

Laboratory Certification Standards Review Council Meeting Minutes From 11/12/2013

Attendance

Council Members: **In Person:** Randy Thater (Chair), Kirsti Sorsa (Vice-Chair), Pat Gorski, Paul Harris, Paul Junio
via LIVE Meeting: Kurt Birkett (Secretary)
absent: Jennifer Peth

DNR Staff: Jack Sullivan, Rick Mealy

Others in Attendance: Sharon Mertens, (Milwaukee MSD), Craig Martin (WE Energies), Steve Geis (WSLH), Tom Hungerford (SF Labs) – via LIVE Meeting

The meeting was called to order at 10:01 am

Jack Sullivan introduced Steve Geis as the new Laboratory Certification Program Section Chief. Steve is currently the Supervisor for Organics and Environmental Toxicology at the State Laboratory of Hygiene. He will assume the chief role on December 2nd.

Minutes from meeting of August 21, 2013

- o **Action:** A motion (Harris/Junio) to approve the August 21, 2013 minutes as presented was unanimously approved.

Program Performance Status Report for FY 2014 Year-to-Date

- Audits - Commercial/Public Health: **15** (34); Municipal/Industrial: **29** (89)
- Reports - Commercial/Public Health: **12** (34); Municipal/Industrial: **27** (89)
 - o Reports Due - Commercial/Public Health: **5**; Municipal/Industrial: **6**
- Closures - Commercial/Public Health: **11** (34); Municipal/Industrial: **27** (89)
 - o Open Cases - Commercial/Public Health: **18**; Municipal/Industrial: **31**
- For the fiscal year 2014 to-date, **78.6%** of reports for audits conducted were issued within 30 days
- o There was also some discussion regarding the large number of open applications and applications from new labs. Most present believed this to be an anomaly rather than an indication of an uptick in the economy.
- o Council members requested a status update on open cases for two labs.

Budget Variance Report for FY 2014

- o The following projections were reported to the Council for the opening of fiscal year 2014:

BEG CASH BAL (7/1/13)	\$ 675,474
+ REVENUE COLLECTED FY14 (fees)	\$ 41,736
- FY14 EXPENDITURES (budget)	\$(132,792)
- FY14 EXPENDITURES (not yet transferred)	\$(14,108)
- FY14 LAPSE	\$ TBD
PROJECTED END CASH BAL (BEG FY15 CASH BALANCE)	\$ 570,311

- The following information represents the status of spending to-date, but does not completely reflect allocable charges incurred. Expenses are expected to be approximately 25% of budget.

	BUDGET	ACTUAL	% of BUDGET
Salary	\$301,882	\$61,834	20%
Indirect labor	\$20,916	\$16,659	80%
Fringe	\$150,779	\$46,662	31%
SALARY+ FRINGE	\$473,577	\$125,155	27%
Contractor	\$24,000	\$4,956	21%
Travel, Supplies & IT	\$44,050	\$9,003	20%
Indirect supplies	\$18,600	\$5,049	27%
Additional Allocables	\$20,175		
SUPPLIES/IT	\$106,825	\$19,008	18%
FY2014 BUDGET	\$580,402	\$144,163	25%
Allocables	\$59,691	\$23,233	39%

Other DNR Business

- Variances - No variance requests were received.
- NR 149 update – Work on NR 149 may begin once NR 219 is on track.
- NR 219 update – Currently on track to go before the Natural Resources Board to request authorization to hold public hearing. Likely will occur in January as well as public comment period. Assuming minimal comments, plan is to turn the rule around and go for adoption in April 2014.
- Reciprocity Issue (SC): South Carolina (SC) has never recognized the agreement we believed was in place. They are willing to maintain an agreement, but it would require us to change the manner in which we conducts audits for labs accredited in South Carolina. The LabCert program position is that it is likely easier to officially terminate the agreement, which covers only two labs.
 - *The Council agreed that what SC proposes does not fit the definition of reciprocity, so the agreement should be terminated. Council members indicated that we should inform the two labs whose accreditations could be affected in writing. Because we do not know which labs may be using their WI accreditation in SC, we may have to inform all labs.*
- ICP training venture (with State Lab of Hygiene) – Progress is being made towards the ICP training session. A survey has been sent out to all labs that will help shape the session.
- Low Level Phosphorus PT– The State Lab of Hygiene (SLH) will be sending out a low level PT to all labs that are certified for total phosphorus and use the SLH as their PT Provider. The low level PT is voluntary and will not be used for accreditation. We are only attempting to evaluate how labs are progressing in lowering their LODs.

Council Member Issues

- Letter from NLS to DNR Secretary Stepp – Paul Junio requested that a letter from Northern Lake Service to Secretary Stepp be included with the minutes. The letter questions accreditations granted to a lab new to the program and is attached to these minutes.

Next Meeting Date

- **Action:** *The next Council meeting was scheduled for 10:00 AM Tuesday, January 7, 2014 at the DNR Science Operations Center (2801 Progress Road, Madison). In the event of inclement weather, Thursday January 9th (same time & location) was reserved as an alternate date.*

The meeting was adjourned at 12:30 PM.

October 10, 2013

Ms. Kathy Stepp, Secretary
Wisconsin Department of Natural Resources
101 South Webster Street, PO Box 7921
Madison WI 53707-7921

Dear Secretary Stepp,

We operate an environmental laboratory, Northern Lake Service, Inc. employing 30 technical and support people here in Crandon, and four more in our Waukesha facility. Since we began in 1974, we've seen many labs come and go in what has been a dynamic marketplace. In the early days, some labs did good work, some not so good, almost none with adequately documented data reliability.

In the mid-1980s the state recognized that accurate, defensible environmental data was crucial for improving and protecting the environment and public health in Wisconsin, and NR 149 was born. The strict standards set forth in the laboratory certification program were welcomed by the more reputable labs. A dedicated crew of knowledgeable, strict, helpful DNR audit chemists was hired and began conducting audits to enforce the new requirements. Some labs saw the handwriting on the wall and voluntarily shut down; others were decertified as a result of their first audit. Almost immediately, the credibility of results from Wisconsin's certified environmental labs set a new standard in the industry.

Wisconsin's lab cert program has not come without considerable cost, and requires that the participating labs fund the program. Our company has paid about \$300,000 into the program in lab certification fees and required associated costs since the inception of the program, and probably five times that much complying with it. This is not a complaint. It is important that our industry is trusted and that our data be beyond reproach by the regulators, clients, the courts, the press, the general public and anyone else whose reporting requirements, health and well-being depend on the certified test results. Any certified lab that does not strive to provide near-perfect, defensible data is a black mark on *all* the labs certified under the program, and on the state's lab cert program itself. We all expect the program to be tough.

Hence, it was extremely disappointing to learn that a start-up laboratory is now advertising that it is certified to operate in Wisconsin, when a recent DNR audit found approximately **300** deficiencies and/or non-compliances in that lab's operations and procedures. And, it's extremely discouraging for our 34 analysts and support staff, who struggle day in and day out to meet the strict requirements of a very complex set of program requirements, to learn that their careful effort might not be looked upon as being all that important after all.

We ask that the Department revisit the importance of the lab cert program and of the Department's responsibility to administer it to the benefit of all Wisconsin citizens, as intended. Millions upon millions of dollars are spent annually on environmental protection projects, the plans for which depend on accurate, credible lab results. Many times a day long- and short term public health decisions regarding drinking waters, ground water, and wastewaters are made after considering data generated by Wisconsin's certified labs. We hope that future certification decisions will reflect the original intent of the lab cert program.

Thank you for your consideration. We would be delighted if you could tour our facilities the next time you visit our area.

Respectfully,

Ronald K Krueger
Founder and Chairman of the Board
Northern Lake Service, Inc.

State of Wisconsin
DEPARTMENT OF NATURAL RESOURCES

1704 North 4th Street
Superior WI 54880
Telephone 715-817-8349

Scott Walker, Governor
Cathy Stepp, Secretary



July 26, 2013

FID: 268633970

Express Analytics LLC
Ms. Karri Warnock
600 W North Shore Drive
Hartland, WI 53209

SUBJECT: Laboratory Evaluation (7/9/13 - 7/11/13)

Dear Ms. Warnock:

On July 9 through July 11, 2013 Rick Mealy and I conducted an on-site evaluation of your laboratory to determine initial compliance with Chapter NR 149, Laboratory Certification and Registration, Wisconsin Administrative Code. Enclosed is a report that summarizes the observations we made while at your laboratory and after reviewing the data and procedures provided to us.

The laboratory must provide a response that includes data that proves that the corrective actions taken have resolved the deficiencies identified in this report. The initial response is to be received within 30 days of the date on this letter. If any corrective actions require more than 30 days to complete then include the planned correctives and expected dates of completion. The deficiencies are listed under the header "**Deficiencies – Supplemental Information**". The deficiencies are highlighted by presenting them in "**bolded**" text. Additional details that clarify the deficient practice are presented directly after the deficiency citation. These details are the "Supplemental Information" for the deficiency. At the very end of this report is a section titled "*Supporting Data*". This section indicates the information that the laboratory needs to provide with the audit response in order to demonstrate that the laboratory has taken the required corrective actions to resolve the deficiencies cited in this report.

It is important to respond to the report in its entirety, with the supporting documents clearly presented, and please send the response to me. The system wide deficiencies must be corrected satisfactorily before any certifications can be granted for any parameters. Once the lab-wide deficiencies are addressed then parameters that have their specific deficiencies also resolved may be granted on a case by case basis.

Both Rick and I would like to thank you, Brian, Ann and Travis for the time and assistance provided to us during the evaluation. We enjoyed meeting and visiting with all of you, discussing your laboratory practices, and seeing your analytical lab. As you read our observations and summaries, if you notice that we misunderstood any of your practices, please let me know so I can note the corrections in the file.

If you have any questions about this report or any other lab issues you can reach me by email or by phone at 715-817-8349. If you do have questions, in the interest of time and efficiency, please list them and send them together. We are also open to having an informal meeting in Madison if you would like. If so, please contact Camille Turcotte as soon as possible to set that up.

Respectfully,

Audit Chemist
Brandy.BakerMuhich@wisconsin.gov

Report of On-Site Evaluation
For
Initial Compliance with Chapter NR 149,
Laboratory Certification and Registration
Wisconsin Administrative Code

Of

FID: 268633970
Express Analytics LLC
600 W North Shore Drive
Hartland, WI 53209

By

Brandy Baker-Muhich
Audit Chemist
Bureau of Science Services
Wisconsin Department of Natural Resources
Environmental Science Services
1701 North 4th Street
Superior, WI 54880

July 9 – July 11, 2013

OVERVIEW

The Express Analytics LLC laboratory was evaluated for initial compliance with NR 149 on July 7 through July 9, 2013. The laboratory is new and this was the laboratory's first evaluation under our program. The Express Analytics LLC laboratory is owned by Karri Warnok and Brian DeJong and is a commercial laboratory that is seeking initial certification to provide a variety of analytical services for water, soil, and hazardous waste matrices.

Karri is the Laboratory Director and acting Quality Manager and Brian is the Operations Manager as well as an analyst. There are two other analysts, Ann and Travis.

All of the requirements set forth in this audit report apply to Wisconsin compliance samples only. If Wisconsin compliance samples are treated differently than other samples then two clear systems must be defined, documented, and maintained so that when Wisconsin compliance samples are received they are handled appropriately. The system wide deficiencies must be corrected satisfactorily before any certifications can be granted for any parameters. Once the lab-wide deficiencies are addressed then parameters that have their specific deficiencies also resolved may be granted on a case by case basis.

The evaluation process necessitates that the following report focus on deficiencies. However, I did observe positive practices at this laboratory:

- The laboratory was spacious and clean
- The equipment is very new and offers updated technology
- The laboratory has an ethics policy
- QC samples, surrogate, method blank, LCS, MS, and MSD (or sample duplicates) results can be reported with sample results
- Quality control limits are housed in the LIMS
- Balances are calibrated on each day of use
- Many of the devices are monitored with min/max thermometers
- Bottle blanks for LLHg are performed per lot
- The analysts were easy to work with and willing to consider recommendations

The deficiencies described in this report are those that were observed during the on-site evaluation and may have been corrected or otherwise addressed in the ensuing time between the evaluation and the receipt of this report. Performing a laboratory evaluation in the course of only a few days limits its scope; therefore, other deficiencies may exist in the laboratory. Their omission from the report does not imply acceptance by the Department. The laboratory is urged to correct all deficiencies even if they were not noted in this report.

Brandy Baker-Muhich
Audit Chemist

Reviewed by: Camille Turcotte, Chief, Environmental Science Services Section

I. FACILITIES and EQUIPMENT

Deficiencies – Supplemental Information

1. Analytical balance – weight verifications are not performed as required.

- NR 149.44 (3) (g) –

All three sets of weights are not currently certified. These sets of weights are used to perform the daily checks on both analytical and non-analytical balances. For two of the weight sets the original ID or lot numbers were not available, and the certification documentation was not available for review at the time of the audit for any of the sets. Weights must be purchased new or re-certified every 5 years. Records must be kept on file to demonstrate that the weights used are currently certified and are Class 1 or S quality. Also be careful of weight handling practices as the condition of weights (i.e. scratches) can affect the calibration.

2. A thermometer is required for the total volatile solids oven.

–SM 2540 G (b) (b) and 2540E (a) -

A NIST traceable thermometer capable of a measuring the muffle furnace at the 550°C temperature is required.

II. SAMPLE HANDLING

Deficiencies – Supplemental Information

1. The laboratory sample acceptance policy does not meet the requirements.

-NR149.46 (2) (a)-

The laboratory has a sample acceptance policy; however the conditions for which the samples will be rejected are not specified. In addition, the SOP includes an option that analysis will continue if the sample container is broken: A sample container was broken in transit. *“Analysis will proceed with existing sample. Reporting limits may be elevated. Contact lab representative with questions”*. Samples that have been significantly compromised or VOCs that are frozen are some examples in which the laboratory policy needs to be re-evaluated.

2. The laboratory has not verified that the sample collection containers used for all analyses do not contribute to the contamination of samples at levels (the limit of detection) that will affect sample determinations.

- NR 149.46 (1) (b) –

The manufacturer “certificates of cleanliness” received with the sample collection containers are generally inadequate to show that the sample collection containers do not contribute to the contamination of samples at the concentration levels that they are used for. Furthermore, the manufacturer “certificates of cleanliness” do not usually address all of the parameters that the containers are used for. As a result, the laboratory will need to perform container blanks for all of the tests it is seeking certification for in order to resolve this issue. For parameters where field blanks and trip blanks are analyzed the results of those samples (as long as they are reported to the limit of detection) would suffice for demonstrating bottle cleanliness.

The minimum requirement is to perform container blanks at least once, for each bottle type and vendor used, for each parameter and method that the containers are used for. The results of these studies (concentrations to the limit of detection) must be kept on file in order to demonstrate that the containers used are acceptable. In addition, the laboratory must document the protocol used in a standard operating procedure.

3. Narrow-bore pipettes are used for BOD, TS, and TSS analysis.

- SM 5210B (5) (c) (2), SM 2540A (2) -

Wide-bore pipettes must be used for preparing all BOD sample dilutions, total solids aliquoting, and total suspended solids aliquoting. A narrow-bore pipette may cause suspended material to be filtered out from the dispensed aliquot – resulting in a non-representative sample aliquot.

III. QUALITY MANUAL and SOPs

Deficiencies – Supplemental Information

1. The Quality Manual does not contain the required information.

-NR149.37 (3) (h) (f) and 149.03(29) (55) (56) –

- The Quality Manual lacks an organizational chart (or narrative) specifying the roles and responsibilities of the current staff.
- Section 10 in the Quality Manual does not include information for some of the analytical activities. While there is information for some techniques (GCMS/ICPMS) there is no information for the SmartChem® discrete analyzer or the Leeman® Mercury analyzer.
- There is no discussion of verification of calibrations, or the use of second source standards.
- There is mention of DFTPP tunes, but the lab lacks any equipment requiring DFTPP.
- There is no reference in the calibration section to the analytical SOPs.

2. Quality Manual does not reflect current, actual practices in the lab.

-NR149.37 (1) & 149.03(60)-

The Quality Manual describes objective quality assurance oversight and supervisory laboratory roles and responsibilities that are non-existent or not consistently followed. Although the goal was to establish this for future capabilities, it must be revised to reflect actual practices so it is clearly understood and ensures that laboratory personnel understand their roles and responsibilities. –NR149.25 (2)-

For example, the Quality Manual includes the information below that needs to be evaluated and clarified:

- There is a statement which seems to indicate there are many more employees than currently employed.
- The manual states that the Quality Manager functions independently of laboratory operations and there is quality assurance oversight to ensure non-bias and objectivity.
- The scope of testing listed is not representative of laboratory capabilities for compliance samples: there are statements of capabilities for public water systems and private wells which include an extensive list of parameters, matrices and services.

- It is lacking a clear system for Wisconsin compliance samples that is defined, documented, and maintained so that when Wisconsin compliance samples are received they are handled appropriately.
- The LOQ is defined in several documents that do not match each other; one description is 3xMDL, another is 2-10x MDL, and verbally was explained the RL would be the lowest calibration standard concentration, and a nominal LOD would then be set to the 3-5x the MDL. Currently the policy that would be used is unclear.
- None of the "Administrative" SOPs, including sample receipt, have been signed by any staff (as required in quality manual).

The above information includes the issues found during the preparation and on site evaluation, if other information is stated in the Quality Manual that is not representative of the current practices these must be corrected as well.

The Quality Manual needs to be reviewed for correctness and the requirements stated and followed. *The Quality Manual must include the elements in NR149.37 (3) with sufficient information that is also included in a consistent manner. The Quality Manual must reflect the actual practices completed at the laboratory. Please send me the updated Quality Manual.*

3. SOPs in use do not contain the required information.

- NR 149.40 (2) (d) (10) (12) (13) & 149.44 (4) (b) –

- The applicable matrices and parameters (and programs) that pertain to compliance samples needs to be referenced (to only include those certified for) or the SOP scope revised.
- The method calibration must be defined. The calibration equation and regression information used for the calibration function must be defined.
- For all methods where an instrument is zeroed (such as the colorimetric tests) the laboratory must define what solution is used to zero the instrument.
- The SOPs for the WestCo SmartChem do not contain specific calibration levels or how the standards are prepared. While some SOPs do specify this, all SOPs must include this information.
- The corrective actions and contingencies for handling out of control or unacceptable data are missing or insufficient in some SOPs.
- The reference for the laboratory policy allowing up to 20% of compounds to exceed the control limits must be included in the VOC SOP.

Reminder - When there are specific software settings known to cause problems with analysis, update the SOP with this information (for example add a clear statement in the 1631E SOP to make sure 3 of the 5 check boxes include a check mark so that all 3 system blanks are evaluated, since data was generated using only 2 blanks).

4. The SOPs in use do not reflect the practices being performed by the laboratory and personnel do not follow the all of the requirements in the standard operating procedures.

-NR149.40 (1) (a) and -NR149.40 (1) (c) (1)-

In general, each of the current SOPs in use need to be re-reviewed by the analysts to ascertain that the procedures being used in the laboratory are accurately reflected in the SOPs. After interviewing the analysts and reviewing the SOPs I have concern that the SOPs in use do not reflect the practices employed by the analysts. In some cases, the SOPs include information that does not apply to the practice being performed and in other cases there are critical practices being performed that are not part of the SOP. See NR 149.40 for the code required information that must be documented in each method SOP. The SOP needs to describe the exact procedures that are performed by the lab.

Currently some of the SOPs in use are missing significant information regarding instrument calibrations. Important information such as the standard concentrations used, the number of standards used, the regression used to reduce the data, the details on zeroing the instruments (is a calibration blank used, what exactly is it made of, is the response used in the regression), the frequency of the calibration, are the initial and continuing calibrations put through a preparation step, how are the CCVs and CCBs handled, etc.). In addition, there is a lack of detail in some SOPs for corrective actions and contingencies for handling out of control or unacceptable data in the SOPs. Quality control sample frequency and control limits, defined in LIMS, does not always concur with the QC requirements defined in the SOPs or methods. Maintenance information is not included in many of the SOPs. Make sure that the methods are re-reviewed for QC frequency and control limits and compare them to what is listed in your SOPs. Make sure that the method referenced is also correct.

There are lab SOPs that reference several different analytical methods, but when this is done, unless the samples are handled and treated differently, then the most stringent QC frequency and criteria must be met. Examples of this are: criteria for metals analysis using SW846 6020 and 6020A; VOC analysis by both 8260B and 624, COD both SM 5220D and 410.4. The reference of 245.7 in the SOP for 1631E indicates this method is utilized, however since this is not the case it should be removed.

The standard operating procedures requirements are not followed or consistently followed in some cases. While these are not inclusive of every SOP evaluated, they provide information the laboratory quality manager can utilize as examples to evaluate if the SOP is correct according to the reference method (and revise as needed); and then ensure the laboratory personnel follow the SOP. *Below are some examples, if others are discovered they must be corrected as well:*

- a. In the SOP for 1631E the following was noted but these practices were not in place at the time of the onsite visit:
 - A Class 100 clean bench is utilized.
 - The system blanks are evaluated for the standard deviation (n-1) and the criteria is <0.10 ng/L.
 - The analytical sequence in the SOP had not been followed in the data sets reviewed.
- b. The SOP for microwave digestion by SW846 3015A inaccurately states:
 - A spike concentration of 1,000 mg/L.
 - There is SOP language that needs to be clarified against statements made during the onsite visit related to if all parameters to be reported are spiked in the LCS, MS/MSD; as well as if the spiking level used is evaluated to the permit level as described.
- c. The SOP for ICPMS inaccurately states:
 - The correction factors stated in the SOP could not be verified.

- The auto tune criteria required in the SOP for minimum cps and oxide criteria is not being verified.
 - All the parameters in the tune solution are not evaluated.
 - The correlation coefficient must be *greater than* 0.998 for all analytes for the curve to be valid.
 - The Operations Manager has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.
 - Although preparation methods 200.8, 3005A, and 3020 was indicated as being utilized and followed in the SOP for ICPMS, these digestion procedures were not evaluated since the laboratory had not developed them at the time of the onsite visit.
- d. The SOP for TP:
- In the SOP for TP by 365.3, the digestion temperature is 120°C, the reference method says 95°C water bath, and the preparation log says 150°C.
 - The laboratory needs to evaluate if the method 365.1 is the applicable reference, since this one is the semi-automated procedure, while 365.3 is the manual method.
 - SOP for TP digestion section references that 1ml of a 0.576N sulfuric acid solution to be used, the method requires 11N sulfuric acid.
- e. The SOP for Hexavalent Cr:
- There is information pertaining to colored and turbid samples, however during the onsite evaluation, the analyst indicated they did not know the requirement for subtracting out the background absorbance of turbid or colored samples prior to calculating the final concentration. –SM4500Cr B (4c Note)-
 - The method used is Hach 8023, versus SM 3500Cr B, but since the requirements for both the methods are combined, the addition of 5N to the sample to achieve a pH of 2 +/-0.5 (from SM method) is done prior to the Hach 8023 Chromaver 3 reagent pillow (which is *on its own* designed to drop the pH to this range). The consequence is that over acidification can lead to exhausting the buffer capacity of the powder reagent. From Hach 8023: Highly buffered or extreme pH may exceed the buffering capacity of the reagents and require sample pretreatment.
- f. The SOP for Ammonia-N:
- The method appears to mirror SM in many aspects, including that the boric acid reagent is not applicable for the phenate method, however the laboratory is using boric acid and a phenate method.
- g. The BOD SOP:
- Is missing sufficient procedural steps and the evaluation for the calibration of the probe. It is critical this is done correctly, and although the SOP references a document, it is not sufficient.
- h. Sample Receipt:
- Preservation protocols for identifying preservatives added to bottles relies on a colored dot sticker system, however there actual color stickers used (of which there are four), did not indicate it meets their requirements of using 6 different colors.

All of the SOPs require review for correctness and the adherence to code and method requirements. Refer to the quality control and analysis sections in the report for addition updates required.

In general the SOPs do not allow for reconstruction of the procedures and quality control requirements being followed. Besides missing information, they are not useable when reference methods, multiple reference methods, vendor method and custom language is combined without fully customizing the document to the actual practices. Another consequence is that the mixed use of reference language can result in mixing procedural steps, such as reagents, which then can affect the chemistry and therefore the quality of the sample results.

One option for the laboratory to consider is once the SOPs reflect correct method requirements, is to have analysts utilizing the printed SOPs at the bench level for a period of time which would allow additional knowledge of the requirements and well as identification of any future updates that are needed.

IV. TRACEABILITY and RECORDS

Deficiencies – Supplemental Information

1. The laboratory does not consistently document all of the records to ensure that method and code traceability requirements are met.

- NR 149.39 (3) -

The laboratory must maintain all records necessary to allow historical reconstruction of all laboratory activities that contributed to generating reported results. Some examples of missing raw data that I noted were:

- a. Records: A thermometer record had been deleted from an excel spreadsheet, so the previous temperature check record was erased and could not be retrieved or reproduced. In cases where spreadsheets are used and paper copies are not printed or saved, the policy for deleting or changing this information must allow for record traceability and historical reconstruction.
- b. Records: There was an additional certification record in the thermometer file but no record of that thermometer was at the laboratory.
- c. Chemical preservation documentation: Where analysts document the preservation of samples is in the preparation log for each test, however these log sheets did not always contain a clear or labeled location to record it (which must be documented for every preserved sample container received).
- d. Sample receipt: When there are other sample receiving anomalies, then ensure this information gets transferred to the report.
- e. BOD: Sample temperature is not documented prior to dilution and set up as required for BOD/cBOD analysis.
- f. BOD: The room temperature and the barometric pressure are not documented for BOD/cBOD analysis.
- g. BOD: The laboratory is using liquid inhibiting agent for cBOD, but the addition is not documented.
- h. Reagents: The GGA preparation from solid reagents was not documented in the LIMS chemical tracking system.
- i. Solids: The temperature of the oven when samples are *put in* and *then taken out* are not documented in data examples.

- j. COD and Cr+6: The sequence of analysis is unknown and raw results are not saved for COD and Cr+6. Absorbance results are transferred from the displayed results on the spectrometer to a computer spreadsheet. The spreadsheet is set up in a format that does not allow the analytical sequence to be reconstructed. In addition, once the absorbance of a sample is entered, it is manually overwritten by the next sample result.
- k. The cBOD/BOD LOD and LOQ need to be 2 mg/L (if sample was not diluted); the test report reviewed included levels of 1 and 3.3 which is not theoretically possible. LOD also needs to be adjusted based on the dilutions run – a theoretical LOD of 2 mg/L is only appropriate if a 300 mL sample volume was analyzed.
- l. The low level Hg digestion bench sheet does not specify the technician/analyst.
- m. The amount of Silica Gel added to samples is not documented for the O&G HEM method.
- n. The RPD criteria for MS/MSD sets must be evaluated for O&G and O&G-SG. The QC limit for this needs to be added to the SOP.
- o. There is no digestion preparation log for COD samples.
- p. The method reference for TP in the SOP is 365.3, but the digestion log says 365.1.
- q. TP: The preparation log sheet includes a header with 4% ammonium persulfate, not 0.4 grams, or 1 ml of 40% solution.
- r. WI (95) GRO: There is no preparation log to document the extraction process, including shake and sonication times and sonicator settings.
- s. The maintenance records do not contain adequate detail, or the reasons completed in some cases.
- t. WestCo methods: Instrument printouts list a calibration lot number and then an expiration date of "12/12/2020", which incorrectly suggests that either the calibration standard or calibration is valid until that date.
- u. The TP digestion procedure has prefilled digestion temperatures. Worksheets are used to collect data that include some pre-filled information. Initials or names, dates, and raw data must never be filled out ahead of time.
- v. When there are temperature requirement of autoclaves, incubators, ovens, and water baths used as part of the method they need to be checked and documented that they meet the temperature requirements each day they are used. -NR149.4 (3)(e)-
- w. There are several issues related to initially incorrect preparation of standards, and then corrections made without traceability to the standard preparation documentation. A few examples noted are the 5/7/13 data for analysis of Silica, and the 5/10/13 analysis for Hexavalent Cr.
- x. The microwave preparation log indicates concentrations of ppm, instead of ppb.
- y. There was no filtration documentation applicable for samples that would be analyzed for dissolved Silica.
- z. Certificates of Accuracy are not retained for gas tight syringes used for organics.
- aa. GC/PVOC Instrument printouts label both the FID and PID chromatograms as "FID".
- bb. The truncated chromatogram quantitation reports indicate that the analytical run time is not extended for 5 minutes beyond the elution of Naphthalene, as required by method. Quantitation reports show a chromatogram run time of about 19-19.5 minutes and Naphthalene elutes at about

18 minutes. While the actual run time is correct, the printed quantitation report is perceived to be incorrect.

- cc. The method indication as labeled on the VOC data is unclear. For the run sequence 04.D from 2/11/13, the "Quant Title" is labeled "8260", but the analyst indicated that this was a drinking water analysis run (i.e., method 524.2).
- dd. NH3-N: There is no information in the reagent section for either the boric acid or 0.04N sulfuric acid.
- ee. TP: the 11N sulfuric acid solution is not in the SOP reagent section.
- ff. There were manual integrations performed on the VOC MDL analytical run, however there were no initials/dates/review (or explanation on the checklist) which are required per the laboratory policy.

Reminder – digital thermometers should not be assumed to be linear. If the thermometer is not checked at the temperature(s) it is used at, then document a check on temperature in the upper range, and one in the lower range.

Reminder – The policy on using correction factors for the NIST reference thermometers is unclear, and there was not a location to record this information in the laboratory record.

Reminder – when there is a pH check requirement, the color change of long range pH strips may not allow for the accuracy needed, therefore the lab should use short range pH indicator strips.

Reminder – Clearly document when thermometers are changed or become inactive.

Reminder- The chemical tracking system did contain at least one error (Mn versus Mg), therefore due to the amount of information here, this documentation needs periodic review.

Reminder- Address how the microwave vessels are evaluated for loss of sample.

Reminder – the COD absorbance results are remembered by the analyst and recorded in a computer excel sheet ~15 feet away – a log book for these observations is needed. Observations need to be documented near where the observation occurs; this was changed for some records (temperatures, balances, pipet checks) during the onsite visit. Update any applicable record policy that is applicable.

Reminder – currently the COD calibrations are completed with each analysis. If COD calibration curves are not completed each time in the future make sure the verification steps are followed per the method.

For each of the items listed above, send me a copy of the records that show you are documenting all of the required information. Also, if SOPs were updated, send me those along with the data that shows staff has been trained in these requirements.

2. Corrective actions are not consistently documented as required.

- NR 149.38 (3) and (4) –

Every time there is a quality control exceedance or deviation from an established procedure, corrective action must be taken to resolve the exceedance or deviation and this action must be documented.

Currently, the laboratory is not recording all of the required information regarding the corrective action. While the lab sometimes notes some corrective actions taken in instrument maintenance log books, or the QA staff inconsistently document some issues, this is insufficient. The following information that must be documented for corrective actions is as follows:

- What failed?
- What was done to try and fix the problem?
- Did the attempted fix correct the problem?
- If yes, how is it known that the attempted fix corrected the problem?

Every time there is a quality control sample failure corrective action must be taken to resolve the failure and this action must be documented. For corrective actions related to acute failures that are properly documented in your method SOPs (*for each quality control sample type, what the routine first course of corrective action is, and if that corrective action resolves the problem*) - then the corrective action course does not need to be documented each time.

Acute failures would be those that occur just once (not occurring on a repeating basis), corrective action is taken, and the failure is resolved.

If the acute failure happens repeatedly (for example more than once per month or more than 10% of the analytical runs) it may be a chronic problem and the established procedures for documenting corrective actions need to be completed.

During the on-site evaluation, it was noted there were quality control failures that required documentation. Some of the examples noted were: BOD blanks and GGA exceedances; ICV exceedances for ICPMS; Surrogate recovery problems for GC analysis; CCV known standard (222 ppm true value) had a recovery of only 84.8% (outside 90-110% criteria); a LDR check that failed requisite 90% (87.6%); blank failures for solids analysis.

It is best to have this information documented on the same form or in the same book for tracking purposes.

Let me know what your plan is for documenting corrective actions in the future. Send me a recent example of how you are recording corrective actions and your updated SOP that addresses this. Let me know how staff has been trained in this new requirement.

V. QUALITY CONTROL

Deficiencies – Supplemental Information

1. The responsibility for the Quality System is not specified clearly.

- NR 149.38 (3) (4) and 149.25 (2)–

As discussed in the Quality Manual and SOP section, the organizational authority and responsibilities requires clarification. Without clear understanding of these responsibilities, and without demonstration that these policies are being duly followed one cannot surmise that an effective quality system has been implemented.

The Quality Manual states there are department managers that have significant responsibilities to maintain the quality system, from assessing data quality, taking corrective action, and evaluating instrument performance, have supervisory roles and are responsible for reporting non-compliance situations to the Quality Manager and Laboratory Director. Since the lab has no Department Managers it is unclear who has these responsibilities and the consequence of this is that these requirements are not currently being met.

2. Corrective action is not taken for quality system or quality control samples, and the effectiveness of the corrective actions is not monitored.

- NR 149.38 (1) (a) (b) & (4)–

The laboratory records indicate that analysis was continued without resolving the reasons for the QC failures, although the laboratory Quality Manual and laboratory SOPs indicate that analysis is stopped immediately and does not continue until the problem is resolved.

During the on-site visit, conflicting information was relayed that if there was a qualifier in the LIMS system, then corrective action was not required. There were qualifiers in LIMS printed on test report examples for the failure of the analytical system, ICV and CCV failures. This does not meet the requirements for taking corrective action when there are departures from established policies and procedures, or there are quality control exceedances of acceptable limits.

Corrective actions for chronic or systemic issues that indicate potential data inaccuracy need to be investigated in greater detail and resolved.

As discussed above in the traceability section, the corrective action documentation in the QA files and analysts maintenance logbooks is incomplete. There were missing records for some PT and major instrument failures; in addition, of the actions for items entered that were in the spreadsheet only one of these was documented as resolved. Therefore it was observed that the implemented corrective actions are not being monitored.

The laboratory needs to have a process for completing corrective actions as required. In addition the system needs to be monitored for the documentation and effectiveness of the corrective actions.

3. The current limit of detection (LOD) and Initial Demonstrations of Capability (IDCs) studies are not valid for some tests:

-NR 149.03 (46) and -NR 149.36 (3) -

The laboratory sent many LOD studies, with the same parameters as before, but different dates, these also included parameters and matrices for which were not applied. Due to how the records were supplied without clear indications for which were used or not, it is not possible with reasonable effort to review this data. The laboratory needs to submit clear and pertinent data only so that it can be properly assessed. The laboratory should organize the records so only the passing study is included, for the specific matrix and parameter applied for. This is common practice for all applications.

- LOD Nitrate by method 300.0 on 4/29/13: the record supplied contains six results at ~ 0.05 mg/L; however one result is included at a level of 0.53 mg/L (which may be an error).
- IDC for TP – the data supplied indicates a concentration of 1ppm for the IDC, however the results are 0.5 ppm. During the onsite the analyst mentioned it was most likely a spiking error, but was not able to confirm.
- LODs and IDCs ICPMS – Many of the parameters have a date of 3/22/13, but I could not find comparable analysis results to the MDL spreadsheet. There are some analysis dates without passing verification or blank levels; the Li internal standard is failing for the ICV; interferences have not been evaluated; Be and TI MDL concentrations levels of 0.25, which are dilutions of 0.5 prepared spike solutions.

- LODs and IDCs for all compounds by method 300.0 must be re-established once the calibration function is reset and not forced through origin.
- The IDC for VOC soils was incorrectly prepared using only 1 gram of soil (980 uL of methanol), rather than following the extraction protocol as required.
- IDCs for TS, TSS, TVS, and TDS have not been completed.

Due to the potential changes which may occur with ICPMS it is possible the IDCs and MDLs for applied for parameters would need to be reanalyzed, however if this is not the case the data applicable to the MDL studies need to be carefully evaluated and explained, and the MDLs resupplied in a form that is reviewable.

Provide me a copy of the new or passing LOD records mentioned above.

Any IDC in which there will be preparation or analytical changes affecting the accuracy of results will need to be redone and these will need to be supplied as well. The IDCs and MDLs need to be established using the proper procedures or they are not applicable.

4. Initial calibration verification (ICV) standards are not analyzed as required in some cases.

- NR 149.44 (6) (i) -

All initial instrument calibrations shall be verified after they are generated with a second source standard - before any quantitative analysis is performed, unless a QCS sample is analyzed three times per year. Some of the data showed the ICV standard is not analyzed before any quantitative analysis is performed.

5. Method blanks are not assessed as required.

- NR 149.48 (3) (d) -

- a. When the method employed does not specify method blank acceptance criteria, the laboratory must refer to NR 149 for the method blank acceptance criteria. NR 149 specifies that samples in a batch be re-analyzed or qualified if the concentration in the associated method blank exceeds the highest of any of the following values:
- The limit of detection
 - Five percent of the regulatory limit
 - Ten percent of the measured concentration in the sample

Currently, the laboratory is assessing method blanks to the reporting limit – not to the limit of detection, as required. An exception to this applies to some methods such as EPA 1631E.

- b. In addition, the acceptance criteria for initial and continuing calibration blanks must be the same as those listed above unless the method specifies otherwise.

At a minimum, the sample results associated with blanks that have detections greater than the limit of detection must be qualified. If the blanks show chronic LOD exceedances then corrective action must be taken.

6. Quality control limits used are incorrect.

- a. EPA 410.4 requires that the matrix spike recovery limits be 90 -110%. - EPA 410.4 (9.4.2) –

- b. EPA 410.4 requires that the matrix spike frequency be 10% of the samples be 90 -110%. - EPA 410.4 (9.4.1) –
- c. SM 5220D requires that the ICV recovery is 95-105% - SM 5220D (4c)-
- d. EPA 350.1 requires that the matrix spike recovery limits be 90 -110%. - EPA 350.1 (9.4.2) –
- e. EPA 1631E requires the LCS limits for 1631E be 77-123% -EPA 1631E Table 2-
- f. EPA 1631E requires the IDC criteria for 1631E be 79-121% -EPA 1631E Table 2-
- g. EPA 1631E requires the blank levels are < 0.5, (the SOP is written as >0.50ng/L).
- h. SM 3500 Cr – B, NR 149 requirements for inorganic ICV and CCV is 90-110% recovery unless other recover limits are specified by the method. The lab uses acceptance criteria of 85-115%. - NR49.44 6ij and 149.44 (7)-
- i. NH3-N analysis by the approved version of EPA 350.1 (1993) for NPDES samples requires that the matrix spike recovery limits be 90 -110%, and the MS frequency to be 1 per 10 samples - 40 CFR Part 136 (May 2012) and EPA 350.1 (9.4.1 & 9.4.2) –
- j. The ICV recovery limits for SW 8260B can be no wider than the CCV limits which are 20%. The SOP included 20% for the CCC compounds, however the other compounds in these check standards must be evaluated. -SW 846 8260B (7.4.5.2) –
- k. In some SOPs there is a requirement to evaluate the MS / MSD recovery, but the limits or a reference to limits are not specified.

Reminder: EPA 365.1 requires that the matrix spike recovery limits be 90 -110%. - EPA 365.1 (9.4.2) –

7. The time requirement for a preparation batch is incorrect.

- NR 149.03 (13) -

The laboratory must prepare the batch samples within a period of 24 hours, from the start of processing the first sample to the start of processing the last sample; an exception is if the laboratory does not analyze more than seven samples for a test per week, then the preparation batch must be processed with one week. The laboratory had understood the preparation batch could exist up to three days except for organic parameters.

Reminder- The LOD/MDL verification check procedure must be evaluated to the requirements in 149.49 (2)(d): Limits of detection shall be determined at least annually unless a laboratory can verify the continued applicability of a previously determined limit of detection by an established and *defensible protocol*. Lab Cert has assessed your protocol for verifying your LOD and has determined it may not be acceptable. The laboratory is encouraged to discuss this further once these procedures are used.

Address each item and update the SOP and QM and provide updated documents.

VI. TEST REPORTS

Deficiencies – Supplemental Information

1. Test reports do not include all of the required elements.

- NR 149.47 -

NR149 lists the minimum elements that must be presented on laboratory test reports. Currently, the laboratory does not include all of those elements on their test reports. Procedures for reporting and qualifying data need to be written. The following elements are missing from the test reports:

- a. The LOD and LOQ values for each parameter must be included in the report. Some results in an example test report were missing.
The majority of WI results need to be reported to the LOD (non-detects are below the LOD as <LOD) (1) (e) (11)
- b. The methods reported must always indicate the exact version that was performed (i.e. SM5220C or EPA 410.4). (1)(e)(5)
- c. When reports are revised they do not always indicate the reason for the revision. (2)(a)
- d. When reports are revised they do not reference the original test report. (2)(a)
- e. The test reports reviewed had some errors that were discussed and reviewed during the closing meeting.

Reminder: When subcontract labs are used the subcontract lab WI certification number must be included in the report.

Reminder: The presence of significant peaks and baseline rises before and after the DRO and GRO retention windows need to be presented on the test reports.

Reminder: Since the data qualifiers are entered by analysts and can be customized the laboratory should make sure these are standardized. In the report example there were qualifiers that indicated system control problems, such as the ICV and CCV failures; the laboratory should have a system in place when these system QC failures are qualified, and if results should be specified as estimated.

Reminder: Tests that require immediate analysis (within 15 minutes of collection) need to be qualified as being performed past hold time unless the analysis was performed after 24 hours of collection. If the analyses are performed past 15 minutes of collection then the sample results need to be qualified as such or the parameter reported as a "lab" value (i.e. "pH, lab" and "TRC, lab"). NR 149.46 (2) (2)

Reminder: The results of oil and grease method blanks must be reported to the LOD with all sample results.

Address each item and update the SOP and QM as required. Provide updated documents that show these are addressed, including the reminder items.

VII. ANALYTICAL TECHNOLOGY

A. Oxygen Demand Assays: BOD, cBOD

Additional training on the requirements of calibration, analysis, reagents and troubleshooting was provided by Rick Mealy during the onsite evaluation.

Deficiencies – Supplemental Information

1. DO meter calibration is not completed and assessed as required for BOD analysis.

-SM5210B 4b4 & SM4500O G and NR 149.39 (3) (c) (11) & NR149.44 3a –

- a. In order to assess DO meter calibration, the mg/L DO saturation value that results from the DO meter automatic calibration function must be documented. The mg/L DO saturation value from the DO meter is then compared to the saturation point value that is based on room temperature and barometric pressure. The saturation point is also used to assess sample DO saturation.
- b. Currently, barometric pressure is not documented. Barometric pressure is a required component used to determine the saturation point. Currently the barometric pressure is obtained from the internet, however the value obtained from the internet is was not adjusted to the laboratory elevation (the internet will report a "corrected to sea level" value). *Due to the elevation of the laboratory of 930 ft., this could result in a positive bias of 0.5 mg/L.* The barometer will need to be checked against an external source at least once annually and documented as such.
- c. The saturation point value, DO meter calibration value, room temperature, and barometric pressure must be measured and documented each time the DO meter is calibrated.
- d. Prior to calibrating, the DO probe needs to be checked for formation of droplets on the probe tip - Pro ODO manual and - SM 4500-O G-
- e. It is not apparent that the analyst allows sufficient time for calibration to be representative of water-saturated air (waits only about 5 minutes before calibrating). This calibration approach (water-saturated air) assumes that the air in contact with the probe is saturated with water vapor which takes significantly longer than 5 minutes.
- f. The probe cap (if this will be continued to be used) must be a proper fit for air calibrations to ensure a closed environment for water saturated air. Using a BOD bottle that has a consistent level of water is a recommended alternative.

2. BOD method blanks are not assessed as required.

- NR 149.48 (3) (c) & (d), SM 5210B (6) (c) -

The laboratory routinely analyzes three BOD blanks. If the average is in control the data is not qualified for a failing BOD blank. If a method blank fails, regardless of how many are analyzed, the data must be qualified for a failing method blank. Method blanks cannot be averaged to determine if they are passing or not. Corrective actions taken to address the cause of the blank result which is outside the method criteria need to be documented.

3. GGA standards analyzed must not be averaged to compensate for instrument problems.

-NR 149.48-

The laboratory routinely analyzes up to 8 GGA standards. If the average is in control, the data may not be qualified for a failing GGA standard. This many GGA standards cannot be averaged to determine if they are passing or not (*the online method does not disallow for up to 3 GGAs to be averaged – if the lab decides to do this*). During the onsite visit, it was discussed that an option would be to analyze one GGA standard per a batch of 20 samples (or at least one per week if the lab analyzes less than 20 samples in a week).

4. GGA standards are not seeded properly.

-SM5210B 6-

The lab inappropriately adds inhibited seed (Polyseed NX) to GGA standards. Polyseed NX contains ATU, and inhibitor used for cBOD analysis. Acceptance criteria for GGA are based on uninhibited GGA.

5. GGA standards are not prepared and stored as required.

-SM5210B 3h-

The lab prepares its own glucose-glutamic acid (GGA) standard, but does not dry the reagents as required before use. In addition the GGA solution is stored in a drawer rather than in refrigerator at ≤ 6 °C.

6. Samples are not treated properly for DO super saturation.

- SM 5210B (4) (b) -

Samples (and QC) must be properly treated for DO saturation. Proper DO saturation is a saturation that is very close to the saturation point. It is easier to remove DO from samples after they are warmed to 22.0 – 23.0 °C. Shaking them vigorously, sometimes multiple times, is often required to remove supersaturated DO from the samples. Currently, the laboratory data sheets included many initial dissolved oxygen values that are greater than 9. This signifies either calibration or super-saturation problem.

7. Extra nutrients are not added to samples as required.

- SM 5210B 4 -

When a sample size of more than 200 mL is used, extra nutrients must be added to the BOD dilution bottle, and this must be documented.

8. Preliminary dilutions are not performed.

-SM 5210B (4) (f) (2)-

For dilutions greater than 1:100 (less than 3ml sample volume) a preliminary dilution is required.

Reminder -Residual Cl strips are available at lower levels than 0.5 mg/L and should be used.

Reminder - results for toxic samples/sliding BODs need to be evaluated, there seemed to be instances where the higher dilution sample had the highest result and was significantly different than the lower dilutions.

Reminder – update the SOP to include the type(s) and amount(s) of inhibitor reagents used; and how under depleted samples are calculated.

Reminder--Duplicate(s) need to be treated in the same manner as the original sample(s).

Address each item and update the SOP. Provide the updated SOP that shows these are addressed, including the reminder items.

B. Colorimetric Technology: Hardness, NH₃-N, SiO₂ - Dissolved, TP, COD, Cr⁺⁶

Deficiencies – Supplemental Information

1. The spectrophotometer is not zeroed as required for COD.

- NR149.48 (1) (c) -

Zeroing the spectrophotometer using the method blank, in essence, is equivalent to subtracting the method blank result from all subsequent measurements – which NR 149 does not allow.

Currently the laboratory is zeroing on the highest concentration standard for this analysis. EPA method 410.4 does not call for zeroing with a high standard. Furthermore, the method SM 5220D specifically

calls for zeroing on an undigested method blank for the mid-high level 620 nm (Cr+3) analysis and reagent water for the low level 420 nm (Cr+6). Instruments should never be zeroed on a standard unless required by an authoritative source. -SM 5220D (4) (c) -

Reminder: For Hexavalent chromium analyses by SM3500 Cr – B, a method blank that is a different sample from the blank used to zero the spectrometer is required.

2. The criteria of samples distilled for ammonia is not met.

- EPA 350.1 (11.3) and SM 4500-NH₃ (a) (1) (4b) –

- a. During the ammonia distillation procedure the sample is adjusted to a pH of “greater than” 9.5; however the method requires that the sample be adjusted to a pH of 9.5 exactly, in addition the pH needs to be documented.
- b. I have included this requirement, since this was discussed during the opening meeting and clarity was requested: Samples must be distilled unless the laboratory performs equivalency studies to demonstrate, for each client and sample type, that distillation is not needed. Unless the laboratory has data to prove that distillation is not necessary - all samples must be distilled. The equivalency study must consist of a minimum of 4 known spikes and 4 sample replicates all performed with distillation and without so that the results can be compared (total of 16 analyses).
- c. The use of boric acid is not applicable to the SM 4500 NH₃-N online method.-SM4500 NH₃ B(1) 1997-

3. The presence of residual chlorine is not checked as required.

- SM 4500 NH₃ B (4b) –

The ammonia method employed requires that samples be assessed for residual chlorine. The use of low level chlorine test strips or DPD coloring reagent are acceptable. If residual chlorine is present, the addition of sodium sulfite is used as the dechlorinating agent.

4. The laboratory has not shown that the water used is ammonia free.

- EPA 350.1 (7.1) & SM 4500 NH₃ B (3a) –

Both the EPA and the SM 4500 method require the use of ammonia free water. The lab reagent water (using purchased distilled water) results have high background optical density results. The source water used by the laboratory for calibration and reagent preparation needs to be free of this analyte on a consistent basis.

In addition the laboratory is currently using different distilled reagent water samples for mixed and multiple purposes (i.e. diluent, rinse and method blanks).

When the laboratory distills water to be used as the diluent, and more than one blank is absolutely needed for enough volume, combining the multiple distilled blanks to create enough blank water allows for equal treatment of samples that need to be diluted.

Each method blank is to be prepared as a single distilled sample tied to the sample batch QC. If multiple blanks are used or combined then it becomes difficult to evaluate if and where contamination above the MDL has occurred.

5. Initial calibrations are not performed as required for TP.

- NR 149.44 (6) (c) – and – EPA 365.1 (10.2) (11)-

All initial calibrations shall be established using standards that are appropriate for the calibration model selected. If procedural standards are utilized then they must be treated the same as samples. Each standard and sample processed through a method procedure is subjected to a bias. As a result, the individual bias of each standard and sample becomes an inherently important component of the calculations that go into generating the results. The protocol of diluting just one standard (subjected to just one bias) after being processed through the method procedure is not an appropriate method for establishing an initial calibration as each of the standards used to establish the initial calibration do not include their own individual bias. Currently, for some general chemistry tests one high concentration standard is processed through the method procedure and then dilutions are made from this one standard to establish an initial calibration curve.

Reminder – The data indicates there is contamination when there is significantly higher background absorbance (optical density) in the zeroing blank than the calibration blank and some of the lower standards. This was observed for the parameters analyzed by the WestCo instrument, the issue needs to be investigated, and the problem resolved.

Reminder – Colorimetric analysis of Silica is applicable to Silica-Dissolved and must be reported as such. -40 CFR part 136, Table IB and SM4500-SiO₂ F 1 –

Address each item and update the SOP(s). Provide the updated SOP(s) that shows these are addressed, including the reminder items.

C. Electrometric Assays: pH

The procedure used for analysis of pH of waters and solids was evaluated using the laboratory data and the pH SOP.

Deficiencies – Supplemental Information

1. **pH analysis of solid and waste samples are not performed as required.**
- SM 4500 – H (4)-
 - a. pH accuracy of the buffers is not evaluated with the required criteria. If the meter response shows a difference greater than 0.1 pH unit from expected value, look for trouble with the electrodes or potentiometer. The criterion of 0.5 pH units on the data review checklist is too wide for some levels. Also the SOP states a different level of 0.5%.
 - b. All pH measurements must be performed with the electrolyte fill plug open to allow for adequate electrolyte flow during measurements, this is not included in the SOP.
 - c. The hold time in the SOP is 28 days, the hold time on the checklist is 24 hours, but the actual hold time for compliance samples is 15 minutes. Additional information for reporting this is in the test report section.

Address each item and update the SOP. Provide the updated SOP that shows these are addressed.

D. Gravimetric Assays – Residue: TDS, TSS, TS, TVS

The procedure used for analysis of solids of waters and solids was evaluated using the laboratory data and the applicable SOPs.

Deficiencies – Supplemental Information

1. The volume of sample required for compliance sample reporting limits is not met.

- Reporting of BOD and TSS Data memo WDNR October 2003 -

Currently the laboratory uses smaller volumes than necessary to meet adequate sensitivity required for reporting limits; the reporting limits established by the laboratory seem to be based more on the accuracy of the balance however, the function of the volume used and the sensitivity of the method are required. This will also help reduce exceedance issues seen with blanks.

To report a value of <2 mg/L for TSS, a volume of at least 500ml must be filtered.

The correct formula to use for determining the TSS (mg/L) reporting limit is (1.0×1000) divided by the mL of sample used.

TDS and TS (mg/L) reporting limits are also a function of the volume of sample used.

The correct formula for determining the TDS and TS (mg/L) reporting limits is (2.5×1000) divided by the mL of sample used.

The current laboratory procedure of using 100 ml for TSS and 50 ml for TDS result would not meet the laboratories stated reporting limits.

Reminder – the blank volume filtered needs to be the maximum volume needed to evaluate them to the reporting level.

Address each item and update the SOP(s). Provide the updated SOP(s) that shows these are addressed, including the reminder items.

E. Gravimetric Assays – Oil & Grease, Hexane Extractable Materials (HEM)

HEM and HEM-SG are completed using SPE equipment.

Deficiencies – Supplemental Information

1. Samples are not dried to a constant weight; the second weight is not completed or documented.

- EPA 1664A (11.4.4) –

After the sample is evaporated and cooled to room temperature in a desiccator for 30 minutes, it is weighed for the initial dry weight. Then it is required to repeat the cycle of drying, cooling, desiccating, and weighing until the weight loss is less than 4 % of the previous weight or less than 0.5 mg, whichever is less.

Address each item and update the SOP. Provide the updated SOP that shows these are addressed.

F. Ion Chromatography Technology: Br, Cl, F, NO₃, NO₂, PO₄, SO₄

Deficiencies – Supplemental Information

1. The IC calibration is forced through the origin.

- NR 149.44 (6) (f) (4)-

The laboratory may not force calibration functions through zero. Results at the low end of the curve are affected when compared to a simple linear regression. MDLs and IDCs need to be reevaluated. Include or reference information concerning software settings that could affect proper selection of calibration functions.

2. Samples analyzed for IC are not diluted as required.

- NR 149.44 (6) (L) -

Valid dilution measurements are those where the response is within the measured responses of the calibration curve standards. If the response is above the response of the highest standard in the calibration curve then the sample must be diluted and reanalyzed. Although establishing a linear range is in the reference method, quantitation must be within the high calibration response.

Reminder- It was discussed with the laboratory quality manager that analysis of NO₃ + NO₂ requires certification if analyzed for compliance samples.

Reminder- The procedure has been recently developed, if there are changes, such as column sizes, that affect the sensitivity, the MDLs must be reanalyzed.

Address each item and update the SOP. Provide the updated SOP that shows these are addressed, including the reminder items.

G. ICP/MS Technology: Al, Sb, As, Ba, Be, Bi, B, Cd, Cr, Co, Cu, Au, Ir, Fe, Pb, Mn, Mo, Ni, Os, Pd, Pt, Rh, Ru, Se, Ag, Sr, Tl, Ti, W, V, Zn, Zr

Lithium and mercury were dropped from consideration due the additional requirements needed to analyze these parameters and the potential lack of request for analysis.

Analysis is completed using 6020A, and it was discussed with the quality manager that the approved EPA method must replace the SW846 method in the future for NPDES / compliance samples.

Samples are prepared by method 3015A, and there are plans to add preparation by 3005 and 3020.

Deficiencies – Supplemental Information

1. Analytical instruments need to be operated by personnel trained in their use.

- NR 149.44 (4) (a) and 6020A-

Additional and potentially formal training is needed for additional knowledge of the principles of ICPMS analysis along with increased experience in the use of the instrument software.

2. Interference effects have not been evaluated properly.

-6020A 2.2 and 7.7-

Currently, the laboratory has correction factors in the SOP; however these calculations need to be confirmed they are applied by the software. Since the instrument is new the analyst needs to work with the vendor to learn the software.

3. The interference check solutions are not evaluated properly.

- 6020A 7, 7.7, 9.7 -

The laboratory analyzes both an ICSA and ICSAB solution. The ICSA is not evaluated to verify the absence of effects or the interference correction for the non-spiked parameters analyzed. The results from the ICSA solution should be less than absolute value of the limit of quantitation for all applicable parameters. The current report format needs to be updated since it does not print the results of the non-spiked parameters.

The ICSAB interference check standards should be prepared at the anticipated maximum concentrations expected to be measured in the samples, and all reported parameters included in the report for evaluation.

4. The development of the ICPMS method requires additional documentation in the SOP for the following:

-6020A-

- Selecting isotopes that are not listed in the method as recommended (for example Mo)
- What multiple isotopes are used and which are reportable
- What is the criteria for selecting which ISs are for specific parameters
- What is included in the pulse analog solution and what is measured in the performance check
- Information for the oxides and double charged ions pertaining to how they are measured and the criteria
- That the Pb result is a summation of the signals of 206, 207 and 208 m/z

5. ICPMS linear dynamic range (LDR) has not been established as required.

- SW846 6020A (10.6) -

At the time of the on-site visit, there was not valid analysis of the LDR.

The SOP discusses an alternative option (see 12.6.2), which still must be within the LDR therefore does not exclude evaluating the LDR.

In additional, the quantitation of results must be limited to 90% of the established LDR, and the SOP needs to be updated.

6. Dilutions need to be taken into account in final calculations.

- SW846 3015a 12.1-

The dilution factors for the digestion dilution of 45 to 50ml have not been used to calculate the results, including the MDL or IDC results.

7. The pH of samples preserved for metals is not verified before digestion or analysis.

- NR 219.04 Table F (Table B) -

Subsamples taken for metals analysis are taken from samples that have not been shown to be preserved for at least 24 hours at a pH < 2. This requirement applies to aqueous samples that will be tested for metals by ICPMS technologies.

8. The relative standard deviation (RSD) is not calculated or evaluated correctly.

The RSD calculation for the 3 replicates pertaining to ICPMS is incorrect. For instance, in the excel table with the three replicates that was supplied, the Be in no-gas mode for the 1ppb standard says the RSD is 4.2, which seems to represent the RSD for the replicates. However, on the report template print out, the %RSD says 0.06 for the same standard on the 6/6/13 data set. When additional information was requested, the vendor told the analyst that it was due to the calculation using 2 significant figures, however this does not appear to be the reason, and the calculation for RSD must be verified to be correct. Typically, RSD results are not 0.00 or 0.01.

Reminder – the policy for reprocessing data for IS failures in the ICPMS SOP in 10.6.1.12 needs to include sufficient detail if used.

Reminder –In Method 6020A the LLCCV concentration is at the lowest calibration standard for multi-point curves, or the quantitation/reporting level (two point curves). -6020A 10.4.3-

Reminder – The microwave digestion procedure recommends dilution of the highly acidic samples, this will also help with getting a closer matrix match to the 2% acid concentration in the ICPMS calibration standards.

Address each item and update the SOP. Provide the updated SOP that shows these are addressed, including the reminder items.

H. Ultra-Low Level Metals Assays: Hg

Deficiencies – Supplemental Information

1. System blanks are used inappropriately.

- EPA 1631E (10.3), (10.3.2.6) -

EPA method 1631E requires that the mean result of three system blank be <0.5 ng/L with a SD < 0.1 ng/L. The average result of the three system blanks is then subtracted from all subsequent measurement raw data before calculations are performed. In one data set the system blank did not meet the SD criteria but was still used.

2. The carryover check had not been completed.

- EPA 1631E (4.3.8.1)-

The carryover check needs to be documented to know the concentration level per 1631E that results in levels greater than or equal to 0.5 ng/L.

3. The SOP lacks sufficient detail addressing how and when sample dilutions are required.

-NR 149.40 (2) (d) (12) –

It is additionally important in this method since samples in which certain reagents have been added are not to be diluted and reanalyzed.

4. The bottle blanks require a minimum oxidation time of 24 hours.

– EPA 1631E 17.3-

The laboratory SOP and practice only used an oxidation time requirement of 12 hours.

5. The MSD RSD requires additional corrective action steps.

– EPA 1631E 9.3.6-

The MSD RSD limit exceedance corrective action steps requires additional detail, since in this method this is used to show the system is in control, and it is not acceptable to not resolve failures.

Reminder - Field blanks are analyzed throughout the run versus prior to the samples, due to the stringent criteria for these samples this is required. – EPA 1631E 9.2.2.1-

Reminder: Quality Control Standards (QCS) for LL Hg must be analyzed.

Laboratories that perform ultra-low level analysis shall analyze QCS samples 3 times per year. These are QC samples that can be ordered from vendors that supply performance evaluation samples. - NR 149.22 (3)-

Reminder: The SOP discusses that the calibration standards should be prepared fresh daily from the working standard. During the onsite visit, the policy was stated to use them unless the response changed, and did not mention any expiration date. There are concerns that due to the newness of the instrument (stability/response may not be sufficiently known yet), and the likelihood that low level standards may not be stable, that calibrations would be used which are not accurate; therefore it is likely necessary to always make these each time to ensure the concentrations remain accurate.

Reminder: The filter equipment must meet the method limit requirements prior to use.
- EPA 1631E (9.4.6.2)-

Reminder: The laboratory needs to choose which MDL result will be used.

Reminder: The laboratory discussed an option of utilizing another vendor to prepare the bottle kits using the laboratory's water for trace level Hg field blanks. There could be consequences for contamination of the blank water used (which does not account for these 'trips' and the vendor environment). The SOP would require revisions for the procedures to be used; in addition, tracking of these bottles, and dates would need to be developed.

Reminder: The laboratory may want to consider using a Class 100 bench top clean room hood for the preparation of reagents, standards, and samples, if required to eliminate potential contamination from the lab environment.

Address each item and update the SOP. Provide the updated SOP that shows these are addressed, including the reminder items.

I. GC Technology: GRO/PVOC

Deficiencies – Supplemental Information

1. Chromatograms do not include sufficient information to determine whether significant peaks outside the GRO window are present.

-WI GRO 9.7.3-

Quantitation reports indicate that the analytical run time is not extended for 5 minutes beyond the elution of Naphthalene, as required by method. Quantitation reports show a chromatogram run time of about 19-19.5 minutes and Naphthalene elutes at approximately 18 minutes. It appears that this is a truncation issue on the printout, as the computer screen clearly shows that the run is properly extended beyond the elution of Naphthalene. Although these are being quantitated correctly, the issue needs to be fixed since analysts typically review data from printouts which could easily lead to a situation in which samples are incorrectly reported as having no peaks beyond the GRO window.

2. The laboratory needs to be able to clearly resolve ethyl benzene from m&p-xylene.

-WI GRO 6.1.2.2-

One option is to evaluate a low and high standard against resolution criteria available in many organic methods. In several methods (such as 8260/70) the criteria is: Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights.

Reminder -Lab routinely has to run instrument blanks due to carryover of Naphthalene. More appropriate corrective action would be to increase the bake time/temperature.

Address each item and update the SOP. Provide the updated SOP that shows these are addressed, including the reminder items.

J. GC/MS Technology: VOC

Deficiencies – Supplemental Information

1. Qualitative identification of target compounds is not performed correctly for all analytes.

-SW-846 8260B 7.6.1-

The instrument is programmed with at least one incorrect quantitation ion that was identified on-site (Trichlorofluoromethane is set up as if it includes m/z 67 as a qualifier ion, but that ion is not present in the mass spectrum.) A review should be performed on all method analytes to verify that the correct quantitation/qualification ions are included in the method.

2. Qualitative identification and accurate quantitation of 1,1,1-Trichloroethane is hampered by coelution with an internal standard which shares common ions.

-SW-846 8260B 7.6.1.5-

Target analyte 1,1,1-Trichloroethane is subject to bias due to coelution with and a shared quantitation ion with Pentafluorobenzene internal standard.

3. The instrument operating conditions have not been optimized to ensure adequate separation and resolution of target compounds.

-SW-846 8260B 2.1-

The analytical run time is significantly compressed which results in problems like coelution and shared ions of 1,1,1-Trichloroethane with the Pentafluorobenzene internal standard. The laboratory use of a 13 minute run time for a full suite of VOCs is significantly different than the reference method run time of approximately 40 minutes. While conditions may be modified, such modifications should not compromise the ability to identify or quantify target analytes.

4. It is not apparent that CCC compounds are evaluated against the correct criteria.

-SW-846 8260B 7.3.6 and 7.3.5-

Instrument printouts of the "Response Factor Report" for calibrations incorrectly flag CCC compounds as being out of range when they actually pass. The ability to access the settings for these compounds in the software is important, for verification and correction; this also applies to the SPCC criteria.

5. The IS recovery for samples is not evaluated.

-SW-846 8000 C (11.4.3)-

The laboratory includes criteria for IS for verification standards, but does not address the criteria for samples. Since this is a diagnostic check for samples (as well as standards), and low internal standards suggests incorrect injection, and high IS indicate other issues, the laboratory needs to assure these are evaluated. Using the established standard criteria (50-200%) for samples is an option that allows for reasonable protection against major errors.

6. The laboratory has developed its own reference mass spectrum library of compounds for VOC analysis. There is not documentation of when or how it was done.

- SW846 8260B-

The analyst was unable at the time of the visit to recall the specifics for the reference library. Additional documentation of the details of how and when this was developed need to be completed.

7. The laboratory must specify a policy for use of alternate ions for quantitation.

- SW846 8260B 7.5.11-

The analyst was unable to recall the reasoning behind the use of alternate ions for quantitation and many compounds had been set up as such.

8. The laboratory policy for manual integration does not apply to samples.

- NR 149.48(1) and 149.39 (3) -

The laboratory has a policy to review these only as they apply to standards. However, incorrect automatic integrations are more likely with real world samples; therefore addressing correctness of sample integration needs to be done.

Reminder: The laboratory should document when and if there is a reason that alternate surrogates or internal standards are chosen. For instance, the laboratory is using dibromofluoromethane as the surrogate standard instead of the recommended d4-1,2 dichloroethane.

Address each item and update the SOP(s). Provide the updated SOP(s) that shows these are addressed, including the reminder items.

K. Waste Characterization Extractions : SPLP, TCLP

Deficiencies – Supplemental Information

1. The SPLP leaching fluid used for wastewater and wastes is not always the correct one as stated in the SOP.

- EPA 1312 (5.4.1) -

Extraction fluid #1 is always to be used for non-volatile wastewaters and wastes regardless of which side of the Mississippi they were collected from.

Reminder – the % TS of soil samples are typically completed by a method such as SM 2540G; when samples have mixed matrices, or contain significant amounts of liquid, additional steps are required. This needs to be clearly written in the SOP.

Reminder – the pressurizing tool that is calibrated is due for another calibration in September 2013.

VIII. SUPPORTING DATA

For each of the deficiencies listed in this report, the laboratory must provide data that demonstrates that the corrective taken has resolved the deficiency. At a minimum, the following supporting data is to be sent with the audit response for the auditor to review.

1. If the deficiency requires a change in procedure that impacts the Quality Manual then a copy of the section in the Quality Manual that was updated must be sent.
2. If the deficiency requires a change in procedure that impacts the way that the laboratory performs their method procedure then a copy of the section in the Method SOP that was updated must be sent.
3. If the deficiency requires that missing documentation be recorded then a copy of a benchsheet, analytical run, or other record needs to be sent. This data is provided to demonstrate that the appropriate records are now kept.

When compiling the audit response make sure to include all of the supporting data that will demonstrate that the corrective actions taken have resolved the cited deficiencies.

One way to help speed up the review of your audit response and the closure of your evaluation is to organize the submittal as follows

- Indicate the corrective action that the laboratory has taken to resolve each deficiency listed in this report
- Provide that information in the order that the deficiencies are listed in this report
- Make sure that the supporting data that demonstrates the corrective action has been implemented is included for each deficiency
- Label the supporting data with the section and number of the deficiency