchased commercially prepared. Stored at room temperature per manufacturer's specifications. Expires upon manufacturer's specified date.
- 8.2 Calibration Curve and Spike, Intermediate Complex Phosphate Standard: 50 mgP/L** Dilute 5.0mL stock complex phosphate solution to 100mL with DI water. Store at $4 \pm 2^{\circ}\text{C}$. Expires 6 months after date of preparation.

8.3 Calibration Curve, Working Standard: 1.0 mgP/L Add 2mL of 50 mgP/L intermediate standard (Section 8.2) to 100mL volumetric flask and dilute to volume with DI water. Prepare fresh on each day of use.

8.4 Calibration Standards: Follow table below. Prepare fresh on each day of use.

| Volume of 1.0 mg/L Working Standard (Section 8.3) | Final Volume (mL) | Calibration Standard Final Concentration (mgP/L) |
|---|-------------------|--|
| 0 mL | 50 | 0 |
| 0.5 mL | 50 | 0.010 |
| 2 mL | 50 | 0.040 |
| 5 mL | 50 | 0.100 |
| 25mL | 50 | 0.500 |
| 50mL | 50 | 1.000 |

8.5 ICV-LCS-CCV Stock Complex Phosphate Standard: 1000 mgP/L Second, independent, source standard. Stored at room temperature per manufacturer's specifications. Expires upon manufacturer's specified date.

8.6 ICV-LCS-CCV Intermediate Complex Phosphate Standard: 50 mgP/L Add 5mL of 1000 mgP/L stock standard (Section 8.5) to 100mL volumetric flask and dilute to volume with DI water. Store at $4 \pm 2^{\circ}\text{C}$. Expires 6 months after date of preparation.

8.7 ICV-LCS-CCV Working Standard: Prepare fresh each day of use.

8.7.1 0.5 mgP/L: Add 0.5mL of 50 mg/L intermediate standard (Section 8.6) to 50mL centrifuge tube and dilute to the 50mL mark with DI water.

8.8 Matrix Spike: Intermediate Phosphate Standard (Section 8.2) is utilized for matrix spike solution. Pipet 0.5mL of the 50 mgP/L standard into 50mL of sample to result in a 0.5mg/L spike concentration.

8.9 Sulfuric Acid, H_2SO_4 , 5N: Dilute 140mL concentrated sulfuric acid to 1L with DI. Store at room temperature. Expires 6 months from date of preparation.

8.10 Potassium Antimonyl Tartrate Solution: Dissolve 1.3715g $\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6 \cdot \frac{1}{2}\text{H}_2\text{O}$ in 400mL DI water in a 500mL volumetric flask and dilute to volume. Store at $4 \pm 2^{\circ}\text{C}$. Expires one month from date of preparation.

8.11 Ammonium Molybdate Solution: Dissolve 10g $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ in 250mL distilled water. Store at $4 \pm 2^{\circ}\text{C}$. Expires one month from date of preparation.

8.12 Ascorbic Acid, 0.1M: Dissolve 3.52g ascorbic acid in 200mL DI water. The solution is stable for about 1 week at $4 \pm 2^{\circ}\text{C}$.

8.13 Orthophosphate 1000ppm solution Independent, source standard. Store at $4 \pm 2^{\circ}\text{C}$. Expires upon manufacturer's specified date.

8.14 Orthophosphate 25ppm spike solution Add 2.5mLs 1000ppm Orthophosphate solution to a clean, glass 100mL volumetric and dilute to volume with DI. Store at $4 \pm 2^{\circ}\text{C}$. Expires 6 months from date of preparation.

8.15 SEAL Working Ascorbic Acid, 15g/L (with orthophosphate spike): Dissolve 1.5g of ascorbic acid in about 80mL DI water. Spike with .15mL 25ppm Orthophosphate standard to produce a spike level of .025mg P/L. Dilute to 100mL and mix well. The solution is stable for one week if stored at $4 \pm 2^{\circ}\text{C}$. Discard if the solution becomes yellowed.

8.16 Combined Reagent: Mix 8.9, 8.10, 8.11, and 8.12 in the following proportions for 100mL of the combined reagent: 50mL 5N H_2SO_4 (Section 8.9), 5mL potassium antimonyl tartrate solution (Section 8.10), 15mL ammonium molybdate solution (Section 8.11), and 30mL ascorbic acid solution (section 8.12). Mix after addition of each reagent. Let all reagents reach room temperature before they are mixed and mix in the order given. If turbidity forms in the combined reagent, shake and let stand for a few minutes until turbidity disappears before proceeding. The reagent is stable for 4 hours. Discard reagent if it turns blue or black in color.

8.17 SEAL Working Coloring Reagent: To a clean 100mL volumetric flask, add 40mL sulfuric acid (8.9), followed by 6.5 mL antimony potassium tartrate (8.10) and swirl to mix. Then, add 20 mL ammonium molybdate (8.11). Swirl the contents, fill the flask up to the mark with DI water and mix well. Expires three weeks from day of preparation if stored at $4 \pm 2^{\circ}\text{C}$. Discard if the reagent turns blue or becomes turbid.

8.18 Sodium Hydroxide, 6N: Dissolve 240 grams of NaOH pellets in 1000mL of DI water. Store at room temperature. Expires one month from date of preparation.

8.19 Phenolphthalein Indicator: Aqueous solution, commercially available. Store at room temperature. Expires upon manufacturer's specified date.

8.20 11N Sulfuric Acid Solution: Dilute 308mL concentrated sulfuric acid to 1000mL with DI. Store at room temperature. Expires 6 months from date of preparation.

8.21 Potassium Persulfate ($\text{K}_2\text{S}_2\text{O}_8$): Commercially available. Store at room temperature. Expires upon manufacturer's specified date.

8.22 Deionized Water

8.23 Soil LCS/SRM ERA Standard Reference Material for Nutrients in soil, catalog no. 542

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Method Blank/Calibration Blank - One method blank, which consists of DI water brought through the entire method, must be analyzed per batch of 20 samples or less. The CCB is analyzed after every 10 samples and at the end of the sequence.

Results of the Blanks must be less than the reporting limit. Otherwise the entire batch of samples must be re-prepared and reanalyzed. Exceptions are samples with results of more than 10 times the positive blank value.

Soil blanks are made with 0.1gm boiling chips and 50mLs of DI water and are analyzed like water blanks.

9.2 Laboratory Control Sample (LCS)

Analyze one per batch of 20 samples or less. The calibration curve must be verified by a second source standard prior to performing any sample analysis. For Total and Dissolved Phosphorus, the LCS is the ICV.

The ICV/LCS must be recovered within 90-110% of the true value. If the ICV/LCS fails, re-analyze. If failure continues, stop analysis, correct problem and re-calibrate.

Soil LCS's are made with approximately 0.15g of Standard Reference Material (SRM) brought up to 50mL with DI water.

9.3 Initial Calibration Verification (ICV)

The calibration curve must be verified by a second source standard prior to performing any sample analysis. For Total and Dissolved Phosphorus, the ICV is the LCS.

The ICV/LCS must be recovered within 90-110% of the true value for water samples and be within vendor criteria for SRM. If the ICV/LCS fails, re-analyze. If failure continues, stop analysis, correct problem and re-calibrate.

9.4 Continuing Calibration Verification (CCV)

The calibration curve must be verified by a second source standard. The CCV is analyzed after every 10 samples and at the end of the sequence to verify the curve.

The CCV must be recovered within 90-110% of the true value. If the CCV fails, re-analyze. If failure continues, stop analysis, correct problem and re-calibrate.

9.5 Matrix Spike

Analyze one per batch of 20 samples or less. Concentration is 0.5 mgP/L. The matrix spike must be recovered within 75 – 125% of the true value. If the matrix spike recovery is outside acceptance criteria, and the LCS is acceptable, a narrative is submitted with the data for inclusion on the client report.

9.6 Laboratory Duplicate

Analyze one sample in duplicate per batch of 20 samples or less. The RPD must be $\leq 20\%$. If this criterion is not met, a narrative is submitted with the data for inclusion on the client report.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

9.8.1 Using spectrophotometer:

- Acid-rinse all glassware
- Calibration curve generation.
- Filter samples if analysis is for Dissolved Phosphorus, then preserve with H_2SO_4 .
- Add 50mL of water sample or 0.1g of soil sample and 50mL DI water to an Erlenmeyer flask.
- Add 1mL H_2SO_4 solution and scoop solid $\text{K}_2\text{S}_2\text{O}_8$ and glass beads
- Boil down to 10mL or less.
- Cool and dilute to 30mL with DI.
- Add 1 drop phenolphthalein indicator solution.
- Neutralize to faint pink color with NaOH.
- Add sample aliquot to a new centrifuge tube and bring up to 50mL with DI.
- Add 4 mL combined reagent to a 25mL aliquot of sample.
- Read sample absorbance after 10-30 minutes.
- Analyze CCV and CCB after every 10 samples to verify curve.
- End sequence with CCV and CCB.
- Calculate results.

9.8.2 Using SEAL AQ2 analyzer:

- Acid-rinse all glassware
- Filter samples if analysis is for Dissolved Phosphorus, then preserve with H_2SO_4 .
- Add 50mL of water sample (soils are not done on the SEAL) to an Erlenmeyer flask, along with a 1.0ppm calibration standard for the calibration curve.
- Add 1mL H_2SO_4 solution and scoop solid $\text{K}_2\text{S}_2\text{O}_8$ and glass beads
- Boil down to 10mL or less.
- Cool and dilute to 50mL with DI.
- Turn on the SEAL AQ2 analyzer.
- Fill out a run sequence and save it.
- Fill cups, including a cup of 1.0ppm digested standard for the curve
- Start the analysis.
- Change names of blank and LCS to be what the LIMS will recognize (ie, what is on the batch sheet).
- Export the data to LIMS by dropping it into the "SEAL on bowzer" folder.

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation for Dissolved Phosphorus Analysis

Prior to preservation, samples to be analyzed for dissolved phosphorus are filtered using new disposable syringes and new 0.45um filter discs. 100mL of sample is filtered, poured into two new centrifuge tubes and preserved with H₂SO₄.

10.2 Initial Calibration

10.2.1:

Preparation of calibration curve, with spectrophotometer: Prepare individual calibration curve from a series of six digested standards (0 mgP/L to 1.0 mgP/L) on each day of analysis. The curve must be digested. Use DI water without the combined reagent to zero the Spectrophotometer. Plot absorbance vs. phosphate concentration to give a straight line. The correlation coefficient must be 0.995 or greater for the curve to be considered valid. Analyze at least one phosphate standard with each batch of 20 samples or less.

All calibration points are back calculated (on excel) and should be within 10% from true concentration, except 2 lowest points of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse than 0.995.

10.2.2:

Preparation of calibration curve, with SEAL AQ2 analyzer: Prepare and digest a 1.0 mgP/L standard and put it into the first slot in the auto-sampler. When prompted, click on "Auto-calibrate" to start calibration. Once the curve is finished, it may be checked in the "calibration" section. The correlation coefficient must be 0.995 or greater for the curve to be considered valid.

. All calibration points are back calculated by SEAL software and should be within 10% from true concentration, except 2 lowest points of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse than 0.995.

10.3 Equipment Operation and Sample Processing, with spectrophotometer

10.3.1 Add 50mL or a suitable portion of thoroughly mixed sample to a prepared (Section 7.2) 125mL Erlenmeyer flask. Use 0.1g of soil sample with 50 ml of DI for soil samples.

10.3.2 Add 1mL H₂SO₄ solution, one scoop solid K₂S₂O₈ and 3 to 5 glass beads.

10.3.3 Boil gently on a preheated hot plate until a final volume of 10mL or less is reached. Organophosphorus compounds such as AMP may require as much as 1-1/2 to 2 hours for complete digestion.

10.3.4 Cool, dilute to 30mL with DI water.

10.3.5 Add 0.05mL (1 drop) phenolphthalein indicator solution.

10.3.6 Neutralize to a faint pink color with NaOH.

- 10.3.7 Pour pink liquid sample into a new (unused) centrifuge tube and bring volume to 50mL with DI. Pour back into a 125mL Erlenmeyer flask.
- 10.3.8 Swirl the sample to mix and pour off 25mL digested sample into centrifuge tube.
- 10.3.9 Add 4.0mL combined reagent to all 25mL sample and QC sample aliquots and mix thoroughly.
- 10.3.10 After at least 10 minutes but no more than 30 minutes, use DI as the reference solution to zero the spectrophotometer at 880nm. Measure absorbance of each sample and record in the electronic laboratory notebook. If samples seem to have high background color before the addition of the coloring reagent, a background color may be checked for (see section 4).

If the sample concentration is greater than the highest concentration of the calibration curve (1.0mg/L), the digested sample is diluted with DI water to a concentration within the range of the calibration curve.

10.4 Equipment Operation and Sample Processing, with SEAL AQ2 Analyzer

- 10.4.1 Acid rinse all glassware twice with 1:1 hydrochloric acid and then twice with DI water.
- 10.4.2 Pour out 50mLs of mixed samples and QC samples, including a 1.0ppm calibration standard.
- 10.4.3 Add 1mL H₂SO₄ solution, one scoop solid K₂S₂O₈ and 3 to 5 glass beads.
- 10.4.4 Boil gently on a preheated hot plate until a final volume of 10mL or less is reached. Organophosphorus compounds such as AMP may require as much as 1-1/2 to 2 hours for complete digestion.
- 10.4.5 Cool and dilute to 50mLs.
- 10.4.6 Turn on SEAL AQ2 analyzer by flipping first the small, and then the large switch in the back.
- 10.4.7 Give the instrument at least half an hour to warm up.
- 10.4.8 If it has not yet been done that day, go through daily start-up, check voltages, and test aspiration.
- 10.4.9 Double click on scheduling to open the schedule form, and insert samples, Method Blanks, LCSs, Duplicates, and Matrix Spikes (the CCVs and CCBs populate automatically) NB: leave the first spot for the calibration curve.
- 10.4.10 Pour a small (approximately 1 mL) aliquot into small tubes and put them into the instrument. Put the 1.0ppm standard into the first spot.
- 10.4.11 Check to see that all the reagent wedges are in the correct spots and that there are sufficient reaction segments.
- 10.4.12 Save the sequence and double click on "run"; check the boxes for curve analysis.
- 10.4.13 Once the instrument is done analyzing the run, check and approve the results in the Data Review section. Then, change the Blank and LCS names to be whatever they are on the batch sheet (ex: WG123456-1) and save the run in the "out" folder.
- 10.4.14 Open the "SEAL on bowzer" folder and drop the run into it from the "out" folder. This saves the data to LIMS.

10.5 Continuing Calibration

The method blank and LCS are used as the CCB/CCV and should be read after every ten samples and at the end of the batch. Recovery for the CCV must be between 85-115% of the true value. Recovery for the CCB must be between the RL and its negative, (i.e: within -.01mg/L and .01mg/L for waters).

10.5 Preventative Maintenance

The Spectrophotometers are calibrated on a semi-annual basis by an instrument service company. Certificates are kept on file.

11. Data Evaluation, Calculations and Reporting

Calculate the concentration value of the sample directly from the standard curve. (Section 10.2).

$$\text{mg P}_{\text{Total}}/\text{L} = \frac{\text{absorbance} - \text{y-intercept}}{\text{slope}} \times \text{Dilution factor}$$

If samples were filtered prior to preservation, report as mg P_{Dissolved} / L.

For soil samples, convert results to mg/kg, by multiplying result in mg/l by extraction volume and dividing by weight. All results must be reported based on dry weight.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedances or improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CCV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan
SOP/1732 MDL/LOD/LOQ Generation
SOP/1739 IDC/DOC Generation
SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.