

12 Steps of Lab Quality Assurance

Parameter	Method	DOC	MDL	Method Blank	LFB	LFM / LFMD	Dup	ICAL / CCV	Control Charts	Corrective Action	QC Acceptance	Batch Size	*QC Frequency
Ammonia	SM4500-NH3 D - 1997	X	X	X	X	X		X, Calibrate meter daily	X	X	X	20	Depends on Permit
BOD ₅ / CBOD ₅	SM5210 B - 2001	X		X	X		X	X, Calibrate meter daily	X	X	X	20	Depends on Permit
Chlorine, TR	SM4500-Cl G - 2000	X	X	X	X		X	X, verify meter daily w Secondary Gel Standards	X	X	X	20	Depends on Permit
pH	SM4500-H+ B - 2000	X					X	X, Calibrate meter daily		X	X	20	Depends on Permit
Oxygen, dissolved	SM4500-O G - 2001	X					X	X, Calibrate meter daily & verify with air-saturated water		X	X	20	Depends on Permit
	Hach Method 10360 Luminescence Oct. 2011	X					X	X, Calibrate meter daily & verify with air-saturated water		X	X	20	Depends on Permit
Phosphorus, total	SM4500-P B and E - 1999	X	X	X	X	X		X, verify meter	X	X	X	20	Depends on Permit
TSS	SM2540 D - 1997	X		X	X		X	X, verify scale daily		X	X	20	Depends on Permit
Sett. Solids	SM2540 F - 1997						X			X		20	Depends on Permit
Temperature	SM2550 B - 2000							X, verify against NIST thermometer		X			Annually

DOC – Demonstration of Capability

- Each analyst should have a file kept from where they have calibrated and analyzed 4 standards to demonstrate they can accurately run this test
- Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods
- Recommend backup analyst do this once a year

MDL – Method Detection Limit

- Annually run at least 7 samples at low levels

Method Blank – aka Laboratory Reagent Blank (LRB)

- Analyze distilled/deionized water as a sample

LFB – Laboratory Fortified Blank

- Analyze a known standard

LFM/LFMD – Laboratory Fortified Matrix/Laboratory Fortified Matrix Duplicate

- Analyze a sample with a known amount of standard added (spike)

Dup – Duplicate

- Analyze the same sample twice

ICAL/CCV – Initial Calibration/Continuing Calibration Verification

- Calibrate meter (DO, pH, ISE) or verify balance, thermometer and colorimeter/spectrophotometer
- Verify the calibration (especially if preset by manufacturer) at beginning of day and/or after every 10 readings, whichever comes first.

Control Charts

- 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.

Corrective Action

- Have corrective action plan in SOP for each method on what to do if QC tests fail or are out of range.
- For example, if standards fail, re-calibrate and run test again.

QC Acceptance

- Have in SOP for each method the acceptance ranges for standards, duplicates, spikes, etc. and make sure they match the method requirements.

Batch Size

- Each batch could be daily, every 10 samples or every 20 samples. Check method.

*QC Frequency (depends on permit) – at least once a month

- 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, we would allow for a duplicate 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!!
 - **Please note that influent and effluent samples count as two separate samples. For example, if you are supposed to run 3 BODs a week, that should be counted as running 6 samples for that week.**
- For samples that need to be analyzed on a 10% basis or once for 10 samples, follow these criteria:
 - If a permit stated that 3 analyses per week, we would allow for a duplicate to be analyzed at least twice per 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!!
 - If a permit stated 5 analyses per week, we would allow a duplicate to be analyzed once per week.
 - Pick a date and be consistent, every Monday or Wednesday. Mark your calendar!!
 - **Please note that influent and effluent samples count as two separate samples. For example, if you are supposed to run 5 TSSs a week, that should be counted as running 10 samples for that week and you should run your duplicates once a week.**

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 the values.
- It must include the QC Acceptance Criteria, the definition of a "Batch" and the minimum frequency of QC checks

Ammonia, SM 4500-NH₃ D, 22nd edition (1997) – Ammonia-Selective Electrode Method

40 CFR 136 Table 1B says the approved methodology is manual distillation⁶ or gas diffusion (pH>11) followed by any of the following: Nesslerization, titration, electrode, manual phenate or automated phenate. Footnote 6 states: “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.”

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.

Initial Demonstration of Capability (DOC)

- 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.
- 4020 B.1.a. - each analyst must run a known standard concentration at least four times and compare limits listed in the method.
- **Real people language – each operator running this test needs to calibrate the probe and analyze 4 samples of an NH₃ Standard at a concentration around 1.0 mg/L**
 - **Keep a folder for each analyst, keep a copy here**
 - **Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.**
 - **Recommend backup analyst do this once a year.**

Method Detection Limit (MDL)

- 1020 B. 4 – As a starting point for selecting the concentration to use when determining the MDL, use an estimate of five times the estimated true detection level ($5 \times 0.03 \text{ mg/L} = 0.15 \text{ mg/L}$).
 - Ideally, prepare and analyze at least seven (7) portions of this solution over a 3-day period to ensure that the MDL determination is more representative of routine measurements as performed in the laboratory.
 - 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 and 150%, with %RSD (relative standard deviation) values $\leq 20\%$.

- 4020 B.1.b. – Verify MDL at least **annually**.
 - Ideally use pooled data from several analysts rather than data from one analyst.
- **Real people language – have several operators, who run this test, analyze an NH₃ Standard at a concentration of 0.15 mg/L over several days with a total of at least 7 samples**
 - **Joe analyzes 3 samples on Monday**
 - **Bob analyzes 3 samples on Tuesday**
 - **Mary analyzes 3 samples on Wednesday**
- **Run this once a year**

Initial Calibration Verification (ICV)

- 1020 B.11.b. – Perform initial calibration using at least three concentrations of standards for linear curves.
- 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 $\pm 10\%$.
- 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
- 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.
- 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.
- **Real people language – calibrate according to manufacturer's instructions with at least 3 standards that will bracket your sample range daily (day of).**
- **Analyze a 10 mg/L standard as a sample after calibration and before samples to verify initial calibration (ICV)**

Method Blank – goes through distillation if you distill

- 1020 B.5.– A reagent blank (method blank) consists of reagent water and all reagents that normally are in contact with a sample during the entire analytical procedure.
- 4020 B.2.d. – Include at least one method blank daily or with each **batch of 20** or fewer samples, whichever is more frequent.
 - If any method blank measurements are at or above the reporting level, take immediate corrective action.
- **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 reporting limit**
 - **Reporting limit will be equal to your Method Detection Limit (MDL)**
 - **Run on a 5% basis (see batch size for more information).**

Laboratory Fortified Blank (LFB) – goes through distillation if you distill

- 1020 B.6.– A laboratory-fortified blank is a reagent water sample to which a known concentration of the analyte of interest has been added.

- o Sample batch = 5% basis = 1 every 20 samples
- o Use an added concentration of at least 10 times the MDL, less than or equal to the midpoint of the calibration curve.
- 4020 B.2.e. – Calculate percent recovery, plot control charts and determine control limits (see Control Charts below)
- **Real people language – analyze an NH₃ standard at a concentration of 5.0 mg/L**
 - o **Run on a 5% basis (see batch size for more information).**

Duplicate

- NONE

Laboratory Fortified Matrix (LFM)/Laboratory Fortified Matrix Duplicate (LFMD) – goes through distillation if you distill

- 1020 B.7.– A laboratory fortified matrix (LFM) is an additional portion of a sample to which a known amount of the analyte of interest is added before sample preparation
 - o The LFM is used to evaluate analyte recovery in a sample
 - o Sample batch = 5% basis
 - o Add a concentration that is at least 10 times the MRL (minimum reporting level), less than or equal to the midpoint of the calibration curve.
 - o Preferably use the same concentration as the LFB
- 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
 - o Add a known concentration of analyte to a randomly selected routine sample
 - o Calculate percent recovery and relative percent difference, plot control charts and determine control limits for spikes at different concentrations (see Control Charts below)
- **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.**
 - o **Run on a 5% basis (see batch size for more information).**
 - o **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%).**
 - o **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.**

Continuing Calibration Verification (CCV)

- 1020 B.11.c. – Analysts periodically use a calibration standard to confirm that the instrument performance has not changed significantly since initial calibration.
 - o Verify calibration by analyzing one standard at a concentration near or at the mid-point of the calibration range.
- 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.
 - o For the calibration verification to be valid, check standards must be within 10% of its true value
- **Real people language – analyze 10 mg/L at the end of all samples daily (day of).**

Control Charts – 1020 B.13.

- **Real people language**
 - **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.**

Corrective Action - 1020 B.5., B.8., & B.15.

QC Acceptance Criteria

- Blanks < MDL
- LFB $\pm$ 15%
- ICV/CCV $\pm$ 10%
- LFM/LFMD $\pm$ 20%
- RPD < 20%
- Reporting limit = MDL

Batch Size

- For samples that need to be analyzed on a 5% basis (1 for every 20 samples or once per month, whichever is more frequent) follow these criteria:
 - o If a permit stated that 3 analyses per week, we would allow for a duplicate 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!!
 - o 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!!
 - o If sampling only once a month, need to run QC once a month.

Calculations

- % Recovery for LFB
 - o
$$= \frac{\text{LFB Result}}{\text{Expected Concentration}} \times 100\%$$
- RPD – relative percent differences for duplicates and LFM/LFMD
 - o
$$= \frac{\text{Difference between sample and duplicate}}{\text{Average of the sample and duplicate}} \times 100\%$$
- % Recovery for LFM – when using less than or equal to 1% spike volume compared to sample volume
 - o
$$= \frac{\text{LFM Result} - \text{Sample Result}}{\text{Actual Concentration of spike}} \times 100\%$$

Initial Demonstration of Capability (DOC)

- 1020 B. 1 – As a minimum, include a reagent blank and at least 4 LFBs
- 4020 B.1.a. – Before analysts run any samples, verify their capability with the method. Run a laboratory fortified blank at least four times and compare to the limits listed in the method.
 - o If no limit is specified, use the following procedure to establish limits.
 - o Calculate the standard deviation of the four samples. Then calculate the LFB's recovery limits (see formula at end)
- **Real people language – each operator running this test needs to analyze 4 samples of GGA at a concentration of 198 ± 30.5 mg/L**
 - o **Keep a folder for each analyst, keep a copy here**
 - o **Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.**
 - o **Recommend backup analyst do this once a year.**

Method Detection Limit (MDL)

- NONE

Initial Calibration Verification (ICV)

- 1020 B.11.b. – Perform initial calibration
- Hach's Method: 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 $20 \pm 3^{\circ}\text{C}$.
- 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 DO probe daily (day of) by following manufacturer's instructions.**

Method Blank

- 1020 B.5.– A reagent blank (method blank) consists of reagent water and all reagents that normally are in contact with a sample during the entire analytical procedure.
- 5020 B.2.d. – Include at least one method blank daily or with each batch of 20 or fewer samples, whichever is more frequent.
- 5210 B.6.c. – With each batch of samples, incubate one or more bottles of dilution water that contains nutrient, mineral and buffer solutions but no seed or nitrification inhibitor.
 - o The DO uptake in 5 days must not be more than 0.20 mg/L and preferably not more than 0.10 mg/L, before making seed corrections.
- **Real people language – analyze dilution water blanks daily (day of), preferably one at beginning and one at end**
 - o **Target value is less than 0.20 mg/L (preferably less than 0.10 mg/L)**

Laboratory Fortified Blank (LFB)

- 1020 B.6.– A laboratory-fortified blank is a reagent water sample to which a known concentration of the analyte of interest has been added.
 - o Sample batch = 5% basis
- 5020 B.2.e. – Calculate percent recovery, plot control charts and determine control limits (see Control Charts below)
 - o When appropriate, include at least one LFB daily or per each batch of 20 or fewer samples.
- **Real people language – analyze GGA sample at a concentration of 198 ± 30.5 mg/L**
 - o **Run on a 5% basis (see batch size for more information).**
 - o **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**

Duplicate

- 1020 B.12.f. – Calculate RPD (relative percent difference)
- 5020 B.2.f. – Randomly select routine samples to be analyzed twice.
 - o Process duplicate sample independently through the entire sample preparation and analysis.
 - o Include at least one duplicate for each matrix type daily or with each batch of 20 or fewer samples.
- **Real people language – analyze 2 samples for BOD or cBOD, run an extra sample dilution bottle**
 - o **Example: if you run 100mL, 200mL and 300mL for your effluent, run a second 300mL sample. You will have 4 bottles total for your effluent dilutions.**
 - o **Target value should be close to the first value (same dilution) and have a small RPD (less than 20%)**
- **For reporting purposes, average results that meet method criteria.**

Laboratory Fortified Matrix (LFM)/Laboratory Fortified Matrix Duplicate (LFMD)

- NONE

Continuing Calibration Verification (CCV)

- Hach Method 10360 7.2 and 9.4 – Calibration Verification for membranes and LDO probes
 - o 7.2.1 – Add approximately 1500 mL of organic-free water or BOD dilution water to a 2-L beaker or PET bottle
 - o 7.2.2 – Allow the water to equilibrate to room temperature. Room temperature should be approximately $20 \pm 3^{\circ}\text{C}$.
 - o 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.
 - o 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 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.

- o 7.2.5 – Transfer the aerated water to a BOD bottle until overflowing and stopper.
- o 7.2.6 – Calculate the theoretical dissolved oxygen concentration using a dissolved oxygen table such as Hitchman...
- o 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 (± 0.2 mg/L).**
 - o Theoretical dissolved oxygen can be found at USGS's website at <http://water.usgs.gov/software/DOTABLES/> or by using a DO Saturation Table.

Control Charts – 1020 B.13.

- **Real people language**
 - **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.**

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:
 - o Dilution water exceeds 0.20 mg/L (5210B.6c)
 - o Glucose-glutamic acid check falls outside of acceptable limits (5210B.6b)
 - o Test replicates show more than 30% difference between high and low values
 - o Seed control samples do not meet the above criteria in all dilutions (5210B.6d) or
 - o Minimum DO is less than 1.0 mg/L (5210B.7a3)

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

Batch Size

- For samples that need to be analyzed on a 5% basis (1 for every 20 samples or once per month, whichever is more frequent) follow these criteria:
- Influent and Effluent are 2 different samples
 - o If a permit stated that 3 analyses per week, that would be 6 samples per week, we would allow for a duplicate to be analyzed at least 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!!
 - o If a permit stated 5 analyses per week, that would be 10 samples per week, we would allow once a week.
 - Pick a date and be consistent, every Monday. Mark your calendar!!

- o If sampling only once a month, need to run QC once a month.

Calculations

- % Recovery for LFB
 - o
$$= \frac{\text{LFB concentration}}{\text{Expected concentration}} \times 100\%$$
- RPD – relative percent differences for duplicates
 - o
$$= \frac{\text{Difference between sample and duplicate}}{\text{Average of the sample and duplicate}} \times 100\%$$
- Unseeded - BOD₅, mg/L
$$= \frac{D_1 - D_2}{P}$$
- Seeded - BOD₅, mg/L
$$= \frac{(D_1 - D_2) - (B_1 - B_2)f}{P}$$
 - o Where:
 - D₁ = Initial Dissolved Oxygen Concentration in Sample, mg/L
 - D₂ = Final Dissolved Oxygen Concentration in Sample, mg/L
 - B₁ = Initial Dissolved Oxygen Concentration in Seed Control, mg/L
 - B₂ = Final Dissolved Oxygen Concentration in Seed Control, mg/L
 - P = Sample Concentration, % (expressed as a decimal)
 - f =
$$\frac{\text{Seed in Sample, \%}}{\text{Seed in Seed Control, \%}}$$

Temp.	Barometric Pressure (mm Hg)																			
(deg C)	685	690	695	700	705	710	715	720	725	730	735	740	745	750	755	760	765	770	775	780
0	13.17	13.27	13.36	13.46	13.56	13.65	13.75	13.85	13.94	14.04	14.14	14.23	14.33	14.43	14.52	14.62	14.72	14.81	14.91	15.01
1	12.81	12.9	12.99	13.09	13.18	13.28	13.37	13.46	13.56	13.65	13.75	13.84	13.93	14.03	14.12	14.22	14.31	14.4	14.5	14.59
2	12.46	12.55	12.64	12.73	12.82	12.91	13.01	13.1	13.19	13.28	13.37	13.46	13.56	13.65	13.74	13.83	13.92	14.01	14.1	14.2
3	12.12	12.21	12.3	12.39	12.48	12.57	12.66	12.75	12.84	12.93	13.02	13.1	13.19	13.28	13.37	13.46	13.55	13.64	13.73	13.82
4	11.81	11.89	11.98	12.07	12.15	12.24	12.33	12.41	12.5	12.59	12.67	12.76	12.85	12.93	13.02	13.11	13.2	13.28	13.37	13.46
5	11.5	11.59	11.67	11.75	11.84	11.92	12.01	12.09	12.18	12.26	12.35	12.43	12.52	12.6	12.69	12.77	12.86	12.94	13.03	13.11
6	11.21	11.29	11.37	11.46	11.54	11.62	11.7	11.79	11.87	11.95	12.04	12.12	12.2	12.28	12.37	12.45	12.53	12.61	12.7	12.78
7	10.93	11.01	11.09	11.17	11.25	11.33	11.41	11.49	11.58	11.66	11.74	11.82	11.9	11.98	12.06	12.14	12.22	12.3	12.38	12.46
8	10.66	10.74	10.82	10.9	10.98	11.06	11.14	11.21	11.29	11.37	11.45	11.53	11.61	11.69	11.76	11.84	11.92	12	12.08	12.16
9	10.41	10.48	10.56	10.64	10.71	10.79	10.87	10.94	11.02	11.1	11.18	11.25	11.33	11.41	11.48	11.56	11.64	11.71	11.79	11.87
10	10.16	10.24	10.31	10.39	10.46	10.54	10.61	10.69	10.76	10.84	10.91	10.99	11.06	11.14	11.21	11.29	11.36	11.44	11.51	11.59
11	9.93	10	10.07	10.15	10.22	10.29	10.37	10.44	10.51	10.59	10.66	10.73	10.81	10.88	10.95	11.03	11.1	11.17	11.25	11.32
12	9.7	9.77	9.84	9.91	9.99	10.06	10.13	10.2	10.27	10.35	10.42	10.49	10.56	10.63	10.71	10.78	10.85	10.92	10.99	11.06
13	9.48	9.55	9.62	9.69	9.76	9.83	9.9	9.97	10.04	10.11	10.19	10.26	10.33	10.4	10.47	10.54	10.61	10.68	10.75	10.82
14	9.27	9.34	9.41	9.48	9.55	9.62	9.69	9.76	9.82	9.89	9.96	10.03	10.1	10.17	10.24	10.31	10.37	10.44	10.51	10.58
15	9.07	9.14	9.21	9.27	9.34	9.41	9.48	9.54	9.61	9.68	9.75	9.81	9.88	9.95	10.02	10.08	10.15	10.22	10.29	10.35
16	8.88	8.95	9.01	9.08	9.14	9.21	9.28	9.34	9.41	9.47	9.54	9.61	9.67	9.74	9.8	9.87	9.94	10	10.07	10.13
17	8.69	8.76	8.82	8.89	8.95	9.02	9.08	9.15	9.21	9.28	9.34	9.41	9.47	9.54	9.6	9.66	9.73	9.79	9.86	9.92
18	8.51	8.58	8.64	8.7	8.77	8.83	8.9	8.96	9.02	9.09	9.15	9.21	9.28	9.34	9.4	9.47	9.53	9.59	9.66	9.72
19	8.34	8.4	8.47	8.53	8.59	8.65	8.72	8.78	8.84	8.9	8.96	9.03	9.09	9.15	9.21	9.28	9.34	9.4	9.46	9.53
20	8.17	8.24	8.3	8.36	8.42	8.48	8.54	8.6	8.66	8.73	8.79	8.85	8.91	8.97	9.03	9.09	9.15	9.21	9.28	9.34
21	8.01	8.07	8.13	8.19	8.25	8.31	8.37	8.43	8.49	8.55	8.61	8.67	8.73	8.79	8.85	8.92	8.98	9.04	9.1	9.16
22	7.86	7.92	7.98	8.04	8.09	8.15	8.21	8.27	8.33	8.39	8.45	8.51	8.57	8.63	8.68	8.74	8.8	8.86	8.92	8.98
23	7.71	7.77	7.82	7.88	7.94	8	8.06	8.11	8.17	8.23	8.29	8.35	8.4	8.46	8.52	8.58	8.64	8.69	8.75	8.81
24	7.56	7.62	7.68	7.73	7.79	7.85	7.9	7.96	8.02	8.08	8.13	8.19	8.25	8.3	8.36	8.42	8.48	8.53	8.59	8.65
25	7.42	7.48	7.53	7.59	7.65	7.7	7.76	7.81	7.87	7.93	7.98	8.04	8.1	8.15	8.21	8.26	8.32	8.38	8.43	8.49
26	7.29	7.34	7.4	7.45	7.51	7.56	7.62	7.67	7.73	7.78	7.84	7.89	7.95	8	8.06	8.11	8.17	8.22	8.28	8.33
27	7.15	7.21	7.26	7.32	7.37	7.43	7.48	7.53	7.59	7.64	7.7	7.75	7.81	7.86	7.91	7.97	8.02	8.08	8.13	8.19
28	7.03	7.08	7.13	7.19	7.24	7.29	7.35	7.4	7.45	7.51	7.56	7.61	7.67	7.72	7.77	7.83	7.88	7.93	7.99	8.04
29	6.9	6.95	7.01	7.06	7.11	7.16	7.22	7.27	7.32	7.38	7.43	7.48	7.53	7.59	7.64	7.69	7.74	7.8	7.85	7.9
30	6.78	6.83	6.88	6.94	6.99	7.04	7.09	7.14	7.2	7.25	7.3	7.35	7.4	7.46	7.51	7.56	7.61	7.66	7.71	7.77

Created at <http://water.usgs.gov/software/DOTABLES/>

The Use of Secondary Standards for Spectrophotometer/Colorimeter Calibration

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.

The analyst is responsible for the following:

- Preparing the calibration curve for each instrument **once per month** at a minimum with chlorine standards or potassium permanganate (see instructions below for KMnO_4), 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
- Verifying the calibration curve using a minimum of one blank and two gel standards that bracket the expected sample concentration
- Recording all verification data

POTASSIUM PERMANGANATE (KMnO_4) 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 an 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.*****

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, and 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.****

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

CALIBRATION STANDARD SOLUTIONS

If using KMnO_4 , four to five calibration standard solutions should be made according to the table below with the addition of DPD to create a calibration curve **once per month** at a minimum. The correlation coefficient of the curve should correlate to 0.995 or better. This curve is then used to check instrument calibration. Gel standards are run against the curve and must agree to within $\pm 10\%$.

****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.****

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. Gel standards are run against the curve and must agree to within $\pm 10\%$.

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

Initial Demonstration of Capability (DOC)

- Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.
- Calibrate daily according to manufacturer's instructions
- Follow Hach Method 10360 9.2.1 – Prepare and measure four samples of air-saturated water according to section 7.2.
 - o 7.2.1 – Add approximately 1500 mL of organic-free water or BOD dilution water to a 2-L beaker or PET bottle
 - o 7.2.2 – Allow the water to equilibrate to room temperature. Room temperature should be approximately $20 \pm 3^{\circ}\text{C}$.
 - o 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.
 - o 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.
 - o 7.2.5 – Transfer the aerated water to a BOD bottle until overflowing and stopper.
 - o 7.2.6 – Calculate the theoretical dissolved oxygen concentration using a dissolved oxygen table such as Hitchman...
- **Real people language – each analyst should calibrate the probe, in accordance with manufacturer's instruction, prepare dilution water that is air-saturated and analyze four bottles and compare to the theoretical dissolved oxygen concentration (± 0.2 mg/L).**
 - o **Theoretical dissolved oxygen can be found at USGS's website at <http://water.usgs.gov/software/DOTABLES/> or by using a DO Saturation Table.**

Method Detection Limit (MDL)

- None

Initial Calibration Verification (ICV)

- 1020 B.11.b. – Perform initial calibration
- **Real people language – calibrate daily (day of) by following manufacturer's instructions.**

Method Blank

- NONE

Laboratory Fortified Blank (LFB)

- NONE

Duplicate

- 1020 B.12.f. – Calculate RPD (relative percent difference)
- 4020 B.2.f. – Randomly select routine samples to be analyzed twice.

- o Process duplicate sample independently through the entire sample preparation and analysis.
- o Include at least one duplicate for each matrix type daily or with each batch of 20 or fewer samples.
- **Real people language – on a 5% basis (see batch size for more information) analyze 2 samples for DO.**
 - o **First sample is result, second sample is duplicate**
 - o **Target value is to be close to the first value and have a small difference (≤ 0.2 mg/L)**
- **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.**

Laboratory Fortified Matrix (LFM)/Laboratory Fortified Matrix Duplicate (LFMD)

- NONE

Continuing Calibration Verification (CCV)

- Follow Hach Method 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 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...
- 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 (± 0.2 mg/L) after daily samples or with each batch of 20 samples or less in an 8-hour period.**
 - o **Theoretical dissolved oxygen can be found at USGS's website at <http://water.usgs.gov/software/DOTABLES/> or by using a DO Saturation Table.**

Control Charts

- NONE

Corrective Action - 1020 B.5., B.8., & B.15.

Batch Size

- For samples that need to be analyzed on a 5% basis (1 for every 20 samples or once per month, whichever is more frequent) follow these criteria:
 - o If a permit stated that 3 analyses per week, we would allow for a duplicate 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!!
 - o 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!!

Initial Demonstration of Capability (DOC)

- Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.
- Calibrate daily according to manufacturer's instructions
- Hach Method 10360 9.2.1 – Prepare and measure four samples of air-saturated water according to section 7.2.
 - o 7.2.1 – Add approximately 1500 mL of organic-free water or BOD dilution water to a 2-L beaker or PET bottle
 - o 7.2.2 – Allow the water to equilibrate to room temperature. Room temperature should be approximately $20 \pm 3^{\circ}\text{C}$.
 - o 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.
 - o 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.
 - o 7.2.5 – Transfer the aerated water to a BOD bottle until overflowing and stopper.
 - o 7.2.6 – Calculate the theoretical dissolved oxygen concentration using a dissolved oxygen table such as Hitchman...
- **Real people language – each analyst should calibrate the probe, in accordance with manufacturer's instruction, prepare dilution water that is air-saturated and analyze four bottles and compare to the theoretical dissolved oxygen concentration (± 0.2 mg/L).**
 - o **Theoretical dissolved oxygen can be found at USGS's website at <http://water.usgs.gov/software/DOTABLES/> or by using a DO Saturation Table.**

Method Detection Limit (MDL)

- None

Initial Calibration Verification (ICV)

- 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 $20 \pm 3^{\circ}\text{C}$.
- 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.**

Method Blank

- NONE

Laboratory Fortified Blank (LFB)

- NONE

Duplicate

- **Real people language – on a 5% basis (see batch size for more information) analyze 2 samples for DO.**
 - **First sample is result, second sample is duplicate**
 - **Target value is to be close to the first value and have a small difference (≤ 0.2 mg/L)**
- **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.**

Laboratory Fortified Matrix (LFM)/Laboratory Fortified Matrix Duplicate (LFMD)

- NONE

Continuing Calibration Verification (CCV)

- 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 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...
- 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 (± 0.2 mg/L) after daily samples or with each batch of 20 samples or less in an 8-hour period.**
 - **Theoretical dissolved oxygen can be found at USGS's website at <http://water.usgs.gov/software/DOTABLES/> or by using a DO Saturation Table.**

Control Charts

- NONE

Batch Size

- 9.3.1 – ... with each analytical batch of 20 samples or less in an 8 hour period.
- For samples that need to be analyzed on a 5% basis (1 for every 20 samples or once per month, whichever is more frequent) follow these criteria:
 - o If a permit stated that 3 analyses per week, we would allow for a duplicate 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!!
 - o 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!!

Initial Demonstration of Capability (DOC)

- 4020 B.1.a. - each analyst must run a known standard concentration at least four times and compare limits listed in the method.
- **Real people language – each operator running this test needs to calibrate instrument and analyze 4 buffers at a pH of 7**
 - Keep a folder for each analyst, keep a copy here
 - Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.
 - Recommend backup analyst do this once a year.
 - Only good for that type of instrument you are using at that plant. If you have a backup instrument made by a different manufacturer or you work at another plant with a different make/model, you would need another DOC.
 - DOCs demonstrate you are proficient with that one instrument.

Method Detection Limit (MDL)

- NONE

Initial Calibration Verification (ICV)

- 1020 B.11.b. – Perform initial calibration
- **Real people language – calibrate daily (day of) with fresh buffers by following manufacturer's instructions.**
- **Analyze 7 buffer solution as a sample after calibration and before samples to verify initial calibration (ICV), should be within ± 0.2 s.u.**

Method Blank

- NONE

Laboratory Fortified Blank (LFB)

- NONE

Duplicate

- 1020 B.12.f. – Calculate RPD (relative percent difference)
- 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 or fewer samples.
- **Real people language – on a 5% basis (see batch size for more information) analyze 2 samples for pH, after reading one, pour out sample and start with a fresh sample**
 - Example, grab sample in bucket and dip pH probe in twice to get a duplicate reading
 - Target value should be close to the first value (within ± 0.2 s.u.)

- 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.

Laboratory Fortified Matrix (LFM)/Laboratory Fortified Matrix Duplicate (LFMD)

- NONE

Continuing Calibration Verification (CCV)

- 1020 B.11.c. – Analysts periodically use a calibration standard to confirm that the instrument performance has not changed significantly since initial calibration.
 - o Verify calibration by analyzing one standard at a concentration near or at the mid-point of the calibration range.
- 4020.B.2.b. – Verify calibration by periodically analyzing a calibration standard during a run – typically after each batch of 10 samples and at the end of the run.
- Real people language – read 7 Buffer after analyzing samples daily (day of and within ± 0.2 pH units).

Control Charts

- NONE

Corrective Action - 1020 B.5., B.8., & B.15.

QC Acceptance Criteria

- ICV/CCV within ± 0.2 s.u.
- Duplicates within ± 0.2 s.u.

Batch Size

- For samples that need to be analyzed on a 5% basis (1 for every 20 samples or once per month, whichever is more frequent) follow these criteria:
 - o If a permit stated that 3 analyses per week, we would allow for a duplicate 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!!
 - o 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!!

Calculations

- NONE

Quality Assurance for E. coli Analysis

Laboratory Equipment and Instrumentation

- Thermometers – 9020B.4.a
 - Annually check accuracy of all working temperature-sensing devices... against a certified 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.
 - For general purposes use thermometers graduated in increments of 0.5°C or less.
- Autoclave – 9020B.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*)
 - Verify the autoclave temperature weekly by using a maximum registering thermometer (MRT) to confirm that 121°C has been reached.
- Refrigerator – 9020B.4.i
 - Maintain temperature at 2-8°C
 - Check and record temperature daily
- Membrane filtration equipment (if MF procedure is used) – 9020B.4.k
 - Wash and rinse filtration assemblies thoroughly after use, wrap in nontoxic paper or foil, and sterilize.
 - UV sterilize or boil funnels between samples
 - If using boiling water, make sure membrane filtration equipment is cool before adding next sample
- Membrane filters and pads (if MF procedure is used) – 9020B.5.i.3
 - Check filters for brittleness if lot is held for one or more years
- Ultraviolet lamps (if used) – 9020B.4.l
 - When used, disconnect lamps monthly and clean bulbs with a soft cloth moistened with ethanol
- Incubator – 9020 B.4.o
 - During usage periods check and record calibration-corrected temperature twice daily (morning and afternoon, separated by at least 4 hours) on each shelf in use to ensure temperature consistency throughout unit.

Laboratory Supplies

- Glassware – 9020 B.5.a
 - 1) pH check - To test clean glassware for alkaline or acid residue add a few drops of 0.04% bromthymol blue (BTB) or other pH indicator and observe the color reaction.
 - BTB should be blue-green (in the acceptable neutral range).
- Dilution water bottles – 9020 B.5.c
 - Dilution waters available commercially are acceptable.

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- o Check one per lot for pH and volume (99 ± 2 mL) and examine bottles for a precipitate
- o Discard by expiration date
- o Before use of each batch or lot conduct sterility (one bottle per lot or quarter with that same lot number, whichever is more frequent)
 - 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
- Sample bottles – 9020 B.5.d.
 - o Check accuracy of 100 mL mark, one per lot and record results.
- Multi-well trays and sealers – 9020 B.5.e
 - o 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.
 - o **Real people language – 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.**
 - o **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 use food-color dye as previously recommended by method.**

General QC Requirements

- Coliforms – Total and E. coli Hach Method 10029 – m-ColiBlue24®
 - o Blank – daily
 - 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.
 - o 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.
 - o Duplicate Analyses – Perform duplicate analyses on a 5% basis (1 in 20 samples) or once a month, whichever is more frequent.
- Enzyme Substrate Test SM 9223 B, 22nd Edition (2004) – Colilert Method
 - o Quality Control

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- 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 $35\pm0.5^{\circ}\text{C}$ as indicated above.
- Duplicate Analyses – Perform duplicate analyses on a 5% basis (1 in 20 samples) or once a month, whichever is more frequent.

Bibliography

American Public Health Association (APHA), American Waterworks Association (AWWA), and Water Environment Federation (WEF). 2012. *Standard Methods for the Examination of Water and Wastewater*. 22nd ed. American Public Health Association, Washington, D.C.

Initial Demonstration of Capability (DOC)

- Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.

Method Detection Limit (MDL)

- NONE

Initial Calibration Verification (ICV)

- NONE

Method Blank

- NONE

Laboratory Fortified Blank (LFB)

- NONE

Duplicate –

- 2540 A.2. "To aid in quality assurance, analyze samples in duplicate.
- **Real people language – analyze 2 samples for Sett. Solids.**
 - 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.
 - Target value should be close to the first value and have a small RPD (less than 20%)
 - Run on a 5% basis (see batch size for more information).
 - For reporting purposes, average sample and duplicate.

Laboratory Fortified Matrix (LFM)/Laboratory Fortified Matrix Duplicate (LFMD)

- NONE

Control Charts

- NONE

Corrective Action - 1020 B.5., B.8., & B.15.

QC Acceptance Criteria

- $RPD < 20\%$
- Reporting Limit = lowest graduation mark on Imhoff cone

Batch Size

- For samples that need to be analyzed on a 5% basis (1 for every 20 samples or once per month, whichever is more frequent) follow these criteria:

- o If a permit stated that 3 analyses per week, we would allow for a duplicate 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!!
- o 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!!

Calculations

- RPD – relative percent differences for duplicates
 - o = $\frac{\text{Difference between sample and duplicate}}{\text{Average of the sample and duplicate}} \times 100\%$

Initial Demonstration of Capability (DOC)

- Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.

Method Detection Limit (MDL)

- NONE

Initial Calibration Verification (ICV)

- 2020 B.2.a.– Verify calibration ***annually***
- 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.
 - o Record calibration results, along with the date and the technician's signature, in a quality control logbook.
 - o Mark the necessary calibration correction factor on each temperature measuring device so that only calibrated-corrected temperature values are recorded.
 - o Verify accuracy of the reference certified thermometer as specified on the certificate of calibration or at least every 5 years.
- **Real people language – have thermometers verified annually by a NIST thermometer.**

Method Blank

- NONE

Laboratory Fortified Blank (LFB)

- NONE

Duplicate

- NONE

Laboratory Fortified Matrix (LFM)/Laboratory Fortified Matrix Duplicate (LFMD)

- NONE

Control Charts

- NONE

Corrective Action - 1020 B.5., B.8., & B.15.

Batch Size

- NONE

Total Phosphorus, SM 4500-P B (Sample Prep) and E, 22nd edition (1999) – Ascorbic Acid Method

Minimum Detectable Concentration – 4500-P E.1.c. – approximately 10 µg/L (0.010 mg/L)

Initial Demonstration of Capability (DOC)

- 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.
- 4020 B.1.a. - each analyst must run a known standard concentration at least four times and compare limits listed in the method.
- **Real people language – each operator running this test needs to analyze 4 samples of Phosphorus standard at a concentration of about 0.5 mg/L**
 - **Keep a folder for each analyst, keep a copy here**
 - **Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.**
 - **Recommend backup analyst do this once a year.**

Method Detection Limit (MDL)

- 1020 B. 4 – As a starting point for selecting the concentration to use when determining the MDL, use an estimate of five times the estimated true detection level ($5 \times 0.010 \text{ mg/L} = 0.050 \text{ mg/L}$).
 - Ideally, prepare and analyze at least seven (7) portions of this solution over a 3-day period to ensure that the MDL determination is more representative of routine measurements as performed in the laboratory.
 - 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 and 150%, with %RSD (relative standard deviation) values $\leq 20\%$.
- 4020 B.1.b. – Verify MDL at least **annually**.
 - Ideally use pooled data from several analysts rather than data from one analyst.
- **Real people language – have several operators, who run this test, analyze a Phosphorus standard at a concentration of 0.05 mg/L over several days with a total of at least 7 samples**
 - **Joe analyzes 3 samples on Monday**
 - **Bob analyzes 3 samples on Tuesday**
 - **Mary analyzes 3 samples on Wednesday**
- **Run this once a year**

Initial Calibration Verification (ICV) – does not go through digestion

- 1020 B.11.b. – Perform initial calibration using at least three concentrations of standards for linear curves.
- 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 $\pm 10\%$.

- **Real people language – prepare a set of Phosphorus standards (4-5 standards) to verify the factory pre-set calibration curve **monthly** or more frequently if reagent lot # changes.**

Method Blank – goes through digestion

- 1020 B.5.– A reagent blank (method blank) consists of reagent water and all reagents that normally are in contact with a sample during the entire analytical procedure.
- 4020 B.2.d. – Include at least one method blank daily or with each batch of 20 or fewer samples, whichever is more frequent.
 - o If any method blank measurements are at or above the reporting level, take immediate corrective action.
- **Real people language – analyze distilled water as a sample by going through all digestion and reagent addition before reading.**
 - o **Target value is less than reporting limit.**
 - **Reporting limit will be equal to your Method Detection Limit (MDL)**
 - o **Run on a 5% basis (see batch size for more information).**

Laboratory Fortified Blank (LFB) – goes through digestion

- 1020 B.6.– A laboratory-fortified blank is a reagent water sample to which a known concentration of the analyte of interest has been added.
 - o Sample batch = 5% basis = 1 every 20 samples
 - o Use an added concentration of at least 10 times the MDL, less than or equal to the midpoint of the calibration curve.
- 4020 B.2.e. – Calculate percent recovery, plot control charts and determine control limits (see Control Charts below)
- **Real people language – analyze Phosphorus standard at a concentration of 0.5 mg/L**
 - o **Run on a 5% basis (see batch size for more information).**

Duplicate –

- NONE

Laboratory Fortified Matrix (LFM)/Laboratory Fortified Matrix Duplicate (LFMD) – goes through digestion

- 1020 B.7.– A laboratory fortified matrix (LFM) is an additional portion of a sample to which a known amount of the analyte of interest is added before sample preparation
 - o The LFM is used to evaluate analyte recover in a sample
 - o Sample batch = 5% basis = 1 every 20 samples
 - o Add a concentration that is at least 10 times the MRL (minimum reporting level), less than or equal to the midpoint of the calibration curve.
 - o Preferably use the same concentration as the LFB
- 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
 - o Add a known concentration of analyte to a randomly selected routine sample
 - o Calculate percent recovery and relative percent difference, plot control charts and determine control limits for spikes at different concentrations (see Control Charts below)

- **Real people language** – add a known amount of phosphorus to a sample and expect that amount to increase your sample concentration
 - o Run on a 5% basis (1 for every 20 samples or once per month, whichever is more frequent), see batch size for more information
 - o 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%).
 - o 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.

Continuing Calibration Verification (CCV) – does not go through digestion

- 1020 B.11.c. – Analysts periodically use a calibration standard to confirm that the instrument performance has not changed significantly since initial calibration.
 - o Verify calibration by analyzing one standard at a concentration near or at the mid-point of the calibration range.
- 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.
 - o For the calibration verification to be valid, check standards must not exceed 10% of its true value
- **Real people language** – analyze mid-range Phosphorus standard **daily** (day of).

Control Charts – 1020 B.13.

- **Real people language**
 - 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.

Corrective Action - 1020 B.5., B.8., & B.15.

QC Acceptance Criteria

- Blanks < MDL
- LFB $\pm$ 15%
- ICV/CCV $\pm$ 10%
- LFM/LFMD $\pm$ 20%
- RPD < 20%
- Reporting Limit = MDL

Batch Size –

- For samples that need to be analyzed on a 5% basis (1 for every 20 samples or once per month, whichever is more frequent) follow these criteria:
 - o If a permit stated that 3 analyses per week, we would allow for a duplicate 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!!
 - o If a permit stated 5 analyses per week, we would suggest 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!!

Calculations –

- % Recovery for LFB
 - o
$$= \frac{\text{LFB concentration}}{\text{Expected concentration}} \times 100\%$$
- RPD – relative percent differences for duplicates and LFM/LFMD
 - o
$$= \frac{\text{Difference between sample and duplicate}}{\text{Average of the sample and duplicate}} \times 100\%$$
- % Recovery for LFM – when using less than or equal to 1% spike volume compared to sample volume
 - o
$$= \frac{\text{LFM concentration} - \text{Sample concentration}}{\text{Concentration of spike}} \times 100\%$$

Minimum Detectable Concentration – 4500-Cl G.1.c. – approximately 10 µg/L (0.010 mg/L)

Initial Demonstration of Capability (DOC)

- 4020 B.1.a. - each analyst must run a known standard concentration at least four times and compare limits listed in the method.
- **Real people language – each operator running this test needs to analyze 4 samples of a chlorine or potassium permanganate (KMnO₄) standard at a concentration of 0.5 mg/L**
 - **Keep a folder for each analyst, keep a copy here**
 - **Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.**
 - **Recommend backup analyst do this once a year.**
 - **Only good for that type of instrument you are using at that plant. If you have a backup instrument made by a different manufacturer or you work at another plant with a different make/model, you would need another DOC.**
 - **DOCs demonstrate you are proficient with that one instrument.**

Method Detection Limit (MDL)

- 1020 B. 4 – As a starting point for selecting the concentration to use when determining the MDL, use an estimate of five times the estimated true detection level ($5 \times 0.010 \text{ mg/L} = 0.050 \text{ mg/L}$).
 - Ideally, prepare and analyze at least seven (7) portions of this solution over a 3-day period to ensure that the MDL determination is more representative of routine measurements as performed in the laboratory.
 - 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 and 150%, with %RSD (relative standard deviation) values $\leq 20\%$.
- 4020 B.1.b. – Verify MDL at least **annually**.
 - Ideally use pooled data from several analysts rather than data from one analyst.
- **Real people language – have several operators, who run this test, analyze chlorine or Potassium Permanganate (KMnO₄) standards at a concentration of 0.05 mg/L over several days with a total of at least 7 samples**
 - **Joe analyzes 3 samples on Monday**
 - **Bob analyzes 3 samples on Tuesday**
 - **Mary analyzes 3 samples on Wednesday**
- **Run this once a year**

Initial Calibration Verification (ICV)

- 1020 B.11.b. – Perform initial calibration using at least three concentrations of standards for linear curves.
- 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 $\pm 10\%$.

- **Real people language – prepare a set of chlorine or potassium permanganate (KMnO₄) standards in accordance with [Guidance for Secondary Standards Use in Calibration 12-19-2013](#) monthly.**

Method Blank

- 1020 B.5.– A reagent blank (method blank) consists of reagent water and all reagents that normally are in contact with a sample during the entire analytical procedure.
- 4020 B.2.d. – Include at least one method blank *daily* or with each batch of 20 or fewer samples, whichever is more frequent.
 - o If any method blanks measurements are at or above the reporting level, take immediate corrective action.
- **Real people language – analyze distilled water as a sample by adding a DPD powder pillow and waiting the 3-6 minutes before reading**
 - o **Target value is less than reporting limit**
 - **Reporting limit will be equal to your Method Detection Limit (MDL)**
 - o **Run on a 5% basis (see batch size for more information).**

Laboratory Fortified Blank (LFB)

- 1020 B.6.– A laboratory-fortified blank is a reagent water sample to which a known concentration of the analyte of interest has been added.
 - o Sample batch = 5% basis = 1 every 20 samples
 - o Use an added concentration of at least 10 times the MDL, less than or equal to the midpoint of the calibration curve.
- 4020 B.2.e. – Calculate percent recovery, plot control charts and determine control limits
- **Real people language – analyze chlorine or potassium permanganate standard at a concentration of 0.5 mg/L**
 - o **Run on a 5% basis (see batch size for more information).**

Laboratory Fortified Matrix (LFM)/Laboratory Fortified Matrix Duplicate (LFMD)

- NONE

Duplicate

- 1020 B.12.f. – Calculate RPD (relative percent difference)
- 4020 B.2.f. – Randomly select routine samples to be analyzed twice.
 - o Process duplicate sample independently through the entire sample preparation and analysis.
 - o Include at least one duplicate for each matrix type daily or with each batch of 20 or fewer samples.
- **Real people language – on a 5% basis (see batch size for more information) analyze 2 samples for chlorine, after reading one, pour out sample and start with a fresh sample**
 - o **For reporting purposes, average sample and duplicate.**
 - o **Target value should be close to the first value and have a small RPD (less than 20%)**

Continuing Calibration Verification (CCV)

- 1020 B.11.c. – Analysts periodically use a calibration standard to confirm that the instrument performance has not changed significantly since initial calibration.
 - o Verify calibration by analyzing one standard at a concentration near or at the mid-point of the calibration range.
- 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.
 - o For the calibration verification to be valid, check standards must not exceed 10% of its true value
- **Real people language**
 - o **Read Secondary Standards in accordance with Guidance for Secondary Standards Use in Calibration 12-19-2013 daily (day of).**
 - o **OR run a chlorine or potassium permanganate standard daily.**

Control Charts – 1020 B.13.

- **Real people language**
 - **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.**

Corrective Action - 1020 B.5., B.8., & B.15.

QC Acceptance Criteria

- Blanks < Method Detection Limit (MDL)
- LFB $\pm$ 15%
- ICV/CCV $\pm$ 10%
- RPD < 20%
- Reporting Limit = MDL

Batch Size

- For samples that need to be analyzed on a 5% basis (1 for every 20 samples or once per month, whichever is more frequent) follow these criteria:
 - o If a permit stated 3 analyses per week, we would allow for a duplicate 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!!
 - o 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!!
 - o If sampling only once a month, need to run QC once a month (when samples are analyzed).

Calculations

- % Recovery for LFB
 - o
$$= \frac{\text{LFB concentration}}{\text{Expected concentration}} \times 100\%$$

Total Residual Chlorine

TDEC – Fleming Training Center

S. Pratt, January 2014

- RPD – relative percent differences for duplicates
 - o = $\frac{\text{Difference between sample and duplicate}}{\text{Average of the sample and duplicate}} \times 100\%$

Total Suspended Solids, SM 2540 D, 22nd edition (1997)

Initial Demonstration of Capability (DOC)

- 2020 B.1 - each analyst must run a known standard concentration at least four times and compare limits listed in the method (under Precision). Table 2020:II lists duplicates and MB for QC only.
- Recommend running replicates and compare results and calculate the standard deviation to compare with that reported in 2540 D.5.
- **Real people language - each operator running this test needs to analyze 4 samples of a TSS Standard**
 - **Keep a folder for each analyst, keep a copy here**
 - **Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.**
 - **Recommend backup analyst do this once a year.**

Method Detection Limit (MDL)

- NONE

Initial Calibration Verification (ICV)

- 2020 B.2.a.– check instrument balance daily as stated below.
- 9020.B.4.b. Service balances annually or more often as conditions change or problems occur...

Check balance 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 technician's initials.

Recertify reference weights as specified in the certificate of calibration or at least every 5 years.

- 2540 B.2. analytical balance, with a sensitivity of 0.1 mg
- **Real people language – check balance 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.**

Method Blank

- 2020 B.2.d.– include at least 1 method blank (MB) daily or with each batch of 20 or fewer samples, whichever is more frequent.
- **Real people language – on a 5% basis (see batch size for more information) filter 100 mL of distilled water.**
 - **Should be less than 2.5 mg/L.**

Laboratory Fortified Blank (LFB)

- 1020 B.6.– A laboratory-fortified blank is a reagent water sample to which a known concentration of the analyte of interest has been added.
 - Sample batch = 5% basis

- 2020 B.2.e. – Using stock solutions, prepare fortified concentrations so they are within the calibration curve.
- **Real people language – analyze TSS Standard sample that can be prepared from recipe below or bought premade.**
 - **Run on a 5% basis (see batch size for more information).**

TSS Standard Samples

To prepare TSS check samples from dry reference material:

1. Dry the reference material* in the desiccator
2. On an analytical balance, weigh 0.1000 gram of the dry powder, put it in a 1000 mL volumetric flask, bring it to the mark with distilled or deionized water and shake well until well suspended.
3. Measure 100 mL and process as usual for environmental samples.
4. A difference of 10 mg should be obtained.
5. 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 Filtler Aid (Powder), Fisher Chemical, 500 gram bottle – Cat#C212-500

Procedure to Omit Re-drying/Re-cooling/Re-weighing Cycle

How to acquire acceptable results for the 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, re-cooled 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 cycle as stated the method (SM 2540 D.3.c.).

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.

Duplicate

- 1020 B.8. states as a minimum to include one duplicate sample with each sample set or on a 5% basis whichever is more frequent.
- 2020 B.2.f. states to include at least one duplicate for each matrix type daily or with each batch of 20 or fewer samples.
- 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.”
- 2540 D.3.c. Analyze at least 10% of all samples in duplicate.
- **Real people language – analyze 2 samples for TSS.**

Total Suspended Solids

TDEC – Fleming Training Center

S. Pratt, January 2014



- For example, filter 100 mL of effluent through filter pad A then filter another 100 mL of effluent through filter pad B. Dry, cool and weigh. Figure RPD for both samples.
- Target value should be close to the first value and have a small RPD (less than 15%)
- Analyze a duplicate at a 10% rate (see batch size for more information).
- For reporting purposes, average sample and duplicate.

Laboratory Fortified Matrix (LFM)/Laboratory Fortified Matrix Duplicate (LFMD)

- NONE

Control Charts

- NONE

Corrective Action - 1020 B.5., B.8., & B.15.

QC Acceptance Criteria

- Blanks < 2.5 mg/L
- LFB $\pm$ 15%
- RPD $\pm$ 15%

Batch Size

- Influent and Effluent are 2 different samples
- 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, that would be 6 samples per week, we would allow for a blank and LFB to be analyzed at least 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!!
 - If a permit stated 5 analyses per week, that would be 10 samples per week, we would allow once a week.
 - Pick a date and be consistent, every Monday. Mark your calendar!!
- For samples that need to be analyzed on a 10% basis or once for every 10 samples follow these criteria:
 - If a permit stated that 3 analyses per week, that would be 6 samples per week, we would allow for a duplicate to be analyzed at least 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!!
 - If a permit stated 5 analyses per week, that would be 10 samples per week, we would allow once a week.
 - Pick a date and be consistent, every Monday. Mark your calendar!!

Calculations

- % Recovery for LFB
 - o
$$= \frac{\text{LFB concentration}}{\text{Expected concentration}} \times 100\%$$
- RPD – relative percent differences for duplicates and LFM/LFMD
 - o
$$= \frac{\text{Difference between sample and duplicate}}{\text{Average of the sample and duplicate}} \times 100\%$$

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Water Resources of the United States

DOTABLES Result

Oxygen Solubility Table

Solubility of oxygen in fresh water at various temperatures and pressures
[Solubility shown in milligrams per liter. Values based on published equations by Benson and Krause (1980 and 1984).
°C, degrees Celsius; mm Hg, millimeters of mercury]

Temp. (°C)	Barometric Pressure (mm Hg)																				
	735	736	737	738	739	740	741	742	743	744	745	746	747	748	749	750	751	752	753	754	755
15.0	9.75	9.76	9.77	9.79	9.80	9.81	9.83	9.84	9.85	9.87	9.88	9.90	9.91	9.92	9.94	9.95	9.96	9.98	9.99	10.00	10.02
15.2	9.70	9.72	9.73	9.75	9.76	9.77	9.79	9.80	9.81	9.83	9.84	9.85	9.87	9.88	9.89	9.91	9.92	9.93	9.95	9.96	9.97
15.4	9.66	9.68	9.69	9.70	9.72	9.73	9.74	9.76	9.77	9.78	9.80	9.81	9.82	9.84	9.85	9.86	9.88	9.89	9.90	9.92	9.93
15.6	9.62	9.64	9.65	9.66	9.68	9.69	9.70	9.71	9.73	9.74	9.75	9.77	9.78	9.79	9.81	9.82	9.83	9.85	9.86	9.87	9.89
15.8	9.58	9.59	9.61	9.62	9.63	9.65	9.66	9.67	9.69	9.70	9.71	9.73	9.74	9.75	9.77	9.78	9.79	9.81	9.82	9.83	9.85
16.0	9.54	9.55	9.57	9.58	9.59	9.61	9.62	9.63	9.65	9.66	9.67	9.69	9.70	9.71	9.73	9.74	9.75	9.76	9.78	9.79	9.80
16.2	9.50	9.51	9.53	9.54	9.55	9.57	9.58	9.59	9.60	9.62	9.63	9.64	9.66	9.67	9.68	9.70	9.71	9.72	9.74	9.75	9.76
16.4	9.46	9.47	9.49	9.50	9.51	9.53	9.54	9.55	9.56	9.58	9.59	9.60	9.62	9.63	9.64	9.66	9.67	9.68	9.70	9.71	9.72
16.6	9.42	9.43	9.45	9.46	9.47	9.48	9.50	9.51	9.52	9.54	9.55	9.56	9.58	9.59	9.60	9.62	9.63	9.64	9.65	9.67	9.68
16.8	9.38	9.39	9.41	9.42	9.43	9.45	9.46	9.47	9.48	9.50	9.51	9.52	9.54	9.55	9.56	9.58	9.59	9.60	9.61	9.63	9.64
17.0	9.34	9.35	9.37	9.38	9.39	9.41	9.42	9.43	9.44	9.46	9.47	9.48	9.50	9.51	9.52	9.54	9.55	9.56	9.57	9.59	9.60
17.2	9.30	9.31	9.33	9.34	9.35	9.37	9.38	9.39	9.41	9.42	9.43	9.44	9.46	9.47	9.48	9.50	9.51	9.52	9.53	9.55	9.56
17.4	9.26	9.28	9.29	9.30	9.31	9.33	9.34	9.35	9.37	9.38	9.39	9.40	9.42	9.43	9.44	9.46	9.47	9.48	9.49	9.51	9.52
17.6	9.23	9.24	9.25	9.26	9.28	9.29	9.30	9.31	9.33	9.34	9.35	9.37	9.38	9.39	9.40	9.42	9.43	9.44	9.46	9.47	9.48
17.8	9.19	9.20	9.21	9.23	9.24	9.25	9.26	9.28	9.29	9.30	9.31	9.33	9.34	9.35	9.37	9.38	9.39	9.40	9.42	9.43	9.44
18.0	9.15	9.16	9.17	9.19	9.20	9.21	9.23	9.24	9.25	9.26	9.28	9.29	9.30	9.31	9.33	9.34	9.35	9.37	9.38	9.39	9.40
18.2	9.11	9.12	9.14	9.15	9.16	9.18	9.19	9.20	9.21	9.23	9.24	9.25	9.26	9.28	9.29	9.30	9.31	9.33	9.34	9.35	9.37
18.4	9.07	9.09	9.10	9.11	9.13	9.14	9.15	9.16	9.18	9.19	9.20	9.21	9.23	9.24	9.25	9.26	9.28	9.29	9.30	9.31	9.33
18.6	9.04	9.05	9.06	9.08	9.09	9.10	9.11	9.13	9.14	9.15	9.16	9.18	9.19	9.20	9.21	9.23	9.24	9.25	9.26	9.28	9.29
18.8	9.00	9.01	9.03	9.04	9.05	9.06	9.08	9.09	9.10	9.11	9.13	9.14	9.15	9.16	9.18	9.19	9.20	9.21	9.23	9.24	9.25
19.0	8.96	8.98	8.99	9.00	9.01	9.03	9.04	9.05	9.06	9.08	9.09	9.10	9.11	9.13	9.14	9.15	9.16	9.18	9.19	9.20	9.21
19.2	8.93	8.94	8.95	8.97	8.98	8.99	9.00	9.02	9.03	9.04	9.05	9.07	9.08	9.09	9.10	9.11	9.13	9.14	9.15	9.16	9.18

DOTABLES Oxygen Solubility Table Result

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Temp. (°C)	735	736	737	738	739	740	741	742	743	744	745	746	747	748	749	750	751	752	753	754	755	756	757	758	759	760
19.4	8.89	8.90	8.92	8.93	8.94	8.95	8.97	8.98	8.99	9.00	9.02	9.03	9.04	9.05	9.07	9.08	9.09	9.10	9.12	9.13	9.14	9.15	9.16	9.18	9.19	9.20
19.6	8.86	8.87	8.88	8.89	8.91	8.92	8.93	8.94	8.96	8.97	8.98	8.99	9.00	9.02	9.03	9.04	9.05	9.07	9.08	9.09	9.10	9.12	9.13	9.14	9.15	9.17
19.8	8.82	8.83	8.85	8.86	8.87	8.88	8.90	8.91	8.92	8.93	8.94	8.96	8.97	8.98	8.99	9.01	9.02	9.03	9.04	9.05	9.07	9.08	9.09	9.10	9.12	9.13
20.0	8.79	8.80	8.81	8.82	8.84	8.85	8.86	8.87	8.88	8.90	8.91	8.92	8.93	8.95	8.96	8.97	8.98	8.99	9.01	9.02	9.03	9.04	9.06	9.07	9.08	9.09
20.2	8.75	8.76	8.78	8.79	8.80	8.81	8.82	8.84	8.85	8.86	8.87	8.89	8.90	8.91	8.92	8.93	8.95	8.96	8.97	8.98	9.00	9.01	9.02	9.03	9.04	9.06
20.4	8.72	8.73	8.74	8.75	8.77	8.78	8.79	8.80	8.81	8.83	8.84	8.85	8.86	8.87	8.89	8.90	8.91	8.92	8.94	8.95	8.96	8.97	8.98	9.00	9.01	9.02
20.6	8.68	8.69	8.71	8.72	8.73	8.74	8.76	8.77	8.78	8.79	8.80	8.82	8.83	8.84	8.85	8.86	8.88	8.89	8.90	8.91	8.92	8.94	8.95	8.96	8.97	8.99
20.8	8.65	8.66	8.67	8.68	8.70	8.71	8.72	8.73	8.74	8.76	8.77	8.78	8.79	8.81	8.82	8.83	8.84	8.85	8.87	8.88	8.89	8.90	8.91	8.93	8.94	8.95
21.0	8.61	8.63	8.64	8.65	8.66	8.67	8.69	8.70	8.71	8.72	8.73	8.75	8.76	8.77	8.78	8.79	8.81	8.82	8.83	8.84	8.85	8.87	8.88	8.89	8.90	8.92
21.2	8.58	8.59	8.60	8.62	8.63	8.64	8.65	8.66	8.68	8.69	8.70	8.71	8.72	8.74	8.75	8.76	8.77	8.78	8.80	8.81	8.82	8.83	8.84	8.86	8.87	8.88
21.4	8.55	8.56	8.57	8.58	8.60	8.61	8.62	8.63	8.64	8.65	8.67	8.68	8.69	8.70	8.71	8.73	8.74	8.75	8.76	8.77	8.79	8.80	8.81	8.82	8.83	8.85
21.6	8.51	8.53	8.54	8.55	8.56	8.57	8.59	8.60	8.61	8.62	8.63	8.65	8.66	8.67	8.68	8.69	8.70	8.72	8.73	8.74	8.75	8.76	8.78	8.79	8.80	8.81
21.8	8.48	8.49	8.51	8.52	8.53	8.54	8.55	8.56	8.58	8.59	8.60	8.61	8.62	8.64	8.65	8.66	8.67	8.68	8.69	8.71	8.72	8.73	8.74	8.75	8.77	8.78
22.0	8.45	8.46	8.47	8.48	8.50	8.51	8.52	8.53	8.54	8.55	8.57	8.58	8.59	8.60	8.61	8.63	8.64	8.65	8.66	8.67	8.68	8.70	8.71	8.72	8.73	8.74
22.2	8.42	8.43	8.44	8.45	8.46	8.47	8.49	8.50	8.51	8.52	8.53	8.55	8.56	8.57	8.58	8.59	8.60	8.62	8.63	8.64	8.65	8.66	8.67	8.69	8.70	8.71
22.4	8.38	8.40	8.41	8.42	8.43	8.44	8.45	8.47	8.48	8.49	8.50	8.51	8.52	8.54	8.55	8.56	8.57	8.58	8.59	8.61	8.62	8.63	8.64	8.65	8.67	8.68
22.6	8.35	8.36	8.38	8.39	8.40	8.41	8.42	8.43	8.45	8.46	8.47	8.48	8.49	8.50	8.52	8.53	8.54	8.55	8.56	8.57	8.59	8.60	8.61	8.62	8.63	8.64
22.8	8.32	8.33	8.34	8.35	8.37	8.38	8.39	8.40	8.41	8.42	8.44	8.45	8.46	8.47	8.48	8.49	8.51	8.52	8.53	8.54	8.55	8.56	8.57	8.59	8.60	8.61
23.0	8.29	8.30	8.31	8.32	8.33	8.35	8.36	8.37	8.38	8.39	8.40	8.42	8.43	8.44	8.45	8.46	8.47	8.49	8.50	8.51	8.52	8.53	8.54	8.55	8.56	8.57
23.2	8.26	8.27	8.28	8.29	8.30	8.31	8.33	8.34	8.35	8.36	8.37	8.38	8.40	8.41	8.42	8.43	8.44	8.45	8.46	8.48	8.49	8.50	8.51	8.52	8.53	8.55
23.4	8.23	8.24	8.25	8.26	8.27	8.28	8.29	8.31	8.32	8.33	8.34	8.35	8.36	8.38	8.39	8.40	8.41	8.42	8.43	8.44	8.46	8.47	8.48	8.49	8.50	8.51
23.6	8.19	8.21	8.22	8.23	8.24	8.25	8.26	8.27	8.29	8.30	8.31	8.32	8.33	8.34	8.36	8.37	8.38	8.39	8.40	8.41	8.42	8.44	8.45	8.46	8.47	8.48
23.8	8.16	8.18	8.19	8.20	8.21	8.22	8.23	8.24	8.26	8.27	8.28	8.29	8.30	8.31	8.32	8.34	8.35	8.36	8.37	8.38	8.39	8.40	8.42	8.43	8.44	8.45
24.0	8.13	8.14	8.16	8.17	8.18	8.19	8.20	8.21	8.22	8.24	8.25	8.26	8.27	8.28	8.29	8.30	8.32	8.33	8.34	8.35	8.36	8.37	8.38	8.40	8.41	8.42
24.2	8.10	8.11	8.13	8.14	8.15	8.16	8.17	8.18	8.19	8.20	8.22	8.23	8.24	8.25	8.26	8.27	8.28	8.30	8.31	8.32	8.33	8.34	8.35	8.36	8.38	8.39
24.4	8.07	8.08	8.10	8.11	8.12	8.13	8.14	8.15	8.16	8.17	8.19	8.20	8.21	8.22	8.23	8.24	8.25	8.27	8.28	8.29	8.30	8.31	8.32	8.33	8.34	8.36
24.6	8.04	8.05	8.07	8.08	8.09	8.10	8.11	8.12	8.13	8.14	8.16	8.17	8.18	8.19	8.20	8.21	8.22	8.23	8.25	8.26	8.27	8.28	8.29	8.30	8.31	8.32
24.8	8.01	8.02	8.04	8.05	8.06	8.07	8.08	8.09	8.10	8.11	8.13	8.14	8.15	8.16	8.17	8.18	8.19	8.20	8.22	8.23	8.24	8.25	8.26	8.27	8.28	8.29
25.0	7.98	7.99	8.01	8.02	8.03	8.04	8.05	8.06	8.07	8.08	8.10	8.11	8.12	8.13	8.14	8.15	8.16	8.17	8.18	8.20	8.21	8.22	8.23	8.24	8.25	8.26
25.2	7.95	7.96	7.98	7.99	8.00	8.01	8.02	8.03	8.04	8.05	8.07	8.08	8.09	8.10	8.11	8.12	8.13	8.14	8.15	8.17	8.18	8.19	8.20	8.21	8.22	8.23
25.4	7.92	7.94	7.95	7.96	7.97	7.98	7.99	8.00	8.01	8.02	8.04	8.05	8.06	8.07	8.08	8.09	8.10	8.11	8.12	8.14	8.15	8.16	8.17	8.18	8.19	8.20
25.6	7.90	7.91	7.92	7.93	7.94	7.95	7.96	7.97	7.98	8.00	8.01	8.02	8.03	8.04	8.05	8.06	8.07	8.08	8.10	8.11	8.12	8.13	8.14	8.15	8.16	8.17

DOTABLES Oxygen Solubility Table Result

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Temp.	Barometric Pressure (mm Hg)																									
(°C)	735	736	737	738	739	740	741	742	743	744	745	746	747	748	749	750	751	752	753	754	755	756	757	758	759	760
25.8	7.87	7.88	7.89	7.90	7.91	7.92	7.93	7.94	7.96	7.97	7.98	7.99	8.00	8.01	8.02	8.03	8.04	8.05	8.07	8.08	8.09	8.10	8.11	8.12	8.13	8.14
26.0	7.84	7.85	7.86	7.87	7.88	7.89	7.90	7.91	7.93	7.94	7.95	7.96	7.97	7.98	7.99	8.00	8.01	8.03	8.04	8.05	8.06	8.07	8.08	8.09	8.10	8.11
26.2	7.81	7.82	7.83	7.84	7.85	7.86	7.88	7.89	7.90	7.91	7.92	7.93	7.94	7.95	7.96	7.97	7.99	8.00	8.01	8.02	8.03	8.04	8.05	8.06	8.07	8.08
26.4	7.78	7.79	7.80	7.81	7.82	7.84	7.85	7.86	7.87	7.88	7.89	7.90	7.91	7.92	7.93	7.95	7.96	7.97	7.98	7.99	8.00	8.01	8.02	8.03	8.04	8.06
26.6	7.75	7.76	7.77	7.79	7.80	7.81	7.82	7.83	7.84	7.85	7.86	7.87	7.88	7.89	7.91	7.92	7.93	7.94	7.95	7.96	7.97	7.98	7.99	8.00	8.02	8.03
26.8	7.72	7.74	7.75	7.76	7.77	7.78	7.79	7.80	7.81	7.82	7.83	7.84	7.86	7.87	7.88	7.89	7.90	7.91	7.92	7.93	7.94	7.95	7.96	7.98	7.99	8.00
27.0	7.70	7.71	7.72	7.73	7.74	7.75	7.76	7.77	7.78	7.79	7.81	7.82	7.83	7.84	7.85	7.86	7.87	7.88	7.89	7.90	7.91	7.93	7.94	7.95	7.96	7.97
27.2	7.67	7.68	7.69	7.70	7.71	7.72	7.73	7.75	7.76	7.77	7.78	7.79	7.80	7.81	7.82	7.83	7.84	7.85	7.86	7.88	7.89	7.90	7.91	7.92	7.93	7.94
27.4	7.64	7.65	7.66	7.67	7.69	7.70	7.71	7.72	7.73	7.74	7.75	7.76	7.77	7.78	7.79	7.80	7.81	7.83	7.84	7.85	7.86	7.87	7.88	7.89	7.90	7.91
27.6	7.61	7.63	7.64	7.65	7.66	7.67	7.68	7.69	7.70	7.71	7.72	7.73	7.74	7.75	7.77	7.78	7.79	7.80	7.81	7.82	7.83	7.84	7.85	7.86	7.87	7.88
27.8	7.59	7.60	7.61	7.62	7.63	7.64	7.65	7.66	7.67	7.68	7.69	7.71	7.72	7.73	7.74	7.75	7.76	7.77	7.78	7.79	7.80	7.81	7.82	7.83	7.84	7.86
28.0	7.56	7.57	7.58	7.59	7.60	7.61	7.62	7.64	7.65	7.66	7.67	7.68	7.69	7.70	7.71	7.72	7.73	7.74	7.75	7.76	7.77	7.79	7.80	7.81	7.82	7.83
28.2	7.53	7.54	7.55	7.57	7.58	7.59	7.60	7.61	7.62	7.63	7.64	7.65	7.66	7.67	7.68	7.69	7.70	7.71	7.73	7.74	7.75	7.76	7.77	7.78	7.79	7.80
28.4	7.51	7.52	7.53	7.54	7.55	7.56	7.57	7.58	7.59	7.60	7.61	7.62	7.63	7.65	7.66	7.67	7.68	7.69	7.70	7.71	7.72	7.73	7.74	7.75	7.76	7.77
28.6	7.48	7.49	7.50	7.51	7.52	7.53	7.54	7.55	7.57	7.58	7.59	7.60	7.61	7.62	7.63	7.64	7.65	7.66	7.67	7.68	7.69	7.70	7.71	7.72	7.73	7.75
28.8	7.45	7.46	7.48	7.49	7.50	7.51	7.52	7.53	7.54	7.55	7.56	7.57	7.58	7.59	7.60	7.61	7.62	7.63	7.64	7.65	7.67	7.68	7.69	7.70	7.71	7.72
29.0	7.43	7.44	7.45	7.46	7.47	7.48	7.49	7.50	7.51	7.52	7.53	7.54	7.55	7.56	7.58	7.59	7.60	7.61	7.62	7.63	7.64	7.65	7.66	7.67	7.68	7.69
29.2	7.40	7.41	7.42	7.43	7.44	7.45	7.47	7.48	7.49	7.50	7.51	7.52	7.53	7.54	7.55	7.56	7.57	7.58	7.59	7.60	7.61	7.62	7.63	7.64	7.65	7.66
29.4	7.38	7.39	7.40	7.41	7.42	7.43	7.44	7.45	7.46	7.47	7.48	7.49	7.50	7.51	7.52	7.53	7.54	7.55	7.56	7.58	7.59	7.60	7.61	7.62	7.63	7.64
29.6	7.35	7.36	7.37	7.38	7.39	7.40	7.41	7.42	7.43	7.44	7.45	7.47	7.48	7.49	7.50	7.51	7.52	7.53	7.54	7.55	7.56	7.57	7.58	7.59	7.60	7.61
29.8	7.32	7.34	7.35	7.36	7.37	7.38	7.39	7.40	7.41	7.42	7.43	7.44	7.45	7.46	7.47	7.48	7.49	7.50	7.51	7.52	7.53	7.54	7.55	7.56	7.57	7.58
30.0	7.30	7.31	7.32	7.33	7.34	7.35	7.36	7.37	7.38	7.39	7.40	7.41	7.42	7.43	7.44	7.46	7.47	7.48	7.49	7.50	7.51	7.52	7.53	7.54	7.55	7.56

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