

Mercury-Free Reactor Digestion Method 25 to 1000 mg/L COD (HR)

Method 10236
TNTplus™ 825

Scope and application: For water and wastewater. Digestion is required.



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
DR 6000, DR 5000	—	—
DR 3900	—	LZV849
DR 3800, DR 2800	—	LZV646
DR 1900	9609900 or 9609800 (A)	—

Before starting

DR 3900, DR 3800, DR 2800: 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.

Run one blank with each set of samples. 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.

Store 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, insert adapter sleeves into the wells.

DR 1900: 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, Mercury-free, HR	1
DRB200 reactor with 13-mm wells	1

Items to collect (continued)

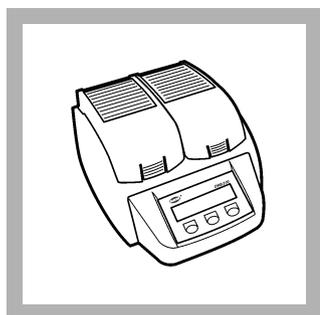
Description	Quantity
Pipet, adjustable volume, 1.0–5.0 mL	1
Pipet tips, for 1.0–5.0 mL pipet	1
Test tube rack	1

Refer to [Consumables and replacement items](#) on page 4 for order information.

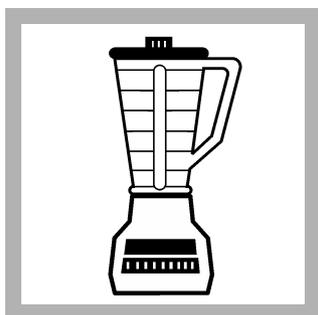
Sample collection and storage

- 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.

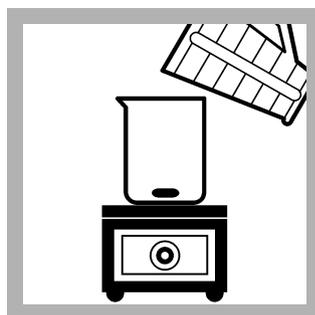
Test procedure



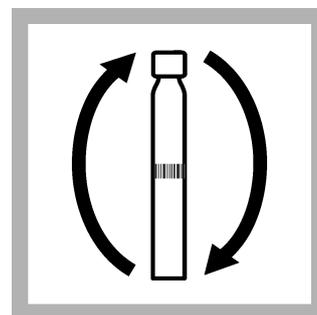
1. Set the DRB200 reactor power to on. Set the temperature to 150 °C.



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.



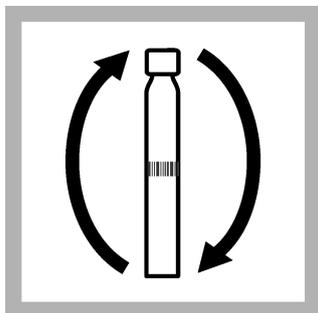
3. Pour the homogenized sample into a 250-mL beaker and stir slowly with a magnetic stir plate.
If the sample does not have suspended solids, ignore this step.



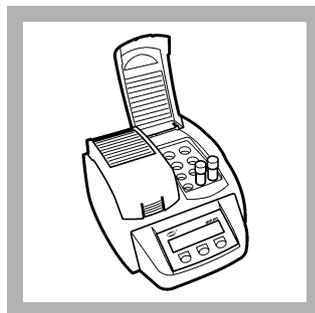
4. Invert a test vial several times to mix.



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



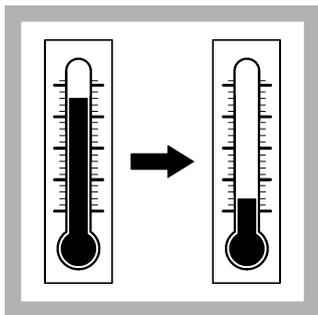
6. Hold the vial by the cap, over a sink. Invert gently several times to mix. **The vial gets very hot during mixing.**



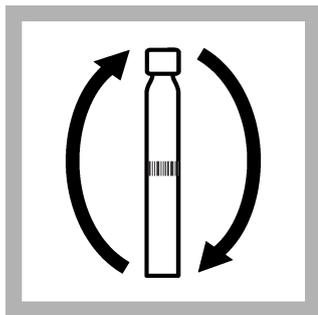
7. Insert the vial in the preheated DRB200 reactor. Close the lid.



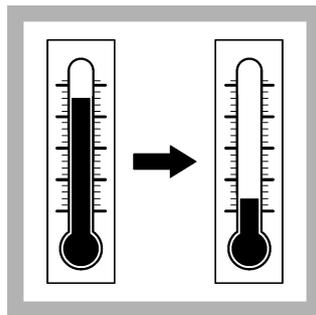
8. Keep the vial in the reactor for 2 hours.



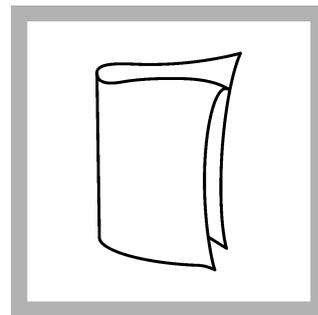
9. When the timer expires, set the reactor power to off. Let the temperature decrease for about 20 minutes to 120 °C or less.



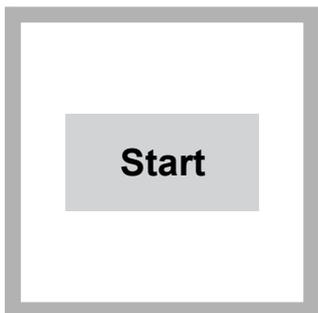
10. Hold the vial by the cap and invert gently several times while the vial is still hot.



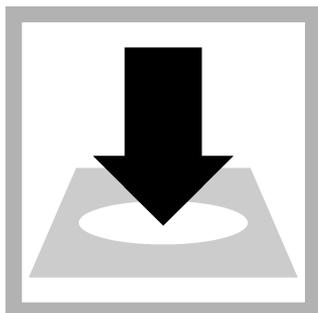
11. Put the vial in a test tube rack. Let the temperature of the vial decrease to room temperature.



12. Clean the vial.



13. DR 1900 only: Select program 825. Refer to [Before starting](#) on page 1.



14. Insert the vial into the cell holder. DR 1900 only: Push **READ**. Results show in mg/L COD.

Blanks for colorimetric determination

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. The interference level is dependent on the chloride and COD concentration. Refer to [Table 2](#). **Example:** If a sample contains 200 mg/L COD and 100 mg/L Cl⁻, the positive interference is 10%. The reading would be 220 mg/L COD.

Table 2 Chloride interference levels

Chloride	COD			
	200 mg/L	400 mg/L	600 mg/L	800 mg/L

Table 2 Chloride interference levels (continued)

100 mg/L	10%	2%	2%	1%
200 mg/L	15%	10%	2%	1%
400 mg/L	20%	10%	5%	2%
600 mg/L	30%	10%	5%	5%
800 mg/L	35%	15%	10%	5%
1000 mg/L	45%	20%	10%	5%

Accuracy check

Standard solution method

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 slight 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	500 mg/L COD	492–508 mg/L COD	20 mg/L COD

Summary of Method

The mg/L COD results are defined as the mg of O₂ consumed per liter of sample under the conditions of this procedure. In this procedure, the sample is heated for 2 hours with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, which reduces the dichromate ion to a green chromic ion. The measurement wavelength is 605 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
COD TNTplus™ Reagent Set, Mercury-free, HR, 25 to 1000 mg/L COD	1	25/pkg	TNT825

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

Required apparatus (continued)

Description	Quantity/test	Unit	Item no.
DRB 200 Reactor, 115 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	1	each	DRB20001
DRB 200 Reactor, 230 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	1	each	DRB20005
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, DR 3800, DR 2800, DR 2700	1	each	LZV646
Light shield, DR 3900	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
Wastewater Influent Standard Solution, Mixed Parameter, for NH ₃ -N, NO ₃ -N, PO ₄ , COD, SO ₄ , 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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