

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

Assessment ID: 2332
Assessment Date: 08/06/09
Assessment Type: General Assessment
Primary Assessor: Zgrodnik, Tom

INTRODUCTION

Deficiency: 508 During the on-site assessment, the laboratory requested withdrawal for a portion of the approved scope. A formal request has not been made to the ELAP Office.

Assessor: Zgrodnik, Tom

Comments: The laboratory does not perform Uranium (activity) by ICP-AES (ELAP ID 3112). A formal written request to drop that certification must be made to the Albany ELAP office. Please submit Form 109, which is available on the ELAP website.

Corrective Action: A letter with Form 109 attached has been written to make several changes to include the withdrawal of the present approved scope, key personnel changes and mailing address changes. The letter with this information was sent to NYSDOH on September 10, 2009. A copy of the letter is attached.

Completed 09/10/09. Documentation previously provided as part of our initial response.

ORGANIZATION AND MANAGEMENT

Deficiency: 5412 The laboratory does not carry out its environmental testing in such a way as to satisfy the needs of the client, the regulatory authorities or organizations providing recognition. (Sec. 5.4.1.2 NELAC 2003)

Assessor: Zgrodnik, Tom

Comments: Lab needs to make a formal request to add Uranium (mass) by ICP-MS to its scope of accreditation. Please submit Form 109, which is available on the ELAP website.

Corrective Action: No corrective action required. NYSDOH (Form 109) only provides PT samples for Uranium (Activity) by ICP-MS. The NWQL wanted to add Uranium (mass) by ICP-MS. Obtaining an additional PT sample from another PT provider is not currently doable, based on current financial constraints. A letter requesting the change was sent to NYSDOH on September 9, 2009. A copy of the letter is attached.

Completed 09/09/09. Documentation previously provided as part of our initial response.

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ORGANIZATION AND MANAGEMENT

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)
Assessor: Zgrodnik, Tom

Comments: The QA department uses checklists when it conducts an internal audit of the laboratory's operations. Those checklists should have the provisions of controlled documents.

Corrective Action: The assessment SOP will be revised to incorporate modified checklists, based on the ones provided. Once approved, the checklists will be part of the SOP and will be considered controlled documents.

Expected completion date: 11/25/09

Completed 11/24/09. The SOP was amended to include the checklists. The SOP is a controlled document. Documentation attached.

RECORDS

Deficiency: 5127 All information relating to the laboratory facilities, equipment, analytical methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are not documented. (Sec. 5.4.12.1.5.a NELAC 2003)

Assessor: Zgrodnik, Tom

Comments: This applies to low-level Hg by fluorescence. The samples undergo digestion, but no bench log or other record of mercury digestion is maintained. The lack of such a record is not legally defensible.

Corrective Action: The SOP will be revised to require that the analysts record the date and times of mercury digestion.

Expected completion date: 9/30/09

Completed 11/24/09. The SOP was amended to require that the analysts record the date and times of mercury digestion. Documentation attached.

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RECORDS

Deficiency: 51215FA Individuals making changes to electronically maintained records are not identified. (Sec. 5.4.12.1.5.f NELAC 2003)

Assessor: Zgrodnik, Tom

Comments: The LIMS program does not provide an audit trail to identify the individual making changes to electronically recorded data. The audit trail feature may have been inadvertently turned off.

Corrective Action: The audit trail functionality currently exists for most of the LIMS tables, including the table where results are stored; however there are some additional reference tables in LIMS in which the audit trail functionality has not yet been applied. The implementation of the audit trail for these tables will be changed in LIMS.

Expected completion date: 11/25/09

Completed 11/20/09. Audit trails have implemented for critical information in order to preclude inadvertent changes. We track the following information: table name, column name, old value, new value, update date, update type, update reason, update user.

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

****REPEAT DEFICIENCY****

Deficiency: 51223I ... standard & reagent origin, receipt, preparation, and use. (Sec. 5.4.12.2.5.3.i NELAC 2003)

Assessor: Zgrodnik, Tom

Comments: In low-level Hg, there is no direct linkage between any chosen analysis and the standards used in that analysis. Records currently indicate only the date received and its expiration date. First use/ last use dates should establish a link.

Corrective Action: The SOP will be revised so that there is complete standards traceability to an analytical result.

Expected completion date: 9/30/09

Completed 11/24/09. The SOP was amended to provide for complete standards traceability to an analytical result. Documentation attached (See previous documentation to deficiency 5127).

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INTERNAL AUDITS

Deficiency: 54131A The internal audit program does not address all elements of the quality system, including testing activities. (Sec. 5.4.13.1 NELAC 2003)

Assessor: Zgrodnik, Tom

Comments: An updated Internal Audit template was given to L. Bressler in the QA Dept. You may find it useful to incorporate the checklists from that template into your own auditing procedure.

Corrective Action: The assessment SOP will be revised to incorporate modified checklists, based on the ones provided. Once approved, the checklists will be part of the SOP and will be considered controlled documents.

Expected completion date: 11/25/09

Completed 11/24/09. The SOP was amended to include the checklists. The SOP is a controlled document. Documentation attached (See previous documentation to deficiency 5431A).

TEST METHODS AND SOPs

Deficiency: 5101 The laboratory does not use appropriate methods and procedures for all test methods and laboratory activities within its scope. (Sec. 5.5.4.1 NELAC 2003)

Assessor: Zgrodnik, Tom

Comments: The wording of the SOP used for routine balance checks of fractional milligram items encourages an analyst to use the less sensitive 3-place balance (milligram range), instead of the 4-place balance (one-tenth mg range) to avoid failures.

Corrective Action: The SOP will be revised to show that switching balances to avoid failures is not an acceptable practice. Personnel will be trained on the revision.

Expected completion date: 11/25/09

Completed 11/16/09. The SOP was amended to include text added to Section 4.3.1 to clarify the responsibility of the balance user conducting balance verification checks. Documentation attached.

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SAMPLE HANDLING

****REPEAT DEFICIENCY****

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

Assessor: Zgrodnik, Tom

Comments: This applies to the pH checks of metals samples. The lab does not indicate on any permanent record whether the pH is found acceptable upon receipt, only if it is NOT acceptable. The lack of such a record is not legally defensible.

Corrective Action: The SOP will be revised to require the results of the pH check done at sample login be recorded.

Expected completion date: 11/25/09

Completed 11/24/09. The SOP was amended to show that the preservation check is recorded at the time of login. Documentation attached.

REPORTS

Deficiency: 5132 The test report does not contain all information necessary for the interpretation of the test results and all information required by the method used. (Sec. 5.5.10.1 NELAC 2003)

Method: USGS 1-1250-85

Assessor: Denicola, Kathie

Comments: [Color] - This refers to the need to specify the result as True (filtered or settled) or Apparent (still cloudy) Color.

Corrective Action: A comment will be added to the sample information in LIMS documenting that the color result is either observed or true. If an RCB sample bottle is used the comment will read "apparent". If an FCC bottle is used the comment will read "true".

Expected completion date: 11/25/09

Completed 11/2/09. As of 11/2/2009 the analyst manually adds a comment in LIMS to each sample specifying if an unfiltered or filtered sample has been analyzed. If an unfiltered sample was used, the comment will read the data is Apparent and if a filtered sample was used the data will be annotated as True.

Additionally, an internal request was initiated October 28, 2009 to the IT department requesting the comment be made automatically based on sample type analyzed in LIMS at the time of data submission. The automation programming is in progress.

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Appendix D - Essential Quality Control Requirements

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 0-2060-01

Assessor: Denicola, Kathie

Comments: [HPLC Herbicides] This refers to the need to use the reference method acceptance criteria for CCV. (Lab could be more stringent than +/- 30%, but not +/- 3 standard deviations if less stringent.)

Corrective Action: The laboratory will review how the control limits for this method were established. The laboratory has method-specific acceptance criteria for continuing calibration acceptance, based on data collected within the laboratory. US EPA methods also allow that alternative acceptance limits may be appropriate. Throughout the NWQL, the use of ± 3 standard deviations is acceptable. Many NWQL analytical systems develop acceptance criteria on an annual basis based on previous CCV year's performance. Individual method requirements are defined in the method SOPs.

Expected completion date: 11/25/09

Completed 11/10/09. At the NWQL, the actual documentation for each analytical procedure is contained in the individual analytical SOPs and/or method documentation. The NWQL typically uses +/- 30% and +/- 20% for calibration standards. The third party check (TPC) has wider limits to account for potential variation in the TPC solutions, which are similar to the EPA standards for drinking water methods.

Specifically, the upper and lower QC limits for USGS Method O-2060-01 are determined based on past performance. The previous year's data (i.e. FY 2009 to generate FY 2010) is transferred from the LIMS "QC Charts" function to a working spreadsheet to obtain the +/- 3 standard deviation values for both the upper and lower limits. In general, analytes must meet the requirements of the mean between 60% - 120% and the standard deviation <25%. We use percent recovery as the units to report data values for CCVs, prep spikes, and prep blanks. After the limits are obtained, comparisons to past limits are discussed within the LCMS group before implementation.

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Appendix D - Essential Quality Control Requirements

Deficiency: 000D13 All essential quality control measures are not incorporated into the laboratory method manual.
(Chapter 5 Appendix 0 NELAC 2003)

Assessor: Zgrodnik, Tom

Comments: Matrix spikes and matrix duplicates are seldom requested by clients and are therefore rarely performed, but when they are requested, the analytical SOPs often do not include provisions to address those elements of the QC protocol.

Corrective Action: No corrective action is required. USGS methods do not require duplicates or spikes. We are recommending to our clients that they request these. Newer USGS methods are beginning to require them, and these methods do show acceptance limits for the spiked compounds.

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: USGS 1-1250-85

Assessor: Denicola, Kathie

Comments: [Color]

Corrective Action: No corrective action is required because according to Chapter 5 App. D.1.1.2.1.b NELAC 2003 the Color analysis is exempt from the LCS requirement because no spiking solutions are available.

Please Note: The catalog requests an RCB bottle type. It is documented that turbidity can increase the true color reading. It is ambiguous for an analyst to decipher whether the sample is cloudy (observed) or not (true) rather bottle type determines designation (RCB or FCC).

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

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. (NELAC Chapter 5, Appendix D.1.1.2.1.e, July 2003.)

Method: USGS 0-1126-95

Assessor: Denicola, Kathie

Comments: [GC/MS Pesticides] This refers to the need to stop using the automatic "E" for estimated result for selected compounds (see Azinphos methyl and Disulfoton).

Corrective Action: The NWQL has reviewed performance data for the compounds identified, and will drop the permanently estimated ("E") status for Azinphos-methyl. Carbaryl remain permanently estimated ("E") in FY10. NWQL will reevaluate the need to "E" code the analytes in this lab schedule annually and make changes as appropriate.

Expected completion date: 10/15/09

Completed 10/15/09. We re-reviewed the method performance for compounds Azinphos-methyl and Carbaryl. Azinphos-methyl was left permanently estimated, because the compound was outside the acceptable limits for compounds as specified in NWQL SOP MRDX0015.3 "Guidelines for method validation and publication at the NWQL". As stated previously, the "E" codes will be reviewed annually.

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

Deficiency: 00D129 Sample results are not quantitated from the initial instrument calibration. (Sec. 5.5.5.2.2 NELAC 2003)

Method: USGS 0-3116-87

Assessor: Denicola, Kathie

Comments: [GC/MS Semi-Vol] This refers to the need to quantify from the initial calibration, and not from any continuing instrument calibration verification standards.

Corrective Action: The SOP will be revised to clarify/note that all sample concentration calculations are to be based on initial calibration.

Expected completion date: 11/25/09

Completed 11/24/09. The SOP was amended to clarify that all sample concentration calculations are to be based on initial calibration. Documentation attached.

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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 for appropriate acceptance criteria in all methods. (TPC +/- 3 standard deviations is not appropriate when used as the initial calibration verification.)

Corrective Action: No corrective action is required. There is no NELAC requirement for this issue. Throughout the NWQL, the use of ± 3 standard deviations is acceptable, and often results in tighter limits than those associated with many published methods. NWQL analytical systems develop acceptance criteria on an annual basis based on the previous year's performance. Individual method requirements are defined in the method SOPs.

Completed 11/10/09. A formal response to the "Second Notice" report of deficiencies (10/07/09) was sent to NYSDOH on 10/10/09.

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)

Method: USGS 1-3561-85

Assessor: Denicola, Kathie

Comments: [COD] This refers to the need to check the spectrophotometer calibration curve at least annually for linearity.

Corrective Action: No corrective action is required because the linearity of the spectrometer is verified during analysis, once using calibration check standards (Certificate of Analysis available) and a second time using third party check standards (made from COD stock verified with NIST). If the check standards do not meet criteria then the instrument is out of linearity and would need service.

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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)

Method: USGS 1-1250-85

Assessor: Denicola, Kathie

Comments: [Color] This refers to the need to check the color wheel at least annually for linearity.

Corrective Action: The color wheels (discs) will be recertified annually through Orbeco-Hellige recertification program. The SOP will be revised. A second set of wheels will be purchased to be used as Third Party Checks.

Please Note: Recertification takes 2-4 weeks. Recertification documents that the wheels (discs) have not changed over time.

Expected completion date: 11/25/09

Completed 11/4/09. A new certified Color disc (0-25 units) was requisitioned on 11/4/09. After receipt, the new color disc will be used as the primary comparison standard. At that time, the current Color disc (0-25 units) will be sent to the vendor for recertification and upon the return, the recertified disc will be used as the Third Party Check verification. The SOP will be revised upon the receipt of the recertified disc. Documentation attached.

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

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. (Sec. 5.5.5.2.2.1.j NELAC 2003)

Assessor: Zgrodnik, Tom

Comments: For ICP-AES, only one standard and a blank is being used for the calibration. Using a blank as a calibration point is not an acceptable practice.

Corrective Action: A reporting level standard will be analyzed each day samples are analyzed. In addition a midpoint calibration verification standard is also analyzed. The SOP will be revised to incorporate the changes.

Expected completion date: 11/15/09

Completed 11/24/09. The SOP was amended to require that a reporting level standard be analyzed each day samples are analyzed and a midpoint calibration verification standard also analyzed. Documentation attached.

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

Deficiency: 00D147 The laboratory does not have established acceptance criteria for continuing calibration verification analysis. (Sec. 5.5.5.10.d NELAC 2003)

Assessor: Denicola, Kathie

Comments: This refers to the need for appropriate acceptance criteria in all methods. (+/- 3 standard deviations is not appropriate.)

Corrective Action: No corrective action is required. The laboratory has method-specific acceptance criteria for continuing calibration acceptance, based on data collected within the laboratory. US EPA methods also allow that alternative acceptance limits may be appropriate. Throughout the NWQL, the use of ± 3 standard deviations is acceptable. NWQL analytical systems develop acceptance criteria on an annual basis, based on previous year's performance. Individual method requirements are defined in the method SOPs.

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

Deficiency: 555221FA Prior to the analysis of samples the zero point/single point calibration must be analyzed and the linear range must be established by analyzing a series of standards, one of which must be at the lowest quantitation level. (Sec. 5.5.5.2.2.1.h.1 NELAC 2003)

Assessor: Zgrodnik, Tom

Comments: For ICP-AES, the linear dynamic range has never been established by the analyst.

Corrective Action: The linear dynamic range will be determined and kept as part of the method history file. This determination will be repeated annually. The SOP will be revised to incorporate the changes based on the NYSDOH comment.

Expected completion date: 11/15/09

Completed 11/24/09. The SOP was amended to require a linear dynamic range be determined annually and kept as part of the method history file. Documentation attached (See previous documentation to deficiency 00D141).

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

Deficiency: 555221FC A standard corresponding to the lowest quantitation level must be analyzed with each analytical batch and must meet established acceptance criteria. (Sec. 5.5.5.2.2.1.h.3 NELAC 2003)

Assessor: Zgrodnik, Tom

Comments: For ICP-AES, a reporting limit standard is not analyzed with each analytical batch.

Corrective Action: A reporting level standard will be analyzed each day samples are analyzed. The SOP will be revised to incorporate the changes to the procedure based on this comment.

Expected completion date: 11/15/09

Completed 11/24/09. The SOP was amended to require that a reporting level standard is analyzed each day samples are analyzed. Documentation attached (See previous documentation to deficiency 00D141).

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

Deficiency: 555221FD The linearity is verified at a frequency established by the method and/or the manufacturer. (Sec. 5.5.5.2.2.1.hA NELAC 2003)

Assessor: Zgrodnik, Tom

Comments: For ICP-AES, the analyst indicated that the linear range has not been established. At a minimum, linearity should be established / verified on an annual basis.

Corrective Action: The linear dynamic range will be determined and kept as part of the method history file. This determination will be repeated annually. The SOP will be revised to incorporate the changes required in response to this NYSDOH comment.

Expected completion date: 11/15/09

Completed 11/24/09. The SOP was amended to require a linear dynamic range be determined annually and kept as part of the method history file. Documentation attached (See previous documentation to deficiency 00D141).