



# LABNOTES *Summer 2007*

## Introducing the 2007 Labs of the Year...

*From DNR Press Release*

On March 28, 2007 at the Natural Resources Board Meeting the 2007 Registered Laboratories of the Year were honored. DNR Secretary Scott Hassett and Environmental Science Services Section Chief David Webb presented the awards to **Stevens Point Wastewater Treatment Plant Laboratory** and the **Fremont-Orihula-Wolf River Joint Sewage**



*From left, members of Stevens Point Wastewater Treatment Plant Laboratory are : Dan Ryskoski , Jeremy Cramer, Frank Suchon, Lee Gostomski, Eric Niffenegger, Kim Halverson, Dave Sobczak).*



*Left. Pictured are (l-r) Mark Corbett, DNR Engineer, Maggie Wilson, and both John Wilson and Mark Meyer from the Fremont-Orihula-Wolf River Joint Sewage Commission*

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*Labs of the Year, continued.*

This is the twelfth year these awards have been presented to registered laboratories that have demonstrated exceptional efforts towards generating high quality data. The data they generate is very important because many programs in the DNR utilize data submitted by these labs to make regulatory decisions. Before the ceremony, DNR Enforcement and Science Division Administrator Amy Smith pointed out that the efforts of staff at wastewater treatment plants is so often “unsung” and that these awards are a chance to recognize what they do for all of us. During the presentation, David Webb stated that this is a great honor because *“over 250 registered laboratories were eligible to be chosen for this award”*. It takes exceptional efforts to be nominated for the Lab of the Year award, and to be chosen as the winner is a great mark of distinction. The Stevens Point Wastewater Treatment Plant Laboratory, located in Stevens Point, was awarded the Laboratory of the Year Award in the Large Registered Facility category. They analyze wastewater samples for biochemical oxygen demand (BOD), carbonaceous BOD, total suspended solids and phosphorus for the municipal wastewater facility.

DNR Audit Chemist, Camille Johnson, nominated the laboratory. In her nomination she wrote that the operators at Stevens Point *“...are extremely proactive about any QC issues that arise. They are also very dedicated and make changes when they learn that there is a better way of doing something”*. She went on to acknowledge that despite the challenge of having four operators rotating duties in the lab; very stringent quality control limits are achieved. Camille Johnson also pointed to the fact that no deficiencies were identified at their last lab evaluation and the operators are all dedicated to doing accurate lab analysis.

When presenting the award, David Webb said that the Stevens Point Lab *“completes lab maintenance before problems arise, something easier said than done.”* The facility was also recognized for analyzing extra quality control samples, taking thorough corrective action, and for excellent records. When accepting the award, Eric Niffenegger, the Stevens Point Superintendent, thanked the Department, the Stevens Point Mayor and the treatment plant staff for their efforts.

The Stevens Point staff honored as part of the award include: Kim Halverson (Director of Water and  
*Continued on next page.*

**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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*Lab of the Year winners, continued.*

Wastewater), Eric Niffenegger (Superintendent), Jeremy Cramer (Chief Operator) and Operators: Dave Sobczak, Dan Ryskoski, Frank Suchon, and Lee Gostomski.

The Fremont-Orihula-Wolf River Joint Sewage Commission (J.S.C.) Laboratory with Operations by Midwest Contract Operations (M.C.O.), located in Fremont, was awarded the Laboratory of the Year Award in the Small Registered Facility category. The Fremont-Orihula-Wolf River J.S.C. Lab analyzes municipal wastewater samples for biochemical oxygen demand and total suspended solids.

DNR Engineer, Mark Corbett and DNR Audit Chemist, Camille Johnson nominated the laboratory. In his nomination Mark Corbett wrote about Fremont's "*.... impressive laboratory self monitoring program, which has clearly led to improved plant performance and the banner compliance record. Impeccable lab procedures and testing protocol is continuously demonstrated at the facility lab. This meticulous attention to testing and recording detail, establishes the on site lab as a true model facility.*" The other nominator, Camille Johnson, wrote that when the sole operator John Wilson encounters rare quality control problems he "*....leaves no stone unturned and really cares about generating the best data possible.*"

When presenting the award to Fremont-Orihula-Wolf River J.S.C. Mr. Webb stated that "*Johns' lab results have clearly led to improved plant performance which has resulted in the plant consistently complying with all permit limits*". The lab was also recognized for having no deficiencies, the operators' strong scientific understanding of the lab and plant and his amazing attention to details.

When accepting the award for the Commission, John Wilson, operator for the plant, remarked "*It is an honor to protect the environment....hats off to the Department for also working hard to protect the environment.*"

Nominations for the 2008 Registered Laboratory of the Year awards are now being accepted. Anyone may nominate a facility as long as they are Registered (not Certified). To obtain a nomination form contact Camille Johnson at (715) 831-3272 or by email at [Camille.Johnson@wisconsin.gov](mailto:Camille.Johnson@wisconsin.gov).



## Exam & Meeting Schedules

### Operator Certification Exams

DNR will hold Wastewater, Drinking Water and Septage Operator Certification exams November 7, 2007 (*postmark deadline October 3, 2007*) in DNR Regions around the state. Check the Operator Certification web site for details, as they become available. Application packets will be mailed, about August 1, 2007, to all operators who have taken an exam in the last 18 months.

[www.dnr.state.wi.us/org/es/science/opcert](http://www.dnr.state.wi.us/org/es/science/opcert)



### 2007 Conferences, Meetings

#### WWA 86<sup>th</sup> Annual Conference & Water Utility Expo

The Wisconsin Water Association's 86<sup>th</sup> annual conference is scheduled for September 12-14, 2007 at the KI Center in Green Bay.

<http://www.wiawwa.org>



#### WWOA 41<sup>st</sup> Annual Conference

The Wisconsin Wastewater Operators Association 41<sup>st</sup> annual conference is to be held October 23 through 26, 2007 at the La Crosse Civic Center & Radisson Hotel La Crosse, WI. Registration and accommodations information available at:

<http://explorelacrosse.com/home/housingbureau/register.asp?btnSubmit=showOptions&cboEventID=94>

[www.wwoa.org](http://www.wwoa.org)



### Training for Lab Analysts

#### Wastewater Lab - Intro

October 29 - 31, 2007

Location: Delafield WWTP

To Register: Dan Tomaro (608) 770-5144

Wastewater Training Solutions

[www.wastewatertrainingsolutions.com](http://www.wastewatertrainingsolutions.com)

Always remember to check out the Operator Certification Program's consolidated training calendar:

[www.dnr.state.wi.us/org/es/science/opcert/training.pdf](http://www.dnr.state.wi.us/org/es/science/opcert/training.pdf)





## Program Administration

### Welcome New Audit Staff!



*Dave Ekern is the new audit chemist stationed in Madison and he will primarily cover the SE Region.*

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#### Dave Ekern

My name is Dave Ekern, and I am very excited to begin my new job at the DNR. I was raised in the La Crosse area and graduated from Viterbo University in 1996 with a B.S. degree. My major was biology and my minors were chemistry and math. I have been married for 8 years. We have two children who are 4 and 1.

For the past 3 years I worked as a chemist at a large cGMP regulated pharmaceutical contract testing facility. I worked primarily on ongoing drug stability testing by performing chemical and physical characterization analysis.

Prior to that experience, I worked in the environmental field for three years in the state of Nevada. I worked in analytical laboratories very similar to the type which I will soon have the opportunity to audit. I tested drinking and wastewater, soil, sludge, etc. I performed a wide variety of the typical tests which are conducted in an environmental lab, and feel I have a solid understanding of the environmental testing field.

I look forward to serving the customers of the lab certification program and will be pleased to meet many of you soon. ☺

#### Tom Trainor

My name is Tom Trainor and I am beginning my new career as an Audit Chemist in the Laboratory Certification and Registration Program. I am looking

very forward to meeting all of you and working with you to help you produce the best quality data you can. My job is to help you, which is why I am here – so feel free to use me as much as you need!

My childhood stomping ground was in Manitowoc, Wisconsin. I have a B.S. degree in Chemical Engineering from the University of Wisconsin-Madison.

My first job out of college was as a chemist in Madison, Wisconsin. On that first job I moved from weighing out corn samples to shaking separatory funnels to managing projects for the FIFRA program following GLP protocols. That lasted about 5 years and then I decided to move closer to home.

My second job was in Green Bay, Wisconsin where I continued my work as a chemist for 5 more years although this time the switch was to the environmental field where I performed work under the SDWA, RCRA, NPDES and UST programs.

An opportunity to work with one of the largest laboratories in the State called me to my last job which was also in Green Bay, Wisconsin. At this job I worked for the last 10 years as a project manager for some very complex projects that were governed by the DOD, DOE and CERCLA programs. I still continued to manage a lot of the SDWA, RCRA, NPDES and UST projects for Wisconsin and other States in the U.S.A.

On a personal side, I have been married for 20 years and I have two wonderful children, a son that is 13 and a daughter that is 16. They are my pride and joy. I am very involved in my kid's sports lives which include basketball, soccer, football and tennis. It seems like there is never an "off season".... My hobbies are sports and music. Go Packers! ☺



*Tom Trainor is the new audit chemist stationed in Green Bay and he will primarily cover the NE region.*

## ***Fiscal Year 2008 Certification and Registration Fees***

The Natural Resources Board on March 28 unanimously approved the department's laboratory certification and registration fee schedule for Fiscal Year 2008. The fee schedule was previously reviewed by the Certification Standards Review Council, who provided their unanimous support and recommended Board approval. The approved fee schedule will allow the Department to fund the laboratory certification and registration program at a level below its spending authority as established under Chapter 20.370(3)(fj), Wis. Stats.

Specifically, the cost per relative value unit (RVU) will increase from \$58.00 to \$64.50, an increase of 11%. Certification renewal fees for the typical commercial laboratory (certified lab base fee + test categories 1-8, 10, 12, & 14-16) will be \$3,805.50. Registration fees for the typical municipal wastewater treatment laboratory (registered base fee + test categories 1-4) will be \$903.00.

The complete fee schedule is provided in the table below:


**Laboratory Fees for FY 2008 (Sept.1, 2007 - Aug. 31, 2008)**

<b>Fee Item</b>	<b>FY 2008 Unit Price</b>	<b>Fee Item</b>	<b>FY 2008 Unit Price</b>
<b>Registered Base Fee</b>	<b>\$645.00</b>	Category 10	\$258.00
<b>Certified Base Fee</b>	<b>\$967.50</b>	Category 11	\$258.00
<b>Reciprocity Fee</b>	<b>\$1,935.00</b>	Category 12	\$258.00
<b>Initial Application Fee</b>	<b>\$387.00</b>	Category 13	\$258.00
<b>Revised Application Fee</b>	<b>\$193.50</b>	Category 14	\$258.00
Category 1	\$64.50	Category 15	\$774.00
Category 2	\$64.50	Category 16	\$258.00
Category 3	\$64.50	Category 17	\$774.00
Category 4	\$64.50	Category 18	\$1290.00
Category 5	\$129.00	Category 18a (Nitrate Only)	\$129.00
Category 6	\$129.00	Category 18b (Nitrate & Fluoride)	\$258.00
Category 7	\$258.00	Category 19	\$258.00
Category 8	\$258.00	Category 20	\$1,677.00
Category 9	\$258.00	Category 21	\$258.00

**Note: fees are effective July 1, 2007**

Fees are calculated using the formula promulgated in s. NR 149.05, Wis. Admin. Code. This formula uses a relative value system to equitably distribute the cost of administering the program across all participating laboratories. Each fee item is assigned a relative value in Ch. NR 149, Table 2. The total number of available RVUs is the sum of the relative values of each fee item multiplied by the number of labs certified or registered for that fee item in the coming fiscal year. The cost per RVU is calculated by dividing the program's operating costs (not including projected travel costs for audits of out-of-state labs, for which these labs are billed directly), by the total number of available RVUs. The cost of each fee item is determined by multiplying its relative value by the cost per RVU.

Certification and registration renewal fees will appear on the environmental fee statements that will be mailed in late May. Payment will be due in full by June 30, 2007. Late fees will be assessed to laboratories that fail to pay renewal fees by this deadline.

Please contact Alfredo Sotomayor at (608) 266-9257 or Alfredo.Sotomayor@Wisconsin.gov if you have any questions about your fees. 

## **Approved Methods for Landfill Analyses under NR 507 – Update**

*By Dave Parsons & Jack Connelly, Waste & Materials Management Program; Camille Johnson, Bureau of Science Services*

There have been questions raised by commercial laboratories as well as Laboratory Certification Program staff regarding the acceptable methods for analyzing landfill samples. Some of these questions were in response to a previous article on “Approved Methods for Landfill Analysis under NR 507” published in the Summer 2006 edition of LabNotes.

The January 2006 revision of NR 507 required that solid waste facilities use only the analytical methods in EPA’s “Test Methods for Evaluating Solid Waste” Third Edition amended through Update IIIA (SW-846) for analyzing groundwater, lysimeter and leachate samples. Prior to January 2006, the NR 507 appendices listed specific methods for each parameter. When the specific method requirements were replaced with the general reference to SW-846, the unintended result was a reduction in analytical methods for some parameters.

Therefore, to better meet our original goal of increasing analytical method flexibility for all parameters, we are now allowing solid waste facilities to use the wastewater test procedures identified in NR 219.04, in addition to the SW-846 methods currently **required in NR 507.17(4)**. Chapter NR 219 is available at:

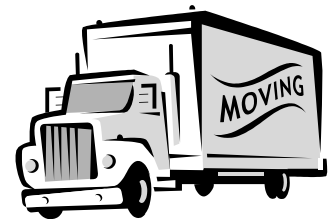
[www.legis.state.wi.us/rsb/code/nr/nr219.pdf](http://www.legis.state.wi.us/rsb/code/nr/nr219.pdf)

This increased flexibility is limited to the handling and analysis of groundwater, lysimeter and leachate samples collected for solid waste facilities, and requires that the methods be “suitable for the matrix, type of analyte, expected level of analyte, regulatory limit, and potential interferences in the samples to be tested” as required in NR 507.17(4). If a solid waste facility is required to monitor water supply wells, samples from those wells must continue to be analyzed by methods equivalent to, or at least as stringent as, the NR 809 Safe Drinking Water methods (NR 507.20).

Because the NR 507 analytical methods requirements legally apply to solid waste facilities, we are separately notifying the facilities and solid waste interested parties of the availability of this increased analytical methods flexibility. In the long-term, we plan to propose revisions to NR 507.17(4) that would replace the reference to SW-846 with a reference to NR 219.04. Until any rule revisions are effective, relying on this increased flexibility will give everyone a chance to try this new approach before it is formally adopted.

For more information, please contact Jack Connelly at (608) 267-7574 or by email at

[johnston.connelly@wisconsin.gov](mailto:johnston.connelly@wisconsin.gov)



### **LabCert On the Move (Again)!!!**

Last May, the LabCert program was re-located back within the GEF buildings (GEF 3) as the first step towards re-integration with the rest of DNR staff in the renovated GEF 2 building.

Currently we are slated to move back into the GEF 2 building sometime this summer, likely in August. Our final destination will be the 7<sup>th</sup> floor.

All of our phone numbers should transfer with us. Mail delivery will change somewhat. Once the move is finalized, our mail “code” (placed after the name of the staff person you are communicating with to facilitate delivery) will be “- SS/7”. In the interim, the current code, “- SS/GEF3”, should still get mail to us. Thanks for your patience.



## Shipping Concentrated Nitric Acid

By Ron Arneson

If you ship concentrated nitric acid (UN2031) to field staff for preservation of environmental samples, be aware that there is **no longer a limit quantity exemption** for this corrosive material. This means that you must follow the requirements in 49 CFR 172 and 173 for packaging, marking and labeling of a non-bulk class 8 packaging group I material. The U.S. Department of Transportation (DOT) rules can be found at <<http://hazmat.dot.gov/regs/rules.htm>>. Please review the following regulations 49 CFR 172.101, 172.301, 172.400, 173.136, 173.137, and 173.158.



Environmental samples that have been preserved with nitric acid are not considered a hazardous material (the acid is not concentrated enough). See the letter from DOT to the U.S. Environmental Protection Agency dated 02/13/2003. A copy of this letter can be found at:

<http://www.epa.gov/epaoswer/hazwaste/test/news.htm#dot> .

If you have any questions on this, contact Ron Arneson at (608) 221-6322 or

[ronald.arneson@wi.gov](mailto:ronald.arneson@wi.gov). 

## EPA Eliminates Freon-113 O&G Methods

Source: EPA QA Newsletter, March 2007-05-08

### Removal of EPA Method 413


The Federal Register Method Update Final Rule, published on March 12, 2007, withdrew the approval of all Oil and Grease methods that use chlorofluorocarbon-113 (CFC-113; Freon-113) as an extraction solvent from the 40 CFR Part 122, 136.

This Rule [took] effect April 12, 2007, and will be implemented by all facilities that monitor for Oil and Grease.

The Clean Air Act Amendments of 1990 require the production phase-out and cessation of import of ozone-depleting substances including chlorofluorocarbons (CFCs), in order to protect Earth's ozone layer. Method 1664A was developed by the U.S. Environmental Protection Agency (EPA) to replace Method 413.1 and Standard Method 5520B, both of which use Freon 113, a Class 1 CFC, as the extraction solvent for the determination of oil and grease and petroleum hydrocarbons. Method 1664A, which uses n-hexane as the extraction solvent is a gravimetric procedure applicable to aqueous samples for the determination of oil and grease and TPH.

The most significant changes in Method 1664A compared to other oil and grease and TPH methods is: (1) the use of n-hexane as the extraction solvent, (2) the use of standards of known composition and purity as the spiking materials for QC analyses, and (3) the introduction of extensive quality control (QC) parameters. For more information regarding Method 1664A, visit EPA web site.

### What it means for permitted facilities

- If your WPDES permit is close to re-issuance, it will likely be reissued listing the hexane method as the required technique.
- If your WPDES permit is not close to re-issuance, you will be contacted regarding the EPA's action and informed of our intent to revise NR219 accordingly.
- We'll be encouraging facilities to voluntarily switch to the HEM procedure as soon as possible.
- The Department will be moving as quickly as possible towards deletion of the Freon methods in the SWAMP database. 



## **Federal Register 3/12/07: Methods Update Rule – What Does it All Mean?**

By Diane Drinkman

*This information was provided to Laboratory Certification Standards and Review Council members and guests on May 17, 2007. It merely summarizes changes at 40 CFR Part 136, that will be incorporated in the various tables of NR 219. Some minor revisions have been made to the original document, for clarity.*

### Changes to 40 CFR Part 136

- Approves new and revised methods from Standard Methods for the Examination of Water and Wastewater. Hard copy methods are identified by the method number (e.g., SM 4500-Cl E) and the edition of the manual in which they appear (e.g., 18th, 19th & 20th ed.), while the electronic versions are identified by method number and year approved (e.g., 4500-Cl E-00). The tables have been reformatted to include analytical technique as a separate column, with Standard Methods 18/19th edition grouped, a separate 20th edition column (to better accommodate the renumbered methods) and a fourth column for SM Online.
- Approves three new methods submitted as ATPs (Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte (D6508, Rev. 2) by Waters Corporation, Digestion and Distillation of Total Cyanide in Drinking and Wastewaters using MICRO DIST and Determination of Cyanide by Flow Injection Analysis (QuikChem Method 10-204-00-1-X) by Lachat Instruments, Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate (Kelada-01).
- Approves EPA Method 200.2 (Revision 2.8, 1994) for use with ICP, ICP-MS, STGFAA (Stabilized Temperature Graphite Furnace) and FLAA.
- Approves three new procedures for ICP-MS: EPA Method 200.8 (Revision 5.4, 1994), AOAC Method 993.14 [16th ed.], ASTM D5673-03.
- Approves EPA Method 200.9 (Revision 2.2, 1994) for STGFAA.
- Approves four new methods for Cr6+ by ion chromatography: EPA Method 218.6, AOAC Method 993.23 [16th ED], ASTM D5257-97, SM 3500-Cr E [18th/19thedition], SM 3500-Cr C [20thedition].
- Approves five new methods for ion chromatography: EPA Method 300.0 (Revision 2.1, 1993), EPA Method 300.1 (Revision 1.0, 1997), AOAC Method 993.30 [16thedition.], ASTM D4327, SM 4110 B [18th/19th/20thedition.].
- Approves six automated cadmium reduction methods for nitrate and nitrite; EPA Method 353.2 (Revision 2.0, 1993), ASTM D3867-99 (A) and (B), SM 4500-NO3 E and F [18th/19th/20th edition], 4500-NO3 E-00 and F-00, USGS Method I-4545-85.
- Approves 2 methods for chlorine by low level amperometry: SM 4500-Cl E [18th/19th/20thedition.], SM 4500-Cl E-00.
- Replaces: EPA Method 180.1 (1978) with EPA 180.1 (Revision 2.0, 1993), EPA Method 200.7 (1990) with EPA 200.7 (Revision 4.4, 1994), EPA Method 245.1 (1974) with EPA 245.1 (Revision 3.0, 1994), EPA Method 335.3 (1978) with EPA 335.4 (Revision 1.0, 1993), EPA Method 350.1 (1978) with EPA 350.1 (Revision 2.0, 1993), EPA Method 351.2 (1978) with EPA 351.2 (Revision 2.0 1993), EPA Method 353.2 (1978) with EPA 353.2 (Revision 2.0, 1993), EPA Method 365.1 (1978) with EPA 365.1 (Revision 2.0, 1993), EPA Method 375.2 (1978) with EPA 375.2 (Revision 2.0, 1993), EPA Method 410.4 (1978) with EPA 410.4 (Revision 2.0, 1993), EPA Method 420.2 (1974) with EPA 420.4 (Revision 1.0, 1993).
- Approves new methods: EPA Method 245.7 [Revision 2.0, 2005] (EPA-821-R-05-001) for mercury, ASTM D6888-04, for available cyanide, ASTM D6919-03, for cations, SM 4500-Cl- D [18th/19th/20thedition] and SM 4500-Cl- D (2000) for chloride by potentiometry, ASTM D512-89 (1999) for chloride by ISE, SM 4500-CN- F [18th/19th/20thedition.], SM 4500-CN F-00, and ASTM D2036-98 A for total cyanide by ISE, SM 4500-S2- G [18th/19th/20thedition.] and ASTM D4658-03 (1996) for sulfide by ISE, SM 4500-NO3 [18th/19th/20thedition.] and SM 4500-NO3 (2000) for nitrite by ISE (*missing specific method, assumed to be "D"*).

( Continued on next page)



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- Approves: Errata sheet to correct typographical errors in toxicity methods manuals, use of 1994, 1996, and 1999 Annual Book of ASTM Standards Vols.11.01 and 11.02, 2000 Annual Book of ASTM Standards, Vol. 11.02 and individual standards published after 2000, Newer versions of 19 methods published by AOAC International, as published in Official Methods of Analysis of AOAC-International, 16th edition, 1995.
- Approves copper sulfate as catalyst for TKN.
- Approves styrene divinyl benzene beads and stabilized formazin for turbidity.
- EPA is adopting a new §136.6 to introduce greater flexibility in the use of approved methods, how approved methods may be modified and the requirements that must be met to use these modified methods without prior EPA approval.
- Includes language at §136.6 (c) to clarify that analysts need only meet method performance requirements for target analytes (those analytes being measured for NPDES reporting) when using multi-analyte methods.
- Includes the language at §136.6 (d) to allow explicitly the use of capillary columns with EPA Methods 601-613, 624, 625, and 1624B and addresses the required retention times as specified in these methods are not method requirements is using capillary columns.
- Withdraws 109 methods from “Methods for the Chemical Analysis of Water and Wastes”.
- Withdraws EPA Methods 612 and 625 as approved methods for the dichlorobenzenes.
- *Withdraws approval of all oil and grease methods that use Freon-113 as an extraction solvent.*
- Revises Required Containers, Preservation Techniques, and Holding Times table and the footnotes, the most significant of the changes are *clarifying "Maximum Holding Time" to "Analyze within 15 minutes" for Total Residual Chlorine, pH, DO*, and interferences by sulfur, sulfide, sulfite, thiocyanate and other compounds, as related to cyanide preservation.
- Updates ATP submittal information.
- Other minor editorial revisions to clarify existing regulations.

**PLEASE NOTE:** *Although the Federal Register states that this regulation was effective on April 11, 2007, laboratories that perform analyses for WPDES permits must continue to use the methods listed in ch. NR 219, Wis. Adm. Code.*

#### NR 219 Code Revision Process

- Working with watershed staff on technical issues (e.g., removing Freon oil & grease monitoring requirements still in facility permits, inclusion of Standard Methods 21st edition).
- May consider removing obsolete methods, or those that generate hazardous wastes when alternatives are available (e.g., brucine method for nitrates, mercuric nitrate titration for chloride, etc).
- All tables in NR 219 will be repealed and recreated. Tables will become landscape orientation to accommodate changes.
- Anticipate "Green Sheet" for public hearing authorization for the October Natural Resources Board meeting.
- Anticipate public hearings in November 2007 (2 hearings in Wausau and Madison).
- Anticipate "Green Sheet" for adoption January 2008.
- Potential effective date, beginning of FY 2009 certification period (September 2008).

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*Diane Drinkman, WDNR Audit Chemist, will be modifying ch. NR219 to reflect these changes, starting this summer. Laboratory and WPDES permittees will be notified as this project progresses. Contact Diane at (608) 264-8950 or via email: [diane.drinkman@wisconsin.gov](mailto:diane.drinkman@wisconsin.gov) if you have questions.*



# Proficiency Testing

## Electronic PT Upload

Mike Kvitrud and Diane Drinkman have worked diligently to finalize the upload system which will be

used by PT Providers to directly upload PT results for labs in our program. This significantly reduces our labor and should minimize PT-related problems.

### PT Providers can access it at:

<http://www.dnr.state.wi.us/org/es/science/lc/PT/PTupload.htm> 

Wisconsin Department of Natural Resources

[Close Window](#)

### PT Provider PT Upload Gateway


NOTE: This page is not for laboratory use.  
This page is reserved for PT providers to upload PT results into the Lab Certification database.

This page has been developed to offer PT Providers a gateway through which PT results for Wisconsin certified and registered laboratories can be uploaded to our system. Once uploaded, we have developed additional tools which will then match the PT results with laboratories in our system and update the PT records appropriately for these labs.

The DNR Switchboard allows a person with environmental involvement to review and update facility and contact information. It connects authorized users to our electronic business tools without having to log in for each.

To facilitate this process, we have developed a [detailed set of instructions](#) (PDF 696KB) to guide PT Providers through the upload process.

## Electronic PT Upload- What Labs will see

With the launch of our new PT upload function, laboratories will automatically receive an e-mail like the one below informing them that our database has received PT results for their facility. The e-mail will be sorted to list any parameters with unacceptable PT performance first. The e-mail will also serve as documentation of which specific PT results we have received, and for which parameters. 

Dear **Labcert Contact**:

This email is to confirm that the Wisconsin DNR Laboratory Certification & Registration Program has received electronic proficiency testing results for **Your Laboratory, Inc.** (Lab ID: 00000000).

Please review your scope of accreditation to ensure that you have submitted PTs for all tests for which you maintain certification or registration. PTs are not required for several tests. A list of these tests is available online, at the program website:  
<http://www.dnr.state.wi.us/org/es/science/lc/pt/>.

If results for all tests that require PTs are passes, you have completed the PT requirement for annual renewal. If results for tests that require PTs are failures, you must analyze another PT and report acceptable results to the program before August 31st of this year. If your laboratory does not submit acceptable PTs for all tests that require them, your certifications or registrations will expire on September 1.

If "Ungraded" is listed in the Pass/Fail column, this PT sample has not been graded to date. Please reply to this email for further information.

WDNR Parameter	PT Study Date	PT Study	Pass/Fail
12 Base/Neutral/Acid Extract	04/14/2006	ERA WP 0133	Ungraded
14 Acid Herbicides	04/14/2006	ERA WP 0133	Ungraded
16 PCBs	04/14/2006	APG WP 0604	Failed
16 PCBs	10/13/2006	APG WP 0604	Failed
02 Ammonia as N	04/14/2006	Wibby WP-138	Passed
08 Cobalt	10/13/2006	Wibby WP-138	Passed
08 Lead	10/13/2006	Wibby WP-138	Passed
08 Manganese	10/13/2006	Wibby WP-138	Passed
13 Explosives by LC	12/08/2005	ERA WP 0129	Passed

## Council Corner

By Kurt Knuth,  
Council Chair



As I began to prepare for my first official duty as the newly elected Council Chair, I read through the past “Council Corner” articles written by my predecessors to get some ideas of what to write about. Many articles are written to welcome and introduce new members to the council or to bid farewell to those who are leaving. As luck would have it, there are no new members joining at this time, and only one member leaving the council (more on that later), so there is no easy way out for me this time.

In between those articles dealing with the membership of the council, it is up to the Council Chair to come up with an idea for the content of this article. For my first foray into writing an article for LabNotes, I decided to delve into the inner workings of a council meeting and explain one of the regular topics that we deal with at every meeting: Laboratory Audits. Because the Council feels that laboratory audits are important to ensuring data quality, time is spent at each meeting to discuss audit numbers, audit backlog, report turn around, and open cases.

At each quarterly meeting the DNR certification staff reports to the council on how many laboratory audits have been conducted, how many reports have been written, and how many cases have been closed. The certification program has set annual goals for each of these items for both the central office and regional program, which I am happy to say are usually met or exceeded. On occasion situations arise such as the NR149 revision or staffing changes that adversely affect the audit numbers for a given quarter, but by the end of the certification period the backlog is usually erased.

The council is also presented with a priority report, which lists all of the labs in the program along with their last seen date, last report date, and date completed. This report allows the council members to quickly see if there have been delays in auditing labs, getting reports back to the labs, or in closing the cases. Council members often inquire about reasons for the audit

backlog, report delays or case closures.

Now just the mere mention of a DNR laboratory audit can send some laboratory personnel running into a dark corner to hide, but in reality the audit process is in place not only to ensure that labs are following the letter of the law, but also to help the analysts become better at the testing they perform. In the end, whether it is the DNR, a commercial lab, or a water/wastewater treatment facility, we should all be working to generate the highest quality data possible to protect Wisconsin’s natural resources.

As I alluded to earlier, one member of the council will be leaving in July as his second term is ending. Randy Herwig, representative for small municipal wastewater plants, has been serving on the council for six years, the past year as the Council Chair. We will miss his contributions and wish him well in the future

### ***New Council member Appointment***

As hard as it is to believe, Randy Herwig (Small Municipal Wastewater Plant representative) is closing out his 2<sup>nd</sup> 3-year term and must now rotate off the Council. All of us in the LabCert Program want to thank him for his hard work and dedication to the Program.

As a new member is appointed, we’ll let people know via the “What’s new” page on the Laboratory Certification website.

Current Council Members		
Representation	Name	Phone# / e-mail
Small Municipal Wastewater Plant	Randy Herwig	(608) 592-3247 rherwig@wppisys.org
Large Municipal Wastewater Plant	Kurt Knuth (Chair)	(608)222-1201 x293 kurtk@madsewer.org
Public Water Utility	Katie Edgington (Vice-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	(414) 475-6700 david.kliber@sflabs.com

# Wastewater Issues

## EPA Memo on Discrete Analyzers

The issue of whether or not Discrete Analyzers are approved for compliance testing—and how approval is obtained—has been tossed around quite a bit.

Recently, the EPA released the following memo which should clarify the role that discrete analyzer methods play in compliance testing.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460

**MEMORANDUM**

**SUBJECT:** Flexibility to Modify CWA Methods - Automated Methods  
**FROM:** Richard Reding, Chief  
Engineering & Analytical Support Branch, EAD, OST  
**TO:** Quality Assurance Managers  
ATP Coordinators  
NPDES Coordinators  
**DATE:** April 2, 2007

OFFICE OF  
WATER

The Methods Update Rule, published March 12, 2007 (72 FR 11200), added a provision that provides the regulated community with the flexibility to automate analysis by an approved Clean Water Act (CWA) methods without further EPA action. Previously, in a Memorandum (Guidance on the Use of Discrete Analyzers Under EPA Clean Water Act Programs), dated January 27, 2005, Geoffrey Grubbs, Director of the Office of Science and Technology (OST), had provided guidance on the use of discrete analyzer instrumentation for permitting and compliance monitoring under the Clean Water Act. The amended regulations at 40 CFR Part 136.6 (72 FR 11239-40) authorize an analyst to modify an approved test procedure in certain circumstances, provided that the chemistry of the method or the determinative technique is not changed. Further, the regulations explicitly state as follows:

Potentially acceptable modifications regardless of current method performance include changes between automated and manual discrete instrumentation 40 CFR 136.6(b)(1)(i).

With respect to Discrete Analyzers, approval letters for the following Discrete Analyzer methods have been released :

### WestCo ATPs

- NO3-0015 (SmartChem# 110-100C = EPA 310.2 Alkalinity)
- NO3-0016 (SmartChem# 230-200C = EPA 325.2 Chloride)
- NO3-0034 (SmartChem# 400-400B = EPA 420.4 Phenols)

### KoneLab ATPs

- NO6-0002 (KoneLab# EST-DWNO201-001 = SM 4500NO2- B (20th ed.) Nitrite)
- NO6-0021 (AquaKem# EST-DWN-01-001 = EPA 353.2 Nitrate)
- NO6-0022 (AquaKem# EST-DWP-02-001 = SM 4500P E Phosphorus)
- NO6-0023 (AquaKem# EST-DWSi-01-001 = SM 4500Si C silica)
- DO6-0001 (KoneLab# EST-DW CN SN4500-E = SM 4500CN E

Cyanide)

NO6-0001 (KoneLab# EST-DW CN SN4500-E = SM 4500CN E Cyanide)

### OI Analytical ATPs

- NO6-0004 (OI# 26930806 = EPA 350.1 for Ammonia),
- NO6-0005 (OI# 27000806 = SM 4500Cl E chloride),
- NO6-0006 (DA 3500 Chromium (VI) = SM 3500Cr B chromium(VI))
- NO6-0007 (OI# 26970806 = EPA 335.2 Cyanide),
- NO6-0008 (OI# 27020806 = SM 4500NO2- B Nitrite),
- NO6-0009 (OI# 26920806 = EPA 353.3 Nitrate/Nitrite),
- NO6-0011 (DA 3500 orthophosphate dual Reagent = SM 4500P E Phosphorus),
- NO6-0012 (DA 3500 Phenol, low range 4AAP = EPA 420.2 Phenol),
- NO6-0013 (DA 3500 Phenol, low range 4AAP = EPA 420.1 Phenol),
- NO6-0014 (DA 3500 Silica = EPA 370.1 Silica),
- NO6-0015 (DA 3500 Sulfate, Turbidimetric = SM 4500SO4= E Sulfate),
- NO6-0016 (DA 3500 Sulfide = SM 4500S2 D for Sulfide),
- NO6-0017 (DA 3500 TKN = EPA 351.2 TKN),
- NO6-0018 (DA 3500 Turbidity = EPA 180.1 Turbidity)

### SEAL ATPs

- NO6-0033 (SEAL#EPA-126-A=353.2 NO3+NO2),
- NO6-0034(SEAL#EPA-125-A=353.2 NO2),
- NO6-0035(SEAL#EPA-126-A=351.2 TKN),
- NO6-0036(SEAL#EPA-111-A=351.2 TKN),
- NO6-0037(SEAL#EPA-120-A=351.2 Total Phosphorus)



# Groundwater Issues

## Changes in Molybdenum Reporting

*Please submit any future monitoring test results, LODs, LOQs & reporting limits for Molybdenum, Total (GEMS parameter code 1062) as ug/L values.*

The program has changed the units for this parameter from mg/L to ug/L, to be consistent with:

- 1) our parameter for Molybdenum, Dissolved (GEMS p-code 1060), &
- 2) the following new Wisconsin public health groundwater quality standards, which have been in effect since December 1, 2006:

Substance	Enforcement Standard (ug/L)	Preventive Action Limit PAL) (ug/L)
Molybdenum	40	8

([www.legis.state.wi.us/rsb/code/nr/nr140.pdf](http://www.legis.state.wi.us/rsb/code/nr/nr140.pdf);  
NR 140.10, Table 1)





## **Changes to NR 140 Preventive Action Limits (PAL)**

A revision to ch. NR 140 (Groundwater Quality standards) was promulgated in January 2007. Of particular relevance are changes to the Enforcement Standard (ES) and Preventative Action Limit (PAL) for the following compounds:

### **Butylate:**

- ES raised to 400 ug/L ( from 67 ug/L )
- PAL raised to 80 ug/L (up from 80)

### **Dacthal:**

- ES lowered to 70 ug/L ( from 4 mg/L)
- PAL lowered to 14 ug/L (from 0.8 mg/L)

### **Molybdenum:**

- New analyte; establishes an ES of 40 ug/L
- New analyte; ; establishes a PAL of 8 ug/L

### **Naphthalene:**

- ES raised to 100 ug/L (up from 40 ug/L)
- PAL raised to 10 ug/L (up from 8).



Watch for updates at:

<http://dnr.wi.gov/org/aw/wm/monitor/Alert.asp>

## **Drinking Water Issues**

### **New Reporting Limit and Calibration Requirements for Disinfection Byproducts**

The Federal Register Notice of January 4, 2006 contained some provisions that became effective April 1, 2007. Many laboratories may have missed these new requirements.

The requirements are part of 40 CFR Part 141.131 (b)(2)(iv) and state the following:

(iv) *“Beginning April 1, 2007, report quantitative data for concentrations at least as low as the ones listed in [Table] for all DBP samples analyzed for compliance...”*

The Table referenced in the Federal Register notice is summarized below:

### **Reporting Limit Requirements**

The rule establishes the following Minimum Reporting limits:

#### **Trihalomethanes (THM): 1 ppb (0.001 mg/L):**

- Chloroform
- Bromodichloromethane
- Dibromochloromethane
- Bromoform

#### **Haloacetic Acids (HAA): 1 ppb (0.001 mg/L):**

- Dichloroacetic Acid
- Trichloroacetic Acid
- Monobromoacetic Acid
- Dibromoacetic Acid

#### **Haloacetic Acids (HAA): 2 ppb (0.002 mg/L):**

- Monochloroacetic Acid

#### **Chlorite: 20 ppb (0.02 mg/L)**

#### **Bromate: 1 ppb (0.001 mg/L)**

for methods 317.0 rev.2, 326.0, or 321.8

#### **Bromate: 5 ppb (0.005 mg/L)**

For all other methods

### **Calibration Requirements**

The calibration curve must encompass the regulatory minimum reporting level (MRL) concentration. Data may be reported for concentrations lower than the regulatory MRL as long as the precision and accuracy criteria are met by analyzing an MRL check standard at the lowest reporting limit chosen by the laboratory.

The laboratory must verify the accuracy of the calibration curve at the MRL concentration by analyzing an MRL check standard with a concentration less than or equal to 110% of the MRL with each batch of samples. The measured concentration for the MRL check standard must be  $\pm 50\%$  of the expected value, if any field sample in the batch has a concentration less than 5 times the regulatory MRL.

Method requirements to analyze higher concentration check standards and meet tighter acceptance criteria for them must be met in addition to the MRL check standard requirement.



## General Interest


### Luminescence DO Update

The Methods Update Rule, recently published as Final Action in the March 12, 2007 Federal Register, unfortunately did not include the addition of luminescence DO (LDO) technology.

The ASTM website indicates that the method just approved by EPA, ASTM D888-92, 03 has been

superseded by method D888-05 (A-C). The LDO procedure, D888-05 (C), received interim approval for use from Bill Telliard last January (see below).

The ATP interim approval states "We recommend that the interim approval be effective from the date of this memorandum..."

If you are interested in using LDO methods for measurement of DO or BOD, contact your auditor for more information. 




UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460

JAN - 3 2006

OFFICE OF  
WATER

#### MEMORANDUM

**SUBJECT:** Recommendation for Interim Approval of ASTM International Standard Test Method D 888-05 (ATP Case No. N05-0046)

**FROM:** William A. Telliard   
Director of Analytical Methods

**TO:** USEPA Regional Administrators (all Regions)

In accordance with the authority specified at 40 CFR part 136.5, I recommend that each EPA Region grant interim approval of ASTM International D 888-05 *Standard Test Methods for Dissolved Oxygen in Water* (ATP Case No. N05-0046) for measurement of dissolved oxygen (DO) in wastewater and in for use in measuring DO in methods approved at Title 40 of the *Code of Federal Regulations*, Part 136.3 (40 CFR Part 136.3) for measuring biochemical oxygen demand (BOD). EPA has reviewed the methods and supporting validation data and determined that the methods, including a new procedure, Test Method C (Luminescence-Based Sensor Procedure), meet all requirements for approval as compliance monitoring methods. We recommend that the interim approval be effective from the date of this memorandum to the effective date of a final rule(s) promulgating the methods in the *Federal Register*.

If I can be of any additional assistance on this matter or others, please contact me at [telliard.william@epa.gov](mailto:telliard.william@epa.gov) or at 202/566-1061 at your convenience.

cc: Quality Assurance Managers (all Regions)  
Water Management Division Directors (all Regions)  
ATP Coordinators (all Regions)  
Len Morrissey, ASTM International  
Kevin Roberts, CSC, SCC

## Excerpts from EPA Region 5's "QA Update" Quarterly Newsletter

The following are two items of note from the May 2007 Issue of Region 5 QA Update Newsletter.

### Revised "Pumpkin Book"

On April 10, the United States Environmental Protection Agency (USEPA) published a new document titled "Solutions to Analytical Chemistry Problems with Clean Water Act Methods". This document is a revision of the "Pumpkin Book". In 1993, USEPA published document titled "Guidance on Evaluation, Resolution, and Documentation of Analytical Problems associated with Compliance Monitoring".

The document was known also as the "Pumpkin Book" because of its pumpkin colored cover.

This new USEPA document represents significant

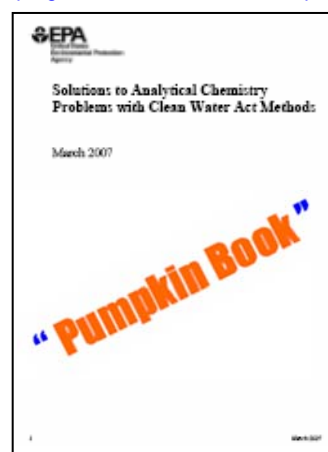
improvements to the earlier document because it includes more information on inorganic chemistry; specific references to regulatory text that supports a performance based approach to method modifications, and offers updated solutions to analytical problems.

This 47-page document is organized into 9 chapters: (1). Introduction, (2). Sampling Requirements, (3). Flexibility to modify an Analytical Method, (4). Data Required to Document Matrix Interference, (5). Case Histories of Reports of Matrix Interferences, (6). Solutions to Matrix Interference

Problems, (7). Review of Data from Analysis of Samples, (8). When a Matrix Interference is Demonstrated, (9). Sources of Additional Help.

This new "Pumpkin Book" can be browsed or downloaded from the USEPA website at:

<http://www.epa.gov/waterscience/methods/pumpkin.pdf>



### Revised EPA Guidance QA/G-6

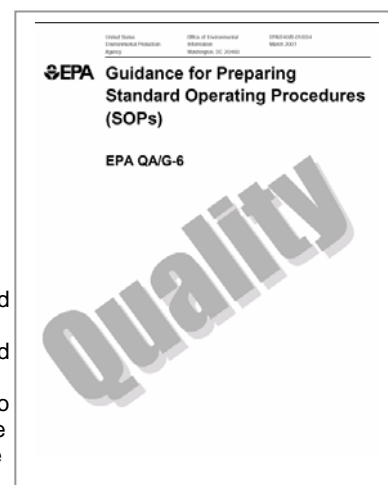
On April 16, 2007, Quality Staff, Office of Environmental Information (OEI), United States Environmental Protection Agency (USEPA), published a guidance document entitled "Guidance for Preparing Standard Operating Procedures (SOPs), EPA QA/G-6, EPA/600-B-07/001, April 2007." This is a revised version of the guidance document EPA QA/G-6, EPA/240/B-01/004, March 2001.

It is USEPA policy (EPA Order 5360.1 A2) that requires all USEPA organizations supporting environmental programs to document their management and technical operating procedures. The development and use of Standard Operating Procedures (SOPs) are an integral part of a successful quality system as SOPs provide the information to

perform job properly and facilitate consistency in the quality and integrity of a product or end results. SOPs describe both technical and administrative operation elements of an organization that would be managed under a work plan or a Quality Assurance Project Plan and under an organization's Quality Management Plan. Guidance for Preparing Standard Operating Procedures is designed to provide guidance in the preparation and use of a SOP within a quality system. It is U.S. EPA's policy (EPA Order 5360 A1, May 2000) that all USEPA requirements and guidance documents are valid for a period of up to five years from the date of publication. After five years, USEPA requirements and guidance documents are either re-issued without change, revised, or

withdrawn from the U.S. Environmental Protection Agency Quality system series documents.

The EPA QA/G-6, March 2001 document has been revised and published as EPA QA/G-6, April 2007. This document will be valid for a period of up to five years from the date of publication. Different example SOPs are provided in Appendix A-E. These example SOPs are provided merely to demonstrate application of SOP format to technical and administrative subjects, and should not be cited or followed as actual procedure specification or guidance.



The new SOP Guidance can be browsed or downloaded from the USEPA website at:

<http://www.epa.gov/quality/qs-docs/g6-final.pdf>



## LabNotes - Summer 2007

Volume 22, Issue 1

Wisconsin Department of Natural Resources

101 South Webster Street

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### Important Dates:

6/30/07: Fee payments due  
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7/1/07: FY08 Fee schedule takes  
effect

8/31/07: All PTs and fee  
payments must be  
received to be renewed

9/1/07: FY08 Certification period  
begins

