

Oxygen, Dissolved

DOC316.53.01179

Azide Modification of Winkler Method

1 to more than 10 mg/L

Method 8215 and 8332

Digital Titrator

Scope and application: For water, wastewater and seawater.



Test preparation

Before starting

As an alternative to stirring by hand, use the TitraStir Titration Stand to hold the Digital Titrator and stir the sample.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

Description	Quantity
Digital Titrator	1
Delivery tube for Digital Titrator, J-hook tip	1
Clippers for plastic pillows	1
For Method 8215, 300-mL BOD bottle	
Dissolved Oxygen Reagent Set	1
Sodium Thiosulfate Titration Cartridge, 2.00 N	varies
Bottle, with stopper, BOD, 300-mL	1
Cylinder, graduated, 250-mL	1
Flask, Erlenmeyer, 250-mL	1
For Method 8332, 60-mL BOD bottle	
Dissolved Oxygen 1 Reagent Powder Pillows	1
Dissolved Oxygen 2 Reagent Powder Pillows	1
Dissolved Oxygen 3 Reagent Powder Pillows	1
Sodium Thiosulfate Titration Cartridge, 0.2000 N	1
Bottle, with stopper, BOD, 60-mL	1
Flask, Erlenmeyer, 125-mL	1
Beaker, polypropylene, 50-mL, low form	1

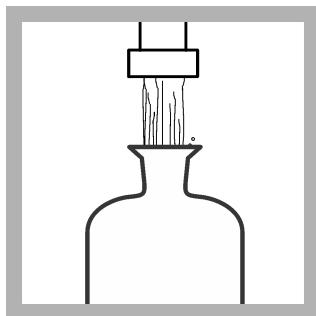
Refer to [Consumables and replacement items](#) on page 6 for order information.

Sample collection

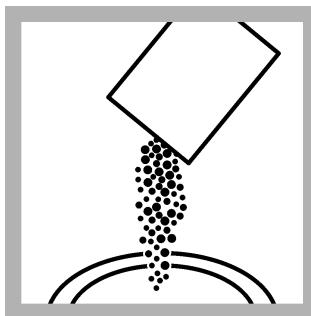
Good sample collection and handling techniques are important to get correct results. The dissolved oxygen content of the sample can change with depth, turbulence, temperature, sludge deposits, light, microbial action, mixing, travel time and other factors. A single dissolved oxygen test frequently is not an accurate reflection of the overall condition of a body of water. Several samples taken at different times, locations and depths are recommended for most reliable results.

- Collect samples in clean BOD Bottles.
- If prompt analysis is not possible, do steps 1 through 4 of the procedure and keep the samples protected from light at 10 to 20 °C (50 to 68 °F).
- Pour a small quantity of water into the flared lip area of the stopper to seal the bottle.
- Use a BOD bottle cap on the flared lip.
- Keep samples for a maximum of 8 hours. For analysis start with step 5.

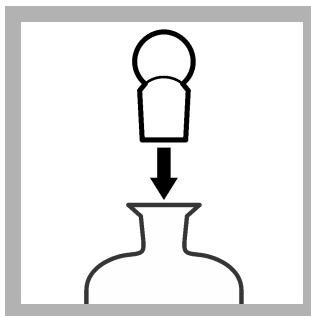
Method 8215, 300-mL BOD bottle



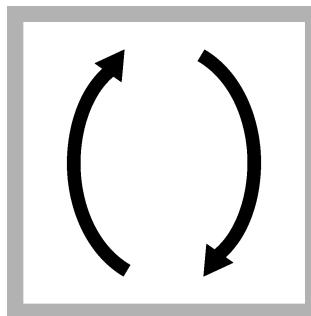
1. Collect a water sample in a clean 300-mL BOD bottle. Let the sample overflow the bottle for 2 or 3 minutes to make sure that a representative sample is collected.



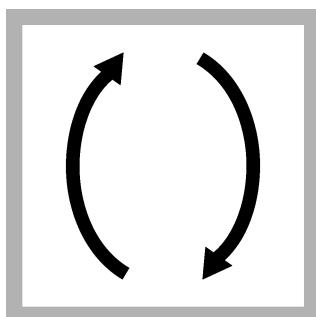
2. Add the contents of one Manganese Sulfate Powder Pillow and one Alkaline Iodide-Azide Reagent Powder Pillow to the sample.



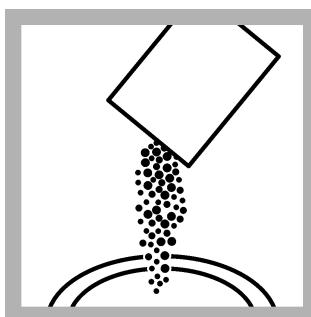
3. Immediately put the stopper in the bottle. Make sure that no air is inside the bottle.



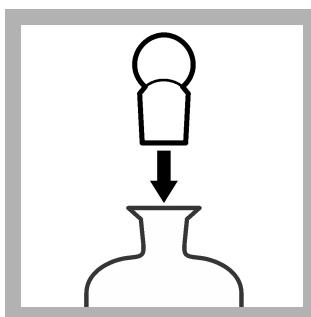
4. Invert the bottle at least 5 times to mix. A flocculent precipitate forms. The floc is orange/brown if oxygen is in the sample or white if there is no oxygen. The floc forms slowly in salt water (approximately 5 minutes more are necessary). When the floc formation is complete, proceed to next step.



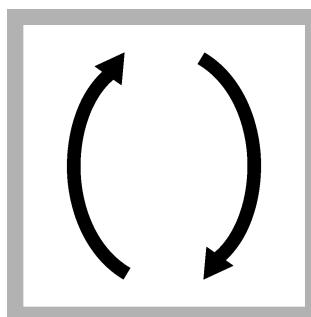
5. Again, invert the bottle at least 5 times to mix. Wait until the top half of the solution is clear and the floc collects at the bottom to make sure that the reaction of the sample and reagents is complete.



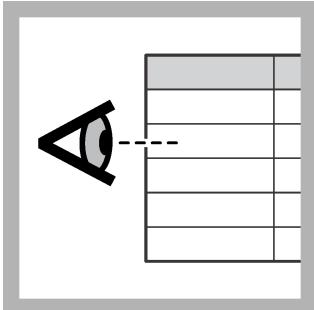
6. Remove the stopper and add the contents of one Sulfamic Acid Powder Pillow to the sample.



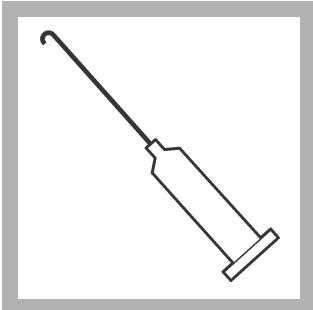
7. Immediately put the stopper in the bottle. Make sure that no air is inside the bottle.



8. Invert the bottle at least 5 times to mix. The floc dissolves and a yellow color develops if oxygen is in the sample.



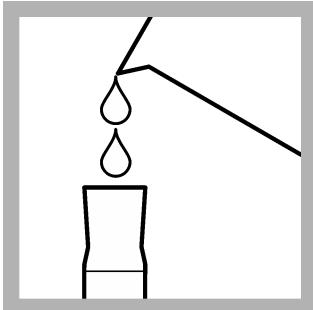
9. Select a sample volume and titration cartridge from [Table 1](#) on page 5.



10. Insert a clean delivery tube into the digital titration cartridge. Attach the cartridge to the Digital Titrator.



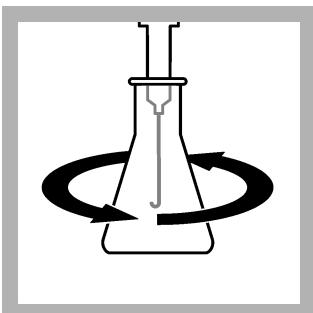
11. Hold the Digital Titrator vertically with the cartridge tip up. Turn the delivery knob to eject air and a few drops of titrant. Reset the counter to zero and clean the tip.



12. Use a graduated cylinder to measure the sample volume from [Table 1](#) on page 5.



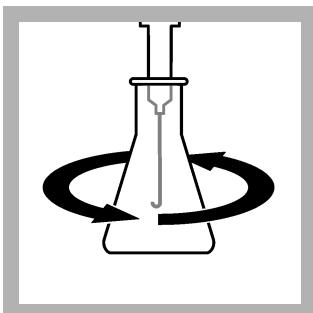
13. Pour the sample into a clean, 250-mL Erlenmeyer flask.



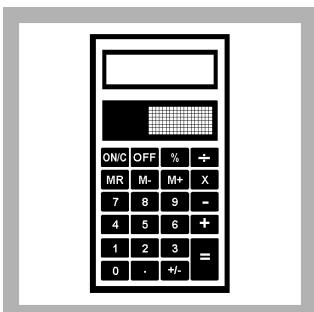
14. Put the delivery tube point fully into the solution and swirl the flask. Turn the knob on the Digital Titrator to add titrant to the solution. Continue to swirl the flask and add titrant until the color changes yellow to a pale yellow.



15. Add 2 mL of Starch Indicator Solution. A dark blue color develops.

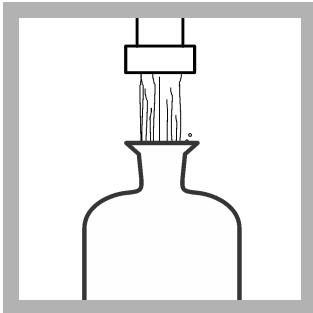


16. Put the delivery tube point fully into the solution and swirl the flask. Turn the knob on the Digital Titrator to add titrant to the solution. Continue to swirl the flask and add titrant until the color changes from a dark blue to a colorless end point. Record the number of digits on the counter.



17. Use the multiplier in [Table 1](#) on page 5 to calculate the concentration. Digits used \times digit multiplier = mg/L Dissolved Oxygen

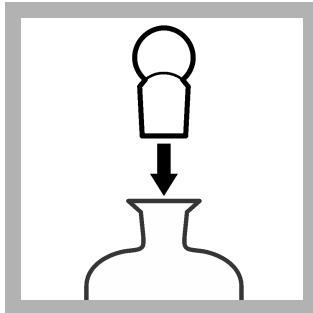
Method 8332, 60-mL BOD bottle



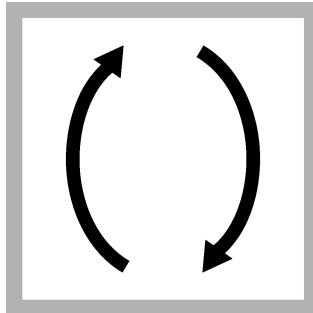
1. Collect a water sample in a clean 60-mL BOD bottle. Let the sample overflow the bottle for 2 or 3 minutes to make sure that a representative sample is collected.



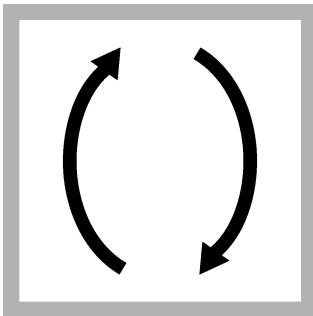
2. Add the contents of one Dissolved Oxygen 1 Powder Pillow and one Dissolved Oxygen 2 Powder Pillow to the sample.



3. Immediately put the stopper in the bottle. Make sure that no air is inside the bottle.



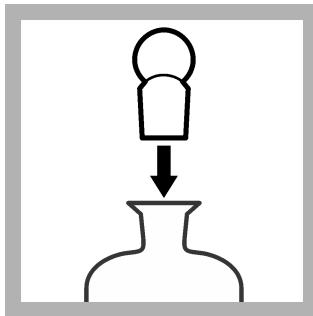
4. Invert the bottle at least 5 times to mix. A flocculent precipitate forms. The floc is orange/brown if oxygen is in the sample or white if there is no oxygen. The floc forms slowly in salt water (approximately 5 minutes more are necessary). When the floc formation is complete, proceed to next step.



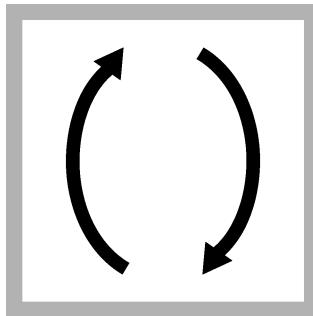
5. Again, invert the bottle at least 5 times to mix. Wait until the top half of the solution is clear and the floc collects at the bottom to make sure that the reaction of the sample and reagents is complete.



6. Remove the stopper and add the contents of one Dissolved Oxygen 3 Powder Pillow to the sample.



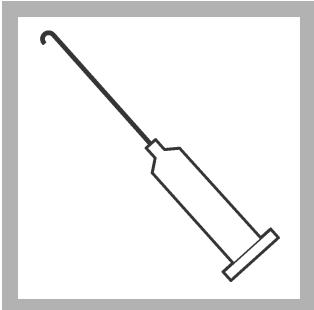
7. Immediately put the stopper to the bottle. Make sure that no air is inside the bottle.



8. Invert the bottle at least 5 times to mix. The floc dissolves and a yellow color develops if oxygen is in the sample.



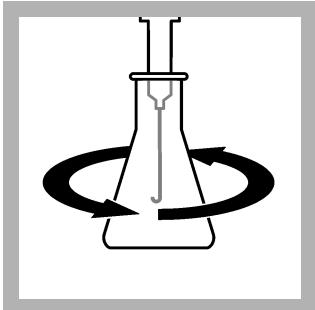
9. Accurately measure 20 mL of the prepared sample and transfer it to a 50-mL Erlenmeyer flask.



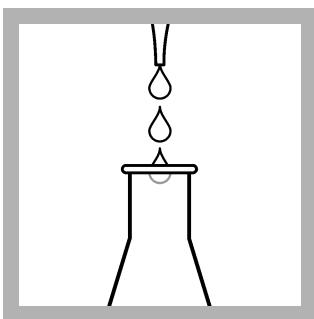
10. Insert a clean delivery tube into a 0.200 N Sodium Thiosulfate Titration Cartridge. Attach the cartridge to the Digital Titrator.



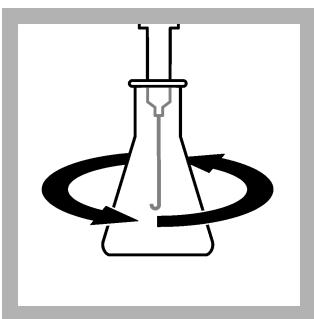
11. Hold the Digital Titrator vertically with the cartridge tip up. Turn the delivery knob to eject air and a few drops of titrant. Reset the counter to zero and clean the tip.



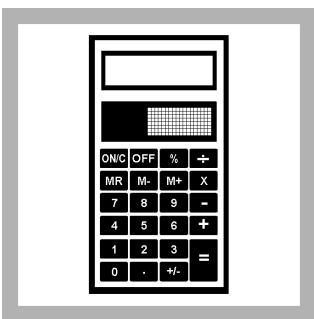
12. Put the delivery tube point fully into the solution and swirl the flask. Turn the knob on the Digital Titrator to add titrant to the solution. Continue to swirl the flask and add titrant until the color changes yellow to a pale yellow.



13. Add 2 mL of Starch Indicator Solution. A dark blue color develops.



14. Put the delivery tube point fully into the solution and swirl the flask. Turn the knob on the Digital Titrator to add titrant to the solution. Continue to swirl the flask and add titrant until the color changes from a dark blue to a colorless end point. Record the number of digits on the counter.



15. Calculate the concentration: digits \times 0.1 = mg/L Dissolved Oxygen.

Sample volume and digit multipliers

Select a range in [Table 1](#), then read across to find the applicable information for this test. Use the digit multiplier to calculate the concentration in the test procedure.

Table 1 Sample volumes and digit multipliers

Range (mg/L DO)	Sample volume (mL)	Titration cartridge	Digit multiplier
1–5	200	0.200	0.01
2–10	100	0.200	0.02
>10	200	2.000	0.10

Interferences

Nitrite interference is removed by the azide in the reagents. Other reducing or oxidizing substances may interfere. If these are in the sample, use an alternative method, such as the High Range Dissolved Oxygen Method (colorimetric, Method 8166) or a dissolved oxygen electrode.

Accuracy check

Standard method solution

Determine the strength of the Sodium Thiosulfate Solution with an Iodate-Iodide Standard Solution, 10 mg/L as dissolved oxygen.

Method 8215, 300-mL BOD bottle

1. Start the analysis at step 6. Add the Sulfamic Acid Powder Pillow to a 200-mL volume of Iodate-Iodide Standard Solution.
 - a. Use a 100-mL sample volume with the 0.200 N Sodium Thiosulfate Titration Cartridge. 500 digits are necessary for this titration. If more than 525 digits are necessary to get to the end point, replace the Sodium Thiosulfate Cartridge.
 - b. Use a 200-mL sample volume with the 2.00 N Sodium Thiosulfate Titration Cartridge. 100 digits are necessary for this titration. If more than 105 digits are necessary to get to the end point, replace the Sodium Thiosulfate Cartridge.

Method 8332, 60-mL BOD bottle

1. Start the analysis at step 6. Add the Dissolved Oxygen 3 Powder Pillow to a 60-mL volume of Iodate-Iodide Standard Solution.
2. Use a 20-mL sample volume with the 2.00 N Sodium Thiosulfate Titration Cartridge. 100 digits are necessary for this titration. If more than 105 digits are necessary to get to the end point, replace the Sodium Thiosulfate Cartridge.

Summary of method

Samples are treated with manganous sulfate and alkaline iodide-azide reagent to form an orange/brown precipitate. After acidification of the sample, this floc reacts with iodide to produce free iodine as triiodide in proportion to the oxygen concentration. The iodine is titrated with sodium thiosulfate to a colorless end point.

Consumables and replacement items

Required reagents for 300-mL BOD bottle

Description	Quantity/test	Unit	Item no.
Dissolved Oxygen Reagent Set (approximately 50 tests)	1	—	2272200
Alkaline Iodide-Azide Powder Pillows		50/pkg	107266
Manganous Sulfate Powder Pillows		50/pkg	107166
Sodium Thiosulfate Titration Cartridge, 0.2000 N		each	2267501
Starch Indicator Solution		100 mL MDB ¹	34932
Sulfamic Acid Powder Pillows		50/pkg	2076266
Sodium Thiosulfate Titration Cartridge, 2.00 N	varies	each	1440101

Required apparatus for 300-mL BOD bottle

Description	Quantity/test	Unit	Item no.
Bottle, with stopper, BOD, 300-mL	1	each	62100
Clippers for plastic pillows	1	each	96800
Cylinder, graduated, 250-mL	1	each	50846
Digital Titrator	1	each	1690001
Flask, Erlenmeyer, 250-mL	1	each	50546
Delivery tube for Digital Titrator, J-hook tip	1	5/pkg	1720500

¹ MDB is Marked Dropper Bottle

Required reagents for 60-mL BOD bottle

Description	Quantity/test	Unit	Item no.
Dissolved Oxygen 1 Reagent Powder Pillows	1	100/pkg	98199
Dissolved Oxygen 2 Reagent Powder Pillows	1	100/pkg	98299
Dissolved Oxygen 3 Reagent Powder Pillows	1	25/pkg	98768
Sodium Thiosulfate Titration Cartridge, 0.2000 N	1	each	2267501

Required apparatus for 60-mL BOD bottle

Description	Quantity/test	Unit	Item no.
Bottle, with stopper, BOD, 60-mL	1	each	190902
Clippers for plastic pillows	1	each	96800
Digital Titrator	1	each	1690001
Flask, Erlenmeyer, 125-mL	2	each	50543
Beaker, polypropylene, 50-mL, low form	1	each	108041
Delivery tube for Digital Titrator, J-hook tip	1	5/pkg	1720500

Recommended standards and apparatus

Description	Unit	Item no.
Iodate-Iodide Standard Solution, 10-mg/L as dissolved oxygen	500 mL	40149
Cap, BOD Bottle Snap-over	6/pkg	241906
BOD Bottle, Serialized (#1–24)	24/pkg	2898700
TitraStir Titration Stand, 115 VAC	each	1940000
TitraStir Titration Stand, 230 VAC	each	1940010
Delivery tube for Digital Titrator, 90-degree bend for use with TitraStir Titration Stand	5/pkg	4157800



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