

2014 Updates for 40CFR136

Method Update Rule
March 24, 2014
Fleming Training Center

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NPDES Permit

- * Section 1.2.3 Test Procedures
 - * b. Unless otherwise noted in the permit, all pollutant parameters shall be determined according to methods prescribed in Title 40, Code of Federal Regulations, **Part 136**, as amended, promulgated pursuant to Section 304 (h) of the Act.
- * Section 2.1.4 Proper O&M
 - * a. ...proper O&M also includes ... appropriate quality assurance procedures.

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You Have Heard it All Before

- * More Rules
- * More Testing
- * More Paperwork
- * More Cost



- * But everything we do is regulated.

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2012 Update of 136

- * Standard Methods approved by date not Edition
- * Section 136.7 Quality Assurance and Quality Control.



Federal Register May 18, 2012

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Routinely run parameter	New approval year for parameter method in Std Methods	Method #	Associated methods	Found in 20 th Ed. Std Methods
Oxygen	2001	4500-O	B,C,D, E, F	No
Phenols	2005	5530-phenols	B, D	No
Oil & Grease	2001	5520-Oil and Grease	B, F	No
Total Phosphorus	1999	4500-P	E,F, G, H	No
Nitrate	2000	4500-NO ₃	D	No
TSS	1997	2540-TSS	B,C,D,E,F	Yes
Ammonia as N	1997	4500-NH ₃	B,C,D,E,F,G,H	Yes
Cyanide	1999	4500-CN	G	No
Hydronium ion, pH	2000	4500-H	B	No
CBOD, BOD	2001	5210	B	No
Total residual Chlorine	2000	4500-Cl	D,E,B,C,F,G	No

40 CFR 136 03-12-2007

TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter	Methodology ¹	Reference (method number or page)					
		EPA ² 816	Standard methods (1980, 1985)	Standard methods (2001)	Standard methods online	ASTM	
1. Acidity, as CaCO ₃ , mg/L	Electrometric endpoint or filtered endpoint endpoint		2310 B(a)	2310 B(a)	2310 B(a)-87	D1087-02, 02	
2. Alkalinity, as CaCO ₃ , mg/L	Electrometric or colorimetric titration to pH 4.5, manual, or automatic		2320 B	2320 B	2320 B-87	D1087-02, 02	
3. Aluminum—Total*, mg/L	Digestion ³ followed by AA direct aspiration ⁴	3102 (Rev. 1974) ¹		3111 D		3111 D-99	
	AA furnace			3113 B		3113 B-99	
	STOFAA	200 B, Rev. 2.2 (1984)					
	ICP-AES ⁵	2007, Rev. 4.4 (1984)	3120 B	3120 B	3120 B-99		
	ICP-MS	2008, Rev. 5.4 (1994)					D5673-03
4. Ammonia (as N), mg/L	Direct Current Plasma (DCP) ⁶		3500-AI D	3500-AI B	3500-AI B-01	D4190-04, 99	
	Colorimetric (Electrode system B)		4000-NH ₃ B	4000-NH ₃ B	4000-NH ₃ B-87		
	Manure distillation (at pH 9.5) ⁷ followed by Resazurin ⁸	3501, Rev. 2.0 (1983)	4000-NH ₃ C (1980)		4000-NH ₃ C	4000-NH ₃ C-87	D1426-06, 03 (A)
	Titration		4000-NH ₃ C (1980)				
	Electrode		4000-NH ₃ C (1980) and 4000-NH ₃ E (1980) and 4000-NH ₃ F or G (1980)	4000-NH ₃ D or E	4000-NH ₃ D or E-87		D1426-06, 03 (B)
Automated phenols, or		3501 ¹ , Rev. 2.0 (1983)	4000-NH ₃ G (1980) and 4000-NH ₃ H (1980)	4000-NH ₃ G	4000-NH ₃ G-87		
Automated electrode or Chromatography						D6319-03	

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THERE'S Waldo!

Section 136.7 Lab QA

- * ... suitable QA/QC procedures...
- * ... QA/QC procedures are generally included in the method or may be found in the methods compendium... (Standard Methods)
- * "The permittee/lab shall follow these QA/QC procedures, as described in the method or methods compendium. (Standard Methods)
- * If the method lacks QA/QC...

Three QA Options

- * A. ... follow equivalent EPA procedures
- * B. Refer to QA/QC in consensus organization compendium. (Follow Standard Methods) didn't we have that on the previous slide?
- * **C. Follow the 12 Steps where applicable.**

12 Quality Control Elements

1. DOC – demonstration of capability
2. MDL – method detection level
3. LRB/MB – method blank
4. LFB – laboratory fortified blank (standard)
5. LFM/LFMD – laboratory fortified matrix/duplicate (spike)
6. Internal standards, surrogate standards or tracer – *only applies to organic analysis and radiochemistry*
7. Calibration- initial and continuing
8. Control charts or other trend analysis
9. Corrective action – root cause analysis
10. QC acceptance criteria
11. Definition of a batch (preparation and analytical)
12. Minimum frequency for conducting all QC elements
13. Unwritten 13th Step – SOP – Standard Operating Procedures need to be written and followed for all lab sampling and analyses

Not all of these items apply to all tests, there are many exceptions!



Can you defend what you do?

- * How do you interpret your Permit language or the Rule?
- * Can you defend that interpretation, will a judge or jury support you?
- * What do Regulators say and what is written?
 - * Is it clear?
 - * Don't be afraid to ask Why?
 - * Don't be afraid to ask for directives in writing.



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Tests Discussed Today

- * Ammonia
- * BOD/cBOD
- * Cl₂
- * pH
- * DO
- * Total Phosphorus
- * TSS
- * Settleable Solids
- * Temperature
- * E-coli



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What You Are Already Doing



- * Most Labs are doing lots of QA/QC stuff
- * Write down what you do...SOP
- * Summarize QC Data
 - * Table Form
 - * Average, Max, Min.
 - * Control Charts

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Demonstration of Capability

- * DOC
- * Standard Methods 1020.B.1
 - * As a minimum, include a reagent blank and at least 4 LFBs at a concentration between 10 times the MDL and the midpoint of a calibration curve.
- * Standard Methods 2020B.1.a, 4020B.1.a. & 5020.B.1.a
 - * Run a laboratory-fortified blank (LFB) at least four times and compare to the limits listed in the method
 - * LFB initial recovery limits = $\text{Mean} \pm (5.84 \times \text{Standard Deviation})$

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Demonstration of Capability

- * What tests does this apply to?
 - * Ammonia, BOD/CBOD, Chlorine, pH, DO, Total Phosphorus, TSS
- * Analyst needs to make up this standard, cannot be bought premade
- * Example: for ammonia, the analyst needs to make up 1.0 mg/L, not purchase pre-made 1.0 mg/L
 - * Analyst can make 1 L of 1.0 mg/L by diluting down from 100 mg/L or 1000 mg/L and then pour up 4-100 mL aliquots

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Demonstration of Capability

- * How often?
 - * Once for each analyst.
 - * Recommended yearly for backup analyst who does not perform tests frequently
 - * EPA highly recommends running every 2-3 years for every analyst
 - * Each analyst should have a file kept on their training within and for the lab.
 - * Something to keep along with these records is a signed form (documentation) that analyst has read and understands all appropriate SOPs and Methods.

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Demonstration of Capability

- * **2014 Update**
 - * **DMRQA's were removed as acceptable DOC**
 - * **Analyst have had a year, there should be at least 4 standards that have been analyzed and within limits to demonstrate capability.**

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Method Detection Level

- * MDL
 - * Standard Methods 1020.B.4
 - * As a starting point for selecting the concentration to use when determining the MDL, us an estimate of five times the estimated true detection level
 - * Ideally, prepare and analyze at least seven portions of this solution over a 3-day period to ensure the MDL determination is more representative of routine measurements as performed in the laboratory

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Method Detection Level

- * Standard Methods 1020.B.4 - continued
 - * Recommended that the replicate measurements should be in the range of one to five times the estimated MDL, and recoveries of the known addition should be between 50-150%, with RSD (relative standard deviation) values \leq 20%
- * Standard Methods 4020.B.1.b
 - * Ideally use pooled data from several analysts rather than data from one analyst

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Method Detection Level

- * What tests does this apply to?
 - * Ammonia, Chlorine, Total Phosphorus
- * How often?
 - * Annually

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What the heck IS an MDL study?

- * It is a calculation that statistically gives the lowest concentration that a lab/facility can “see”, that is detect an analyte
- * Not practical for many analyses
- * It is a bit tricky the first time, but KEEP RECORDS so next year it will be a breeze.
- * Fresh samples prepared daily are preferred and it is recommended that samples are run over 3 days to give a more accurate account of how samples are run.

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How MDL Studies are Performed

- * Make seven very low level blank spikes (can be lower than the lowest point on your curve)
- * Analyze all seven over several days and calculate the standard deviation
- * Multiply the standard deviation by the “student t” for 7 values (3.14)
- * You cannot “cherry pick” your results, they must be 7 samples in a row

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MDL Calculations

- * The result is the MDL (method detection level)
- * The MDL must be greater than 1/10 the concentration of each spike
 - * Example: if the spike was 3, the MDL cannot be lower than 0.3 (3 divided by 10)
- * Keep up with the best spike value used for your MDL study so you don't have to go through several attempts each year
- * **2014 Update – this is your reporting limit**

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Good MDL Values for Cl₂

Date	Analyst	Number	True Value	Value Read	% Recovery (50-150%)
1/28/2013	SEP	1	0.05	0.09	180.00
1/28/2013	SEP	2	0.05	0.07	140.00
1/28/2013	SEP	3	0.05	0.07	140.00
1/30/2013	SEP	4	0.05	0.08	160.00
1/30/2013	SEP	5	0.05	0.08	160.00
2/1/2013	SEP	6	0.05	0.07	140.00
2/1/2013	SEP	7	0.05	0.08	160.00
Standard Deviation			0.007559289		
Relative Standard Deviation (RSD)			9.7990789 (Needs to be ≤ 20%)		
MDL			0.0237362		

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Laboratory Fortified Blank

- * Standard Methods 2020.B.2.e – TSS
 - * Using stock solutions, prepare fortified concentrations so they are within the calibration curve
- * Standard Methods 4020.B.2.e – Ammonia, BOD/CBOD, Chlorine, Phosphorus
 - * Calculate percent recovery, plot control charts and determine control limits
 - * **More control chart info later**
- * What tests does this apply to?
 - * Ammonia, BOD/CBOD, Chlorine, Total Phosphorus, TSS

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Laboratory Fortified Blank

- * How often?
- * For samples that need to be analyzed on a 5% basis or once for every 20 samples follow these criteria:
 - * *Influent and Effluent are 2 different samples*
 - * If a permit stated that 3 analyses per week, we would allow for a LFB to be analyzed at least once per month.
 - * Pick a date and be consistent, the 1st of every month or the 1st Thursday of every month. Mark your calendar!!
 - * If a permit stated 5 analyses per week, we would allow twice a month.
 - * Pick a date and be consistent, the 1st and 15th of every month or the 1st and 3rd Thursday of every month. Mark your calendar!!

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Laboratory Fortified Matrix and Duplicate

- * LFM/LFMD
- * Also known as a spike and spike dup
- * Standard Methods 1020.B.7
 - * A laboratory matrix (LFM) is an additional portion of a sample to which a known amount of the analyte of interest is added before sample preparation
 - * The LFM is used to evaluate analyte recovery in a sample
 - * Sample batch = 5% basis
 - * Add a concentration less than or equal to the midpoint of the calibration curve
 - * Preferably the same concentration as the LFB (laboratory fortified blank)

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Laboratory Fortified Matrix and Duplicate

- * Standard Methods 4020.B.2.g
 - * When appropriate for the analyte, include at least one LFM/LFMD ... with each batch of 20 samples
 - * Add a known concentration of analyte (ideally from a second source) to a randomly selected routine sample. Calculate percent recovery and relative percent difference, plot control charts and determine control limits for spikes at different concentrations
- * What tests does this apply to?
 - * Ammonia and Total Phosphorus

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Laboratory Fortified Matrix and Duplicate

- * How often?
 - * For samples that need to be analyzed on a 5% basis or once for every 20 samples follow these criteria:
 - * *Influent and Effluent are 2 different samples*
 - * If a permit stated that 3 analyses per week, we would allow for a spike to be analyzed at least once per month.
 - * Pick a date and be consistent, the 1st of every month or the 1st Thursday of every month. Mark your calendar!!
 - * If a permit stated 5 analyses per week, we would allow twice a month.
 - * Pick a date and be consistent, the 1st and 15th of every month or the 1st and 3rd Thursday of every month. Mark your calendar!!

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Laboratory Fortified Matrix and Duplicate

- * Also called a Matrix Spike/Matrix Spike Duplicate (MS/MSD)
 - * Calculate RPD between Spike and Spike Dup
- * Shows if there are interferences in the effluent matrix
- * **2014 Update – Spike volume should be less than 1% of the volume.**
 - * **Example: spike with 1 mL of 1000 mg/L into 100 mL sample will equal a 10 mg/L increase in ammonia concentration.**

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Duplicate

- * Dup
- * Not a part of the 12 Steps of QA, an addition from the State of TN
- * Standard Methods 1020.B.8
 - * As a minimum, include one duplicate sample with each sample set or on a 5% basis
- * Standard Methods 1020.B.12
 - * Calculate the RPD (relative percent difference)
 - * Equal to or less than 20% RPD

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Duplicate – TSS & Sett. Solids

- * Standard Methods 2020.B.2.f
 - * Include at least one duplicate for each matrix type daily or with each batch of 20 or fewer samples
- * Standard Methods 2540.A.2
 - * To aid in quality assurance, analyze samples in duplicate.
 - * Dry samples to constant weight if possible.
 - * This entails multiple drying-cooling-weighing cycles for each determination... more info later in presentation.
- * Standard Methods 2540.D.3.c
 - * Analyze at least 10% of all samples in duplicate

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Duplicate – Cl₂, pH and DO

- * Standard Methods 4020.B.2.f
 - * Randomly select routine samples to be analyzed twice
 - * Process duplicate sample independently through the entire sample preparation and analysis
 - * Include at least one duplicate for each matrix type daily or with each batch of 20

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Duplicate – BOD/CBOD

- * Standard Methods 5020.B.2.f
 - * Randomly select routine samples to be analyzed twice
 - * Process duplicate sample independently through the entire sample preparation and analysis
 - * Include at least one duplicate for each matrix type daily or with each batch of 20

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Duplicate

- * What tests does this apply to?
 - * BOD/CBOD, Chlorine, pH, DO, TSS and Settleable Solids
- * How often?
 - * For samples that need to be analyzed on a 5% basis or once for every 20 samples follow these criteria: (10% would be once every 10 samples for TSS)
 - * If a permit stated that 3 analyses per week, we would allow for a dup to be analyzed at least once per month.
 - * Pick a date and be consistent, the 1st of every month or the 1st Thursday of every month. Mark your calendar!!
 - * If a permit stated 5 analyses per week, we would allow twice a month.
 - * Pick a date and be consistent, the 1st and 15th of every month or the 1st and 3rd Thursday of every month. Mark your calendar!!

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Initial Calibration Verification & Continuing Calibration Verification

- * ICV
 - * Standard Methods 1020.B.11.b
 - * Perform initial calibration using at least three concentrations of standards for linear curves
 - * Calibrate meter (DO, pH or ISE) or verify balance, thermometer and colorimeter/spectrophotometer

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Initial Calibration Verification & Continuing Calibration Verification

- * CCV
 - * Standard Methods 1020.B.11.c
 - * Analysts periodically use a calibration standard to confirm that the instrument performance has not changed significantly since initial calibration.
 - * Verify calibration by analyzing one standard at a concentration near or at the mid-point of the calibration range.
 - * Verify the calibration (especially if preset by manufacturer) at beginning of day, after every 10 readings and at the end of the batch
 - * Daily (day of)

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ICV - TSS

- * Standard Methods 2020.B.2.a
- * Check instrument balances daily
- * Standard Methods 9020.B.4.b
 - * Service balances annually or more often as conditions change or problems occur
 - * Check balances routinely, preferably daily before use, with at least two working weights that bracket the normal usage range (e.g. ANSI/ASTM Class 1 or NIST Class S accompanied by appropriate certificate) for accuracy, precision and linearity.
 - * Record results along with date and technicians initials
 - * Recertify reference weights as specified in the certificate of calibration or at least every 5 years.



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ICV - Temperature

- * Standard Methods 9020.B.4.a
 - * Annually, or preferably, semiannually check accuracy of all working temperature sensing devices, such as liquid-in-glass thermometers, thermocouples and temperature-recording instruments at the use temperature against a certified National Institute of Standards and Technology (NIST) thermometer or one traceable to NIST and conforming to NIST specifications.
 - * Record calibration results, along with the date and the technician's signature, in a quality control logbook.
 - * Mark the necessary calibration correction factor on each temperature measuring device so that only calibrated-corrected temperature values are recorded.
 - * Verify accuracy of the reference certified thermometer as specified on the certificate of calibration or at least every 5 years.

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ICV –Ammonia, BOD/CBOD, Chlorine, pH, DO, Phosphorus

- * Standard Methods 4020.B.2.a
 - * Calibrate initially with at least one blank and three calibration standards
 - * The appropriate linear correlation coefficient for standard concentration-to-instrument response should be greater than or equal to 0.995.
 - * The back-calculated and true concentrations should agree within $\pm 10\%$.

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CCV –Ammonia, BOD/CBOD, Chlorine, pH, DO, Phosphorus

- * Standard Methods 4020.B.2.b
 - * Verify calibration by periodically analyzing a calibration standard and calibration blank during a run – typically after each batch of 10 samples and at the end of the run.
 - * For the calibration verification to be valid, check standards must be within 10% of its true value

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ICV –Probes for Ammonia

- * Standard Methods 4500-NH₃ D.4.a.
 - * Prepare a series of standard solutions covering the concentrations of 1000, 100, 10, 1 and 0.1 mg NH₃-N/L
- * Standard Methods 4500-NH₃ D.4.b.
 - * Calibrate from lowest to highest concentration.
 - * Wait until the reading has stabilized (at least 2-3 min) before recording millivolts for standards and samples containing ≤ 1 mg NH₃-N/L.
- * Standard Methods 4500-NH₃ D.4.c.
 - * If the electrode is functioning properly a tenfold change of NH₃-N concentration produces a potential change of about 59 mV.

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ICV/CCV –Chlorine

- * Prepare a set of Chlorine Standards or Potassium Permanganate (KMnO_4) standards in accordance with “Guidance for Secondary Standards use in Calibration” monthly.
- * Initial – Chlorine Standards or Potassium Permanganate (KMnO_4) standards monthly.
- * Continuing – Chlorine Standards, Potassium Permanganate (KMnO_4) standards or gel standards daily (day of).

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ICV/CCV –Chlorine

- * Secondary standards (gel standards) are specifically designed to verify the instrument's calibration and to check the instrument's performance.
- * They are not intended to be used to create calibration curves or to calibrate the instrument.
- * Because the DPD reagent cannot be mixed with the gel standards, the quality and the reaction time of the reagent cannot be assessed.
- * For these reasons gel standards cannot take the place of primary standards.

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ICV/CCV –Chlorine

- * The analyst is responsible for the following:
 - * Preparing the calibration curve for each instrument once per month at a minimum, before the use of new DPD reagents, or the use of new gel standards
 - * Recording reagent lot #'s for reagents and standards
 - * Recording calibration concentrations
 - * Verified the calibration curve using a minimum of one blank and two gel standards that bracket the expected sample concentration
 - * Recording all verification data

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ICV/CCV –Chlorine

- * Stock Standard Solution
 - * 0.891 grams of reagent grade $KMnO_4$ in 1000 mL vol. flask made to mark with deionized water.
 - * Deionized water must never be stored in plastic containers or exposed to airborne contamination.
 - * Store the stock solution in amber bottle in a cool area.
 - * The typical shelf life of the stock solution is six (6) months.
 - * If solids appear in the solution, do not use.
 - * ***Avoid leaving the cap off for extended periods of time and avoid contamination.***

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ICV/CCV –Chlorine

- * Intermediate (Working) Standard Solution (10 mg/L)
 - * 10 mL of STOCK made in 1000 mL vol. flask made to mark with deionized water.
 - * The flask should be labeled with the name, $KMnO_4$, date of preparation, initials of who made it.
 - * This information should also be entered into a logbook.
 - * **The intermediate stock solution should be stable for approximately 5 days if kept cool and away from light.**

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ICV/CCV –Chlorine

- * Calibration Standard Solutions
 - * Four to five calibration standard solutions should be made according to the table below to create a calibration curve once per month at a minimum.
 - * The R^2 (regression) of the curve should correlate to 0.995 or better.
 - * This curve is then used to check or calibrate the instrument.
 - * Gel standards are run against the curve and must agree to within + 10%.
 - * **The working solution should be stable for approximately 2 hours and will fade rapidly (within 15 minutes) if chlorine demand-free water is not used.**

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ICV/CCV –Chlorine

* A target value (e.g. permit value for a facility) should be known and three gel standards, 0.00 mg/L, blank, and two other standards (a low and a high standard) that bracket the target value should be chosen.



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ICV/CCV –Chlorine

mL Working Standard Diluted w/Deionized water	Chlorine Equivalent mg/L
20 mL (vol. Pipette) to 100 mL (vol. flask)	2.0 mg/L
10 mL (vol. Pipette) to 100 mL (vol. flask)	1.0 mg/L
5 mL (vol. Pipette) to 100 mL (vol. flask)	0.5 mg/L
1 mL (vol. Pipette) to 100 mL (vol. flask)	0.1 mg/L
1 mL (vol. Pipette) to 200 mL (vol. flask)	0.05 mg/L
1 mL (vol. Pipette) to 500 mL (vol. flask)	0.02 mg/L
100 mL of deionized water	0.00 mg/L

Don't forget to use DPD on Potassium Permanganate standards

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ICV/CCV – Phosphorus

- * ICV/CCV – does not go through digestion
- * LFM and LFB – does go through digestion

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Accuracy Control Charts

- * Standard Methods 1020 B.13.a
 - * The accuracy chart for QC samples (e.g., reagent blanks, LFBs, calibration check standards and LFM) is constructed from the average and standard deviation of a specified number of measurements of the analyte of interest.
 - * The accuracy chart includes upper and lower warning levels (WL) and upper and lower control levels (CL).
 - * Common practice is to use $\pm 2s$ and $\pm 3s$ limits for the WL and CL, respectively, where s represents standard deviation.

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BOD Accuracy Control Charts

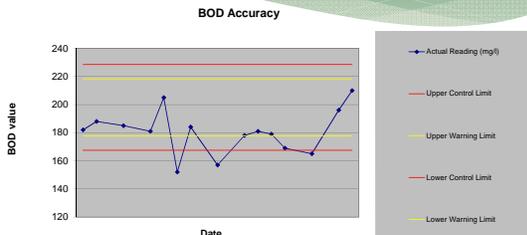
Date	Reading (mg/l)	Standard Value	Difference	*Upper Limit	*Lower Limit	*2/3 Upper	*2/3 Lower
8/22	182	198	-16	228.5	167.5	218.33	177.67
8/23	188	198	-10	228.5	167.5	218.33	177.67
8/25	185	198	-13	228.5	167.5	218.33	177.67
8/27	181	198	-17	228.5	167.5	218.33	177.67
8/28	205	198	7	228.5	167.5	218.33	177.67
8/29	152	198	-46	228.5	167.5	218.33	177.67
8/30	184	198	-14	228.5	167.5	218.33	177.67
9/1	157	198	-41	228.5	167.5	218.33	177.67
9/3	178	198	-20	228.5	167.5	218.33	177.67
9/4	181	198	-17	228.5	167.5	218.33	177.67
9/5	179	198	-19	228.5	167.5	218.33	177.67
9/6	169	198	-29	228.5	167.5	218.33	177.67
9/8	165	198	-33	228.5	167.5	218.33	177.67
9/10	196	198	-2	228.5	167.5	218.33	177.67
9/11	210	198	12	228.5	167.5	218.33	177.67

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BOD Accuracy Control Charts



* Control and warning limits are based on ± 30.5 mg/l, the standard value of the glucose solution.

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Precision Control Charts

- * Standard Methods 1020 B.13.b
- * The precision chart also is constructed on the average and standard deviation of a specified number of measurements (e.g., %RSD [relative standard deviation] or RPD) for a replicate or duplicate analyses of the analyte of interest.

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BOD Precision Control Charts

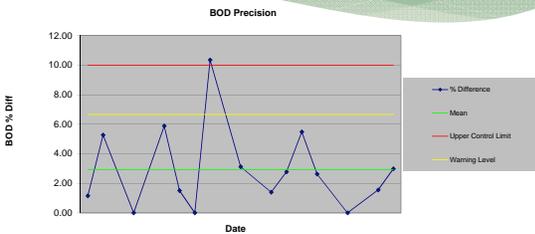
Date	#1 (mg/l)	#2 (mg/l)	Avg. (mg/l)	Diff. (Range)	% Diff.	2 X SD	3 X SD	Mean	Max Variat.	2/3 Max Var.
8/22	87	86	86.5	1.0	1.16	5.63	8.45	2.94	10.00	6.67
8/23	78	74	76.0	4.0	5.26	5.63	8.45	2.94	10.00	6.67
8/25	62	62	62.0	0.0	0.00	5.63	8.45	2.94	10.00	6.67
8/27	70	66	68.0	4.0	5.88	5.63	8.45	2.94	10.00	6.67
8/28	67	66	66.5	1.0	1.50	5.63	8.45	2.94	10.00	6.67
8/29	76	76	76.0	0.0	0.00	5.63	8.45	2.94	10.00	6.67
8/30	61	55	58.0	6.0	10.34	5.63	8.45	2.94	10.00	6.67
9/1	65	63	64.0	2.0	3.13	5.63	8.45	2.94	10.00	6.67
9/3	72	71	71.5	1.0	1.40	5.63	8.45	2.94	10.00	6.67
9/4	73	71	72.0	2.0	2.78	5.63	8.45	2.94	10.00	6.67
9/5	75	71	73.0	4.0	5.48	5.63	8.45	2.94	10.00	6.67
9/6	77	75	76.0	2.0	2.63	5.63	8.45	2.94	10.00	6.67
9/8	83	83	83.0	0.0	0.00	5.63	8.45	2.94	10.00	6.67
9/10	65	64	64.5	1.0	1.55	5.63	8.45	2.94	10.00	6.67
9/11	66	68	67.0	2.0	2.99	5.63	8.45	2.94	10.00	6.67
					2.94	mean				
					2.82	St.Dev.				

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BOD Precision Control Charts



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Control Charts

- * **2014 Update** - Create and maintain control charts if you have 20-30 data points within 90 days.
- * If you do not meet the above criteria, follow QC Acceptance Criteria below.
 - * Blanks < MDL
 - * LFB $\pm 15\%$
 - * ICV/CCV $\pm 10\%$
 - * LFM/LFMD $\pm 20\%$
 - * RPD < 20%
 - * Reporting limit = MDL

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Corrective Action

- * Standard Methods 1020 B.15
- * QC data that are outside the acceptance limits or exhibit a trend are evidence of unacceptable error in the analytical process.
- * Take corrective action promptly to determine and eliminate the source of error.
- * Do not report data until the cause of the problem is identified and either corrected or qualified (see Table 1020:II)

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Corrective Action

- * The corrective action plan needs to be in your SOP for each method on what to do if your QC tests fail or are out of range
- * If you have a "boo boo", write down how you fixed it
- * Any issues should be recorded and a sentence on how it can be prevented, if possible, in the future
- * Common problems and their corrections should be covered in your Standard Operating Procedures (SOP)
 - * If you see things frequently, you can give them qualifiers that are noted in your SOP;
 - * R = rain event
 - * D = bad dilution, etc.

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QC Acceptance

- * Have in SOP for each method the acceptance ranges for standards, duplicates, spikes, etc. and make sure they match the method requirements.
- * If not mentioned in method, these are the accepted criteria for QC:
 - * Blank < reporting limit
 - * LFB $\pm 15\%$
 - * MS/MSD $\pm 20\%$
 - * ICV/CCV $\pm 10\%$
 - * RPD < 20%
 - * Reporting limit = MDL

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Batch Size

- * Each "Batch" could be daily (day of), every 10 samples or every 20 samples.
- * Check method
- * *Influent and Effluent are 2 different samples*
- * If you sample only once a month, need to run QC each time.
 - * Once per month is minimum requirement

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Batch Size

- * For samples that need to be analyzed on a 5% basis or once for every 20 samples follow these criteria:
 - * If a permit stated that 3 analyses per week, how many samples would that be a week?
 - * TSS and BOD would be 6, Cl₂ would be 3
 - * If a permit stated 5 analyses per week, how many samples would that be a week?
 - * TSS and BOD would be 10, Cl₂ would be 5

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QC Frequency

- * Usually lumped in with the definition of a “batch” and should be in the SOP of some kind

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Standard Operating Procedure

- * Here's that “13th Step”, your SOP
- * All procedures must be documented in some type of SOP
- * It can be very simple but must provide the information necessary for someone who is not familiar with the test to perform it
 - * Step by step instructions on how and where to collect the samples, how to run the test and how to report values.
- * It must include the QC Acceptance Criteria, the definition of a “Batch” and the minimum frequency of QC checks

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Ammonia SM4500-NH₃ D -1997

- * 136 Table 1B
 - * Distillation Required
 - * Footnote #6
 - * Comparability Study
 - * Follow Standard Methods
- * See page 29,784 of the 136 Rule, Footnote #6



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40 CFR 136 05-21-2012 Table 1B

- 21st & 22nd Ed. Method 4500-NH₃ D: "Sample distillation is unnecessary."
- Footnote 6 – "Manual distillation is not required if comparability data on representative effluent samples are on file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies. In general, the analytical method should be consulted regarding the need for distillation. If the method is not clear, the laboratory may compare a minimum of 9 different sample matrices to evaluate the need for distillation. For each matrix, a matrix spike and matrix spike duplicate are analyzed both with and without the distillation step. (A total of 36 samples, assuming 9 matrices). If results are comparable, the laboratory may dispense with the distillation step for future analysis. Comparable is defined as < 20% RPD for all tested matrices). Alternatively the two populations of spike recovery percentages may be compared using a recognized statistical test."

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Ammonia SM4500-NH₃ D -1997

- * Standard Methods
 - * 4500-NH₃ A.1 – In general, direct manual determination of low concentrations of ammonia is confined to drinking waters, clean surface or groundwater and good-quality nitrified wastewater effluent.
 - * 4500-NH₃ D.1.b. – Sample distillation is unnecessary.
- * Tennessee recommends that one sample is run yearly to compare the distilled and undistilled results and that the results are within 20% of each other.
 - * Note – if distilled sample and undistilled sample are below detection limit, you cannot calculate the percent difference.

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Ammonia SM4500-NH₃ D -1997

- * DOC
- * MDL
- * LRB
- * LFB
- * LFM/LFMD
- * ICAL/CCV
- * Control Charts
- * Corrective Action
- * QC Acceptance
- * Batch Size
- * QC Frequency



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Ammonia SM4500-NH₃ D -1997

- * Demonstration of Capability (DOC)
 - * Run a laboratory-fortified blank (LFB) at least four times and compare to the limits listed in the method
 - * No limits listed for ammonia
- * Real people language: each operator running this test need to analyze 4 samples of an Ammonia Standard at a concentration around 1.0 mg/L.
- * Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.
- * Recommend backup analyst do this once a year.

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Ammonia SM4500-NH₃ D -1997

- * MDL- Estimated Detection Level=0.03mg/L
- * From SM 1030 C.
 - * 0.03mg/L * 5= 0.15 mg/L- MDL
 - * Make a 0.15mg/L standard
 - * Analyze 7 portions over ≥ 3 days
 - * Calculate standard deviation (s)
 - * $n1 \Sigma + n2 \Sigma + n3 \Sigma + \dots + n7 \Sigma + 2^{nd} \sigma n = s$
 - * $s * 3.14 = MDL$

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Ammonia SM4500-NH₃ D -1997

- * Method Blank
 - * Real people language: analyze distilled water as a sample by going through distillation (if you still distill samples) and using ISA (ion strength adjuster)
 - * Target value is less than MDL (reporting limit)
 - * Run on a 5% basis, one for every 20 samples
- * Laboratory Fortified Blank
 - * Real people language: analyze an ammonia standard at a concentration around 5 mg/L
 - * Run on a 5% basis (see batch size for more information).

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Ammonia SM4500-NH₃ D -1997

- * Lab fortified matrix and duplicate (spike& spike dup)
- * Real people language – add a known amount of ammonia to a sample and expect that amount to be added to your sample concentration and repeat process for LFMD.
- * Run on a 5% basis (see batch size for more information).
- * Calculate RPD between spiked sample and spiked duplicate, target value should be close to the first value and have a small RPD (less than 20%).
- * **2014 Update** - Spike volume should be less than 1% of the volume.
 - * Example: spike with 1 mL of 1000 mg/L into 100 mL sample will equal a 10 mg/L increase in ammonia concentration.

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Ammonia SM4500-NH₃ D -1997

- * Initial Calibration
 - * Standards that bracket the expected concentrations
 - * Standards should not exceed an order of magnitude such as 1,10,100,1000
 - * Real people language: calibrate probe daily (day of) with at least 3 standards
 - * **2014 Update** – analyze a 10 mg/L standard as a sample after calibration and before samples to verify initial calibration (ICV)
- * Calibration Verification
 - * Real people language: analyze 10 mg/L at the end of samples daily (day of) to verify calibration is still valid

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Ammonia SM4500-NH₃ D -1997

- * **2014 Update** - Create and maintain control charts if you have 20-30 data points within 90 days.
- * If you do not meet the above criteria, follow QC Acceptance Criteria below.
 - * Blanks < MDL
 - * LFB ± 15%
 - * ICV/CCV ± 10%
 - * LFM/LFMD ± 20%
 - * RPD < 20%
 - * Reporting limit = MDL

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BOD₅/cBOD₅ SM5210 B – 2001 & Hach Method 10360

- * DOC
- * LRB
- * LFB
- * Dup
- * ICAL/CCV
- * Control Charts
- * Corrective Action
- * QC Acceptance
- * Batch Size
- * QC Frequency



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BOD₅/cBOD₅ SM5210 B – 2001 & Hach Method 10360

- * Minimum DO depletion (including seed bottles) of 2.0 mg/l
- * Minimum residual DO of at least 1.0 mg/l
- * Dilution water quality check (nutrient, mineral, buffer) must not be more than 0.2 mg/l (0.1 is preferred)
- * Seed control of three dilutions. Smallest to give at least 2.0 mg/l depletion and the largest to at least 1.0 mg/l residual...

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BOD₅/cBOD₅ SM5210 B – 2001 & Hach Method 10360

- * Demonstration of Capability (DOC)
 - * Run a laboratory-fortified blank (LFB) at least four times and compare to the limits listed in the method
 - * Real people language: Each operator running this test needs to analyze 4 samples of GGA at a concentration of 198±30.5 mg/L
 - * Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.
 - * Recommend backup analyst do this once a year.

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BOD₅/cBOD₅ SM5210 B – 2001 & Hach Method 10360

- * Method Blanks
 - * Real people language: analyze dilution water
 - * Preferably one at the beginning and one at end
 - * **2014 Update – Removed** – For reporting CBOD₅ results, then also add one Nitrification Inhibitor (NI Blank)
 - * Run on daily (day of)
 - * Target value is less than 0.20 mg/L (preferably less than 0.10 mg/L)

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BOD₅/cBOD₅ SM5210 B – 2001 & Hach Method 10360

- * Laboratory Fortified Blank
 - * Real people language: analyze a Glucose/Glutamic Acid (GGA) standard at a concentration of 198±30.5 mg/L
 - * Run on a 5% basis, one for every 20 samples
 - * **2014 Update** - If permit requires cBOD, add nitrification inhibitor (NI) to one GGA bottle once/quarter (or more often if the Lot # of NI changes), which should be equal to 164 ±30.7 mg/L

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BOD₅/cBOD₅ SM5210 B – 2001 & Hach Method 10360

- * Duplicate
 - * Analyze 2 samples for BOD or CBOD
 - * Example, if you run 6, 9 and 12 mL on your raw/influent sample, run a second 9 mL sample.
 - * You would end up with a total of 4 bottles for your raw/influent sample
 - * Run on a 5% basis, one for every 20 samples
 - * Calculate %RPD, ≤ 20%
 - * **2014 Update - For reporting purposes, average results that meet method criteria.**

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BOD₅/cBOD₅ SM5210 B – 2001 & Hach Method 10360

- * Initial Calibration (ICV)
 - * Calibrate daily (day of) by following manufacturer's instructions
 - * Using barometric pressure is best
- * Continuing Calibration (CCV)
 - * Prepare dilution water that is air-saturated and analyze bottles and compare to the theoretical dissolved oxygen concentration (± 0.2 mg/L).
 - * Same as DO CCV if using a different probe



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BOD₅/cBOD₅ SM5210 B – 2001 & Hach Method 10360

- * Corrective Action - 1020 B.5., B.8., & B.15.
- * 5210 B.7.b. – Identify results in the test reports when any of the following quality control parameters is not met:
 - * Dilution water exceeds 0.20 mg/L (5210B.6c)
 - * Glucose-glutamic acid check falls outside of acceptable limits (5210B.6b)
 - * Test replicates show more than 30% difference between high and low values
 - * Seed control samples do not meet the above criteria in all dilutions (5210B.6d) or
 - * Minimum DO is less than 1.0 mg/L (5210B.7a3)

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BOD₅/cBOD₅ SM5210 B – 2001 & Hach Method 10360

- * QC Acceptance Criteria
 - * Blanks < 0.20 mg/L
 - * GGA = 198 ± 30.5 mg/L (if running cBOD, add NI to one bottle once/quarter or more often if NI Lot# changes, and it should = 164 ± 30.7 mg/L)
 - * RPD < 20%
 - * Minimum of three dilutions for each sample, at least one sample must have valid data with at least 2.0 mg/L depletion and a residual of 1.0 mg/L

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Total Residual Chlorine SM4500-Cl G - 2000, DPD

- * DOC
- * MDL
- * LRB
- * LFB
- * Dup
- * ICAL/CCV
- * Control Charts
- * Corrective Action
- * QC Acceptance
- * Batch Size
- * QC Frequency



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Total Residual Chlorine SM4500-Cl G - 2000, DPD

- * Demonstration of Capability (DOC)
 - * Run a laboratory-fortified blank (LFB) at least four times and compare to the limits listed in the method
 - * No limits listed for chlorine
 - * Real people language: each operator running this test need to analyze 4 samples of a Chlorine Standard or Potassium Permanganate (KMnO₄) at a concentration around 0.5 mg/L.
 - * Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.
 - * Recommend backup analyst do this once a year.

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Total Residual Chlorine SM4500-Cl G - 2000, DPD

- * Method Detection level
 - * HACH- Estimated Detection Level=0.02mg/L
 - * From SM 1030 C.
 - * 0.02mg/L * 5= 0.10 mg/L- MDL
 - * Make a 0.10 mg/L standard
 - * Analyze 7 portions over ≥ 3 days
 - * Calculate standard deviation (s)
 - * $n1 \Sigma + n2 \Sigma + n3 \Sigma + \dots + n7 \Sigma + 2^{nd} \sigma_{xn} = s$
 - * $s * 3.14 = MDL$

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Total Residual Chlorine SM4500-Cl G - 2000, DPD

- * Method Blank
 - Real people language: analyze distilled water as a sample by adding DPD powder pillow and waiting the 3-6 minutes before reading
 - * Target value is less than MDL
 - * **2014 Update – run on a 5% basis instead of daily**
- * Laboratory Fortified Blank
 - Real people language: analyze a chlorine standard or potassium permanganate (KMnO₄) at a concentration around 0.5 mg/L
 - * Run on a 5% basis, one for every 20 samples
- * Duplicates
 - Run on a 5% basis, one for every 20 samples
 - Calculate %RPD, ≤ 20%
 - **2014 Update – For reporting purposes, average sample and duplicate.**

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Total Residual Chlorine SM4500-Cl G - 2000, DPD

- * Initial Calibration
 - Prepare a set of chlorine standard or potassium permanganate (KMnO₄) in accordance with the Guidance for Secondary Standards Use in Calibration monthly.
 - * Once per month at minimum, before the use of new DPD reagents, or the use of new gel standards



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Total Residual Chlorine SM4500-Cl G - 2000, DPD

- * Stock Standard Solution
 - 0.891 grams of reagent grade KMnO₄ in 1000 mL vol. flask made to mark with deionized water.
 - Deionized water must never be stored in plastic containers or exposed to airborne contamination.
 - Store the stock solution in amber bottle in a cool area.
 - The typical shelf life of the stock solution is six (6) months.
 - If solids appear in the solution, **do not use**.

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Total Residual Chlorine SM4500-Cl G - 2000, DPD

- * Intermediate (Working) Standard Solution
 - * 10 mL of STOCK made in 1000 mL vol. flask made to mark with deionized water.
 - * The flask should be labeled with the name, $KMnO_4$, date of preparation, initials of who made it.
 - * This information should also be entered into a logbook.
 - * **The intermediate stock solution should be stable for approximately 5 days if kept cool and away from light.**

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Total Residual Chlorine SM4500-Cl G - 2000, DPD

- * Potassium Permanganate Standard Solution
 - * Care should be taken that the pipette and glassware are clean and thoroughly rinsed with deionized water to avoid contamination.
 - * Store only in glass container (preferably amber glass) never in plastic containers.
 - * The working solution should be remade if solids appear in the bottom of the container.

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Total Residual Chlorine SM4500-Cl G - 2000, DPD

- * Calibration Standard Solutions
 - * Four to five calibration standard solutions should be made according to the table below to create a calibration curve once per month at a minimum.
 - * The linear regression of the curve should correlate to 0.995 or better.
 - * This curve is then used to check or calibrate the instrument.
 - * Gel standards are run against the curve and must agree to within + 10%.
 - * **The working solution should be stable for approximately 2 hours and will fade rapidly (within 15 minutes) if chlorine demand-free water is not used.**

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Total Residual Chlorine SM4500-Cl G - 2000, DPD

- * Calibration Standard Solutions
 - * A target value (e.g. permit value for a facility) should be known and three gel standards, 0.00 mg/L, blank, and two other standards (a low and a high standard) that bracket the target value should be chosen.

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Total Residual Chlorine SM4500-Cl G - 2000, DPD

mL Working Standard Diluted w/Deionized water	Chlorine Equivalent mg/L
20 mL (vol. Pipette) to 100 mL (vol. flask)	2.0 mg/L
10 mL (vol. Pipette) to 100 mL (vol. flask)	1.0 mg/L
5 mL (vol. Pipette) to 100 mL (vol. flask)	0.5 mg/L
1 mL (vol. Pipette) to 100 mL (vol. flask)	0.1 mg/L
1 mL (vol. Pipette) to 200 mL (vol. flask)	0.05 mg/L
1 mL (vol. Pipette) to 500 mL (vol. flask)	0.02 mg/L
100 mL of deionized water	0.00 mg/L

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Total Residual Chlorine SM4500-Cl G - 2000, DPD

- * Calibration Verification
 - * Verify meter daily (day of) with secondary gel standards using a minimum of one blank and two gel standards that bracket the expected sample concentration



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Total Residual Chlorine SM4500-Cl G - 2000, DPD

- * **2014 Update** - Create and maintain control charts if you have 20-30 data points within 90 days.
- * If you do not meet the above criteria, follow QC Acceptance Criteria below.
 - * Blanks < MDL
 - * LFB \pm 15%
 - * ICV/CCV \pm 10%
 - * RPD < 20%
 - * Reporting limit = MDL

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pH SM4500-H⁺ B – 2000 Electrometric Method

- * DOC
- * Dup
- * ICAL/CCV
- * Corrective Action
- * QC Acceptance
- * Batch Size
- * QC Frequency



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pH SM4500-H⁺ B – 2000 Electrometric Measurement

- * Read to 1/10th units only, 0.0 s.u.
- * Demonstration of Capability (DOC)
 - * Run buffer at least four times and compare to the limits listed in the method
- * Real people language: each operator running this test need to calibrate and analyze 4 buffers at a pH of 7
- * Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.
- * Recommend backup analyst do this once a year.

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pH SM4500-H+ B – 2000 Electrometric Measurement

- * Initial Calibration
 - * Calibrate per manufactures instructions with fresh buffers daily (day of).
 - * **2014 Update – Analyze a 7 buffer solution as a sample after calibration and before samples to verify initial calibration (ICV), should be within ± 0.2 s.u.**
- * Calibration Verification
 - * Read 7 buffer after analyzing samples daily



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pH SM4500-H+ B – 2000 Electrometric Measurement

- * Duplicates of the sample
 - * Run on a 5% basis, one for every 20 samples
 - * Within ± 0.2 s.u.
 - * **2014 Update – For reporting purposes, all duplicates should be reported according to your permit limits. If your permit sets a minimum or maximum limit such as pH, then the minimum or maximum value should be reported even if falls outside your permit limit.**

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Reporting Examples Typical Permit Limits = 6.0 min & 9.0 max

- * Example 1:
 - * Sample read 5.9 and } Report Sample, fell outside of limits
 - * Duplicate read 6.1 }
- * Example 2:
 - * Sample read 8.9 and } Report Duplicate, fell outside of limits
 - * Duplicate read 9.1 }
- * Example 3:
 - * Sample read 6.2 and } QC failed, outside of 0.2 s.u.
 - * Duplicate read 6.5 } Reread sample and duplicate

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DO Hach Method 10360, LDO Measurement Oct. 2011

- * DOC
- * Dup
- * ICAL/CCV
- * Corrective Action
- * QC Acceptance
- * Batch Size
- * QC Frequency



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DO Hach Method 10360, LDO Measurement Oct. 2011

- * Demonstration of Capability
 - * Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.
 - * Hach Method 10360 9.2.1 – Prepare and measure four samples of air-saturated water according to section 7.2.
 - * 7.2.1 – Add approximately 1500 mL of organic-free water or BOD dilution water to a 2-L beaker or PET bottle
 - * 7.2.2 – Allow the water to equilibrate to room temperature. Room temperature should be approximately $20 \pm 3^\circ\text{C}$.
 - * 7.2.3 – With a steady gentle stream of filtered air ($\approx 10\text{-}40$ mL per minute), aerate the water for a minimum of 30 minutes. Alternatively, vigorously shake the reagent water or BOD dilution water for several minutes.

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DO Hach Method 10360, LDO Measurement Oct. 2011

- * Hach Method 10360 continued
 - * 7.2.4 – At the completion of aeration, let water re-equilibrate to room temperature ($20 \pm 3^\circ\text{C}$) for 30 minutes and note the barometric pressure **[uncorrected]** of the laboratory during preparation. The barometric temperature reading is used in the calculation and determination of the theoretical DO concentration for the preparation of air-saturated water.
 - * 7.2.5 – Transfer the aerated water to a BOD bottle until overflowing and stopper.
 - * 7.2.6 – Calculate the theoretical dissolved oxygen concentration using a dissolved oxygen table such as Hitchman...
- * Real people language – prepare dilution water that is air-saturated and analyze four bottles and compare to the theoretical dissolved oxygen concentration.

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Temp.	Barometric Pressure (mm Hg)																							
Temp. (C)	31.2	31.3	31.4	31.5	31.6	31.7	31.8	31.9	32.0	32.1	32.2	32.3	32.4	32.5	32.6	32.7	32.8	32.9	33.0	33.1	33.2	33.3	33.4	33.5
0	10.95	11.14	11.33	11.53	11.72	11.91	12.11	12.31	12.50	12.69	12.88	13.07	13.27	13.46	13.65	13.85	14.04	14.23	14.43	14.62				
1	10.64	10.83	11.02	11.21	11.39	11.58	11.77	11.96	12.15	12.34	12.52	12.71	12.9	13.09	13.28	13.46	13.65	13.84	14.03	14.22				
2	10.35	10.53	10.72	10.9	11.08	11.27	11.45	11.63	11.82	12	12.18	12.37	12.55	12.73	12.91	13.1	13.28	13.46	13.65	13.83				
3	10.07	10.25	10.43	10.61	10.79	10.96	11.14	11.32	11.5	11.68	11.86	12.03	12.21	12.39	12.57	12.75	12.93	13.11	13.28	13.46				
4	9.81	9.98	10.15	10.33	10.5	10.68	10.85	11.02	11.2	11.37	11.54	11.72	11.89	12.07	12.24	12.41	12.59	12.76	12.93	13.11				
5	9.55	9.72	9.89	10.06	10.23	10.4	10.57	10.74	10.91	11.08	11.25	11.42	11.59	11.75	11.92	12.09	12.26	12.43	12.6	12.77				
6	9.31	9.47	9.64	9.8	9.97	10.14	10.3	10.47	10.63	10.8	10.96	11.13	11.29	11.46	11.62	11.79	11.95	12.12	12.28	12.45				
7	9.08	9.24	9.4	9.56	9.72	9.88	10.04	10.2	10.37	10.53	10.69	10.85	11.01	11.17	11.33	11.49	11.65	11.82	11.98	12.14				
8	8.85	9.01	9.17	9.33	9.48	9.64	9.8	9.95	10.11	10.27	10.43	10.58	10.74	10.9	11.06	11.21	11.37	11.53	11.69	11.84				
9	8.64	8.79	8.95	9.1	9.25	9.41	9.56	9.71	9.87	10.02	10.18	10.33	10.48	10.64	10.79	10.94	11.1	11.25	11.41	11.56				
10	8.43	8.58	8.73	8.88	9.03	9.18	9.33	9.49	9.64	9.79	9.94	10.09	10.24	10.39	10.54	10.69	10.84	10.99	11.14	11.29				
11	8.24	8.38	8.53	8.68	8.82	8.97	9.12	9.26	9.41	9.56	9.71	9.85	10	10.15	10.29	10.44	10.59	10.73	10.88	11.03				
12	8.05	8.19	8.33	8.48	8.62	8.77	8.91	9.05	9.2	9.34	9.48	9.63	9.77	9.91	10.06	10.2	10.35	10.49	10.63	10.78				
13	7.86	8.01	8.15	8.29	8.43	8.57	8.71	8.85	8.99	9.13	9.27	9.41	9.55	9.69	9.83	9.97	10.11	10.26	10.4	10.54				
14	7.69	7.83	7.97	8.1	8.24	8.38	8.52	8.65	8.79	8.93	9.07	9.2	9.34	9.48	9.62	9.76	9.89	10.03	10.17	10.31				
15	7.52	7.66	7.79	7.93	8.06	8.2	8.33	8.47	8.6	8.74	8.87	9	9.14	9.27	9.41	9.54	9.68	9.81	9.95	10.08				
16	7.36	7.49	7.62	7.76	7.89	8.02	8.15	8.28	8.42	8.55	8.68	8.81	8.95	9.08	9.21	9.34	9.47	9.61	9.74	9.87				
17	7.2	7.33	7.46	7.59	7.72	7.85	7.98	8.11	8.24	8.37	8.5	8.63	8.76	8.89	9.02	9.15	9.28	9.41	9.54	9.66				
18	7.05	7.18	7.31	7.43	7.56	7.69	7.81	7.94	8.07	8.2	8.32	8.45	8.58	8.7	8.83	8.96	9.09	9.21	9.34	9.47				
19	6.91	7.03	7.16	7.28	7.41	7.53	7.66	7.78	7.9	8.03	8.15	8.28	8.4	8.53	8.65	8.78	8.9	9.03	9.15	9.28				
20	6.77	6.89	7.01	7.13	7.26	7.38	7.5	7.62	7.75	7.87	7.99	8.11	8.24	8.36	8.48	8.6	8.73	8.85	8.97	9.09				
21	6.63	6.75	6.87	6.99	7.11	7.23	7.35	7.47	7.59	7.71	7.83	7.95	8.07	8.19	8.31	8.43	8.55	8.67	8.79	8.91				
22	6.5	6.62	6.74	6.85	6.97	7.09	7.21	7.33	7.45	7.56	7.68	7.8	7.92	8.04	8.15	8.27	8.39	8.51	8.63	8.74				
23	6.37	6.49	6.61	6.72	6.84	6.95	7.07	7.19	7.3	7.42	7.53	7.65	7.77	7.88	8	8.11	8.23	8.35	8.46	8.58				
24	6.25	6.36	6.48	6.59	6.71	6.82	6.94	7.05	7.16	7.28	7.39	7.51	7.62	7.73	7.85	7.96	8.08	8.19	8.3	8.42				
25	6.13	6.24	6.35	6.47	6.58	6.69	6.81	6.92	7.03	7.14	7.25	7.37	7.48	7.59	7.7	7.81	7.93	8.04	8.15	8.26				
26	6.02	6.13	6.24	6.35	6.46	6.57	6.68	6.79	6.9	7.01	7.12	7.23	7.34	7.45	7.56	7.67	7.78	7.89	8	8.11				
27	5.9	6.01	6.12	6.23	6.34	6.45	6.56	6.67	6.77	6.88	6.99	7.1	7.21	7.32	7.43	7.53	7.64	7.75	7.86	7.97				
28	5.8	5.9	6.01	6.12	6.23	6.33	6.44	6.54	6.65	6.76	6.87	6.97	7.08	7.19	7.29	7.4	7.51	7.61	7.72	7.83				
29	5.69	5.8	5.9	6.01	6.11	6.22	6.32	6.43	6.53	6.64	6.74	6.85	6.95	7.06	7.16	7.27	7.38	7.48	7.59	7.69				
30	5.59	5.69	5.79	5.9	6	6.11	6.21	6.31	6.42	6.52	6.63	6.73	6.83	6.94	7.04	7.14	7.25	7.35	7.46	7.56				

Can be made at <http://water.usgs.gov/software/DOTABLES/>

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DO Hach Method 10360, LDO Measurement Oct. 2011

- * Calibration
 - * 7.1.1 – Add approximately 1 inch (2.54 cm) of reagent water to a clean BOD bottle and stopper).
 - * 7.1.2 – Shake vigorously for ~ 10 seconds.
 - * 7.1.3 – Allow for the BOD bottle and its contents to equilibrate to room temperature. Room temperature should be approximately 20 ± 3°C.
 - * 7.1.4 – The stopper may now be removed from the BOD bottle and the LBOD probe inserted for calibration purposes.
- * Real people language – calibrate daily (day of) by following manufacturer's instructions.

TDEC - Fleming Training Center 113

DO Hach Method 10360, LDO Measurement Oct. 2011

- * Duplicate
 - * Real people language – analyze 2 samples for DO, grab sample in a bucket and dip probe twice to get two readings
 - * Target value is to get close to the first value and have a small RPD
 - * **2014 Update – For reporting purposes, all duplicates should be reported according to your permit limits. If your permit sets a minimum, then the minimum value should be reported even if falls outside your permit limit.**

TDEC - Fleming Training Center 114

DO Hach Method 10360, LDO Measurement Oct. 2011

- * Continuing Calibration Verification (CCV) - daily (day of)
 - * 7.2.1 – Add approximately 1500 mL of organic-free water or BOD dilution water to a 2-L beaker or PET bottle
 - * 7.2.2 – Allow the water to equilibrate to room temperature. Room temperature should be approximately $20 \pm 3^\circ\text{C}$.
 - * 7.2.3 – With a steady gentle stream of filtered air ($\approx 10\text{-}40$ mL per minute), aerate the water for a minimum of 30 minutes. Alternatively, vigorously shake the reagent water or BOD dilution water for several minutes.
 - * 7.2.4 – At the completion of aeration, let water re-equilibrate to room temperature ($20 \pm 3^\circ\text{C}$) for 30 minutes and note the barometric pressure **[uncorrected]** of the laboratory during preparation. The barometric pressure reading is used in the calculation and determination of the theoretical DO concentration for the preparation of air-saturated water.

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DO Hach Method 10360, LDO Measurement Oct. 2011

- * Continuing Calibration Verification (CCV) continued
 - * 7.2.5 – Transfer the aerated water to a BOD bottle until overflowing and stopper.
 - * 7.2.6 – Calculate the theoretical dissolved oxygen concentration using a dissolved oxygen table such as Hitchman...
 - * 9.3.1 – Upon air calibration, prepare a calibration verification standard with each analytical batch of 20 samples or less in an 8 hour period.
 - * 9.4.3 – Initially and at the end of each analytical batch of samples, analyze a dilution water sample that is air-saturated

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DO Hach Method 10360, LDO Measurement Oct. 2011

- * Continuing Calibration Verification (CCV) continued
 - * Real people language – prepare dilution water that is air-saturated and analyze bottles and compare to the theoretical dissolved oxygen concentration (± 0.2 mg/L) after daily samples or with each batch of 20 samples or less in an 8-hour period.
- * Batch Size
 - * 9.3.1 – ... with each analytical batch of 20 samples or less in an 8 hour period.

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DO SM4500-O G – 2001 Membrane Electrode Method

- * DOC
- * Dup
- * ICAL/CCV
- * Corrective Action
- * QC Acceptance
- * Batch Size
- * QC Frequency



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DO SM4500-O G – 2001 Membrane Electrode Method

- * Demonstration of Capability
 - * Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.
 - * Follow Hach Method 10360 9.2.1 – Prepare and measure four samples of air-saturated water according to section 7.2.
 - * 7.2.1 – Add approximately 1500 mL of organic-free water or BOD dilution water to a 2-L beaker or PET bottle
 - * 7.2.2 – Allow the water to equilibrate to room temperature. Room temperature should be approximately 20 ± 3°C.
 - * 7.2.3 – With a steady gentle stream of filtered air (≈ 10-40 mL per minute), aerate the water for a minimum of 30 minutes. Alternatively, vigorously shake the reagent water or BOD dilution water for several minutes.

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DO SM4500-O G – 2001 Membrane Electrode Method

- * Hach Method 10360 continued
 - * 7.2.4 – At the completion of aeration, let water re-equilibrate to room temperature (20 ± 3°C) for 30 minutes and note the barometric pressure **[uncorrected]** of the laboratory during preparation. The barometric pressure reading is used in the calculation and determination of the theoretical DO concentration for the preparation of air-saturated water.
 - * 7.2.5 – Transfer the aerated water to a BOD bottle until overflowing and stopper.
 - * 7.2.6 – Calculate the theoretical dissolved oxygen concentration using a dissolved oxygen table such as Hitchman...
- * Real people language – prepare dilution water that is air-saturated and analyze four bottles and compare to the theoretical dissolved oxygen concentration.

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DO SM4500-O G – 2001 Membrane Electrode Method

- * Continuing Calibration Verification (CCV) continued
- * 7.2.5 – Transfer the aerated water to a BOD bottle until overflowing and stopper.
- * 7.2.6 – Calculate the theoretical dissolved oxygen concentration using a dissolved oxygen table such as Hitchman...
- * 9.3.1 – Upon air calibration, prepare a calibration verification standard with each analytical batch of 20 samples or less in an 8 hour period.
- * 9.4.3 – Initially and at the end of each analytical batch of samples, analyze a dilution water sample that is air-saturated
- * Real people language – prepare dilution water that is air-saturated and analyze bottles and compare to the theoretical dissolved oxygen concentration.

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Phosphorus SM4500-P B and E -1999

- * DOC
- * MDL
- * LRB
- * LFB
- * LFM/LFMD
- * ICAL/CCV
- * Control Charts
- * Corrective Action
- * QC Acceptance
- * Batch Size
- * QC Frequency



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Phosphorus SM4500-P B and E -1999

- * Demonstration of Capability (DOC)
- * Run a laboratory-fortified blank (LFB) at least four times and compare to the limits listed in the method
- * Real people language: each operator running this test need to analyze 4 samples of a Phosphorus Standard at a concentration around 0.5 mg/L.
- * Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.
- * Recommend backup analyst do this once a year.

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Phosphorus SM4500-P B and E -1999

- * MDL- Estimated Detection Level=0.01mg/L
 - * From SM 1030 C.
 - * 0.01 mg/L * 5= 0.05 mg/L~ MDL
 - * **Make a 0.05 mg/L standard**
 - * **Analyze 7 portions over ≥ 3 days**
 - * Calculate standard deviation (s)
 - * $n_1 \Sigma + n_2 \Sigma + n_3 \Sigma + \dots + n_7 \Sigma + 2^{nd} \sigma x n = s$
 - * $s^* 3.14 = MDL$

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Phosphorus SM4500-P B and E -1999

- * Method Blank – goes through digestion
 - * Real people language: analyze distilled water as a sample by going through digestion and reagent addition before reading
 - * Target value is less than reporting limit
 - * Reporting limit will be equal to your Method Detection Limit (MDL)
 - * Run on a 5% basis, one for every 20 samples
 - * Laboratory Fortified Blank – goes through digestion
 - * Real people language: analyze a phosphorus standard at a concentration around 0.5 mg/L
 - * Run on a 5% basis, one for every 20 samples

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Phosphorus SM4500-P B and E -1999

- * Lab fortified matrix & duplicate (spike& spike dup)
 - * 4020 B.2.g. – When appropriate for the analyte, include at least one LFM/LFMD daily or with each batch of 20 or fewer samples
 - * Add a known concentration of analyte (ideally from a second source) to a randomly selected routine sample without increasing its volume by more than 5%
 - * Calculate percent recovery and relative percent difference, plot control charts and determine control limits for spikes at different concentrations
 - * Real people language – add a known amount of phosphorus to a sample and expect that amount to increase your sample concentration
 - * Run on a 5% basis (1 for every 20 samples or once per month, whichever is more frequent)
 - * Calculate RPD between spiked sample and spiked duplicate, target value should be close to the first value and have a small RPD (less than 20%).
 - * Spike volume should be less than 1% of the volume.
 - * Example: spike with 0.1 mL of 100 mg/L into 10 mL sample will equal a 1 mg/L increase in phosphorus concentration.

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Phosphorus SM4500-P B and E -1999

- * Initial Calibration – does not go through digestion
 - * Analyze 2-3 different standards within the curve
 - * Run on a 5% basis, one for every 20 samples
- * Calibration Verification – does not go through digestion
 - * Analyze a mid-range phosphorous standard daily (day of)
 - * Hach's method range is 0.2-2.50 mg/L, a 1 mg/L would work at your daily check standard

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Phosphorus SM4500-P B and E -1999

- * **2014 Update** - Create and maintain control charts if you have 20-30 data points within 90 days.
- * If you do not meet the above criteria, follow QC Acceptance Criteria below.
 - * Blanks < MDL
 - * LFB $\pm 15\%$
 - * ICV/CCV $\pm 10\%$
 - * LFM/LFMD $\pm 20\%$
 - * RPD < 20%
 - * Reporting limit = MDL

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TSS SM2540 D – 1997 Dried at 103-105°C

- * DOC
- * LRB
- * LFB
- * Dup
- * ICAL
- * Corrective Action
- * QC Acceptance
- * Batch Size
- * QC Frequency



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TSS SM2540 D – 1997 Dried at 103-105°C

- * Demonstration of Capability (DOC)
 - * Run a laboratory-fortified blank (LFB) at least four times and compare to the limits listed in the method
- * Real people language: each operator running this test need to analyze 4 samples of an Total Suspended Solids Standards
- * Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.
- * Recommend backup analyst do this once a year.



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TSS SM2540 D – 1997 Dried at 103-105°C

- * Blanks
 - * Filter 100 mL of deionized/distilled water through a pre-washed/pre-dried/pre-weighed filter with each batch of 20 or fewer samples
 - * Run on a 5% basis, one for every 20 samples
 - * **2014 Update – Should be less than 2.5 mg/L**
- * Laboratory Fortified Blank
 - * Real people language: analyze a TSS standard that can be prepared from recipe (next slide) or bought premade
 - * Run on a 5% basis, one for every 20 samples

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TSS SM2540 D – 1997 Dried at 103-105°C

- * To prepare TSS check samples from dry reference material:
 - * Dry the reference material* in the desiccator
 - * On an analytical balance, weigh 0.1000 gram of the dry powder, put it in a 100.0 mL volumetric flask, bring it to the mark with distilled or deionized water and shake well until well suspended.
 - * Measure 100 mL and process as usual for environmental samples.
 - * A difference of 10 mg should be obtained.
 - * Calculation: $\frac{(A-B)(1000)}{\text{Vol. used}} = \frac{(10 \text{ mg})(1000)}{100 \text{ mL}} = 100 \text{ mg/L}$
- * Example of material available from Fisher
 - * Celite 545 Filter Aid (Powder), Fisher Chemical, 500 gram bottle – Cat#C212-500

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TSS SM2540 D – 1997 Dried at 103-105oC

- * Initial Calibration
 - * Check balances daily (day of) with at least 2 working weights that bracket the normal usage range and record results on bench sheet or separate log book
- * Duplicates
 - * Run on a 10% basis, one for every 10 samples
 - * Calculate %RPD
 - * Target value should be close to the first value and have a small RPD (less than 15%)
 - * **2014 Update - For reporting purposes, average sample and duplicate.**

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Total Suspended Solids Analysis Procedure to Omit Re-drying/Re-cooling/Re-weighing

How to acquire acceptable results for total suspended solids comparability data:

- The maximum holding time for a total suspended solids sample prior to analysis is 7 days if stored at temperatures of 6°C and below (not 0°C). (*40CFR part 136, Table II*)
- EPA recommends that 4-7 different samples, in duplicate, be collected and analyzed for this procedure in order to prove that the step for "reheating, recooling, and reweighing" is unnecessary. "Different" could mean samples collected 4-7 consecutive days or 4-7 samples run in one day. These 4-7 samples are dried **overnight** at 103-105°C.
- The next morning, the filters are removed from the oven, allowed to cool in the desiccator and weighed.
- The samples are then returned to the drying oven for one hour, recooled and reweighed.
- The resulting data should be examined to determine if the difference between the overnight values and the redried values are less than 4% or 0.5 mg, whichever is less. If so, the redrying step may be omitted for a normal set of samples.
- This procedure excludes atypical samples. (i.e. high fat, oil and grease samples).
- The operator may choose not to perform this study and continue to follow the procedure for redrying/recooling/reweighing as stated the method (Std Methods, 2540 D).

The study should be re-evaluated at least once per year or whenever a change in sample characteristics occurs and kept on file at the treatment plant.

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TSS SM2540 D – 1997 Dried at 103-105oC

- * **2014 Update** - Create and maintain control charts if you have 20-30 data points within 90 days.
- * If you do not meet the above criteria, follow QC Acceptance Criteria below.
 - * Blanks < 2.5 mg/L
 - * LFB ± 15%
 - * RPD < 15%

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SS SM2540 F – 1997 Settleable Solids

- * Dup
- * Corrective Action
- * Batch Size
- * QC Frequency



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SS SM2540 F – 1997 Settleable Solids

- * Duplicates
 - * For example, pour up 1000 mL of effluent into Imhoff then pour up another 1000 mL of effluent in another Imhoff. Wait 45 min, stir, wait 15 min, read. Figure RPD for both samples.
 - * Calculate RPD, (less than 20%)
 - * Run on a 5% basis (see batch size for more information).
 - * **2014 Update – For reporting purposes, average sample and duplicate.**



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SS SM2540 F – 1997 Settleable Solids

- * **2014 Update** - QC Acceptance Criteria below.
- * RPD < 20%
- * Reporting Limit = lowest graduation mark on Imhoff cone

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Temperature SM2550 B – 2000 Thermometric Measurement

- * ICAL
 - * Have thermometers verified **annually** by an NIST thermometer
- * Corrective Action
- * QC Frequency



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E. coli



- * Two Approved Methods
 - * SM 9223 B – 2004 IDEXX Colilert Quanti-Tray
 - * Hach Method 10029 – m-ColiBlue24[®] -

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SM 9020 B. QC Guidelines

- * General Considerations
 - * The program must be practical and require only a reasonable amount of time or it will be bypassed.
- * Facilities
 - * Provide a dust and draft free lab that has a stable temperature that does not have extreme temperature variations.
 - * Minimize through traffic and visitors
 - * Provide adequate space for conducting the analysis
 - * Keeps work area clean and disinfected

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SM 9020 B. QC Guidelines

- * Lab Equipment
 - * Verify thermometer accuracy annually. (-LabtronX or other certification vendor) 9020 B.4.a
 - * UV lamps – 9020 B.4.l (if used)
 - * Clean monthly with soft cloth moistened with ethanol
 - * Recommend replacing bulbs annually
 - * Incubators – 9020 B.4.o
 - * Verify thermometers – annually
 - * Record temperature twice daily (day of), at least 4 hours apart
 - * Verify that cold samples incubate for specified time. May need to warm samples in very cold weather
 - * Protect incubator from extreme room temperatures. Ideal is 60-80°F

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SM 9020 B. QC Guidelines

- * Lab Equipment - continued
 - * Media
 - * Check reagent media appearance with each use and discard if there is a color change.
 - * Protect reagent media from light
 - * Refrigerators – 9020 B.4.i
 - * Maintain temperature at 2-8°C
 - * Check and record temps daily (day of)



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SM 9020 B. QC Guidelines

- * Lab Equipment – continued
 - * Membrane Filtration Equipment (if MF procedure is used) – 9020 B.4.k
 - * Wash and rinse filtration assemblies thoroughly after use, wrap in nontoxic paper or foil, and sterilize
 - * UV sterilize or boil funnel apparatus between samples
 - * If using boiling water, make sure membrane filtration equipment is cool before adding next sample

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SM 9020 B. QC Guidelines

- * Lab Equipment – continued
- * Autoclave – 9020 B.4.h
 - * For routine use, verify the autoclave temperature weekly by using a maximum registering thermometer (MRT) to confirm that 121°C has been reached
 - * Test **monthly** for sterilization efficacy (with *Geobacillus stearothermophilus*)
 - * If any media, bottles, filters or other equipment that comes into contact with the samples are sterilized in the autoclave, the sterilization efficacy test must be performed **monthly**
 - * If you are only using your autoclave to sterilize waste, you just need an MRT (maximum registering thermometer)

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SM 9020 B. QC Guidelines

- * Lab Equipment – continued
- * Membrane filters and pads (if MF procedure is used) – 9020 B.5.i.3
 - * Check filters for brittleness if lot is held for one or more years

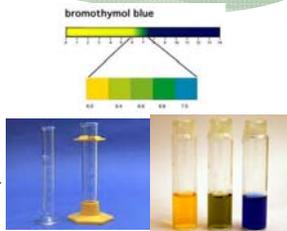
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SM 9020 B. QC Guidelines

- * Lab Supplies
- * Glassware – 9020 8.5.a
 - * pH check to test clean glassware for alkaline or acid residue, add a few drops of 0.04% bromothymol blue (BTB) or other pH indicator and observe the color reaction.
 - * BTB should be blue-green in the acceptable neutral range



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SM 9020 B. QC Guidelines



- * Lab Supplies – continued
- * Dilution water bottles – 9020 B.5.c
 - * Dilution waters available commercially are acceptable.
 - * Check one per lot for pH and volume (99 ± 2 mL) and examine bottles for a precipitate
 - * Discard by expiration date
 - * Before use of each batch conduct sterility (one bottle per batch) – **More information on slide #154**

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SM 9020 B. QC Guidelines

- * Lab Supplies – continued
- * Dilution water bottles sterility check - continued
- * Sterility Checks – 9020B.9.d
 - * Check each new batch (or lot) of buffered water for sterility before first use by adding 50 mL of water to 50 mL of a double-strength broth (e.g. tryptic soy, trypticase soy or tryptose broth).
 - * Alternatively, aseptically pass 100 mL of dilution water through a membrane filter and place filter on nonselective medium.
 - * Incubate at $35 \pm 0.5^\circ\text{C}$ for 24 hours and observe for growth.
 - * For membrane filter tests, check the sterility of the entire process by using sterile reagent or dilution water as the sample at the beginning and end of each filtration series of samples and test for growth

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SM 9020 B. QC Guidelines

- * Lab Supplies – continued
- * Sample bottles – 9020 B.5.d
 - * Minimally test for sterility one sample bottle per batch sterilized in the lab. Document results. – **More information on slide #154**
 - * Check accuracy of 100 mL mark, one per lot and record results.



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SM 9020 B. QC Guidelines

- * Lab Supplies – continued
- * Multi-well trays and sealers – 9020 B.5.e
 - * Check sterility of multi-well trays one per lot (**one tray per lot or quarter with that same lot number, whichever is more frequent**) by aseptically adding 100 mL of tryptic soy broth, seal, and incubate at 35±0.5°C for up to 48 hours.
 - * No growth indicates sterility
 - * Note that if the wells become very turbid (indicating nonsterile condition), there could be gas production and contaminant blowout between wells.
 - * Evaluate sealing performance of heat sealer unit quarterly by adding one to two drops of food-color dye to 100 mL deionized water sample, run through sealer and visually check each well for leakage.
 - * **2014 Update – analyze a method blank once per lot (of sterile water, media, bottles and trays) or once per quarter, whichever is more frequent, to demonstrate sterility.**

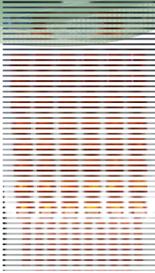


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SM 9020 B. QC Guidelines

- * Lab Supplies – continued
- * Multi-well trays and sealers – 9020 B.5.e
 - * Evaluate sealing performance of heat sealer unit **monthly** by adding one to two drops of food-color dye to 100 mL deionized water sample, run through sealer, and visually check each well for leakage.
 - * **2014 Update – As a monthly check of a sealer efficacy, perform and document a visual check that trays are properly sealed. If all sample wells are positive for total coliform and sufficient contrast, visually examine the tray cells for leakage and document the check. If insufficient color contrast is present us food-color dye as previously recommended by method.**
 - * Perform cleaning and maintenance on sealer annually or more frequently, if needed.



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SM 9020 B. QC Guidelines



- * Coliforms – Total and E. coli Hach Method 10029 – m-ColiBlue24®
- * Blank – daily (day of)
 - * Run at least one membrane filter blank at the beginning and the end of each filtration series by filtering 20-30 mL of dilution water through the membrane filter, placing in a petri dish with mColiBlue broth and testing for growth.
- * Positive and Negative Controls – Check certified control cultures with each lot of media and petri dishes with pads OR once a quarter, whichever is more frequent.
 - * Pseudomonas aeruginosa is recommended as a negative control and Escherichia coli as a positive control.
- * Duplicate Analyses – Perform duplicate analyses on a 5% basis (1 in 20 samples) or once a month, whichever is more frequent.

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SM 9020 B. QC Guidelines

- * Enzyme Substrate Test SM 9223 B, 22nd Edition (2004) – Colilert Method
- * Quality Control
 - * Test **each lot of media or quarterly (whichever is more frequent)** purchased for performance by inoculation with two certified control bacteria: *Escherichia coli* and a noncoliform.
 - * Also add a sterile water control. If a sterile water control exhibits faint fluorescence or faint positive coliform, discard use and use a new batch of substrate.
 - * Incubate these controls at 35±0.5°C as indicated above.
- * Duplicate Analyses – Perform duplicate analyses on a 5% basis (1 in 20 samples) or once a month, whichever is more frequent.

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SM 9020 B. QC Guidelines

- * Reporting Duplicates
 - * Both results should be documented on bench sheet.
 - * All duplicates should be reported according to your permit limits. If your permit sets a maximum limit, then the maximum value should be reported even if falls outside your permit limit.
 - * If both values fall below your daily max, average (arithmetically) the daily duplicates to get one result and then using that averaged result as part of the monthly geometric mean calculation.

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Questions/Comments



Shannon Pratt
Fleming Training Center
(615) 898-6506
Shannon.Pratt@tn.gov

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