

**National Water Quality Laboratory Response
to the
NYS Department of Health – Wadsworth Center
Environmental Laboratory Approval Program
Assessment Report**

Lab: 11642 USGS NATIONAL WATER QUALITY LABORATORY Assessment ID: 1161
Address: MS 407 Bldg 95- Denver Fed Ctr Assessment Date: 07/19/05
Denver CO 80225-0046 Assessment Type: General Assessment
Lead Assessor: Denicola, Kathie

Lab Personnel Participating in Assessment
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Assessors Participating in Assessment
Denicola, Kathie
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Comment: Radon in water only

QUALITY SYSTEM

Deficiency: 5510 The quality manual and related quality documentation does not include a list of all the methods under which the laboratory performs its accredited testing. (Sec. 5.4.2.3.h NELAC 2003)

Comments: This refers to the Quality Manual.

Corrective Action:
The NWQL will prepare an amendment to the Quality Management System document (QMSd), the equivalent of a Quality Manual, which will identify all the methods under which the laboratory performs its accredited testing.

Completion Date:
October 15, 2005

Deficiency: 5520 The quality manual does not define in detail the data integrity procedures, including; a.)__data integrity training, b.)__signed data integrity documentation for all laboratory employees, c.)__in depth periodic monitoring of data integrity, d.)__data integrity procedure documentation (subject to document control procedure). (Sec. 5.4.2.6 NELAC 2003)

Comments: This refers to c.

Corrective Action:
The NWQL will amend the QMSd to include in-depth monitoring of data integrity. A detailed examination of a sample request from sample receipt at the NWQL through data release and retrieval by the customer will be conducted as part of the NWQL internal audits.

Completion Date:
The QMSd will be amended by December 31, 2005.

DOCUMENT CONTROL

Deficiency: 5431A The laboratory does not establish and maintain procedures to control all documents that form part of its quality system, whether internally generated or from external sources. (Sec. 5.4.3.1 NELAC 2003)

Comments: This refers to SOPs.

Corrective Action:

The most recent revision of NWQL SOP QUAX0001.3, "Writing and Approving Standard Operating Procedures at the NWQL" specifies that as SOPs are created, revised, and/or reapproved they will become controlled documents with the master copy placed in the QAS official SOP file. Additional controlled copies will be limited to one for the section's master files and the copy that is available at the official workstation for a procedure.

Completion date:

The implementation of the controlled copy procedure will be completed by June 30, 2006.

SERVICES AND SUPPLIES

Deficiency: 5464 The laboratory does not maintain records of evaluations of all suppliers from whom it obtains support services or supplies required for tests and list those approved. (Sec. 5.4.6.4 NELAC 2003)

Comments: This refers to a need for a list

Corrective Action:

The NWQL maintains a listing of qualified vendors and manufacturers for supplies and services that could affect the quality of the environmental data generated by the laboratory. This listing is maintained by the Administrative Section of the laboratory, the group that performs the actual purchasing function and interfaces with the external contracting office when a contract or purchase agreement is required. As of 09/28/05, this list is on file in the Quality Assurance Section (QAS).

Supplies are purchased from historically reputable manufacturers/vendors. Chemical assays of reagents are supplied by the manufacturer on the labels and suitability for use is verified by routine analysis. When certificates of quality associated parameters are available from the manufacturer, they are kept by the acquiring group with the laboratory.

Services such as maintenance agreements for instrumentation are purchased from the manufacturer for an initial period of time following acquisition of the equipment, usually during the warranty period, and then added to a competitively awarded contract service provider(s) for longer-term needs. These contract instruments are under continuous quality surveillance with regard to compliance with contract specifications including quality and timeliness. Any problems or issues are referred to the government's contracting officer for adjudication and corrective action.

CORRECTIVE ACTION

Deficiency: 54104 The laboratory does not monitor the results to ensure that the corrective actions taken have been effective. (Sec. 5.4.10.4 NELAC 2003)

Comments: This refers to the need to specify how this is to be documented.

Corrective Action:

Quality control corrective actions as outlined in section 2.7 of the QMSd are monitored at the instrument level to ensure that procedures meet acceptance criteria and are documented with the data packages. If there are multiple instances of the same problem, the supervisor is informed and the procedure is investigated, appropriate corrective actions are determined and implemented, and the procedure is monitored to ensure that the problem was fixed. Internal audits are tracked in a database which includes the problem, corrective action, responsible party, due dates, and implementation dates. The Chief of Analytical Services and the Chief of the QAS meet on a weekly basis to discuss data quality issues in the laboratory. Analytical Services Section supervisors meet on a weekly basis to discuss data quality issues in the laboratory. Internal audit findings and corrective actions are reviewed at by the Analytical Services Section on a monthly basis and by the QAS within one month of completion of all audit findings via a follow-up audit.

Deficiency 5532 The laboratory has no general procedures to be followed when there are departures from documented policies, procedures and QC. (Sec. 5.4.10.6 NELAC 2003)

Comments: This refers to the need to specify how this is to be documented.

Corrective Action:

The procedure has been established in the QMSd (sec. 2.6) which was approved in July 2005. The Analytical Services Section will further develop documentation procedures and identify data qualifiers when exceptions to documented methods, procedures, and QC occur. The Analytical Services Section and QAS will work together to modify policies and the QMSd.

Completion Date:

The additional documentation procedures and amendments to the QMSd will be completed by June 2006.

QUALITY SYSTEM: There are no procedures to be followed when there is a departure from documented policies, procedures and QC to include but not limited to:

Deficiency: 5533E There are no procedures specified for management (including the QA officer) to review corrective action reports. (Sec 5.4.10.6.a.5 NELAC 2003)

Comments: This refers to the need to review all corrective action reports.

Corrective Action:

The QAS employs a Quality Investigation (QI) procedure (NWQL SOP QX0315.0, "The QAS Quality Investigation Procedure for Corrective Actions") that utilizes a spreadsheet to track commitments made by NWQL personnel in their responses to in-house audits, external audits, and Performance Testing (PT) study failures. These data are entered into a spreadsheet by QAS personnel and monitored on a periodic basis.

All entries require investigations by QAS personnel and generate commitments from other NWQL personnel to remedy the situations. The commitments usually are associated with a date of expected completion of a task. The QI procedure tracks this information to facilitate follow-up audits followed by follow-up audit reports. When PT failures occur, the NWQL schedules Quality Review meetings to discuss the PT failure, determine the root cause, and develop an effective corrective action.

The tracked categories listed above have been expanded to include analytical supplies problems (field and in-house supplies), district or in-house inquiries, and departures from established procedures as encountered by QAS personnel.

Beginning October 2005, QAS will produce a monthly report to the chief of the QAS regarding the status of the contents of the spreadsheet.

The NWQL is currently developing a program that will compile bench-level quality control data that will allow the NWQL to track trends in on-line QC sample data. It is projected that this program will be in place by April of 2006.

PREVENTIVE ACTION

Deficiency: 54111A The laboratory does not develop, implement and monitor action plans where preventive action is required. (Sec 5.4.11.1 NELAC 2003)

Comments: This refers to the need for documentation.

Corrective Action:

The procedure has been established in the QMSd (sec. 2.7.2) which was approved July 2005.

Completion Date:

Laboratory personnel will be trained to the new version of the QMSd by October 19, 2005.

PREVENTIVE ACTION

Deficiency: 54112 Procedures for preventive action do not include the initiation of such actions and application of controls to ensure that they are effective. (Sec 5.4.11.2 NELAC 2003)

Comments: This refers to the need for documentation

Corrective Action:

The procedure has been established in the QMSd (sec. 2.7.2) which was approved July 2005. The initiation of actions is described in communication, administrative actions, and customer interactions sections. The application of controls is described in the Laboratory Information Management System (LIMS) section of 2.7.2. Additionally Quality System Reviews described in section 2.8.2 of the QMSd are applicable for ensuring preventive actions taken are effective.

Completion Date:

Laboratory personnel will be trained to the new version of the QMSd by October 19, 2005.

RECORDS: The laboratory does not retain records of the following procedures to which a sample is subjected while it is in the lab's possession:

Deficiency: 51221A Sample preservation, appropriateness of sample container, & compliance with holding time requirements. (Sec. 5.4.12.2.5.1. a NELAC 2003)

Comments: This refers to the need for a complete record of preservations performed in the lab.

Corrective Action:

Procedures including records management for checking proper preservation of samples and preservations performed in the laboratory are described in SOP TX0076.3, Login Unit of the National Water Quality

Laboratory. Samples not properly preserved upon receipt at the laboratory are preserved and information recorded on the Sample Preservation Verification Form.

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

Deficiency: 51223F Analyst's or operator's initials/signature. (Sec. 5.4.12.3.3.f NELAC 2003)

Comments: This refers to instrument printouts such as from the auto analyzer instrument

Corrective Action:

Analysts will be instructed to initial all instrument printouts. The instructions will be added to the 4-channel SOP as an amendment report by October 31, 2005.

Completion Date:

The amendment report for the Four channel SOP will be completed by October 31, 2005.

Deficiency: 51223I Standard & reagent origin, receipt, preparation, and use. (Sec. 5.4.12.3.3.i NELAC 2003) [The correct NELAC Standard is 5.4.12.2.5.3.i]

Method: USGS 1-2057-85

Comments: This refers to the need for records of the origin of internal QC samples from BQS (USGS).

Corrective Action:

The date of receipt, date opened, expiration date, and unique identifier of the Branch of Quality Systems (BQS) QC sample bottles will be recorded in the appropriate logbook for IC analyses. The analysis report provided by BQS for the QC samples will also be included in the logbook.

Completion Date:

November 1, 2005.

Note that section 5.4.12.3.3.i does not exist in NELAC 2003. This refers to 5.4.12.2.5.3.i

MANAGERIAL REVIEWS

Deficiency: 5526 The laboratory does not have a procedure for the annual management review of the quality system. (Sec. 5.4.14.2 NELAC 2003)

Comments: This refers to the need for records from 2004

Corrective Action:

A management review of the quality system was conducted in 2003, but was overlooked in 2004. A review of the NWQL's quality system will be presented to the NWQL Management Team by QAS and will take place before December 31, 2005. Such a review will take place at least annually thereafter. Records of review findings and subsequent action assignments will be filed.

Completion date:

December 31, 2005

DATA INTEGRITY

Deficiency: 54155 The data integrity procedures are not signed and dated by senior management. (Sec. 5.4.2.6 NELAC 2003)

Comments: This refers to the need for specific SOP.

Corrective Action:

An SOP modeled after the *Data Integrity Plan* template obtained from the NYSDOH assessor will be prepared. This SOP will describe the NWQL Data Integrity System and include responsibilities and procedures for ethics training, written ethics agreements, data integrity monitoring and documentation. This SOP will be reviewed and signed off annually by senior management. Additionally, the NWQL QMSd will be amended to reflect these Data Integrity Procedures.

Completion Date:

The SOP will be drafted by and the QMSd will be amended by December 31, 2005.

PERSONNEL

Deficiency: 565 The laboratory’s management does not maintain records to assure that all technical laboratory staff have demonstrated and documented initial and ongoing proficiency in the activities for which they are responsible. (Sec 5.5.2.6.b NELAC 2003)

Method: ASTM D-3648 (USGS Only)

Comments: This refers to need for records for Rn 222 analyst’s annual on-going proficiency.

Corrective Action:

The analyst has completed a continuing demonstration of capability for Rn 222. The form was filed with the Records Management Office on September 29, 2005.

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

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)

Method: ASTM D-3648 (USGS Only)

Comments: This refers to latest revisions to Rn 222 SOP.

Corrective Action:

The analyst has completed a training form for the latest version of the SOP. The form was filed with the Records Management Office on September 19, 2005.

PERSONNEL: Data integrity training does not include; (Sec. 5.5.2.7 NELAC 2002)

Deficiency 5527D Record keeping.

Comments: This refers to need for inclusion of slide in training presentation.

Corrective Action:

A slide has been prepared for inclusion in new employee NWQL Data Integrity Training and will be included in annual ethics refresher training. This slide details the proper procedure when making changes to records and includes the following points:

- The individual responsible for the record change draws a single line through the record so that the record remains legible.
- The responsible individual initials and dates change, makes correction and gives the reason adjacent to the change
- Use of white-out, pencil, erasures, or obliteration of information is prohibited

Completion Date:

September 26, 2005

Deficiency: 5527H Requirement for emphasis on the importance or proper written narration on the part of the analyst with respect to those cases where analytical data may be useful, but are in some way partially deficient.

Comments: This refers to need for inclusion of slide in training presentation.

Corrective Action:

Three slides have been prepared for inclusion in new employee NWQL Data Integrity Training. These slides emphasize the importance of using proper data narration with the remark codes and value qualifiers available in the NWQL LIMS. The slides give examples of remark codes and value qualifiers and refer analysts to the appropriate section of the NWQL QMSd for more detailed information.

Completion Date:

September 26, 2005

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)

Comments: This refers to need for expiration date of COD reagent vials. *This deficiency actually refers to the traceability of reagents and standards. Reagent solutions were not discreetly identified with purchased same-lot stock solutions from separate containers.*

Corrective action:

If two or more containers of a purchased stock or neat compound have identical lot numbers or other identifiers they will be labeled with a unique identifier such as the day opened and that identifier will be added to the information recorded on subsequent reagents and in the record book. The analyst has been informed and is trained in this procedure. Effective date: 8/25/05

SAMPLE HANDLING: The following information is not recorded in the laboratory chronological log.

Deficiency: 51122b Date and time of laboratory receipt of sample. (Sec. 5.5.8.3.1.d.1.ii NELAC 2003)

Comments: This refers to time.

Corrective Action:

The actual time the sample is unpacked and logged in is now being recorded on the Analytical Services Request (ASR) form. The date of receipt is reflected on the laboratory identification number which is assigned when the sample is unpacked and logged in. If the date differs from the date the sample was received, the actual receipt date is entered on the ASR and LIMS. The ASR is scanned into the LIMS and is available for review by internal employees and external customers. The NWQL "Sample Status Page", available via the web, provides access to scanned ASRs.

The following information is not easily retrievable upon request and readily available to individuals who will process the sample.

Deficiency: 51123b Date and time of sample collection linked to the sample container and to the date and time received in the laboratory. (Sec. 5.5.8.3.1.d.2ii NELAC 2003)

Comments: This refers to routine samples.

Corrective Action:

The actual time the sample is unpacked and logged in is now being recorded on the ASR form. The date of receipt is reflected on the laboratory identification number which is assigned when the sample is unpacked and logged in. If the date differs from the date the sample was received, the actual receipt date is entered on the ASR and LIMS. The ASR which includes the date and time of sample collection is scanned into the LIMS and is available for review by internal employees and external customers. The NWQL "Sample Status Page", available via the web, provides access to scanned ASRs.

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: USGS 1-2545-90

Comments: This refers to need for cadmium column reduction efficiency checks.

Corrective Action:

Cadmium efficiency check samples will be run at the beginning and end of each run. An amendment report to the nutrient 4-channel SOP will be completed by October 31, 2005 and will include the frequency of use and acceptable limits to accept data for nitrate + nitrite analysis.

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: USGS O-4127-96

Comments: This refers to the need to describe allowable marginal LCS exceedences for VOC SOP.

Corrective Action:

The VOC SOP for USGS method O-4127-96 and the QMSd will be updated to allow for marginal exceedence for laboratory control samples by November 30, 2005.

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

Method: USGS I-4472-97

Comments: This refers to the need for corrective actions (attachment 5) added to the ICP-OES SOP. *There was a typo in the text of NWQL SOP IM0386.1, "Trace metals and Potassium determination by Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES)". The reference to attachment 5 in the SOP should have been attachment 6.*

Corrective action:

The ICP-OES SOP was updated and posted to the NWQL Intranet Page on September 19, 2005.

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

Deficiency: 00D112 An LCS (a sample matrix free of analytes of interest spiked with a verified known amount of analyte) is not performed at a frequency of one per preparation batch, per matrix type. For analyses in which there is no separate preparation method, the LCS is not performed per batch of up to twenty samples. (Chapter 5 App. D.1.1.2.1.b NELAC 2003)

Method: EPA 1664A

Comments: This refers to need for Oil and Grease LCS to include all sample processing steps.

Corrective Action:

The LCS samples are now run through the entire process, blank water is spiked in the appropriate amber glass bottle and the spiked water is then processed. Effective date: 7/28/05. An amendment report will be added to the Oil and Grease SOP OW0086.1, "EPA Method 1664, N-hexane extractable material (HEM) or silica gel treated N-Hexane extractable material (SGT-HEM) by extraction and gravimetry (Oil and grease or total petroleum hydrocarbons)" to document the change in procedure.

Completion date:

November 1, 2005

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)

Comments: This refers to automated colorimetric methods SOP.

Corrective Action:

The details of the calibration procedures including the type of calibration curve fit will be better defined in NWQL SOP ID0163.4, "Ammonia, Orthophosphate, Nitrite, and Nitrate plus Nitrite (4-channel Nutrient analyses, regular level) by automated, segmented-flow analysis" by amendment report by October 31, 2005.

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-3116-87

Comments: This refers to BNA SOP need for appropriate calibration curve regression.

Corrective Action:

Power curve calibration is used for the specific compounds in schedule 1383 (BNA's). This calibration technique provides better agreement with expected concentrations in the lower range of the calibration curve than using quadratic or other available curve fitting algorithms. The accuracy of the power curve is evaluated by analyzing a third-party check-standard around the mid point of the calibration range. The calibration is deemed acceptable if the analyses of the third party check standard solutions, calibration standards, and continuous calibration standard verifications produce results that are within acceptance criteria as denoted in NWQL SOP#OM0389.1. If a compound concentration exceeds the calibration range for the method, the extract is diluted to within the calibration range and reanalyzed. The calibration evaluation procedure is documented in SOP#OM0389.1. Data from the internal NWQL QC process, the USGS BQS organic blind sample program, and external performance testing samples indicate that the calibration procedure produces acceptable data.

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

Comments: This refers to TOC method. *The TOC method uses a point to point calibration to determine concentrations, and a third order least squares curve to determine quality of fit. (0, 1, 2, 3, 4, 5, 7.5, 10, 15, 20, 30, 40, 50 mg/L) are the calibration standards. Current QC is a set spike at 5mg/L, a CCV at 20 mg/L, and a TPC at 5 mg/L.*

Corrective Action:

Point-to point calibration is used for the Total Organic Carbon analyses. The calibration curve is evaluated using a third order least squares regression, with a required R² value ≥ 0.9975. Calibration using quadratic or other available curve fitting algorithms alone have been found to be unacceptable. The accuracy of the point-point curve is evaluated by analyzing a third-party check-standard and a set spike each at a concentration of 5 mg/L, and continuing calibration verification (CCV) standard at 20 mg/L. The calibration is deemed acceptable if the analyses of the third party check standard solution, the set spike and CCV produce results that are within statistically determined control limits. The calibration evaluation procedure is documented in NWQL SOP OM0194.1, "Total Organic Carbon (TOC) Analysis". Data from

the internal NWQL QC process, the USGS BQS inorganic blind sample program, and external performance testing samples indicate that the calibration procedure produces acceptable data.

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. 5.5.5.2.2.1a NELAC 2003)

Method: USGS I-2057-85

Comments: This refers to IC. The calibration range should be divided into high and low range, to keep a linear calibration

Corrective Action:

Point-to point calibration is used for the ion chromatographic analyses. The calibration curve is evaluated using a quadratic curve fit, with a required R² value of 0.999. Calibration using quadratic or other available curve fitting algorithms alone have been found to be unacceptable. The accuracy of the point-point curve is evaluated by analyzing two third-party check-standards, one at a concentration of 150 mg/L and one at 1.5 mg/L. The calibration is deemed acceptable if the analyses of the third party check standard solutions produce results that are within statistically determined control limits. The calibration evaluation procedure is documented in NWQL SOP ID0056.5, "Chloride and sulfate ions by ion chromatography". Data from the internal NWQL QC process, the USGS BQS inorganic blind sample program, and external performance testing samples indicate that the calibration procedure produces acceptable data.

Deficiency: 00D133 All initial calibrations are not verified with a standard obtained from a second source. (Sec. 5.5.5.2.2.1.d NELAC 2003)

Comments: This refers to the need for second manufacturer source of different lot # if the manufacturer demonstrates independence of preparation to use as IC check standards.

Corrective Action:

New calibration standards will be purchased and prepared from a source different from the third party check.

Completion Date:

November 1, 2005.

Lab Director: Merle Shockey (signed)

Date: October 4, 2005