#### DOC316.53.01525

## Oxygen Demand, Chemical

# USEPA Reactor Digestion Method 5 to 60 g/L COD (UHR)

Method 10212 TNTplus® 824

Scope and application: For wastewater and process waters; 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.

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.

Keep new (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.

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

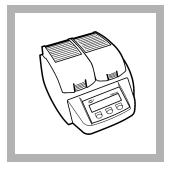
Description	Quantity
COD TNTplus Reagent Set, 5 to 60 g/L	1
DRB200 reactor with 13-mm wells	1
Blender, 2-speed	1
Pipet, adjustable volume, 0.2–1.0 mL	1
Pipet tips, for 0.2–1.0 mL pipet	1
Test tube rack	1

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.

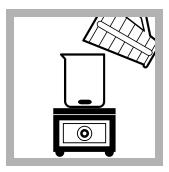
## **Test procedure**



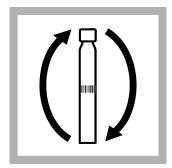
**1.** Set the DRB200 reactor power to on. Set the temperature to 150 °C.



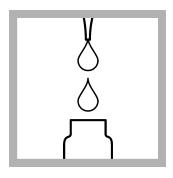
2. Measure 100 mL of sample in a blender. Blend for 30 seconds or until homogenized. If the sample does not have suspended solids, ignore this step.



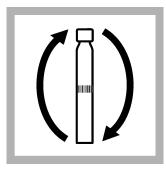
3. Pour the homogenized sample into a 250-mL beaker and stir slowly with a magnetic stir plate. If the sample does not have suspended solids, ignore this step.



**4.** Invert a test vial several times to mix.



**5.** Use a pipet to add 0.2 mL of sample to the test vial.



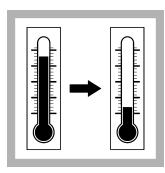
Hold the vial by the cap, over a sink. Invert gently several times to mix. The vial gets very hot during mixing.



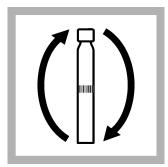
7. Insert the vial in the preheated DRB200 reactor. Close the lid.



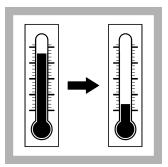
**8.** Keep the vial in the reactor for 2 hours.



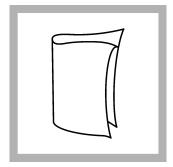
9. When the timer expires, set the reactor power to off. Let the temperature decrease for about 20 minutes to 120 °C or less.



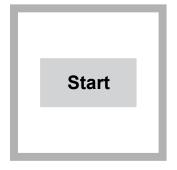
**10.** Hold the vial by the cap and invert gently several times while the vial is still hot.



**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 824. 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.

#### 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 10,000 mg/L Cl<sup>-</sup>.

## **Accuracy check**

#### Standard solution method

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

#### Items to collect:

- · Potassium Acid Phthalate (KHP), ACS
- 1-L volumetric flask, Class A
- Laboratory balance
- Deionized water
- 1. Prepare a 30-g/L COD standard solution as follows:
  - a. Weigh 25.5 g of KHP. Add the KHP to a 1-L volumetric flask.
  - **b.** Dilute to the mark with deionized water. Mix well.
- **2.** Use the test procedure to measure the concentration of the prepared standard solution.
- **3.** Compare the expected result to the actual result.

## **Summary of Method**

Chemical oxygen demand measures the mg of  $O_2$  that is consumed per liter of sample under the conditions of this procedure. In this procedure, the sample temperature is increased to 150 °C for 2 hours with sulfuric acid and a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds in the sample react and reduce the dichromate ion  $(Cr_2O_7^{2-})$  to green chromic ion  $(Cr^{3+})$ . The green color from the  $Cr^{3+}$  ion is measured. The COD reagent also contains silver as a catalyst and mercury to remove interference from chloride. The measurement wavelength is 620 nm.

## Consumables and replacement items

#### Required reagents

Description	Quantity/Test	Unit	Item no.
COD TNTplus Reagent Set, 5 to 60 g/L COD	1 vial	24/pkg	TNT824

### 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
DRB 200 Reactor, 115 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	1	each	DRB200-01
DRB 200 Reactor, 230 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	1	each	DRB200-05
Pipet, adjustable volume, 0.2–1.0 mL	1	each	BBP078
Pipet tips, for 0.2–1.0 mL pipet	1	100/pkg	BBP079
Light shield, DR 3800, DR 2800, DR 2700	1	each	LZV646
Light shield, DR 3900	1	each	LZV849

#### Recommended standards

Description	Unit	Item no.
Potassium Acid Phthalate (KHP), ACS	500 g	31534

#### Optional reagents and apparatus

Description	Unit	Item no.
Flask, volumetric, Class A, 1000 mL glass, Certified	each	2636653
Reactor adapter sleeves, 16 mm to 13 mm diameter, for TNTplus vials	5/pkg	2895805

Optional reagents and apparatus (continued)

Description	Unit	Item no.
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

