

Biochemical Oxygen Demand Do's and Don'ts



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Classic



Historic



Outline

- Definition and Overview
- Procedure
 - Sampling
 - Blanks
 - Controls
 - Seed
 - Nitrification Inhibitors
 - Dilutions
 - Meter calibration
- Calculations
- Troubleshooting common problems
- Resources, Links...

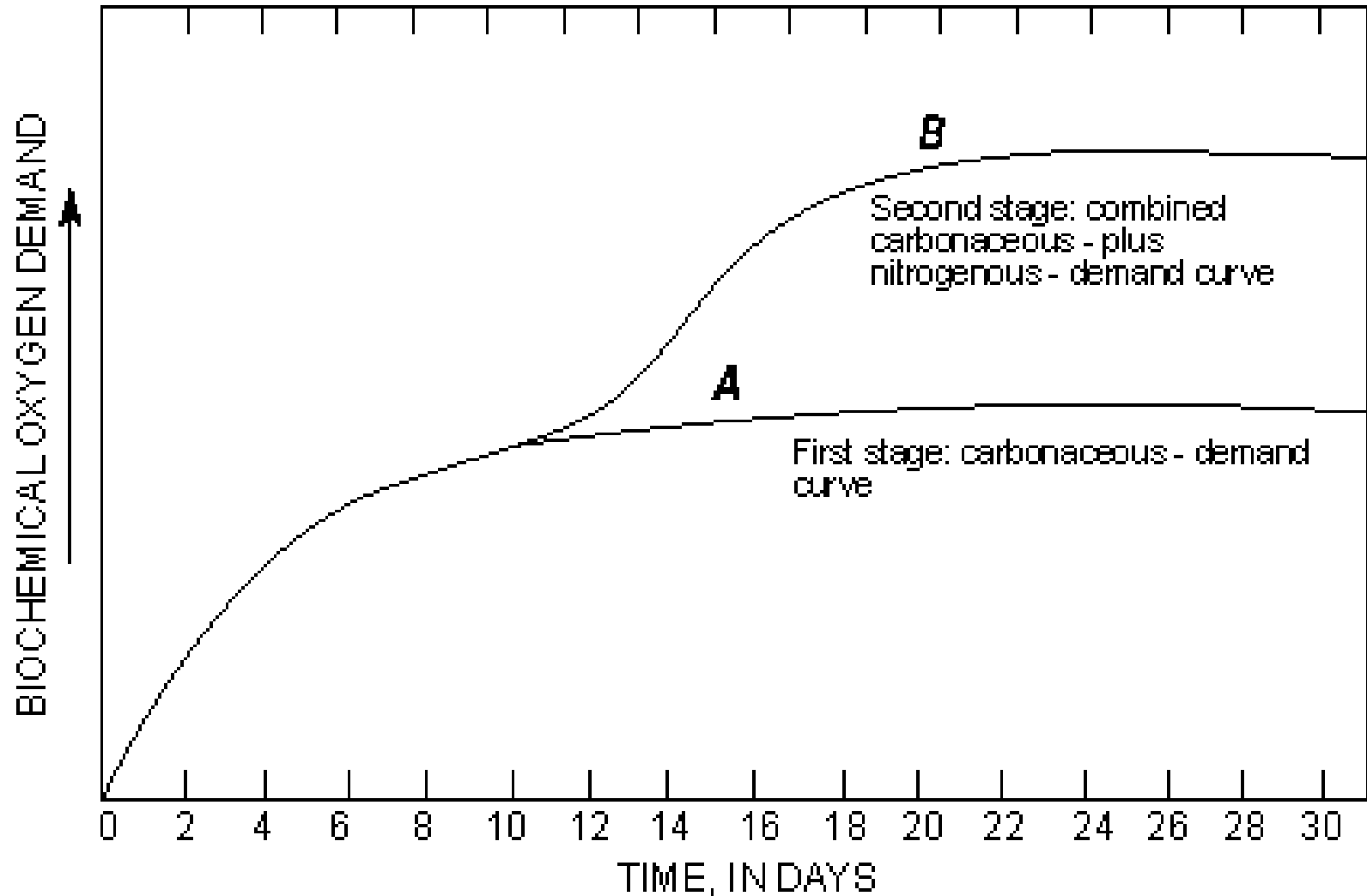
Approved Methods:

- **Standard Methods 5210 B – 2001**
- If/When new EPA Method Update Rule is published the latest approved BOD method will be 5210B – 2011 (22nd Edition)
- Hach Method 10360 Rev 1.2 – 2011 allows the use of a Luminescence Based sensor.

What does BOD measure?

- The Biochemical Oxygen Demand is an empirical test that measures the amount of **oxygen** used by **bacteria** as they metabolize **organic matter** at **20 °Celsius**, in the dark, usually over a **5 day** period.
- (pH 6.5 to 7.5 is best)
- Two types of bacterial activity:
 - Carbonaceous
 - Nitrogenous

The BOD Curve



BOD's are simple

- Calibrate your DO meter.
- Put samples in BOD bottles, add dilution water and measure DO.
- Put bottles in incubator.

- Repeat again in 5 days

Murphy's Law

- IF ANYTHING CAN GO WRONG, IT WILL!
- H. Tim Neketin: “Murphy was an optimist.”

Samples

- Clean effluent sampler hoses at least weekly with bleach to remove build up of nitrifying bacteria
- Warm samples to room temp: $20 \pm 3^{\circ}\text{C}$
- Check pH of samples. If not between 6.0 and 8.5, then adjust to between 6.5 and 7.5 with 1 Normal H_2SO_4 or 1 Normal NaOH ...
- *Recent method says adjust to 7.0 to 7.2*

Bottles, jugs, glassware

- Use **non-phosphate detergent** that leaves glassware sparkly clean.

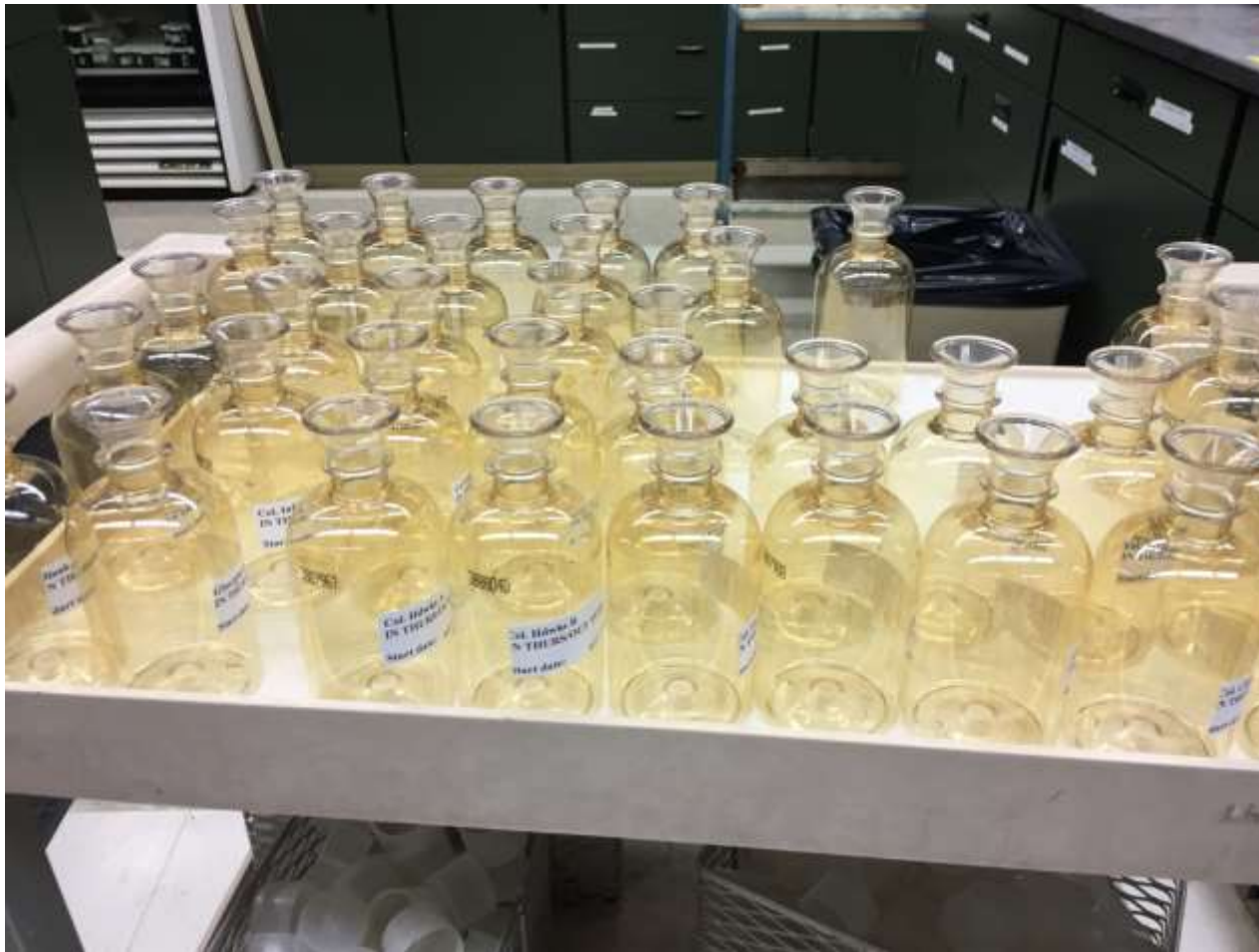
Contrad liquid

Neodisher (from Miele) or Tergajet(?)

Don't use Alconox, Versatone

- To remove dried on film from bottles use 1:1 Sulfuric Acid (or HCl in a hood).
 - Don't use Chromic Acid (honest!)
- *Disposable BOD bottles avoid cleaning problems*

Disposable BOD Bottles



Dilution Water

- Source:

 - DI+carbon filtered water

 - Steam-distilled commercial water

 - Distilled is risky as is straight DI or RO/DI water

 - Tap Water is a no-no! (Chlorine, Copper, etc.)

- Aerate & store at room temp. Add nutrients only 1 day ahead of use.
- Bleach storage jugs and hose at least every 2 weeks.

Dissolved Oxygen Meters



(Museum Quality circa 1980.... Please Do Not Touch)

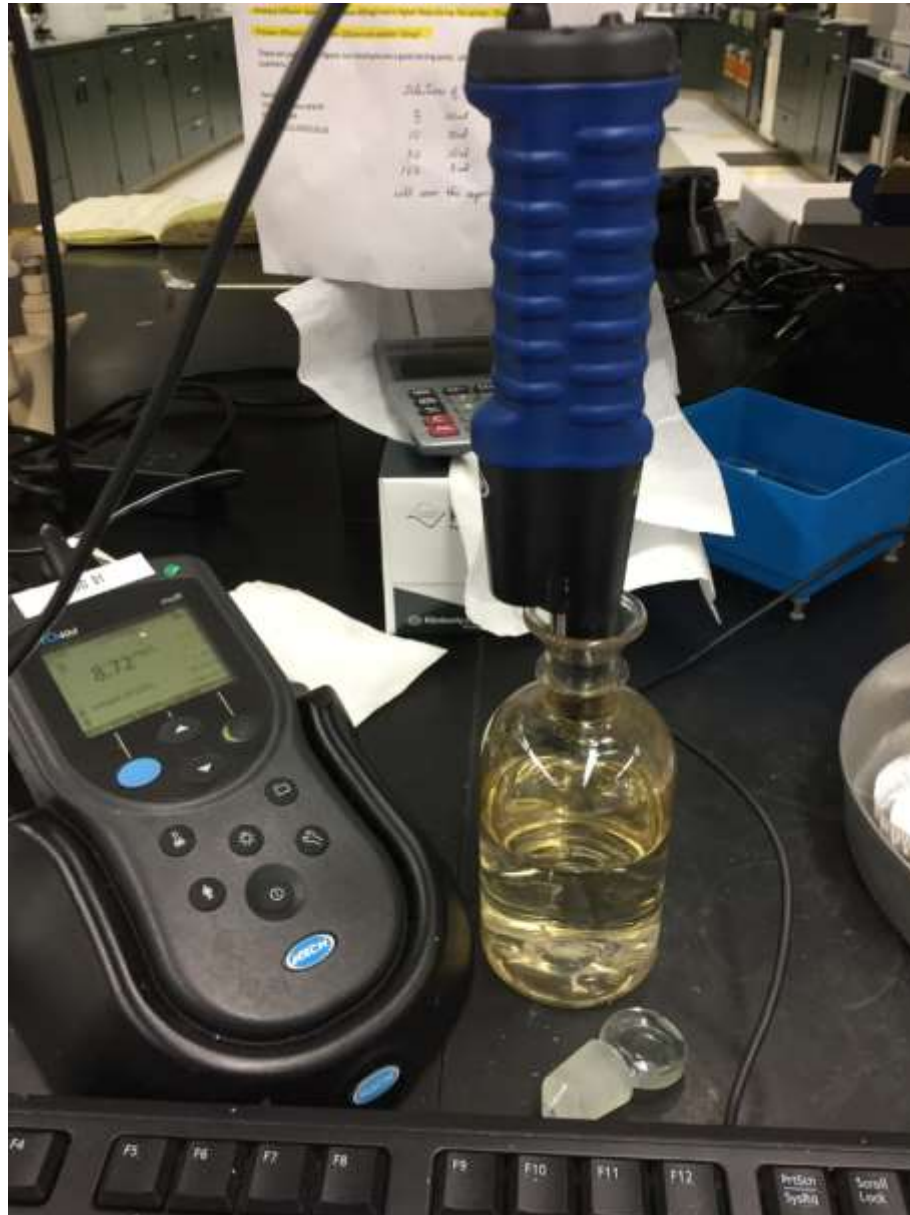
YSI-Polarographic Probe



D.O. Meter and Probe

- **YSI type:** change membrane/cap and filling solution every two to four weeks or when scratched, bubbles under membrane or leaking
- Check for Black AgS on electrode. Soak with 10% Ammonia and buff lightly.
- Store in BOD bottle with a little H₂O.
- Keep membrane dry (no water drops) when calibrating.

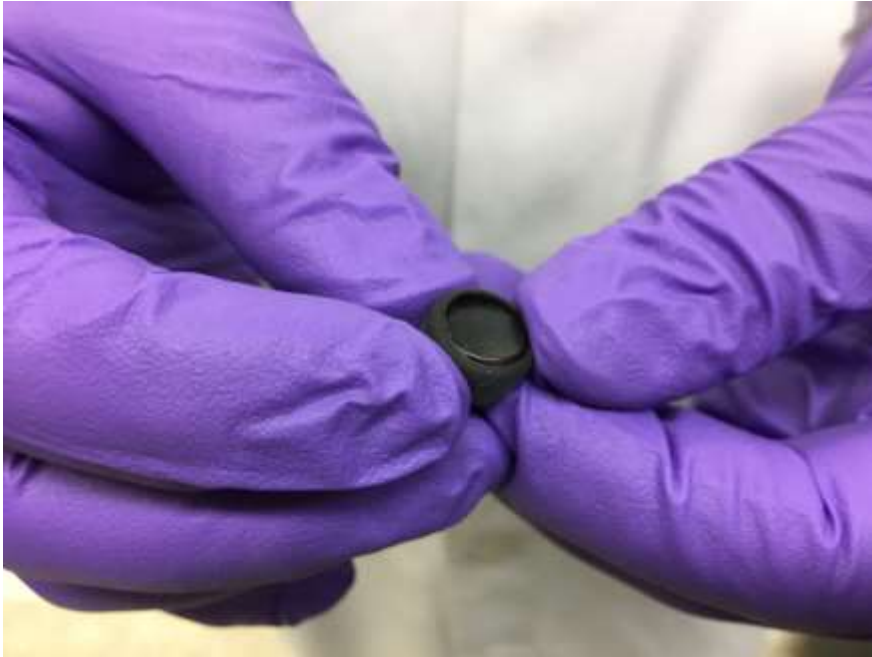
Luminescent (LDO) Probes



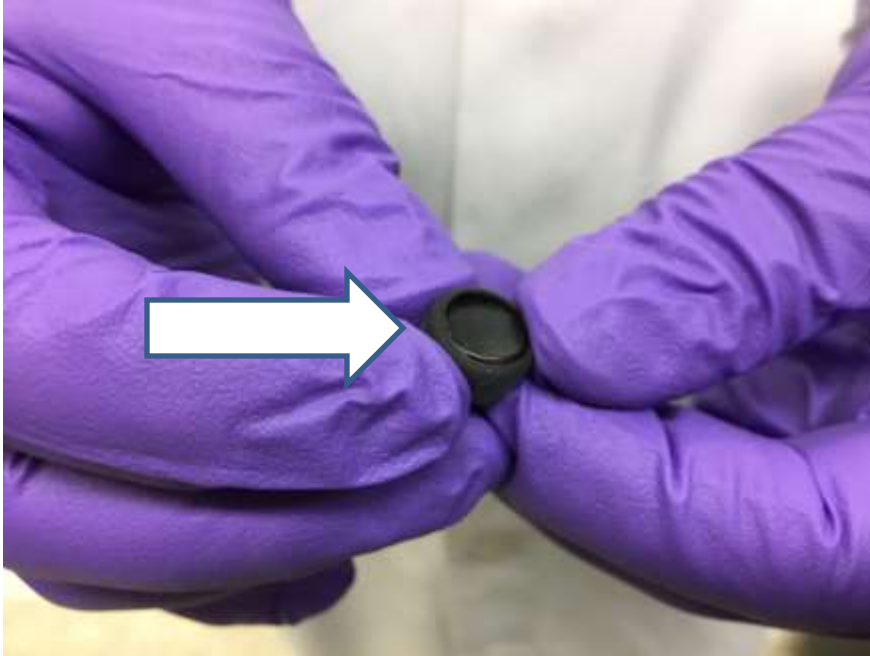
D.O. Meter and Probe

- **LED type:** change cap annually (6-12 mos)
- Store in BOD bottle with a little H₂O.
 - Check for leaks periodically
 - Replace stirrer as needed
- Keep membrane dry (no water drops).

LED Probe Maintenance



LED Probe Maintenance



Inside LED Cap



Calibration and Checks

- Air calibration.
 - wipe membrane of water drops
 - Allow enough time to stabilize
 - Check against Saturation Tables
 - DO sat is 9.09 mg/l at 20°C and 760 mm Hg
- Check Bottle.
 - Two bottles of aerated dilution water
 - Check DO in Bottle #1 after calibration complete.
 - Run Winkler on Bottle #2. (Agree within 0.5 mg/l?)
 - Check Bottle#1 again at end of readings (within 0.2?)

Seed

- Settled Raw Influent
- Primary Effluent
- Avoid polymer residues, high ammonia
- Aim for seed correction of 0.6 to 1.0 mg/l

- Add to dechlorinated effluent
- Some labs add seed to all industrial discharges

Quality Controls

- **GGA:**

150 mg Dextrose, anhydrous,

+

150 mg L-(+)-Glutamic Acid **per liter**

Make your own: pre-measure 5 mls into vials and freeze

or

Purchase already made and refrigerate

Nitrification Inhibitors

- TCMP:

2-chloro-(TriChloroMethyl)Pyridine, 2.2%
(powder)

Available from Hach, North Central Laboratories

- N-Allylthiourea

(liquid in its pure state)

(contained in Polyseed NX)

Available from Hach, other lab suppliers

Set'm Up

	Volume (ml)	Seed vol.(ml)
• Blank	300	0
• Seed	4 to 8	0
• Seed + TCMP	4 to 8	0
• GGA	5	1 or 2
• Influent 1	6	0
• Influent 2	4	0
• Influent 3	2	0
• Effluent 1+TCMP	250	1 or 2
• Effluent 2+TCMP	200	1 or 2
• Effluent 3+TCMP	150	1 or 2



Dilution Water









Calculations

- No seed:

$$\frac{(DO_0 - DO_5) * 300}{(\text{ml sample})}$$

- Seeded:

$$\frac{[(DO_0 - DO_5) - (\text{seed corr})] * 300}{(\text{ml sample})}$$

Valid dilution has DO depletion of at least 2.0 mg/l

AND

has at least 1.0 mg/l DO left

Calculations

- See Sample Benchsheet

SAMPLE	BOTTLE #	SAMPLE ML	ML SEED	D.O. in mg/l	D.O. out mg/l	Depletion mg/l	Seed corr'n	B.O.D. mg/l	B.O.D. mg/l
BLANK	100	300	-	8.35	8.15	0.2			
Seed	200	6	-	8.25	3.45	4.8	0.80/ml		
Seed+TCMP	201	6	-	8.3	4.7	3.6	0.60/ml		
GGA	300	5	2	8.4	3.2	5.2	1.6		
Influent	401	10	-	8.3	0.99	DO5<1	-		
	402	6	-	8.25	3.25	5	-		
	403	3	-	8.19	6.4	Depl<2.0	-		
Effluent	501	250	1	10.1	3	7.01	0.8		
	502	200	1	10	4	6	0.8		
	503	150	1	9.9	5	4.9	0.8		
Effluent + TCMP	601	250	1	9.5	5.5	4	0.6		
	602	200	1	9.5	6	3.5	0.6		
	603	150	1	9.5	6.5	3	0.6		
Industry A	701	1.5	1	8.4	2.6	5.8	0.8		
	702	1	1	8.4	4.6	3.8	0.8		
COD=2000	703	0.5	1	8.4	6.4	2	0.8		
Industry Z	801	0.8	1	8.4	4.4	4	0.8		
	802	0.5	1	8.4	4.6	3.8	0.8		
COD=4000	803	0.3	1	8.4	4.8	3.6	0.8		
DO check in, finish				DO check out finish					

SAMPLE	BOTTLE #	SAMPLE ML	ML SEED	D.O. in mg/l	D.O. out mg/l	Depletion mg/l	Seed corr'n	B.O.D. mg/l	B.O.D. mg/l
BLANK	100	300	-	8.35	8.15	0.2			
Seed	200	6	-	8.25	3.45	4.8	0.80/ml		
Seed+TCMP	201	6	-	8.3	4.7	3.6	0.60/ml		
GGA	300	5	2	8.4	3.2	5.2	1.6	216	216
Influent	401	10	-	8.3	0.99	DO5<1	-		
	402	6	-	8.25	3.25	5	-	250	
	403	3	-	8.19	6.4	Depl<2.0	-		
Effluent	501	250	1	10.1	3	7.01	0.8	7.45	7.82
	502	200	1	10	4	6	0.8	7.8	
	503	150	1	9.9	5	4.9	0.8	8.2	
Effluent + TCMP	601	250	1	9.5	5.5	4	0.6	4.08	4.41
	602	200	1	9.5	6	3.5	0.6	4.35	
	603	150	1	9.5	6.5	3	0.6	4.8	
Industry A COD=2000	701	1.5	1	8.4	2.6	5.8	0.8	1000	873 ???
	702	1	1	8.4	4.6	3.8	0.8	900	
	703	0.5	1	8.4	6.4	2	0.8	720	
Industry Z COD=4000	801	0.8	1	8.4	4.4	4	0.8	1200	1933 ???
	802	0.5	1	8.4	4.6	3.8	0.8	1800	
	803	0.3	1	8.4	4.8	3.6	0.8	2800	
DO check in, finish				DO check out finish					

TNI Documentation Requirements

- Method ID/SOP ID
- Date/time/Analyst Initials
- Equipment ID
- Before/After Calibration Verification
- Reagent Lot ID's
- Maintenance (Meter Logbook)

Troubleshooting

- Bad Blanks: depletion > 0.20 mg/l
 - bad dilution water
 - dirty bottles or other labware
 - calibration problems (meter/probe)

Other questions?

- how does reporting limit depend on dilution?
- when do you average results?

Troubleshooting

- Bad GGA controls: < 167.5 or > 228.5 mg/l

GGA-BOD: 198 ± 30.5 mg/l. source: Std Methods

[GGA-CBOD: 164 ± 26 mg/l. source: TNI PT]

- Bad Seed: source? age?
- Forgot seed, forgot GGA (Steve Martin defense)
- Bad Dilution Water, meter, probe, etc.

My Thanks To:

- James Loftis & Steve Hall, City of Salem
- Chris Dennis, City of Portland, BES
- John Hoppner, City of Corvallis
- Perry Brake



Resources:

Perry Brake:

- <http://www.perrybrake.com/BODSolutions.html>
- <http://slideplayer.com/slide/4769603/>

Oregon DEQ:

<http://www.deq.state.or.us/lab/techrpts/docs/09lab0071gag.pdf>

If all else fails, then try:

- Keith.chapman6023@gmail.com

Standard Methods 21st Edition BOD Changes

By Perry Brake

While the 21st Edition of Standard Methods has not yet been approved for Clean Water Act monitoring, it is worthwhile to know what some of the changes are in the BOD test (SM 5210B). Some of these changes may be allowed or even encouraged by regulators and lab accreditors. This article points out some of the more significant changes in the order they appear in the method. Comments are those of the author.

- **-Paragraph 3g(2)** adds allylthiourea (ATU) as an approved nitrification inhibitor.
- **-Paragraph 4b(4) and 5b** allow sample temperature prior to dilution to be $20 \pm 3^\circ \text{C}$ rather than $\pm 1^\circ \text{C}$ (but the incubation temperature remains $20 \pm 1^\circ \text{C}$.)
- **-Paragraph 4b(5)** adds pretreatment procedures for samples containing hydrogen peroxide

22nd Edition BOD Changes - by Perry Brake

The only substantial change to the BOD test in the most recent (spring 2012) edition of *Standard Methods* 5210B is that the QC tests applying to the procedure are summarized in a table (5020:1). That table calls for three tests: 1) a *lab fortified blank* which is the 50:50 glucose/glutamic acid solution required by previous editions of 5210B; 2) a *lab fortified matrix*, often called a matrix spike; and 3) a *lab fortified matrix duplicate*. The matrix spike and duplicate are new



The BOD Song!

- Oh B-O-D, Oh B-O-D
- You are so biochemical
- A little sample and then some seed
- So plain yet still empirical
- To set you up is such a breeze
- Just five short days at 20 degrees
- Oh B-O-D, Oh B-O-D
- Pray the blanks don't deplete on me.

- First get the bottles all lined up
- Then calibrate the meter
- Aerate the water; prep the seed
- And don't forget the Winkler
- Pipet your samples in “just so”
- Make sure they have enough D.O.
- Now load the samples on a cart
- Incubate them five days in the dark.

- If Wednesday morn you put them in
- Next Monday all your woes begin
- All the dilutions that you tried
- Were either too low or else too high
- All your controls are out of range
- The calculations just too strange
- Oh B-O-D you are so great
- The test that we all love to hate

Thank You

