

## USEPA<sup>1</sup> Reactor Digestion Method<sup>2</sup>

Method 8000

3 to 150 mg/L COD (LR)

TNTplus<sup>®</sup> 821/822

20 to 1500 mg/L COD (HR)

**Scope and application:** For water, wastewater; digestion is required.

<sup>1</sup> COD ranges 3–150 mg/L and 20– 1500 mg/L COD are USEPA approved (5220 D) for wastewater analyses, Federal Register, April 21, 1980, 45(78), 26811-26812

<sup>2</sup> Jirka, A.M.; Carter, M.J., Analytical Chemistry, 1975, 47(8), 1397



## Test preparation

### Instrument-specific information

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
DR3800, DR2800	—	LZV646
DR1900	9609900 or 9609800 (A)	—

### Before starting

DR3900, DR3800, DR2800: 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.

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

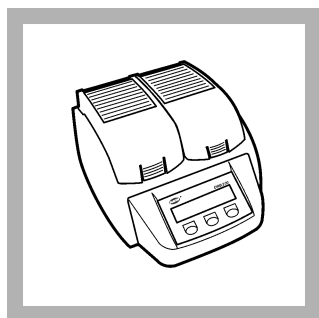
Description	Quantity
COD TNTplus <sup>®</sup> Reagent Set, LR or HR	1
DRB200 reactor with 13-mm wells	1
Blender, 2-speed	1
Pipet, adjustable volume, 1.0–5.0 mL	1
Pipet tips, for 1.0–5.0 mL pipet	1
Test tube rack	1

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

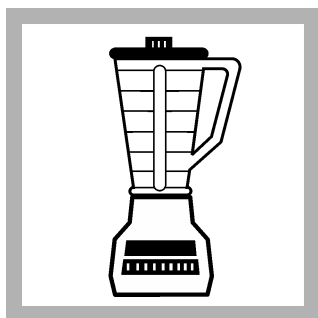
## Sample collection and storage

- 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.

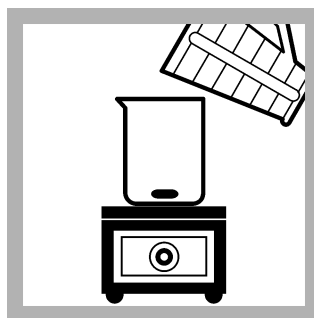
## Test procedure



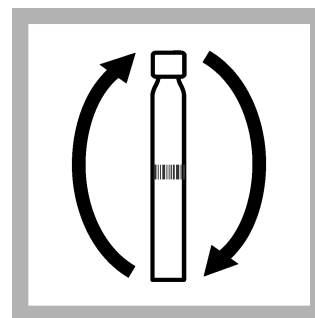
1. Set the DRB200 reactor power to on. Set the temperature to 150 °C.



2. Measure 100 mL of sample in a blender. Blend for 30 seconds or until homogenized. If the sample does not have suspended solids, ignore this step.



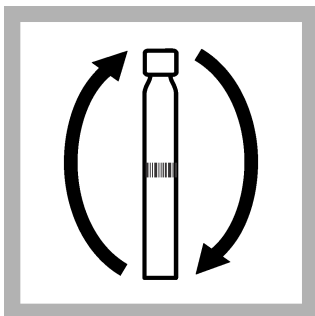
3. Pour the homogenized sample into a 250-mL beaker and stir slowly with a magnetic stir plate. If the sample does not have suspended solids, ignore this step.



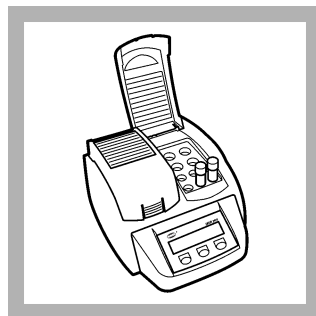
4. Invert a test vial several times to mix.



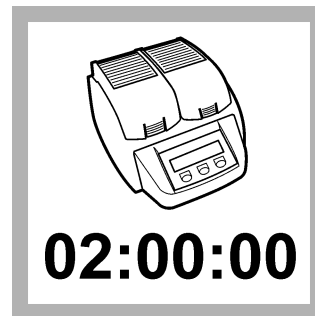
5. Use a pipet to add 2.0 mL of sample to the test vial.



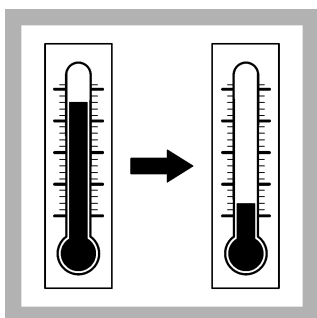
6. Hold the vial by the cap, over a sink. Invert gently several times to mix. **The vial gets very hot during mixing.**



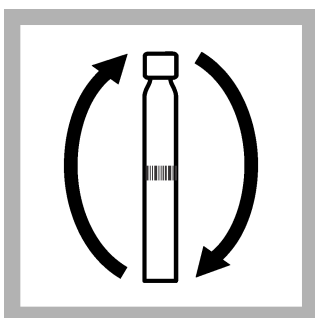
7. Insert the vial in the preheated DRB200 reactor. Close the lid.



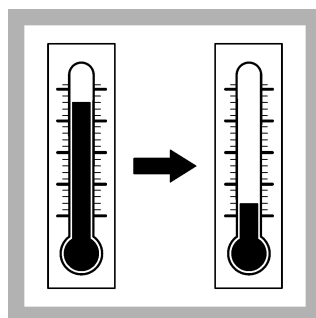
8. Keep the vial in the reactor for 2 hours.



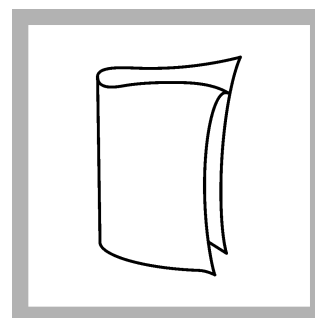
9. When the timer expires, set the reactor power to off. Let the temperature decrease for about 20 minutes to 120 °C or less.



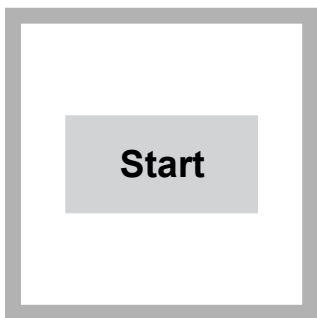
10. Hold the vial by the cap and invert gently several times while the vial is still hot.



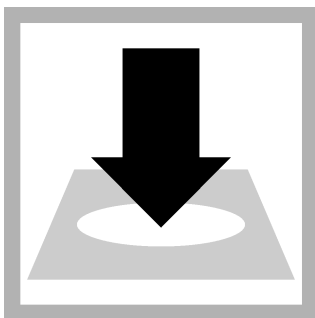
11. Put the vial in a test tube rack. Let the temperature of the vial decrease to room temperature.



12. Clean the vial.



13. DR1900 only: Select program 821 (LR) or 822 (HR). 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

Chloride is the primary interference in this test method and results in a positive interference. Each COD vial contains mercuric sulfate that will eliminate chloride interference to a maximum of 2000 mg/L Cl<sup>-</sup>.

## Accuracy check

### Standard solution method—LR

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
- 10.0-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water

1. Prepare a 100-mg/L COD standard solution as follows:
  - a. Use a pipet to add 10.0 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 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—HR

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

Items to collect:

- COD Standard Solution, 300-mg/L, 800-mg/L or 1000-mg/L COD or Oxygen Demand Standard (contains 617-mg/L COD) or Wastewater Influent Standard Solution, Mixed Parameter (contains 500-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 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 (TNTplus 821, LR)	75 mg/L COD	72–78 mg/L COD	—
barcode (TNTplus 822, HR)	750 mg/L COD	736–764 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 organic compounds react, reducing the dichromate ion (Cr<sub>2</sub>O<sub>7</sub><sup>2-</sup>) to green chromic ion (Cr<sup>3+</sup>). When the 3–150 mg/L colorimetric method is used, the amount of Cr<sup>6+</sup> that remains is measured. When the 20–1500 mg/L colorimetric method is used, the amount of Cr<sup>3+</sup> that is produced is measured. The COD reagent also contains silver and mercury ions. Silver is a catalyst, and mercury is used to complex chloride interferences. The measurement wavelength is 420 nm for the LR or 620 nm for the HR.

## Consumables and replacement items

### Required reagents

Description	Quantity/Test	Unit	Item no.
COD TNTplus <sup>®</sup> Reagent Set, LR, 3 to 150 mg/L COD	1–2 vials	25/pkg	TNT821
COD TNTplus <sup>®</sup> Reagent Set, HR, 20 to 1500 mg/L COD	1–2 vials	25/pkg	TNT822

### Required apparatus

Description	Quantity/test	Unit	Item no.
Blender, 2-speed, 120 VAC option	1	each	2616100
Blender, 2-speed, 240 VAC option	1	each	2616102
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, DR3800, DR2800, DR2700	1	each	LZV646
Light shield, DR3900	1	each	LZV849

### Recommended standards

Description	Unit	Item no.
COD Standard Solution, 300-mg/L	200 mL	1218629
COD Standard Solution, 800-mg/L	200 mL	2672629
COD Standard Solution, 1000-mg/L	200 mL	2253929
Oxygen Demand Standard (BOD, COD, TOC), 10-mL ampules	16/pkg	2833510

**Recommended standards (continued)**

Description	Unit	Item no.
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
Wastewater Influent 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	2833149

**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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