



Catherine E. Heigel, Director

Promoting and protecting the health of the public and the environment

MEMORANDUM

Date: February 9, 2016

To: Mr. Michael V. Peyton, Director
U. S. EPA Region IV

Through: Renee Shealy, Bureau Chief *RS*
Bureau of Environmental Health Services

Through: Sandra A. Flemming, Asst. Bureau Chief *SAF*
Bureau of Environmental Health Services

From: Mr. Micheal Mattocks, Director *MM*
Analytical and Radiological Environmental Services Division

Ms. Carol Smith, Director *CS*
Office of Environmental Laboratory Certification

Subject: Response to Drinking Water On-Site SC DHEC Laboratory and Laboratory Certification Program

This memorandum is a response to the findings and recommendations of the EPA during the audit of the South Carolina DHEC Columbia Laboratory and Laboratory Certification Program conducted August 25 - August 28, 2015. The responses are listed below and are numbered in accordance with the audit report. Your findings and recommendations have been written verbatim as stated in the audit report. The laboratory would like to thank EPA for the recommendations intended to improve the work being performed. However, it would be very helpful to the laboratory if EPA could provide funding to support these recommendations. Please let us know if you have questions or concerns regarding any of the responses.

3.0 GENERAL LABORATORY QUALITY, TRAINING AND DOCUMENTATION

- 3.1 **Recommendation:** Due to the varying timeframes used for the certification of equipment such as thermometers, pipettes, balances and autoclaves, it is recommended that the certification expiration date be recorded on the equipment.

Response: The laboratory ensures calibration of all equipment and apparatus as required. This includes accompanying documentation. However, the laboratory does see some potential of this recommendation enhancing overall QA goals and will where feasible and found to be efficient within the work processes of the lab, try to implement this practice.

- 3.2 **Recommendation:** The laboratory should consider entering PT samples and other QC samples such as MDL samples into the LIMS. This will create consistency across the laboratory with sample login and QC data management.

Response: The laboratory understands the intent of this recommendation. However, PT samples as received from the vendor and collected samples from the field are not "apples to apples". Samples collected in the field are whole volume samples. Proficiency Test (PT) samples are received from the vendor in varied volumes and containment (ampules, etc.) specific to a given parameter. Additionally, the paperwork as received from the vendor is specifically intended for the analyst with unique instructions specific for a given parameter. This changes the receiving logistics as compared to a sample collected in the field. Having a limited number of staff dedicated to sample receiving duties and the huge number of different parameters analyzed in our laboratory would make implementing this practice impossible. A general receiving duty would become too involved for this level of staff at this point in the analysis process. Method Detection Limit (MDL) studies would likewise create a challenge logistically. The MDL process would have to start with the analyst and result in a process where the analyst would have to work with the sample receiving group to have these study samples entered into LIMS. This would create a backward process opposite of how samples are normally received and ultimately result in an inefficient circular process which would only eat valuable time that analysts need to ensure that MDLs are performed and completed annually. The laboratory does not see this recommendation as providing anything which would enhance the overall QA or prove to be a benefit at this time.

- 3.3 **Recommendation:** The laboratory should consider purchasing disposable containers for VOA, SVOA and pesticide analyses. There is possible risk of contamination when reusing sample containers. However, if this is not economically feasible, the laboratory should designate groups of bottles cleaned together with a unique lot number and analyze one bottle to demonstrate lack of contamination.

Response: The laboratory has a defined system of cleaning sample collection containers in place that has proven to be adequate. The laboratory has never identified a contamination issue attributed to the sample containers. However, the laboratory will continue to evaluate its current process relevant to regulatory requirements and good laboratory practices accepted by the science community as advances are made.

4.0 SAMPLE RECEIVING, REPORTING AND STORAGE

4.1 Finding: The ID of the certified thermometer used in the sample receipt area was not documented in the receipt logbook.

Corrective Action: In accordance with Chapter IV, Sect. 8.1 of the Certification Manual, compliance monitoring data should be made defensible by keeping thorough and accurate records. Begin recording the unique identifier of the thermometer on sample receipt forms so that a link can be made to the thermometer calibration.

Response: The unique identifier for the thermometer was being recorded in the logbook. However, when a new book was started, this information was not carried over to the new book. The unique identifier is recorded and now documented on each page and traceable to each sample (see Attachment 1).

4.2 Recommendation: The laboratory indicated that samples were dropped off at a loading dock. It is recommended that samples be secured in a locked area when they are dropped off.

Response: Samples are dropped off at the lab at the loading dock area located at the rear entrance of the building. However, it is believed that this was misunderstood by the auditors that this meant on the dock itself. Samples are placed inside of the building at the loading dock area. This area is locked and at no time are samples in an unsecured location.

5.0 ORGANIC CHEMISTRY

General Organic Chemistry

The findings described under the General Organic Chemistry category are those that span over multiple methods.

5.1 Finding: Residual Chlorine checks were not performed for organic analysis to verify that the preservative added to the sample was sufficient to neutralize any chlorine that may have been present.

Corrective Action: The Drinking Water Certification Manual, Chapter IV, Section 6.3, requires that laboratories verify that samples' preservation was sufficient to neutralize residual chlorine. This should be performed for all associated methods, by a chlorine test strip, and documented in the sample preparation logbook or bench sheet.

Response: Item 6.3 of the Drinking Water Certification Manual does not state that residual chlorine checks must be performed. It states that: *Samples must be analyzed within the maximum holding times required by the method and references Table IV-6.* The laboratory is following this requirement as stated and exemplified in Table IV-6 with regard to preservatives, sample holding times, extract holding time and storage conditions, suggested sample size, and type of container for each parameter. The sample collection process is a shared endeavor where the lab provides bottles to the field staff. Samples which require dechlorinating agents are pre-prepared by the laboratory with the appropriate amount of dechlorinating agent already added as required by the methodology. The laboratory verifies appropriate preservation of samples as required by controlling this part of the sample collection process. Additionally, the Federal Register does not require that the residual chlorine be checked with test strips, but only requires that appropriate preservation as required for a given parameter be done. The laboratory has looked at a number of various versions of test strips to perform residual chlorine checks and this would incur a huge cost for the laboratory. The laboratory is of the opinion that since there is no document which clearly states or mandates that residual chlorine be checked, but only that the appropriate preservation be added, this finding should have been a recommendation. Each method in its "Sample Collection, Preservation, and Storage" section states the amount of dechlorinating agent to be added for appropriate elimination of potential chlorine presence.

Method Specific Organic Chemistry

The findings described under the method specific organic chemistry category are those that relate specifically to a particular method and each finding is referenced by a citation for requirements found in the method, Code of Federal Regulations, and/or Certification Manual.

EPA Method 515.3, Chlorinated Herbicides

- 5.2 **Finding:** The laboratory was raising the pH of the sample extract to greater than 12 as required in the method but this was not documented.

Corrective Action: All steps in a method or procedure must be documented during the process by the analyst which performs the step(s). See Chapter IV, Section 8.4 of the DW Cert Manual.

Response: This pH is performed as part of the procedural steps listed in the SOP. The laboratory will update the sample batch worksheet for this method, which are included with each data pack, to include a place for the recording of this pH adjustment (see Attachment 11). The laboratory however, does not agree that 8.4 implies or states that steps of a procedure must be documented as stated in the Finding 5.2. Part 8.4 of the Drinking Water Certification Manual states: *Data should be recorded in ink with any changes lined through such that original entry is visible. Changes need to be initialed and dated. The following information should be readily available:*

8.4.1 Laboratory and persons responsible for performing analysis;

8.4.2 Analytical Techniques/methods used

8.4.3 Date and time of analysis

8.4.4 Results of sample and quality control analyses

8.4.5 Calibrations and standards information

8.4.6 Analyst and technicians Initial Demonstration of Capability documentation should be kept on file as results of proficiency testing.

The laboratory is of the opinion that since there is no document which clearly states or mandates that this step needs to be documented as stated in Finding 5.2, this finding should have been a recommendation.

EPA Method 524.2, Volatile Organics

5.3 Finding: The laboratory was using a sample surrogate (4-BFB) which is also used to verify the tuning criteria for the Gas Chromatograph/Mass Spectrometer, to verify that the instrument was acceptable for further analysis.

Corrective Action: The method requires that the tune criteria check be performed with a blank standard which is spiked with the tune check criteria solution (4-BFB). A sample is inappropriate as there may be positive or negative interferences which would affect the results of the tune criteria verification. See Section 10.3.1 of EPA Method 524.2.

Response: The laboratory will implement the use of blanks for the BFB tune check. The SOP associated with this finding will be updated by March 1, 2016.

EPA Method 525.2, Semi-Volatile Organics

- 5.4 **Finding:** The acceptable limits designated in the laboratory SOP for the Laboratory Fortified Blank (LFB) and the Laboratory Fortified Matrix (LFM) were 37 - 68% recovery and 32 - 74% recovery respectively. These are outside of the acceptable limits required by the method (70 - 130% recovery).

Corrective Action: The method requires that the control charts be generated that establish an acceptable range for each analyte of interest. However, these limits cannot exceed the maximum allowable limits set in the method. See Chapter IV, Section 7.2.8 of the DW Cert Manual.

Response: The calculated limits are only used for the compound metribuzin. Metribuzin, as indicated in the method, is a poor responder when fortified with other compounds in this method as indicated in Section 13, Part 13.2.7 of EPA Method 525.2. It is also given a very conservative $\pm 50\%$ recovery for PT studies as an unregulated compound which also points toward this compound as a poor responder when fortified with other compounds. The laboratory feels that based on this and years of data where this compound has always been consistent with the methodology theory of a poor responder, it is unreasonable to expect this compound to meet the set limits. The more realistic approach would be to calculate limits which are reflective of the poor response of this compound. All other compounds analyzed for by this procedure by the laboratory do follow the EPA method set limits for quality control. The laboratory is currently and will continue to test the use of different consumables used in the procedure to improve the recovery of metribuzin. However, any guidance that can be provided would be appreciated by the laboratory regarding improving recovery for this compound.

EPA Method 549.2 Diquat and Paraquat

- 5.5 **Finding:** The pH meter used to determine appropriate pH measurements during the sample preparation was calibrated before each use but was not documented.

Corrective Action: All steps in a method or procedure must be documented during the process by the analyst which performs the step(s). See Chapter IV, Section 8.4 of the DW Cert Manual.

Response: The pH meter calibration was documented, although the lot numbers of each buffer solution were not included. We have updated the batch sheet to include the buffer lot numbers (see Attachment III).

EPA Method 551.1, Trihalomethanes (THMs)

- 5.6 Finding:** Section 6.11 of the laboratory SOP describes the Laboratory Performance (LPC). It notes that four compounds (not listed) are used to determine the performance of the instrument. However, the SOP has a photocopy of the LPC table that is in the method and it lists seven compounds that must be analyzed for various performance acceptance criteria before the analysis of samples can begin. Data packages verify that only four compounds, Lindane, Hexachlorocyclopentadiene, Bromodichloromethane and Trichloroethylene are checked. The remaining three, Bromacil, Alachlor, and Endrin are not evaluated.

Corrective Action: The method requires that all seven compounds must pass various acceptance criteria for the instrument to be properly calibrated. See section 7.5 and table 7 of the method.

Response: The laboratory is only analyzing the trihalomethane compounds with this procedure. EPA Method 551.1 states in section 7.5.1 that the three compounds; bromacil, alachlor, and endrin can be omitted from the LPC if not analyzing for the high molecular weight compounds. The LPC table copied into the laboratory's SOP will be updated to exclude these as to prevent confusion. The associated SOP will be updated by March 1, 2016. Please see below the referenced excerpt from EPA Method 551.1.

7.5.1 If a laboratory is not conducting analyses for the high molecular weight pesticides and herbicides, a modified LPC may be prepared. This modified LPC can omit the endrin analyte breakdown component as well as the resolution requirement for bromacil and alachlor under column performance. In addition, substitute analytes in place of lindane for the sensitivity check and hexachlorocyclopentadiene for chromatographic performance can be selected. These substitute compounds must meet the same criteria as listed in Table 7 with the concentration for sensitivity check near the substitute analyte's EDL and the concentration for chromatographic performance near 50 times the substitute analyte's EDL. The column performance criteria for resolution between bromodichloromethane and trichloroethylene cannot be modified.

- 5.7 Finding:** Sample extracts were stored in the same freezer as calibration standards.

Corrective Action: Chapter IV, Section 2 of the DW Lab Cert Manual requires that "The analytical and sample storage areas should be isolated from all potential sources of contamination." Sample extracts should be relocated to an area free of potential contamination.

Response: The laboratory does not analyze samples for this method, but only maintains primacy. There were no samples analyzed during this evaluation period, only PT samples. Moving forward the laboratory will provide a separate freezer unit for extracts for this analysis.

- 5.8 Recommendation:** The laboratory should consider purchasing disposable sample containers for VOA, SVOA, and pesticide analyses. The risk of contamination is possible when reusing sample containers. However, if this is not economically feasible, the laboratory should designate groups of bottles cleaned together with a unique lot number and analyze one bottle to demonstrate lack of contamination.

Response: (Same response as provided for 3.3) - The laboratory has a defined system of cleaning sample collection containers in place that has proven to be adequate. The laboratory has never identified a contamination issue attributed to the sample containers. However, the laboratory will continue to evaluate its current process relevant to regulatory requirements and good laboratory practices accepted by the science community as advances are made in science.

6.0 METALS AND INORGANIC CHEMISTRY

General Inorganic Chemistry

The findings described under the general chemistry category span over multiple methods.

- 6.1 Finding:** The metals and general chemistry analysts are capturing most of the information on preparation logs needed to trace standards back to the manufacturer and on run logs that allow for the reconstruction of the data. However, there were some gaps in documentation and traceability that prohibit the unambiguous reconstruction of data and analytical conditions. Examples include:
- a. The preparation of the matrix spike was not documented for nearly all areas of inorganic chemistry. The spike ID, volume, and final volume needs to be documented.
 - b. Hot blocks and thermometers used for metals analysis did not have unique IDs for analyst to definitively determine calibration status.
 - c. For metals analysis by EPA Method 200.7, the working QCS prepared on 07/15/15 was made from High Purity standards, but AccuStandard was hardcoded (typed) into the preparation logbook. Auto-pipettes used for dilution did not contain a unique ID for analyst to definitively determine calibration status. The unique ID of ICP (B) was missing from analytical run logs.
 - d. For metals analysis by EPA Method 200.8, the preparation of working QC solutions was not documented.
 - e. For metals analysis by EPA Method 200.9, the balance ID was not documented for the preparation of palladium/magnesium nitrate matrix modifier. The exact weight shown on the balance was not recorded, only the nominal weight required, which was hardcoded (typed) into the preparation logbook.

- f. For mercury analysis by SM 3112B, the preparation of reagents, QC standards, and the matrix spike was not documented. The balance ID was not documented in preparation logbooks. The addition of reagents for sample pretreatment was not documented for each sample batch. The heating of samples for 2 hrs at 90-95 °C was not documented for each sample batch. The certified thermometer ID used for monitoring temperature was not recorded.
- g. For disinfection by product analysis by EPA Method 300.1, the preparation of the working Quality Control Solution (QCS) was not documented.
- h. For cyanide analysis by SM 4500 CN E, the distillation logbook did not document the temperature and heating time.
- i. For fluoride analysis by Lachat QuikChem 10-109-12-2-A, the balance ID was not documented on the preparation log, and only a nominal weight was being recorded, instead of all places on the balance.

Corrective Action: In accordance with Chapter IV, Sect. 8.1 of the Certification Manual, compliance monitoring data should be made defensible by keeping thorough and accurate records. Also, in accordance with Chapter IV, Sect. 8.5 of the Certification Manual, adequate information should be available to allow the auditor to reconstruct the final results for compliance samples and PT samples. A procedure should be developed to ensure that samples, reagents, calibration standards, and QC standards are unambiguously traceable from data system printouts, run logs, sample preparation logs, and standard preparation logs back to the original sample containers or chemical lot numbers. Sample preparation logs should document all measurements and required procedures performed in the utilized method such as interference checks, heating at a certain temperature, and weighing.

Response: See below the laboratory's response to the itemized Finding 6.1 (a-i).

**Please Note, each itemized sub-finding under Finding 6.1 has been intentionally presented a second time below, above the response for convenience of the reader.*

***Please Note, the nomenclature for reagent lot numbers are as follows: analyst initials, date of preparation, and reagent acronym. As an example, for a Mercury Rinse solution prepared on 01/15/16 by John Black would be; JB011516MR. This nomenclature applies only to the inorganic chemistry metals laboratory.*

Finding 6.1(a): The preparation of the matrix spike was not documented for nearly all areas of inorganic chemistry. The spike ID, volume, and final volume need to be documented.

Response 6.1(a): The lot number of the spike is captured either on the batch sheet or solution preparation log depending on the analysis. However, we did not have a sheet for the mercury analyses. A sheet for this area has been created (see Attachments IV-A through IV-D). The laboratory will add the additional

information to the batch sheet. We are temporarily handwriting the required information. All forms will be updated by March 1, 2016.

Finding 6.1(b): Hot blocks and thermometers used for metals analysis did not have unique IDs for analyst to definitively determine calibration status.

Response 6.1(b): The laboratory has added unique identifiers to the digestion book for the hot block. However, thermometers in our laboratory have always had unique identifiers (see Attachment V). The laboratory will add the additional information to the forms. We are temporarily handwriting the required information. All forms will be updated by March 1, 2016.

Finding 6.1(c): For metals analysis by EPA Method 200.7, the working QCS prepared on 07/15/15 was made from High Purity standards, but AccuStandard was hardcoded (typed) into the preparation logbook. Auto-pipettes used for dilution did not contain a unique ID for analyst to definitively determine calibration status. The unique ID of ICP (B) was missing from analytical run logs.

Response 6.1(c): The laboratory has corrected the preparation logbook for the 07/15/15 standard which was incorrectly recorded. Auto-pipettes used for dilution will use their serial numbers as the unique identifier to associate the pipette used with its calibration status. The ICP instrument used will be captured on the analytical run logs (see Attachments VI-A and VI-B). The laboratory will add the additional information to the logbook and forms. We are temporarily handwriting the required information. The forms and logbook will be updated by March 1, 2016.

Finding 6.1(d): For metals analysis by EPA Method 200.8, the preparation of working QC solutions was not documented.

Response 6.1(d): The laboratory feels the auditor may have not been clear regarding the process for the preparation of standards and QC. The QC solutions are documented and were shown to the auditors (see Attachment VII). Please note that the dark spots on the document are not strike-overs, but are the result of converting the documents (containing gray-shaded areas) to JPEG files.

Finding 6.1(e): For metals analysis by EPA Method 200.9, the balance ID was not documented for the preparation of palladium/magnesium nitrate modifier. The exact weight shown on the balance was not recorded, only the nominal weight required, which was hardcoded (typed) into the preparation logbook.

Response 6.1(e): The laboratory has updated the balance logbook and the balance ID is now being documented in the preparation logbook. The laboratory will now record the exact weight. An updated form will be implemented to better accommodate this request. Attachment VIII-B will be the new form used by the laboratory, (see Attachments VIII-A and VIII-B). The laboratory will add the additional information to the logbook. We are temporarily handwriting the

required information. The logbook will be updated by March 1, 2016.

Finding 6.1(f): For mercury analysis by SM3112B, the preparation of reagents, QC standards, and the matrix spike was not documented. The balance ID was not documented in preparation logbooks. The addition of reagents for sample pretreatment was not documented for each sample batch. The heating of samples for 2 hours at 90-95 °C was not documented for each sample batch. The certified thermometer ID used for monitoring temperature was not recorded.

Response 6.1(f): The preparation of reagents, QC standards, and matrix spikes will be more clearly documented. The balance ID is now documented in the preparation logbook (see Attachments IX-A through IX-J). The laboratory has temporarily edited the forms by handwriting the required information in the established form documents. A new form for mercury has been included to capture the heating times for samples. All other associated forms to this finding will be updated. We are temporarily handwriting the required information. The forms will be updated by March 1, 2016. It should be noted, that Attachments IX-H through IX-J will appear to have dark spots on first line of entered information. This is not a strike-over but the result of converting the original document to a JPEG document and the gray shadow on the original document.

Finding 6.1(g): For disinfection by product analysis by EPA Method 300.1, the preparation of the working Quality Control Solution (QCS) was not documented.

Response 6.1(g): The QCS will be documented in the QCS logbook (see Attachment X).

Finding 6.1(h): For cyanide analysis by SM 4500 CN E, the distillation logbook did not document the temperature and heating time.

Response 6.1(h): The laboratory will document the temperature and heating time in the distillation logbook (see Attachment XI). The laboratory will add the additional information to the logbook. We are temporarily handwriting the required information. The logbook will be updated by March 1, 2016.

Finding 6.1(i): For fluoride analysis by Lachat QuikChem 10-109-12-2-A, the balance ID was not documented on the preparation log, and only a nominal weight was being recorded, instead of all places on the balance.

Response 6.1(i): The laboratory will now record the balance ID and the exact weight (see Attachment XII).

6.2 Finding: Residual Chlorine checks were not performed for inorganic analysis by EPA Method 200.7 and 200.8, and SM3112B to verify that the preservative added to the sample was sufficient to neutralize any chlorine that may have been present.

Corrective Action: The Drinking Water Certification Manual, Chapter IV, Section 6.4, requires that laboratories verify that samples' preservation was sufficient to neutralize residual chlorine. This should be performed for all associated methods, by a chlorine test strip, and documented in the sample preparation logbook or bench sheet.

Response: Residual Chlorine checks are not required for these methods and Section 6.4 does not state that residual chlorine checks must be performed. Section 6.4 states: *There must be strict adherence to correct sampling procedures, sample handling, complete identification of the sample, and prompt transfer of the sample to the laboratory when required by the method. When the laboratory is not responsible for sample collection and transport, it must verify that the paperwork, preservatives, containers, and holding times are correct as required by the methods or reject the sample. The rejection criteria should (EPA Order 5360.1) be documented in writing. Methods 200.7, 200.8, and SM3112B are preserved with the appropriate acid in the field and the pH of the sample is checked once the sample is received by the laboratory verifying the sample has been acidified accordingly. The laboratory does not feel this should have been a finding as residual chlorine does not apply to any of the inorganic parameters as such.*

SM 3112B Mercury

6.2 Finding: The laboratory not analyzing a minimum reporting level (MRL) check standard every time the instrument was calibrated.

Corrective Action: In accordance with Standard Methods 3020 B Sect. 2e governing method QC requirements, begin analyzing a QC standard at the minimum reporting level every new calibration.

Response: The laboratory will begin analyzing a Minimum Reporting Level (MRL) standard each time the instrument is calibrated. The associated SOP will be updated by March 1, 2016.

11.0 FINDINGS AND RECOMMENDATIONS - MICROBIOLOGY

11.1 Finding: There was no record of contents or analyst's initials each time the autoclave was used.

Corrective Action: The following information should be recorded each time the autoclave is used: date, contents, sterilization time and temperature, total time in the autoclave, and analyst's initials. Refer to Chapter 5, Section 3.5.3 of the Certification Manual.

Response: Autoclave services for the microbiology laboratory are provided by

the Media, Reagent, and Glassware section in the Bureau of Laboratories. The Media, Reagent, and Glassware section has updated their records to reflect autoclave operator initials, process date, and who services were provided to. Each autoclave strip will reflect this information (see Attachment XIII).

11.2 Finding: All drinking water samples were not shaken 25 times before analysis.

Corrective Action: All water samples should be shaken vigorously at least 25 times before analyzing. The laboratory should add this procedure to their SOP. Refer to Chapter 5, Section 5.1.3 of the Certification Manual.

Response: The microbiology laboratory will shake samples 25 times before analysis. The associated SOP will be updated by March 1, 2016.

11.3 Finding: The instrument services laboratory had questionable readings when using the UV light meter. There were no standard procedures on how to accurately take readings each time, therefore the laboratory needs to create a SOP for the UV light meter.

Corrective Action: The laboratory should have a Standard Operating Procedure available, pertaining to its own calibration of equipment or supplies. Refer to Chapter 5, Section 7.1 of the Certification Manual.

Response: Instrument Services performs this calibration service for the laboratory. The Instrument Service group has revised their quarterly UV light check SOP. The SOP has been revised requiring the technician to allow the UV lamp to warm up for a minimum of 10 minutes before taking a reading. Once the lamp has warmed up, the technician will place the UV sensor directly on the bulb in the middle and then take the reading. This will result in uniform readings.

11.4 Finding: During the review of the pre-audit data packages, it was noted for:
. EM15-PT - The wrong expiration date for Simplate Multi-dose was recorded.
. EM15-PT 6F - The wrong 48 hour incubation date for Simplate was recorded.

Corrective Action: The laboratory should peer review data to ensure that all data reported by the laboratory meets the required quality assurance and regulatory criteria. Refer to Chapter 5, Section 1.1 of the Certification Manual.

Response: No response required, as noted in the report, this finding was corrected at the time of the audit.

11.5 Recommendation: The laboratory should start recording/documenting the pre-warming incubation times for Colilert-18 samples.

Response: The required pre-warm incubation time for colilert-18 is stated in the laboratory's SOP. The laboratory feels no other documentation is necessary.

- 11.6 Recommendation:** The laboratory should follow the manufacturer's recommendation concerning the subculture of commercial stock cultures.

Response: The laboratory is currently using one swab every six months to maintain stock cultures. The manufacturer recommends a swab be done each month. Following the manufacturer's recommendation would result in substantial cost for the laboratory. This recommendation will not be implemented. The laboratory will continue to evaluate this recommendation.

- 11.7 Recommendation:** The laboratory should take the actual pH reading of any commercial reagents or media if the manufacturer's certificate of analysis (COA) lists a range instead of a specific pH for that lot number.

Response: The laboratory uses commercial media that is accompanied with a COA. Efforts will be made to purchase media that has a specific pH listed rather than a pH range, but this may not be possible in all cases. QC is run on all media before it is used and if the QC passes, the media should be fine for use.

- 11.8 Recommendation:** When analyzing annual Proficiency Testing (PT) samples, EPA auditors recommend occasionally alternating between Colilert and Colilert 18 samples since the laboratory is certified for both.

Response: The laboratory will implement this practice. The associated SOP will be updated by March 1, 2016.

- 11.9 Recommendation:** The laboratory should modify the Calibration of Thermometers SOP to include:
- a. Allowing time for equilibration before comparing the NIST thermometer to the laboratory thermometer.
 - b. Checking several degree increments instead of just one (i.e at, below and above the temperature at which the thermometer will be used) when verifying thermometers to obtain a more accurate correction factor.

Response:

- a. The Instrument Services laboratory has revised the thermometer calibration check SOP to allow a 1 hour equilibration time before checking the NIST thermometer against the lab thermometer being checked.
- b. Due to limited staff and the large volume of thermometers checked, the instrument services laboratory will only check the thermometer at one increment.

14.0 EVALUATION OF THE SOUTH CAROLINA DRINKING WATER LABORATORY CERTIFICATION PROGRAM

14.5 Recommendation: It is recommended that the Certification program retain the auditor's checklists, which contain the original observations, and that they be available for review. This is noted in Chapter 11, Section 15 of the EPA's "Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth Edition".

Response: The Office of Environmental Laboratory Certification will ensure that records for the on-site laboratory assessments and certification program reviews be maintained in an easily accessible location for a period of six years to include the last two on-site audits. This includes records/correspondence used to determine compliance with the requirements in the certification manual. Records may include checklists, corrective action reports, final reports, certificates, PT study results and related documents. These records will be maintained as paper documents or electronically in an easily accessible location.

Table 4 of the on-site evaluation report documented the incorrect numbers of certified drinking water laboratories in South Carolina. An updated Table 4 has been provided with the number of certified laboratories.

Table 4 - South Carolina Certified Laboratories

State	# Chemistry Laboratories Certified in State (# Out of State)	# Microbiology Laboratories Certified in State (# Out of State)	# Radiochemistry Laboratories Certified in State (# Out of State)	# <i>Cryptosporidium</i> Laboratories Certified in State (# Out of State)
SC	187 (5)	117 (6)	1 (3)	0 (5)

Attachment 1

IR Thermometer
 Model 21521, SN: 1771
 Calibrated Thermometer 609 5.270
 TS, HCSL, WST 50 105 1, Corr. Factor 0.0
 To Use Unless Otherwise Noted

Date	Analyst	IR Thermometer Temperature (°C)	Calibrated Thermometer Temperature (°C)
12/17/15	Chin Lee	3.9	3.8
12/18/15	Chin Lee	4.1	4.0
12/21/15	Amber Leonard	4.2	4.2
12/22/15	Chin Lee	4.2	4.1
12/23/15	Chin Lee	4.2	4.0
12/29/15	Chin Lee	4.2	4.1
12/30/15	Chin Lee	4.3	4.2
12/31/15	Chin Lee	4.5	4.5
1/4/16	Amber Leonard	4.2	4.0
1/5/16	Chin Lee	4.4	4.4
1/6/16	Eric Soren	4.7	4.4
1/7/16	Eric Soren	4.1	4.2
1/8/16	Amber Leonard	4.6	4.4
1/11/16	Amber Leonard	4.7	4.5
1/12/16	Chin Lee	4.5	4.5
1/12/16	Chin Lee	4.5	4.5
1/12/16	Chin Lee	4.5	4.5
1/15/16	Chin Lee	4.5	4.5
1/19/16	Chin Lee	4.5	4.5
1/20/16	Eric Soren	4.7	4.6
1/21/16	Eric Soren	4.5	4.5
1/22/16	Eric Soren	4.6	4.6
1/25/16	Amber Leonard	4.6	4.7

The temperature readings of the IR and calibrated thermometers must agree within 0.5°C.

Comments: _____

DHEC 2834 (06/2010)

Attachment II



SC DHEC/ARESD – Organic Sample Batch Worksheet

Acid Herbicides IX-E-3(n) EPA 515.3

Date: _____
Chemist: _____
QC#: _____

Standards Used (Lot Number)

Surrogate Spike: _____
Analyte Spike: _____
Internal Standard Spike: _____

Sample Identification pH>12

- 1. _____
- 2. _____
- 3. _____
- 4. _____
- 5. _____
- 6. _____
- 7. _____
- 8. _____
- 9. _____
- 10. _____
- 11. _____
- 12. _____
- 13. _____
- 14. _____
- 15. _____
- 16. _____
- 17. _____
- 18. _____
- 19. _____
- 20. _____
- 21. _____
- 22. _____
- 23. _____
- 24. _____
- 25. _____
- 26. _____
- 27. _____
- 28. _____
- 29. _____

Reagents Used (Lot Number)

4N NaOH: _____
H₂SO₄: _____
CuSO₄·5H₂O: _____
Na₂SO₄: _____
Diazald: _____
Carbitol: _____
Ethyl Ether: _____
37% KOH: _____
Silica Gel: _____
Florisil: _____
Acidified Na₂SO₄: _____

Solvents Used (Lot Number)

MtBE: _____
Ethyl Ether: _____
DI Water System Used: _____

Batch Comments:

Verification Analyst: _____ Date: _____

This Form is to be included in Data Pack
Batch Worksheet IX-E-3(n) Acid Herbicides 2016 01 12.doc

Attachment III



SC DHEC/ARESD – Organic Sample Batch Worksheet

Diquat IX-E-5(d) EPA 549.2

Date: _____
 Chemist: _____
 QC#: _____

Standards Used (Lot Number)
 Analyte Spike: _____

<u>Sample Identification</u>	<u>pH</u> <u>Initial</u>	<u>pH</u> <u>Adj.</u>
1. _____	_____	_____
2. _____	_____	_____
3. _____	_____	_____
4. _____	_____	_____
5. _____	_____	_____
6. _____	_____	_____
7. _____	_____	_____
8. _____	_____	_____
9. _____	_____	_____
10. _____	_____	_____
11. _____	_____	_____
12. _____	_____	_____
13. _____	_____	_____
14. _____	_____	_____
15. _____	_____	_____
16. _____	_____	_____
17. _____	_____	_____
18. _____	_____	_____
19. _____	_____	_____
20. _____	_____	_____
21. _____	_____	_____
22. _____	_____	_____
23. _____	_____	_____
24. _____	_____	_____

3M C8 Disks (Lot Number): _____

Reagents Used (Lot Number)
 10% NaOH: _____
 10% HCl: _____
 Cond. Solution A: _____
 Cond. Solution B: _____
 Disk Eluting Solution: _____
 Ion-pair Concentrate: _____
 Mobile Phase: _____

Solvents Used (Lot Number)
 Methanol: _____

Batch Comments:

pH Meter Calibration:
 Buffer 4: _____ Lot# _____
 Buffer 7: _____ Lot# _____
 Buffer 10: _____ Lot# _____
 Slope: _____

Verification Analyst: _____ Date: _____

This Form is to be included in Data Pack
 Batch Worksheet IX-E-5(d) Diquat 2015 09 08

Attachment IV-A



Checklist for Nitrite in Water Analysis
Analytical & Radiological Environmental Services Division
Automated Analysis Section

THE DATA SET SUBMITTED ADHERES TO THE FOLLOWING:

- Calibrated with at least 5 standards producing a curve with an R value of 0.995 or higher. _____
- A LRB, LFB, MRL and QCCS were analyzed after the calibration curve and prior to any samples. The results for each fell within the established acceptance criteria. _____
- A LFM and duplicate LFM were analyzed with each set of 10 samples or less. _____
- Prepared by adding 100 μ L of 100 mg/L stock solution to a 25 mL volumetric flask and brought to volume with the sample. Recoveries (Accuracy) and RPD (Precision) fell within the established control limits. _____
- A Blank and Calibration Check Standard (CCC) was analyzed with each set of 10 samples. _____
- The integration of peaks appeared consistent and reasonable. _____

REAGENTS USED IN THIS BATCH:


REAGENT	REAGENT #	DATE MADE:	EXP.
BUFFER	RAA-16-20	01/13/16	28 DAYS
COLOR REAGENT	RAB-16-13	01/22/16	28 DAYS

STANDARDS USED IN THIS BATCH:

CONC. (mg/L)	S= Stock I=Intermediate W=Working			DATE MADE:	EXP.
	TYPE	STANDARD #			
100	S	S-NO2-	16-220	01/28/16	5 DAYS
10	I	S-NO2-	16-212	01/14/16	1 DAY
2.0	W	S-NO2-	16-213	01/14/16	1 DAY
1.5	W	S-NO2-	16-214	01/14/16	1 DAY
1.0	W	S-NO2-	16-215	01/14/16	1 DAY
0.5	W	S-NO2-	16-216	01/14/16	1 DAY
0.1	W	S-NO2-	16-217	01/14/16	1 DAY
0.05	W	S-NO2-	16-218	01/14/16	1 DAY
0.02	W	S-NO2-	16-219	01/14/16	1 DAY
QCCS # 163	S	ERA Lot #S210-695		11/05/15	11/30/2016

Batch analyzed by: _____ Date: _____
 Results entered in LIMS and QC documented by: _____ Date: _____
 Batch verified by: _____ Date: _____
 Results validated in LIMS and QC verified by: _____ Date: _____

Attachment IV-B

		Preparation of QCS High Line Working Solution Inorganic Section Analytical & Radiological Environmental Services Division			
<input type="checkbox"/> Intermediate <input checked="" type="checkbox"/> Working					
DI Water: MORM324			Lot #: FE122115 QCS HL		
Date Prepared: 7/2/15			Prepared By: FE		
Vendor of Parent Solution: High-Purity Standards					
Compound	Item #	Lot #	Stock Conc. (mg/L)	Amt. Used (mL)	Final Conc. (mg/L)
1. Sodium (Na)	INFCS-7	1429612	1000	10	10.0
2. Magnesium (Mg)	INFCS-7	1429612	1000	10	10.0
3. Aluminum (Al)	INFCS-7	1429612	1000	10	10.0
4. Calcium (Ca)	INFCS-7	1429612	1000	10	10.0
5. Iron (Fe)	INFCS-7	1429612	1000	10	10.0
			Pipette	EHS3805	
In addition to the above listed aliquot of standards, 10 mL of concentrated nitric acid and 50 mL of concentrated hydrochloric acid were added to a 1000 mL class A volumetric flask containing about 500 mL of deionized water. The solution was then diluted to volume with deionized water.					
Nitric Acid Lot #: 1114070			Hydrochloric Acid Lot #: 4114100		
Page _____					


Attachment IV-C

D H E C		Preparation of Silver Spike Stock			
		Inorganic Section			
Analytical & Radiological Environmental Services Division					
<input type="checkbox"/> Intermediate <input checked="" type="checkbox"/> Working		Lot #: FE 021015DAg			
Date Prepared: 07/01/15		Prepared By: FE			
Vendor for Silver is UltraScientific.		DI Water: MORM324			
Compound	Stock Lot #	Stock Conc. (mg/L)	Amt. Used (mL)	Final Vol. (mL)	Final Conc. (mg/L)
Silver (Ag)	K00662-08/31/10	10,000	1.0	1000	10
In addition to the above listed aliquot of standard, 50 mL of 1:1 nitric acid was added to the 1000 mL class A volumetric flask containing about 500 mL of deionized water. The solution was then diluted to volume with deionized water.					
1:1 Nitric Acid Lot#: FE 020315N		Remake annually or as needed.			

<input type="checkbox"/> Intermediate <input checked="" type="checkbox"/> Working		Lot #:			
Date Prepared:		Prepared By:			
Vendor for Silver is UltraScientific.		DI Water: MORM324			
Compound	Stock Lot #	Stock Conc. (mg/L)	Amt. Used (mL)	Final Vol. (mL)	Final Conc. (mg/L)
Silver (Ag)		10,000	1.0	1000	10
In addition to the above listed aliquot of standard, 50 mL of 1:1 nitric acid was added to the 1000 mL class A volumetric flask containing about 500 mL of deionized water. The solution was then diluted to volume with deionized water.					
1:1 Nitric Acid Lot#:		Remake annually or as needed.			

<input type="checkbox"/> Intermediate <input checked="" type="checkbox"/> Working		Lot #:			
Date Prepared:		Prepared By:			
Vendor for Silver is UltraScientific.		DI Water: MORM324			
Compound	Stock Lot #	Stock Conc. (mg/L)	Amt. Used (mL)	Final Vol. (mL)	Final Conc. (mg/L)
Silver (Ag)		10,000	1.0	1000	10
In addition to the above listed aliquot of standard, 50 mL of 1:1 nitric acid was added to the 1000 mL class A volumetric flask containing about 500 mL of deionized water. The solution was then diluted to volume with deionized water.					
1:1 Nitric Acid Lot#:		Remake annually or as needed.			

<input type="checkbox"/> Intermediate <input checked="" type="checkbox"/> Working		Lot #:			
Date Prepared:		Prepared By:			
Vendor for Silver is UltraScientific.		DI Water: MORM324			
Compound	Stock Lot #	Stock Conc. (mg/L)	Amt. Used (mL)	Final Vol. (mL)	Final Conc. (mg/L)
Silver (Ag)		10,000	1.0	1000	10
In addition to the above listed aliquot of standard, 50 mL of 1:1 nitric acid was added to the 1000 mL class A volumetric flask containing about 500 mL of deionized water. The solution was then diluted to volume with deionized water.					
1:1 Nitric Acid Lot#:		Remake annually or as needed.			

		Metals Digestion Workbook for Water, Sediment, and Fish Analytical & Radiological Environmental Services Division							8
Beaker Number	Sample Number	Charge Code	Weight (g)	Parameters Requested	Beaker Number	Sample Number	Charge Code	Weight (g)	Parameters Requested
1	RB		50ml						
2	LEPZM LFB(HS)								
3	LFB(F)								
4	ADG8309	WSR							
5	Duplicate								
6	ADG8333	WSR							
7	LPM(HS)								
8	ADG8778	WSR							
9	LFM(F)								
10	ADG7857	WSR							
11	68315								
12	68536								
13	69168								
	Digestion blank	FT677			Pipette	HH93536			Thermometer: B3.768
						HH16897			

Date: 01/21/16
 Matrix: H₂O
 Analyst: JF
 Lot # HCl: F2D114164
 Lot # HNO₃: R6D11716N
 Lot # of Digestion Tubes: 150436-1A-536-140
 Lot # of Spike Stocks: F2D2415165 (2.5ml) R6D10116561 (5D₂L)
 Initial Digestion Temperature: 91.2 °C
 Final Digestion Temperature: 92.5 °C
 Start Time: 11:00 Ending Time: 4:00
 Digestion SOP Used: IX-D-1(b)



ICAP 6500B
FY 2016
Book 1



Analytical & Radiological Environmental Services Division

I.C.A.P Water Supply Workbook

Date: 07/15/15 → 10/29/15

Attachment VI-B

		Preparation of QCS High Line Working Solution Inorganic Section Analytical & Radiological Environmental Services Division			
<input type="checkbox"/> Intermediate <input checked="" type="checkbox"/> Working					
DI Water: MQRM324		Lot #: FE071515 DCSHL			
Date Prepared: 07/15/15		FED22515		Prepared By: [Signature]	
Vendor of Parent Solution: AccuStandard High Purity Standards					
Compound	Item #	Lot #	Stock Conc. (mg/L)	Amt. Used (mL)	Final Conc. (mg/L)
1. Sodium (Na)	INFCS-7	1429612	1000	10	10.0
2. Magnesium (Mg)	INFCS-7	1429612	1000	10	10.0
3. Aluminum (Al)	INFCS-7	1429612	1000	10	10.0
4. Calcium (Ca)	INFCS-7	1429612	1000	10	10.0
5. Iron (Fe)	INFCS-7	1429612	1000	10	10.0
<p>In addition to the above listed aliquot of standards, 10 mL of concentrated nitric acid and 50 mL of concentrated hydrochloric acid were added to a 1000 mL class A volumetric flask containing about 500 mL of deionized water. The solution was then diluted to volume with deionized water.</p>					
Nitric Acid Lot #: 1115010			Hydrochloric Acid Lot #: 4114121		
Page 1					

Attachment VII

Preparation ICP/MS Working Standard Solutions							
Analytical & Radiological Environmental Services Division							
<input type="checkbox"/> Intermediate		<input checked="" type="checkbox"/> Working					
Date Prepared: 4-12-14		Prepared By: JH		Remake Daily			
DI Water ID: MORM324		1+1 Nitric Acid lot#: TD0724150PN					
Compound	Stock Conc. (ug/L) Se 5x Conc	Amount used (ml)	Volume made (ml)	Final Conc. ug/l Se 5x Conc.	ml 1+1 Nitric Acid	Standard Lot #	
0.5 ppb (Ultra Std A & B)	10000	0.0025	50	0.5	1.0	DH0012150.SWS	
1.0 ppb (Ultra Std A & B)	10000	0.005	50	1	1.0	DH0012151.SWS	
10 ppb (Ultra Std A & B)	10000	0.05	50	10	1.0	DH00121510.SWS	
20 ppb (Ultra Std A & B)	10000	0.1	50	20	1.0	DH00121520.SWS	
100 ppb (Ultra Std A & B)	10000	0.5	50	100	1.0	DH001215100WS	
200 ppb (Ultra STD A & B)	10000	1	50	200	1.0	DH001215200WS	
Calibration Blank (Reagent Blank)	N/A	N/A	50	0	1.0	DH001215WSCB	
QCS Part A (Part B)(LFB) (AccuStandard)	10000	0.25(0.25)	50	50	1.0	DH001215WSQCS	
ICS Part A(Part B) (AccuStandard)	See Chart	0.15(0.010)	50	See Chart	1.0	DH001215WSICS	
	Ultra/AccuStandard Conc. (ug/L)	Ultra Lot/Exp. Date	AccuStandard Lot/Exp. Date	AccuStandard Interferent Check	Conc. (ppm)	AccuStandard Lot/Exp. Date	Final Conc. (ug/L)
Std. A: 10000 ug/L Se 5x Conc	10	T00079	212063118-02	Part A		21303902-01	
1. Aluminum (Al)	10	02/28/18	7/16	1. Aluminum (Al)	1000	2/16	300
2. Antimony (Sb)	10			2. Carbon (C)	2000		600
3. Arsenic (As)	10			3. Chlorine (Cl)	3000		900
4. Beryllium (Be)	10			4. Chlorine (Cl)	18000		4500
5. Cadmium (Cd)	10			5. Iron (Fe)	2500		750
6. Chromium (Cr)	10			6. Magnesium (Mg)	1000		300
7. Cobalt (Co)	10			7. Molybdenum (Mo)	20		60
8. Copper (Cu)	10			8. Phosphorus (P)	1000		300
9. Lead (Pb)	10			9. Potassium (K)	1000		300
10. Manganese (Mn)	10			10. Sodium (Na)	2500		750
11. Molybdenum (Mo)	10						
12. Nickel (Ni)	10						
13. Selenium (Se)	50			1. Arsenic (As)	10	212025002-02	10
14. Vanadium (V)	10			2. Cadmium (Cd)	10	2/16	10
15. Barium (Ba)	10			3. Carbon (C)	20		20
16. Uranium (U)	10			4. Chlorine (Cl)	20		20
17. Vanadium (V)	10			5. Copper (Cu)	20		20
18. Zinc (Zn)	10			6. Magnesium (Mg)	20		20
				7. Nickel (Ni)	20		20
				8. Selenium (Se)	10		10
				9. Silver (Ag)	20		20
Std. B		CL-2515	212083074-02	10. Vanadium (V)	20		20
19. Silver (Ag)	10	07/31/17	6/16				
20. Barium (Ba)	10						

In addition to the above listed aliquots of standards, 1.0 ml of 1+1 ultra high purity nitric acid was added to the 50 ml class A Digestion tube containing about 20 ml of deionized water. The solution was then diluted to volume with deionized water. Note: In Ultra Scientific Standards for calibration and AccuStandard Standards for QCS/MCV Se is at 5 times the concentration of the other analytes.

Attachment VIII - A



Palladium/Magnesium Matrix Modifier

Metals Section
Analytical and Radiological Environmental Services
Division

Alfa Aesar Palladium Lot # /exact weight (300 mg)	DI Water	Conc. Nitric Lot # (1 mL)	Alfa Aesar Magnesium Nitrate Lot # /exact weight (200 mg)	Modifier Lot # (Final volume = 100 mL)
23960	MQRM324	1114110	23106	CH062315PMM
N28 Yoss wt. 0.3007	MQRM324	1114070	23106 (0.927)	RG092815PMM
N28 Yoss wt. 0.6419	MQRM324	1114110	23106 (0.402)	G-102416PMM
N28 Yoss wt. 0.6414	MQRM324	1115080	23106 (0.407)	G-011416PMM
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
MQRM324				
Remake every 6 months		GFAAS LOT #: analyst initials, date prepared, solution acronym		
DHEC Asset ID of balance used: F7678				


Attachment IX-A

MERCURY DIGESTION

STANDARD METHODS 3112B

<u>STEP</u>	<u>COMPLETED</u>
1. Turn on water bath and allow to reach 95 +/- 2 deg. C.	_____
2. Add standards and samples to proper digestion vessels.	_____
3. Add Sulfuric acid solution to all digestion vessels.	_____
4. Add Nitric acid solution to all digestion vessels.	_____
5. Add Potassium Permanganate solution to all digestion vessels.	_____
6. Wait 15 minutes. START: _____ FINISH: _____	_____
7. Add Potassium Persulfate to all digestion vessels.	_____
8. Transfer all digestion vessels to 95 +/- 2 degree water bath.	_____
9. Digest for two(2) hours. START: _____ FINISH: _____	_____
10. Remove and allow to cool to room temperature(may be held overnight).	_____
11. Turn on Cetac Analyzer(A or B) and allow lamp to warm.	_____
12. Just prior to analysis add Sodium Chloride-Hydroxylamine Hydrochloride solution and mix. Add extra if Potassium Permanganate color does not dissipate.	_____
13. Pour into appropriate sample tubes and analyze.	_____


Attachment IX-C

 Preparation of 5% Potassium Persulfate Solution Analytical & Radiological Environmental Services Division			
<input type="checkbox"/> Intermediate <input checked="checked" type="checkbox"/> Working			
Potassium Persulfate Lot#	Amount Added	DI Water	5% Potassium Persulfate Lot #
083939	200 grams	RO332	GP030915PPS
084173	200 grams	RO332	GP061915PPS
084173	200 grams	RO332	GP090315PPS
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
	200 grams		
Date of Manufacture included in Lot #		Final Volume = 4000 mL	
Balance FD - F7678			
		Page <u> 1 </u>	

Attachment IX-I

Preparation Mercury 1 ppm Solution Analytical & Radiological Environmental Services Division					
(X) Intermediate () Working			Remake every month.		
DI Water ID: RO332					
Vendor of Parent Solution is Ultra Scientific					
Acid Lot	100 ppm Lot #	Working Standard Lot#	Stock Conc.	Amount used	Final Conc. mg/L
1114080	6P051915 M1100	6P070615 M11	100 ppm	1 ml	1.00
1114080	L	6P090515 M11	100 ppm	1 ml	1.00
1114080	6P090315 M1100	6P090315 M11	100 ppm	1 ml	1.00
1114080	6P090315 M1100	MA100715 M11	100 ppm	1 ml	1.00
1115010	6P090315 M1100	MA110915 M11	100 ppm	1 ml	1.00
1115010	6P090315 M1100	MA120715 M11	100 ppm	1 ml	1.00
1115010	6P090315 M1100	MA111615 M11 <i>MA 1116-16</i>	100 ppm	1 ml	1.00
In addition to the above listed aliquats of standards, 0.5 ml of concentrated nitric acid was added to the 100 ml volumetric flask containing about 50 ml of deionized water. The solution was then diluted to volume with deionized water.					
Vendor is <u>VWR</u>					
DI Water: RO332					
Date of Manufacture is included in the Working Lot#.					Page _____

Attachment IX-J

 Preparation Mercury Working 100 ppb Standard Solution Analytical & Radiological Environmental Services Division					
<input type="checkbox"/> Intermediate <input checked="" type="checkbox"/> Working		Remake weekly.			
DI Water ID: RO332					
Vendor of Parent Solution is UltraScientific					
Acid Lot #	1 ppm Lot #	Working Standard Lot#	Stock Conc.	Amount used	Final Conc. ug/L (ppb)
114080	GPO70615 MII	GPO706715 1100PPB	1 ppm	10	100
114080	GPO70615 MII	GPO71415 100 ppb	1 ppm	10 ml	100
114080	GPO70615 MII	GPO72715 100 ppb	1 ppm	10 ml	100
114080	GPO80515 MII	GPO80515 100 ppb	1 ppm	10 ml	100
114080	GPO90515 MII	GPO81015 100 ppb	1 ppm	10 ml	100
114080	GPO80515 MII	GPO8715 100 ppb	1 ppm	10 ml	100
114080	GPO80515 MII	GPO82515 100 ppb	1 ppm	10 ml	100
114080	GPO80515 MII	GPO8115 100 ppb	1 ppm	10 ml	100
114080	GPO90315 MII	MA091415 100 ppb	1 ppm	10 ml	100
114080	GPO90315 MII	MA092115 100 ppb	1 ppm	10 ml	100
114080	GPO90315 MII	MA092215 100 ppb	1 ppm	10 ml	100
114080	GPO90315 MII	MA092815 100 ppb	1 ppm	10 ml	100
114080	MA100715 MII	MA100715 100 ppb	1 ppm	10 ml	100
114080	MA100715 MII	MA101315 100 ppb	1 ppm	10 ml	100
114080	MA100715 MII	MA101915 100 ppb	1 ppm	10 ml	100
114080	MA100715 MII	MA102615 100 ppb	1 ppm	10 ml	100
115010	MA100715 MII	MA110315 100 ppb	1 ppm	10 ml	100
115010	MA110915 MII	MA110915 100 ppb	1 ppm	10 ml	100
115010	MA110915 MII	MA112315 100 ppb	1 ppm	10 ml	100
115010	MA110915 MII	MA113015 100 ppb	1 ppm	10 ml	100
115010	MA120815	MA120815			
115010	MA120715 MII	MA120815 100 ppb	1 ppm	10 ml	100
115010	MA120715 MII	MA121415 100 ppb	1 ppm	10 ml	100
115010	MA122915 MII	MA122915 100 ppb	1 ppm	10 ml	100
115010	MA011116 MII	MA011116 100 ppb	1 ppm	10 ml	100
115010	MA011116 MII	MA011916 100 ppb	1 ppm	10 ml	100

In addition to the above listed aliquats of standards, 2 ml of 1:1 nitric acid was added to the 100 ml volumetric flask containing about 20 ml of deionized water. The solution was then diluted to volume with deionized water.

The 1:1 nitric acid vendor is: VWR

Page _____



Reference Materials

• Certificate of Analysis •

Product: WatR™ Supply Inorganic Disinfection #1
 Catalog Number: 5272
 Lot No. S195-5272 - QCCS- Disl-15-1
 Certificate Issue Date: January 22, 2013
 Expiration Date: February 28, 2015
 Revision Number: Original

CERTIFICATION

Parameter	Certified Value ¹	Uncertainty ²	QC Performance Acceptance Limits ³	PT Performance Acceptance Limits ⁴
	µg/L	%	µg/L	µg/L
Chlorate	83.1	5.90	62.5 - 101	58.2 - 108
Chlorite	317	3.05	217 - 425	222 - 412

ANALYTICAL VERIFICATION

Parameter	Certified Value ¹	Proficiency Testing Study			NIST Traceability	
		Mean	Recovery ³	n	SRM Number	Recovery
	µg/L	µg/L	%			%
Chlorate	83.1	84.2	101	9	-	-
Chlorite	317	306	96.6	10	-	-

Prepared using 1mL of lot # S195-5272 diluted to 200mL DI water
 DI-water - synergy UV Serial # F8MN174578 (room 320)
 prepared by CDM 1/23/15



Cyanide Workbook
Analytical & Radiological Environmental Services Division

5

Distillation Date: 1-19-16 Colorimetric Analysis Date: 1-19-16 Wavelength: 578 nm Cell Path Length: 1 cm

Sample Number	Reported Value mg/L	Sampling Location	mL of Spike added	Source of Standard (1°/2°)	mL Original Sample	mL Absorbing Solution	Absorbance	µg CN from Curve	Calculated CN Value mg/L
Blank	<u>< 0.01 mg/L</u>		<u>0</u>	<u>N/A</u>	<u>50</u>	<u>50</u>	<u>0.007</u>	<u>0.0917</u>	<u>0.00195</u>
<u>0.5</u>	<u>108 % Rec</u>		<u>0.5</u>	<u>1°</u>	<u>50</u>	<u>50</u>	<u>0.024</u>	<u>0.5423</u>	<u>0.01085</u>
<u>1.0</u>	<u>93 % Rec</u>		<u>1.0</u>	<u>1°</u>	<u>50</u>	<u>50</u>	<u>0.039</u>	<u>0.9346</u>	<u>0.01869</u>
<u>2.0</u>	<u>100 % Rec</u>		<u>2.0</u>	<u>1°</u>	<u>50</u>	<u>50</u>	<u>0.080</u>	<u>2.0068</u>	<u>0.04014</u>
<u>5.0</u>	<u>96 % Rec</u>		<u>5.0</u>	<u>1°</u>	<u>50</u>	<u>50</u>	<u>0.188</u>	<u>4.8311</u>	<u>0.09662</u>
<u>10.0</u>	<u>100 % Rec</u>		<u>10.0</u>	<u>1°</u>	<u>50</u>	<u>50</u>	<u>0.389</u>	<u>10.0875</u>	<u>0.20175</u>

1° Working Cyanide 1 ppm Standard ID: 16-404 1:1 H2SO4 Acid ID: 15-313 Date Curve Analyzed: 1-18-16 Temp: 125°
 2° Working Cyanide 1 ppm Standard ID: 16-405 NaOH 0.25N ID: 16-401 Curve Slope: 0.038 Distillation Time start: 10:30
 Acetate Buffer ID: 16-48 Color Reagent ID: 16-406 Curve Intercept: 0.0033 Stop: 11:32
 Chloramine-T ID: 16-407 DI Water Source: 20167 R value: 0.9996 Curve Passed/Failed: Pass

Calculation - Colorimetric Analysis: CN, mg/L = AX50 Where: A = µg CN read from calibration curve
 BXC B = mL original sample used in distillation
 C = mL absorbing solution used in colorimetric analysis

Analyst: gs Date Analyzed: 1-19-16 Recorded Results Verified By: _____ Date: _____
 Results Recorded By: N/A Date: _____ QC Recorded By: _____ Date: 1-19-16
 Results Verified By: _____ Date: _____ QC Verified By: _____ Date: _____

Floating Block ID: H4747 Spectrophotometer ID: H4748

ARESD - Automated Analysis Section
Reagent Prep Log for Fluoride Buffer Solution
(Fluoride Analysis)

<p>Reagent ID number RBJ - <u>10</u> - <u>5</u> . Fluoride Buffer Solution was prepared on <u>12/7/15</u> at <u>10:20am</u> by <u>CFG</u> . Using Balance id <u>H10827</u> 114.0 mL of glacial acetic acid, CH₃COOH was added to a 2.000 liter volumetric flask containing about 1000 ml of deionized water. While stirring 60.0 grams of sodium hydroxide, NaOH, 116.0 grams of sodium chloride, NaCl, and 8.0 grams of trans 1,2-diaminocyclohexane, CDTA, was slowly added to the volumetric flask. The solution was stirred for a minimum of 30 minutes. 1.0 mL of 100.0 mg/L Stock Standard was added. The solution was then brought to volume with deionized water The vendor for the glacial acetic acid, CH₃COOH, is <u>Fisher Scientific</u> . The Lot # for the glacial acetic acid, CH₃COOH, is <u>100221</u> . The vendor for the sodium hydroxide, NaOH, is <u>Fisher Scientific</u> . The Lot # for the sodium hydroxide, NaOH, is <u>152171</u> . Actual weight for NaOH <u>120.0000</u> The vendor for the sodium chloride, NaCl, is <u>VWR</u> . The Lot # for the sodium chloride, NaCl, is <u>144828</u> . Actual weight for NaCl is <u>116.0000</u> The vendor for the trans 1,2-diaminocyclohexane, CDTA is <u>GFS Chemicals</u> . The Lot # for the trans 1,2-diaminocyclohexane, CDTA is <u>C4109241</u> . Actual weight for trans 1,2-diaminocyclohexane, CDTA is <u>8.0000</u></p>
<p>Reagent ID number RBJ - <u>10</u> - <u>10</u> . Fluoride Buffer Solution was prepared on <u>1/8/16</u> at <u>9:46am</u> by <u>CFG</u> . Using Balance id <u>H10827</u> 114.0 mL of glacial acetic acid, CH₃COOH was added to a 2.000 liter volumetric flask containing about 1000 ml of deionized water. While stirring 60.0 grams of sodium hydroxide, NaOH, 116.0 grams of sodium chloride, NaCl, and 8.0 grams of trans 1,2-diaminocyclohexane, CDTA, was slowly added to the volumetric flask. The solution was stirred for a minimum of 30 minutes. 1.0 mL of 100.0 mg/L Stock Standard was added. The solution was then brought to volume with deionized water The vendor for the glacial acetic acid, CH₃COOH, is <u>Fisher Scientific</u> . <u>CFG 2/3/16</u> The Lot # for the glacial acetic acid, CH₃COOH, is <u>100221 100221</u> . The vendor for the sodium hydroxide, NaOH, is <u>Fisher Scientific</u> . The Lot # for the sodium hydroxide, NaOH, is <u>152171</u> . Actual weight for NaOH <u>120.0000</u> The vendor for the sodium chloride, NaCl, is <u>VWR</u> . The Lot # for the sodium chloride, NaCl, is <u>144828</u> . Actual weight for NaCl is <u>116.0000</u> The vendor for the trans 1,2-diaminocyclohexane, CDTA is <u>GFS Chemicals</u> . The Lot # for the trans 1,2-diaminocyclohexane, CDTA is <u>C4109241</u> . Actual weight for trans 1,2-diaminocyclohexane, CDTA is <u>8.0000</u></p>

Page # 3

Attachment XIII

```

=====
===== WASTE =====
=====
CYCLE START AT 0:34:13A
ON 11/05/15

CYCLE COUNT 11-5-15
OPERATOR EQC EIC
STERILIZER GRAVITY
CYCLE TYPE GRAVITY
CYCLE NO. 1
    
```

```

CYCLER TEMP = 124.0C
CONTROL TEMP = 123.3C
STER TIME = 0:30:00
DRY TIME = 0:05:00
    
```

```

- TIME T= C P= S=
U=10H9
-----
C 8:54:50A 74.9 1.2P
    
```

```

C 8:55:51A 88.1 10.8P
S 8:59:10A 124.8 29.1P
S 9:04:11A 125.8 25.6P
S 9:09:11A 125.7 23.2P
S 9:14:11A 125.7 22.7P
S 9:19:11A 125.5 22.4P
S 9:24:11A 125.5 22.1P
E 9:29:11A 125.5 21.5P
4 ALARM
TOO LONG IN EXHAUST
9:34:12A 102.1 4.2P
E 9:34:21A 102.0 3.9P
E 9:34:22A 101.9 3.9P
    
```

```

E 9:39:23A 92.3 11.6P
Z 0:39:46A 92.7 2.0P
    
```

```

LOAD 110501
CHAMBER TEMP MAX=126.4C
CHAMBER TEMP MIN=124.0C
    
```

```

CONDITION = 4:20
STERILIZE =30:00
EXHAUST =10:35
TOTAL CYCLE =44:55
    
```

```

=====
= READY TO UNLOAD =
=====
    
```