Thermo SCIENTIFIC

Meter Log # 68

Parameter and Sample Type
Total Chlorine Residual in Wastewater by Colorimetry

Introduction
Total chlorine residual is a regulated parameter in wastewater effluents and a process parameter in the treatment plant. Orion AC4P72 Total Chlorine DPD Method is EPA-approved for total chlorine reporting in wastewater samples.

References

1. Orion AC4P72 Total Chlorine DPD Method, rev 3, July 9, 2002; EPA accepted on June 7, 2004. www.thermo.com/waterapps.
2. Standard Methods for the Examination of Water and Wastewater, 20th edition, 1998; Part 4500-Cl G., DPD Colorimetric Method.

Recommended Equipment
Orion AQ3070 Colorimeter; Orion AC4P72 Total Chlorine reagent powder packs; Orion AC3V25 25mm vials; AQ3070 Colorimeter User Guide; timer.

Required Solutions
Chlorine standard - stock standard and 1 mg/L as chlorine; deionized (DI) water, free of chlorine or chlorine demand.

Solutions Preparation
1. Option 1: Purchase chlorine stock standard commercially and dilute or spike directly to prepare 1 mg/L standard; or
2. Option 2: Prepare chlorine surrogate standard at 1000 mg/L (as chlorine) by dissolving 445.5 mg of reagent grade potassium permanganate with DI in a 500 mL volumetric. Mix well and store in a light-blocking bottle.
3. Weekly: Dilute 10.0 mL of chlorine surrogate standard with DI in a 100 mL volumetric flask for a 100 mg/L standard.
4. Daily: When 1.0 mL of weekly solution is diluted with DI in a 100 mL volumetric flask, a chlorine equivalent of 1.0 mg/L will be produced in the DPD reaction.

Meter Setup
Turn on the meter, press the mode key to select the total chlorine mode, and press Read/Enter key to enter standby mode. Fill a clean vial to the line with DI water. Cap and wipe w/ lint-free wiper until dry and free of smears. Handling the vial by the cap, insert the vial into the meter aligning the mark on the vial with the mark on the meter. Zero the meter on DI water.

Testing Procedure
Fill a cleaned vial to the line with 10 mL of sample, standard, or blank water for testing. Use a pipet for best accuracy. (Note: If the sample is colored or turbid, zero the meter with sample to compensate for that. Cap and wipe the vial clean. Insert the vial and zero the meter. Before testing another sample, rezero the meter with DI or the new sample). Tear or cut open one AC4P72 total chlorine powder pack and empty the entire contents into the vial. Start the timer. Cap the vial and swirl vigorously or shake gently for 30 seconds. It is not necessary for all the powder to dissolve. Wipe the vial until clean and dry. Insert, aligning marks. After 2 minutes, press the Read/Enter key to obtain the total chlorine reading.

Meter Performance Check/Calibration Verification
Check meter accuracy by reading a chlorine standard at 1 mg/L and a reagent blank. The reagent blank should read <0.0.3 mg/L and the 1 mg/L standard should read within +/- 15%, e.g., 0.85 – 1.15 mg/L.
If the meter performance check fails, take corrective actions as follows: 1) wipe the vial carefully with a lint-free wipe to remove all fingerprints and liquid drips from the exterior, handle the vial by the cap only, and remeasure; 2) if the powder is not white and free flowing, use another powder pack or another lot of powder packs; 3) using a clean vial, rezero the meter with DI water. Using the same vial, fill with 1.0 mg/L standard or DI, add a powder pack, and retest; 4) prepare a fresh 100 mg/L standard and fresh 1.0 mg/L standard and retest.

Sample Vial Storage and Cleaning
Clean and store vials per instructions in the user guide. Do not allow reacted samples to remain in the vials overnight.

Sample Storage and Preparation
Analyze samples immediately – do not store samples. Allow the samples to warm to room temperature before measurement. Mix the sample well, but do not shake, which could cause loss of chlorine due to volatilization and/or oxidation by air.

Calibration
The meter is shipped precalibrated. The meter performance is very stable and does not require frequent calibration. If a standard reading is not within criteria, take all necessary corrective actions (as described in the Meter Performance Check section) to improve meter readings. If corrective actions fail and recalibration is necessary, perform the recalibration according to the meter user guide. Before recalibration, it is important to verify the calibration standard by an alternate test procedure.

Quality Control (QC)
Recommended QC procedures include: calibration verification, reagent blank analysis, QC samples, sample duplicates & spikes.

Notes for Improved Accuracy of Low-Level Samples
If improved accuracy is desired, pay close attention to: 1) completely rinse the sample vial(s) between samples; 2) keep vials used for total chlorine separate from vials used for free chlorine; 3) set the meter zero correctly; 4) handle samples properly to avoid loss of chlorine; 5) use the same vial for zero and for sample.

Interferences
See the meter manual.

Results Statistics
Results for blanks, standards, two wastewater samples (chlorine contact and dechlorination tanks) with duplicates and spikes, and a commercially-available performance evaluation (PE) sample:

Parameter	Criteria	AQ3070 Orion 1	AQ3070 Orion 2	Brand A
Blanks	<0.03 mg/L	0.00	0.00	0.00
1.0 mg/L	85-115%R	108%	107%	96%
1.0 mg/L	15%RSD	2.6%	2.5%	4.2%
0.1 mg/L	85-115%R	105%	108%	109%
MDL	< 0.03 mg/L	0.02	0.02	0.01
0.03 mg/L	0.02-0.04	0.04	0.04	0.03
2.0 mg/L	85-115%R	98%	98%	90%
4.0 mg/L	85-115%R	91%	89%	95%
WW dechlor		0.01	0.01	0.02
Dupl	+/- 0.01 mg/L	0.00	0.00	0.02
0.1 spike	85-115%R	89%	89%	90%
WW chlorine contact		1.59	1.61	1.32
0.32 spike	85-115%R	106%	97%	108%
0.64 spike	85-115%R	108%	97%	84%
PE	1.44-1.95 mg/L	1.85	1.85	1.62
PE duplicate	1.44-1.95 mg/L	1.80	1.80	1.59
PE duplicate	15% D	2.7%	2.7%	1.9%

green shaded cells meet performance criteria