

# Oxygen Demand in Salt Water, Chemical

DOC316.53.01534

Method 10299

7 to 70 mg/L COD (LR)

TNTplus™ 815

**Scope and application:** For wastewater, sea water and surface water that contains chloride and process analysis with 1.0 to 20 g/L chloride. For lower chloride concentration, use TNTplus 820.



## Test preparation

### Instrument specific table

Table 1 shows all of the instruments that have the program for this test. The table also shows the adapter and light shield requirements for the applicable instruments that can use TNTplus vials.

To use the table, select an instrument, then read across to find the applicable information for this test.

**Table 1 Instrument-specific information for TNTplus vials**

Instrument	Adapters	Light shield
DR6000, DR5000	—	—
DR3900	—	LZV849
DR1900	9609900 or 9609800 (A)	—

### Before starting

DR3900: Install the light shield in Cell Compartment #2 before this test is started.

Review the safety information and the expiration date on the package.

The recommended temperature for samples and reagents is 15–25 °C (59–77 °F).

The recommended temperature for reagent storage is 15–25 °C (59–77 °F).

The reagent that is used in this test is corrosive and toxic. Use protection for eyes and skin and be prepared to flush any spills with running water.

Spilled reagent will affect test accuracy and is hazardous to skin and other materials. Be prepared to wash spills with running water.

The reagents that are used in this test contain mercury. Collect the reacted samples for safe disposal.

The vial gets very hot when the sample is added.

Digestion is required.

Identify the chloride concentration of the sample with TNTplus 879, 1–1000 mg/L Cl<sup>-</sup> or chloride Quantab® test strips, 300–6000 mg/L (2751340). The photometer shows COD values for the three different chloride ranges that follow:

- 1 to less than 5 g/L chloride in sample—mg/L COD 1 to 5 g/L Cl<sup>-</sup>
- 5 to 10 g/L chloride in sample—mg/L COD 5 to 10 g/L Cl<sup>-</sup>
- More than 10 to 20 g/L chloride in sample—mg/L COD 10 to 20 g/L Cl<sup>-</sup>

Analyze reagent blanks as a quality check for accurate results at low concentrations or when the reagents were in storage for long periods of time. For the best results, analyze one blank (at minimum) per lot of reagents. Refer to [Blanks for colorimetric determination](#) on page 3. Run all tests (the samples and the blank) with the same lot of vials. The lot number is on the container label.

Keep unused (light sensitive) vials in a closed box.

Use the DRB reactor with 13-mm wells for the digestion. If the reactor has 16-mm wells, put adapter sleeves into the wells.

DR1900: Go to All Programs>LCK or TNTplus Methods>Options to select the TNTplus number for the test. Other instruments automatically select the method from the barcode on the vial.

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

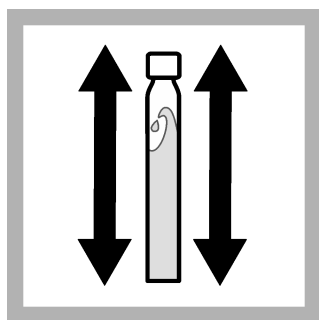
Description	Quantity
COD TNTplus™ Reagent Set, LR in salt water	1
DRB200 reactor with 13-mm wells	1
Pipet, adjustable volume, 1.0–5.0 mL	1
Pipet tips, for 1.0–5.0 mL pipet	1
Test tube rack	1

Refer to [Consumables and replacement items](#) on page 5 for order information.

## Sample collection and storage

- Collect samples in clean glass bottles. Use plastic bottles only if they are known to be free of organic contamination.
- Test biologically active samples as soon as possible.
- Homogenize samples that contain solids to get a representative sample.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated sulfuric acid (approximately 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep the preserved samples at 2–6 °C (36–43 °F) for a maximum of 28 days.
- Correct the test result for the dilution caused by the volume additions.

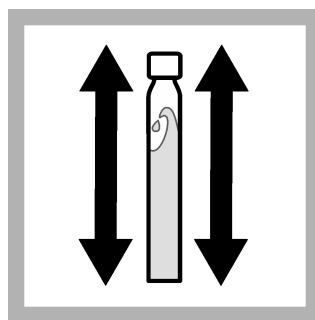
## Test procedure



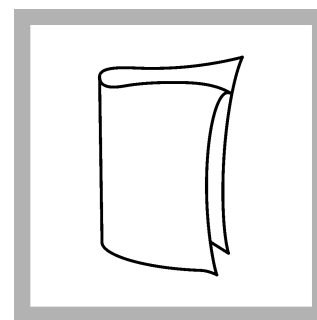
1. Shake the vial to fully mix the sediment and liquid.



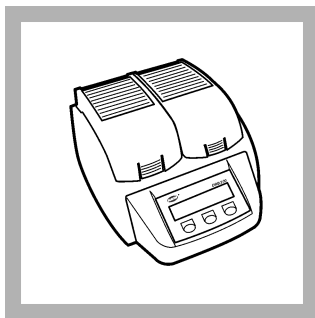
2. Use a pipet to add 1.8 mL of sample to the test vial.



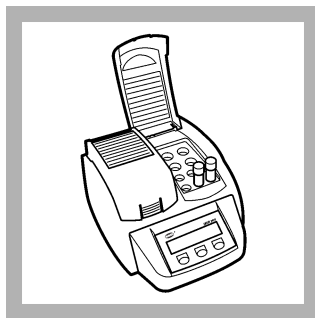
3. Immediately put the cap on the vial. Hold the vial by the cap, over a sink. Shake the vial quickly. **The vial gets very hot during mixing.**



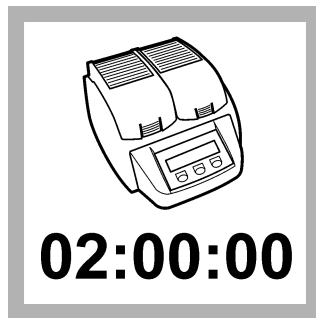
4. Clean the vial.



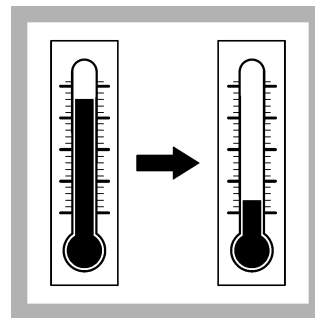
5. Set the DRB200 reactor power to on. Set the temperature to 150 °C (302 °F).



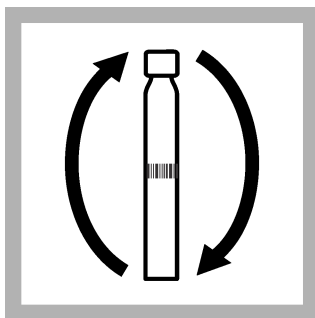
6. Insert the vial in the preheated DRB200 reactor. Close the lid.



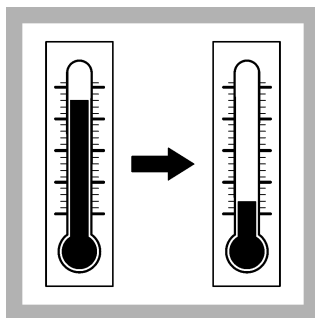
7. Keep the vial in the reactor for 2 hours.



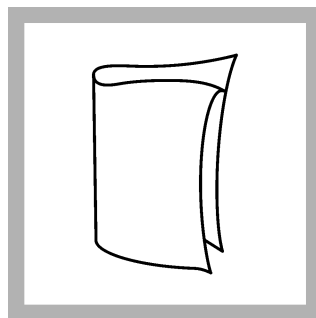
8. When the timer expires, set the reactor power to off. Let the temperature decrease for about 20 minutes to 120 °C (248 °F) or less.



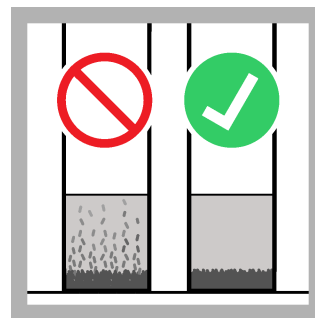
9. Hold the vial by the cap and invert gently several times while the vial is still hot.



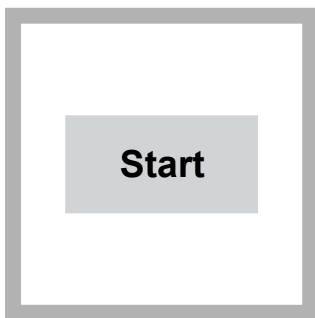
10. Put the vial in a test tube rack. Let the temperature of the vial decrease to room temperature.



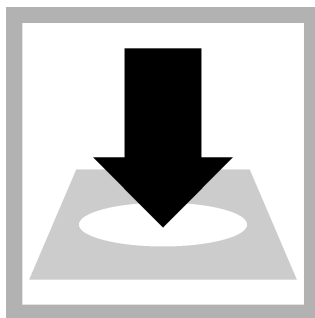
11. Clean the vial.



12. Wait for the sediment to fully settled to the bottom of the vial. If necessary, put the vial in a centrifuge at 4000 rotations/minute.



13. DR1900 only: Select program 815. Refer to [Before starting](#) on page 1.



14. Insert the vial into the cell holder. DR1900 only: Push **READ**. Results show in mg/L COD.

## Blanks for colorimetric determination

Analyze reagent blanks as a quality check for accurate results at low concentrations or when the reagents were in storage for long periods of time. For the best results, analyze one blank (at minimum) per lot of reagents. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option. Use the blank again for other measurements with the same lot of vials. For storage, keep the blanks in a dark location. Monitor the decomposition of the blanks by periodically measuring its concentration. Measure the reagent blank value when a new lot of reagent is used.

To subtract the value of the blanks from a series of measurements:

1. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Clean the vial, then put it in the cell holder. Close the lid.
2. Set the reagent blank function to on. The measured value of the reagent blank is shown.

**Note:** As an alternative, record or enter the reagent blank value at a different time. Push the highlighted reagent blank box and use the keypad to enter the value.

## Interferences

Use this method for samples (or diluted samples) with chloride concentrations of 1.0 to 20 g/L. For lower chloride concentration, use TNTplus 820.

Fully mix the sediment and liquid before the sample is added or high-biased results can occur.

Wastewater can contain ingredients for which the oxidation potential of the reagent in the vial is not sufficient.

## Accuracy check

### Standard solution method—Mixed parameter standard

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- Wastewater Effluent Standard Solution, Mixed Parameter (contains 25-mg/L COD)
1. Use the test procedure to measure the concentration of the standard solution.
  2. Compare the expected result to the actual result.

**Note:** The factory calibration can be adjusted slightly with the standard calibration adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are small variations in the reagents or instruments.

### Standard solution method—COD standard

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- COD Standard Solution, 1000-mg/L COD
  - 100-mL volumetric flask, Class A
  - 5.0-mL volumetric pipet, Class A and pipet filler safety bulb
  - Deionized water
1. Prepare a 50-mg/L COD standard solution as follows:
    - a. Use a pipet to add 5.0 mL of a 1000-mg/L COD standard solution into the volumetric flask.
    - b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
  2. Use the test procedure to measure the concentration of the prepared standard solution.
  3. Compare the expected result to the actual result.

**Note:** The factory calibration can be adjusted slightly with the standard calibration adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are small variations in the reagents or instruments.

## Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% confidence interval)	Sensitivity Concentration change per 0.010 Abs change
barcode	50 mg/L COD	48–52 mg/L COD	—

## Summary of Method

The results in mg/L COD are defined as the milligrams of O<sub>2</sub> consumed per liter of sample under the conditions of this procedure. The sample is heated for 2 hours with sulfuric acid and a strong oxidizing agent, potassium dichromate. Oxidizable compounds react with the sulfuric acid and potassium dichromate solution. The COD reagent also contains silver and mercury sulfate ions. Silver is a catalyst, and mercury sulfate is used to complex chloride interferences. The reduction in the yellow color of Cr<sup>6+</sup> is measured. The measurement wavelength is 420 nm.

## Consumables and replacement items

### Required reagents

Description	Quantity/Test	Unit	Item no.
COD TNTplus™ Reagent Set, LR in salt water, 7 to 70 mg/L COD	1-2 vials	25/pkg	TNT815

### Required apparatus

Description	Quantity/test	Unit	Item no.
DRB200 Reactor, 115 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	1	each	DRB200-01
DRB200 Reactor, 230 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	1	each	DRB200-05
Pipet, adjustable volume, 1.0–5.0 mL	1	each	BBP065
Pipet tips, for 1.0–5.0 mL pipet	1	75/pkg	BBP068
Light shield, DR3900	1	each	LZV849

### Recommended standards

Description	Unit	Item no.
COD Standard Solution, 1000 mg/L	200 mL	2253929
Wastewater Effluent Standard Solution, Mixed Parameter, for NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> <sup>3-</sup> , COD, SO <sub>4</sub> <sup>2-</sup> , TOC	500 mL	2833249

### Optional reagents and apparatus

Description	Unit	Item no.
Flask, volumetric, Class A, 100 mL, glass	each	1457442
Reactor adapter sleeves, 16 mm to 13 mm diameter, for TNTplus vials	5/pkg	2895805
Sampling bottle with cap, low density polyethylene, 500 mL	12/pkg	2087079
Sulfuric Acid, concentrated, ACS	500 mL	97949
Test tube rack, polyethylene, for 13-mm OD vials, 90 holes	each	2497900
Water, deionized	4 L	27256



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