Hach Company TNTplus™ Phosphorus – Spectrophotometric Measurement of Phosphorus in Water and Wastewater

Hach Company TNTplus™ Phosphorus

Method 10209/10210
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Spectrophotometric Measurement of ortho - and Total Phosphorus in Water and Wastewater

1.0 Scope and Application

1.1 These procedures cover the determination of specified forms of phosphorus in drinking water, surface and saline waters, domestic and industrial wastes. The methods have been determined to be equivalent to EPA Method 365.3, following 40 CFR 136.6.

1.2 The procedures are based on reactions that are specific for the orthophosphate ion. Thus, depending on the prescribed pre-treatment of the sample, the various forms of phosphorus given may be determined. These forms are defined in Section 4.0.

1.3 The method is applicable in the range from 0.01 to 20 mg P /L, depending on the test range of the TNTplus Phosphorus kit.

2.0 Summary of Method

2.1 Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration and is measured at 650 or 880 ± 5 nm.

2.2 Only orthophosphate forms a blue color in this test. Polyphosphates (and some organic phosphorus compounds) may be converted to the orthophosphate form by sulfuric acid hydrolysis. Organic phosphorus compounds may be converted to the orthophosphate form by persulfate digestion.

3.0 Interferences

3.1 There are no interferences caused by copper, iron, or silicate at concentrations many times greater than their reported concentration in seawater. However, high iron concentrations can cause precipitation of and subsequent loss of phosphorus.

3.2 The salt error for samples ranging from 5 to 20% salt content was found to be less than 1%.

3.3 Arsenate is determined similarly to phosphorus and should be considered when present in concentrations higher than phosphorus. However, at concentrations found in seawater, it does not interfere.

4.0 Definitions

4.1 Total Phosphorus (P)--all of the phosphorus present in the sample, regardless of form, as measured by the persulfate digestion procedure.

4.1.1 Total Orthophosphate (P, ortho)--inorganic phosphorus [$(PO_4\text{-})^{-3}$] in the sample as measured by the direct colorimetric analysis procedure.

4.1.3 Total Organic Phosphorus (P, org)--phosphorus (inorganic plus oxidizable organic) in the sample measured by the persulfate digestion procedure, and minus hydrolyzable phosphorus and orthophosphate.

4.2 Dissolved Phosphorus (P-D)--all of the phosphorus present in the filtrate of a sample filtered through a phosphorus-free filter of 0.45 micron pore size and measured by the persulfate digestion procedure.

4.2.1 Dissolved Orthophosphate (P - D, ortho)--as measured by the direct colorimetric analysis procedure.

4.2.2 Dissolved Organic Phosphorus (P - D, org)--as measured by the persulfate digestion procedure, and minus dissolved hydrolysable phosphorus and orthophosphate.

4.3 The following forms, when sufficient amounts of phosphorus are present in the sample to warrant such consideration, may be determined:

4.3.1 Insoluble Phosphorus (P - I) = (P) - (P - D).

4.3.1.1 Insoluble orthophosphate (P - I, ortho) = (P, ortho) - (P - D, ortho).

4.3.1.2 Insoluble Organic Phosphorus (P - I, org) = (P, org) - (P - D, org).

5.0 Safety

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. It is suggested that the laboratory perform personal hygiene monitoring of each analyst using this method and that the results of this monitoring be made available to the analyst.

5.2 Unknown samples may contain high concentrations of volatile toxic compounds. Sample containers should be opened in a hood and handled with gloves to prevent exposure.

5.3 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of any chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses. Additional information on laboratory safety can be found in Sections 16.6 and 16.7.

6.0 Equipment

Note: *Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here, but demonstration of equivalent performance that meets the requirements of this method is the responsibility of the laboratory.*

6.1 Sampling equipment

6.1.1 Sample collection bottles—Glass, approximately 1-L, with PTFE-lined screw cap.
Note: *In those instances necessitating collection of a smaller aliquot, a smaller sample container may be used.*

6.1.2 Cleaning

6.1.2.1 All glassware used should be washed with hot 1:1 HCl and rinsed with distilled water. Preferably, this glassware should be used only for the determination of phosphorus and after use it should be rinsed with distilled water and kept covered until needed again. If this is done, the treatment with 1:1 HCl is only occasionally required. Commercial detergents should never be used.

6.2 Equipment for glassware cleaning

6.2.2 Oven - Capable of maintaining a temperature within ± 5°C in the range of 100–250°C.

6.3 Equipment for sample analysis

6.3.1 Hach DR 6000, DR 5000, DR 3800, DR3900, DR 2800 spectrophotometer, or equivalent.

6.3.2 DRB200 Digital Reactor Block for TNTplus: 30x13mm vial wells, 115 Vac (P/N DRB200-03)

6.4 Equipment for standard preparation

6.4.1 Volumetric flask – Glass, 1000-mL.

6.4.2 Volumetric flask – Glass, 50-mL.

6.4.3 Volumetric pipette – glass, assorted sizes.

7.0 Reagent and Standards

7.1 Reagent water – Water in which phosphorus is not detected at or above the method level of this method. Bottled distilled water, or water prepared by passage of tap water through ion exchange and activated carbon have been shown to be acceptable sources of reagent water.

7.2 Hach Company TNTplus Phosphorus Kits (TNT843, 0.01 – 1.5 mg P/L; TNT844, 0.5 – 5.0 mg P/L; TNTplus 845, 2.0 – 20 mg P/L

7.3 Hach Company Phosphate Standard Solution, 50 mg/L as PO_4, Cat. No. 171-49.

7.3.1 Prepare a secondary standard spiking solution by diluting 30.0 mL of standard solution (Section 7.3) to 1000 mL. Final concentration = 0.0005 mg P/mL.

7.4 Method detection limit solution

7.4.1 Prepare 7 or more replicate MDL solutions by diluting 1.0 mL of standard spiking solution (Section 7.3.1) to 50 mL. Final concentration = 0.01 mg P/L.

7.5 Initial precision and recovery solution

7.5.1 TNTplus 843, 0.01 – 1.5 mg P/L.

7.5.1.1 Prepare 4 or more replicate IPR solutions by diluting 10.0 mL of standard spiking solution (Section 7.3.1) to 50 mL. Final concentration = 0.10 mg P/L.

7.6 On-going precision and recovery

7.6.1 TNTplus 843, 0.01 – 1.5 mg P/L.

7.6.1.1 Prepare 1 or more solutions by diluting 1.0 mL of standard spiking solution (Section 7.3.1) to 50 mL. Final concentration = 0.10 mg P/L.

8.0 Sample Collection Preservation and Storage

8.1 If benthic deposits are present in the area being sampled, great care should be taken not to include these deposits.

8.2 Sample containers may be of plastic material, such as cubitainers, or of Pyrex glass.

8.3 If the analysis cannot be performed the day of collection, the sample should be preserved by the addition of 2 mL conc. H_2SO_4 per liter and refrigeration at 4°C.

9.0 Quality Control

9.1 It is recommended that each laboratory that uses this method be required to operate a formal quality assurance program (Section 16.1). The minimum requirements of this program consist of an initial demonstration of laboratory capability and ongoing analyses of laboratory prepared water standards as a test of continued performance to assess accuracy and precision. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.
The laboratory shall, on an ongoing basis, demonstrate through analysis of the ongoing precision and recovery sample that the analysis system is in control. This procedure is described in Sections 8.3.

9.1.2 Accompanying QC for the determination of P is required per analytical batch. An analytical batch is a set of samples processed during a contiguous 8-hour period. Each analytical batch must be accompanied by an ongoing precision and recovery sample, matrix spike sample, and matrix spike duplicate sample resulting in a minimum of four analyses (1 OPR, 1 sample, MS, and MSD).

9.2 Initial demonstration of laboratory capability.

9.2.1 To establish the ability to detect nitrite the analyst shall determine the MDL and ML per the procedure in 40 CFR 136, Appendix B (Section 16.5) using the apparatus, reagents, and standards that will be used in the practice of this method. An achieved MDL and ML less than or equal to the MDL in Section 13.0 is recommended prior to the practice of this method.

9.2.1 Prepare and measure seven replicates of the MDL standard according to the procedure beginning in Section 7.4.1.

9.2.2 Initial precision and recovery (IPR) - To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

9.2.3.1 Prepare and measure four samples of the IPR standard according to the procedure beginning in Section 7.5.

9.2.3.2 Using the results of the set of four analyses, compute the average percent recovery (X) and the standard deviation of the percent recovery (*s*) for nitrite. Use the following equation for calculation of the standard deviation of the percent recovery:

$$s = \sqrt{\frac{\sum x^2 - \frac{\left(\sum x\right)^2}{n}}{n-1}}$$

where:

n = Number of samples
X = % recovery in each sample

9.2.3.3 Compare *s* and X with the corresponding limits for initial precision and recovery in Table 1. If *s* and X meet the acceptance criteria, system performance is acceptable, and analysis of samples may begin. If, however, *s* exceeds the precision limit or X falls outside the range for recovery, system performance is unacceptable. In this event correct the problem and repeat the test.

9.3 Ongoing precision and recovery - To demonstrate that the analysis system is in control, and acceptable precision and accuracy is being maintained with each analytical batch, the analyst shall perform the following operations:

9.3.1 Prepare a precision and recovery standard with each analytical batch according to the procedure beginning in Section 7.6.

9.3.2 At the end of each analytical batch of samples, analyze a precision and recovery standard and compare the concentration recovery with the limits for ongoing precision and recovery in Table 3. If the recovery is in the range specified, measurement process is in control and analysis of samples may proceed. If, however, the recovery is not in the specified range, the analytical process is not in control. In this event, correct the problem, re-analyze analytical batch, repeating the ongoing precision and recovery test.

9.3.3 The laboratory should add results that pass the specification in Section 13.0 to IPR and previous OPR data and update QC charts to form a graphic representation of continued laboratory performance. The laboratory should also develop a statement of laboratory data quality for each analyte by calculating the average percent recovery (R) and the standard deviation of the percent recovery (sr). Express the accuracy as a recovery interval from R - 2sr to R + 2sr. For example, if R = 95% and sr = 5%, the accuracy is 85% to 105%.

9.4 Depending upon specific program requirements, field replicates may be required to assess the precision and accuracy of the sampling and sample transporting techniques.

10.0 Calibration and Standardization

10.1 The Hach Company DR series spectrophotometers have a built-in calibration that is automatically used when the TNTplus Phosphorus sample vial is placed in the cell holder of the instrument. No further initial calibration is required. However, the instruments have the capability of developing a user-calibration. See manufacturer's manual for instructions.

11.0 Procedure

11.1 Instrument Setup – follow the instrument manufacturer's instructions for instrument setup.

11.2 Sample Preparation and Analysis

11.2.1 Ortho-phosphate Low Range - Follow the instruction on Hach DOC316.53.01124

11.2.1.1 Adjust sample pH to 7 with 5 N sodium hydroxide solution.

11.2.1.2 Sample temperature should be 15 – 25^0C.

11.2.1.3 Use a pipet to add 2.0 mL of sample to the test vial.

11.2.1.4 Use a pipet to add 0.2 mL of Solution B to the test vial.

11.2.1.5 Place a grey DosiCap C on the vial, tighten and invert the vial 2-3 times.

11.2.1.6 Allow the reagents and sample to react for 10 minutes.

11.2.1.7 Invert the vial 2-3 times then place in the spectrophotometer and analyze.

11.2.1.8 Place test vial in spectrophotometer and read result.

11.2.2 Ortho-phosphate High Range - Follow the instruction on Hach DOC316.53.01125

11.2.2.1 Adjust sample pH to 7 with 5 N sodium hydroxide solution.

11.2.2.2 Sample temperature should be 15 – 25^0C.

11.2.2.3 Use a pipet to add 0.5 mL of sample to the test vial.

11.2.2.4 Use a pipet to add 0.2 mL of Solution B to the test vial.

11.2.2.5 Place a grey DosiCap C on the vial, tighten and invert the vial 2-3 times.

11.2.2.6 Allow the reagents and sample to react for 10 minutes.

11.2.2.7 Invert the vial 2-3 times then place in the spectrophotometer and analyze.

11.2.2.8 Place test vial in spectrophotometer and read result.

11.2.3 Ortho-phosphate Ultra High Range - Follow the instruction on Hach DOC316.53.01126

11.2.3.1 Adjust sample pH to 7 with 5 N sodium hydroxide solution.

11.2.3.2 Sample temperature should be 15 – 25^{0}C Adjust sample pH to 7 with 5 N sodium hydroxide solution.

11.2.3.3 Use a pipet to add 0.4 mL of sample to the test vial.

11.2.3.4 Use a pipet to add 0.5 mL of Solution B to the test vial.

11.2.3.5 Place a grey DosiCap C on the vial, tighten and invert the vial 2-3 times.

11.2.3.6 Allow the reagents and sample to react for 10 minutes.

11.2.3.7 Invert the vial 2-3 times then place in the spectrophotometer and analyze.

11.2.3.8 Place test vial in spectrophotometer and read result.

11.2.4 Total phosphate Low Range - Follow the instruction on Hach DOC316.53.01124

11.2.4.1 Adjust sample pH to 7 with 5 N sodium hydroxide solution.

11.2.4.2 Set digester block temperature to 120^{0}C.

11.2.4.3 Remove the lid from the DosiCap, then remove the cap from the test vial.

11.2.4.4 Use a pipet to add 2.0 mL of sample to the test vial.

11.2.4.5 Turn the DosiCap over so that the reagent side is placed on the test vial and tighten cap.

11.2.4.6 Invert the vial 2-3 times to dissolve the reagent in the cap.

11.2.4.7 Place test vial in heating block and digest at 120^0 C., for 30 minutes.

11.2.4.8 When the digestion time has expired, remove the vial from the reactor and cool to room temperature.

11.2.4.9 Invert the vial 2-3 times, then use a pipet to add 0.2 mL of Solution B to the test vial.

11.2.4.10 Place a grey DosiCap C on the vial, tighten, and invert the vial 2-3 times.

11.2.4.11 Allow the vial to reaction with Solution B for 10 minutes.

11.2.4.12 When the reaction time has expired, invert the vial 2-3 times.

11.2.4.13 Place test vial in spectrophotometer and read result.

11.2.5 Total phosphate High Range - Follow the instruction on Hach DOC316.53.01125

11.2.5.1 Adjust sample pH to 7 with 5 N sodium hydroxide solution.

11.2.5.2 Set digester block temperature to 120^0C.

11.2.5.3 Remove the lid from the DosiCap, then remove the cap from the test vial.

11.2.5.4 Use a pipet to add 0.5 mL of sample to the test vial.

11.2.5.5 Turn the DosiCap over so that the reagent side is placed on the test vial and tighten cap.

11.2.5.6 Invert the vial 2-3 times to dissolve the reagent in the cap.

11.2.5.7 Place test vial in heating block and digest at 120^0 C., for 30 minutes.

11.2.5.8 When the digestion time has expired, remove the vial from the reactor and cool to room temperature.

11.2.5.9 Invert the vial 2-3 times, then use a pipet to add 0.2 mL of Solution B to the test vial.

11.2.5.10 Place a grey DosiCap C on the vial, tighten, and invert the vial 2-3 times.

11.2.5.11 Allow the vial to reaction with Solution B for 10 minutes.

11.2.5.12 When the reaction time has expired, invert the vial 2-3 times.

11.2.5.13 Place test vial in spectrophotometer and read result.

11.2.6 Total phosphate Ultra High Range - Follow the instruction on Hach DOC316.53.01126

11.2.6.1 Adjust sample pH to 7 with 5 N sodium hydroxide solution.

11.2.6.2 Set digester block temperature to 120^0C.

11.2.6.3 Remove the lid from the DosiCap, then remove the cap from the test vial.

11.2.6.4 Use a pipet to add 0.4 mL of sample to the test vial.

11.2.6.5 Turn the DosiCap over so that the reagent side is placed on the test vial and tighten cap.

11.2.6.6 Invert the vial 2-3 times to dissolve the reagent in the cap.

11.2.6.7 Place test vial in heating block and digest at 120^0 C., for 30 minutes.

11.2.6.8 When the digestion time has expired, remove the vial from the reactor and cool to

room temperature.

11.2.6.9 Invert the vial 2-3 times, then use a pipet to add 0.5 mL of Solution B to the test vial.

11.2.6.10 Place a grey DosiCap C on the vial, tighten, and invert the vial 2-3 times.

11.2.6.11 Allow the vial to reaction with Solution B for 10 minutes.

11.2.6.12 When the reaction time has expired, invert the vial 2-3 times.

11.2.6.13 Place test vial in spectrophotometer and read result.

12.0 Data Analysis and Calculations

12.1 Phosphorus concentration is calculated automatically against internal instrument calibration.

13.0 Method Performance

Acceptance Criterion	Section	Limit
Method Detection Limit	9.2.1	0.003 mg/L P
Minimum Limit	9.2.1	0.01 mg/L P
Initial Accuracy	9.2.2	90% - 110%
On-going Accuracy	9.3	90% - 110%

14.0 Pollution Prevention

14.1 Follow guidelines in Section 15.

15.0 Waste Management

15.1 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect air, water, and land by minimizing and control all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

15.2 For further information on waste management, consult "The Waste Management manual for Laboratory Personnel", and Less is Better: "Laboratory Chemical Management for Waste Reduction", both available from the American Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036.

16.0 References

16.1 "Handbook of Analytical Quality Control in Water and Wastewater Laboratories," USEPA, EMSL-CI, Cincinnati, OH 45268, EPA-600-4-79-019, March 1979.

16.2 Standard Methods for the Performance of Water and Wastewater, 20th Edition, p 443, Method 5210B (1998).

16.3 International Oceanographic Tables, Vol. 1, National Institute of Oceanography of Great Britain, Womley, Godaming, Surrey, England and Uncesco, Paris 1971.

16.4 40 CFR 136, Appendix A, Methods 1624 and 1625.

16.5 40 CFR 136, Appendix B.

16.6 "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976)

16.7 "Safety in Academic Chemistry Laboratories," American Chemical Society, Committee on Chemical Safety, 3rd Edition, 1979.

17.0 Tables

17.1 Acceptance Criteria for Performance tests – The QC performance criteria for this method was performed with a Hach Company DR 5000 spectrophotometer using Hach Company TNTplus Phosphorus Kit.

Table 1. Initial Precision and Recovery Method Performance

IPR Concentration	Average Recovery (%)	Rel. Standard Deviation (%)
0.10 mg/L P	88.6	0.44

Table 2. Method Detection Limit and Method Limit Performance

MDL Test Concentration	MDL	ML
0.01 mg P/L	0.003 mg P/L	0.01 mg P/L

Table 3. On-going Recovery Performance

OPR Concentration	Average % Recovery
0.10 mg P/L	98%

18.0 Glossary of Definitions and Purposes

The definitions and purposes are specified to this method but have been conformed to common usage as much as possible.

18.1 Units of weight and measure and their abbreviations

18.1.1 Symbols

°C degrees Celsius

18.1.2 Alphabetical characters

mg/L milligram per liter

18.2 Definitions, acronyms, and abbreviations

18.2.1 MDL: Method detection limit

18.2.2 ML: Method limit

18.2.3 IPR: Initial precision and recovery

18.2.4 OPR: On-going precision and recovery

18.2.5 MS: Matrix spike

Phosphorus
Method Comparison Tables

	Method 365.3 /SM 4500-P E	Hach TNTplus Phosphorus (10209/10210)
Scope and Application	0.01 – 0.50 mg P/L	0.01 – 20 mg P/L
Summary of Method	Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. The complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration. Only orthophosphate forms a blue color in this test. Organic phosphorus compounds may be converted to the orthophosphate form by persulphate digestion.	Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. The complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration. Only orthophosphate forms a blue color in this test. Organic phosphorus compounds may be converted to the orthophosphate form by persulphate digestion.
Interference	No interference is caused by copper, iron, or silicate at concentrations many times greater than their reported concentration in seawater. However, high iron concentrations can cause precipitation of and subsequent loss of phosphorus.	No interference is caused by copper, iron, or silicate at concentrations many times greater than their reported concentration in sea water. However, high iron concentrations can cause precipitation of and subsequent loss of phosphorus.
Equipment	Spectrophotometer	Spectrophotometer
Sample Handling/ Preservation	If the analysis cannot be performed the day of collection, the sample should be preserved by the addition of 2 mL conc. H_2SO_4.	If the analysis cannot be performed the day of collection, the sample should be preserved by the addition of 2 mL conc. H_2SO_4.
Reagents and Standards	Sulfuric Acid (source of acidity) Antimony potassium tartrate (complexing reagent) Ammonium molybdate (complexing reagent) Ascorbic acid (reducing reagent)) Persulfate (digestion reagent)	Sulfuric Acid (source of acidity) Antimony potassium tartrate (complexing reagent) Ammonium molybdate (complexing reagent) Ascorbic acid (reducing reagent) Lithium sulfate (stabilizer) Sulfamic acid (interference reagent) Tartaric acid (solubility enhancer) Persulfate (digestion reagent)
Method Performance	Precision and Accuracy: Orthophosphate Conc. mg P/L / Stdev. / % Bias 0.29 / 0.10 / - 4.95 0.038 / 0.008 / - 6.00 0.335 / 0.018 / - 2.75 0.383 / 0.023 / - 1.76 Organic Phosphate (Total Phosphate) 0.110 / 0.033 / + 0.003 0.132 / 0.051 / + 0.016 0.772 / 0.130 / + 0.023 0.882 / 0.128 / - 0.008	Precision and Accuracy: Orthophosphate Initial Precision and Recovery 99% (0.10 mg P/L Stdev. 0.86 Ongoing Precision and Recovery – 97% Effluent #1 (Loveland, CO) Average Matrix Spike Recovery – 99% Stdev – 0.51 (0.20 mg P/L spike) Effluent #2 (Boston, MA) Average Matrix Spike Recovery – 94% Stdev – 2.6 (0.20 mg P/L spike)

		Effluent #3 (Ventura, CA Average Matrix Spike Recovery – 99% Stdev – 0.01 (0.20 mg P/L spike) Method Detection Limit – 0.003 P/L @ (0.01 mg P/L spike) Organic Phosphate (Total Phosphate) Conc. mg P/L % Recovery % Bias 0.03 97 - 3 0.05 98 - 2 0.10 104 + 4 0.20 102 + 2 0.30 100 0 0.40 99 - 1 0.50 97 - 3 1.00 98 - 2 1.20 98 - 2 1.30 98 - 2 1.40 98 - 2 1.50 99 - 1 Effluent #1 (Loveland, CO) Average Matrix Spike Recovery – 98% Stdev – 1.17 (0.20 mg P/L spike) Effluent #2 (Boston, MA) Average Matrix Spike Recovery – 99% Stdev – 2.3 (0.20 mg P/L spike) Effluent #3 (Ventura, CA Average Matrix Spike Recovery – 101% Stdev – 0.29 (0.20 mg P/L spike) Method Detection Limit – 0.003 mg P/L (0.01 mg P/L spike)