**Attachment D - Procedure for Residual Measurement**

**Louisiana Department of Health, Office of Public Health (LDH/OPH)**

**RFx #3000024507**

**PURPOSE:** The following procedure must be followed for the Residual Measurement (free or total chlorine) when collecting routine samples for bacteriological (total coliform/E. coli) analysis.

1. **Sampling Procedure:**
   1. Remove any attachments from the sample tap, such as hoses, filters, screens, or aerators, and disinfect the sample tap by spraying the sample tap thoroughly with Isopropyl alcohol (91%). Allow to air dry for 2 minutes.
   2. Flush the sample tap for 5 minutes until the water temperature reaches a constant temperature, and then reduce water flow to a smooth (laminar) stream about the size of a pencil if possible.
   3. The disinfectant (chlorine) residual must be measured at the time of collection for the total coliform sample at the LDH/OPH-approved sample sites (*e.g.,* TCR-001, MRT-001, etc.) in the Public Water Systems (PWS) distribution system.
   4. The disinfectant (chlorine) residual must be measured using an LDH/OPH-approved N,N-diethyl-p-phenylenediamine (DPD) colorimeter (e.g., HACH Colorimeter).
   5. Measure the residual type (free or total) that is specified for the water system. Total chlorine is measured for systems that use chloramines. Measure the residual using an appropriate method, taking into account any interfering substances that may be present. For example, both manganese and chloramine can interfere with the DPD colorimetric method, but do not interfere with the indophenol method. As a result, for systems that operate using free chlorine, that have manganese and/or naturally occurring ammonia, the indophenol method or a DPD method that specifically accounts for the interference of concern should be selected
   6. Only EPA [see 40 CFR Part 136.3, Table II] or State-approved methods for potable water shall be used. When running the test, the analyst should follow the specific directions provided with the test kit. In general, the analyst will need to measure out a known volume of sample and will need to add the reagent(s) in the order described, wait a specific reaction time, and then measure the color that develops in the sample. The intensity of the color that develops after the addition of a reagent is measured using a spectrophotometer and relates directly to the amount of disinfection residual present in the sample.
   7. Record the sample collection date (if changed) and time and residual measurement (and Meter#) on the sample chain-of-custody form (COC). The COC must be signed and included with the samples to the LDH/OPH Lab or Depot.
   8. Enter the sample collection date (if changed) and time and residual measurement into STARLIMS by 8:00 A.M. (CT) on the day after sample was collected. Initially this will be done by LDH/OPH staff until the Contractor is setup to access STARLIMS.
2. **Meter and Methods:**

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| **Meter** | **Test** | **Sample Size** | **Sample Container** | **Reagents** | **Test Procedure** | **Range** |
| Pocket Colorimeter™ II, Chlorine (Free and Total)  #5870000 | DPD Free  (Low Range) | 10-mL | Glass  #2427606 | DPD  10 mL  Powder Pillows | Refer to directions provided with test kit. | 0.02 – 2.00 mg/l (will max out and flash at 2.20 mg/l) |
| DPD Total  (Low Range) |
| DPD Free  (High Range) | 5-mL | Plastic  #4864302 | DPD  2-10 mL or  1-25 mL  Powder Pillows | Refer to directions provided with test kit. | 0.1-8.0 mg/l for 2-10 mL  0.1-10.0 mg/l for 1-25 mL |
| DPD Total  (High Range) |
| Pocket Colorimeter™ II, Chloramine, Mono and Free Ammonia (Free)  #5870026 | Indophenol  Free Cl  Hach Method 10241 | 10-mL | Plastic  #4864302 | Freechlor F Reagent Solution  (5 drops)  Monochlor F Reagent Powder Pillows | Refer to Hach Indophenol Method 10241 | * 1. - 4.50 mg/L |
| DR900 Multiparameter Portable Colorimeter  #9385100 | DPD Free  (Low & High) | 5-mL  10-mL | Plastic  #4864302  Glass  #2401906 | DPD Powder Pillows  Free 10069, 10245  Total 10070, 10250 | Refer to directions provided with test kit. | High 0.1-10 mg/L  Mid 0.05 – 4.00 mg/L |
| DPD Total  (Low & High) |
| Indophenol  Free Cl  Hach Method 10241 | 10-mL | Glass  #2401906 | Freechlor F Reagent Solution  (5 drops)  Monochlor F Reagent Powder Pillows | Refer to Hach Indophenol Method 10241 | 0.04 – 4.50 mg/l |
| Pocket Colorimeter™ II, Chlorine, (Free and Total) plus pH**– not recommended for compliance reporting.** | DPD Free  (High Range) | 5-mil | Plastic  #4864302 | DPD 1-25 mL Powder Pillow | Refer to directions provided with test kit. | 0.2 – 10.0 mg/L  Requires LR procedure when result is < 2 mg/L; **instrument does not support LR procedure**. |
| DPD Total  (High Range) |
| **Notes**: | | | | | | |
| 1. Under Range – flashing number below test range; Verify instrument cap is correctly seated and Check zero by measuring a blank | | | | | | |
| 1. Over Range - flashing number above test range; Check for Light Blockage; Switch meter to HR and retest sample; if the sample is still over range, dilute the sample to bring it within the test range. See Section IV. HOW TO DILUTE SAMPLES | | | | | | |
| 1. Field test results that are suspected of being incorrect or are unexpected may be resampled for confirmation. | | | | | | |

1. **METER VERIFICATION:**
   1. The disinfectant (chlorine) residual meter must be verified weekly with chlorine standards. There are 2 sets, Low Range and High Range; each with 1 blank and 3 standards.
   2. Check that the chlorine standards are not expired.
   3. Verify the meter with both Low Range and High Range by zeroing with the blank and reading each standard. See below examples.
   4. If the meter fails by reading outside the range of the standard, do not use the meter for chlorine measurement. Contact the central office for another meter.

Meter: SN 13080E228638 Verification Date: 1/6/17

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| Standard Lot: A3242 | | Exp Date: 8/2017 | |
| Low Range | Standard Value | Range | LR Meter Reading | Pass/Fail |
| Standard 1 | 0.22 ± 0.09 | 0.13-0.31 | 0.21 | Pass |
| Standard 2 | 0.92 ± 0.10 | 0.82-1.02 | 0.93 | Pass |
| Standard 3 | 1.61 ± 0.14 | 1.47-1.75 | 1.63 | Pass |

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| --- | --- | --- | --- | --- |
| Standard Lot: A3229 | | Exp Date: 8/2017 | |  |
| High Range | Standard Value | Range | HR Meter Reading | | | Pass/Fail |
| Standard 1 | 2.1 ± 0.2 | 1.9-2.3 | 2 | | | Pass |
| Standard 2 | 3.7 ± 0.3 | 3.4-4.0 | 3.7 | | | Pass |
| Standard 3 | 6.7 ± 0.6 | 6.1-7.3 | 6.8 | | | Pass |

1. **HOW TO DILUTE A SAMPLE WHEN THE RESULT IS OVER RANGE:**

The simplest dilution is a one-to-one or 2-fold dilution, where one part sample is mixed with one part deionized or distilled water. This reduces the concentration of anything in the sample by half. Follow the usual test procedure using the diluted sample, and then multiply the result by 2 to find the concentration of the sample before it was diluted.

Example: Your sample reads off-scale on your kit or instrument display. Mix 5 mL sample with 5 mL deionized water and repeat the test. The result using this diluted sample is 1.9 mg/L. The concentration of the sample before dilution is (1.9 x 2) = 3.8 mg/L.

If the sample is still over-range after dilution, a larger dilution will need to be made. For example, mix one part sample to two parts deionized water for a 3-fold dilution (multiply result by 3), one part sample to three parts deionized water for a 4-fold dilution (multiply result by 4), and so on. If your test uses the sample as a blank, use the diluted sample as the blank.

It is very important to measure the amount of sample and deionized water accurately when making dilutions. An error in these measurements will lead to an error in the result, which will then be multiplied by the dilution factor. At minimum, measure the sample and deionized water in a graduated cylinder. For best results use a TenSette pipet for measuring the sample, add it to a Class A volumetric flask, and fill to the mark with deionized water. Mix thoroughly before testing.

You can adjust the measured volumes to accommodate the pipets and flasks available to you. For instance, if you need to make a 20-fold dilution, you can measure 1 mL of sample and add 19 mL of dilution water. Or you can measure 5 mL of sample and add 95 mL of water (or dilute to the mark on a 100-mL volumetric flask) to get the same dilution. If only 10 mL is required for running the test, use just 10 mL of this mixture. Use the extra for the blank or duplicate measurements if desired.