EXPRESS NEWS

Volume 1, Number 1 *Fall 2003*

Meeting the NELAC Accreditation Requirements

By Jerry L. Parr

Over 1,000 laboratories in the United States are now accredited to the standard developed by the National Environmental Laboratory Accreditation Conference (NELAC), with 11 state organizations implementing the program. This article describes the benefits of the NELAC Program and outlines the steps a laboratory can take to meet NELAC requirements.

Questions that need to be answered when considering applying for NELAC Accreditation are:

- Do my customers and regulators expect NELAC Accreditation?
- Does my state currently implement this program?
- Will accreditation be beneficial to my lab when working within other states where NELAC is required?
- Would standardization of processes within my laboratory result in improved data quality?
- What are the direct and indirect costs?
- How long will the process take?

Benefits of NELAC

Results from a 2001 survey of NELAC accredited laboratories indicated that implementation of the program improved both the laboratories' internal processes as well as data quality. The reason for this improvement: NELAC laboratories standardize routine operations (e.g., sample handling, instrument calibration, reagent purchasing). This standardization results in more efficient and improved laboratory operations.

Requirements for Accreditation

1. Analyze four proficiency test (PT) samples per year
2. Designate a Technical Director
3. Implement a Quality System
4. Pass on-site assessment of the laboratory to verify other elements accomplished
5. Application filed and appropriate fees paid

Costs Associated with NELAC Standards

The direct costs of the program are the purchase of PT samples and fees paid to the state agency providing the accreditation. Indirect costs include activities required to implement the program, as well as ongoing costs for analyzing PT samples and meeting the Quality System requirements.

Steps to Implement the Program:

1. Become familiar with the requirements, especially those in Chapters 2, 4 and 5 of the NELAC standard (http://www.epa.gov/ttn/nelac/2002standards.html). Take a training course or come to a NELAC meeting.

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Baltimore, MD; Oct. 11-15

FACSS
Ft. Lauderdale, FL; Oct. 19-23

BOD

Common Questions & Answers

PART ONE

The following are questions often asked by wastewater and other labs about the BOD/CBOD test. The answers are provided by Perry Brake, manager of Washington's Environmental Laboratory Accreditation Program. Brake reminds readers that the regulatory and lab accreditation staffs in their own states have the final say on any controversial issues. For more information, e-mail Brake at PBRA461@ECY.WA.GOV.

Q: What are the important quality control tests for the BOD/CBOD analysis?

A: *The blank, the standard (50:50 solution of glucose/glutamic acid), and an occasional duplicate to check within-batch precision.*

Q: My blanks are usually less than 0.2 mg/L depletion, but every few months, I get a 0.6 mg/L and sometimes higher. Should I invalidate all the data for that test?

A: *If your blanks are usually within the expected range (<0.2 mg/L), you can safely assume that the rare failure is a problem for only that one bottle, perhaps resulting from contamination. There is no need to disqualify all data in that batch. Your blanks usually should be less than 0.1, with an occasional 0.2, and a very rare >0.2. If they are higher than that, you need to take corrective action.*

Q: What causes blanks to be high?

A: *As mentioned above, the occasional high blank might be contamination of a single bottle. If blanks are usually too high, it can be contamination in any part of the system, or it can be and often is a problem with the source water or dilution water. Very seldom is it a problem with measurement of the DO, because that would cause both high and negative blanks. If you determine that water might be a source of high blanks, try buying "steam distilled" water at a supermarket. If that solves the problem, you can either upgrade your reagent grade water system in the lab, or continue to buy the water. At times, contamination of bottles is a problem. One of the labs in our accreditation program ran a side-by-side test using glass and disposable plastic BOD bottles. All data was very comparable, except for blanks, where results were good for both types of bottles, but consistently lower for plastic. NPDES Methods 5210B does not specify what type of bottle must be used.*

Q: How about potassium hydrogen phthalate (KHP) as the standard?

A: *KHP contains no nitrogen. If you are doing CBOD, that is not a problem, except that the data quality objectives for analysis of a KHP standard aren't as well established as those for a 50:50 G/GA solution.*

Q: Analysts often hear that G/GA results should be "198 ± 30.5 mg/L." What does that mean?

A: *Many think it means all your G/GA results have to be within the range of 198 ± 30.5 mg/L. That isn't what* Standard Methods *5210B says, and I don't think that is what it means. Those numbers come from a nationwide study where several labs averaged 198 mg/L, and the standard deviation of those same results was 30.5 mg/L. The 20th Edition of* Standard Methods *tells you the 30.5 mg/L should be considered an action limit, and since action limits are normally three standard deviations, a single lab should have a standard deviation of approximately 10 mg/L. I think that is a bit optimistic. Anywhere in the mid-teens or lower is good. The guideline for CBOD of a solution of 150 mg/L each of glucose/glutamic acid, where the nitrogen portion of the glutamic acid is not oxidized, is 172 ± 27 mg/L.*

Q: What if the G/GA result was outside the acceptable range for a given batch? Does that disqualify all data in that batch?

A: *It certainly does for all seeded bottles, and most regulators (permit managers) would say it disqualifies the entire batch.*

Q: Is there a good source of basic information that a non-chemist can use?

A: *I am a bit biased in that area, but I personally think the BOD/CBOD book offered by North Central Labs of Wisconsin (1.800.648.7836) provides easy-to-understand advice, including a troubleshooting guide for common problems.*

Trace Mercury — EPA Method 1631 Update

By Robert Benz, Environmental Express

The EPA has approved and subsequently implemented Method 1631 Revision E. North Carolina and South Carolina have joined California, Wisconsin, and Michigan in noting that Method 1631 soon will be appearing on NPDES permits. Other states are considering the same. Given these market place changes, many laboratories are now facing the decision of whether or not to venture into atomic fluorescence analysis.

Instrument Technique

Atomic fluorescence spectroscopy (AFS) is a very sensitive and selective technique for the detection of a number of environmentally important elements, including mercury, arsenic, selenium, bismuth, antimony, and tellurium. AFS is based on the radiational deactivation process which occurs after the excitation of free atoms by the absorption of radiation of a certain wavelength from an excitation source. For the analysis of Hg, the benefits of AFS over other common spectroscopy processes, such as cold vapor atomic absorption spectroscopy (CVAAS) and AES, include greater sensitivity, greater linearity, and less spectral interference.

A typical AFS instrument for EPA Method 1631 consists of three basic sections: a vapor generator, a concentrator (gold amalgamation) and an AFS detector. Gold amalgamation pre-concentrates the Hg, which is thermally desorbed and flushed into an AFS detector. Pre-concentration allows accurate and reproducible results in the sub parts-per-trillion (ppt) range.

New Sampling Requirements

In addition to new instrumentation, EPA 1631 requires the use of EPA Method 1669 for the collection of samples. This rigorous sampling method calls for a two-person sampling team, certified sampling equipment, field blanks, backup sampling materials, and close attention to the elimination of contamination and interference sources, such as automotive exhaust, rain and tobacco smoke.

Sample Preservation, Digestion and Elemental Conversion

Once samples are collected, EPA 1631 specifies sample preservation with 5mL/L of pre-screened HCl. BrCl solution is added to oxidize all Hg compounds to Hg^{+2}, and the sample is pre-reduced with NH_2OH-HCl to destroy free halogens. The sample is then reduced with SnCl, converting Hg^{+2} to Hg^0. The Hg^0 is separated from solution by Argon purging, and thereby deposited onto the gold trap of the AFS instrument.

Supplies Required for Method 1631

Environmental Express manufactures concentrated BrCl solution, pre-screened HCl, stannous chloride concentrate, and Hg standards for EPA 1631. Certifications are included. The Environmental Express HotBlock™ and SC475 digestion cups have been proven acceptable for 1631 use. PS Analytical (www.psanalytical.com) and Leeman Labs (www.leemanlabs.com) manufacture AFS instrumentation.

Cost to Implement

Costs to implement Method 1631 vary. They include: instrumentation costs (average $25,000-$30,000); sampling equipment ($1,500-$2,500); documentation, training, certification ($1,000-$3,000); creation of a clean lab environment ($0-$100,000). Frank Pasztor of Meritech Labs (Reidsville, NC) estimates his company's cost to up-fit for 1631 at $45,000 to $55,000.

Market Size and Value

The total number of samples requiring 1631 analysis is limited. An example of the market can be seen in North Carolina, which will implement Method 1631 on Sept. 1, 2003.

NC Market Size for 2004:	
Permit Holders	155
Average # of Samples per Permittee	26
Average # of QA/QC Samples per Permittee	22
Yearly Samples	7,440

Estimated NC Market Value for 2004:	*low range*	*high range*
Revenue, per 1631 sample analyzed	$65	$100
Revenue, per 1669 sampling event	$60	$150
Yearly Value	$725,400	$1,348,500

As of July 8, 2003, the NC DNR Lab Certification section states that three labs in North Carolina are certified for this parameter, with four more labs participating in the 1631 certification process. Additional information specifically on Method 1631 may be found on the EPA web site (www.epa.gov/waterscience/methods).

NELAC Requirements continued from page 1

Note: I would use the 2003 Standard, assuming a 2-3 year implementation cycle. The 2003 Standard currently is available to *Catalyst* subscribers online at www.CatalystInfoResources.com. The EPA has yet to publish it.

2. Start the process by developing a Quality Manual that addresses all of the requirements in Section 5.4.2.3 of the NELAC standard.
3. Designate a Technical Director. If necessary, use the grandfather or exemption clauses in Chapter 4 of the NELAC standard.
4. Implement the remaining requirements in Chapter 5.
5. Purchase and analyze PT samples. Use the data to identify problem areas.
6. Submit an application and associated fees.
7. Schedule the on-site assessment. Note that a laboratory has 2 years to correct any deficiencies identified in this process.

Assistance Implementing the Program

The Institute for National Environmental Laboratory Accreditation (INELA, www.inela.org) has been formed to develop training and related tools to help laboratories with the accreditation process. A committee in INELA, Small Organizations, also has been formed specifically to assist small labs.

When the NELAC effort began in 1995, its goal was one national, standardized program for all laboratories. For this to be accomplished, laboratories must recognize the benefits of the program and realize that the requirements can be met in a fiscally sound manner. For more information, go to http://www.epa.gov/ttn/nelac/.

We want to hear from you!

We hope you've found the articles in our first issue of *Express News* both interesting and informative. We plan to publish several times a year, and invite your input. Please e-mail your comments, suggestions or ideas for future articles to: paulab@environmentalexpress.com.

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In This Issue:

L A B P R O F I L E

State of Florida Multi-tasks with the AutoBlock™

Located 30 miles south of downtown Miami in the heart of the South Dade County agricultural area, is the Tropical Research and Education Center of the University of Florida. This facility is dedicated to research on the nutritional needs of plants for farmers and homeowners in the state of Florida. In a typical growing season, the research facility tests nutrient levels of truck crops, such as tomatoes, pepper and squash, as well as more exotic crops, such as papaya, guava and mango. In addition to testing plants and soil nutrient levels, the laboratory now tests water quality for natural resource monitoring. Associate Professor Dr. Yuncong Li was charged with the task of managing the new, higher workload with his existing support staff.

Laura Rosado loads samples into the AutoBlock™.

Li investigated automated digestion systems and installed an AutoBlock™ from Environmental Express to help meet his goals of increasing productivity and reducing error. "One of our focuses is low-level phosphorus testing," Li said. "The standard for discharge into the Everglades is only 10ppb, and to get the RSD we need to report at that level, we have to standardize the digestion procedure and avoid human error." The digestor allows his lab to adhere to NELAP standards in providing precise soil and water nutrient data to ensure a safe and healthy environment. According to Li, "Our lab has digested nearly 500 soil and water samples for both total phosphorus and total metals, and has obtained a 98% recovery with a 9% RSD." Li said that the method storage capability of the AutoBlock™ makes it easy to switch from one digestion procedure to another without operator error while maintaining analysis uniformity.

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The Tropical Research and Education Center is located on 160 acres complete with greenhouses, orchards and vegetable fields. To learn more about the center and its work, visit http://ifas.ufl.edu/.