Collection and Analysis of Lake Bottom Sediments for Phosphorus Concentration Quality Assurance Project Plan
EPA Tracking # RFA 17003

Eastern Connecticut Conservation District
238 West Town Street, Norwich, CT 06360
860-887-4163 x 400

1.0 Title and Approval Sheet

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Signature/date

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ROSELAND LAKE, WOODSTOCK, CT

NUTRIENT SOURCE STUDY

Quality Assurance Project Plan

Collection and Analysis of Lake Bottom Sediments for Phosphorus Concentration

US EPA RFA # 17003

Eastern Connecticut Conservation District

Approved October 21, 2016

This document outlines the protocols for collecting and analyzing lake bottom sediments to determine the amount of available Phosphorus.

This project is funded in part by an US EPA Clean Water Act § 319 NPS grant through the CT DEEP. Project support also provided by the Town of Putnam, CT and The Last Green Valley, Inc.

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3.0 Distribution List

The following agencies, organizations and individuals will receive a copy of the approved QAPP and any subsequent revisions. A complete copy of the approved Quality Assurance Project Plan will be kept on file by the project QA Manager at the Eastern Connecticut Conservation District, and will be available upon request.

- Eastern Connecticut Conservation District: Dan Mullins, Jean Pillo
- Connecticut Department of Energy and Environmental Protection (CT DEEP): Chris Bellucci, Eric Thomas
- Solitude Lake Management, Dominic Meringolo
- US Environmental Protection Agency (EPA): Steve Winnett, Bryan Hogan
- Town of Putnam: Jerry Beausoleil
- Northeast Laboratories: Garrett Johnson

4.0 Project/Task Organization

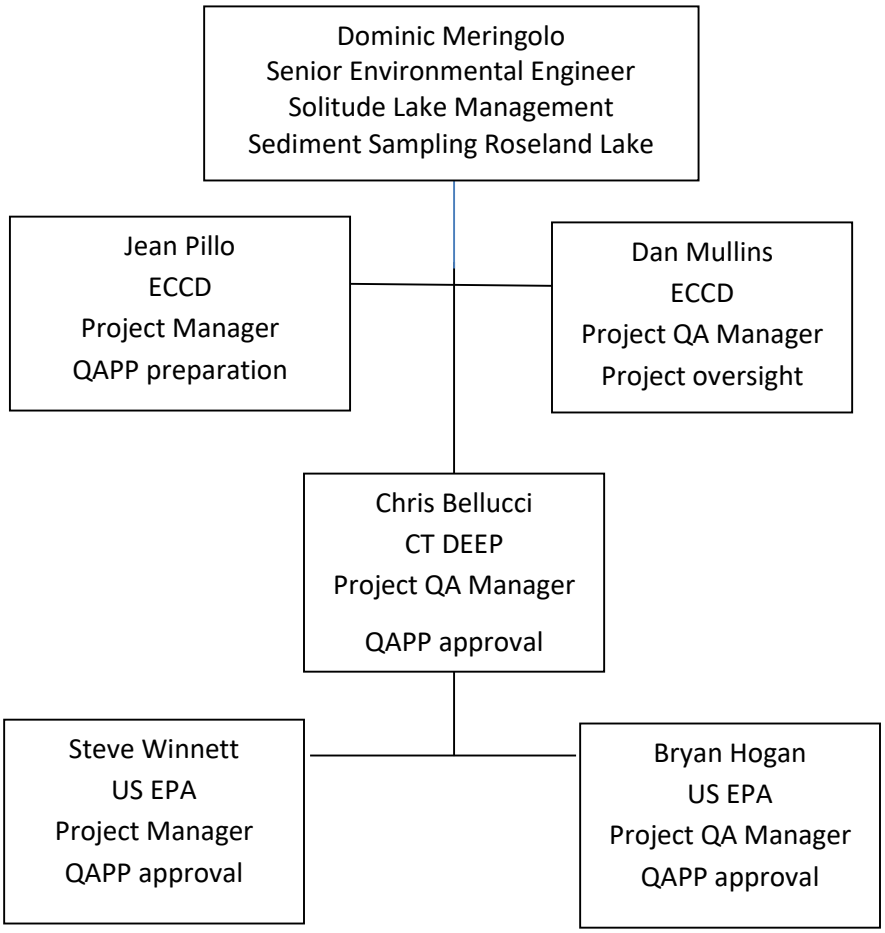


Figure 1 Project/Task Organization

5.0 Problem Identification/Background:

5.1 Problem Definition:

Roseland Lake, located in the Little River watershed in Woodstock, CT, has been listed as impaired for Recreation use in the CT DEEP Water Quality Assessment Report, most recently in 2014. The functional definition of recreation is swimming, water skiing, surfing or other full body contact activities (primary contact), as well as boating, canoeing, kayaking, fishing, aesthetic appreciation or other activities that do not require full body contact (secondary contact). The cause of impairment for Roseland Lake is Nutrient/Eutrophication and Biological Indicators.

5.2 Background Information:

Roseland Lake is in the Little River watershed (HUC12 011000010401), and is approximately two miles upstream of the surface water intake of a drinking water supply surface intake serving approximately 4000+ people in the neighboring town of Putnam. The lake was last evaluated for sediment and nutrient contributions from the watershed in 1991 based on data collected from 1981 – 83. During that period, it was estimated that an average of 427 tons of sediment were being deposited into Roseland Lake annually by Muddy Brook, the main tributary to the lake. Nitrogen and phosphorus concentrations were consistent with eutrophic category in Water Quality Standards.

Water quality in Roseland Lake has declined from mesotrophic conditions in the late 1930s to eutrophic/highly eutrophic conditions in more recent decades.

In freshwater ecosystems, phosphorus is typically the limiting nutrient for promoting algal and plant growth. The main sources of phosphorus in surface waters upstream of Roseland Lake in this watershed include erosion, fertilizer, animal waste (including waterfowl) and failing septic systems. In shallow thermally stratified lakes, phosphorus normally bonded with metals in the sediments can be released back into the water if the bottom layer lacks dissolved oxygen. Therefore, legacy phosphorus, deposited over time, may also be contributing to the eutrophication of Roseland Lake.

The Putnam Water and Sewer Department has been managing Roseland Lake for excess algal growth by treatment with copper sulfate since approximately 1980. Their staff had been using a subjective method to decide when to apply the algaecide. Copper sulfate is applied one to several times during the summer months. Roseland Lake is observed to experience numerous algae blooms during the warm weather season, including blue green algae blooms. Identification of the types and concentration of algae in Roseland Lake is important to protect recreational boaters and fishermen from coming in contact with potentially toxic harmful algae blooms and is important data for developing appropriate treatment methods for control of algae blooms. Beginning in 2015, two ongoing studies are in progress to identify and enumerate phytoplankton populations in Roseland Lake.

Improper application of copper sulfate can be both damaging the aquatic habitat and can actually exacerbate the problem they are trying to treat. A 1984 study on the Side Effects of 58 Years of Copper Sulfate Treatment of Fairmont Lakes, Minnesota (Stephan, 1984) is summarized in the Executive Summary of the report:

"The shallow Fairmont Lakes in southern Minnesota have been treated with copper sulfate for 58 years to reduce excessive algal growth. Data collected since treatment of the Fairmont Lakes began in 1921 provide alarming insights into lake responses to sustained chemical treatment with copper sulfate. Short-term and long-term effects have occurred. Short-term effects include: a) the intended temporary killing of algae, b) dissolved oxygen depletion by decomposition of dead algae, c) accelerated phosphorus recycling from the lake bed and recovery of the algal population within 7 to 21 days, and d) occasional fish kills due to oxygen depletion or copper toxicity or both. Long-term effects are shown to include: a) copper accumulation in the sediments, b) tolerance adjustments of certain species of algae to higher copper sulfate dosages, c) shift of species from green to blue-green algae and from game fish to rough fish, d) disappearance of macrophytes, and e) reductions in benthic macroinvertebrates. The conclusion is that while copper sulfate treatments enjoy great popularity because they kill and remove algae almost instantaneously, other immediate or cumulative side effects can be harmful to many other aquatic organisms."

Data needed to determine alternate treatment methods for management of the Roseland Lake as a drinking water supply is currently not available. If alternate treatment options for algae blooms are to be considered, an evaluation of the upper watershed nutrient contributions as well as in-lake source analysis is essential and ongoing. The in-lake source analysis needs to include an evaluation of the phosphorus load in the surface of the lake bottom sediments through the analysis of bottom sediment samples and an understanding of how much of the lake bottom experiences anoxic conditions. An outcome of this project will be that the data generated will improve Putnam's ability to manage the upstream waters as a public drinking water supply and the potential to consider alternate treatment methods in the lake that will have long term benefits with less environmental impacts.

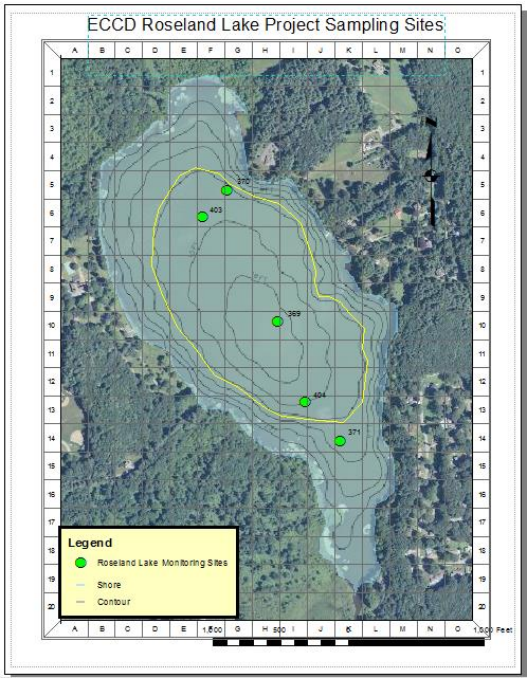


Figure 2 Roseland Lake Water Quality Monitoring Sites

Beginning in May 2015 and through July 2016, ECCD monitored Roseland Lake in three locations to demonstrate the period of time the lake was thermally stratified and determined the approximate duration and the depth of the anoxic layer. See Figure 2 Roseland Lake Water Quality Monitoring Sites. The outcome of this study was used to determine potential bottom sediment sampling locations. The yellow line estimates the area with summer anoxic conditions on the lake bottom.

6.0 Project/Task Description:

This project will involve the collection of sediment samples from the bottom of Roseland Lake in Woodstock by a qualified contractor. Samples will be collected using an Ekman grab sampler. The lake bottom will be sampled in four locations and based on data collected in 2015 and 2016 demonstrating the area of the lake bottom that experienced seasonal anoxic conditions. Refer to Attachment B. Roseland Lake Project Sediment Sampling Sites. Each sample will be analyzed for loosely sorbed Phosphorus and Iron-bound phosphorus. A composite of the four samples will be dosed with aluminum to determine the treatment requirements to immobilize the existing phosphorus in the sediment. The Standard Operating Procedures for collecting sediment samples are outlined Attachment A, Lake Sediment Sampling Standard Operating Procedures.

The sediment samples will be transported in an ice filled cooler to a qualified laboratory (NE Laboratory, Berlin, CT) for Phosphorus Fractionation, to determine the available phosphorus released during anoxic conditions on the lake bottom. The sample will also be analyzed to determine how much alum would be required to immobilize the available phosphorus.

Aluminum dosing will consist of re-testing of Fe-Bound phosphorus at 4-5 different doses of aluminum. Aluminum dosing standard operating procedures are located in Attachment E. The purpose of the aluminum dosing will be to:

- See how heterogeneous sediment conditions and concentrations are,
- Compare the available sediment P concentrations with the peak hypolimnetic concentrations
- Compare the reported dry weight basis concentrations with some values of the porosity/bulk density of the sediment to be able to look at the concentrations in an area basis for the lake.

The laboratory results will be interpreted by the qualified contractor, who will prepare a report on their data analysis.

7.0 Data Quality Objectives for Measurement Data:

All data must meet quality objectives for completeness, representativeness, and comparability, and also meet measurement performance criteria for accuracy and precision.
Completeness: It is expected that sediment samples will be collected in all four locations selected in consultation with the project consultant.
Representativeness and Comparability: Sediment sampling sites have been selected to characterize quality of the lake bottom sediments within the hypolimnion beneath the anoxic zone. Depth profile data demonstrating the depth of the oxygen depleted layer
Data Accuracy and Precision: Data accuracy (conformity to the true value) will be ensured by Quality Assurance (QA) procedures (e.g., inspection, maintenance and calibration of instruments), and Quality Control (QC) checks (e.g., analysis of laboratory blanks, standards and spiked samples). Precision (repeatability of measurements) will be ensured by consistent

methods and practices, and evaluated by field and laboratory replicate samples. Accuracy and precision goals for parameters to be measured for this study are provided in Table A7-1 for laboratory tests.

Table A7-1 Data Quality

Parameter	Method ID	Method Name	MDL	Preservation method	Sample Hold time
Phosphorus	SM4500-P E	colorimetric ascorbic acid reduction method	0.1 mg/l	4˚C	48 hours

8.0 Measurement/Data Acquisition

8.1 Sampling locations:

In 2015 and 2016, ECCD staff sampled in Roseland Lake and determined the lake to be nearly anoxic at 3.5 M and completely anoxic at 4.0 M from May through August. Using a bathymetric map of Roseland Lake, sediment sampling locations were selected in areas of the lake where the water is greater than 4.0 M deep. Using divisions of 100' by 100', a grid system was laid out over the project area. Whole grids over the anoxic area were numbered sequentially in a west to east, north to south pattern and a random number generator was used to select 4 of the 31 grids for sample collection. See Attachment B Roseland Lake Project Sediment Sampling Location Map.

8.2 Data Collection:

Benthic sediments samples will be collected by a qualified contractor (Solitude Lake Management). Four sediment samples will be collected and analyzed independently for loosely sorbed Phosphorus and Iron-bound phosphorus at a certified lab qualified to conduct the analysis (Northeast Laboratory, Berlin, CT). At one random sampling location, duplicate samples will be prepared from one grab sample for quality control purposes. A composite sample containing equal volumes of sediment samples from the four sampling locations will be dosed with aluminum to determine the treatment requirements to immobilize the existing phosphorus in the sediment. The Lake Sediment Sampling Standard Operating Procedures are located in Attachment A.

8.3 Data Management:

Solitude Lake Management will keep a record of their field sampling experience in a field notebook. The field notes will include information regarding the date, time and weather conditions at the time the sampling took place, and the GPS coordinates of each sample location. Observations regarding the sediment composition and the number of samples collected at each

site will also be recorded. Field notes will be copied and included with the raw data used to generate the final report for this project.

8.3.1 Sample chain of custody documentation:

The Northeast Laboratory Form will serve as the sample chain of custody documentation. A sample of the Northeast Laboratory Form is located in Attachment D.

8.3.2 Laboratory Results:

Laboratory results will be transferred electronically to Solitude Lake Management and copied to ECCD. The data will be transcribe into an excel spreadsheet and reviewed to assure there were no data transcription errors. Raw data will be used by Solitude Lake Management for interpretation and reporting.

8.3.3 Data Storage:

ECCD will retain electronic copies of the data on an external hard drive. Hard copies of the data sheets will be retained for 5 years in a secure location.

8.4 Quality Controls:

Laboratory quality controls include a laboratory blank and a laboratory duplicate. See Attachment C: Northeast Laboratory Standard Operating Procedures for *Phosphorus Fractionation of Sediments for Loosely-sorbed and Iron Bound Phosphorus* for a complete description of their laboratory quality control practices.

9.0 Assessment/Oversight

9.1 Assessment:

Assessment of the laboratory results will be completed by the project consultant, Solitude Lake Management.

9.2 Reports:

The Contractor (Solitude Lake Management) will prepare a report on the outcomes of the sediment sampling and give a presentation of the results to CT DEEP, ECCD and the other project partners.

10.0 Data Review, Validation and Verification

The Project Manager and QA Officer (ECCD) will review data to determine if QAPP data objectives are being met. Other resource professionals with a particular expertise may be consulted as needs arise. The Project Manager and QA Officer (ECCD) make the final determination about whether results will be accepted, qualified or rejected.

Attachment A: Lake Sediment Sampling Standard Operating Procedures
Site selection:

Determine the depth to the anoxic zone of the lake. Use a bathymetric map of the lake to create a shapefile outlining the edge of the anoxic zone. Overlay a grid pattern using 100 foot by 100 foot grid size. Beginning at the northwest corner of the lake, number the grids over the anoxic zone sequentially using a west to east, north to south pattern. Incomplete grid squares are not included. Using a random number generator, select for stations to collect samples. Determine the GPS coordinates for the center of each grid. In the field, use a hand held GPS unit to locate those coordinates and direct the vessel over each sampling location.

Sample collection:

The Ekman Bottom Grab is a light weight bottom grab designed to take samples in soft, finely divided littoral bottoms free from vegetation and intermixtures of sand, stones and other coarse debris. It is useful for sampling lake bottoms composed of finely divided muck, mud, ooze or fine peaty materials.

Figure 3 Ekman Dredge

Preparation:

Equipment List

1. 6" x 6" Ekman Dredge
2. Light line
3. Weighted messenger
4. Four clean pails
5. Four 250 ml acid washed polypropylene jars with tops (one per sampling site)
6. Sample labels with serial identification numbers.
7. Chain of Custody Form for sampling records

Sample collection standard operating procedures

1. Inspect the dredge to make sure it is in good working condition.
2. Using a GPS unit, locate the first sampling site. Pilot the boat above it and drop anchor.
3. Prepare the sampler for collecting samples.
    A. Thread the weighted messenger on the line, attach the line securely to the dredge by passing the line through the trip mechanism and knotting it securely below the underlying plate.
    B. Fasten the opposite end of the line to a boat or hoist.
    C. Set the springs.

a. At the sampling site, when ready to sample, close the scoops
b. Hook the end of one spring onto one scoop button
c. Stretch the spring to reach the second scoop button.
d. Repeat with the second spring on the other side of the grab.

D. Arm the scoops
a. Just before lowering the Ekman grab into the water, hook one cable loop to one Twin-Pin pin.
b. Repeat for the second cable loop.

Caution: The dredge is now armed and dangerous! Keep fingers clear of potentially moving parts.

E. Use the white ball on the cable for a hand grip.
F. Lower the sampler to the desired sampling spot on the bottom
a. Lower the dredge in the open form into the water so that it will descend vertically.
b. The sampler should not be dropped too rapidly; otherwise it will tend to descend in a diagonal direction.
c. When lowered properly, the sampler hits the bottom correctly.
d. On arrival at the bottom, allow the dredge to settle; holding the line with just enough tension to keep the line straight but not lift the sampler off the bottom surface.
G. Release the messenger on the line and let it slide down to the sampler. The impact of the messenger depresses the Twin-Pin™ strike pad, which swipes the cables off the pins to close the scoops.

7. Retrieve your sample

A. After the dredge has closed, lift it to the surface with moderate, steady speed.

8. Remove your sample.

A. Gently drain any water in the Ekman before scooping the sample.
B. Scoop the top 2" of the sample while it's still in the Ekman dredge using a large spoon. Scooped material is placed in a pail for mixing with other samples for the composite.
C. Discard the remaining sediment.
D. Rinse the dredge with clean water.

9. Change position of the boat to insure adequate sampling by using a very long anchor rope, and pulling a portion onboard to relocate from the previously sampled area.

10. Repeat steps 3 – 8 two additional times at each sampling location, adding the top two inches of sediment in the dredge to the same bucket.

11. Collecting a representative sample:

A. After each location has been sampled three times, thoroughly mix the combined sediment samples in the bucket.
B. Fill a 250 ml acid washed wide mouth polyethylene container with the composite sample from that site.
C. Label the container with the name of the lake, grid number, and date and time the sampling took place.
D. Place the labeled sample in a cooler with ice packs for shipment to the laboratory.
E. Complete the Northeast Laboratory, Inc. Chain of Custody Sheet, completing one line for each sample collected.

12. After completing the process at a sampling location, unhook the springs from their scoop button. This is a safety precaution!

13. Repeat steps 2 – 10 for the each predetermined sampling location. Be sure to keep accurate records of number of samples taken.

To eliminate sample leakage from the dredge.

A. If on reaching the surface, the dredge shows leakage through the jaws, inspect for grit lodged between the side jaws and the wall of the dredge body.
B. Hard objects caught between the edges of the two opposing jaws also cause leaking. Discard these samples. Thoroughly clean parts before continuing sampling.

Sampling Tips:

Use boats with a high degree of stability, especially on large, rough bodies of water.

On-Site Maintenance:

1. Inspect the sampler before each use. Replace or repair damaged parts.
2. Avoid impacts with the survey vessel and other objects. Damage in the form of bent or dented sides, jaws and lids is likely to result in leaks, seriously impairing its effectiveness.
3. Avoid collecting fine grains of sand between the sides of the box and the closely opposed sides of the jaws. Sand can prevent the jaws from closing properly, resulting in sample loss and contact with overlying water.
4. After each day's operations, rinse all parts with fresh water before adhering bottom muds become dry. Do the same with all equipment/
5. Before Leaving a Boat Launch, practice the Clean, Drain, Dry technique[1].

Clean:

- Inspect and remove all visible plant, fish, and animals as well as mud or other debris at the launch. Do not transport them home.

[1] http://www.ct.gov/deep/cwp/view.asp?a=2696&q=322690&deepNav_GID=1630

- Check trailer, including axle and wheel areas - in and around the boat itself: anchor, props and jet engines, ropes, boat bumpers, paddles (anything that came in contact with the water).

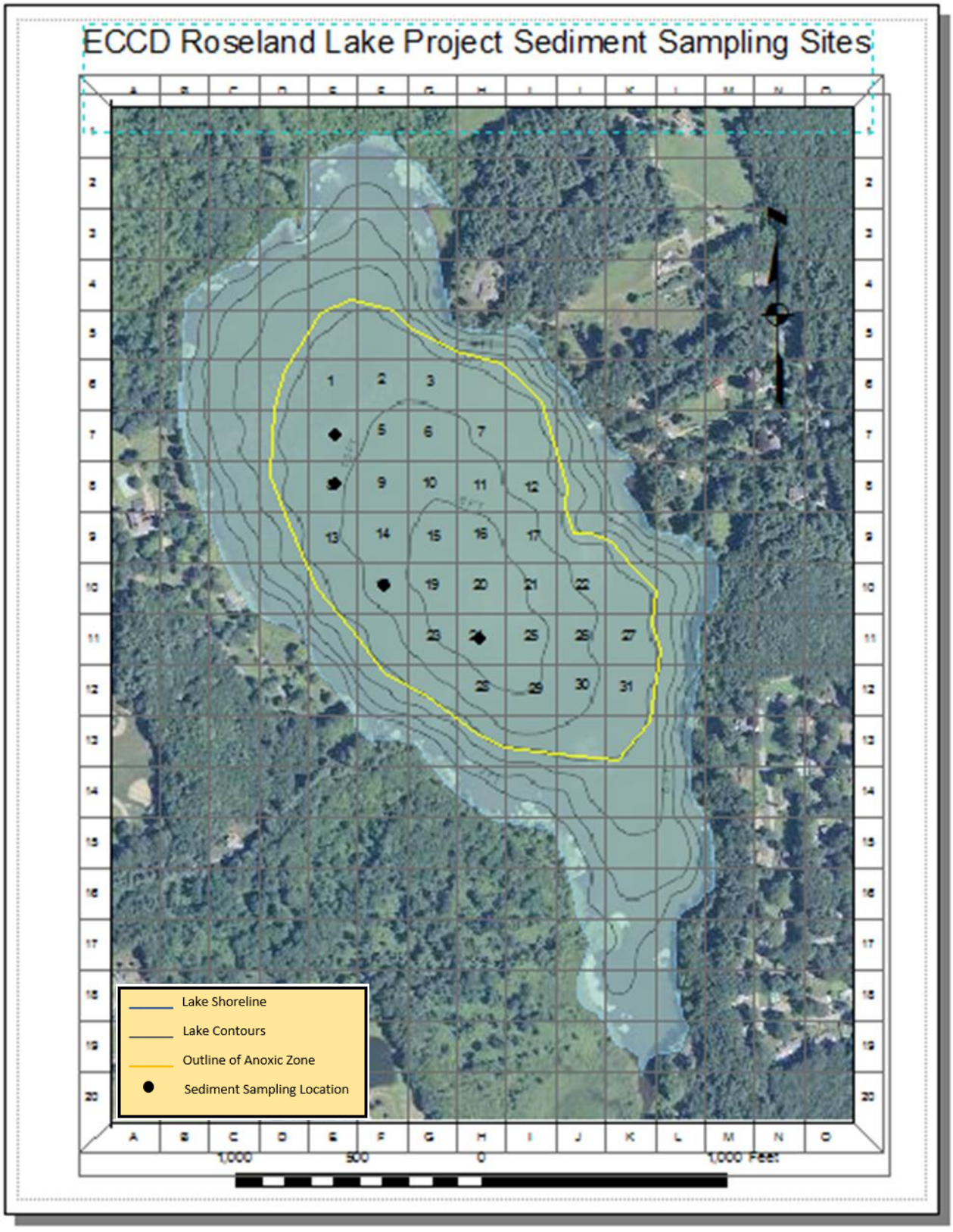
Drain:

- Eliminate all water from every conceivable space and item before you leave the area you
- Remove the drain plug from boats and put boat on an incline so that the water drains out
- Drain all water in live-wells, bilge, ballast tanks, transom wells, kayaks, canoes, rafts, motors, jet drives, boat hulls, scuba tanks and regulators, boots, waders, bait buckets, seaplane floats and swimming floats.

Dry:

- Dry Equipment, if possible, allow for 5 days of drying time before entering new waters.

Attachment B. Roseland Lake Project Sediment Sampling Sites



Attachment C: Northeast Laboratories Standard Operating Procedures

Northeast Laboratory of Danbury	Title: SOP for Phosphorus and Ortho Phosphate	
129 Mill Street	Doc. No. 1	Rev. No. 2
Berlin, CT 06037	Date: 4/12/2002	Page: 1 of 6

STANDARD OPERATING PROCEDURE

ANALYSIS OF PHOSPHORUS

AND ORTHO PHOSPHATE

IN WATER

Approved: _____
Technical Director & Quality Manager

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to drinking, surface and saline waters, domestic and industrial waters.

1.2 This method is based on the reactions that are specific for the orthophosphate ion, thus depending on the sample pretreatment, the various forms of phosphorus may be determined.

2.0 SUMMARY OF METHOD

2.1 Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute phosphorus solutions to form an antimony-phospho-molybdate complex. This complex is reduced by ascorbic acid to an intensely blue-colored complex, which is proportional to the phosphorus concentration.

2.2 The reporting of results as orthophosphate or total phosphorus is dependent on whether or not the digestion procedure was used.

3.0 INTERFERENCES

3.1 High iron concentrations can cause precipitation and a subsequent loss of phosphorus.

3.2 Arsenate is determined similarly to phosphorus and should be considered when its presence is known.

4.0 EQUIPMENT AND SUPPLIES

4.1 Spectrophotometer (880 nm)
4.2 Volumetric flasks; 50, 100, 1000ml
4.3 Pipets (class A), graduated
4.4 Beakers; 150ml, 500ml
4.5 Graduated cylinders; 25ml, 50ml, 100ml
4.6 Hot plate

Note: Glassware should be washed with nitric acid and rinsed with distilled water prior to use.

5.0 REAGENTS AND STANDARDS

5.1 Sulfuric acid solution (5N): Dilute 70mL of concentrated H_2SO_4 with 500mL of distilled water.

5.2 Ammonium molybdate solution: Dissolve 10g $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 250mL of distilled water.

5.3 Antimony potassium titrate solution: Purchased or dissolve 1.3715g $K(SbO)C_4H_4O_6 \cdot \frac{1}{2} H_2O$ in 400 mL of distilled water in a 500mL volumetric flask, and dilute to volume. Store at 4ºC.

5.4 Ascorbic acid (0.1M): Dissolve 1.76g of ascorbic acid in 100mL of distilled water. Expires:1 week

5.5 Combined reagent: (100mls) Add 5mL of antimony potassium tartrate solution, and add 15mL of ammonium molybdate solution to 50mL of 5N H_2SO_4, mixing after each reagent is added. Add 30mL of 0.1M ascorbic acid. Prepared combined reagent fresh daily. Double to make 200mls for larger batches of phosphorus.

5.6 Stock phosphorus solution (1.0mL = 50µg P; 50 ppm)
Dissolve 0.2195g of anhydrous potassium dihydrogen phosphate, KH_2PO_4 in distilled water and dilute to 1L.

5.7 Standard phosphorus solution (1.00mL = 0.5µg P; 0.5ppm)
Dilute 10.00 mL stock phosphorus solution to 1 liter.

5.8 Sodium hydroxide (1N): Dissolve 40g NaOH in 600mL of distilled water and dilute to 1 liter.

5.9 Phenolphthalein indicator.

5.10 Potassium persulfate ($K_2S_2O_8$).

6.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

6.1 Collect at least 200ml of sample in a 250 ml acid washed wide mouth polyethylene.

6.2 Samples are to be stored preserved with H_2SO_4 at 4°C for total phosphorus. Samples to be analyzed for orthophosphate are stored unpreserved at 4°C.

6.3 Samples are to be analyzed within 28 days of sampling for total phosphorus, or within 48 hours of sampling for orthophosphate.

7.0 PROCEDURE

7.1 Phosphorus

7.1.1 Add 0.7 mL of concentrated H_2SO_4 to a 100mL sample in a 150mL beaker.

7.1.2 Add 1.0g of potassium persulfate.

7.1.3 Boil gently on a hot plate until a final volume of 10mls has been reached. Do not allow sample to go to dryness! Cool and dilute the sample to approximately 30mL.

7.1.4 Add few drops of phenolphthalein and neutralize to a faint pink with a strong NaOH. Dilute to 100mL.

7.1.5 Bring a standard, blank, LCS, duplicate and spike through the digestion procedure with the samples.

7.1.6 Determine as orthophosphate as below.

7.2 Orthophosphate

7.2.1 Add two drops phenolphthalein and neutralize to a faint pink with 6 N NaOH, if necessary.

7.2.2 Add 8.0mL of combined reagent to the sample or standard and mix thoroughly. After 10 minutes, read the color developed on a spectrophotometer at 880nm.

Note: Samples must be read on the spectrophotometer within 30 minutes of adding the color reagent.

7.2.3 Spectrophotometer calibration instructions are available in Attachment I.

7.2.4 A new curve is to be prepared whenever a new phosphorus stock solution is made from a series of 6 standards.

7.3 Dissolved Phosphorus

7.3.1 Filter samples for dissolved phosphorus through a 0.45µm membrane filter or a glass fiber filter.

7.3.2 Proceed as in 7.1 or 7.2

8.0 QUALITY CONTROL

8.1 All stock solutions and standard preparations are logged and coded. All solutions are labeled with the following: analyte, concentration, analyst's initials, date prepared and expiration date.

8.2 A prep blank (reagent water) is to be analyzed with every batch of twenty or fewer samples.

8.3 One sample duplicate must be analyzed from each batch of ten or fewer samples. The relative percent difference (RPD) between the sample and duplicate must be less than or equal to 20%. No RPD criteria is applied if both results are less than five time the MDL. No MDL criteria is applied if one result is lower than five times the MDL or one above, if the difference between the two results is no more than the MDL.

8.4 A sample spike is performed on every batch of ten or fewer samples. The acceptable spike recovery range is 75 to 125%.

8.5 A laboratory control sample (LCS) must be analyzed in every batch of 10 or fewer samples. The LCS recovery must be between 85 –115%.

9.0 CALCULATIONS

9.1 Phosphorus Calculation:

mg/L as P = A x B

Where: A = mg/L P (from curve)
B = Dilution factor (if any)

9.2 The relative percent difference is calculated:

$$RPD = \frac{(S - D)}{(S + D)/2} \times 100$$

Where: S = Original sample result
D = Duplicate sample result

9.3 The Lab Control and standard recoveries

%Recovery = mg/L found x 100 mg/L true value

9.4 The sample matrix spike is calculated:

$$\text{Percent Recovery} = \frac{(SSR - SR)}{SA} \times 100$$

Where: SSR = Spike Sample Result (mg/L)
SR = Sample Result (mg/L)
SA = Spike Added (mg/L)

10.0 REPORTING OF RESULTS

10.1 Results are computed for samples and recorded in the chemistry wastewater logbook and also in the file/lab sheet for each sample.

11.0 DOCUMENTATION OF PROBLEMS

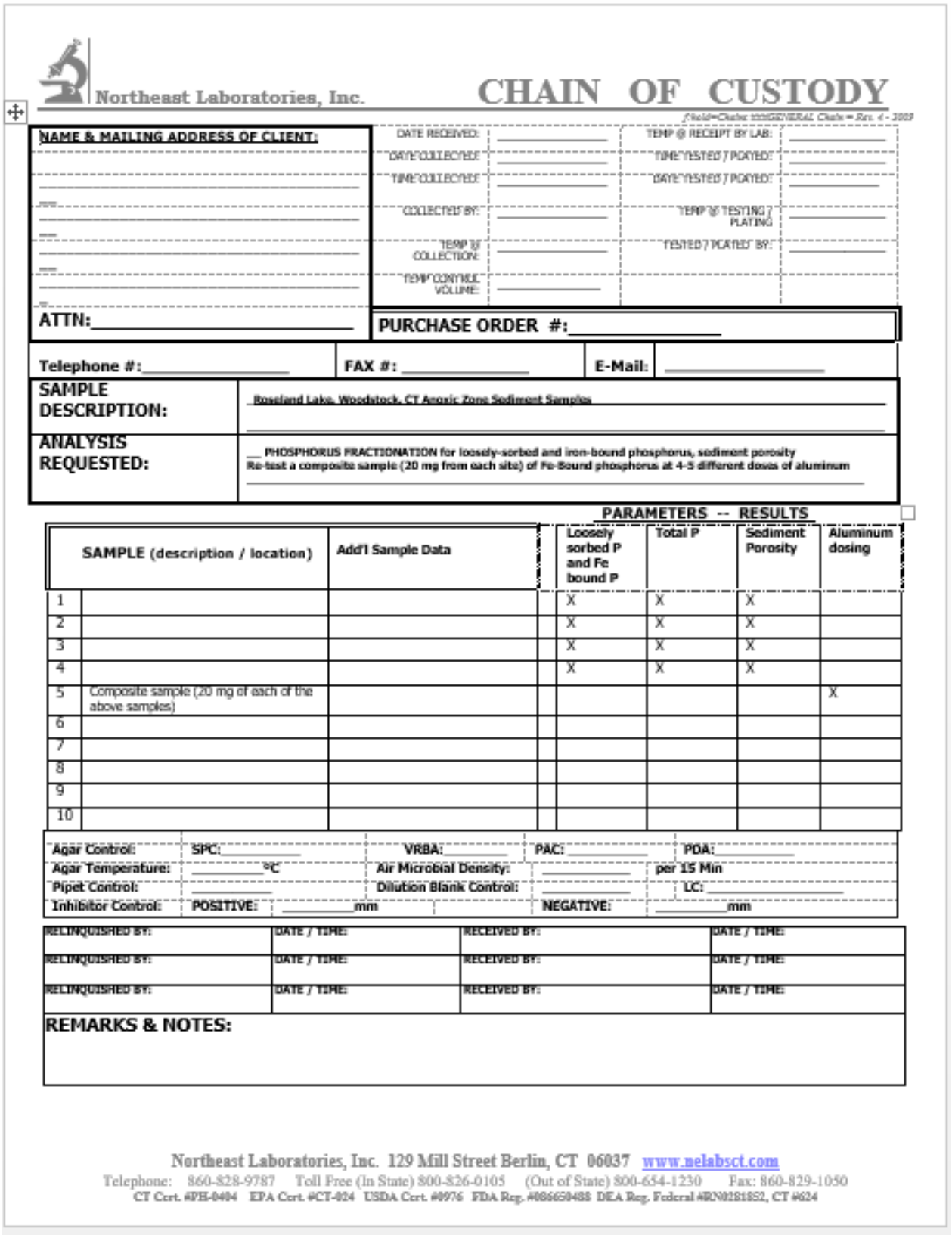
11.1 Corrective action reports must be filed if any problems were associated with the analysis.

11.2 Case narratives – should include any deviation from this SOP or any difficulties encountered in the procedure.

12.0 REFERENCES

Standard Methods for the Examination of Water and Wastewater, 18th edition, 1992**;** 4500-P B,E**.**

Attachment D: Chain of Custody Form Northeast Laboratory



Northeast Laboratories, Inc.

CHAIN OF CUSTODY

(Yield=Choice ####GENERAL Chain = Rev. 4 - 2009

NAME & MAILING ADDRESS OF CLIENT:	DATE RECEIVED:		TEMP @ RECEIPT BY LAB:	
	DATE COLLECTED:		TIME TESTED / PLATED:	
	TIME COLLECTED:		DATE TESTED / PLATED:	
	COLLECTED BY:		TEMP @ TESTING / PLATING	
	TEMP @ COLLECTION:		TESTED / PLATED BY:	
	TEMP CONTAINER VOLUME:			

ATTN:_____ PURCHASE ORDER #:_____

Telephone #:_____ FAX #: _____ E-Mail: _____

SAMPLE DESCRIPTION:	Roseland Lake, Woodstock, CT Anoxic Zone Sediment Samples
ANALYSIS REQUESTED:	__ PHOSPHORUS FRACTIONATION for loosely-sorbed and iron-bound phosphorus, sediment porosity Re-test a composite sample (20 mg from each site) of Fe-Bound phosphorus at 4-5 different doses of aluminum

PARAMETERS -- RESULTS

	SAMPLE (description / location)	Add'l Sample Data	Loosely sorbed P and Fe bound P	Total P	Sediment Porosity	Aluminum dosing
1			X	X	X	
2			X	X	X	
3			X	X	X	
4			X	X	X	
5	Composite sample (20 mg of each of the above samples)					X
6						
7						
8						
9						
10						

Agar Control:	SPC:	VRBA:	PAC:	PDA:
Agar Temperature:	°C	Air Microbial Density:	per 15 Min	
Pipet Control:		Dilution Blank Control:	LC:	
Inhibitor Control:	POSITIVE: mm		NEGATIVE:	mm

RELINQUISHED BY:	DATE / TIME:	RECEIVED BY:	DATE / TIME:
RELINQUISHED BY:	DATE / TIME:	RECEIVED BY:	DATE / TIME:
RELINQUISHED BY:	DATE / TIME:	RECEIVED BY:	DATE / TIME:

REMARKS & NOTES:

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CT Cert. #PH-0404 EPA Cert. #CT-024 USDA Cert. #0976 FDA Reg. #086650488 DEA Reg. Federal #RN0281852, CT #624

Attachment E: Aluminum dosing Standard Operating Procedures

Northeast Laboratories, Inc.

STANDARD OPERATING PROCEDURES FOR Determination of Phosphorus Inactivation by Aluminum in Sediment Samples

Prepared by:	Alan C. Johnson	Digitally signed by Alan C. Johnson Date: October 18, 2016
Reviewed by:		Digitally signed by Date: October 18, 2016
Laboratory Director Signature Signifies Effective Date:	Alan C. Johnson	Digitally signed by Alan C. Johnson Date: October 18, 2016

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STANDARD OPERATING PROCEDURES FOR Determination of Phosphorus Inactivation by Aluminum in Sediment Samples

Reference
Phosphorus Fractionation of Sediments for Loosely Sorbed and Iron Bound Phosphorus.

Aluminum dose testing is conducted in the laboratory to determine the appropriate dose of aluminum as a rehabilitation technique for inactivating phosphorus in sediments.

Summary
A wet sediment sample of a known solids level is reacted with aluminum at a known level to establish dosing levels needed for the lake, pond or water body phosphorus content inactivation.

Aluminum dose testing is conducted in the laboratory to determine the appropriate dose of aluminum as a rehabilitation technique for inactivating phosphorus in sediments.

Treatment Solutions

Aluminum Sulfate	4.4% Al (58.27 g/L)
Sodium Aluminate	10.38% Al (1498 g/L)

Standard ratio is a mix of Aluminum Sulfate and Sodium Aluminate at 2:1 by volume.

Testing
Set up reactions tubes (as per Phosphorus Fractionation Test) including a blank and a range of doses (e.g. 5-200 g/sq.m.). Tumble 2 hours minimum, then allow to stand for 16-24 hours to ensure complete reaction.

Analyze for loosely sorbed and iron bound phosphorus.

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