CENTER FOR DRUG EVALUATION AND RESEARCH

APPLICATION NUMBER:

205109Orig1s000

ENVIRONMENTAL ASSESSMENT
CLAIM FOR CATEGORICAL EXCLUSION OF NEED TO FILE AN ENVIRONMENTAL ASSESSMENT

Date: 13 November 2012
Version Number: 1.0
Invented Name: [Redacted] (Company code: PA21 drug product)
Active Substance: Mixture of polynuclear iron(III)-oxyhydroxide, sucrose and starches (PA21 drug substance)
ATC Code: V03AE05 (proposed)
Applicant: Vifor Fresenius Medical Care Renal Pharma France
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Reference ID: 3422105
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LIST OF ABBREVIATIONS

ESRD  End-stage renal disease
NDA   New drug application
pn-FeOOH Polynuclear iron(III)-oxyhydroxide
US    United States
1. **Claim for Categorical Exclusion of Need to File an Environmental Risk Assessment**

1.1 **Claim for Categorical Exclusion for Substance that Occurs Naturally in the Environment**

In accordance with 21 CFR § 2.5.31(c), Vifor Fresenius Medical Care Renal Pharma France claims that approval of this new drug application (NDA) qualifies for categorical exclusion of the need to file an environmental assessment, since the active moiety, polynuclear iron(III)-oxyhydroxide (pn-FeOOH), occurs naturally in the environment and the disposal from human use upon NDA approval will not significantly alter the concentration or distribution of iron, its metabolites or degradation products in the environment. To the best of the applicant’s knowledge, no extraordinary circumstances exist that may significantly affect the quality of the human environment.

1.2 **Background Information on PA21**

Hyperphosphatemia is a common and serious complication in patients with chronic kidney disease, particularly those in end-stage renal disease (ESRD) requiring dialysis. Vifor Pharma is developing a new oral phosphate binder, PA21, for therapeutic use in the control of serum phosphorus levels in patients with ESRD.

PA21 acts by binding the dietary phosphate in the gastrointestinal tract, preventing its uptake into the blood, thereby reducing the serum level of phosphorus.

PA21 drug substance is a mixture of pn-FeOOH, sucrose and starches. It is a dry powder formulation containing approximately [redacted] of iron (equivalent to approximately [redacted] of iron(III)-oxyhydroxide (FeOOH)), [redacted] of sucrose, [redacted] of starches, [redacted] The active component in PA21 drug substance is the pn-FeOOH moiety. The PA21 drug product is presented as a chewable tablet with a content of 500 mg iron, corresponding to approximately 2,500 mg drug substance. The tablet also contains silica, woodberry flavor, neohesperidin dihydrochalcone, and magnesium stearate.

1.3 **Occurrence of Iron and Estimation of Exposure of PA21 in the Environment**

Iron is a universal constituent of the environment, constituting approximately 5% of the earth’s crust. It is found widely in soils and ores in the form of various salts, the most abundant being iron oxide. Iron hydroxide is mainly linked to the formation of rust and is practically insoluble in water. Iron is essential to almost all living organisms, from microorganisms to humans. Remarkably, chemical precipitation with, for example, iron salts, is widely used to remove phosphorus at wastewater treatment plants [1].
The estimation of exposure is limited to the pn-FeOOH moiety of the PA21 drug substance, as the carbohydrates (sucrose and starches) are dietary components, which are metabolized and are not considered to result in a significant risk to the environment.

The highest quantity of the drug substance expected to be produced for direct use in the corresponding to approximately \( \text{(b)(4)} \) of pn-FeOOH (approximately \( \text{(b)(4)} \) or approximately \( \text{(b)(4)} \) of iron.

Considering the 5% iron in the earth’s crust, \( \text{(b)(4)} \) iron will be present in the top of a field of \( \text{(b)(4)} \) which is negligible compared to the overall surface of the US.

Thus, the concentration of iron in the environment will not be significantly increased following the use of PA21.

1.4 Conclusion

PA21 drug substance contains iron in the form of pn-FeOOH, as well as sucrose and starches. These ingredients and their degradation products are unlikely to represent a risk to the environment and it is therefore concluded that no additional experimental data are required.
2. REFERENCES

Nutrient Control Design Manual

by

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Scientific, Technical, Research, Engineering, and Modeling Support (STREAMS)
Task Order 68

Contract No. EP-C-05-058

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3. Principles of Phosphorus Removal by Chemical Addition

Chapter 3 covers:

3.1 Introduction
3.2 Available Forms of Metal Salts and Lime
3.3 Equations and Stoichiometry
3.4 Solids Separation Processes
3.5 Effects on Sludge Production and Handling
3.6 Two Factors that May Limit the Ability of Plants to Achieve Very Low Effluent Levels
3.7 References

3.1 Introduction

Chemical precipitation is widely used to remove phosphorus at wastewater treatment plants (WWTPs). Chemicals such as metal salts or lime can be added to primary, secondary, or tertiary processes or at multiple locations within the plant. Solids removal following chemical precipitation can be conventional, such as primary or secondary clarification, or advanced, such as tertiary filtration or alternative technologies. Chemical precipitation can be used alone or in conjunction with biological phosphorus removal (BPR) to reduce the costs associated with the chemical dose and sludge production.

This chapter describes the principles of phosphorus removal by chemical addition. It includes a description of chemicals available, the latest research on the mechanisms of phosphorus removal, and factors affecting performance such as point of application and solids separation. Lastly, it presents information on sludge production and limits of removal. Chapter 9 follows with more detailed recommendations for designing a chemical phosphorus removal treatment system.

3.2 Available Forms of Metal Salts and Lime

Chemicals used for phosphorus precipitation are typically either metal salts or lime. The two most common metal salts are aluminum sulfate (commonly known as alum) and ferric chloride. Sodium aluminate can serve as a source of alum, although it can increase the pH substantially (WEF and ASCE 2009). Various forms of polyaluminum chloride (PAC) can also be used for chemical precipitation. Ferrous sulfate and ferrous chloride, which are available as byproducts of steel-making operations (pickle liquor), are also used. Lime is typically available in solid form as either quicklime (CaO) or hydrated lime Ca(OH)₂. Table 3-1 summarizes the most common chemical precipitants used for phosphorus removal. Chapter 9 provides additional information on chemical properties and guidance for chemical selection.
Table 3-1. Common Chemicals Used for Phosphorus Removal

<table>
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<tr>
<th>Chemical</th>
<th>Formula</th>
<th>Description</th>
<th>Typical Weight Percent in Commercial Solutions²</th>
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<tbody>
<tr>
<td>Aluminum Sulfate (Alum)</td>
<td>$\text{Al}_2\text{(SO}_4\text{)}_3 \cdot 14\text{(H}_2\text{O)}$</td>
<td>Crystallized, dry form or liquid</td>
<td>48%</td>
</tr>
<tr>
<td>Sodium Aluminate</td>
<td>$\text{Na}_2\text{Al}_2\text{O}_4$</td>
<td>Powder or liquid form. Granular thydrate is common commercial form</td>
<td>20%</td>
</tr>
<tr>
<td>Polyaluminum Chloride (PAC)</td>
<td>$\text{Al}<em>n\text{Cl}</em>{3n-m}\text{(OH)}<em>m$ EX: $\text{Al}</em>{12}\text{Cl}_{24}\text{(OH)}_24$</td>
<td>Range in the degree of basicity and aluminum concentration</td>
<td>51%</td>
</tr>
<tr>
<td>Ferric Chloride¹</td>
<td>$\text{FeCl}_3$</td>
<td>Orange-brown aqueous solution</td>
<td>37 – 47%</td>
</tr>
<tr>
<td>Pickle liquor (Ferrous sulfate or Ferrous iron)</td>
<td>$\text{Fe}_2\text{SO}_4 \text{ or Fe}^{2+}$</td>
<td>Green aqueous solution</td>
<td>Varies</td>
</tr>
<tr>
<td>Lime</td>
<td>CaO, Ca(OH)₂</td>
<td>Dry white powder or liquid form as quicklime, CaO, or hydrated lime, $\text{Ca(OH)}_2$</td>
<td>NA</td>
</tr>
</tbody>
</table>

1. *Ferric is also common trade name for FeCl₃ and also 40% FeCl₃ solution
2. Source: WEF and ASCE 2006 Tables 8.6 and 8.9

3.3 Equations and Stoichiometry

This section describes the forms of phosphorus that can be removed by chemical treatment. It follows with a detailed discussion of the chemical reactions among metal salts, lime, and phosphorus including factors affecting removal efficiency.

3.3.1 Removable Phosphorus

Chemical precipitation will remove only the phosphate (i.e., orthophosphate) fraction of total phosphorus in wastewater. Influent phosphate is typically 50 to 80 percent of total phosphorus and generally exists in one of two forms, $\text{H}_2\text{PO}_4^-$ and $\text{HPO}_4^{2-}$, with the first being dominant at pH below 8.3. Polyphosphates will not react with metal salts or lime; however, they will be converted to phosphate during biological treatment¹. Organically bound phosphorus typically makes up the smallest fraction of total influent phosphorus ($1 < \text{mg/L}$). The colloidal and particulate portion will generally be removed during solids separation processes. The soluble organic fraction may either be hydrolyzed into orthophosphate during the treatment process (if biodegradable) or will pass through a WWTP (if non-biodegradable).

See Chapter 2, Section 2.2.2 for additional information on the forms of phosphorus in influent wastewater. See Section 3.8 for a discussion on the implications of recalcitrant phosphorus on achieving low effluent total phosphorus (TP) concentrations.

¹ Colorimetric techniques used to quantify phosphate concentrations give results for "reactive" phosphorus, which is primarily orthophosphate but includes a small condensed phosphate fraction.
3.3.2 Reactions of Metal Salts and Phosphorus

The metal salt dose for chemical phosphorus removal has been recognized as a critical design and economic parameter for chemical treatment systems. In addition to dose, the wastewater characteristics, method of chemical addition, chemical addition feeding point(s), reaction pH, flocculation method, and time after chemical addition are important design and performance issues that can affect the relationship between dose and phosphorus removal efficiency.

The metal salt dose is commonly described in terms of the moles of metal added (Me_dose) per mole of soluble phosphorus in the influent (P_in). The term "stoichiometric dose" is based on 1.0 Me added per Mole of P removed (i.e., 1.0 Me_dose/P_in) which would be the molar ratio requirement for strictly a chemical reaction between aluminum or iron salts to form a M-P04 precipitate as shown in Equations 3-1 and 3-2 (WEF and ASCE 2009, charges omitted).

\[
\text{Al}_2(\text{SO}_4)_{3.14} + 2\text{H}_3(\text{PO}_4) \rightarrow 2\text{Al}(\text{PO}_4) + 3\text{H}_2\text{SO}_4 + 18\text{H}_2\text{O} \quad \text{Eq. 3-1}
\]

\[
\text{FeCl}_3(6\text{H}_2\text{O}) + \text{H}_2\text{PO}_4 + 2\text{HCO}_3 \rightarrow \text{FePO}_4 + 3\text{Cl} + 2\text{CO}_2 + 8\text{H}_2\text{O} \quad \text{Eq. 3-2}
\]

At relatively high effluent P concentrations (above 1.0 mg/L), the stoichiometric relationship for metal salt dose is commonly observed. The molar ratio typically increases well above the stoichiometric ratio as lower effluent phosphorus concentrations are needed. This has commonly been attributed to substantial metal hydroxide formation in addition to metal-phosphorus precipitates (Sedlak, 1991).

Research by Szabó et al. (2008) and Smith et al. (2008) defined a mechanism for phosphorus removal by ferric chloride based on a surface complexation model. Conceptually, the addition of ferric chloride results in the precipitation of hydrous ferric oxide (HFO), which provides surface sites for reaction with phosphorus. The basis of the phosphorus removal mechanism is that phosphate and iron can share an oxygen molecule and that interactions can be represented by the following symbolic reaction (charges omitted) (Smith et al. 2008):

\[
\text{FeOOH} + \text{HOPO}_3 \rightarrow \text{FeOOPO}_3 + \text{H}_2\text{O} \quad \text{Eq. 3-3}
\]

The reactive oxygens are termed "surface sites," and their availability is related to mixing and aging conditions. Under rapid mixing, surface sites are readily available. With slow mixing, however, much of the HFO would form in the absence of phosphorus and result in less efficient phosphorus removal.

As the HFO forms, phosphate is simultaneously removed through (1) co-precipitation of phosphate into the HFO structure, and (2) adsorption of the phosphate onto pre-formed HFO particles (Smith et al. 2008). After the initial HFO formation and phosphorus removal, additional phosphorus can be removed over time by diffusion of phosphorus into the floc. The effect of aging is to reduce reactive sites and HFO phosphorus removal capacity. Additional research is needed to confirm a similar removal mechanism for aluminum.

Chemical Dose and Phosphorus Removal Efficiency

In all observations on chemical removal of phosphorus, the percent phosphorus removal increases and effluent phosphorus concentration decreases as the molar chemical dose for metal salts increases, but the incremental removal diminishes with increased dosages (Sedlak 1991, Szabo et al.)
Smith et al. (2007) found that for typical influent phosphorus concentrations, ferric doses above 1.5 to 2.0 Me_{dose}/P_{in} ratios are sufficient to remove 80 to 98 percent of soluble phosphorus. Reaching very low effluent TP concentrations, i.e. below 0.10 mg/L, requires significantly higher ratios of about 6 or 7 Me_{dose}/P_{in}. A similar dose was reported for 75 to 95 percent phosphorus removal from wastewater using alum (WEF and ASCE 2009). For similar phosphorus removal efficiencies using pre-polymerized salts such as PACl or sodium aluminate, a higher dose is needed. Factors that influence dose and removal efficiency may include pH, alkalinity, competing substances in the wastewater, initial mixing conditions and flocculation.

Mixing at the Dosage Point

Mixing at the dosage point is necessary to ensure that the metal and phosphate molecules react. Mixing intensity can be represented by the velocity gradient, G, in units of second^{-1}. Smith et al. (2007) reported that the reactions of phosphates at the surface of the hydrous ferric oxides depend highly on the mixing conditions. Rapid mixing means that the surface sites are available, whereas with slower mixing, many of the metal oxides would form in the absence of phosphate and render internal oxygen atoms unavailable for binding.

Bench-scale kinetic experiments by Szabo et al. (2008) revealed that a majority of phosphate will react with iron in the first 10 to 20 seconds under ideal mixing conditions (G = 425 second^{-1}). At wastewater plants, mixing at the dosage point is typically poor, with G values ranging from 20 to 100 second^{-1} (Szabó et al. 2008). Szabó et al. recommend that plants attempt to achieve a very high mixing intensity at the dosage point (G values between 200 and 300 second^{-1}). Mixing times at high energy are generally in the range of 10 to 30 seconds.

After the initial rapid kinetics, phosphorus removal can continue with slow reaction kinetics between the phosphate and iron over many hours and even days (Szabo et al. 2008). The slow reaction rate removal is more important for alum or ferric addition to activated sludge basins with solids retention times in days.

Flocculation

After rapid mixing at the dosage point, gentle mixing is needed to form flocs that can be settled or removed through a solids separation process. This is critical for meeting low effluent phosphorus requirements. Often, movement of the wastewater through the treatment plant is sufficient for floc formation (USEPA 2008b). Flocculation can be limited by insufficient time or conditions that disrupt floc formation such as pumping and aeration.

pH and Alkalinity

The highest removal efficiency for chemical precipitation is within a pH range of 5.5 to 7.0 (Szabó et al. 2008). Between pH 7 and 10, phosphorus removal efficiency declines because the surfaces of metal hydroxides are more negatively charged, and soluble iron hydroxides begin to form. At low pH values, the solubility of the precipitant is reduced, and at extremely low pH values, metal hydroxide precipitation is limited. Szabó et al. (2008) reported similar relationships between phosphorus removal efficiency and pH for both alum and ferric chloride.
COD and TSS in Raw Wastewater

The efficiency of metal salt addition in the primary treatment step can be affected by the wastewater characteristics. Based on the results of jar tests using municipal wastewater, Szabo et al. (2008) observed a relationship between organic content and efficiency of phosphorus removal using metal salts. Between chemical oxygen demand (COD) values of 300 to 700 mg/L, the phosphate removal efficiency decreased with increasing COD. Similar results were observed for total suspended solids (TSS), with lower phosphorus removal efficiency at higher TSS concentrations. In addition to reduced efficiency during primary treatment, organic content can reduce the efficiency of metal salt removal of phosphorus in activated sludge reactors. Iron and aluminum ions can react with humic and fulvic acid substances to form insoluble complexes with the metal ions and their mineral oxides, thereby blocking the reactive sites for phosphate precipitation (WERF 2009).

3.3.3 Reactions of Lime with Phosphorus

When lime is added to wastewater, it first reacts with the bicarbonate alkalinity to form calcium carbonate (CaCO₃). As the pH increases to more than 10, excess calcium ions will react with phosphate to precipitate hydroxyapatite [Ca₅(OH)(PO₄)₃] as shown in the reaction below.

\[ 5Ca^{2+} + 4OH^- + 3HPO_4^- \rightarrow Ca_5(OH)(PO_4)_3 + 3H_2O \]  Eq. 3-4

Because it reacts first with alkalinity, the lime dose is essentially independent of the influent phosphorus concentration. Tchobanoglous et al. (2003) estimates the lime dose to typically be 1.4 to 1.6 times the total alkalinity expressed as CaCO₃.

Lime addition can raise the pH to greater than 11. Because activated sludge processes require pH levels below 9, lime cannot be added directly to biological treatment processes.

3.4 Solids Separation Processes

Solids separation methods are critical for determining phosphorus removal efficiency with chemical precipitation as a large portion of the effluent phosphorus is contained in chemical precipitates. Solids separation technologies, such as clarification and filtration are often used in combination to achieve low effluent TP levels. Polymers can be used in addition to the metal salts for phosphorus precipitation to enhance removal for fine particles and colloids.

Gravity separation in primary or secondary clarifiers is a traditional solids separation method at WWTPs. Clarifiers used in chemical precipitation systems differ very little from those employed in conventional treatment, although use of flocculation zones is recommended to provide flocculation time after chemical addition.

For secondary clarification, flocculation can occur in aeration basins or channels preceding clarification. The use of flocculation zones in secondary clarifiers is a recommended practice to allow flexibility in the point of chemical addition and to provide a zone in which direct control can be exercised over velocity gradients to achieve optimum flocculation.
Membrane bioreactors for activated sludge wastewater treatment provide maximum solids separation with effluent turbidity values typically less than 0.30 NTU and non-detect effluent TSS concentrations.

Tertiary filtration following secondary clarification is increasingly being used as a polishing step, with chemical to reduce TSS and to achieve effluent TP concentrations below 0.50 mg/L. The types of filters available include traditional media filters, upflow continuous backwash filters, cloth filters, and membranes. Several patented ballasted high-rate clarifiers (BHRC) using different types of ballast such as recycled sludge, microsand, and magnetic ballast (USEPA 2008b) have been developed in recent years. Examples include DensaDeg®, Actiflo®, and the CoMag™ process. Chapter 6 of this design manual provides an overview of the different tertiary filter technologies and Chapter 11 provides recommendations for design.

3.5 Effects on Sludge Production and Handling

Sludge production and handling is generally considered to be one of the downsides of chemical phosphorus removal. Chemical precipitation methods always produce additional solids due to generation of metal- or calcium- phosphate precipitates and metal hydroxide sludge. The amount of increased sludge production will depend on the location of chemical addition, the chemical dose used, and the constituents present in the wastewater.

The stoichiometric relationships shown below can provide a good first estimate of additional solids production from chemical precipitation (WEF and ASCE 2009). For alum addition the removal of P with Al can be represented by Al₁₀(NO₃)₁₂(OH)₂₄ and the remaining aluminum added will be described by aluminum hydroxide production in accordance with Eq. 3-5. For ferric removal with iron the sludge production from P removal can be estimated as Fe₅(NO₃)₉(OH)₁₈ and the remaining ferric added will be described by ferric hydroxide production in Eq. 3-6.

\[
\text{Al}^{3+} + 3\text{H}_2\text{O} \leftrightarrow \text{Al(OH)}_3 + 3\text{H}^+ \quad \text{Eq. 3-5}
\]

\[
\text{Fe}^{3+} + 3\text{H}_2\text{O} \leftrightarrow \text{Fe(OH)}_3 + 3\text{H}^+ \quad \text{Eq. 3-6}
\]

Typically, the addition of metal salts to the primary clarifier for the purposes of complete phosphorus removal will increase primary treatment sludge production by 50 to 100 percent due to phosphorus and hydroxide precipitates and increased suspended solids removal. In this case the secondary sludge production is lower due to removal of additional TSS and BOD in the primary clarifier. The total overall plant sludge production can be expected to increase by 60 to 70 percent (WEF and ASCE 2009). For metal addition to the secondary treatment process to achieve effluent P concentrations in the range of 0.50 to 1.0 mg/L with the stoichiometric metal salt dose in the range of 2.0, the sludge production may increase by 35 to 45 percent and the overall plant sludge production may increase by 5 to 25 percent (WEF and ASCE 2009). For tertiary applications to achieve effluent P concentrations of less than 0.10 mg/L, the chemical stoichiometric dose can be 2 to 3 times that indicated in the previous sentence for secondary treatment, but the amount of P to be removed is much less, so that the effect on sludge production can be estimated to be increased by 45 to 60 percent for secondary/tertiary treatment and by 10 to 40 percent for the overall plant sludge production. See Chapter 9, Section 9.8.3, for an example calculation of sludge production increase resulting from the addition of metal salts.
Lime typically produces a much higher sludge volume compared to metal salts because of its reaction with natural alkalinity. The following reactions are important for determining the sludge produced from lime addition (Tchobanoglous et al. 2003):

\[ 10Ca^{2+} + 6PO_4^{3-} + 2OH^- \leftrightarrow Ca_{10}(PO_4)_6(OH)_2 \]  
Eq. 3-7

\[ Mg^{2+} + 2OH^- \leftrightarrow Mg(OH)_2 \]  
Eq. 3-8

\[ Ca^{2+} + CO_3^{2-} \leftrightarrow CaCO_3 \]  
Eq. 3-9

An advantage of lime sludge is that some stabilization can occur due to the high pH levels required. One disadvantage is that lime can cause scaling in mechanical thickening and dewatering systems. Although alum tends to produce less sludge than ferric salts, alum sludge can be more difficult to concentrate and dewater compared to ferric sludge.

The use of metal salts can result in increased inorganic salts (salinity) in the sludge and in the effluent. Salinity can create problems when biosolids are land applied or when the effluent is returned to existing water supply reservoirs. Biological phosphorus removal was developed in South Africa due to the high rate of indirect recycling of wastewater effluent, which led to excessive total dissolved solids (TDS) in the water supply during dry periods. High total salts can reduce germination rates for crops and negatively affect the soil structure, in addition to potential taste and odor problems.

3.6 Two Factors that May Limit the Ability of Plants to Achieve Very Low Effluent Levels

Two factors may limit the ability of plants to achieve very low levels: the presence of recalcitrant phosphorus and challenges in measuring very low effluent concentrations.

A portion of total phosphorus in wastewater can be recalcitrant, meaning that it passes through the chemical, biological, and physical treatment processes and into the effluent unchanged. Lancaster and Madden (2008) reported atypical recalcitrant phosphorus spikes as high as 0.5 mg/L in municipal wastewater, which were suspected to originate from an industrial source. The authors speculated that the recalcitrant fraction was made up of either dissolved acid-hydrolyzable phosphorus, organic phosphorus, or a combination of the two. Neethling et al. (2007) postulated that recalcitrant phosphorus is mostly the dissolved organic variety.

Studies conducted in Washington State and Nevada revealed significant variability in measured phosphorus concentrations less than 0.020 mg/L. The City of Las Vegas Pilot Study found that the accuracy of the measurement depended on several factors including the sample matrix and digestion procedure (Eleuterio and Neethling 2009). In 2007, WERF initiated a project to evaluate the capabilities of commercial laboratories to accurately measure phosphorus concentrations less than 0.020 mg/L and determine factors that affect method accuracy. Key findings, which were presented at the WEF 2009 specialty conference on nutrient removal, are as follows:

- Total phosphorus measurements exhibited a wide variability. This range was attributed to sample digestion procedures.
• Orthophosphate measurements were accurate for de-ionized water, but not for wastewater effluent samples. Variability in results was attributed to sample matrix and digestion procedures.

• The Ascorbic Acid method (either the USEPA or Standard Methods procedure) was the most often used and was found to be a reliable technique for measuring orthophosphate at low concentrations.

Eleuterio and Neethling (2009) concluded that additional research is needed to identify the compounds interfering with the method and develop techniques to correct these interferences.

3.7 References


Neethling, J.B., M. Benisch, D. Clark, A. Gu. 2007. Phosphorus Speciation Provides Direction to Produce 10 µg/L. In Nutrient Removal 2007. WEF.


