

## Biochemical Oxygen Demand Audit Checklist \* (BOD and CBOD)

Based on NR 219 (2004), NR 149 (2008) and Standard Methods 5210 B (18th, 19th and 20th Editions)

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	Sample Storage and Pretreatment	Y	N	Notes	Citation
1	Are BOD samples set up within 2 hours or stored at $\leq 6$ °C prior to analysis?				NR 219; Table F
2	Are samples set up within hold time ( $\leq 48$ hours)?				NR 219; Table F; 5210 B
3	Are samples checked for residual chlorine if applicable?				5210 B; 4.e.(2)
4	If residual chlorine is found is the sample neutralized?				5210 B; 4.e.(2)
5	Is the pH of samples checked prior to set up?				20th 5210B; 4.e.
6	Are samples pH adjusted to pH 6.5 - 7.5 (if not in pH 6.0 - 8.5 initially)?				20th 5210B; 4.e.
7	If pH adjustment is done is the amount of acid or base used limited to $\leq 0.5\%$ of sample volume?				5210B; 4.e.(1)
8	Are samples warmed to 20 +/-3 °C before analysis?				20th 5210B; 1.b.
9	Are samples over the 100% DO saturation value identified and treated for super saturation?				5210B; 4.e.(4)

	Equipment	Y	N	Notes	Citation
10	Are all necessary reagents and glassware available? Reagents purchased _____ or prepared _____?				5210 B; 2. & 3.
11	Is the DO meter properly calibrated on each analysis day? Water sat.air _____ Air sat. water _____ or Winkler _____				NR149.44(6)(d)(1); NR149.44(6)(n)
12	Are thermometers used in the lab for incubators, refrigerators, samplers, sample measurement etc. calibrated annually with a NIST traceable thermometer?				NR149.44(3)(d)
13	Does the incubator maintain samples at 20 +/- 1 °C during the 5 day test period?				5210B; 2.b.
14	Is the room temperature sufficiently controlled to meet the test requirements of 20 +/- 3 °C?				20th 5210B; 4.

	Sample Seeding	Y	N	Notes	Citation
15	What is the seed source and which samples are seeded?				5210B; 4.d.
16	Is the lot of seed recorded and linked to analysis.				149.45(1)
17	Is the seed properly prepared and measured?				5210B; 4.d.(1)
18	Are industrial, disinfected (UV or chlorine), or pH-adjusted samples seeded?				5210B; 4.e.(1&2)
19	Are at least two seed controls run that regularly meet depletion criteria?				5210B; 4.d.(2)
20	Are seed correction factors properly calculated and used to adjust results of seeded samples?				5210B; 4.d.(2)

BOD/CBOD Checklist

	General Procedural Requirements	Y	N	Notes	Citation
21	Are the proper reagents prepared _____ or purchased _____ for dilution water preparation?				5210B; 3.
22	Are reagents properly labeled, recorded & good condition?				5210B; 3.
23	Is the dilution water properly made and stored?				5210B; 4.a.& b.
24	For sample dilutions of greater than 1:100 is a preliminary dilution done and recorded?				5210 B; 4.f.(2)
25	Are sample volumes adjusted so that depletion criteria are met as often as possible?( <i>depletion of &gt; 2 mg/L DO &amp; remainder of &gt; 1 mg/L DO</i> )				5210B; 4.f.
26	Are only samples and seed controls that meet criteria ( <i>depletion of &gt; 2 mg/L DO &amp; remainder of &gt; 1 mg/L DO</i> ) used on the results?				5210B; 5.
27	Do at least two sample volumes meet the depletion criteria?				20th 5210 B; 4.f.
28	Are at least two sample dilutions run for each sample?				5210B; 4.f.
29	For samples over 201 mL are additional nutrients added? Nutrients:powder _____ or liquid _____(0.33 mL per 300 mL)?				20th 5210B; 4.f.
30	Are sample bottles water sealed prior to incubation?				20th 5210B; 4.f. 18th/19th 5210B; 2.a.
31	If nitrification inhibitor is used, does the lab have certification or registration for CBOD?				NR149.13 Table 2
32	Are CBOD samples properly labeled and the results reported as CBOD?				NR 149.39(3)C(7)
33	Have sliding BODs been observed?				5210B; 4.e.(3)
34	If sliding BODs have been observed have steps been taken to identify the source of the toxicity?				5210B; 4.e.(3)
35	Are BOD values properly calculated for all samples?				5210B; 5.

	LCS - Glucose-Glutamic Acid (GGA) Standard	Y	N	Notes	Citation
36	Is GGA standard properly prepared or commercially purchased?				5210B; 3.h.
37	Is the lot of GGA standard recorded and linked to analysis.				149.45(1)(a)
38	Is GGA standard analyzed at a 2% dilution (6 mL to 300 mL) using a concentration that yields 3 mg/L glucose and 3 mg/L glutamic acid in the GGA test bottle?				5210B; 4.c.
39	Are GGA standards one per batch or weekly at a minimum (if < 20 samples are run in a week)?				NR149.48(4)(b)
40	Are seed controls run and correctly applied to GGA data?				5210B; 4.d.(2)
41	Do GGA results meet the 198 +/- 30.5 mg/L BOD standard? (167.5 - 228.5) Multiple GGA standards cannot be averaged.				5210B; 6.

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<b>BOD-Specific Quality Control</b> (refer to the QC and Records checklist for other QC/records requirements)		<b>Y</b>	<b>N</b>	<b>Notes</b>	<b>Citation</b>
42	Do all samples, standards and seed controls used to calculate results meet the depletion criteria?				5210B; 5.
43	If criteria are not met are data excluded from calculations or qualified if there are no acceptable dilutions to use?				5210B; 5.
44	Is a dilution water blank run with each batch of samples and/or batch of dilution water?				5210B; 4.h.
45	Do dilution water blanks meet the depletion limit of < 0.2 mg/L DO?				5210B; 4.h.

<b>Other Observations</b>

## Total Suspended Solids (TSS) Checklist \*

Based on NR 149 (2008), NR 219 (2004) and Standard Methods (18th, 19th and 20th Editions)

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Sample Storage and Pretreatment		Y	N	Notes	Citation
1	Are TSS samples stored at $\leq 6$ °C until analyzed?				NR 219; Table F
2	Are samples analyzed within the hold time of 7 days?				NR 219; Table F
3	Is sample well stirred using a magnetic stirrer prior to analysis?				2540D; 3.c.

Equipment		Y	N	Notes	Citation
4	Are glass fiber filter disks (without organic binder) approved brands or other products that give equivalent results?				2540 D; 2. 2540C; 2a.
5	Does the lab have an analytical balance capable of weighing to 0.1 mg?				2540 D; 2. 2540C; 2f.
6	Is a monthly balance-weight check done with a certified mg and a gram weight?				NR 149.44(3)g
7	Are balance verification weights sent in every 5 years for certification?				NR 149.44(3)(g)(2)
8	Is a suitable desiccator with color indicating desiccant or an instrumental indicator used?				2540 D; 2. 2540C; 2d.
9	Is the drying oven capable of maintaining 103 - 105 °C?				2540 D; 2. 2540C; 2e.

General Procedural Observations		Y	N	Notes	Citation
10	Is the fiber filter disk rinsed with three 20mL portions of reagent-grade water prior to drying?				2540D; 3.a.
11	Is the filter dried at 103-105°C for at least an hour, cooled in desiccator, and then weighed?				2540D; 3.a.
12	Is the filter re-dried and re-weighed until weight change is less than 0.5 mg?				2540D; 3.a.
13	If filter is not re-dried is the filter dried overnight with a quarterly redry done to prove dryness is achieved?				Lab Cert Allowance
14	Is sample size appropriate to yield between 1.0 and 200 mg of residue?				Lab Cert Allowance 2540D; 3.b.
15	Is the sample filtered and then washed with at least three 10mL portions of reagent-grade water?				2540D; 3.c.
16	Are very large particles removed from the sample if it is deemed that they are not representative?				2540D; 1.b.
17	Is the filter with sample dried at 103-105°C for at least an hour, cooled in desiccator and then weighed?				2540D; 3.c.
18	Is sample re-dried and re-weighed until residue weight change is less than 0.5 mg?				2540D; 3.c.
19	If sample is not re-dried is it dried overnight (8hrs+) with a quarterly redry done to prove dryness is achieved?				Lab Cert Allowance
20	Are calculations of suspended solids done correctly?				2540D; 4.

## Total Phosphorus - Ascorbic Acid Method Checklist \*

Based on NR 219 (2004), NR 149 (2008), HACH 8190 and Standard Methods (18th, 19th and 20th Editions)

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Sample Storage and Pretreatment		Y	N	Notes	Citation
1	Are phosphorus samples stored at $\leq 6^{\circ}\text{C}$ prior to analysis?				NR 219; Table F
2	If analysis is not started immediately ( $\leq 15$ minutes) is the sample preserved at pH of $<2$ using sulfuric acid?				NR 219; Table F
3	Are samples analyzed within the hold time of 28 days (preserved at pH $<2$ and $\leq 6^{\circ}\text{C}$ )?				NR 219; Table F
4	What is digested? ___ samples ___ Cal standards ___ cal blank ___ instr.blank ___ MB ___ LCS ___ CCV	NA	NA		NA

Equipment		Y	N	Notes	Citation
5	Are all apparatus, reagents and glassware available?				4500-P-E; 2&3.
6	Does glassware cleaning include a hydrochloric acid rinse or is clean glassware stored filled with distilled water and subjected to occasional acid rinse?				4500-P-E; 2.

Standard Persulfate Digestion		Y	N	Notes	Citation
7	Is combined reagent made fresh daily & used w/in 4 h?				4500 P-E; 3.
8	Are reagent to sample volumes proportional to those required by the method?				4500-P-B(5)c.
9	Is mixed sample measured into a flask, then phenolphthalein indicator added? If red color appears is sulfuric acid solution added until it clears?				4500-P-B(5)c.
10	Then is sulfuric acid solution and either ammonium persulfate or potassium persulfate added to the flask?				4500-P-B(5)c.
11	Are samples gently boiled on a hot plate for 30-40 minutes or until the volume is 10 mL?				4500-P-B(5)c.
12	Alternatively, are samples heated for 30 minutes in an autoclave or pressure cooker at 98 to 137 kPa?				4500-P-B(5)c.
13	Is the sample then cooled, diluted with RW, phenolphth added & then neutralized w/NaOH to a faint pink color?				4500-P-B(5)c.
14	Is digested sample pipetted into a flask, phenolphthalein indicator added, and if red 5N sulfuric acid added until the solution is clear?				4500-P-E(4)a.
15	Is combined reagent then added and mixed?				4500-P-E(4)a.

HACH Persulfate Digestion		Y	N	Notes	Citation
16	If samples are preserved prior to analysis, are they warmed to room temperature and adjusted to pH 7 before beginning digestion (record volumes of base added)?				HACH 8190
17	Is 5 mL of neutral sample added to a test vial (already contains acid) & 1 K persulfate pillow added.				HACH 8190
18	Vial is capped tightly and shaken, then placed in the pre-heated block digester at $150^{\circ}\text{C}$ for 30 minutes?				HACH 8190
19	Is the sample cooled, 2 mL of 1.54N sodiumOH std solution added to the vial, then capped and shaken?				HACH 8190
20	Is contents of one phosver3 phosphate pillow then added to vial, capped, shaken & 2 min timer started?				HACH 8190

Calibration/Sample Measurement		Y	N	Citation
21	Is the spectrophotometer calibrated annually with at least 3 standards and a blank? Also calibrated after CCV failure or non-routine maintenance?			NR149.44(6)(d)
22	Is the calibration curve acceptable (0.995 or better), and properly used to derive sample concentrations based on absorbance data?			NR149.44(6)(g)(2)
23	Are samples above the upper end of the calibration curve diluted and re-analyzed with minimal dilution?			NR 149.44(6)(L)
24	For samples digested using autoclave/hotplate Is the sample absorbance measured between 10 and 30 minutes after the addition of the combined reagent?			4500-P-E(4)a.
25	For samples digested following HACH method is the vial wiped clean and the absorbance measured between 2 and 8 minutes after phosver3 pillow packet was added?			HACH 8190
26	Is absorbance measured at the appropriate wavelength - (610, 660, 880, or 890 depending on the method)?			4500-P-E HACH 8190
27	Is an instrument blank (RW no CR) used to zero and a calibration blank (RW with CR) used as the 0 point in the curve?			149.44(5)(h)
28	Is a method blank run with each sample batch and processed in the same manner as the samples?			NR149.48(3)(b)
29	Is method blank below highest of: LOD, 5% regulatory limit or 10% sample concentration?			NR149.48(3)(d)
30	On days when ICAL (initial calibration) is analyzed is a 2nd source ICV analyzed right after calibration?			NR149.44(6)(i)
31	If 2nd source ICV is not run, is a QCS run three times a year?			NR149.48(5)(a)
32	Are CCVs analyzed after every 20 samples and before sample analysis if no ICAL run that day?			NR149.44(7)(a)
33	Does CCV meet the 10% recovery criteria?			NR149.44(7)(d)(1)
34	Is an LCS analyzed with each batch (unless ICAL, ICV and CCV are digested then the CCV can cover LCS)?			NR149.48(4)(a)

Other Observations				

## Ammonia Ion-Selective Electrode (ISE) Method Checklist \*

Based on NR 149 (2008), NR 219 (2004), EPA Method 350.3, and Standard Methods (18th, 19th and 20th editions)

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Sample Storage and Pretreatment		Y	N	Notes	Citation
1	Are ammonia samples stored at $\leq 6^{\circ}\text{C}$ prior to analysis?				NR 219; Table F
2	If analysis is not started immediately ( $\leq 15$ minutes) is the sample preserved at pH of $<2$ using sulfuric acid?				NR 219; Table F
3	Are samples analyzed within the hold time of 28 days (stored at pH $<2$ and $\leq 6^{\circ}\text{C}$ )?				NR 219; Table F
4	Are samples and standards at room temperature before analysis starts?				19 & 20:4500 NH3-D; 4b. 18:4500 NH3-F; 4b.
5	Are samples distilled unless they are wastewater samples and a copy of the SLH distillation study is on file?				Lab Cert Allowance NR 219 Table B
6	If sample concentration is above the top standard is the sample diluted and re-analyzed?				4500 NH3-B; 4.e.

Equipment		Y	N	Notes	Citation
7	Are all the necessary apparatus, reagents and glassware available?				19&20: 4500-NH3-D; 2&3. 18: 4500 NH3-F; 2 & 3 350.3 (5&6)
8	Is ammonia -free water being used for analysis?				4500-NH3-B;3.a. 350.3 (6.0)
9	Is the ammonia probe maintained properly? <i>Including regular membrane changes, filling solution changes, proper storage (in 100 or 1000 ppm std.), etc.?</i>				NR149.44(2)(a)

Calibration/Sample Measurement		Y	N	Notes	Citation
10	Is 100 mLs of sample/standard used to do the analysis?				4500 NH3-B; 4.e.
11	Are samples slowly stirred using a thermally insulated magnetic stirrer?				4500 NH3-B; 4.b.
12	Is the NaOH buffer solution added to samples and standards <u>after</u> the probe is immersed in the sample?				19&20:4500 NH3-D; 4b 18:4500 NH3-F;4b. 350.3 (7.2)
13	Is enough buffer used to raise the sample pH above 11?				19&20:4500 NH3-D; 4b. 18:4500 NH3-F;4b. 350.3 (7.2)
14	If more than 1mL of buffer is added to samples (and less is added to the calibration standards) is the amount noted and used in the calculation?				19&20:4500 NH3-D; 4b. 18:4500 NH3-F;5.
15	Is the meter allowed sufficient time to stabilize (about 3 minutes on the low level standards/samples)?				4500 NH3-B; 4.b.& e.
16	Is the meter calibrated daily with at least 2 standards?				NR 149.44(6)(d)(2)
17	Is the meter calibrated starting with the lowest standard and progressing to higher standards?				19&20:4500 NH3-D; 4b. 18:4500 NH3-F;4b. 350.3 (7.2)
18	Does the calibration yield a slope of 59 mV (54-60) or slope required by meter manufacturer? <small>Orion -54 to -60 mV, Corning -50 to -60 mV, Acumet -55 to -63 mV Hach -54 to -62 mV</small>				19&20:4500 NH3-D; 4c. 18:4500 NH3-F;4c.

## Quality Control (QC) and Record Requirements Checklist \* for BOD, TSS, Ammonia and Phosphorus

Based on NR 149 (2008). Indicate yes (Y), no (N) or not applicable (NA) for each analyte. SC indicates that you should see checklist (SC) that is specific to that analyte.

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	Quality Control	BOD	TSS	NH3	P	Notes	Citation
1	Method blank per analytical batch. Must meet MB criteria.		NA				NR149.48(3)(a)
2	Is corrective action taken if the method blank is above the LOD, 5% of regulatory limit or 10% of the sample concentration (whichever is <u>highest</u> )?	SC	NA				NR149.48(3)(d)
3	ICAL daily BOD, NH3, monthly TSS, annually TP (minimum). 1 std BOD, minimum 2 for NH3 and minimum 3 for TP.						NR 149.44(6)(d)(2)
4	Second source initial calibration verification (ICV) immediately after ICAL (not required if run QCS)	NA	NA	NA			NR149.44(6)(i)
5	If 2nd source ICV is not run, is a QCS run three times a year?	NA	NA	NA			NR149.48(5)(a)
6	Continuing calibration verification (CCV) if ICAL then after every 20 samples. If no ICAL then run before analysis and after every 20 samples.	SC	NA				NR 149.48(4)
7	Lab control sample (LCS) 1 per prep batch unless ICAL, ICV and CCV are digested then CCV=LCS	NA	NA				NR149.48(4)(a)
8	LOD determined annually or with major change.	NA	NA				NR 149.48(2)(d)
9	Replicates for TSS 20th edition only. 1/10	NA		NA	NA		2540(3)c
10	When QC Limits are exceeded is corrective action taken and followed up on?						NR149.38



QC/Records Checklist

	Records	BOD	TSS	NH3	P	Notes	Citation
11	Are all records available for last 3 years of analysis?						NR149.39(1)c
12	Are records kept in secure manner, recorded in ink or stored electronically w/ safeguards?						NR149.39(1)(f)
13	Are SOPs documenting all methods and lab procedures available, up to date and followed?						NR149.39(1)(f)
14	Are clear sample handling and preservation records kept?						NR149.39(c)(1-6)
15	Are standards and reagents used in analysis fully traceable?						NR149.39
16	Is the raw data (i.e. absorbance, millivolts) recorded for all samples and standards?						NR149.39(c)(12)
17	Are sample results clearly traceable to the calibration curve that was used to generate them?						NR149.39(c)(11)
18	Are equipment maintenance records for all analytical equipment kept?						NR149.39(c)(15)
19	Records of Initial demonstration of capability (IDC) are kept for each analysts and each parameter.						NR149.36(3)(b)
20	Chain of custody documents available unless the lab only collects, transports and analyzes their own samples.						
21	Are records associated with QCS (if chosen to run) and PT/reference samples available?						NR149.39(2)(d)
22	Are records of corrective actions taken in response to QC failures available?						NR149.39(c)(14)
23	Does corrective action include qualification of data on data report or DMR?						NR149.47(1)(e)(13)

Other Observations	