NR 149: 1st Anniversary

*Tempus fugit* is Latin for “time flees”, which the world has morphed into “time flies”; and how quickly time does fly.

Since the revisions to NR 149, fully 134 of you laboratories out there have already been audited under the new requirements. That means you’ve likely gotten up close and personal with NR149. You may have your Quality Manuals to guide you, but we rely on NR 149.

Trying on a new set of rules is not wholly unlike trying on a new pair of pants. They looked right in the store, and may have even felt OK if you bothered to head for a dressing room to actually try them on. Of course, some of you just buy by looks, color, and essential measurements, without ever bothering to try them on. Until you get home, that is. You know what we’re talking about don’t you? Especially with jeans. They just don’t feel comfortable, like your old jeans with the gaping holes at the knees. They need time to be worn in.

That’s the way it is with the new NR149; we’ve had a year-long “shake-down cruise” of sorts to try it on for size and get it worn in.

We know some of you out there, especially those closest to it, are clamoring to re-open the rule and fine-tune it. And that will happen—in good time. We need to ensure we know what adjustments are needed to make it feel right. Like a fine beer, NR 149 needs a little krausening.

Everything You Always Wanted To Know About PTs *

*...and some things you didn’t even know you didn’t know!*

The accreditation renewal period which ended on September 1, 2009 was the first in which the relationship between new accreditation structure and PT results was put to the test.

We learned that that we all have some corrective measures to implement in order to ensure a smoother renewal next spring. Consequently, we’ve decided to dedicate the majority of this issue to the details of PT requirements. The program is making some changes for next year and labs have some work to do to be ready.
At an early age we all learned that we are identified by our social security number. In the world of proficiency testing, the identifier is the “EPA ID”.

Yes, your 9-digit WDNR LabCert ID identifies you, but each state has its own ID system. PT providers need a singular means of identifying labs and that means is the EPA ID.

For providers other than the State Lab of Hygiene, if you do not submit an EPA ID with your PT results, your results will end up in the electronic home for wayward PT results. We use your EPA ID, as uploaded by the PT provider with your EPA IDs: Get one. Use it.

EPA IDs: Get one. Use it.

We Don’t Do Solid PTs

The program has opted not to allow use of solid or hazardous waste PTs for certification or registration in the solids matrix. The concentration ranges tend to be somewhat high and the acceptance criteria for some analytes are quite lenient, when compared to the WP.

Solids PTs routinely cost double or more than the corresponding WP and their analysis really only demonstrates the effectiveness of the digestion, distillation or extraction. Since WP PTs demonstrate laboratory capability at lower concentrations, they are more likely to indicate laboratory quality. Finally, PTs are a significant expense for all laboratories, this will help to keep costs down.

Successful results for an analyte-technology combination on a “WP” will satisfy PT requirements for both the aqueous and solid matrix.

WS PTs are valid only for Drinking Water

In a nutshell, WS PTs count only for drinking water, even though many analytes are the same as in WP PTs.

Drinking water analysis has always required submittal of an acceptable Water Supply (WS) PT sample. Since 2005, drinking water certification has been by method/analyte (or VOC analyte group) and laboratories have been required to submit acceptable PTs for each method/analyte combination. Laboratories that are certified or registered in the either the aqueous or solids matrix must submit acceptable PTs from a Water Pollution (WP) study. If a laboratory maintains the same analytical technology/analyte or analyte group certification in both matrices, only a single PT result is required.
Report [Appropriate] Method Codes for PT results

As EPA IDs are required to identify which lab “belongs” to a particular set of PT results, so “method codes” are used to identify the particular analytical technology used to generate a set of PT results.

If we can’t identify which technology was used to report PT results, how would we know which combination of analyte-technology to credit with acceptable PT results?

For example, we receive acceptable PT results for ACME labs for ammonia nitrogen, with no method description or method code selected. ACME Labs is registered to perform ammonia by ion-selective electrode. Sure, it may seem appropriate to credit ACME with an acceptable PT for ammonia by ISE, but how can we be sure that this is really how the PT was analyzed?

The issue becomes further clouded when we consider a class of analytes like metals. A PT result for lead (Pb) could have been analyzed by flame AA, graphite furnace AA, ICP, or ICP/MS. If ACME labs happened to be certified for all of these technologies, which one gets “credited”? And, if the lab actually uses flame AA for the PT, flame AA is specifically exempted from PT requirements!

Most PT providers offer either a “look-up” function (for on-line reporting of PT results) or a list of available method codes. Make sure that you are reporting a method code with your PT results, and that the method code you report is appropriate for your accreditation(s).

The consequences are that you run the risk of not having one or more accreditations renewed annually, resulting in a potential lapse in accreditation.

Do NOT Report Preparatory Method Codes

During this year’s renewal, we found a large number of laboratories’ PT results did not get uploaded because the method code reported was associated with a preparatory technique, like a digestion or distillation.

One good example is Total Kjeldahl Nitrogen, TKN. A fair number of labs reported no method code, but the method description, SM4500N Org B. If you look up this method in Standard Methods, you will find that “B” refers to the digestion procedure. You must report a method code and description associated with a determinative, or specific analytical, technology. Since the TKN procedure converts all organically bound nitrogen to ammonia (NH3), a determinative method for TKN is any approved method for ammonia. Therefore, if you analyze the digestate from TKN by ion-selective electrode, the determinative method should be SM4500NH3 D. The method code for 20th ed. SM 4500NH3 D is 20109006.

Total phosphorus is another great example of where this issue rears its head. Many labs reported the method description, 4500P B 5, for their total phosphorus results. Like TKN, the “B” method is the digestion procedure used. Almost every lab uses a colorimetric procedure, and the method code for SM 4500P E (20th ed.) is 20123802.

Please note that a unique method code has been created for each method and each edition of Standard Methods, including the SM Online methods. Therefore there are as many as 5 or 6 method codes for a single Standard Methods method.

Did you know?

PT “method codes” were established as a NELAC requirement for PT providers. PT method codes are 8 digit numbers. Those beginning with 1 are EPA methods. Standard Methods codes begin with “2”.

“If we can’t identify which technology was used to report PT results, how would we know which combination of analyte-technology to credit with acceptable PT results?”
Applying? PTs must be less than 6 months “old”

The requirements for initial and revised applications have not substantively changed with the revision of ch. NR 149, Wisconsin Administrative Code.

Laboratories that are seeking new or updated accreditations in the aqueous or solid matrix, must submit acceptable PT results from either a routine or “rapid response” WP study, analyzed within 6 months, for each analytical technology/analyte combination under application.

Laboratories seeking initial or additional accreditations in the drinking water matrix applying? PTs must be less than 6 months “old” must submit acceptable PT results from either a routine or “rapid response” WS study, analyzed within 6 months, for each method/analyte combination under application.

When determining whether your PTs are sufficiently recent, the Lab Certification starts with the date on which the application was officially received and then counts back 6 months (180 d). Any PT results submitted with the application must have a study close date no earlier than this date.

August 15th is the deadline for renewal PTs

During renewal this past summer, many labs seemed surprised by the new PT submittal deadline for accreditation renewal. We made an allowance this year, but that was a one-time consideration.

If a PT is required for any analytical technology/analyte or analyte group, an acceptable WP study PT result must be submitted for renewal. For annual renewal of accreditation, laboratories must analyze PTs with study close dates after January 1. PTs with study close dates between September and December can only be used for applications). What has changed is the cut-off date for PT submittal. All PT results must be received by WDNR by August 15 for renewal.

Many commercial PT providers offer WP and WS studies each month. Every PT study is open for 45 days from the day the PTs are shipped to laboratories. After a routine study closes, PT providers can take up to 30 days to issue reports to participating laboratories. For example, one provider shipped samples to laboratories on February 4, 2008. All results had to be reported by March 20, 2008. These results were provided to the WDNR on April 18, 2008. Reports to laboratories were sent the same day. Because it can take up to a month to receive PT results, laboratories are strongly encouraged to participate in PT studies that close well before July 15.

Because it can take up to a month to receive PT results, laboratories are strongly encouraged to participate in PT studies that close well before July 15.

Since laboratories must submit an acceptable result for each analytical technology/analyte or analyte group that has a PT requirement, participating in studies earlier in the year will allow more time to address any failures. If a laboratory fails a PT in a study that closes in July, they will be required to utilize the "rapid response" type samples for renewal. These PTs typically cost significantly more than a routine study.

Those laboratories that participate in the Wisconsin State Laboratory of Hygiene PT Program typically have a shorter timeframe in which to analyze samples. This allows the WSLH to offer three studies between the months of January and July. Laboratories that fail WSLH PTs are automatically shipped the next study.
PT Grading & Evaluation: Not Like NELAC

At the heart of this issue lies a difference between Wisconsin's PT requirements, and the NELAC requirements adopted by approved PT Providers.

Under the NELAC rules, one or more analytes for multi-component analyses may be considered "negative challenge" analytes. These analytes are not "spiked" (assigned concentration value of =0) in the PT sample, and the challenge is to correctly identify that these analytes are not present. Any lab that correctly reports one of these analytes as being < LOD, receives a grade of "Acceptable".

Wisconsin, however, requires an analyte to be spiked in a PT sample in order to obtain credit. Our program requires successful identification and quantitation for each analyte requiring a PT. In addition, labs are penalized for false positives (reporting an analyte to be present above the LOD when it's assigned value is zero) and false negatives (reporting an analyte as not detected, when its assigned value is greater than the lab's LOD).

To a "NELAC" lab, our example VOC [see pg 6] PT result would be scored as "Pass" 47 Acceptable, 6 Not acceptable. That comes to a pass rate of 47/53, or 88.7%

Our program, however, scores this as a "Fail". Here's how we scored the VOC example on the next page. This is a software subroutine applied to uploaded PT provider files.

Step 1 counts all the "spiked" analytes (assigned value>0) = 26
Step 2 counts all of the spiked analytes for which "Acceptable/Check for Error" results were obtained = 22
Step 3 subtracts any "penalties" for false positives or false negatives = -2
Step 4 adjusts the number of correct analytes and calculates a percentage. (22-2) = 20 correct out of 26 = 76.9%

The program then applies the "80% rule"...meaning that a passing grade for the PT requires a score of at least 80%.

The bottom line is that NELAC gives labs "credit" for every unspiked analyte (about 27 in this case) correctly identified as "ND". We don't do that. Our requirements for an acceptable PT results are accurate identification and quantitation for each spiked analyte.

We acknowledge receipt of PTs with an E-mail

Our database has been set up to send an "auto" e-mail anytime to all those labs whose PT files are updated upon a PT Provider uploading data for a particular study. The system sends these e-mails to the individual e-mail address associated with the laboratory individual identified in our database as the "LabCert Contact".

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Method</th>
<th>PT Date</th>
<th>EPA WP</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>VOC ANALYTE GROUP by GC</td>
<td>10/19/2009</td>
<td>EPA WP 107098K</td>
<td>Ungraded</td>
<td></td>
</tr>
<tr>
<td>1,2-Dibromoethane (EDB) by GC</td>
<td>10/19/2009</td>
<td>EPA WP 107098K</td>
<td>Failed</td>
<td></td>
</tr>
<tr>
<td>1,2-Dibromo-3-chloropropane (DBCP) by GC</td>
<td>10/19/2009</td>
<td>EPA WP 107098K</td>
<td>Passed</td>
<td></td>
</tr>
</tbody>
</table>

"Anytime PT results for your lab are uploaded by a PT provider, you will receive a confirmation report via e-mail."
At first glance, the following VOC may look to be worthy of an Acceptable” evaluation for the VOC analyte group. 47 analytes are “Acceptable”; only 6 are “Not Acceptable”. That appears to be a passing rate of 47/53 or 88.7%

Unfortunately, by our grading protocol, it is not acceptable. The number of spiked analytes is 26. Four (4) analytes that were spiked were scored “Not Acceptable”. In addition, there were two (2) false positives receiving an evaluation of “Not Acceptable”.

That leaves us with (26-4-2)=20, which simplifies to 20/26, or 76.9%. A passing score for “analyte groups” is 80%.

"The WI DNR LabCert program does not subscribe to the NELAC approach to scoring PT results, particularly for multi-component analyses certified as analyte 'groups' (e.g., VOCs, BNAs)."

"Unlike NELAC, analytes that are not present in the sample, are not evaluated and do not ‘count’ towards the PT evaluation."
Changes to List of Analytes Requiring a PT

The list of analytes for which a PT was required in 2009-10 was inconsistent between technologies and was insufficiently comprehensive. Subsequently, we are modifying these lists for accreditation renewal 2010.

Wisconsin’s program requires both successful identification and quantitation of any analyte in order to pass a PT for a given analyte. This is in conflict with the NELAC approach that allows a lab to be credited with passing a PT for an analyte whose assigned value is “0”. We believe both identification and quantitation are requisites for passing a PT sample. Consequently, for a number of analytes, labs’ certifications could not be renewed.

**EXCEPTION: Organochlorine Pesticide, VOC and BNA PT samples.** We have also included the lists of VOC, and BNA analytes for which a PT is required. This requirement applies ONLY to those labs that select certification for an individual customized list of analytes, rather than our “group” certification.

New for 2010: Acid Herbicides Requiring a PT

Effective January 1, 2010, the following acid pesticides (herbicides) will require a PT. If you are certified or registered for any of these analytes, a PT will be required. Note that to be acceptable, these analytes must be present at a non-zero concentration.

- 2,4,5-T
- 2,4,5-TP (Silvex)
- 2,4-D
- 2,4-DB
- 3,5-Dichlorobenzoic acid
- 4-Nitrophenol
- Acifluorfen
- Bentazon
- Chloramben
- Chlorthal (DCPA Diacid, Dacthal Diacid)
- Dalapon
- Dicamba
- Dichlorprop
- Dinoseb (2-sec-butyl-4,6-dinitrophenol)
- Pentachlorophenol
- Picloram

New for 2010: OrganoP Pesticides Requiring a PT

Effective January 1, 2010, the following organophosphate pesticides will require a PT. If you are certified or registered for any of these analytes, a PT will be required. Note that to be acceptable, these analytes must be present at a non-zero concentration.

- Azinphos-methyl (*Guthion*)
- Carbophenothion
- Chlorpyrifos
- Demeton (O.S. *Total*)
- Diazinon
- Dichlorovos (*DDVP*)
- Dimethoate
- Dioxathion
- Disulfoton
- Ethion
- Ethoprop
- Famphur
- Fonophos
- Malathion
- Parathion, ethyl
- Parathion, methyl
- Phorate
- Phosmet (*Imidan*)
- Ronnel
- Terbufos
- Tetachorvinphos (*Stirophos, Gardona*)
New for 2010: Nitrogen Pesticides Requiring a PT

Effective January 1, 2010, the following Nitrogen pesticides will require a PT. If you are certified or registered for any of these analytes, a PT will be required. Note that to be acceptable, these analytes must be present at a non-zero concentration.

- Bromacil
- Butachlor
- Metolachlor
- Metribuzin
- Alachlor
- Butylate
- EPTC (Eptam)
- Hexazinone
- Napropamide
- Pronamide
- Propachlor
- Terbacil
- Trifluralin

New for 2010: Triazine Pesticides Requiring a PT

Effective January 1, 2010, the following Triazine pesticides will require a PT. If you are certified or registered for any of these analytes, a PT will be required. Note that to be acceptable, these analytes must be present at a non-zero concentration.

- Ametryn
- Anilazine
- Atraton
- Atrazine
- Cyanazine
- Deethyl atrazine
- Deisopropyl atrazine
- Diaminoatrazine (Deethyl-deisopropyl atrazine)
- Prometon
- Prometryn
- Propazine
- Simazine

New for 2010: Carbamate Pesticides Requiring a PT

Effective January 1, 2010, the following carbamate (or urea-based) pesticides will require a PT. If you are certified or registered for any of these analytes, a PT will be required. Note that to be acceptable, these analytes must be present at a non-zero concentration.

- 3-Hydroxycarbofuran
- Aldicarb
- Aldicarb sulfone
- Aldicarb sulfoxide
- Baygon (Propoxur)
- Carbofuran
- Diuron
- Methomyl
- Oxamyl (Vydate)
- Propham
- 1,3,5-Trinitrobenzene (1,3,5-TNB)
- 1,3-Dinitrobenzene (1,3-DNB)
- 2,4,6-Trinitrotoluene (2,4,6-TNT)
- 2,4-Dinitrotoluene (2,4-DNT)
- 2,6-Dinitrotoluene (2,6-DNT)
- 3-Nitrotoluene
- 4-Amino-2,6-dinitrotoluene
- 4-Nitrotoluene
- HMX (Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)
- Nitrobenzene
- RDX (Hexahydro-1,3,5-trinitro-1,3,5-triazine)
- Tetryl (Methyl-2,4,6-trinitrophenylnitramine)
### VOCs

A PT is required for each of these analytes **ONLY** if you are accredited for the individual analytes. Labs certified for the VOC “analyte group” need only analyze a VOC PT subject to our grading criteria (pgs 5-6).

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Acetone</th>
<th>Ethylbenzene</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,1,1,2-Tetrachloroethane</td>
<td>Acrolein</td>
<td>Hexachlorobutadiene</td>
</tr>
<tr>
<td>1,1,1-Trichloroethane</td>
<td>Acrylonitrile</td>
<td>m/p-Xylenes</td>
</tr>
<tr>
<td>1,1,2,2-Tetrachloroethane</td>
<td>Benzene</td>
<td>Methyl tert-Butyl Ether (MTBE)</td>
</tr>
<tr>
<td>1,1-Dichloroethane</td>
<td>Bromodichloromethane</td>
<td>Methylene chloride</td>
</tr>
<tr>
<td>1,1-Dichloroethylene</td>
<td>Bromoform</td>
<td>Naphthalene</td>
</tr>
<tr>
<td>1,2,3-Trichloropropane</td>
<td>Bromomethane (Methyl bromide)</td>
<td>o-Xylene</td>
</tr>
<tr>
<td>1,2,4-Trichlorobenzene</td>
<td>Carbon disulfide</td>
<td>Styrene</td>
</tr>
<tr>
<td>1,2-Dibromo-3-chloropropylene (DBCP)</td>
<td>Carbon tetrachloride</td>
<td>Tetrachloroethylene</td>
</tr>
<tr>
<td>1,2-Dibromoethane (EDB)</td>
<td>Chlorobenzene</td>
<td>Toluene</td>
</tr>
<tr>
<td>1,2-Dichlorobenzene</td>
<td>Chloroethane</td>
<td>trans-1,2-Dichloroethylene</td>
</tr>
<tr>
<td>1,2-Dichloroethene</td>
<td>Chloroform</td>
<td>trans-1,3-Dichloropropylene</td>
</tr>
<tr>
<td>1,2-Dichloropropane</td>
<td>Chloromethane (Methyl Chloride)</td>
<td>Trichloroethylene</td>
</tr>
<tr>
<td>1,3-Dichlorobenzene</td>
<td>cis-1,2-Dichloroethylene</td>
<td>Trichlorofluoromethane</td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td>cis-1,3-Dichloropropylene</td>
<td>Vinyl acetate</td>
</tr>
<tr>
<td>2-Butanone (MEK)</td>
<td>Dibromochloromethane</td>
<td>Vinyl chloride</td>
</tr>
<tr>
<td>2-Hexanone</td>
<td>Dibromomethane</td>
<td>Xylenes, Total</td>
</tr>
<tr>
<td>4-Methyl-2-pentanone (MIBK)</td>
<td>Dichlorodifluoromethane</td>
<td></td>
</tr>
</tbody>
</table>

### BNAs – Base/Neutrals

A PT is required for each of these analytes **ONLY** if you are accredited for the individual analytes. Labs certified for the BNA “analyte group” need only analyze a BNA PT subject to our grading criteria (pgs 5-6).

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Anthracene</th>
<th>Di-n-butylphthalate</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,2,4-Trichlorobenzene</td>
<td>Benzo(a)anthracene</td>
<td>Di-n-octylphthalate</td>
</tr>
<tr>
<td>1,2-Dichlorobenzene</td>
<td>Benzo(a)pyrene</td>
<td>Fluoranthen</td>
</tr>
<tr>
<td>1,3-Dichlorobenzene</td>
<td>Benzo(b)fluoranthen</td>
<td>Fluorene</td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td>Benzo(k)fluoranthen</td>
<td>Hexachlorobenzene</td>
</tr>
<tr>
<td>1-Chloronaphthalene</td>
<td>Benzo(b)fluoranthen</td>
<td>Hexachlorobutadiene</td>
</tr>
<tr>
<td>2,4-Dinitrotoluene</td>
<td>Benzo(g,h,i)pyrrole</td>
<td>Hexachlorocyclopentadiene</td>
</tr>
<tr>
<td>2,6-Dinitrotoluene</td>
<td>Benzo(k)fluoranthen</td>
<td>Hexachloroethene</td>
</tr>
<tr>
<td>2-Chloronaphthalene</td>
<td>Benzyl alcohol</td>
<td>Indeno(1,2,3-cd)pyrene</td>
</tr>
<tr>
<td>2-Methylnaphthalene</td>
<td>Benzyl butyl phthalate</td>
<td>Isophorone</td>
</tr>
<tr>
<td>2-Nitroaniline</td>
<td>bis(2-Chloroethoxy)methane</td>
<td>Naphthalene</td>
</tr>
<tr>
<td>3,3’-Dichlorobenzidine</td>
<td>bis(2-Chloroethyl)ether</td>
<td>Nitrobenzene</td>
</tr>
<tr>
<td>3-Nitroaniline</td>
<td>bis(2-Chloroisopropyl)ether</td>
<td>N-Nitrosodiethylamine</td>
</tr>
<tr>
<td>4-Bromophenyl phenylether</td>
<td>bis(2-Ethylhexyl)phthalate</td>
<td>N-Nitrosodimethamine</td>
</tr>
<tr>
<td>4-Chloroaniline</td>
<td>Carbazole</td>
<td>N-Nitroso-di-n-propylamine</td>
</tr>
<tr>
<td>4-Chlorophenyl phenylether</td>
<td>Chrysene</td>
<td>Phenanthrene</td>
</tr>
<tr>
<td>4-Nitroaniline</td>
<td>Dibenzo[a,h]anthracene</td>
<td>Pyrene</td>
</tr>
<tr>
<td>Acenaphthene</td>
<td>Dibenzo(furan)</td>
<td>Pyridine</td>
</tr>
<tr>
<td>Acenaphthylene</td>
<td>Diethylphthalate</td>
<td></td>
</tr>
<tr>
<td>Aniline</td>
<td>Dimethylphthalate</td>
<td></td>
</tr>
</tbody>
</table>

### BNAs – Acid Extractables

<table>
<thead>
<tr>
<th>Analyte</th>
<th>2-Chlorophenol</th>
<th>4-Chloro-3-methylphenol</th>
</tr>
</thead>
<tbody>
<tr>
<td>2,4,5-Trichlorophenol</td>
<td>2-Chlorophenol</td>
<td>4-Chloro-3-methylphenol</td>
</tr>
<tr>
<td>2,4,6-Trichlorophenol</td>
<td>2-Methyl-4,6-dinitrophenol</td>
<td>4-Methylphenol (p-Cresol)</td>
</tr>
<tr>
<td>2,4-Dichlorophenol</td>
<td>2-Methylphenol (O-Cresol)</td>
<td>4-Nitrophenol</td>
</tr>
<tr>
<td>2,4-Dimethylphenol</td>
<td>2-Nitrophenol</td>
<td>Benzoic acid</td>
</tr>
<tr>
<td>2,4-Dinitrophenol</td>
<td>3-Methylphenol (m-Cresol)</td>
<td>Pentachlorophenol</td>
</tr>
<tr>
<td>2,6-Dichlorophenol</td>
<td></td>
<td>Phenol</td>
</tr>
</tbody>
</table>
The following is a list of some of the methods which have been deleted as approved methods for compliance testing.

If you report method codes associated with these methods for your PT results, they will not be acceptable and will not be uploaded into the Lab Certification database.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Deleted Clean Water Act Methods</th>
<th>Parameter</th>
<th>Deleted Clean Water Act Methods</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acidity</td>
<td>Titrimetric: EPA 305.1</td>
<td>Manganese (Mn)</td>
<td>FLAA: EPA 243.1</td>
</tr>
<tr>
<td></td>
<td>Titrimetric: USGS I-2030-85</td>
<td>Molybdenum (Mo)</td>
<td>FLAA: EPA 246.1</td>
</tr>
<tr>
<td>Alkalinity</td>
<td>Titrimetric: EPA 310.1</td>
<td>Nickel (Ni)</td>
<td>FLAA: EPA 249.1</td>
</tr>
<tr>
<td></td>
<td>Titrimetric: SM 2310 B (4a)</td>
<td>Nitrate+Nitrite (NO3+NO2)</td>
<td>Colorimetric: EPA 353.1</td>
</tr>
<tr>
<td>Aluminum (Al)</td>
<td>FLAA: EPA 202.1</td>
<td>Nitrite (NO2)</td>
<td>Colorimetric: EPA 353.3</td>
</tr>
<tr>
<td></td>
<td>GFAA: EPA 202.2</td>
<td>Oil&amp;Grease</td>
<td>Gravimetric: EPA 413.1</td>
</tr>
<tr>
<td>Ammonia</td>
<td>Colorimetric: EPA 350.2</td>
<td>TOC (Total Organic Carbon)</td>
<td>Combustion/Oxidation: EPA 415.1</td>
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<tr>
<td></td>
<td>ISE: EPA 350.3</td>
<td>Orthophosphate (cPO4)</td>
<td>Colorimetric: EPA 365.2</td>
</tr>
<tr>
<td>Antimony (Sb)</td>
<td>FLAA: EPA 204.1</td>
<td>Phenolics, Total</td>
<td>Colorimetric: EPA 420.2</td>
</tr>
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<td></td>
<td>GFAA: EPA 204.2</td>
<td>Phosphorus, Total</td>
<td>Colorimetric: EPA 365.2</td>
</tr>
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<td>Arsenic (As)</td>
<td>GFAA: EPA 206.2</td>
<td>Potassium (K)</td>
<td>FLAA: EPA 258.1</td>
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<td></td>
<td>Hyd A: EPA 208.3</td>
<td>Total Solids (Residue, Total)</td>
<td>Gravimetric: EPA 160.3</td>
</tr>
<tr>
<td>Barium (Ba)</td>
<td>FLAA: EPA 208.1</td>
<td>TDS Total Dissolved Solids</td>
<td>Gravimetric: EPA 160.1</td>
</tr>
<tr>
<td></td>
<td>GFAA: EPA 208.2</td>
<td>(Residue, Filtrable)</td>
<td></td>
</tr>
<tr>
<td>Beryllium (Be)</td>
<td>FLAA: EPA 210.1</td>
<td>TSS Total Dissolved Solids</td>
<td>Gravimetric: EPA 160.2</td>
</tr>
<tr>
<td></td>
<td>GFAA: EPA 210.2</td>
<td>(Residue, Non-Filtrable)</td>
<td></td>
</tr>
<tr>
<td>Bod (Biochemical Oxygen Demand)</td>
<td>BOD Assay: EPA 405.1</td>
<td>Sulfate</td>
<td>Gravimetric: EPA 375.3</td>
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<td>Boron</td>
<td>Colorimetric: 212.3</td>
<td>Settleable Solids</td>
<td>Gravimetric: EPA 160.5</td>
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<td>Bromide</td>
<td>Titrimetric: EPA 320.1</td>
<td>Selenium (Se)</td>
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<td>Cadmium (Cd)</td>
<td>FLAA: EPA 213.1</td>
<td>Silver (Ag)</td>
<td>FLAA: EPA 272.1</td>
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<td>GFAA: EPA 213.2</td>
<td>Sodium (Na)</td>
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<td>Sulfate</td>
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<td>Gravimetric: EPA 375.3</td>
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<td>COD (Chemical Oxygen Demand)</td>
<td>Colorimetric: EPA 410.1</td>
<td>Surfactants (MBAS)</td>
<td>Colorimetric: EPA 425.1</td>
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<td>Thallium (Tl)</td>
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<td>Tin (Sn)</td>
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<td>Titrimetric: EPA 330.2</td>
<td></td>
<td>GFAA: EPA 282.2</td>
</tr>
<tr>
<td></td>
<td>Titrimetric: EPA 330.3</td>
<td>Titanium (Ti)</td>
<td>FLAA: EPA 283.1</td>
</tr>
<tr>
<td></td>
<td>Titrimetric: EPA 330.4</td>
<td>Vanadium (V)</td>
<td>FLAA: EPA 286.1</td>
</tr>
<tr>
<td></td>
<td>Colorimetric: EPA 330.5</td>
<td></td>
<td>GFAA: EPA 286.2</td>
</tr>
<tr>
<td>Chromium, Hexavalent</td>
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<td>Zinc (Zn)</td>
<td>FLAA: EPA 289.1</td>
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<td></td>
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<td>Titanium (Ti)</td>
<td>FLAA: EPA 283.1</td>
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<td></td>
<td></td>
<td>Vanadium (V)</td>
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<td>GFAA: EPA 286.2</td>
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<td></td>
<td></td>
<td>Zinc (Zn)</td>
<td>FLAA: EPA 289.1</td>
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</tbody>
</table>

“Most of the old EPA ‘200 series’ methods have been eliminated as approved methods. Use only approved methods!”

“Refer to NR 219 for wastewater approved methods. Refer to NR 809 for drinking water methods.”

202.1
202.2
204.1
206.1
206.2
208.1
208.2
210.1
210.2
212.3
215.1
215.2
Report PT Results by the Correct Technology

One other problem that cropped up during accreditation renewal was that labs analyzed PTs, but analyzed them using a different technology than what they were actually accredited for.

**Example:** Lab X reported PT results using SM 3111B, an FLAA method. The lab is certified for these metals by GFAA. The lab had to do another PT, and use GFAA this time.

A number of labs were initially (9/1/08) accredited for various pesticides by both GC and GC/MS, but only submitted PT data analyzed using one of the technologies. Check your Scope of Accreditation before reporting PT results and make sure you have all technologies covered for a given analyte before submitting your results to the PT Provider.

Remember: you can report results from several different technologies for any given PT. The only limitation is the volume of PT sample available for analysis.

This happens frequently with wet chemistry. A cross-reference list of technologies based on common wet chemistry methods is provided on our website at: 

WEP (Water Extractable Phosphorus) Reporting

There is a long standing problem with reporting Parameter 686 Water Extractable Phosphorus on Characteristic Report (49) forms.

Often the confusion facilities have is the outcome of not knowing how to interpret test results on lab reports. Those most involved believe that the situation can be greatly improved if labs would spell out the parameter 686 as it is required to be reported on 49 forms, which is **Water extractable P as a percent of total P**.

A number of issues have been raised regarding how permittees report Water Extractable Phosphorus results for their biosolids. There appears to be some confusion in that some are reporting the results as Percent of P per kg of biosolids. This equals the portion of total phosphorus which is water extractable.

**Percent WEP (PWEP) = (WEP ÷ PT) × 100. P_T = total P as mg of P per kg of biosolids or other P-source (dry weight) via acceptable method (e.g. EPA Digestion Method 3050 or 3051 and analytical Method 6010 or 6020, or Standard Method 4500-P; etc.)**

This means that 3.3% of the Total P is water extractable; this is the result that should be reported to the agency.

**Example:**

Standard WEP = 1,000 mg P/kg biosolids (dry weight) = 0.1% dry weight

Total P = 30,000 mg P/kg biosolids (dry weight) = 3.0% dry weight

PWEP = \((1,000 ÷ 30,000) \times 100 = 3.3\%\.) This means that 3.3% of the Total P is water extractable; this is the result that should be reported to the agency.

An attempt to clarify this has been made by changing the units in the nutrient 'picklist' to % of Total P for the parameter Phosphorus, Water Extractable.

"WEP (Water Extractable Phosphorus must be reported in units of % of the total Phosphorus.)"
Sharing Lab Data and the Open Records Laws

The State of Wisconsin’s policy is to have the government accountable. Hence, most records that the State maintains are open to the public. The “Open Records Law” can be found in Chapter 19, subchapter II of the State Statutes. When State Agencies receive data they need to consider the following:

- Trade Secrets
- Privileged information
- Confidential information
- Personally identifiable Information

Trade Secrets
Trade secrets in most cases do not apply to environmental testing data. It could apply in regard to waste treatment information and data.

Privileged Information
Certain information is protected as privileged information. These include attorney-client and doctor-patient information. This type of data in general is not submitted to DNR. Privileged information would also be information gathered by the State as part of an enforcement investigation.

Confidential Information
When data is collected by the State, one can request that the information be treated as confidential. That request, if granted, would mean that the data would not be shared with the public under the “Open Records Law”. The State Agency must make a decision as to whether the request should be granted. State Statutes set standards for confidentiality for a number of programs (e.g., metallic mining prospecting data). There are no provisions in State Statutes for confidentiality for drinking water. However, a request can still be made for the data to be confidential and if it meets the criteria set in State Adm. Code NR 2, then the request can be granted.

Personally Identifiable Information
Data collected by the State regarding private wells is stored in a secure location behind a firewall. Some of this information is available to the public. However, the personally identifiable information is not available to the general public. State Statutes prohibits sharing personally identifiable information.

Do I need to submit all of my monitoring data or can I choose what data to submit?
Many monitoring programs require that you submit all the data collected. A laboratory or facility can not pick and choose which data to provide to determine compliance.

Is information I submit to one State Agency shared with other agencies?
Yes, it can be. Many times different state and local agencies have overlapping missions. An example is protecting public health. DNR, Department of Health Services, the State Laboratory of Hygiene, and local health departments all share this same mission. So if Madison & Dane County Health Department is investigating arsenic in the county’s groundwater, then the state agencies will provide the information when requested. Much of the work that is done by state agencies is funded by federal agencies. A condition of federal funding may include providing the funding agency with our monitoring data.

Open Records Information Resources
Visit the DNR web page on open records at:
Department of Justice’s information on the “Open Records Law”:
Request for confidential status under NR 2.19, Adm. Code
http://www.legis.state.wi.us/rsb/code/nr/nr002.pdf

“A condition of federal funding may include providing the funding agency with our monitoring data.”
VOCs – Changes on the Landscape

There are some recent changes (some proposed) regarding VOCs. NR 140 is proposing the addition of:
- 1,4-dioxane (PAL 0.3 ug/l)
- chlorodifluoromethane (PAL 700 ug/l)
- ethyl ether (PAL 100 ug/l)
- tertiary butyl alcohol (PAL 1.2 ug/l)

In addition some of the enforcement standards (ES) and preventive action limits (PAL) will be changing for other VOCs.

NR 809 is proposing addition of MtBE as a special monitoring contaminant. Hearings for NR 809 will be in October and NR 140 this winter.

EPA published method 524.3 in June 2009 and added:
- Chlorodifluoromethane
- diisopropyl ether
- methyl acetate
- t-amyl ethyl ether
- t-amyl methyl ether
- t-butyl alcohol.

GEMS – Blank Detections and Reporting QC Flags

Reporting Groundwater Monitoring Results to GEMS - New QC Failure Criteria

As of December 1, 2009 the Department is asking labs to change the criteria they use to report quality control (QC) failures for samples when a parameter is detected in an associated method, trip or field blank. For all data submitted to the groundwater and environmental monitoring system (GEMS), please use the following new relaxed criteria:

Only report a QC Flag I failure (“F”) for a sample if the concentration of a parameter in the associated method blank, trip blank or field blank exceeds the highest of any of the following values for that parameter:

1. The limit of detection;
2. Five percent of the lowest applicable regulatory limit (e.g., NR 140 groundwater quality preventive action limit); or
3. Ten percent of the measured concentration in the sample.

Before this change, a QC Flag I failure had to be reported for a sample if the concentration of a parameter in the associated method, trip or field blank exceeded the limit of detection for that parameter. This criteria was more restrictive than that specified by EPA or the Laboratory Certification program. The new requirement is consistent with the Lab Certification program’s criteria.

Jack Connelly, the Solid Waste Program Coordinator, sent a November 10 email to the environmental contact at each of the facilities submitting data to GEMS informing them of this change. If you have any questions about this change, please contact Jack at 608-267-7574 or johnston.connelly@wisconsin.gov.

Before this change, facilities had to report a failure for a quality control flag any time a blank had a detect for a given parameter above the LOD.
NR 219 Update Finalized

On June 1, 2009, changes to NR 219, the administrative rule that governs approved methodologies for wastewater monitoring took effect.

In March 2007, the EPA published final rules that promoted sweeping changes related to analytical methods for NPDES monitoring. Revisions to NR 219 were made to bring Wisconsin into compliance with these federal standards which were promulgated two years previously.

One of the major impacts of this rule change was to delete numerous methods considered obsolete (see page 10).

Although the rule revision deleted many analytical methods, the rule retains at least one method for any regulated analyte. The rule also reduces the use of reagents containing mercury with the elimination of 14 additional methods. In sum, the rule maintains 504 methods from the previous version, deletes 62, and approves 366 additional ones.

One key method addition is the approval of luminescence technology (LDO) for the analysis of dissolved oxygen. LDO and similar “electro-optical” technologies are now also approved for BOD analysis.

The affected analytes are: cBOD (carbonaceous BOD), HEM (Hexane Extractable Material), TDS (Total Dissolved Solids, or filterable residue), alkalinity, acidity, orthophosphate, and nitrite.

Please note that these parameters DO require a PT for each technology. The State Lab of Hygiene will be providing PTs for each of these parameters in 2010.
Drinking Water Lab Data Entry Reminders

The following are some updates and reminders related to drinking water lab data submittals.

User ID and Password
In order to transmit your public drinking water monitoring data to the DNR you must have a user ID from the Wisconsin Access Management System. These IDs are not meant to be used lab wide. Rather each person should have there own ID and password. Information Technology (IT) folks are sensitive about this subject and are very concerned with security. One should never share a password! Also, you should have more than one person in your lab to do this. Having backups is always a good idea. If you need an ID go to https://on.wisconsin.gov/WAMS/SelfRegController. Once you have done this go to http://www.dnr.wi.gov/environmentprotect/switchboard/ebp.html to register with DNR to use the drinking water data entry system. Both registrations are easy to do.

Confirmation E-mail
After you submit your data via web form or file drop, you should get an e-mail giving you the status of the data. Be sure to read these e-mails to see if the system accepted your results. If it did not, it should tell you what needs to be corrected.

Data Available on the Web
Also note that your public drinking water results should be available on the DNR website the next morning. So if you want to double check your results go to: http://prodoasext.dnr.wi.gov/inter1/pws25$startup and enter the PWS number of the facility in question.

Monitoring Sites
The Public Drinking Water Program will be requiring that Monitoring Site IDs also be reported for all samples. Changes will be made to the online forms and the Drinking Water Lab Sample Entry system. The file drop system can already handle the information, but you will need to add it to your exported files. Keep your eyes open for more information on this.

Private Well Water
DNR is in the process of allowing laboratories to enter or drop files for private well testing. The web form is being developed and it is planned to use the same system for dropping files as the public water system (just need the well ID rather than the PWS #). The new system should be available before next summer. We will let you know when it is available. We hope laboratories will use this system for both the compliance data on new wells and pump work, but also for non-compliance results. Data from the non-compliance work will help us all better understand the quality of our state’s groundwater and protect the health of our citizens. If you are interested in helping us test the new system call or e-mail Ron Arneson (608.221.6322) (Ronald.Arneson@wi.gov).

“The Public Drinking Water Program will soon require that Monitoring Site IDs be reported for all samples.”
Application Fee Reminders

Many labs are overpaying when it comes to applications.

**Base Fees**
If you are already an accredited lab, then you have already paid an annual base fee; you do NOT have to pay it again. The only two cases where you need to pay a base fee are for a new lab, or a lab switching from registration to certification (must pay the difference in base fee between certification and registration).

**Matrix Fees**
If you are already accredited for any technology/class for a given matrix, then you do NOT pay that matrix fee.

**Example 1:** Lab is certified only for drinking water matrix, wants to add GC/MS under aqueous and solid matrices. Pay the matrix fee for aqueous and solid matrices.

**Example 2:** Lab is adding new technologies to a matrix for which they are already accredited. Pay no matrix fee.

**Technology (Class) fees**
If you already are accredited for a given technology in a given matrix (or “class” for drinking water), then you do NOT pay that technology fee for that matrix.

**Example 1:** Lab is certified for GC and ICP in aqueous and solid matrices; wants to add GC/MS. GC/MS technology fee for both aqueous and solid is required.

**Example 2:** Lab is certified for VOCs by GC/MS in aqueous and wants to add BNAs by GC/MS. GC/MS technology fee is NOT required.

A fee calculation spreadsheet is available at:
www.dnr.state.wi.us/org/es/science/lc/APPLICATION/Application%20Fee%20Calculator.xls

NOTE: Conversion from Certified to Registered ($172.50) or Registered to Certified ($460.00) does require an application and fee.

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