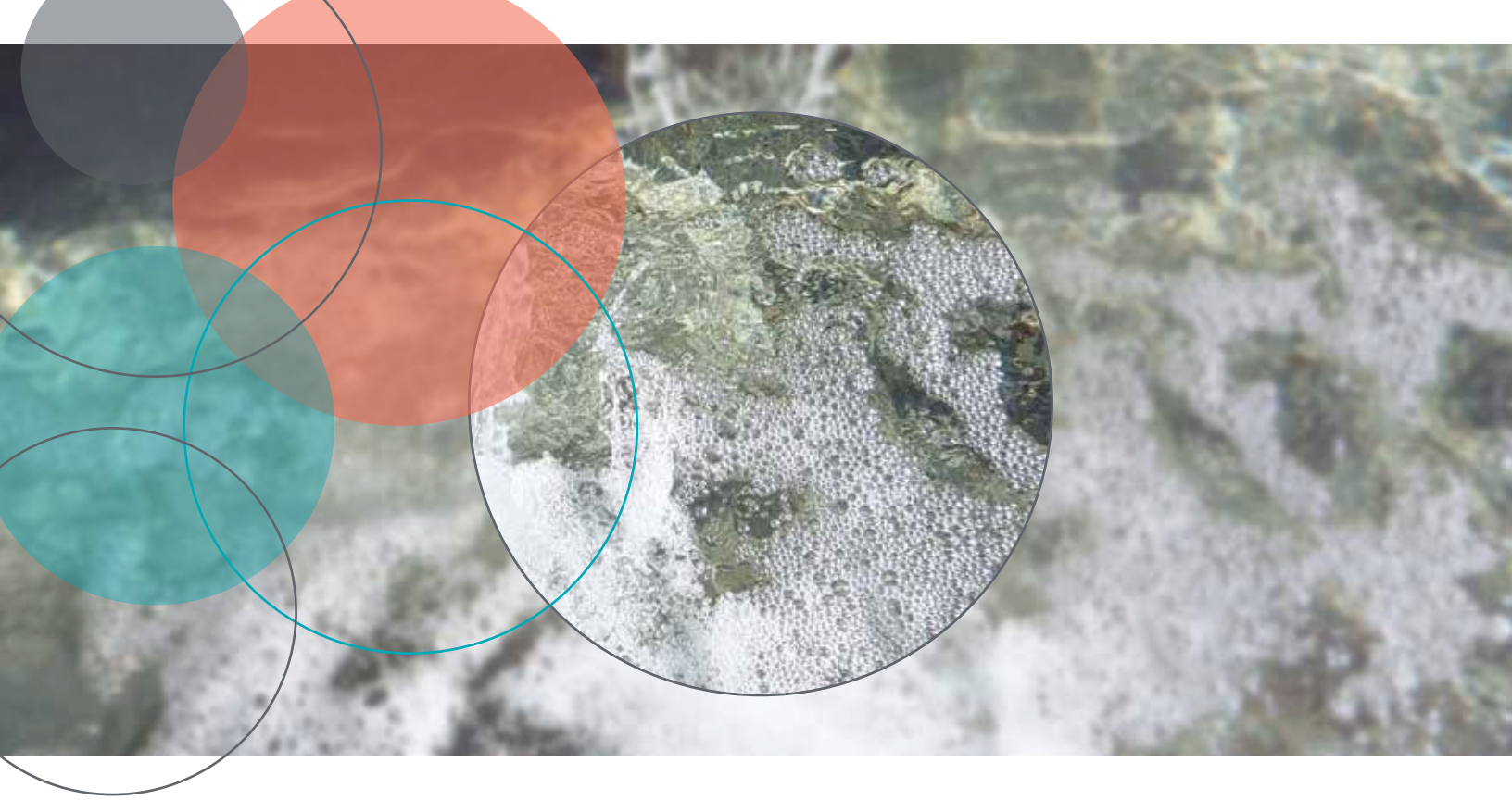

Measuring Low Phosphorus Concentrations

APRIL 2019

The 2008 *Low Phosphorus Concentration Measurement Compendium* was written to identify knowledge gaps to be addressed by the Nutrient Removal Challenge. The 2008 compendium included a number of questions and challenges related to the ability and accuracy to measure phosphorus species. This 2019 compendium revision contains a summary of the findings presented in reports and documents generated by the Nutrient Removal Challenge researchers and contributors.

THE Water Research FOUNDATION

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The findings from the Nutrient Removal Challenge were incorporated into this 2019 edition by:

Bryce Figdore
HDR Inc.

JB Neethling
HDR Inc.

David Stensel
University of Washington

The following individuals contributed to earlier versions of this compendium:

Alan Bingham
Clean Water Services

Lazaro Eleuterio
HDR Inc.

JB Neethling
HDR Inc.

Scott Smith,
Laurier University,
Canada

The reader is also referred to two WRF projects related to phosphorus measurement. Eleuterio and Neethling (2009) sent samples containing less than 20 µg/L phosphorus to ten different laboratories for analysis of reactive phosphorus (orthophosphate) and total phosphorus. Results showed highly variable results, particularly for lower concentrations and total phosphorus measurements in treated effluent. Best practices for phosphorus analytical measurements and the impact of reaction time, path length and other analytical metrics on the accuracy of measurements are described by Smith (2015).

Please use the following citation for this document:

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1199 North Fairfax Street, Suite 900
Alexandria, VA 22314-1445

6666 West Quincy Avenue
Denver, CO 80235-3098

info@waterrf.org
www.waterrf.org

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WHAT ARE THE PHOSPHORUS SPECIES IN WASTEWATER?

Phosphorus appears in wastewater in many forms, as indicated in Table 1. Many of the phosphorus forms change in wastewater treatment due to chemical and biological actions, some intentional but many unintentional. The most dominant phosphorus species in wastewater is orthophosphate, a weak acid, and the species used in biological metabolism. Other forms of phosphorus are converted to orthophosphate and are then available to support biological growth.

Table 1 - Phosphorus Species in Water

Category	Species	Solid/Liquid	Comment
I. Orthophosphate	PO_4^{3-}, HPO_4^{2-}, H_2PO_{4-}, H_3PO_4	Liquid	Weak acid (pKa's ~ 2.15, 7.2, 12.35), most dominant form, reactive.
II. Polyphosphates/ Condensed Phosphate	Pyrophosphate, tripoly-phosphate, metaphosphate	Liquid	Complex large molecule. Precipitate in condensed form or hydrolysis to orthophosphate. Hydrolysis rates high in presence of microorganisms (sludge).
III. Organic Phosphorus	Cell material, intracellular phosphate, intracellular granules	Solid or Soluble	Linked to biological growth, enhanced biological phosphorus removal, etc. Phosphorus in organic compounds.
IV. Chemical Phosphorus	Phosphorus precipitants, typically Fe, Al, Ca. Struvite and other compounds also contribute to chemical phosphorus.	Solid	Particle size important. Reactions slower and could change with time.
V. Adsorbed Phosphorus	Adsorption to sorbant or to metal hydroxides, form complex	Solid	Could be considered a chemical phosphorus species.

Source: Neethling et al. 2007

Table 1, above, also indicates whether the species are soluble or particulate. Particulate fractions are removable in treatment using solids separation processes such as settlers, conventional filters, membrane processes, and others. Soluble phosphorus must be converted into a particulate form in order to be removed from the wastewater.

Differentiating between soluble and particulate species is an operational and analytical challenge (see What Is the Operational Definition of Soluble/Filterable Phosphorus?). Colloidal particles can pass through a filter and be measured as a soluble species.

What Is Phosphorus Speciation?

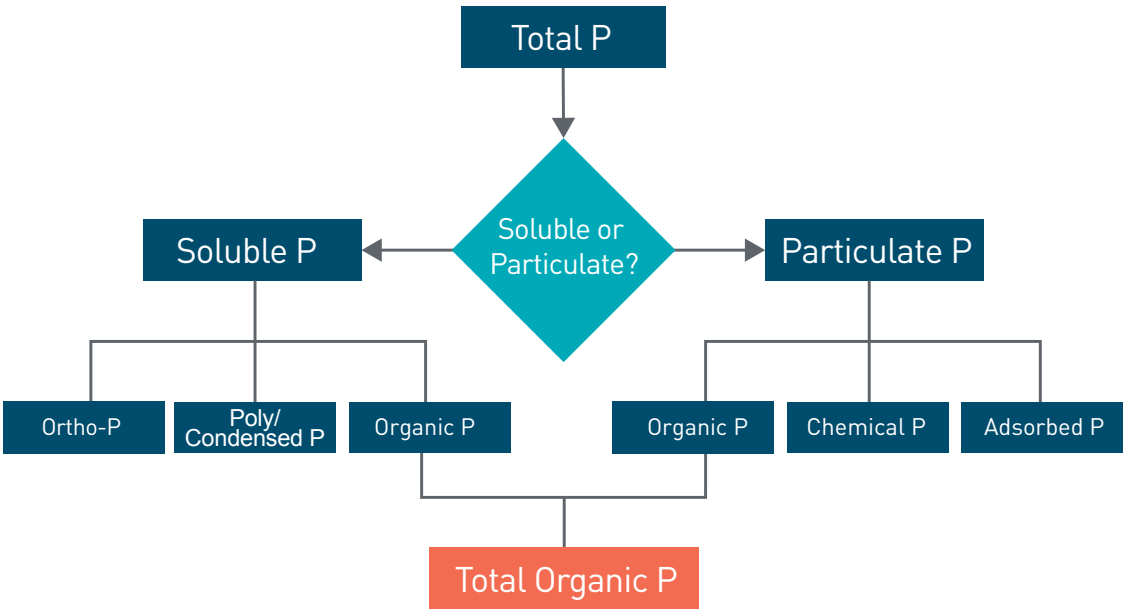
Phosphorus appears in wastewater in many different forms (see above). Standard measurements for phosphorus typically detect several of the species present in the sample. Phosphorus speciation

is the categorization of the chemical and physical forms of phosphorus in a given sample. These forms of phosphorus can be speciated in terms of "boxes" such as soluble and particulate, where soluble (filterable) is the total phosphorus passing through a filter and particulate (non-filterable) is retained by the filter and determined as difference between total phosphorus analyzed on an unfiltered sample and the soluble concentration.

How Is Phosphorus Speciation Determined Analytically?

Figure 1 shows a speciation diagram for the common phosphorus species. The challenge with this speciation diagram is that the analytical methods to measure the species are not readily available.

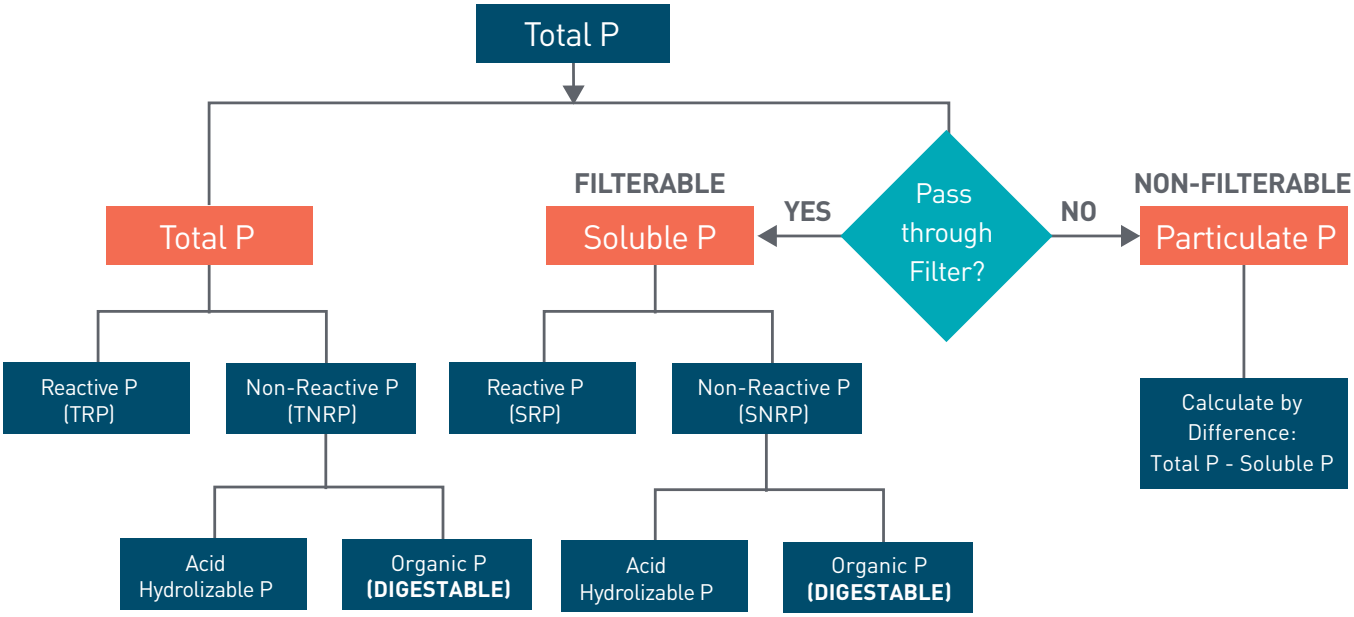
Figure 1 - Phosphorus Speciation Diagram



A practical approach to speciation is to categorize the species in terms of the analytical method used to detect the phosphorus. Figure 2 shows a speciation diagram based on the analytical method used. The species are categorized in terms of:

- **Soluble and Particulate Species.** Soluble (filterable) and particulate (non-filterable) have been used as an industry standard to designate solutes (and fine particles) passing through a filter or particles being retained by a filter (see What Is the Operational Definition of Soluble/Filterable Phosphorus?).
- **Reactive and Non-reactive Phosphorus.** Based on the species reactivity towards the analyte, it will measure in a test as reactive (or not). The reactive phosphorus test is intended to measure orthophosphate, but other compounds may react and be included in the analysis. The soluble reactive phosphorus is typically used as a measure of the orthophosphate in the sample.
- **Hydrolysis by Weak Acid or Digestion.** This distinction is not commonly used. It will distinguish between poly/condensed phosphates and complex phosphates such as organic phosphorus.

Figure 2 - Practical Speciation Diagram for Phosphorus



The analytical approach allows detailed speciation. Neethling et al. (2007) presented a method for separating the phosphorus species in terms of the analytical method, as shown in Table 2.

Table 2 - Typical Analytical Methods for Phosphorus Measurement

Fraction	Filter?	Acid?	Heat?	I	II	III	IV	V
A. Total Reactive Phosphorus	N	N	N	Y	N?	N	?	?
B. Total Acid Hydrolyzable Phosphorus[a]	N	Y	N	N[c]	Y	N?	Y	Y?
C. Total Phosphorus	N	Y	Y	Y	Y	Y	Y	Y
D. Total Organic Phosphorus	Calculate as D = C-B-A							
E. Soluble Reactive Phosphorus	Y	N	N	Y	N?	N	?	?
F. Soluble Acid Hydrolyzable Phosphorus[b]	Y	Y	N	N[c]	Y	N?	?	?
G. Soluble Total Phosphorus	Y	Y	Y	Y	Y	N	?	?
H. Soluble Organic Phosphorus	Calculate as H = G-E-F							
Particulate Phosphorus	Calculate as Difference of Total and Soluble							

Note:
I - Phosphate
II - Polyphosphate / Condensed Phosphate
III - Organic Phosphorus
IV - Chemical Phosphorus
V - Adsorbed Phosphorus

a. Calculated from measurement that includes (A)+(B)
b. Calculated from measurement that includes (E)+(F)
c. Subtracted from measurement
Y = Yes
N = No
P = Partial

How Is a Detailed Phosphorus Speciation Determined Analytically?

More detailed analyses of fractionation schemes are possible, as well as the analysis of specific phosphorus species using ion chromatography and phosphorus nuclear magnetic resonance (NMR) spectroscopy.

Uhlmann et al. (1990) present an alternate fractionation technique compared to the standard methods. By this method, the following phosphate fractions are defined: a) redox-sensitive phosphate, mainly bound to HFO; b) phosphate adsorbed to surfaces, exchangeable against OH-, and alkali-soluble phosphate; c) phosphate bound to CaCO3, MgCO3 and in apatite; and (d) a fraction which mainly composed of polyphosphate. In the work by Uhlmann et al., unique spectroscopic 31P NMR signatures for polyphosphates, as well as orthophosphate, are presented. Jing et al. (1992) also utilized phosphorus NMR to distinguish orthophosphate and polyphosphates. In this case, phosphorus NMR is used before and after sonification of bacteria to indicate cytoplasmic versus external fractions.

Spivakov et al. (1999) present a comprehensive analysis of phosphorus speciation techniques for both solid and liquid phases. Separation techniques are presented, as well as a discussion of phosphorus NMR and ion chromatography. Ion chromatographic analysis of phosphorus species is also detailed in Ruiz-Calero and Galcaran (2005).

WHAT IS THE OPERATIONAL DEFINITION OF SOLUBLE/ FILTERABLE PHOSPHORUS?

The amount of particulate (non-filterable) phosphorus or soluble (filterable) species depends on the type and size of filter used in the analytical procedure. The most commonly used filter is a membrane filter with 0.45 um pore openings. While this is small enough to exclude all bacteria, colloidal particles will still pass through the filter—meaning that very small chemical precipitant particles will pass through the filter paper and be measured as filterable or "soluble."

The terms "filterable" (soluble) and "non-filterable" (particulate) may seem counter-intuitive. Following ASTM D5907-18 "Standard Test Methods for Filterable Matter (Total Dissolved Solids) and Nonfilterable Matter (Total Suspended Solids) in Water," we accept the definition of soluble or dissolved as "filterable" and particulate species as "non-filterable." In this case filterable (soluble) refers to the fact that the compound is able to pass through the filter, as opposed to be retained by the filter. Non-filterable (particulate) are retained by the filter.

The term "soluble" is preferred over "dissolved" or "filterable" and the term "particulate" is preferred over "non-filterable."

What Are the Artifacts Associated with Filtration?

This is an area of active research. In addition to the obvious pore size, there are other factors affecting the separation process. These include the following:

- Selection of membrane pore filter or depth filter
- Passage of colloidal particles
- Flocculation during the test could increase particle size and improve removal
- Chemical solids captured on the filter paper can serve to adsorb more phosphate from the sample during the test.
- Biological solids from an EBPR (Enhanced Biological Phosphorus Removal) processes retained on the filter paper could release phosphate during the filtration process and increase the soluble phosphorus concentration.

What Is an Appropriate Filter Size?

The most commonly used operational definition of soluble phosphorus is particles that will pass 0.45 um pore filter. For clarity, the filter pore size should always be stated when reporting soluble or filterable phosphorus.

ORTHOPHOSPHATE ANALYTICAL METHODS & DETECTION LIMITS

The methods most commonly used for the determination of total phosphorus and reactive (ortho-) phosphorus and their respective detection limits are shown in Table 3. The circles indicate whether the method is designed exclusively for total phosphorus, orthophosphorus, or for both. These methods comprise two procedural steps. First, the phosphorus form of interest is converted to dissolved orthophosphate. Second, the dissolved orthophosphate is determined colorimetrically.

Even though a number of analytical tests exist for the measurement of phosphorus, the ascorbic acid method is the most commonly used test. In this method, the molybdate reagent reacts with orthophosphate producing phosphomolybdic acid, which forms the blue colored molybdenum upon reduction with ascorbic acid. While the compound appears blue, the peak absorbance at 885 nm is in the infrared region. The method detects phosphate concentrations of 5 to 1300 µg/L, with a cuvette pathlength of 1 cm (Eaton et al. 2005).

Table 3 - Summary of Orthophosphate Analytical Methods

Method	TP	OP	MDL (µg/L)	Reference
Vanadomolybdophosphoric Acid	●	●	200	Eaton et al. 2005
Stannous Chloride	●	●	3	Eaton et al. 2005
Ascorbic Acid:				Eaton et al. 2005
SM 4500-PE	●	●	10	Eaton et al. 2005
SM 4500-PF		●	<1	Eaton et al. 2005
EPA 365.1		●	10	EPA
EPA 365.2	●	●	10	EPA
EPA 365.3	●	●	10	EPA
EPA 365.4	●		10	EPA
D 515 – 88 A		●	10	ASTM Standards
D 515 – 88 B	●		100	ASTM Standards
Flow Injection Analysis Orthophosphate		●	0.7	Eaton et al. 2005
Manual Digestion and Flow Injection Analysis for TP	●		2	Eaton et al. 2005
In-line UV/Persulfate Digestion and Flow Injection Analysis	●	●	7	Eaton et al. 2005
Persulfate Method for Determination of TP	●		2	Eaton et al. 2005
I-2601-90 (Automated-segmented Flow)		●	10	USGS
Kjeldahl Digestion Method	●		10	USGS

Total phosphorus and total soluble phosphorus require a digestion step prior to the measurement of the orthophosphate form. The three digestion methods most commonly used during total phosphorus analysis include perchloric acid, nitric acid-sulfuric acid, and persulfate oxidation methods. Phosphorus occurs in association with organic matter, which must be effectively oxidized to release phosphorus as orthophosphate. The perchloric acid method was exclusively designed for sample matrices difficult to digest, such as soil and sediments. This method has been considered very time consuming and requires special facilities and safety precautions.

Even though the acid-sulfuric acid method is widely recommended for most sample matrices, the persulfate oxidation method is by far the simplest method. During the digestion, organo-phosphorus compounds and polyphosphates are oxidized to orthophosphate.

How Are Orthophosphate Samples Properly Preserved for Low-level Determination?

According to EPA (2007), complete stability for water constituent cannot be achieved. It means that after removing the water from the parent source, there is no guarantee that the constituents will remain in the original form. At best, preservation methods are designed to delay chemical and biological changes that certainly continue after sampling. Ideally, samples should be analyzed immediately after collection. The holding times listed in Table 4 are the maximum periods of time that samples may be held before analysis and still be considered valid.

Table 4 - Preservation Methods for Various Phosphorus Analytical Methods

Phosphorus	Vol. req. (mL)	Container	Preservative	Holding Time
Ortho-P	50	Glass	Cool at ≤ 6°C	48 hours
Dissolved OP	50	Glass	Filter on Site Immediately After Sampling, Cool at ≤ 6°C	48 hours
Hydrolyzables	50	Glass	Cool at ≤ 6°C, Acidify (Sulfuric Acid, pH ≈ 2)	28 days
Total Phosphorus	50	Glass	Cool at ≤ 6°C, Acidify (Sulfuric Acid, pH ≈ 2)	28 days
Total Dissolved	50	Glass	Filter on Site Immediately After Sampling, Cool at ≤ 6°C, Acidify (Sulfuric Acid, pH ≈ 2)	24 hours

U.S. Environmental Protection Agency. 2007. Federal Register/ Vol. 72, No. 57/ Guidelines Establishing Test Procedures for the Analysis of Pollutants, Analytical Methods for Biological Pollutants in Wastewater and Sewage Sludge, Final Rule.
Note: Some laboratories require larger sample volumes.

Define the Instrument Detection Limit (IDL)

The instrument detection limit (IDL) is the smallest signal above baseline noise that an analytical instrument can detect with a 99 percent confidence level. The IDL is determined from the analysis of a spiked deionized water matrix. The average of the standard deviations of seven non-consecutive measurements performed on each of three non-consecutive days on a spike deionized water multiplied by 3.14 equals the instrument detection limit (Eaton et al. 2005).

Define the Method Detection Limit (MDL)

The method detection limit (MDL) is the minimum concentration of an analyte that can be measured and reported at a 99 percent confidence. It is also called the minimum detection limit. It must be determined using a water sample prepared at a concentration of two to three times the estimated instrument detection level (Eaton et al. 2005).

Define the Minimum Reporting Level (MRL)

The minimum reporting level (MRL) is defined as the minimum signal level required to quantify an analyte at a desired confidence level (95 percent). The MRL is computed by multiplying the MDL by 3.18 and rounding to a routinely usable value (USGS 1999).

Define the Practical Quantification Limits (PQL)

The practical quantification limit (PQL) represents a practical and routinely achievable limit with a high degree of certainty (> 99.9 percent confidence) in the results. The PQL is determined simply as about 5 times the MDL (Eaton et al. 2005).

Define the Lower Level of Detection (LLD)

The Lower Level of Detection is defined as the amount of the analyte that generates a signal sufficiently large that 99 percent of the trials with that amount will reproduce a detectable signal (Easton 2005). The LLD can be determined by multiplying the concentration of the analyte (no greater than five times the IDL) by the standard deviation (Eaton et al. 2005).

How Is the Method Detection Limit (MDL) Determined?

Prior to analyzing total and ortho-phosphorus, instrument detection, method detection, minimum reporting, practical quantification, and lower level of detection limits must be determined.

The method detection limit (MDL) (Define the Method Detection Limit) is computed by taking at least seven replicate aliquots of a water sample and processing them through the complete

analytical method. The aliquots comprise spiked deionized water matrix. The MDL is computed according to the following equation.

$$MDL = SD \times t_{0.99}$$

where: $SD = \{ \sum_{i=1}^{n} (xi - X)^2 / (n-1) \}^{1/2}$

SD = standard deviation of the replicate analyses

$t_{0.99}$ = t-distribution table value for 99 percent confidence level and a standard deviation computed with n-1 degree of freedom. For seven replicates, t is found to be 3.143. (See Table 5.)

xi = spiking replicates concentration (µg/L) (i = 1 . . . n) (n = 7)

X = the mean of spiking concentrations (µg/L).

Table 5 - T-Distribution Values

Number of Replicates	Degree of Freedom (n-1)	T (n-1, 99%)
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.002
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
∞	∞	2.326

Source: Wisconsin Department of Natural Resources 1996. PUBL-TS-056-96

What Are the Complications Associated with Low Phosphorus Measurement in Wastewater Effluent Samples?

Several substances can interfere with the phosphorus analysis in wastewater effluent samples. For instance, arsenate, silica, barium, lead, sodium chloride and humic substances are constituents that can interfere with phosphorus measurement. Water sample turbidity may also affect phosphorus measurement at low levels. It is important to carefully observe the physical characteristics of water samples. The phosphorus methods are highly sensitive to trace amounts of contamination commonly present in tap water, on fingers, in soap and some detergents, and in buffer solutions and other reagents.

Glassware must be cleaned thoroughly, and maximum cleanliness in the field collection and in the laboratory must be observed. The glassware must be soaked with a phosphate-free soap, rinsed at least five times with deionized water, and three times with hot dilute hydrochloric acid (HCl) followed by three rinses with nitric acid. It is also very important to run distilled water blanks and standards, and keep records of the absorbance in order to detect changes in distilled water quality or reagents.

Another challenge associated with low phosphorus analysis is the correction of the interferences. A good laboratory QA/QC protocol is fundamental; however, what is done when the substances that interfere with phosphorus analysis are already in the parent water source? For instance, arsenate ion is isomorphic with phosphate ion and forms similar molybdate complexes. To minimize arsenate interference on low phosphorus analysis, the reduction of arsenate to arsenite with thiosulfate is appropriate since arsenite does not form molydate complexes.

LABORATORY CERTIFICATION

An accredited laboratory is a laboratory that has proficiency or competence in determining the concentration of the analyte of interest accurately at the levels established by the accreditation authority. The laboratory accreditation process and the accrediting authorities are as follows:

- **NELAC/NELAP - National Environmental Laboratory Accreditation Conference**
- **A2LA – American Association for Laboratory Accreditation**
- **State and Federal Organizations/Agencies**

NELAC/NELAP (National Environmental Laboratory Accreditation Conference)

The National Environmental Laboratory Accreditation Conference (NELAC) is an association of states and federal agencies, created to establish and promote acceptable performance standards for the operation of environmental laboratories (NELAC 2003). The standards include both analytical testing of environmental samples and the laboratory accreditation process. The National Environmental Laboratory Accreditation Program (NELAP) implements the NELAC standards under the supervision of the EPA. The specific objectives of the NELAP, include: a) to evaluate and approve NELAC standards implementation; b) to establish and maintain national database on environmental laboratories; c) to report to NELAC on the evaluation of the conformance of state and federal accreditation program activities; d) to report to NELAC on the evaluations of proficiency testing sample providers and assessor training program; and e) to approve supplemental accreditation requirements.

Accreditation is mutually recognized by state and federal accrediting authorities that meet all NELAC requirements. These authorities use NELAC standards as a reference to assess the qualifications of the laboratories applying for initial and continuing NELAC accreditation, review and approve applications, evaluate the results on proficiency testing samples, and enforce laws and rules regarding accreditation (NELAC 2003). Prior to NELAP initial accreditation and to maintain continuing accreditation, laboratories are required to meet all EPA regulatory requirements, including quality assurance/quality control protocols. Laboratories must also meet the general requirements of the NELAP Quality System.

NELAC accrediting authorities can be either primary or secondary. A primary accrediting authority ensures directly that the laboratories are in conformance with the NELAC standards. A secondary accrediting authority accepts the accreditation of a primary authority. Currently, NELAC has 13 primary recognized accrediting authorities located in 12 states (California, Florida, New York, Illinois, Louisiana, New Hampshire, New Jersey, Oregon, Pennsylvania, Texas, and Utah). For instance, the EPA Division of Laboratories, QA Section in Illinois, is a NELAC-recognized accrediting authority.

Table 6 - NELAC Recognized Accrediting Authorities	
State	**Recognized Accrediting Authority**
California	Environmental Laboratory Accreditation Program
Florida	Florida Department of Health, Bureau of Laboratories
Illinois	EPA, Div. of Lab., QA Section
Kansas	KS Department of Health & Environment
Louisiana	LA Department of Health & Hospitals
Louisiana	LA Department of Environmental Quality
New Hampshire	NH Environmental Lab. Accreditation Program
New Jersey	Department of Environmental Protection
New York	NY State Department of Health
Oregon	Oregon Health Division
Pennsylvania	Bureau of Labs, Department of Environmental Prot.
Texas	Texas Comm. on Environmental Quality
Utah	Utah Department of Health

Source: U.S. Environmental Protection Agency 2007. (www.nemc.us)

The accreditation process includes a review of personnel qualifications, on-site assessment proficiency testing, and quality assurance/quality control standards. The major requirements for the personnel qualifications are: a) the person in charge of the laboratory must be a technical director with a bachelor's degree in chemical, environmental, biological sciences, physical sciences or engineering, with at least 24 college semester credit hours in chemistry, and b) he or she must have a minimum of two years of experience in environmental analysis of organic and inorganic analytes for which the laboratory requests or maintains accreditation (NELAC 2003).

The on-site assessment consists of all the fields of accreditation and methods for which the laboratory wants to obtain accreditation. The on-site assessment must be performed at least once every two years and is considered by NELAC as the primary means to determine a laboratory's capabilities and qualifications. During the on-site assessment, information regarding personnel and a laboratory's physical condition is collected and evaluated (NELAC 2003).

Laboratories interested in obtaining and maintaining accreditation have to perform analyses of proficiency testing samples twice per year for each field of accreditation for which it has applied for accreditation and for which it is currently accredited. For total phosphorus and ortho-phosphorus analysis, the minimum concentration required by the NELAP proficiency test is 340 µg/L. **This concentration is too high when low phosphorus measurements (at 5 µg/L level) are required.** Finally, laboratories are required to ensure consistency and promote the use of quality assurance/quality control standards to generate quality data.

U.S. Geological Survey (USGS) laboratories are accredited by the NELAP. USGS does not provide accreditation to other laboratories. However, since 1962, USGS has conducted an inter-laboratory comparison study that occurs twice per year. The major objective of this study is to ensure quality control of environmental samples analysis. The study provides a wide spectrum of standard reference samples (SRS) prepared with water from natural streams. Water samples are spiked with known concentrations of the analyte of interest and are, thus, analyzed by the laboratories enrolled in the USGS program. The results of SRS analysis are sent to USGS with the purpose of evaluating the performance of the enrolled laboratories, identifying analytical problems and ascertaining accuracy and precision of the analytical methods. To date, approximately 250 USGS and non-USGS laboratories participate in the SRS study. The lowest concentration of total phosphorus and ortho-phosphorus in the SRS ranges from 10 – 80 µg/L and 10 – 70 µg/L, respectively (https://qsb.usgs.gov/srs_study/).

American Association for Laboratory Accreditation (A2LA)

The American Association for Laboratory Accreditation (A2LA) is a non-governmental organization that recognizes the competence of a laboratory to perform a wide range of testing in the Biological, Environmental, Chemical and Geotechnical fields (A2LA 2007a). Other fields of accreditation include Mechanical, Thermal and Electrical. In contrast with NELAC, A2LA does not accredit or review accreditation of a laboratory that fails to meet its requirements. The A2LA accreditation requirements are the ISO/IEC 17025:2005 General Procedures for the Competence of Testing and Calibration Laboratories (A2LA 2007b).

ISO/IEC 17025 states that laboratories seeking accreditation must have quality control procedures for monitoring the validity of tests and calibrations carried out. Laboratories are required to pass the proficiency testing for each field of accreditation for which it has applied for accreditation and for which it is currently accredited (A2LA 2007a,b). According to ISO/IEC 17025 general requirements, the results of the proficiency testing indicate the laboratory competence and constitute an integral portion of the on-site assessment and accreditation process. Proficiency tests must have been performed by personnel trained and qualified for the relevant tests. On-site assessment indicates whether the laboratories comply with the A2LA requirements for accreditation and can proficiently perform the tests for which the accreditation is desired (A2LA 2007a,b).

State and Federal Organizations/Agencies

Some State and Federal Agencies may not follow either the NELAC nor the A2LA standards. They tend to follow the EPA procedures, which focus primarily on the quality of the analyses, instead of the NELAC and A2LA accreditations that focus on the laboratory management and quality assurance. They establish their own requirements and constitute accreditation authorities. A number of states certify laboratories for the analyses of Drinking Water (Safe Drinking Water Act), Wastewater (Clean Water Act), and Hazardous Waste. These states include Idaho, Washington, Nevada, California, Arizona, Colorado, Wyoming and Florida. For instance, in Washington and Nevada, the Department of Ecology and Nevada Department of Environmental Protection (NDEP), respectively, are the laboratory accreditation authority.

How Do I Find a Certified Laboratory?

In order to identify an accredited laboratory, contact the NELAC, A2LA, state or federal agency located in the state where the laboratory is located. If the state or federal agency does not constitute a laboratory accreditation authority, it may be capable of directing you to an accreditation authority in the state where the laboratory is located. For example, NELAC published a list of accredited laboratories nationwide on the internet (https://nelac-institute.org/content/NELAP/accred-bodies.php).

RESEARCH NEEDS

There remains uncertainty as to the species that are detected using the different analytical techniques. While orthophosphate is known to be "reactive," the degree of reactivity of poly-phosphates and condensed phosphates are not established. There is also no clear method for measuring the organic phosphate content because it is calculated based on other measurements. No standard filter (type or pore size) has been selected. Finally, the presence of small particles that pass the filter makes it impossible to differentiate between chemical phosphorus colloids and soluble species.

Research at Laurier University (Smith 2008) is showing that filterable phosphate depends on the volume filtered as well as the specific brand/type of filter used.

The artifacts listed above need to be addressed and culminate in a standard procedure to attain reproducible and reliable results. Research at Laurier (Smith 2008) shows that nano-sized iron colloids containing phosphate initially pass through a filter but begin to remove phosphate from the sample as they accumulate on the inside of a filter.

Measurements from various laboratories for phosphorus concentrations less than 50 µg/L have shown a large variability and have proven to be a challenge. While laboratories follow standard procedures, some variability in results still exist. Variations above 100 percent are not uncommon. A new standard procedure for phosphorus analysis is needed to eliminate this variability.

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