US Environmental Protection Agency
Office of Pesticide Programs

Office of Pesticide Programs
Microbiology Laboratory
Environmental Science Center, Ft. Meade, MD

Standard Operating Procedure for

Quality Assurance of Purified Water

SOP Number: QC-01-07

Date Revised: 03-02-17

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Title	Quality Assurance of Purified Water
Scope	This protocol outlines the procedures for monitoring the quality of the deionized water used in the laboratory to make media. The vendor will perform the following tests: Specific heavy metals (cadmium [Cd], lead [Pb], nickel [Ni], zinc [Zn], copper [Cu], and chromium [Cr]), total heavy metals (cumulative value of the six specific heavy metals), total organic carbon, conductivity, heterotrophic plate counts, and water quality/suitability. The total chlorine residual test is performed in-house.
Application	Verifying water quality is an important component of a laboratory's internal quality control program. Poor water quality can significantly affect the outcome of procedures.

	Approval Date
SOP Developer:	_____ Print Name: _____
SOP Reviewer	_____ Print Name: _____
Quality Assurance Unit	_____ Print Name: _____
Branch Chief	_____ Print Name: _____

Date SOP issued:	
Controlled copy number:	
Date SOP withdrawn:	

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1. Definitions	Abbreviations/definitions are provided in the text.
2. Health and Safety	1. Follow procedures specified in SOP MB-01, Laboratory Biosafety. The Study Director and/or lead analyst should consult the Safety Data Sheet for specific hazards associated with products. 2. Several of the tests to be performed on the purified water require that the sample water be preserved with sulfuric or nitric acid. To protect against possible chemical burns, the laboratory worker must wear a lab coat, gloves, and protective eyewear (e.g., glasses or goggles) while filling water sample bottles containing sulfuric or nitric acid.
3. Personnel Qualifications and Training	Refer to SOP ADM-04, OPP Microbiology Laboratory Training.
4. Instrument Calibration	Not Applicable.
5. Sample Handling and Storage	Refer to section 12 for sample handling and storage conditions.
6. Quality Control	None
7. Interferences	1. Each new lot of the DPD Powder Pillows reagent (i.e., DPD [salt of N, N-Diethyl-p-Phenylenediamine Potassium Iodide Sodium Phosphate, Dibasic] Total Chlorine Reagent Powder Pillows) should be checked for reagent accuracy. If the DPD Powder Pillows do not demonstrate reagent accuracy, the total chlorine residual test may be jeopardized. See section 12.4 for a description of the method to check reagent accuracy. 2. Discard expired DPD Total Chlorine Reagent Powder Pillows. 3. All test parameters will be conducted at the monitoring frequency indicated in Table 1: Monitoring Frequency and Acceptable Limits for Water Quality Parameters (section 12.1). 4. Individual test parameters will be tested more frequently than described in Table 1: Monitoring Frequency and Acceptable Limits for Water Quality Parameters (section 12.1) if a problem with water quality is identified (see section 8, Non-Conforming Data and section 13, Data Analysis and Calculations). The laboratory may opt to test the water whenever the source of the water is changed, if there is an upgrade, or there is a known failure of the building's deionized water system.

8. Non-conforming Data	1. Procedures will be consistent with SOP ADM-07, Non-Conformance Reports. 2. If the water quality, including residual chlorine, falls outside of acceptable limits (Table 1), take corrective actions (in consultation with the Facility Engineer) and retest the water to demonstrate that the water quality parameter(s) that previously fell outside of acceptable limits is/are now within acceptable limits. 3. Disinfectant efficacy tests may have been performed between the time the water quality fell outside of acceptable limits (i.e., potentially one day after the date of the previous sampling for that test parameter) and the time that results of successful retesting are received. If the media and reagent controls associated with each test indicate that the media and reagents perform(ed) acceptably, the test data are considered valid. 4. If any water quality parameter is outside of the acceptable limits consult with the Facility Operation Branch Mechanical Engineer.
9. Data Management	1. Data will be archived consistent with SOP ADM-03, Records and Archives. 2. Reports are e-mailed, printed, and placed in the Quality Assurance of Purified Water Record Book. A carbon copy of the vendor chain of custody form is archived.
10. Cautions	1. Let the water run for approximately 30 seconds prior to filling sample collection bottles. 2. The water collected for the heterotrophic plate counts must be kept cold during transit by packing the container with ice packs. Heterotrophic plate counts are analyzed within 30 hours of collection. Hence, collect the water sample as close to the mail pick up time as practical. 3. Ship water samples to vendors on Mondays, Tuesdays, and Wednesdays only to ensure that the samples arrive prior to a weekend. 4. Legibly complete the vendor chain of custody forms and sample identification labels to maintain evidence of chain of custody and to ensure that the testing laboratory performs the analyses requested by the ESC/OPP Microbiology Laboratory.
11. Special Apparatus and Materials	1. *Building deionized water spigots.* Located in each laboratory sink. There are two in B206. 2. *Vendor-supplied test request/chain of custody forms and water collection bottles.* Contact vendor to obtain additional forms and

<table>
<tr><td></td><td>bottles.
3. Styrofoam shipping container. To ship the water sample to the testing laboratory.
4. Refrigerant packs. To keep the water samples cool in shipment.
5. Hach Total Chlorine Test Kit. 0-3.5 mg/L (Hach catalog number 2231-03), for determination of chlorine concentration in the water sample.
6. Chlorine Standard Solution. 50-75 mg/L, 2-mL PourRite ampule (Hach catalog number 14268-20), for calibration of the Hach Total Chlorine Kit.
7. Concentrated Nitric Acid. For heavy metal test</td></tr>
<tr><td>12. Procedure and Analysis</td><td>The vendor will perform the following tests: Specific heavy metals (cadmium [Cd], lead [Pb], nickel [Ni], zinc [Zn], copper [Cu], and chromium [Cr]), total heavy metals (cumulative value of the six specific heavy metals), total organic carbon, conductivity, heterotrophic plate counts, and water quality/suitability.

The total chlorine residual test is performed in-house.</td></tr>
<tr><td>12.1 Frequency of Analysis and Acceptable Limits</td><td>Table 1. Monitoring frequency and acceptable limits for water quality parameters.
<table>
<tr><th>Test</th><th>Monitoring Frequency</th><th>Accepted Limits</th></tr>
<tr><td>Heavy Metals, Total</td><td>Annually</td><td><0.10 mg/L*</td></tr>
<tr><td>Heavy Metals, Single (Cd, Cr, Cu, Ni, Pb, and Zn)</td><td>Annually</td><td>< 0.05 mg/L</td></tr>
<tr><td>Water Suitability Test</td><td>Annually</td><td>0.8 to 3.0 ratio</td></tr>
<tr><td>Total Organic Carbon</td><td>Twice a year</td><td>< 1.0 mg/L</td></tr>
<tr><td>Conductivity</td><td>Twice a year</td><td>< 2 umhos/cm at 25°C</td></tr>
<tr><td>Total Chlorine Residual</td><td>Twice a year</td><td><0.1 mg/L**</td></tr>
<tr><td>Heterotrophic Plate Counts</td><td>Twice a year</td><td>< 500 Colony Forming Units/mL (CFU/mL)</td></tr>
</table>
*The accepted limit for total heavy metals is determined by finding the cumulative value of the six specific heavy metals. If the testing result for the specific heavy metals are “not detected”, determine the cumulative value of the reportable limits (RL). This value must be below 0.10 mg/L.

**As detected by Hach Total Chlorine Test Kit (0.0-3.5 mg/L)</td></tr>
<tr><td>12.2 Collection and Preparation</td><td>a. Either deionized water spigot in B206 can be used to collect the water in the vendor supplied bottles. Fill each bottle to the top. Refer to</td></tr>
</table>

of Water for Vendor Performed Tests

Table 2.

Table 2. Collection specifications for water quality parameters.

Test	Type of Collection Bottle/Preservative
Heavy Metals, Single (Cd, Cr, Cu, Ni, Pb, and Zn)	One Pint/Dropper full of Nitric Acid
Total Organic Carbon	One Pint/Sulfuric Acid
Conductivity	One Pint/No Preservative
Heterotrophic Plate Counts	Sterile Bottle/No Preservative
Water Suitability Test	Square, Glass Bottle/No Preservative

b. To the Heavy Metal bottle add a dropper full of concentrated nitric acid in the fume hood using appropriate PPE.

c. Water Suitability Test: Collect deionized water sample in the glass bottle supplied by the vendor.

d. Complete the test request/chain of custody form.

e. Once all sample collection bottles have been filled and the collection time recorded on the vendor sample identification labels (in military time) and the test request/chain of custody form, place the bottles in a styrofoam shipping container. Place the samples in the shipping container and ice packs and add packing material to prevent the bottles from moving during shipment.

f. Place the vendor test request/chain of custody form in the box. Tape the box closed. Ship the package by priority overnight shipping.

12.3 Chlorine Residual Test

a. Monitor the total chlorine residual of the water twice a year using the Hach Total Chlorine Test Kit, 0-3.5 mg/L (Hach catalog number 2231-03).

b. Clean a beaker and the kit's plastic viewing tubes and caps with isopropyl alcohol or a non-abrasive detergent prior to commencing the test. Rinse several times with the deionized water. Use a soft cloth for wiping or drying the plastic viewing tubes. Do not use paper towels or tissue as this may scratch the plastic.

c. Collect sample water from a deionized water spigot in B206. Pour water from the beaker into one plastic viewing tube until the water level reaches the first line (bottom edge of the frosted area-equals 5 mL). This is the blank.

d. Place the blank in the top left opening of the color comparator.

e. Fill the second plastic viewing tube to the first line (bottom edge of the

	frosted area) with sample water from the beaker. f. Add the contents of one DPD (salt of N,N-Diethyl-p-Phenylenediamine Potassium Iodide Sodium Phosphate, Dibasic) Total Chlorine Reagent Powder Pillow to the second tube. (Note: Check reagent accuracy of each new lot of DPD Total Chlorine Reagent Powder Pillows. See section 12.12). g. Cap the second tube and swirl to mix. Accuracy of the test is not affected by undissolved powder. h. Wait 3 minutes. The result of the test must be read within 6 minutes of the addition of the powder. i. Place the second tube in the top right opening of the color comparator. Hold the comparator up to a light source such as a window or lamp. Look through the openings in the front of the comparator. j. Rotate the color disc until the color matches in the two openings. k. Read and record the mg/L total chlorine in the scale window. l. Rinse the viewing tubes several times with de-ionized water and allow to dry before putting them back in the kit.
12.4 Accuracy check of a new lot of DPD Powder Pillows	a. When a new lot of DPD Powder Pillows is received, conduct the following test prior to using the DPD Powder Pillows to monitor the total chlorine residual of the laboratory's deionized water. b. Clean two beakers and the kit's plastic viewing tubes and caps with isopropyl alcohol or a non-abrasive detergent prior to commencing the test. Rinse several times with deionized water from a deionized water spigot in B206. Use a soft cloth for wiping or drying the plastic viewing tubes. Do not use paper towels or tissue as this may scratch the plastic. c. In a beaker, collect approximately 100 mL sample water from a deionized water spigot in B206. Pour water from the beaker into one plastic viewing tube until the water level reaches the first line (bottom edge of the frosted area-equals 5 mL). This is the blank. d. Cap the blank and place it in the top left opening of the color comparator (containing color disc). e. Snap open an ampule of the Chlorine Standard Solution, (50-75 mg/L, 2-mL PourRite ampule). Note the amount of free chlorine at the time that the ampules were filled. This value can be found on the first page of the instructions accompanying the chlorine standard. f. Prepare a dilution of the standard to yield anywhere from 1 to 3 mg/L

	free chlorine. For example, if the amount of free chlorine in the standard is 64.2 mg/L, adding 1 mL of the chlorine standard to 49 mL water will yield a water sample with approximately 1.3 mg/L chlorine (calculation: [64.2 mg/L][1 mL]=[X mg/L][50 mL]; solving for X yields X=1.28 mg/L). Using a sterile 25 mL pipet, pipet 49 mL of water from the beaker and add it to a second, empty beaker. Using a sterile 1 mL pipet, add 1 mL of the chlorine standard to the 49 mL of water. Swirl to mix. g. Fill the second plastic viewing tube to the first line (bottom edge of the frosted area) with chlorinated water from the second beaker. h. Add the contents of one DPD Total Chlorine Reagent Powder Pillow to the second tube. i. Cap the second tube and swirl to mix. Accuracy of the test is not affected by undissolved powder. j. Wait 3 minutes. The result of the test must be read within 6 minutes of the addition of the powder. k. Place the second tube in the top right opening of the color comparator. Hold the comparator up to a light source such as a window or lamp. Look through the openings in the front of the comparator. l. Rotate the color disc until the color matches in the two openings. m. Read the mg/L total chlorine in the scale window. The mg/L total chlorine read from the color comparator must approximate the chlorine concentration (mg/L) in the prepared dilution in order for the reagents to be used in the total chlorine residual test. n. Record the results in the DPD Reagent Accuracy Form. o. Discard the remaining chlorine standard in the ampule by pouring it down the sink. Rinse the ampule with water and place it in a box for broken glass. p. Rinse the viewing tubes several times with deionized water and allow to dry before putting them back in the kit.
13. Data Analysis/ Calculations	1. When the report of water sample analysis is received from the vendor laboratory, record the testing results legibly and in indelible ink under the “Test Results” column of the Quality Assurance of Purified Water Form (see section 14). Compare the testing results with the “Accepted Limits” (see Table 1). For each parameter, if the testing results for that parameter fall within the accepted limits, indicate in the form that the water quality is acceptable. If the testing results for that parameter fall outside of the accepted limits, indicate that the water quality is not

	acceptable and record the corrective action taken. 2. Record the results of the total chlorine residual test in the Quality Assurance of Purified Water Form (see section 14). Compare the testing results with the "Accepted Limits" (see Table 1). If the testing results fall within the accepted limits, indicate in the form that the water quality is acceptable. If the testing results fall outside of the accepted limits, indicate that the water quality is not acceptable, and record the corrective action taken. The water quality is acceptable only if the results for all parameters fall within the accepted limits.
14. Forms and Data Sheets	Test Sheets. Test sheets are stored separately from the SOP under the following file names: DPD Reagent Accuracy Form — QC-01-07_F1.docx Total Chlorine Residual of Purified Water Form — QC-01-07_F2.docx Quality Assurance of Purified Water Form — QC-01-07_F3.docx
15. References	1. Eaton, A.D., Clesceri, L.S., Rice, E.W., Greenberg, A.E. eds. Standard Methods for the Examination of Water and Wastewater, Online Edition. American Public Health Association, American Water Works Association, Water Environment Federation, Section 9020 approved 2005. 2. U.S. EPA Region III and OPPTS Environmental Science Center Specifications, Volume 2, November 7, 1996.