Wisconsin Department of Natural Resources

The Newsletter of the Wisconsin Laboratory Certification and Registration Program

LabNotes *Spring 2008*

Touchdown! NR149 crosses the goal line.

On October 24, 2007, the Natural Resources Board voted unanimously to approve promulgation of revisions to ch. NR149, the Wisconsin Administrative code which governs the chemistry segment of environmental laboratory. On February 18, 2008, the rule cleared Wisconsin's legislature. This action culminates a lengthy effort to bring Wisconsin's laboratory certification regulations in line with national standards and those adopted by other states.

It may have taken a long time to reach this point, but sometimes it takes a long time to get something right. With a code change of this magnitude, we needed to get this right from the start. "Getting it right" required a game plan the likes of which Bill Belichick would want to purloin. Bellicose Bill may have his staff of coordinators, position coaches, and camera operators; but we relied on a lot of hard work by program staff, the Certification Standards Review Council, and other key stakeholders that made up the Rule Advisory Committee (RAC) to come up with a draft version for public hearings.

The next phase of the gameplan was to solicit public comment from all of the other stakeholders in the program—you, the laboratory community. We received a substantial volume of comments, but that's a good thing. Responding to those public comments allowed us to tweak NR149 into a winning formula. The net result of the effort was to find compromise that allows us to update our rules while offering flexibility to the laboratory community with respect to how to comply with the new rules. We also incorporated a delayed effective date—September 1, 2008 in order that we can provide outreach to help laboratories prepare for the changes ahead. In addition, we are dedicating a large portion of this edition of LabNotes to the changes in NR149.

Also in this Issue:

Contact us at: (608) 267-7633 or LabCert@Wisconsin.gov
Web Site: www.dnr.state.wi.us/org/es/science/lc/

Exams, Meetings & Training Opportunities

Operator Certification Exams

DNR will hold Wastewater, Drinking Water, and Septage Operator Certification (OC) exams on May 7, 2008 (postmark deadline April 9, 2008) and November 5, 2008 (postmark deadline October 9, 2008) in DNR Regions around the state. Check the Operator Certification web site for details, as they become available. The DNR's Central Office in Madison will send an exam application 3 months prior to the upcoming exam date to those operators that have taken an exam(s) in the last 3 exam cycles.

www.dnr.state.wi.us/org/es/science/opcert

Training for Lab Analysts

* Wastewater Lab-Intro *

March 18-20, 2008

Stevens Point

Wastewater Training Solutions (Dan Tomaro)

www.wastewatertrainingsolutions.com

* Wastewater Lab-Advanced *

April 1-2, 2008

Fond du Lac

Wastewater Training Solutions (Dan Tomaro)

www.wastewatertrainingsolutions.com

* Math for Wastewater *

April 3, 2008

Fond du Lac

Wastewater Training Solutions (Dan Tomaro

Check the DNR OpCert Training Calendar

www.dnr.state.wi.us/org/es/science/opcert/training.pdf

LabNotes

Newsletter of the Laboratory Certification Program

LabNotes is published twice annually by the Wisconsin DNR Laboratory Certification and Registration Program. For information about distribution or to make suggestions for future articles, contact the editor.

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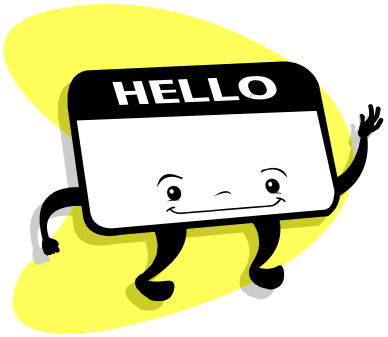
2008 Conferences & Meetings



Government Affairs Seminar

The Government Affairs Seminar (jointly sponsored by Wisconsin DNR, the Wisconsin Section of the Central States WEA, Wisconsin Wastewater Operators Association, Municipal Environmental Group and Wisconsin League of Municipalities) will be held February 28, 2008 at the Marriott in Madison. To register, call 608-263-1672

FET's Environment '08 Conference

The Federation of Environmental Technology's (FET) annual conference will be held March 10-12, 2007 at the Wyndham Milwaukee Airport & Convention Center, in Milwaukee.

www.fetinc.org/

26th Annual Spring BioSolids Symposium

The Spring BioSolids Symposium will be held March 18, 2008 at the Country Springs & Convention Center (fka Holiday Inn) in Stevens Point. Joint sponsored by DNR, CSWEA, WWOA and UW-EX No contact information available.

Rural Water (WRWA) Association

The Wisconsin Rural Water Association holds its 20th annual technical conference April 8 - 11, 2008 at the Green Bay Regency Suites and KI Convention Center complex. Call (715) 344-7778 or visit their web site for more information.

www.wrwa.org

Central States Water Environment Association

The Central States Water Environment Association's (CSWEA) 81st annual meeting is scheduled for May 19-22, 2008 at the Sheraton Hotel in Bloomington, Minnesota.

www.cswea.org

Wisconsin Water Association

The Wisconsin Water Association (formerly AWWA WS) 87th annual conference is scheduled for September 17-19, 2008 in Stevens Point. Contact Jack Albrechtson at (608) 831-6554 for more information.

www.wih2oassoc.org

Wastewater Operators Association (WWOA)

The 43rd Wisconsin Wastewater Operators Association annual conference will be held September 30 through October 3, 2008 at the Holiday Inn in Stevens Point. Check the WWOA web site for more details.

www.wwoa.org

Council Corner

By Katie Edgington, Council Chair

My second term concludes in July, which means my tenure is unfortunately coming to an end. It was a good time to serve on the council. Over the past years, many positive things have happened in the laboratory certification/registration program. This is a timely opportunity to reflect on the accomplishments over the years.

When I began, the NR 149 revision process had already begun. No one thought this would be an easy task. This is the most technical administrative code which addresses a variety of stakeholders. The makeup of labs can vary from a large commercial lab which serves hundreds of clients to the small wastewater lab where the operator must wear the hats of lab tech, plant operator, and snow plow driver (especially this past winter). The revision process was long and arduous to both the lab certification/registration program staff and the stakeholders; however, the final product is one that all can embrace. I commend the program staff for thoroughly addressing the concerns of the stakeholders during the public comment period. The code has a delayed implementation of September 1, 2008; so, I strongly encourage all to take advantage of the many outreach opportunities that the DNR is offering to roll out the new code. The website is a great place to start.

Judy Tholen appointed to (Laboratory) Certification Standards Review Council.

Hi everyone. I am pleased and honored to announce that I have been selected to represent the Small Municipal Plant Labs on the Laboratory Certification Council. My predecessor, Randy Herwig, did an outstanding job during his tenure on the Council, especially since most of it was spent debating NR149 proposals, commenting on NR 149 drafts, and recommending changes to NR149. During the next three years I can only hope to match his dedicated efforts to protect and verbalize the interests of our small municipal labs.

For those of you who may not know my background, I'll fill you in. My humble start in wastewater was basically a money issue….I needed a job during my final two years of college. A professor suggested I try the local wastewater treatment plant lab. Lucky for me, Whitewater WWTP hired me as assistant lab technician. I found myself enjoying the work so much (or maybe I needed more money), that I was hired as assistant lab technician for the Walworth County Metropolitan Sewerage District. I alternated weekends between the plants to help pay for college. Well by graduation, I was hooked. So off to Fort Atkinson WWTP I go as …..guess what? An assistant lab technician! However, after a couple of years I was able to transfer to a different area for more wastewater operations experience. Biosolids.

I really did enjoy driving those trucks amongst other duties. During my years at Fort Atkinson I was involved on the WWOA Board, served on the NR204 biosolids regulation revamp committee, and served on the proposed nutrient trading committee which stemmed from the Rock River Partnership. As if this wasn't enough experience, I decided to see what life was like on the private side of things. It was a positive experience working for companies that serviced our small wastewater plants. But my true heart was in my beginnings, the lab. So here I am at Watertown WWTP, but this time not as an assistant, but the Lab Manager. Whoopee!

But seriously, after 20+ years of working for and working with our small wastewater facilities, I feel I can and will represent and protect our small labs' interests by making your voice(s) heard to the LabCert Council.

Current Council Members

Representation	Name	Phone# / e-mail
Small Municipal Wastewater Plant	Judy Tholen	(920) 262-4085 judy.tholen@ci.watertown.wi.us
Large Municipal Wastewater Plant	* vacant *	---------- ----------
Public Water Utility	Katie Edgington *(Chair)*	(608) 755-3115 edgingtonk@ci.janesville.wi.us
Demonstrated Interest in Lab Certification	Chris Groh	(715) 344-7778 cgroh@charter.net
State Laboratory of Hygiene	Susan Hill *(Secretary)*	(608) 224-6282 hill@mail.slh.wisc.edu
Industrial Laboratory	Steve Jossart	(920) 438-2898 steve.jossart@GAPAC.com
Commercial Laboratory	David Kliber *(Vice-Chair)*	(414) 475-6700 david.kliber@sflabs.com

Council information and meeting information on the web:
www.dnr.state.wi.us/org/es/science/lc/contacts/council/

FAQ – Frequently Asked Questions regarding changes to NR149

Where can I obtain a copy of the new rule

The revised ch. NR149 can be obtained here:

www.dnr.state.wi.us/org/es/science/lc/NR149.pdf

The official version of the final rule will also be printed in the Wisconsin Administrative Register. Because of the size of the rule, it will likely appear in either the April 1, 2008 or April 15, 2008 edition of the Wisconsin Administrative Register available at:

(http://www.legis.state.wi.us/rsb/regindex.htm).

Where can I find information updates?

Information updates regarding the rule will be posted here:

www.dnr.state.wi.us/org/es/science/lc/RULES/NR149/149update.htm

Where can I find outreach or training information?

Training tools and documents related to the rule will be posted here:

www.dnr.state.wi.us/org/es/science/lc/RULES/NR149/149update.htm

When do I have to have all these changes made?

The rule includes a delayed effective date of September 1, 2008. As of that date, labs will be expected to have implemented the changes required.

Should I keep a copy of the old NR149?

You should. Remember that on-site evaluations are performed every 3 years, and the scope of the audits is to review data generated and practices in place for the prior 3 years. Consequently, until September 1, 2011 (*at which point all data will have been generated in accordance with the 2007 revisions*), all on-site evaluations will include a review of protocols under the existing NR149 rule (for data generated prior to 9/1/2008) as well as protocols established under the 2007 revisions.

Am I really exempt from having to run matrix spikes & replicates?

Very likely you are, particularly if your facility is a small wastewater treatment plant lab. The revised NR149 now requires analysis of laboratory control standards (LCS) to evaluate laboratory performance. If the approved method you use does not specify the analysis of matrix spikes and or replicates (as nearly all Standard Methods procedures) then you will only need to analyze LCS samples. Note also that if required by a QAPP or by a client, you may still have to analyze spikes and replicates.

There are many new analytes and technologies available for certification. How can I add these to my certification?

Because the rule does not take effect until 9/1/2008, additional parameters cannot be added until that time. Certificates for the 9/1/08 to 8/31/09 certification period will initially represent a conversion of your existing certifications to the new structure. We will accept applications beginning 9/1/2008 to add new technologies and analytes to your scope of accreditation.

What outreach efforts are you planning?

Current outreach efforts include the following:

- 2/14/08 WWOA NC mtg (Antigo, WI)
- 2/28/08 Government Affairs (Madison, WI)
- 4/24/08 Wisconsin Environmental Lab Assoc. (WELA) forum (Madison, WI)
- 5/22/08 WWOA Joint LM/SO mtg (Berlin, WI)
- (TBA) April-June. 1-day sessions around the state through WWOA and WRWA
- (TBA) Summer statewide forum (WELA)
- 9/30/2008 WWOA conference (Stevens Point, WI)

Will you develop hardcopy materials and templates to help labs comply with the new rule?

Yes. We are currently working on a dynamic "Implementation Guidance" document, which will replace former "Program Guidance" and "QA Manual for a Small WWTP Lab" documents. This documented is intended to clarify provisions of the rule and to provide templates and spreadsheets wherever possible.

Where should questions about the rule be directed?

Direct any questions about the rule content, outreach efforts or comments regarding specific outreach materials you would like to see, to either Rick Mealy [(608) 264-6006; Richard.Mealy@Wisconsin.gov] or David Webb [(608) 266-0245; David1.Webb@Wisconsin.gov].

NR149 Program Structure Changes

From its inception in 1986, the Laboratory Certification & Registration Program has used a "test category-test" certification structure. In addition, there were basically two certifications, one for drinking water (test category 18), and the other to cover virtually all other matrices (all categories except 18). With the 2007-2008 revisions to NR 149, we are changing to a 3-tier structure which begins with matrix. Certification matrices offered will be Aqueous, Solid, and Drinking Water. **The complete 3-tier structure for Aqueous and Solid matrices will be "matrix—technology—analyte (*or analyte group*)". For Drinking Water, the structure will be "matrix—method—analyte (*or analyte group*)".** The EPA required states to certify by individual methodology for drinking water parameters several years ago, so for labs testing drinking water, program structural changes will be more cosmetic.

What does this change mean for labs? For small wastewater labs analyzing only wastewater samples, your certificate will look different, but it should be a relatively seamless transition. The changes will be most noticeable for those labs that analyze both aqueous and solid (e.g., soil, sediment, dewatered sludges, waste) samples. Where the existing certification allowed analysis of both aqueous and solid samples if you maintained certification or registration for a given test category (again, other than category 18), now labs will have to maintain specific certification to perform testing on solid matrices.

Labs will only see a significant difference if either they analyze solid samples as well as aqueous samples, or if they choose to be certified to perform multiple analytical technologies for a given analyte (or analyte group). The best example of this would be to consider a lab that is currently certified to analyze lead. The lab analyzes both waters (aqueous) and solids**. Under the "old" rule, the lab would need to be certified for the test "Lead" under test category 08, "Metals I".** Pretty simple.

Note that this example covers *ONLY* lead. For a large-scale commercial laboratory performing multiple technologies for a broad spectrum of analytical parameters, this certification array can become quite complex. In this case, the lab went from one generic certification for lead to 6 specific certifications covering two matrices. In addition, maintenance of these certifications each year will require some a similar array of PT samples. This will be discussed in greater detail in the "Changes to PT Requirements" section.

For a small wastewater lab analyzing BOD, TSS, ammonia (by ion-selective electrode), and total phosphorus (colorimetric), the change will look something like:

EXISTING NR149 CERTIFICATION/REGISTRATION	**9/1/08 REVISIONS TO NR149 CERTIFICATION/REGISTRATION**
	Matrix: Aqueous
Test Category 01 Oxygen Utilization	**Technology: Oxygen Demand assays (BOD or cBOD)**
Biochemical Oxygen Demand	Analyte: Biochemical Oxygen Demand (BOD)
Test Category 02 Nitrogen	**Technology: Electrometric Assays**
Ammonia	Analyte: Ammonia as N
Test Category 03 Phosphorus	**Technology: Colorimetric or Nephelometric**
Total Phosphorus	Analyte: Total Phosphorus
Test Category 04 Physical	**Technology: Gravimetric Assays – Residue**
Total Suspended Solids	Analyte: Total Suspended Solids

For aqueous samples, a lab wishes to be certified to perform lead testing by graphite furnace atomic absorption (GFAA), flame atomic absorption (FLAA), ICP, and ICP/MS. For solid matrices, the lab generally will use flame AA or ICP. Under the new rule, the lab might choose to establish the following certification array:

CERTIFICATION
Matrix: Aqueous
Class: Metals
Technology: FLAA
Analyte: Lead
Technology: GFAA
Analyte: Lead
Technology: ICP
Analyte**:** Lead
Technology: ICP/MS
Analyte: Lead

CERTIFICATION
Matrix: Solid
Class: Metals
Technology: FLAA
Analyte: Lead
Technology: ICP
Analyte: Lead

NR149 Fee Structure Changes

NR149 Fee Changes

As with most other aspects of NR 149, changes related to program fees are mostly directly related to program structural changes. The Laboratory Certification & Registration Program operates under a simple balanced budget. Fees are collected to equal the cost of running the program each year. Each test category is assigned a number of "relative value units" (RVU) that roughly equate to the technical difficulty associated with performing tests under that test category. The cost to run the program for a given certification period is simply divided by the total number of RVUs for the labs in the program to obtain an individual RVU cost. The DNR's Natural Resources Board approves the Lab Certification & Registration Program's budget annually in March. Lab fees are then assessed each May by multiplying the number of lab RVUs by the cost per RVU.

While the basic RVU process for determining fees is not changing, the change from category—test structure to a matrix—technology—analyte system required us to establish RVUs for each technology, rather than each test category. This allowed us to build in greater equity. One example was the BOD test. BOD has historically been considered an "entry-level" test in many labs, whereby entry level analysts are assigned BOD testing. In practice, however, BOD is more of a bioassay than a "bucket chemistry" type of test, and the Standard Methods procedure requires a significant level of detail and procedure to perform the test correctly. Consequently, we have increased BOD from 1 RVU to 3 RVU to reflect the difficulty level associated with performing this test correctly.

Transitioning: Fees for FY09 (9/1/08 - 8/31/09)

Since the revisions to NR149 do not take effect until September 1, 2008 and fees for the September 1, 2008 to August 31, 2009 certification period must be invoiced in May of 2008 (due July 1, 2008**) the program will assess fees based on the category—test certification structure as it exists currently**. As this issue went to press, the proposed fee schedule slated to go before the Natural Resources Board in March was $68.00 per RVU.

NR149 PT Changes

The 2007-08 changes to NR 149 incorporate both a relatively minor timetable change and then procedural changes to match program structural amendments. The schedule change affects the deadline for submitting PT results to complete annual certification/registration renewal. Historically, with the certification year (and new certificates printed) beginning September 1 of each calendar year, PT sample results were required to be submitted prior to the close of business on August 31. **With the changes to 149, this deadline is moved a little more than 2 weeks back to August 15**.

This may seem like a small change, but we typically receive a large volume of "last minute" PT results required to "fill a hole". As much as 10% or more of laboratories routinely find themselves submitting results for one or more parameters in the days leading up to the new certification year. This change will require labs to pay closer attention to their PT sample needs and minimize the time that we have to spend generating new and replacement certificates in the last two weeks of a certification period.

The most significant change for PTs relates to the new "2nd tier" (technology) of certification program structure and is the **requirement to analyze a PT for each analyte AND technology**. This change does not affect smaller wastewater treatment plant type labs that only perform a single technology and matrix. And many larger laboratories that perform testing across the nation are already meeting this requirement.

Higher PT costs?

Does this mean a lab needs to purchase a separate PT sample for each analyte /technology combination? **NO.** Most PT samples, upon preparation, provide sufficient sample volume to perform multiple analyses. While unique PT results are required for each analyte/technology combo, Revisions to NR 149 allow a laboratory to separate a given PT sample into multiple aliquots and then analyze each aliquot by a different technology.

NR149 QC Changes

As with everything else, in the 10 years since the last revision to NR149, many things have changed in the world of quality control for environmental testing. This rule change incorporates concepts that keep us in pace with national trends.

Quality System

This subchapter contains the core of operational requirements that laboratories need to follow. It includes:

- provisions for demonstrating capability of analysts to perform testing (offering flexibility in options),
- requirements for content of quality manual and standard operating procedures,
- types of documents to be maintained,
- method selection protocols,
- equipment considerations,
- traceability of standards and reagents,
- handling of samples,
- required information for test reports, and
- essential quality control requirements.

The subchapter offers in most cases alternative means to comply with the stated requirements. Laboratories should be able to select alternatives that match their needs from the choices given.

Initial Demonstration of Capability

The subchapter does not specify requirements for education, experience, or training of analytical personnel. It requires all analysts to complete an initial demonstration of capability **ONLY** *for any tests they perform in which the referenced methodology specifies a procedure for demonstrating capability*. If referenced methods do NOT specify a procedure for demonstrating capability (e.g. Standard Methods procedures for BOD, TSS, ammonia by ISE, and manual colorimetric phosphorus), then the laboratory is required to develop and document their own internal procedures used to determine that a given analyst has demonstrated the ability to produce quality analytical results.

This approach offers flexibility necessary for the concern to be addressed by laboratories of all types and sizes. The critical requirement is that laboratories have some protocol in place to ascertain that a given employee is qualified to perform a given test in a manner consistent with data quality needs. A whole range of options is available, and any option will satisfy the requirement.

Quality Manual

Formerly termed "Quality Assurance Manual", now known as the "Quality Manual". Note, however, that the code allows a lab to name the manual however they choose, but it will be referenced as the "quality manual" in any program related documents. The single most significant change is that the code language now specifies that labs must not only have a Quality Manual, but they are required to adhere to it.

Since the program originated, NR 149 has really only specified that laboratories *have* a quality assurance document. There were no codified requirements for what the QA document should or must contain, or even that, once developed, the labs be accountable for adhering to the document. Clearly, it only follows that if a lab is going to generate a "quality manual", that manual must serve as the basis for performing all lab activities including sample analysis. What these revisions do is to clearly state that requirement.

The revision allows flexibility in the format of the manual as long as it addresses a set of content elements. The content elements, for the most part, are items that are customarily included in existing quality assurance plans. The quality manual shall include, address, or refer to, at a minimum, the following elements:

- Organization and management structure of the laboratory.
- Procedures for retention, control, and maintenance of documents used in or associated with analyses.
- Procedures for achieving traceability of standards, reagents, and reference materials used to derive any results or measurements.
- Procedures for handling samples.
- Lists of major analytical instruments and support equipment.
- Procedures for calibration, verification, and maintenance of major analytical instruments and support equipment.
- Procedures for evaluating quality control samples, including, but not limited to, method blanks, laboratory control samples, matrix fortified samples and replicates.

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- Procedures for initiating, following up on and documenting corrective action addressing quality assurance and quality control failures, discrepancies or nonconformance.
- Procedures for reviewing analytical data and reporting analytical results

Manuals must be kept current and the revision dates must be documented within the manual.

The section also instructs laboratories that are analyzing drinking water to follow the content elements in the EPA's "Manual for the Certification of Laboratories Analyzing Drinking Water".

Standard operating procedures (SOPs)
This section requires laboratories to maintain written procedures that can consist of copies of published documents, annotated published documents with modifications, or in-house written procedures. SOPs have become a staple in labs over the years; but they are new to NR149.

The section also recognizes analytical methods as a subset of the standard operating procedures of a laboratory. Unless the laboratory follows a referenced procedure "to the letter" an addendum is required indicating any differences between the laboratory's procedure and the referenced method. When a laboratory decides to create its own written procedures, the section describes the content elements that need to be addressed.

Laboratory equipment
This section introduces the concept of splitting equipment into two types: support equipment (e.g., ovens, balances, thermometers) and analytical equipment (e.g., DO meters, ion meters, spectrophotometers, GCs). Procedures for verifying the accuracy of support equipment are included, such as verifying the calibration status of balances, thermometers, and auto-pipets.

Requirements for calibration of analytical instruments are discussed in detail, something that was lacking in previous versions of the rule.

Calibration
Since the program originated, NR 149 has really only specified "*thou shall calibrate to maintain accuracy*". Yet, over the years, labs have struggled with the details of the calibration process:

- ▸ which calibration function should I use? (linear?, quadratic?, cubic?, point-to-point?)
- ▸ How many calibration points do I need?
- ▸ What levels should I choose for the calibration standards?
- ▸ Can I (should I) use weighting? *[Maybe]*
- ▸ Can I (should I) force the calibration through the origin? *[No]*
- ▸ How do I evaluate the resultant calibration function?
- ▸ How long does a calibration curve last? (when do I need to do another?)

Answering all these questions certainly expanded the volume of NR149, but in this case, more language is a good thing, because the language now provides clarity of requirements.

Calibration is broken down into initial (full) calibrations and then the process of verifying that an existing initial calibration remains valid *("continuing calibration verification" or CCV*). A maximum one-year time limit is established between initial calibrations.

The initial calibration sequence is laid out logically:

- select a calibration function,
- determine the number of calibration levels and specific concentrations for them,
- identify any additive functions, such as weighting factors
- evaluate the calibration result

This section also introduces the concept of using second source standards to evaluate an initial calibration.

Second source standards
A "second source" standard is either a standard purchased from an alternate vendor, or one purchased form a single vendor but from a different lot than the one designated for use in generating calibrations. Second source standards are then used to prepare standards used to verify an initial calibration. As long as initial calibrations are verified using 2nd source standards, the requirement to (*purchase and*) analyze QCS (blind standards) is waived. From a strict cost comparison and workload perspective, second source standards are a logical advancement.

Measurement traceability
Laboratory must ensure that results of analyses can

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be linked to all the standards and reagents used to derive results.

In addition, laboratories need to document the identity, source, and purity of all standards and reagents used in tests methods performed. Why? Because it's only logical that if a critical reagent is associated with a specific "shelf-life", then we need documentation that the shelf-life wasn't exceeded.

If standards or reagents are accompanied by a certificate of analysis, these records must be retained when necessary to establish the identity, source, or purity of standards and reagents. Other specific reagent traceability documentation required includes:

1. Original containers of standards and reagents must be labeled with a receipt and an expiration date.

2. Laboratories need to document the lot number, manufacturer, date of receipt, and the date of expiration of stock standards and reagents separately from their containers to ensure this information will be retained when the containers are discarded. (*This really means maintaining some sort of reagent logbook, which we've been discussing during audits and training sessions for some time now*.)

3. Laboratories need to maintain records detailing the preparation of intermediate and working standards and reagents. These records must link the intermediate and working standards and reagents to their respective originating stocks or neat compounds.

Spikes & replicates out (*in many cases*); LCS in

The 2007 revisions to NR149 represent a quantum leap forward in terms of the quality control (QC) samples required to evaluate the data quality of a given analytical batch. The major difference is a philosophical change from basing sample batch quality evaluations on matrix QC (matrix spikes, replicates) to a system that evaluates batch quality on a sample that is free of matrix interference (Laboratory Control Standard or LCS).

New rules only require the analysis of matrix spikes &replicates (or matrix spike duplicates) if:

- ***The test method you are using (referencing) require their analysis***. [NOTE: *The lab must also have received sufficient quantity of sample necessary prepare them*].
- Sample analysis is being done in accordance with a ***project plan that requires their analysis***.
- ***By choice***: a lab may choose to use them in place of LCS to evaluate the level of control of an analytical system.

Blind standards ⇨ **QCS (**Quality Control Standards)

QCS become optional in most cases. Analysis of blind standards has historically been required, at specified intervals, 3 times a year. QCS samples are essentially PT samples for which the lab receives the "true values" and acceptance criteria in advance. These standards are both costly and represent a workload burden.

First we re-named "blind standards" to the more widely used term, Quality Control Standards (QCS). Then **we waived the requirement to analyze blind standards for any laboratory that opts to incorporate the use of "second source" standards in its initial calibration verification procedures**. For analyses such as total phosphorus, the need for labs to purchase (or prepare) stock standards used to generate calibrations is nothing new. Now all you need to do is purchase a 2nd standard from a different lot, or from an alternate vendor, and you can forego paying the cost of QCS.

Control limits – a new approach

Let's be honest; the task of collecting data and updating control limits doesn't rate very high on anyone's list of the most enjoyable aspects of lab analysis. Consequently, with these revisions to NR 149, we included the ability for the LabCert Program to establish acceptance criteria for the analysis of LCS, matrix spikes (if required), and replicates (if required). In the past, we codified acceptance criteria for the analysis of "known standards" (± 10% of true value for inorganic parameters; ± 15% of true value for organic parameters.)

Laboratory control samples are identical to the former "known standards" in terms of their make-up, so it makes a lot of sense to use similar fixed control limits with which to evaluate them. It is likely that the program will establish specific, fixed acceptance criteria for LCS samples. **If these limits are established, the limits will be communicated to the laboratory community and posted on the LabCert website.**

SOPs – a "new" requirement

Those of you in larger, commercial laboratories are likely thinking, "SOPs? New? But….we've had them for years." Standard operating procedures (SOP) have indeed become a staple of any quality system over the past 10 years. Prior to this revision to NR149, however, a search of the code for "SOP" would have come up empty. So, while the concept may not be new, the requirement to have SOPs is a new one.

Listed below is a distillation of specific SOPs which are now required under NR149. This list should be considered minimum requirements. Certainly other SOPs can and should be developed; but these are SOPs clearly specified by rule and ones which your auditor will be looking for once audits under the new rule begin in September 2008.

SUBJECT	SOP Required	NR149 ref.
CALIBRATION	Procedures for calibration, verification and maintenance of major analytical instruments and support equipment.	149.37(3)(f)
EQUIP MAINTENANCE	The laboratory shall establish procedures for the maintenance of analytical instruments to prevent contamination or deterioration that may affect reported results.	149.44(4)(b)
ANALYTICAL METHOD SOPS	Laboratories shall maintain written standard operating procedures that document or reference activities needed to maintain their quality systems and that enable performing or reproducing an analysis in its entirety as performed at the laboratory.	149.40(1)(a)
QC: BLANKS & ZEROING	The laboratory shall establish procedures for zeroing an instrument and the treatment of calibration blanks, when the referenced analytical method used by the laboratory requires the response of a calibration blank to be part of a calibration function.	149.44(6)(h)
QC: CONFIRMATION OF ORGANIC ANALYTES	The laboratory shall establish procedures to confirm the results of organic analytes determined by techniques that, unlike mass spectrometry, do not provide a positive unique identification when… *1. The history of a sample source does not suggest the likely presence of the detected analyte.* *2. A client or approved project plan requires it.)*	149.48(9)(a)
QC: CORRECTIVE ACTION	Procedures for initiating, following up on and documenting corrective action addressing quality assurance and quality control failures, discrepancies or nonconformance.	149.37(3)(h)
QC: LOD & LOQ	Laboratories shall establish procedures to relate limits of detection to limits of quantitation.	149.48(2)(f)
QC SAMPLES	Procedures for evaluating quality control samples, including, but not limited to, method blanks, laboratory control samples, matrix fortified samples and replicates.	149.37(3)(g)
QC SAMPLE BATCH REQUIREMENTS	Laboratories shall establish procedures for identifying and documenting preparation batches that facilitate determining compliance with the frequencies of quality control samples required by this subchapter.	149.48(1)(d)

continued from page 11

SUBJECT	SOP Required	NR149 ref.
RECORDS	Procedures for retention, control and maintenance of documents used in or associated with analyses.	149.37(3)(b)
RECORDS	Procedures to control and manage all records and documents that form part of its quality system and that are required to demonstrate compliance with this chapter. *The procedures shall ensure that documents required to perform analyses and to ensure the quality of generated data are available to laboratory personnel, and that records and documents are reviewed periodically for continuing suitability and, when necessary, revised to facilitate compliance with the requirements of this chapter.*	149.39(1)(a)
RECORDS	Procedures to prevent unauthorized access or amendments to records and documents.	149.39(1)(g) 4.
REPORTING	Procedures for reviewing analytical data and reporting analytical results. *(Required to be in Quality Manual)*	149.37(3)(i)
REPORTING	The laboratory shall establish procedures and rules for reporting results for samples analyzed by dual column and dual detector systems. *These procedures must declare:* *(1)Under what conditions a presumptive identification is confirmed.* *(2) Under what conditions a presumptive identification is reported.* *(3) The value that will be reported when the dual systems both provide quantitative confirmed results*	149.48(9)(b)
SAMPLE HANDLING	**Sample Acceptance Policy.** The laboratory shall have and follow a written policy that clearly outlines the conditions under which samples will be accepted or rejected for analysis, or under which associated reported results will be qualified.	149.46(2)(a)
SAMPLE HANDLING	Procedures for handling samples. *(Required to be in Quality Manual)*	149.37(3)(d)
SAMPLE HANDLING	The laboratory shall establish and follow procedures for identifying samples uniquely. *(The procedures shall ensure that the identity of samples cannot be confused physically or when referenced in records or other documents.)*	149.46(3)(a)
SAMPLE STORAGE	The laboratory shall have procedures and appropriate facilities for avoiding deterioration, contamination, loss or damage of samples during storage.	149.46(6)(a)
SAMPLE CONTAINERS	*When the laboratory provides containers and preservatives for sample collection, including bulk sampling containers such as "carboys",* the laboratory shall have standard operating procedures in place which address concerns that the containers are adequately cleaned and not contributing to contamination of samples, do not contain analytes of interest at levels which will affect sample determinations and that the preservatives used are sufficiently pure to maintain the validity of reported results. *NOTE:* The laboratory should establish procedures to ensure and document that the sample containers it provides do not contribute contaminants before they are used for collecting samples.	149.46(1)(b)
TRACEABILITY	Procedures for achieving traceability of standards, reagents, and reference materials used to derive any results or measurements. *(Required to be in Quality Manual)*	149.37(3)(c)

SOPs – Can I just reference a specific method?

NR149 does allow a laboratory to substitute a referenced method as its SOP. In order to do so, however, the lab must include an addendum that indicates any modifications made to the referenced method and include clear protocols where the referenced method offers multiple options.

To provide an example, we have re-printed USGS procedure I-3765-85 for total suspended solids (TSS), which is available online at http://infotrek.er.usgs.gov/pls/nemi_pdf/nemi_data.download_pdf?p_file=1274.

While this is certainly one of the less complex methods, it lends itself well as an example of how labs can quite easily develop an addendum which will allow them to satisfy the NR149 requirements for method SOPs.

The "addendum" below clarifies differences between a lab's procedures and the USGS authoritative reference and satisfies the requirements of NR149 revisions.

Solids, residue at 105°C, suspended, gravimetric

Parameter and Code:
Solids, residue at 105°C, suspended, I-3765-85 (mg/L): 00530

1. Application
This method may be used to determine the suspended-solids concentration of any natural or treated water or industrial waste.

2. Summary of method
2.1 Suspended solids are those that are retained on a glass-fiber filter. The determined value is fairly representative of the sample but does not accurately represent the suspended sediment concentration of a stream; suspended solids values should not be confused with sediment concentration, which is the more accurate measure of material in suspension.
2.2 The unfiltered sample is mixed thoroughly and an appropriate volume is rapidly poured into a graduated cylinder. The suspended solids are collected on a glass-fiber filter, and the insoluble residue is dried and weighed.

3. Interferences
Precipitation in the sample during storage, such as iron, will produce erroneously high results.

4. Apparatus
4.1 *Desiccator,* charged with indicating silica gel or other efficient desiccant.
4.2 *Filtration apparatus,* consisting of suction flask, gooch crucible, glass-fiber filter disk, and suitable holder.
4.3 *Oven,* 105°C, uniform temperature throughout.

6. Procedure
6.1 Shake the sample bottle vigorously and rapidly pour a suitable volume into a graduated cylinder. Record the volume.
6.2 Quantitatively collect the suspended material from the sample on a tared glass-fiber filter disk. A blank should be determined with each set of samples.
6.3 Wash the suspended material on the filter sparingly with demineralized water.
6.4 Dry the residue and filter disk overnight at 105°C.
6.5 Cool in a desiccator and weigh the filter disk containing the dry residue to the nearest 0.1 mg. Record the weight.

7. Calculations
7.1 Apply a correction for any loss shown by the blank.
7.2 Determine suspended solids in milligrams per liter as follows:

$$\text{Suspended solids, mg/L} = \frac{1000}{\text{mL sample}} \times \text{mg residue}$$

8. Report
Report solids, residue at 105°C, suspended (00530), concentrations as follows: less than 1,000 mg/L, whole numbers; 1,000 mg/L and above, three significant figures.

9. Precision
Precision data are not available for this method.

Reference

Guy, H. P., 1969, Laboratory theory and methods for sediment analysis: Techniques of Water-Resources Investigations of the U.S. Geological Survey, book 5, chapter C1, 58 p.

Addendum to SOP # TSS (I-3765-85)

ACME Labs follows USGS method I-3765-85 for the analysis of TSS with the exception of the following modifications or clarifications where the method presents flexibility in terms of options.

- 4.3, 6.4 - Oven temperature is allowed to be 103-105 ºC (104 ± 1 ºC).
- 6.1 - Generally use 500 mL for effluent, 25 mL for influent. Sample is stirred continuously with magnetic stir bar until sample aliquot is removed.
- 6.2 - Blanks are not analyzed, an exemption afforded in ch. NR 149.14 (3) (d)
- 6.4- Samples are dried overnight (at least 8 hours). The laboratory performs a verification of drying effectiveness quarterly as per DNR letter (May 2001).
- NOTE 1. Preservation requirement is samples not to exceed 6 ºC and not to be frozen.
- NOTE 2. The lab uses filters, deemed to be equivalent to method specifications, from North Central Labs.
- NOTE 3. Use 3 x 25-mL portions of reagent water to wash filters.
- NOTE 4. Filters are subjected to a triple final wash using 20, 20, and 10 mL of reagent water.

EPA pushes closer to a new standard for determining LODs

An EPA FACA committee released its final report on Detection and Quantitation in December 2007. Tom Mugan, of the DNR's Watershed Management Program, is a member of the FACA. The report affects NPDES permitting and how WQBELs (Water Quality-Based Effluent Limitations) below detection should be handled.

15 objectives related to "What We Need A Procedure To Do" were identified. The term "limit" is used generally to refer to Detection and Quantitation Limits since the Committee had not yet defined them:

1. Does the procedure provide an explicit estimate of bias at LQ for limits that must be verifiable by labs at those limits?
2. Does the procedure provide an explicit estimate of precision at LQ for limits that must be verifiable by labs at those limits?
3. Does the procedure provide an explicit false positive rate for LC?
4. Does the procedure provide an explicit false negative rate at LC for the true value at LD or LQ that must be observed in labs at LC for the estimated values of LD or LQ?
5. Does the procedure provide that qualitative identification criteria defined in the analytical method are met at the determined Detection and Quantitation Limits?
6. Does the procedure adequately represent routine variability in laboratory performance?
7. Does the procedure perform on-going verification of estimates?
8. Is the procedure capable of calculating limits using matrices other than laboratory reagent grade water?
9. Does the procedure use only data that results from test methods conducted in their entirety?
10. Does the procedure explicitly adjust or account for situations where method blanks always return a non-zero result/response?
11. Does the procedure explicitly adjust or account for situations where method blanks are intermittently contaminated?
12. Is the procedure clearly written with enough detail so most users can understand and implement it?
13. Is the procedure cost-effective?
14. Does the procedure assess multi and inter-laboratory variability when data from more than one laboratory is used?
15. Is the procedure applicable to all users and test methods?

The report can be downloaded at
http://www.epa.gov/waterscience/methods/det

Lab-doku

A laboratory twist to the popular logic-based number placement puzzle.

			ICP		HPLC			
				GRO		ASTM		
CFR		ICP		DMR		FIA		GRO
HPLC	FIA						GRO	EPA
ICP	EPA						ASTM	BOD
ASTM		BOD		HPLC		ICP		FIA
		EPA		FIA				
			CFR		GRO			

Complete the grid so that every row, column, and 3x3 box contains each of the following acronyms only once.

Or, if you prefer, solve the puzzle the more conventional way by substituting the digits in parentheses after each acronym.

ASTM (1)
BOD (2)
CFR (3)
DMR (4)
EPA (5)
FIA (6)
GRO (7)
HPLC (8)
ICP (9)

Drinking Water Monitoring Data and Open Records Laws

In May 2007, the National Rural Water Association commissioned a "white paper" entitled, "Applicability of Laboratory Data Generated for Compliance With Safe Drinking Water Regulations". One of the study's key conclusions was,

> "*This study required the unnecessary expenditure of resources just to obtain drinking water compliance data in a format usable for even simple statistical manipulations. Given the importance of these data, the need for accessibility by reasonably skilled persons, and the mandates of the state and federal regulatory agencies, it seems almost negligent that they are not easily available from a regional or national database with easy access by interested parties.*"

These are certainly serious concerns. We cannot speak to national or individual state policies, but Wisconsin has a tradition of strong commitment to the concepts of open government and open records. To address these issues, we offer the following information regarding Wisconsin's Open Records laws and the availability of drinking water compliance data.

Wisconsin's Open Records Law

Wisconsin State Law requires that most records be open to the public. The State Statutes with this requirement are found in 19.31 to 19.39.
http://www.legis.state.wi.us/statutes/Stat0019.pdf

The Wisconsin Department of Justice states: "*Effective citizen oversight of the workings of government and government employees is essential to democratic government and confidence in that government. Access to public records by citizens is a vital aspect of this principle*." Other information on the open records law can be found on the Department of Justice website at the link listed below.
http://www.doj.state.wi.us/AWP/2007OMCG-PRO/2007_PR_Outline.pdf

Much of the information that is of interest to the public is available on the DNR's website. If you can not find the information you are interested in, you can make an open records request. Information on DNR's policy for open records requests can be found at the following address.
http://dnr.wi.gov/aboutdnr/legal/openrecords.html

Not all records are open to the public and you can request that records not be public. Further information on this can be found in chapter 19 of the State Statutes (a link can be found at the beginning of this article) and in s. NR 2.19, Wis. Adm. Code (http://www.legis.state.wi.us/rsb/code/nr/nr002.pdf).

Drinking Water Monitoring Data Available on the Web

Yes, public water supply monitoring data is available on the DNR website. You can search for a public water supply system by name or water supply ID and see the laboratory results.

When a laboratory transmits a file to DNR or enters the data on a web form, the data is available the next day for viewing. This is one way facilities and laboratories can verify that the data has gotten into the DNR Public Drinking Water System. Laboratories should also get an e-mail form the DNR data system telling them if the data loaded or not. If the user does not receive a confirmatory e-mail message the likely cause is one of the following: user e-mail address change, changes in servers, server problems. In addition the messages can be unintentionally filtered by anti-spam software.

Below are instructions on how to access this data.
Go to
http://prodoasext.dnr.wi.gov/inter1/pws2$.startup.

- Enter the facility name or PWS ID number.
- Click on the "Find" button.
- Scroll to the bottom of the page where you will see a number of links; pick the appropriate link.
- A list of samples will appear, click on the sample link to see the detail.

If you have questions on this, contact Ron Arneson at 608-221-6322.

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Action Items

1. Download a copy of the NR149 revisions.
2. Check our website for updates:

www.dnr.state.wi.us/org/es/science/lc/

3. Get your SOPs in order!