

Lab: 11642 USGS NATIONAL WATER QUALITY LABORATORY
Address: BLDG 95-DENVER FEDERAL CENTER
DENVER, CO 80225-0585

Assessment ID: 3242
Assessment Date: 08/08/13
Assessment Type: General Assessment
Primary Assessor: Zgrodnik, Tom

SUGGESTION(S)

Deficiency: SGST The following is an observation on areas for possible improvement and is not considered a deficiency:

Assessor: DeNicola, Kathie

Comments: The long term method detection limit (LTMDL) studies have been discontinued by the USGS's QA group. A procedure should be developed that documents the annual limit of detection (LOD) and / or limit of quantitation (LOQ) determinations for the methods.

Corrective Action:

To replace the LT-MDL program, the NWQL initially evaluated 2 different programs — the ASTM program (DQCALC)[1] and EPA LC-MRL program [2]. An initial test was done to compare the results of both programs with the LT-MDLs that had been previously established, in addition to evaluating the programs on data input ease, general ease of use, and information provided as output.

Once we were able to identify the appropriate input for both programs, side by side testing was done on a variety of analytes, including both organic and inorganic methods. Note that the DQCALC program uses the entire calibration curve data and requires seven different data sets for each point along the curve. The LC-MRL program uses the low standard from the calibration curves, but still requires seven different data points.

The initial data pulls started with methods/analytes that have had an LT-MDL in the past, with a priority work based on those analytes for which we hold an accreditation. The priority of methods initially was identified as: volatiles; nutrients; metals; GC methods; pesticides, new pesticide method, and others to be determined. The key was identifying if calibration data exists in LIMS for all methods.

Both programs output an MDL based on the same calculation that is contained in 40 CFR Section 136 Part B, the EPA/industry standard. The DQCALC program was identified to have the added advantage of providing precision and bias information for all the tested analytes for the entire length of the curve, i.e., concentration-specific data. The NWQL thought this added advantage makes the DQCALC program more worthwhile to pursue, and has begun identifying detection limits (LOD) based on the program. The section supervisors are given the opportunity to review the data and can identify an LOQ anywhere in the range of 2–5 times the LOD. For consistency, QAS suggests the LOQ as a multiple of 2.

The program is currently in use and further refinements of the procedure are expected to take place as the NWQL implements the LOD and LOQ verification procedures.

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Deficiency: SGST continued

1. ASTM D7510 – 10 Standard Practice for Performing Detection and Quantitation Estimation and Data Assessment Utilizing DQCALC Software, based on ASTM Practices D6091 and D6512 of Committee D19 on Water (<http://www.astm.org/Standards/D7510.htm>)
2. Statistical Protocol for the Determination of the Single-Laboratory Lowest Concentration Minimum Reporting Level (LCMRL) and Validation of Laboratory Performance at or Below the Minimum Reporting Level (MRL) (http://www.epa.gov/safewater/methods/pdfs/methods/methods_lcmrl.pdf)

No corrective action required

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INTRODUCTION

Deficiency: 504 The laboratory does not make accurate statements concerning their NELAP accreditation fields of testing and NELAP accreditation status. (Sec. 4.6.1 & 6.8.a.2) [M2,5.10.11(a)]

Assessor: Zgrodnik, Tom

Comments: The laboratory is reporting results for analytes that ELAP offers certification for. If a given analyte is offered by NYS ELAP for accreditation, then accreditation must be gained by submitting an application form to ELAP. For those analytes not offered by NYS ELAP, then a qualifier must appear on the report, indicating those specific analytes are not accredited by NYS ELAP.

Corrective Action:

The U.S. Geological Survey (USGS) gathers data for determining the location, amount, availability, and quality of groundwater and surface water throughout the United States and its territories. The NWQL determines organic and inorganic constituents in samples of groundwater and surface water, river and lake sediment, aquatic plant and animal material, and precipitation.

The USGS NWQL presently has 483 analytical test codes for about 2600 compounds. As a science agency, samples are obtained by USGS satellite science centers and the subsequent data are reported back to them for use in tracking and trending purposes. Because of the nature of its work, our USGS methods are developed to get the lowest reporting level possible. These levels are much lower than the required drinking levels of most of the methods listed in 40 CFR 136.

The laboratory does report results for analytes for which ELAP is certified for; however, the ELAP certification does not cover all methods that the NWQL utilizes. In August 2012, the NWQL submitted an application to ELAP identifying additional analytes that needed to be added to its accreditation, with supporting data; and some of those analytes were rejected (see email attached). The basis for the rejection seems to have been the fact that the method used by the NWQL differs from the methods listed in the Federal Register and NEMI. If approval is provided to apply for analytes that are on the ELAP list, but not listed in the CFR, we would provide method analysis and analyst demonstration of capability. In addition, the NWQL is working with the EPA and NEMI to get additional USGS/NWQL methods listed. In its customer catalog, the NWQL has identified to its customers which analytes are NELAP certified.

No corrective action required

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CORRECTIVE ACTION

Deficiency: 54103 The laboratory does not identify potential corrective actions and select and implement the action(s) most likely to eliminate the problem and to prevent recurrence. (Sec. 5.4.10.3)[M2,4.11.3]

Assessor: Zgrodnik, Tom

Comments: The corrective action report for the PT#345 failure of color was examined. The root cause was identified as the use of non-Class A glassware. The minor differences between the use of Class A glassware and non-class A glassware would not yield the significant difference in the result reported (35.0) and the target value (63.6). In addition, the TOC failure for PT #345 was also examined. There was no root cause determined for the failure, but a later run yielded satisfactory results. Root cause investigations must document all the factors examined and explain the logic for the failure.

Corrective Action:

Color: The root cause of this finding is that the subject failures were not fully investigated, documented, or explained at the time the failures were initially identified. Chapter 6, Corrective Actions, of NWQL's Quality Assurance and Quality Control Manual outlines measures that should have been taken. Failure in this regard will be specifically discussed during NWQL's Ethics, Data Integrity, and Scientific Integrity training. During recent investigations it appears that the Color SOP was not being strictly followed in establishing the sample dilution required. The dilution choice was misjudged.

TOC: The sample was analyzed initially without dilution on July 22, 2011. Using a curve prepared on July 8, 2011, the result (68.3 mg/L) exceeded the high calibration standard. Reanalyzed using a 2x dilution, a concentration of 70.5 mg/L (35.2595 x 2) was determined and reported for PT study 345. All three replicates (both diluted and undiluted) were within 1 RSD. All QC was acceptable: blank, 0.18 (-0.6-0.3); TPC 4.81 (4.10-5.74); CRM 4.36 (1.67-5.89); CCV 10 std, 9.96 (8.83-11.03), LOQ, 0.54 (0.396-0.865); and 5 std, 4.88 (4.24-5.89).

The analyst prepared the sample again and reanalyzed it on October 25, 2011. The result for the undiluted sample was 57.4 mg/L and the 2x dilution was 59.8 mg/L (29.9 x 2). All QC was again acceptable.

The rerun, however, was performed when the sample had already exceeded the NWQL's acceptable holding time for TOC. Because TOC has been shown to decrease with time, the concentration in the rerun would be expected to be lower than the original result. Furthermore, the rerun was analyzed using a new calibration curve, which was necessary due to a new catalyst and the instrument's being down during relocation of the carbon laboratory. The subsequent review of the laboratory procedures did not identify any problems with the sample's preparation or analysis.

Although the rerun was past its holding time, it was analyzed on October 25, 2011, using a curve from October 24, 2011. Both calibration curves consisted of standards that were (all) analyzed on the same day. The QC associated all analyses was acceptable and the calculations verified. All calibration standards, CCV standards, and CRM QC were analyzed within the approved expiration times.

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Deficiency 54103 continued

The same stock standard was used to prepare both calibration curves. The CCVs were prepared less than 3 months prior to the analyses. The catalyst was within 2 months of its expiration when the NY PT was analyzed originally and had just been replaced when the NY PT was reanalyzed.

Failure is most likely attributable to the catalyst's starting to fail at the high end.

No corrective action required.

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DATA INTEGRITY PROCEDURES

Deficiency: 54151 Reviews are not conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity..(Sec. 5.4.15)[M2,4.16]

Assessor: DeNicola, Kathie

Comments: This refers to review of TOC raw data. Calibration curve of 10/12/12 showed that standards were not analyzed on the same day. Some were replaced by previously run standards in order for the correlation to pass. Even if the RSD passed between triplicate injections, the reported results were picked based on assumptions and not according to the SOP. Also, the raw data for the calibration curve of 10/12/12 was not in the calibration log book where it is normally kept.

Corrective Action:

The analyst has been retrained and understands that the calibration curve data must be analyzed on the same date of analysis. The NWQL SOP ORGW0483.1-1, Total organic carbon (TOC) analysis will revise section 7.3.2.6 to state that the calibration data must be from the same calibration curve.

Additionally, the analyst will create a new book that contains the calibration curves instead of placing the curves with the analytical run data. The book will start with June 2013 curves. The missing calibration curve from September 12, 2012 (10/12/12) has been located and placed in the book.

Due date – December 12, 2013

Completed: November 26, 2013; Respective SOP pages included to demonstrate procedure change for TOC analytical line. Additionally, copy of the new calibration curve book cover and the calibration curve from September 12, 2012 (10/12/12).

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ENVIRONMENTAL TEST METHODS and METHOD VALIDATION

Deficiency: 55462 The laboratory does not have a procedure to estimate the uncertainty of measurement. (Sec. 5.5.4.6.1)

Assessor: Zgrodnik, Tom

Comments: For example, use historical LCS recoveries, calculating the standard deviation of those recoveries, and expressing a 95% confidence limit using 2-sigma limits. Avoid using 3-sigma limits and expressing the 99% confidence limits.

Corrective Action:

The laboratory is in the process of recalculating uncertainty based on 2 SD and the 95% confidence level value. In addition, use of the DQCALC program to establish method detection and reporting limits also provides uncertainty information throughout the span of the calibration curve, and will help in establishing a procedure for the evaluation of measurement uncertainty.

No corrective action required

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EQUIPMENT AND REFERENCE MATERIALS

Deficiency: 5911 All support equipment is not calibrated annually, using NIST traceable references when available, over the entire range in which the equipment is used. (Sec. 5.5.5.2.1.b)[M2,5.5.13.1(b)]

Method: USGS I-3561-85

Assessor: DeNicola, Kathie

Comments: There is no record of wavelength accuracy check for the HACH spectrophotometer used for COD analysis.

Corrective Action:

The wavelength accuracy check was performed on September 26, 2013 by the analyst and recorded in the COD logbook. NWQL SOP INCW0119.6, Chemical oxygen demand by HACH DR/4000 colorimeter, will be amended to include a wavelength accuracy check with each analytical run. In addition, the instrument will be serviced annually to verify instrument stability and accuracy. A copy of this service will be maintained in the instrument logbook.

Please note, the lab has a very low analysis demand for this procedure. The NWQL is in the process of determining whether it will continue this analysis. If the lab does not continue performing the analysis, it will submit form DOH-109 to delete it from its accreditation prior to the completion of the corrective action stated.

Due date – December 12, 2013

Completed: December 6, 2013; Respective SOP pages included to demonstrate procedure change for Chemical Oxygen Demand by HACH DR/4000 colorimeter. Additionally, copy of the COD logbook cover, page 5 of the logbook showing the wavelength accuracy check, and the Certificate of Instrument Performance documenting the instrument service.

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QUALITY CONTROL MEASURES

Deficiency: 000D13 All essential quality control measures are not incorporated into the laboratory method manual.
(D) [M2,5.9.3(c)]

Assessor: DeNicola, Kathie

Comments: 1. In general, there is no concept of batch QC (Method Blank and Lab Control Sample) with acceptance criteria and corrective actions that meet the ELAP Certification requirements in the SOPs. See example from the MBAS SOP, "9.2.8 If spike, CCV, blank, TPC, or standards do not meet criteria, rerun that QC. If it fails again, prepare new working stock and rerun."
2. Some SOPs do not include acceptance criteria for LOQ verification standards. See COD SOP for example. Annual processed LOQ samples are required annually.

Corrective Action:

1. The NWQL Laboratory Quality Manual identifies batch QC and contains the following information:

Section 12.1 Internal QC Samples – Internal QC samples are samples prepared and implemented by the laboratory as part of the analytical process. Thus, QC samples are a part of each sample batch. At the NWQL a batch is defined as the number of environmental samples that can be prepared at one time. For methods that do not have a separate preparation step, a batch is defined as those samples that are analyzed together with the same method and personnel, using the same lots of reagents, and not to exceed the analysis of 20 environmental samples.

Section 12.1.1 contains the following wording – The method blank shall be analyzed at a minimum of one per preparation set or analytical batch of samples. The method blank shall consist of a matrix that is similar to the associated samples and known to be free of the analytes of interest.

Section 13.1.4-2 contains the following wording – The CCV standard must be:

- a. analyzed at the beginning of each analytical run unless a new calibration curve has been produced prior to sample's being analyzed in the same analytical batch;
- b. used to bracket each group of no more than 20 samples.

Section 13.1.4-5 states – Samples associated with a failed CCV standard shall be reanalyzed, unless the following conditions occur: [conditions not listed in this response].

NWQL SOP ORGW0060.3-1, Determination of methylene blue active substances, will be modified to reflect the batch acceptance criteria in the NWQL Laboratory Quality Manual and to verify that the samples associated with the batch QC will be rerun when appropriate.

2. The NWQL SOP INCW0119.6, Chemical oxygen demand by HACH DR/4000 colorimeter, will also be modified to show the requirement to analyze a standard at the LOQ and to have acceptability criteria for that standard based on 2-sigma limits and a 95% confidence level. The SOP wording will be modified to show that the LOQ is verified annually.

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Deficiency: 000D13 continued

Please note, the lab has a very low analysis demand for this procedure. The NWQL is in the process of determining whether it will continue this analysis. If the lab does not continue performing the analysis, it will submit form DOH-109 to delete it from its accreditation prior to the completion of the corrective action stated.

Due date – December 12, 2013

Completed: November 21, 2013; Respective SOP pages included to demonstrate procedure change for Determination of Methylene Blue Active Substances and Chemical Oxygen Demand by HACH DR/4000 colorimeter.

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LIMIT OF DETECTION (LOD) & LIMIT OF QUANTITATION (LOQ)

Deficiency: 00C119 The validity of the LOQ is not confirmed by successful analysis of a QC sample containing the analytes of concern in each approved matrix 1-2 times the claimed LOQ.
(C.3.2.c)[M4,1.5.2.2(c)]

Method: USGS I-4471-97

Assessor: Zgrodnik, Tom

Comments: An element analyzed by ICP-OES was selected at random for review of this requirement. For example, LTMDL established for Na was at 0.06 mg/L, and a reporting limit of 0.11 mg/L was established. However, the lowest standard on the calibration curve is 40 mg/L.

Corrective Action:

To avoid confusion the concentration units on the LT-MDL bottle labeling will be changed to coincide with reporting units. The ICP-OES instrument utilizes a single-point calibration, which is an accepted practice for ICP-OES instruments. The NWQL does analyze an LT-MDL solution with a concentration for sodium at 0.05 mg/L with each analytical run. NWQL SOP INCM0386.5, Trace metals and potassium determination by inductively coupled plasma-optical emission spectrometry will be modified to ensure that a standard at the LOQ (no more than 2x the LOQ) will be analyzed during calibration runs. Acceptability ranges for this standard will be identified and verified.

Due date – December 12, 2013

Completed: December 2, 2013; Respective SOP pages included to demonstrate procedure change for Trace metals and potassium determination by inductively coupled plasma-optical emission spectrometry.

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CHEMICAL TESTING DETAILED METHOD REVIEW (D.1)[M4]

Deficiency: 00D111A Procedures are not in place to determine if a method blank is contaminated. (D.1.1.1.a)
[M4,1.7.3.1(a)]

Method: USGS I-3561-85

Assessor: DeNicola, Kathie

Comments: The middle concentration of the COD method blanks is used to zero the spectrophotometer. Each blank is then compared to a method blank. Therefore, contamination could be masked.

Corrective Action:

The NWQL SOP INCW0119.6, Chemical oxygen demand by HACH DR/4000 colorimeter, will be amended to include a reference blank with each run using a processed DI water blank in the COD vial as suggested by HACH. The instrument will then be zeroed in absorbance mode with the first blank and verification of the pH check will be documented (recorded) in the data packet and included in the SOP.

Please note, the lab has a very low analysis demand for this procedure. The NWQL is in the process of determining whether it will continue this analysis. If the lab does not continue performing the analysis, it will submit form DOH-109 to delete it from its accreditation prior to the completion of the corrective action stated.

Due date – December 12, 2013

Completed: November 21, 2013; Respective SOP pages included to demonstrate procedure change for Chemical Oxygen Demand by HACH DR/4000 colorimeter.

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CHEMICAL TESTING DETAILED METHOD REVIEW (D.1)[M4]

Deficiency: 000D18A A method blank is not performed one per preparation batch, per matrix type. (D.1.1.1.b)
[M4,1.7.3.1(b)]

Method: USGS I-2522-90

Assessor: DeNicola, Kathie

Comments: This refers to all the Kone lab analyses: Ammonia, Nitrite, and Ortho Phosphate. Method blanks are not analyzed with every batch. A batch is at most 20 samples.

Corrective Action:

The NWQL SOP INCF0452.3, Colorimetric determination of ammonium, nitrate plus nitrite, nitrite, and orthophosphate in water by automated discrete analysis ("Kone") will be amended to include a method blank with every batch of environmental samples to measure contamination. Each method blank will relate to a specific batch and the batch will not exceed 20 environmental samples.

Due date – December 12, 2013

Completed: November 13, 2013; Respective SOP pages included to demonstrate procedure change for Colorimetric determination of ammonium, nitrate plus nitrite, nitrite, and orthophosphate in water by automated discrete analysis ("Kone").

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CHEMICAL TESTING DETAILED METHOD REVIEW (D.1)[M4]

Deficiency: 00D110 Each method blank is not critically evaluated as the nature of the interference and the effect on the analysis of each sample within the batch. (D.1.1.1.d) [M4,1.7.4.1]

Assessor: DeNicola, Kathie

Comments: In general, for colorimetric measurement tests, the method batch blank is used to zero the instrument. So, it cannot be used to determine (measure) contamination. Also, the blank does not relate to a specific batch of samples.

Corrective Action:

All colorimetric line SOPs will be amended to include method blanks every 20 environmental samples to measure contamination. Each method blank will relate to a specific batch of 20 environmental samples. The SOPs will be amended to define the method blank and wash, which is used to correct the baseline. These are separate samples. Both will be run as batch QC.

Due date – December 12, 2013

Completed: November 13, 2013; Respective SOP pages included to demonstrate procedure change for Colorimetric determination of ammonium, nitrate plus nitrite, nitrite, and orthophosphate in water by automated discrete analysis (“Kone”) and Determination of ammonium plus organic nitrogen and phosphorus in Kjeldahl digests.

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CHEMICAL TESTING DETAILED METHOD REVIEW (D.1)[M4]

Deficiency: D1121E2 The same analyte exceeds the LCS control limit repeatedly, indicating a systemic problem. The source of the error is not located and corrective action taken. (D.1.1.2.1.e)[M4,1.7.4.2(b)]

Method: USGS O-4127-96

Assessor: DeNicola, Kathie

Comments: There is a practice of automatically qualifying certain GC-MS volatile and semi-volatile compounds as E for estimated regardless of the QC acceptance criteria.

Corrective Action:

Analytes that do not perform ideally in a method or whose data are reported less than the lowest calibration standard (between the lowest reporting level and the LT-MDL), have data qualified with an estimated ("E") code.

The NWQL reviews the application of qualifiers for compounds on an annual basis. Although there is a correlation with sample set QC for the performance of the specific sample matrix in a procedure, there are times where the set QC information may not be reflective of the performance in a specific sample matrix. The NWQL reviews the application of qualifiers for overall performance of analytes on an annual basis as well as for specific QC samples associated with the environmental sample. Application of overall performance qualifiers will use the guidance in the following NWQL SOP MRDX0015.3, Guidelines for method validation and publication.

No corrective action required

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CHEMICAL TESTING DETAILED METHOD REVIEW (D.1)[M4]

Deficiency: D1121E3 The laboratory does not have a written procedure to monitor the application of marginal exceedence allowance to the LCS. (D.1.1.2.1.e) [M4,1.7.4.2(b)]

Method: USGS O-4127-96

Assessor: DeNicola, Kathie

Comments: This refers to the use of the automatic qualifier (E for estimated) regardless of the QC acceptance criteria.

Corrective Action:

There is a written procedure for the applications of marginal exceedences in the NWQL Quality Laboratory manual to evaluate specific QC samples associated with the environmental sample. The application of yearly performance qualifiers for a specific analyte will use the guidance in the NWQL SOP MRDX0015.3, Guidelines for method validation and publication.

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CHEMICAL TESTING DETAILED METHOD REVIEW (D.1)[M4]

Deficiency: 00D133A All standards used in initial calibration are not traceable to a national standard, when available.
(5.5.5.2.2.1.d)[M4,1.7.1.1(d)]

Method: USGS I-1250-85

Assessor: DeNicola, Kathie

Comments: Refers to Color by USGS I-1250-85. All of the discs on the color wheel need to be traceable to a national standard (platinum-cobalt reference standard) at least annually.

Corrective Action:

Platinum-cobalt standards will be prepared to verify the color wheel at 5 different levels annually and NWQL SOP INCW0032.5, Determination of color, will be amended to reflect the change. Levels will include 5.0, 10, 15, 20, and 25 Hazen units. Third party checks (second source initial calibration standard) will be used to verify the color wheel by alternating levels of the platinum-cobalt verification standards every 20 environmental standards.

Due date – December 12, 2013

Completed: November 22, 2013; Respective SOP pages included to demonstrate procedure change for Determination of color.

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CHEMICAL TESTING DETAILED METHOD REVIEW (D.1)[M4]

Deficiency: 00D133 All initial calibrations are not verified with a standard obtained from a second source manufacturer or lot if the lot can be demonstrated from the manufacturer as prepared independently from other lots. (5.5.5.2.2.1.d)[M4,1.7.1.1(d)]

Assessor: DeNicola, Kathie

Comments: 1. Refers to COD by USGS I-3561-85 and MBAS by USGS O-3128-95 - The second-source initial calibration verification standard acceptance criteria was based on the mean of lab results instead of the true certified value. This practice masked an analytical bias which almost resulted in a failed MBAS PT sample.
2. Refers to Phosphate, Total by USGS I-4610-91 and TKN by USGS I-4515-91 - There is no second source initial calibration verification standard that includes the simple form of the analyte to check the calibration standards. The complex or organic forms of the analytes are to check the efficiency of the digestion for a batch of samples.

Corrective Action:

MBAS: Open-File Report 95-189, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory — Determination of methylene blue active substances by spectrophotometry (Burkhardt and others, 1995 p. 15) lists the third party check (TPC) acceptance criteria at $\pm 15\%$ from the known MBAS concentration. The current TPC acceptance criteria have been re-evaluated and new limits have been set at $\pm 15\%$ from the known MBAS concentration. NWQL SOP ORGW0060.3-1, Determination of methylene blue active substances will be revised to reflect this change.

COD: The second source initial calibration verification standard acceptance criteria have been changed to $\pm 20\%$ of the true value. The NWQL SOP INCW0119.6, Chemical oxygen demand by HACH DR/4000 colorimeter, will be amended accordingly.

Phosphate: The NWQL will purchase the simple form ammonia and orthophosphate from a second source to be used as a TPC.

Due date – December 12, 2013

Completed: December 2, 2013; Respective SOP pages included to demonstrate procedure change for the following analytical lines; Determination of methylene blue active substances, Chemical Oxygen Demand by HACH DR/4000 colorimeter, and Determination of ammonium plus organic nitrogen and phosphorus in Kjeldahl digests. Additionally, documentation of COAs that will be used as the TPC and the new standard. We had purchased these standards for another analytical line and will be used for TKN.

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CHEMICAL TESTING DETAILED METHOD REVIEW (D.1)[M4]

Deficiency: 00D137 Corrective actions are not performed if the results of the initial calibration are outside of established acceptance criteria. (5.5.5.2.2.1.i)[M4,1.7.1.1(i)]

Method: SM 18-21 5310B (00)

Assessor: DeNicola, Kathie

Comments: This refers to the lab's practice discovered during the review of the 10/12/12 TOC calibration curve. The lab's practice was to replace calibration standards with previously run standards instead of performing the corrective action procedure.

Corrective Action:

The analyst has been retrained and understands that the calibration curve data will be analyzed on the same date. The NWQL SOP ORGW0483.1-1, Total organic carbon (TOC) analysis, will revise section 7.3.2.6 to state that calibration data must be from the same calibration curve.

Additionally, the analyst will create a new book that contains the calibration curves instead of placing the curves with the analytical run data. The book will start with June 2013 curves. The missing calibration curve from September 12, 2012 (10/12/12) has been located and placed in the book.

Due date – December 12, 2013

Completed: November 26, 2013; Respective SOP pages included to demonstrate procedure change for Total organic carbon (TOC) analysis. Additionally, copy of the new calibration curve book cover and the calibration curve from September 12, 2012 (10/12/12).

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Assessment ID: 3242
Assessment Date: 08/08/13
Assessment Type: General Assessment
Primary Assessor: Zgrodnik, Tom

CHEMICAL TESTING DETAILED METHOD REVIEW (D.1)[M4]

Deficiency: 00D141 If the reference or mandated method does not specify the number of calibration standards, the minimum used is not two, not including a blank or zero standard. (5.5.5.2.2.1.j)[M4,1.7.1.1(j)]

Method: USGS I-4471-97

Assessor: Zgrodnik, Tom

Comments: The analyst is using a 1 point calibration curve. The SOP for this method does not specify how many points are to be used for the calibration. A minimum of 3 points should be used to construct calibration curves for ICP-AES

Corrective Action:

The NWQL ICP-AES method uses a 1-point calibration curve forced through zero. Table 2 of NWQL SOP INCM0386.5, Trace metals and potassium determination by inductively coupled plasma-optical emission spectrometry, is confusing on the number of calibration points used. The perceived "2-point calibration" is actually the background correction. The SOP will be clarified to relate the background correction and the calibration type. The NWQL is planning to analyze 5-point calibration curves monthly to support the MDL determination process.

Due date – December 12, 2013

Completed: December 2, 2013; Respective SOP pages included to demonstrate procedure change for Trace metals and potassium determination by inductively coupled plasma-optical emission spectrometry. NWQL evaluates and updates MDLs at the end of the calendar year. Institution of the 5-point standard calibration curve will begin in January 2014.

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CHEMICAL TESTING DETAILED METHOD REVIEW (D.1)[M4]

Deficiency: 00D143 The details of the continuing instrument calibration procedure, calculations and associated statistics are not included or referenced in the test method SOP. (5.5.5.10.a)[M4,1.7.2(a)]

Assessor: Zgrodnik, Tom

Comments: See comment for 00D141. The SOP should specify how to construct a calibration curve.

Corrective Action:

The NWQL ICP-AES method currently uses a 1-point calibration curve forced through zero. Table 2 of the SOP is confusing on the number of calibration points used. The perceived "2-point calibration" is actually the background correction. The NWQL SOP INCM0386.5, Trace metals and potassium determination by inductively coupled plasma-optical emission spectrometry, will be clarified to relate the background correction and the calibration type. The NWQL is planning to analyze 5-point calibration curves monthly to support the MDL determination process. The ICP-AES procedure discusses the QC requirements and process for acceptance limits in Attachment 6. The NWQL will review and revise the attachment to clarify the information.

Due date – December 12, 2013

Completed: December 2, 2013; Respective SOP pages included to demonstrate procedure change for Trace metals and potassium determination by inductively coupled plasma-optical emission spectrometry.

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For ICP and/or ICP-MS the following does not occur:

Deficiency: 555221FE The linearity is verified at a frequency established by the method and/or the manufacturer.
(5.5.5.2.2.1.h.4)[M4,1.7.1.1(h)(iv)]

Method: USGS I-4471-97

Assessor: Zgrodnik, Tom

Comments: For ICP-AES, the linearity was not established in the year 2012.

Corrective Action:

The analyst found the linearity check that was completed on August 1, 2012, however, there were issues with the dilutions and the instrument QC, therefore the linearity check failed for several elements. The linearity check was not rechecked until January 2013. The 2012 linearity check was printed out and placed in the note book on November 6, 2013. The NWQL will monitor the annual linearity checks more closely, so that if a linearity check fails, the check is repeated immediately.

Completed – November 6, 2013; We have included a copy of the 2012 linearity check.

I understand that under Section 55-2.6 of 10 NYCRR, a Certificate of Approval may be revoked, suspended or denied if any material fact pertinent to obtaining or retaining such certificate is misrepresented. Such misrepresentation may also violate NYS Penal Law Article 175 and subject parties who file a false instrument to criminal prosecution. I attest that this plan of correction has been/or will be implemented by the date(s) indicated.

Lab Director: _____ Date: _____