



Chemical Oxygen Demand V.1

William Brigman¹

¹USDA

Version 1

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Works for me

This protocol is published without a DOI.

PDI Test

Josh Birlingmair
USDA-ARS

SUBMIT TO PLOS ONE

ABSTRACT

Version 2.0 (COD station 1st lab and analytical trailer)

Developed by: William Brigman 9-25-2014; Filename: COD protocol v2.0.doc

Certified by: Matias Vanotti 8-4-2011

Experts: William Brigman (ext. 302)

Revised: 1/18/2017

PROTOCOL CITATION

William Brigman 2021. Chemical Oxygen Demand. **protocols.io**

<https://protocols.io/view/chemical-oxygen-demand-bs78nhrw>



KEYWORDS

Chemical, Oxygen, Demand, COD

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GUIDELINES

QA/QC

1. Make sure you check pipette calibration before using. Since we are using so little sample the smallest error could be critical. In order to check calibration, see pipette protocol.
2. QA/QC standard checks. Use at least 1 QA/QC check sample besides your 1,000 mg/L standard. You can use a 300 mg/L standard or a 800 mg/L standard. Prepare just like a normal standard.
3. Always use (2) 1,000 mg/L standards. These values should be very similar and should be in the range of (950-1050 mg/L).

Instrument Log Book

A log sheet will be kept next to the instrument documenting instrument use and QA/QC:

Date, technician initials, # samples (high & low), standard values obtained

Waste Disposal

COD vials should be disposed of in the hazardous waste shed that is located next to the Chemical waste shed.

The used vials can be kept in your lab until you have a significant amount to put in the COD drum, usually a box full is significant. Make sure your box is clearly labeled "USED COD VIALS".

The drum, located in the hazardous waste shed (Novak has the key), should have nothing in it but used COD vials; no vermiculite or any other packing material is needed in the barrel.

Put the used COD vials in the barrel with caution (don't just throw them in there) because you don't want to break one. If one gets broken, contact Jeff Novak, Don Watts, or William Brigman immediately.

After adding the used COD vials, there is a on the p:drive ([..\..\..\..\Safety\EZ COD drum 7212344.xlsx](#)) that needs to be filled out. Put the date, # vials disposed, and your initials. Make sure the drum # and the drum # on the sheet match up.

When the barrel is getting close to full please let Jeff Novak-Chemical Hygiene Officer (ext. 110) know so that he can schedule a hazardous waste pick-up.

We will have to schedule a pick up from Heritage Environmental Services to pick up the barrel. All cost are included in the purchase of the barrel from Hach Company. Whenever the barrel was purchased from Hach, paper work should have been sent with all the information needed to schedule the pick-up.

After the company has picked up the drum, give all paper work to either Jeff Novak or Don Watts to file. The paper work will be kept in the safety room file cabinet in building 1.

MATERIALS TEXT

1. 1,500 COD digestion vials (20-1,500mg/L), 150 per pack, part # (2125915) Hach Company
2. 15,000 COD digestion vials (200-15,000 mg/L), 150 per pack, part # (2415915) Hach Company
3. 1,000mg/L (± 50 mg/L) COD standard, 200mL bottle, part # (2253929) Hach Company
4. 300mg/L (± 15 mg/L) COD standard, 200mL bottle, part # (1218629) Hach Company
5. 800mg/L (± 40 mg/L) COD standard, 200mL bottle, part# (2672629) Hach Company
6. EZ COD Recycling Service, 20 gallon barrel, part # (2895420) Hach Company – Heritage Environmental Services will pick up. (\$584)

SAFETY WARNINGS

**Wear safety clothing, COD vials contain mercury and dichromate!
Goggles, lab coat, and gloves are required!!!**

DISCLAIMER:

Method's citation in a publication: "Wastewater analyses were performed according to Standard Methods for the Examination of Water and Wastewater (APHA, AWWA & WEF, 1998). Chemical oxygen demand (COD) was measured with the closed reflux, colorimetric method (Standard Method 5220 D)".

Reference List: APHA, AWWA, WEF. 1998. Standard Methods for the Examination of Water and Wastewater. 20th Edition. Washington, D.C., USA.

- 1 Fill out the COD data sheet, record the sample identification number and COD vial number.
- 2 For samples with moderate to low COD (such as constructed wetlands) use the 1,500 mg/L COD vials. For samples with high COD (such as lagoons) use the 15,000 mg/L COD vials.
- 3 Turn on COD block heater digester (DRB 200-HACH) (toggle switch on back) and allow it to reach set temperature (~20 minutes).
- 4 If using **1,500 mg/L COD vials**, use **2mL** of sample (measured by pipette). If using **15,000 mg/L COD vials**, use **0.2mL** of sample (measured by pipette).
- 5 Use the COD standard (1000 mg/L) located in the refrigerator. Pipette 2mL of standard into a 1,500 mg/L COD vial and 0.2mL into 15,000 mg/L COD vial.
- 6 For a blank, pipette the appropriate amount of deionized water into COD vial.
- 7 Remember to shake your samples and standards up before sampling them so that we get a uniform sample.
- 8 For a QA/QC check sample, get the COD standard (300 mg/L and/or 800 mg/L) from the refrigerator to use as a check.
- 9 After adding sample, put cap back on tight and shake COD vials to mix the sample.
- 10 You should always have at least **1 blank, 2 standards, and 1 QC check**.
- 11 Place vials in COD block heater. Set timer on COD block heater for 2 hours.
- 12 After 2 hours, let the vials cool in the block heater for 10 minutes and then remove the vials from the block heater, place in a dark area, and allow the vials to cool. Caution, these will be hot!!!! Allow to cool for 10-15 minutes.
- 13 After COD vials have cooled, wipe off COD vial with a kimwipe to remove any fingerprints and smudges:

- 14 Place the blank COD vial into the COD reader, press "zero" on the colorimeter keyboard. This will set the blank. The blank should read 0 mg/L.
- 15 Next, place the vial containing the 1000 mg/L standard into the colorimeter. Press read and record the value.
- 16 Measure COD values for all standards, samples, and QAQC standards. Follow the same procedure in step 15 for each sample. Record colorimeter data on the COD data sheet. The output will be in mg/L.
- 17 After completion of measurements, turn off COD block heater and COD colorimeter.

DR-2700 Colorimeter

- 18 Press button on the back of colorimeter to turn on and allow 30min to warm-up.
- 19 The colorimeter will go thru a self check once when its first turned on.
- 20 After 30min, click on "Favorite Program" button and select the correct program. The program we are using is "435 COD HR 1500 mg/L."
- 21 Once selected hit start.
- 22 Next hit "options" button and change the current units to mg/L (hit 3rd button down from the top unit the correct units are displayed on the big screen).
- 23 Insert the blank vial and press "zero", wait for beep and reading to be displayed.
- 24 Insert the standard vial and press "read", record value and continue into all samples have been analyzed.
- 25 Hold button on back of colorimeter to turn off.
- 26 See **WASTE DISPOSAL** section under Guidelines.