

Lab: 11642 USGS NATIONAL WATER QUALITY LABORATORY
Address: MS 407 – BLDG 95 – DENVER FED CTR
DENVER, CO 80225-0046

Assessment ID: 1805
Assessment Date: 08/02/07
Assessment Type: General Assessment
Primary Assessor: Dersham, Randy

INTRODUCTION

Deficiency: 504 The laboratory does not make accurate statements concerning their NELAP accreditation fields of testing and NELAP accreditation status. (Chapter 4 Sec. 4.6.1; Chapter 6 Sec. 6.8.a.2, NELAC 2003)
Assessor: Dersham, Randy

Comments: Database of reported data needs to identify what analytes are covered by environmental certifications.

Corrective Action: New data fields will be added to the LIMS that will allow for NELAP accredited analyte/method combinations to be identified; the fields will include state identifier, analyte name, test code, effective date, and expiration date. A flag will then be added to the NWQL catalog and NWQL sample status web sites to identify those analyte/method combinations that are NELAP accredited. This information will also be added to all sample reports in a comment field. The corrective action will be implemented in accordance with NWQL's Change Control Board scheduling, and will be completed no later than December 31, 2007.

ORGANIZATION AND MANAGEMENT

Deficiency: 5432 The laboratory does not nominate deputies in the case of absence of the technical director or QA officer. (Sec. 5.4.1.5.j NELAC 2003)
Assessor: Dersham, Randy

Comments: Needs to be in writing in quality manual.

Corrective Action: An amendment report to the Quality Management System document (QMSd) that documents the designation of a deputy technical director and a deputy quality assurance officer has been prepared and approved. A copy of this approved amendment report is attached.

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DOCUMENT CONTROL

Deficiency: 54321 All documents are not issued to personnel in the laboratory as part of the quality system reviewed and approved for use by authorized personnel prior to issue.
(Sec. 5.4.3.2.1 NELAC 2003)

Method: USGS I-3561-85

Assessor: Denicola, Kathie

Comments: This refers to the need to control "bench notes" used by the analyst.

Corrective Action: Bench Notes have been pulled from the COD method and instrument area. If the use of bench notes is required in the future they will be published in the form of an amendment report or a revision of the SOP.

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REVIEW of REQUESTS, TENDERS and CONTRACTS

Deficiency: 5541B1 The laboratory does not inform the client of the results of the review if it indicates any potential conflict, deficiency, lack of appropriate accreditation status, or inability on the laboratory's part to complete the clients work. (Sec. 5.4.4.1.b NELAC 2003)

Assessor: Dersham, Randy

Comments: Review of requests, tenders and contracts does not include evaluation of state certification requirements.

Corrective Action: The National Water Quality Laboratory (NWQL) analyzes samples submitted by U.S. Geological Survey Water Science Centers in every state. Samples are generally associated with USGS national water-quality programs. However, Water Science Centers can submit samples that are associated with cooperative studies in the state in which they reside. When cooperative studies require a NELAP accredited laboratory, the Water Science Center notifies NWQL requesting that secondary accreditation be obtained. It is only under these circumstances that NWQL obtains secondary accreditation. Secondary accreditation must be obtained prior to the start of the project or the analysis of associated samples. Samples associated with cooperative studies are identified using a unique project number. Even though the NWQL analyzes samples from every state that has NELAP accrediting authority, we only have primary and secondary accreditation from states that have cooperative programs with the Water Science Centers.

To ensure that State certification requirements are fulfilled, NWQL will provide Water Science Centers with the following information. When joint projects involve State cooperators, the State can require the laboratory that analyzes the samples associated with the joint project be NELAP accredited. If the State requires NELAP accreditation, the Water Science Center shall contact NWQL to inquire whether it has the required accreditation. If the NWQL is accredited by that State, samples can be submitted for analysis. If the NWQL does not have the required State accreditation, primary or secondary accreditation must be established before samples can be submitted to NWQL for analysis.

Water Science Centers also have the option of having the NWQL review their Quality Assurance Project Plans (also called Sampling and Analysis Plans). NWQL staff examine these plans to ensure that NWQL possesses the necessary personnel, equipment, and informational resources, that analytical methods meet the plan's data quality objectives, and that analysts have the skills and expertise necessary for the performance of the requested environmental tests.

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SUBCONTRACTING

Deficiency: 5142 Where a laboratory subcontracts any part of the testing covered under its NELAP accreditation, records do not indicate that this work is placed with a laboratory accredited under NELAP for the tests performed. (Sec. 5.4.5.1 NELAC 2003)

Assessor: Dersham, Randy

Comments: Radiochemistry subcontracted lab does not have certification in NY.

Corrective Action: The National Water Quality Laboratory's (NWQL) contract laboratory for radiochemical analyses has primary NELAP accreditation from the state of California. The NWQL is not aware of any cooperative program in New York that submits radiochemical samples through the U.S. Geological Survey Water Science Center that would require secondary accreditation from New York. If cooperative studies require secondary accreditation, the New York Water Science Center will notify NWQL requesting that secondary accreditation be obtained prior to the start of the project. After the reciprocal agreement is obtained, samples associated with the cooperative study are identified using a unique project number.

To ensure that State certification requirements are fulfilled, NWQL will provide Water Science Centers with the following information. When joint projects involve State cooperators, the State can require the laboratory that analyzes the samples associated with the joint project be NELAP accredited. If the State requires NELAP accreditation, the Water Science Center shall contact NWQL to inquire whether it has the required accreditation. If NWQL is accredited by that State, samples can be submitted for analysis. If NWQL does not have the required State accreditation, primary or secondary accreditation must be established before samples can be submitted to NWQL for analysis.

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RECORDS

Deficiency: 5124 The record keeping system does not allow historical reconstruction of all laboratory activities that produced the resultant sample analytical data. (Sec. 5.4.12.1.5 NELAC 2003)

Method: USGS I-1750-85

Assessor: Denicola, Kathie

Comments: This refers to the need to record the times and temperatures of evaporating and drying cycles (ROE from FU & RU bottles). Evaporate below boiling (98 degrees C). Temperature validation cannot be performed.

Corrective Action: The total error for the probe wire, calibrating thermometer, and oven temperature control is $\pm 4.3^\circ \text{C}$. The temperature monitor trace for each analysis is printed and attached to each analytical run data packet. Average temperatures at holding periods will be within $\pm 4.3^\circ \text{C}$ of the expected temperature for the run to be approved. Temperature criteria and tracking and documentation procedures are published in the attached amendment report to the SOP. Also attached is a copy of an oven temperature profile.

RECORDS: strip charts, tabular printouts, computer data files, analytical notebooks, and/or run logs do not include:

Deficiency: 51223G sample preparation including clean-up & separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations & reagents used. (Sec. 5.4.12.3.3.g NELAC 2003)

Method: USGS O-3116-87

Assessor: Denicola, Kathie

Comments: This refers to the need to include the Batch QC in the prep. records (there was no prep sheet for BNA LCS or Blank)

Corrective Action: While this finding is specific to the BNA line, the NWQL has incorporated the use of Sample Preparation Data Sheets for all organic sample preparation lines at this time. Method blanks, laboratory control samples, and other QC samples will be treated the same as environmental samples and a Sample Preparation Data Sheet will be filled out completely for each one. See the attached documentation for an example Sample Preparation Data Sheet.

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Deficiency: 51223I standard & reagent origin, receipt, preparation, and use. (Sec. 5.4.12.3.3.i NELAC 2003)

Method: USGS O-3116-87

Assessor: Denicola, Kathie

Comments: This refers to the need to track calibration standard origin on raw data (run logs).

Corrective Action: The NWQL will include identifications of calibration standards, second source calibration verification standards, and continuing calibration verification standards in the raw data packages on the data review summary sheets. The approved amendment report for NWQL SOP ORGM 389 (NWQL Method O-3116-87) is included as an attachment.

RECORDS: strip charts, tabular printouts, computer data files, analytical notebooks, and/or run logs do not include:

Deficiency: 51223I standard & reagent origin, receipt, preparation, and use. (Sec. 5.4.12.3.3.i NELAC 2003)

Method: USGS O-3100-83

Assessor: Denicola, Kathie

Comments: This refers to record the source of the "third party check".

Corrective Action: A new Third Party Check Standard was ordered in August 2007, and the expired standard has been replaced. When new standards are prepared the analyst will be reminded to record the preparation in the appropriate standards logbook. See the attached information.

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DATA INTEGRITY

Deficiency: 54158 Senior managers acknowledge their support of these procedures by upholding the spirit and intent of the organizations data integrity procedures, and effectively implementing the specific requirements of the procedures. (Sec. 5.4.2.7 NELAC 2003)

Assessor: Denicola, Kathie

Comments: This refers to the need to effectively perform the "in-depth data monitoring" requirement of Data Integrity. Many of the deficiencies relate to data integrity (data validation) problems.

Corrective Action: The National Water Quality Laboratory's (NWQL) Ethics and Data Integrity Plan was implemented on July 12, 2007. The Plan was signed by all senior management as well as every unit supervisor. An integral part of the Plan is quarterly in-depth monitoring of records associated with sample analysis, from sample receipt at the NWQL through data release and retrieval by the customer. Inorganic and organic methods are randomly reviewed every quarter. See the attached documentation for evidence of the NWQL's in-depth review. The Plan also requires that annual data integrity training be provided to all staff by the unit supervisors and the ethics coordinator. The training includes discussion of elements in the current NELAC Standards and technical training tailored specifically for the staff. Specific data integrity issues discovered during data reviews, internal and external audits, and investigations will be discussed. Updates to the QMS and Ethics and Data Integrity Plan will also be discussed. The first training sessions are in the planning stage and will be conducted before the end of the calendar year.

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PERSONNEL

Deficiency: 5522A The training program is not relevant to the present and anticipated tasks of the laboratory.
(Sec 5.5.2.2 NELAC 2003)

Method: USGS I-4610-91

Assessor: Denicola, Kathie

Comments: This refers to the need to train the analyst (using the Alpkem analyser) to select calibration method according to the TKN and Total Phosphate SOP (linear, non-forced, should be the preferred selection).

Corrective Action: The NELAC standard (5.5.5.2.2 Instrument Calibration) does not specify the type of calibration, but requires the type of curve fit to be documented in the SOP. The SOP (INCM0164.4) for TKN and total phosphate documents the type of curve to be used in calculating results in section 8.1, Data Acquisition and Calculation Instructions as follows: “The Faspac software automatically sets peak markers and calculates concentrations based upon the type of curve selected. In this case, the curve is a second order fit. Instrument calculations use a calibration function that is estimated from a least-squares fit of nominal calibrant concentrations (x) in relation to peak absorbance (y). A second-order polynomial function ($y = a+bx+cx^2$) usually provides improved concentration estimates at the upper end of the calibration range than the more conventional linear model ($y = a+bx$). There is no loss of accuracy when a second-order fit is used even if the calibration function is strictly linear, because, in this case, the value estimated for the quadratic parameter, c, will approach zero. The analyst uses a second order fit to calculate results and has been trained on this function. The calculation is not forced through zero. No corrective action is necessary.

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PERSONNEL: The training records available for all technical staff do not include:

Deficiency: 566C annual training courses in ethical and legal responsibilities including the potential punishments & penalties for violations. (Sec 5.5.2.7 NELAC 2003)

Assessor: Dersham, Randy

Comments: Not completed in 2005.

Corrective Action: Staff did not receive a formal training course covering ethical and legal responsibilities in 2005. However, in 2005, all staff provided signature for the record that they had read, acknowledged, and understood their personal and legal responsibilities including potential punishments and penalties for violations as described in Appendix D – Code of Ethics in the National Water Quality Laboratory's Quality Management System document (QMS) (see the attached documentation). The Code of Ethics discusses scientific misconduct, fabrication, falsification, and plagiarism and requires staff to comply with the Federal Policy on Research Misconduct. The Code also defines specific punishments and penalties. The training course for 2007 is scheduled for December 13th and 14th.

PERSONNEL: The training records available for all technical staff do not include:

Deficiency: 566D annual signature evidence for each employee demonstrating that the employee has read; acknowledges and understands their personal & legal responsibilities including potential punishments & penalties for violations. (Sec. 5.5.2.7 NELAC 2003)

Assessor: Dersham, Randy

Comments: Not completed in 2005.

Corrective Action: In 2005, all staff provided signature for the record that they had read, acknowledged, and understood their personal and legal responsibilities including potential punishments and penalties for violations as described in Appendix D – Code of Ethics in the National Water Quality Laboratory's Quality Management System document (QMS). The Code of Ethics discusses scientific misconduct, fabrication, falsification, and plagiarism and requires staff to comply with the Federal Policy on Research Misconduct. See the attached documentation for an example of the signature page.

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Deficiency: 566E documentation certifying that the employee has read, understands and agrees to use the latest version of a test method used. (Sec. 5.5.2.6.c.3 NELAC 2003)

Assessor: Dersham, Randy

Comments: A few files missing training record when minor updates to SOPs are done.

Corrective Action: The current method used to track and file training records will be replaced with an electronic system within the next 8 to 12 months. The Integrated Document Management System (IDMS) is currently in a testing stage that will be followed by a training phase. Once the system is implemented, training needs will be automatically sent to staff via email and completion of the required training will be documented and tracked in a database. See the attached documentation for further information.

TEST METHODS AND SOPs

Deficiency: 5109 The laboratory does not use appropriate test methods and procedures for all tests and related activities within its responsibility (including sample collection, handling, transport, storage, preparation and analysis. (Sec. 5.5.4.2 NELAC 2003)

Assessor: Dersham, Randy

Comments: Using unapproved method for NW metals digestion.

Testing for some analytes in NY samples that the lab is not certified for such as TPH, color, Br, F, some metals, and U by ICP/MS.

Corrective Action: National Water Quality Laboratory (NWQL) U.S. Geological Survey Methods I-4471-97 and I-4472-97 for the determination of elements in whole-water digests using inductively coupled plasma-atomic emission spectrometry and inductively coupled plasma-mass spectrometry, respectively, specifically cites the use of the in-bottle digestion procedure for sample preparation (see the attached documentation). Therefore, it was our understanding that since the procedure is an integral part of these methods and that these methods are accredited, then the digestion procedure is acceptable. Furthermore, since these methods and the in-bottle digestion procedure were implemented in 1998, 266 out of 277 inductively coupled plasma-atomic emission spectrometry and inductively coupled plasma-mass spectrometry proficiency test results have been satisfactory.

The NWQL analyzes samples submitted by U.S. Geological Survey Water Science Centers in every state. Samples are generally associated with national water-quality programs. However, Water Science Centers can submit samples that are associated with cooperative studies in the state in which they reside. When cooperative studies require the determination of an analyte that is not accredited at NWQL, the Water Science Center requests that NWQL obtain accreditation. If accreditation is available through the primary accreditation authority, NWQL obtains the required proficiency test samples and submits application for accreditation. The NWQL will pursue accreditation for all ELAP-offered analytes appropriate to the NWQL.

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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 NELAC 2003)

Assessor: Dersham, Randy

Comments: Thermometer calibrations not completed in 2006.

Corrective Action: We had already found this deficiency prior to the audit and had completed the 2007 calibration checks by the end of July 2007. Attached is the log used to track the date of calibration, the person who performed the calibration, required adjustments (if needed), and other pertinent data.

The failure to check the calibrations in 2006 was due to the loss of the individual performing that task. Once we noted the deficiency we assigned the task to a new person and provided training for a backup person to ensure that this duty will continue to be performed even if the primary leaves.

EQUIPMENT AND REFERENCE MATERIALS

Deficiency: 5912 The results of support equipment calibration within the specifications are not requires of the application for which it is used. (Sec. 5.5.5.2.1.b NELAC 2003)

Assessor: Dersham, Randy

Comments: Sample receipt IR guns did not have correction factors of calibration applied.

Corrective Action: The IR devices in Login were calibrated June 5, 2007; however, they do not measure to the tenth of a degree. New IR devices will be purchased that measure to the tenth of a degree and the Laboratory Management Information System will be updated to accommodate the decimal point and tenth of a degree.

Completion Date: November 30, 2007

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Deficiency: 5916 Balances, ovens, refrigerators, freezers, incubators and water baths are not checked on each working day with NIST traceable references (where available) before use in the expected use range. (Sec 5.5.5.2.1.d NELAC 2003)

Method: EPA 1664A

Assessor: Denicola, Kathie

Comments: This refers to the need to check the balance in the range of the sample measurements (about 1500g).

Corrective Action: NIST traceable weights will be used to check the balance at 1000 to 1200 g. Balance checking procedures are published as the attached amendment report to the SOP.

EQUIPMENT AND REFERENCE MATERIALS

Deficiency: 5918 The acceptability for use or continued use is not in accord with the needs of the analysis or application for which it is used. (Sec. 5.5.5.2.1.d NELAC 2003)

Method: USGS I-1750-85

Assessor: Denicola, Kathie

Comments: This refers to the need to include and use the correct temperature range for the Residue Analyses (ROE from FU & RU bottles). The oven temperature records were out of method range (180 +/- 2 & 103-105).

Corrective Action: The total error for the probe wire, calibrating thermometer, and oven temperature control is $\pm 4.3^{\circ}$ C. The temperature monitor trace for each analysis is printed and attached to each analytical run data packet. Average temperatures at holding periods will be within $\pm 4.3^{\circ}$ C of the expected temperature for the run to be approved. Temperature criteria and tracking and documentation procedures are published in the attached amendment report to the SOP.

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Deficiency: 5919 Mechanical volumetric devices, including burettes, are not checked for accuracy on a quarterly basis.
(Sec. 5.5.5.2.1.e NELAC 2003)

Assessor: Dersham, Randy

Comments: Not performed quarterly in 2006 for pipettes.

Corrective Action: Pipettes accuracy checks were missed in 2006 during a change of personnel. The primary coordinator left the laboratory and during the transition period the accuracy checks for various pipettes were missed. We re-evaluated the process and determined that it would be more effective to have an outside contractor complete the pipette accuracy checks on a quarterly basis.

We have revised the service contract so that the service representative will now complete the accuracy checks for all pipettes on a quarterly basis as is stated in the NWQL Standard Operating Procedure.

MEASUREMENTS TRACEABILITY AND CALIBRATION

Deficiency: 51028 The records of reagent and standard preparation do not indicate traceability to purchased stocks or neat compounds and include the date of preparation and preparer's initials.
(Sec. 5.5.6.4.c NELAC 2003)

Method: USGS O-3100-83

Assessor: Denicola, Kathie

Comments: This refers to the need for "third party check standard" traceability to a certified standard that is not expired.

Corrective Action: A new Third Party Check Standard was ordered in August 2007, and the expired standard has been replaced. When new standards are prepared the analyst will be reminded to record the preparation in the appropriate standards logbook. See the attached information.

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SAMPLE HANDLING

Deficiency: 51118 The results of all checks are not recorded. (Sec. 5.5.8.3.1.b NELAC 2003)

Assessor: Dersham, Randy

Comments: Sample login needs a permanent record for preservation checks.

Corrective Action: The NWQL's Laboratory Management Information System (LIMS) does record the condition of each sample to include if the sample was received at the proper temperature, with the bottle intact, and with the proper preservation. When an improperly preserved sample is identified, the exception is noted and LIMS is updated to reflect the discrepancy. See the attached documentation for a LIMS excerpt.

SAMPLE HANDLING

Deficiency: 51118 The results of all checks are not recorded. (Sec. 5.5.8.3.1.b NELAC 2003)

Method: USGS I-3561-85

Assessor: Denicola, Kathie

Comments: This refers to the need to record the preservation checks (COD).

Corrective Action: Current laboratory login procedures initially record the samples as being preserved in the LIMS if the preservation check is to be performed by the analyst. If a preservation check reveals that a sample was not properly preserved then procedures outlined in the Login SOP are followed (see attached documentation) and the sample's status as not being preserved is recorded in LIMS. The procedures for preservation checking, follow up, and the Login SOP reference are included in the attached amendment report to the SOP.

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SAMPLE HANDLING: The sample acceptance policy criteria does not include the following:

Deficiency: 51110a Proper, full and complete documentation, which includes sample ID, location, date, time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample.
(Sec. 5.5.8.3.2.a NELAC 2003)

Assessor: Dersham, Randy

Comments: Not using EPA acceptance criteria when more stringent than lab criteria.

Corrective Action: The NWQL was unaware of this special requirement. Section 5.5.8.3.2 of the 2003 NELAC standards states that "The laboratory must have a written sample acceptance policy that clearly outlines the circumstances under which samples shall be accepted or rejected." Section 4.5, "Login sample acceptance policy" of NWQL SOP #SSSX0076.4 "Login Unit of the National Water Quality Laboratory" contains the following subsections that outline sample acceptance: 4.5.1 Warm Samples; 4.5.2 Improperly Preserved Samples; 4.5.3 Improper Sample Container; 4.5.4 Non-Standard Frozen Samples; 4.5.5 Hazardous Samples; 4.5.6 Broken Samples; and 4.5.7 Problem Samples. All criteria listed in section 5.5.8.3.2.a are documented as evidenced by the USGS Analytical Services Request form (see attached documentation). The login sample acceptance policy was written for NWQL processes - the NELAC standards do not require that a laboratory use EPA login acceptance criteria if the EPA login acceptance criteria are more stringent.

ASSURING the QUALITY of TEST RESULTS

Deficiency: 5535 All quality control measures are not assessed or evaluated on an ongoing basis nor quality control acceptance limits used to determine the usability of the data. (Sec. 5.5.9.2.b NELAC 2003)

Method: USGS O-4127-96

Assessor: Denicola, Kathie

Comments: This refers to the need to put control limits that were in use at the time of the analysis on the control charts (VOC & COD LCS charting were some of the examples found).

Corrective Action: During the audit, many of the control charts that were shown to the assessor had the default control limits of 70-130%. The VOC group has since learned the procedure for updating the control limits in LIMS so that they are accurately represented on the control charts. See the attached documentation for an updated control chart for chloroform.

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REPORTS: The report does not contain:

Deficiency: 5133I date of receipt of sample, date and time of sample collection, dates of performance of tests, and time of sample preparation and/or analysis if the required holding time for either activity is less than or equal to 72 hours. (Sec. 5.5.10.2.g NELAC 2003)

Assessor: Dersham, Randy

Comments: need time of analysis

Corrective Action: The NWQL nutrients unit follows the 30 day holding time criteria for all nutrient tests including ortho-phosphate and nitrite as established in USGS WRIR 98-4118, U.S. Geological Survey Nutrient preservation Experiment-Experimental Design, Statistical Analysis, and Interpretation of Analytical Results. This publication was supplied to ELAP some years ago. As such, EPA holding time requirements for ortho-P and nitrite of 48 hours are not met since the USGS study established a 30 day holding time for both analytes. Due to the 30 day holding time requirement, the time of analysis is not recorded with the analytical run data. No corrective action is required.

Appendix D - Essential Quality Control Requirements

Deficiency: 000D11 The laboratory does not demonstrate that it meets all requirements contained in a mandated test method or by regulation, even if the requirement is more stringent than the corresponding NELAC standard.
(Sec. 5.1.1.b NELAC 2003)

Method: EPA 1664A

Assessor: Denicola, Kathie

Comments: This refers to the need to rinse, with n-hexane, the glass rod used for pH check into the separatory funnel used to extract the sample (EPA 1664 section 8.2).

Corrective Action: The glass rod will be rinsed with n-hexane back into the sample bottle and the bottle stored in an explosion proof refrigerator. The procedures are published in the attached amendment report to the SOP.

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Deficiency: 000D11 The laboratory does not demonstrate that it meets all requirements contained in a mandated test method or by regulation, even if the requirement is more stringent than the corresponding NELAC standard.
(Sec. 5.1.1.b NELAC 2003)

Method: AOAC 973.55

Assessor: Denicola, Kathie

Comments: This refers to the need to pre-rinse filters with three 20-ml portions of water (SUSO). The lab is not presently certified for this analyte.

Corrective Action: Pre-rinsing and post rinsing procedures will be followed as described in the source method. New procedures are published in the attached amendment report to the SOP.

Appendix D - Essential Quality Control Requirements

Deficiency: 000D11 The laboratory does not demonstrate that it meets all requirements contained in a mandated test method or by regulation, even if the requirement is more stringent than the corresponding NELAC standard. (Sec. 5.1.1.b NELAC 2003)

Assessor: Denicola, Kathie

Comments: This refers to the need to follow EPA 1664 section 8.1.1 for collecting samples to establish volume of preservative needed if samples are to be stored (so pH checking can then be done at time of analysis).

Corrective Action: Samples are preserved in the field upon collection with 2 mL of concentrated sulfuric acid. Samples are checked for pH upon delivery to the analyst and if the pH is greater than 2 then 2 mL of concentrated sulfuric is added to the sample and the appropriate action regarding non-preserved samples is taken as referenced in the SOP and Amendment Reports. No corrective action is necessary.

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Assessment Date: 08/02/07
Assessment Type: General Assessment
Primary Assessor: Dersham, Randy

Deficiency: 000D12 The quality control protocols specified by the laboratory's method manual are not followed by all analysts. (Chapter 5 Appendix D NELAC 2003)

Method: USGS I-4464-01 (Hg)

Assessor: Dersham, Randy

Comments: Not using linear curve as per SOP. Dropping ICAL points not allowed in SOP.

Corrective Action: The method file in the instrument has been changed to use a linear curve for all future analyses. An example of the new curve is attached.

Section 7.3.2 of the mercury SOP discusses the calibration curve, but doesn't have any guidance on dropping points. Attachment 4 of the SOP provides directions for dropping ICAL points (see attachment #4 from the SOP). In addition, the new *USGS National Water Quality Laboratory Protocol for the Calibration of Analytical Instrumentation* has guidance on when ICAL points may or may not be dropped (see paragraph h in section *Initial Instrument Calibration*).

We have re-processed some random files to determine the impact of running with a second order curve verses the required linear curve. The files checked to date do not show significant changes in the results, but we will check several packets to ensure that this is true (see attached example). If the results are found to have significant changes (differences from the reported value that are larger than normal method variance) we will update all affected results using the linear model. Since we are analyzing samples within the linear range of this method, we expect that there will be no significant changes.

Appendix D - Essential Quality Control Requirements

Deficiency: 000D13 All essential quality control measures are not incorporated into the laboratory method manual. (Chapter 5 Appendix D NELAC 2003)

Method: EPA 1664A

Assessor: Denicola, Kathie

Comments: This refers to the need for collecting extra sample bottles every ten samples to be used for spike and duplicate analyses (EPA 1664 section 8.2 & 9.3).

Corrective Action: NWQL customers submit and request "matrix spike and, if used, matrix spike duplicate," sample analysis as described in Section 8.2, "Sample Collection, Preservation, and Storage," of EPA Method 1664A when their data quality and quality control objectives require such results. By doing so, matrix spike results provide recovery information directly related to the particular study's matrix (as stated in section 9.3). In lieu of a matrix-spike sample, a laboratory fortified reagent blank, or spike, is analyzed at a minimum frequency of 1 for every 10 environmental samples. No corrective action necessary.

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Primary Assessor: Dersham, Randy

APPENDIX D.1 - CHEMICAL TESTING AND AIR TESTING DETAILED METHOD REVIEW

Deficiency: 00D112A The LCS is not used to evaluate the performance of the total analytical system including all preparation and analysis steps. (D.1.1.2.1.a NELAC 2003)

Method: EPA 1664A

Assessor: Denicola, Kathie

Comments: This refers to the need to include the pH check step in the IDL sample analysis.

Corrective Action: All QC samples, including the IDL, are acidified by the addition of 2 ml of sulfuric acid immediately prior to preparation and analysis. Since all QC samples are prepared in deionized-water matrix, the lack of buffering capacity ensures that the pH is less than 2. No corrective action necessary.

APPENDIX D.1 CHEMICAL TESTING AND AIR TESTING DETAILED METHOD REVIEW

Deficiency: 00D130 The continuing instrument calibration verification is not used to confirm the continued validity of the initial calibration. (Sec. 5.5.5.2.2 NELAC 2003)

Assessor: Denicola, Kathie

Comments: This refers to the need to verify non-linear curves at more than one mid-point (as determined by the policy for determining the number of standards necessary to define each curve type).

Corrective Action: The National Water Quality Laboratory has established a lab-wide policy with regard to the number of Continuing Calibration Verification standards required to verify calibration curves based on different regression models. The policy is described in the new *USGS National Water Quality Laboratory Protocol for Calibration of Analytical Instrumentation* in paragraph d in section *Continuing Calibration Verification (CCV) Standards* (see attachment page 15b).

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Assessment Type: General Assessment
Primary Assessor: Dersham, Randy

Deficiency: 00D131 The SOPs or the test method SOPs do not reference the details of the initial calibration procedures, including calculations, integrations, acceptance criteria and associated statistics. (Sec. 5.5.5.2.2.1.a NELAC 2003)

Method: USGS O-2060-01

Assessor: Denicola, Kathie

Comments: This refers to the need to include the curve type and number of calibration standards used for HPLC Herbicides.

Corrective Action: The National Water Quality Laboratory has established a lab-wide policy with regard to the number of standards required to establish calibration curves based on different regression models. The policy is described in the new *USGS National Water Quality Laboratory Protocol for Calibration of Analytical Instrumentation* in paragraph h in section *Initial Instrument Calibration* (see attachment page 15b).

APPENDIX D.1 CHEMICAL TESTING AND AIR TESTING DETAILED METHOD REVIEW

Deficiency: 00D131 The SOPs or the test method SOPs do not reference the details of the initial calibration procedures, including calculations, integrations, acceptance criteria and associated statistics. (Sec. 5.5.5.2.2.1.a NELAC 2003)

Method: USGS O-4127-96

Assessor: Denicola, Kathie

Comments: This refers to the need for the acceptance correlation coefficient in the SOP.

Corrective Action: The acceptance criterion for the correlation coefficient has been added to the SOP.

REPEAT DEFICIENCY

Deficiency: 00D131 The SOPs or the test method SOPs do not reference the details of the initial calibration procedures, including calculations, integrations, acceptance criteria and associated statistics. (Sec. 5.5.5.2.2.1.a NELAC 2003)

Method: USGS I-2057-85

Assessor: Denicola, Kathie

Comments: This refers to IC. Point-to-point calibration is not an approved option for initial calibration calculation. The calibration method used for validation should also be used to calculate sample results.

Corrective Action: Corrective action submitted on September 26, 2007.

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Assessment Type: General Assessment
Primary Assessor: Dersham, Randy

APPENDIX D.1 - CHEMICAL TESTING AND AIR TESTING DETAILED METHOD REVIEW

****REPEAT DEFICIENCY****

Deficiency: 00D131 The SOPs or the test method SOPs do not reference the details of the initial calibration procedures, including calculations, integrations, acceptance criteria and associated statistics. (Sec. 5.5.5.2.2.1.a NELAC 2003)

Method: USGS O-3100-83

Assessor: Denicola, Kathie

Comments: This refers to TOC. Point-to-point calibration is not an approved option for initial calibration calculation. The calibration method used for validation should also be used to calculate sample results.

Corrective Action: Corrective action submitted on September 26, 2007.

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. (Sec. 5.5.5.2.2.1.d NELAC 2003)

Method: ASTM 05072-92 (Radon-222)

Assessor: Dersham, Randy

Corrective Action: The NWQL uses a calibration standard supplied by the instrument manufacturer (Beckman Coulter). It is an unquenched standard, part number 566321, Lot Number S408021 with a control number of 012074 (see attached certificate). We verify the Beckman Coulter using standards from Analytics (see attached certificates). Since we have had trouble finding standards we currently use two different activities (0.395 and 20.0 dps) from the same Analytics lot number. We have requested a quote from Analytics to provide a second source standard near 10.0 dps from a different lot.

APPENDIX D.1 CHEMICAL TESTING AND AIR TESTING DETAILED METHOD REVIEW

Deficiency: 00D134 The criteria for the acceptance of an initial calibration is not established or appropriate to the calibration technique employed. (Sec. 5.5.5.2.2.1.e NELAC 2003)

Assessor: Denicola, Kathie

Comments: This refers to the need to establish appropriate acceptance criteria that does not allow discretionary dropping of standards.

Corrective Action: The National Water Quality Laboratory has established a lab-wide policy with regard to rules for dropping calibration points. The policy is described in the new *USGS National Water Quality Laboratory Protocol for Calibration of Analytical Instrumentation* in paragraph h in section *Initial Instrument Calibration* (see attachment page 15b).

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Primary Assessor: Dersham, Randy

Deficiency: 555221F The lowest calibration standard is not the lowest concentration for which quantitative data are to be reported (see Appendix C.) Any data reported below the lower limit of quantitation should be considered to have an increased quantitative uncertainty and shall be reported using defined qualifiers or flags or explained in the case narrative. (NELAC Chapter 5, section 5.5.5.2.2.1.f, July 2003.)

Assessor: Dersham, Randy

Comments: ICP and ICP/MS are not flagging data reported below first ICAL point.

Corrective Action: The ICP is changing to a single point calibration as described in section 5.5.5.2.2.1, Initial Instrument Calibration, of the 2003 NELAC Standards. The new SOP (attached) was forwarded for approval on 10/11/2007.

The ICP/MS methods are being modified to conform to the new *USGS National Water Quality Laboratory Protocol for the Calibration of Analytical Instrumentation* (see *Initial Instrument Calibration* section).

APPENDIX D.1 - CHEMICAL TESTING AND AIR TESTING DETAILED METHOD REVIEW

Deficiency: 00D136 The lowest calibration standard of the initial calibration is not above the detection limit.
(Sec. 5.5.5.2.2.1.h NELAC 2003)

Assessor: Denicola, Kathie

Comments: This refers to the need for a lab-wide policy that applies to the certified testing that is reflected in the SOPs.

Corrective Action: The National Water Quality Laboratory has established a lab-wide policy that the lowest calibration standard of the initial calibration is above the detection limit. The policy is described in the new *USGS National Water Quality Laboratory Protocol for Calibration of Analytical Instrumentation* in paragraph b in section *Initial Instrument Calibration* (see attachment page 15b).

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Primary Assessor: Dersham, Randy

Deficiency: 00D136 The lowest calibration standard of the initial calibration is not above the detection limit.
(Sec. 5.5.5.2.2.1.h NELAC 2003)

Assessor: Denicola, Kathie

Comments: This refers to need to not include or force calibration through zero.

Corrective Action: The National Water Quality Laboratory has established a lab-wide policy stating that regression equations should not be forced through zero. The policy is described in the new *USGS National Water Quality Laboratory Protocol for Calibration of Analytical Instrumentation* in paragraph c in section *Initial Instrument Calibration* (see attachment page 15b).

APPENDIX 0.1 CHEMICAL TESTING AND AIR TESTING DETAILED METHOD REVIEW

Deficiency: 00D140 The laboratory does not have an SOP for determining the number of points for establishing the initial calibration. (Sec. 5.5.5.2.2.1.2 NELAC 2003)

Assessor: Denicola, Kathie

Comments: This refers to the need to establish a policy for the number of standards necessary to define each curve type that does not allow for discretionary dropping of standards.

Corrective Action: The National Water Quality Laboratory has established a lab-wide policy with regard to the number of standards required to establish calibration curves based on different regression models. The policy is described in the new *USGS National Water Quality Laboratory Protocol for Calibration of Analytical Instrumentation* in paragraph h in section *Initial Instrument Calibration* (see attachment page 15b).

Deficiency: 00D156 When the protocol for determining detection limits is not specified, the selection made by the laboratory does not reflect instrument limitations and the intended application of the test method.
(Chapter 5 App. D.1.2.1 NELAC 2003)

Method: USGS I-1750-85

Assessor: Denicola, Kathie

Comments: This refers to the need to use detection limits for analysis and reporting based on minimum measurable residue (ROE from FU & RU bottles).

Corrective Action: Detection limits for this method are derived from statistical analysis of blank sample concentration as there is a distribution around zero for blank sample final weights. See attached blank concentration file and associated control chart. No corrective action is necessary.

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Primary Assessor: Dersham, Randy

APPENDIX D.1 - CHEMICAL TESTING AND AIR TESTING DETAILED METHOD REVIEW

Deficiency: 00D120 The LOD is not verified annually for each quality system matrix, method and analyte according to the procedure specified in C.3. (NELAC Chapter 5, Appendix D.1.2.d, July 2003)
Assessor: Dersham, Randy

Comments: Some metals were not evaluated because they were not included in LT-MDL spike program.

Corrective Action: The referenced metals are non-routine analytes that are determined on a custom basis, and the LODs are established based on customer's needs, method availability, cost, and time constraints. The USGS Branch of Quality Systems includes all NELAP-accredited metals in the LT-MDL program.

APPENDIX D.4 - Radiochemistry Testing Detailed Method Review

Deficiency: 00D42 The quality control protocols are not specified by the laboratory's method manual followed by all analysts.

Method: ASTM 05072-92

Assessor: Dersham, Randy

Comments: Low level CCV 509 had failures without a corrective action as per SOP.

Corrective Action: CCV 509 is one of several in-house QC standards produced in 2005. Over time all of these low-level CCVs have degraded and are no longer valid. We continued to run this CCV knowing it falls outside the original control limits, along with our acceptable CCVs, since it provided consistent results and provided an extra reference value in the low range. This was done, in part, since it is difficult to find sources with low activities that meet the needs of our low analytical range. We agree with the NELAC Assessor that this practice introduced confusion, and we have stopped using this CCV.

Since we have had trouble finding standards we currently use two different activities (0.395 and 20.0 dps) from the same Analytics lot number as noted in deficiency 00D133. We have requested a quote from Analytics to provide a standard near 10.0 dps from a different lot.