

Direct Method

Method 10273

30–300, 300–3000 or 3000–30,000 mg/L C

TNTplus 811

Scope and application: For oil and gas field waters, municipal wastewater and industrial wastewater.



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.

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

Make sure to digest the samples at 100 °C. Higher temperatures may cause the vials to break apart.

Be careful with the vials after the digestion. Pressure increases in the vials during the digestion and can cause the vials to break apart.

Use only the TOC-X5 shaker to remove total inorganic carbon (TIC) from the sample.

Carbon dioxide from the air can contaminate the sample. Do not open the indicator vial before the shaker operation is complete. Immediately install the double cap on the indicator vial after the cap is removed, then immediately install the other side of the double cap on the sample vial.

The formation of crystals in the sample vial does not affect the result.

The recommended temperature for reagent storage is 2–8 °C (35–46 °F).

The recommended sample pH is 3–10.

If the sample contains particles, dilute the sample. Use the diluted sample in the test procedure. Multiply the test result by the dilution factor.

After both vials are attached to the double cap, keep the vial assembly together. Put the vial assembly in the plastic packaging after the analysis.

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

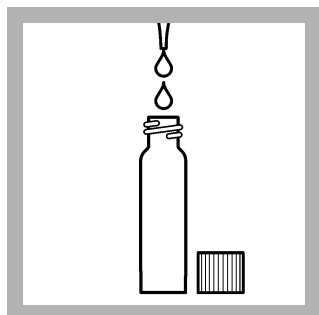
| Description | Quantity |
|--|----------|
| Total Organic Carbon, LR TNTplus 810 Reagent Set | 1 |
| DRB200 reactor with 13-mm wells | 1 |
| TOC-X5 shaker | 1 |
| Pipet, adjustable volume, 1.0–5.0 mL | 1 |
| Pipet, adjustable volume, 1.0–10.0 mL | 1 |
| Pipet tips, for 1.0–5.0 mL pipet | 1 |
| Pipet, adjustable volume, 1.0–10.0 mL | 1 |
| Test tube rack | 1 |
| Flask, volumetric, Class A, 100-mL | 1 |

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

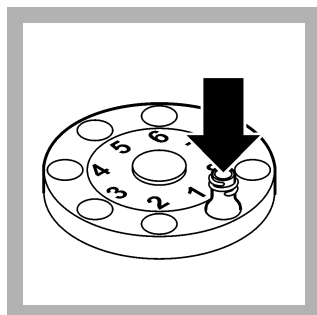
Sample collection

- Collect samples in clean glass bottles.
- Homogenize samples that contain solids to get a representative sample.
- Rinse the sample bottle several times with the sample to be collected.
- Fill the bottle completely full, then tighten the cap on the bottle.
- Analyze the samples as soon as possible for best results.
- Acid preservation is not recommended.

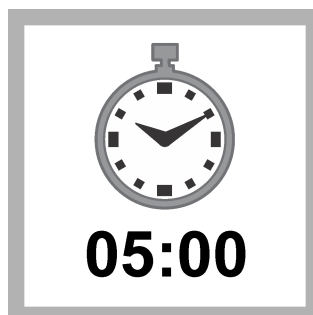
Test procedure



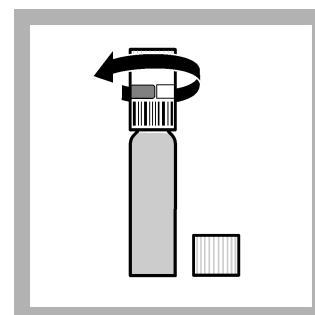
1. Remove the cap from a clear vial. Use a pipet to add 1 mL of sample to the vial. For the 300–3000 and 3000–30,000 mg/L concentration range, add 1 mL of the diluted sample to the vial. Refer to [Sample dilution](#) on page 3.



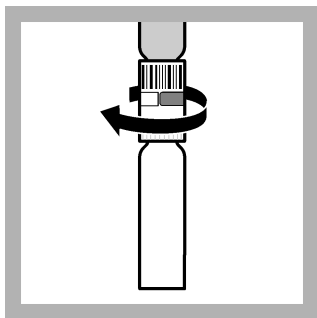
2. Insert the uncapped sample vial into the TOC-X5 shaker. Make sure that the vial is pushed all the way down into the shaker. Move the fan over the vial.



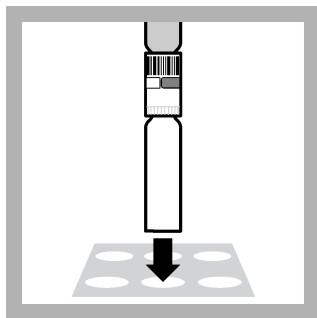
3. Push the on/off switch to start the shaker. Operate the shaker for 5 minutes.



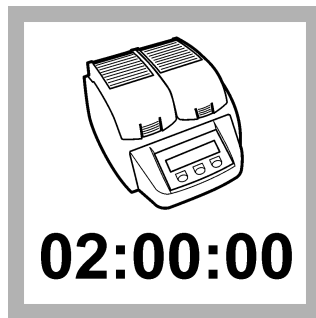
4. When the shake time is complete, remove the cap from a blue indicator vial. Immediately install and tighten a double cap on the indicator vial with the barcode label toward the vial.



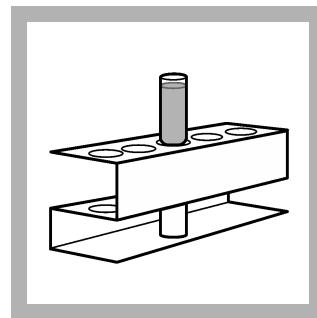
5. Immediately invert the indicator vial, then install and tighten the other side of the double cap on the sample vial. Hold the vial assembly vertically.



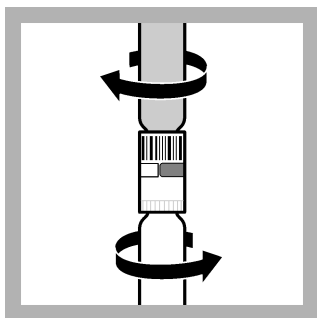
6. Insert the vial assembly into the DRB reactor (indicator vial on top).



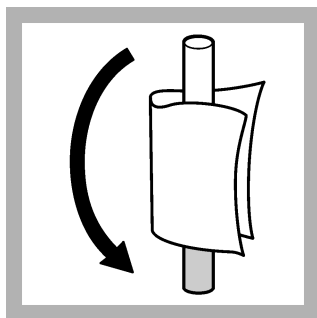
7. Increase the vial assembly temperature for 2 hours at 100 °C.



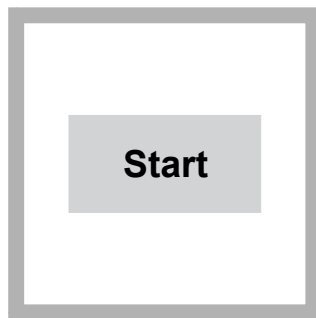
8. Let the vial assembly cool completely to room temperature. Make sure that the vials cool completely. Warm vials will give high results.



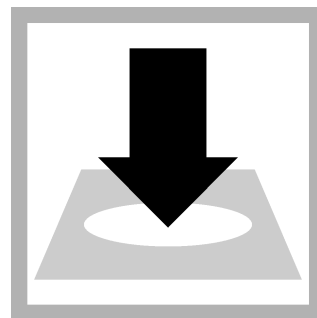
9. Tighten the double cap on both vials.



10. Invert the vial assembly so the indicator vial is on the bottom. Clean the indicator vial.



11. DR 1900 only: Select program 811. Refer to [Before starting](#) on page 1.



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

Sample dilution

Dilute the sample to increase the concentration range of this method and decrease the chloride interference. Refer to [Chloride interference](#) on page 4.

Do the steps that follow to make a 10x (10-fold: Sample is diluted by a factor of 10) or 100x (100-fold) dilution:

1. Use a pipet to add the selected sample portion to a clean volumetric flask. Fill the flask to the necessary volume with deionized TOC-free water.
 - a. Prepare a 10x dilution—Add 1 mL of sample and fill to 10 mL with deionized water (1 mL sample + 9 mL of deionized water).
 - b. Prepare a 100x dilution—Add 1 mL of sample and fill to 100 mL with deionized water (1 mL sample + 99 mL of deionized water).
2. Mix well.
3. Use the diluted sample to complete the test.

Interferences

The table that follows shows the substances that were tested for interference and did not interfere up to the levels shown.

| Interfering substance | Interference level |
|-----------------------|--------------------------------|
| Ammonium | 2000 mg/L |
| Calcium | 2000 mg/L as CaCO ₃ |
| Chloride | 3000 mg/L |

| Interfering substance | Interference level |
|-----------------------|--------------------------------|
| Magnesium | 2000 mg/L as CaCO ₃ |
| TIC | 250 mg/L |

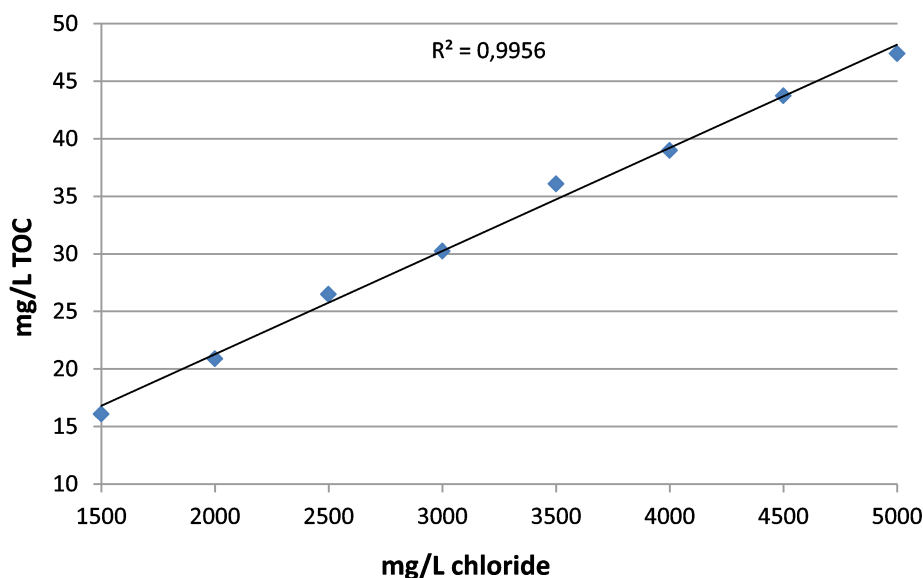
Chloride interference

Chloride ion is frequently found in produced and flowback waters. High levels of chloride¹ interfere with the test because the chloride changes to chlorine during the digestion process. The chlorine gas goes through the membrane and bleaches the color indicator. This causes a false-positive result.

For the 30–300 mg/L TOC range, chloride interferes with the TOC as shown in [Figure 1](#). At 3000 mg/L, chloride causes an interference of 30 mg/L TOC, which is at the minimum permitted of the concentration range for undiluted samples. If the sample is diluted the chloride interference concentration increases. Refer to [Sample dilution](#) on page 3.

- For a 10x dilution, the chloride interference concentration increases from 3000 to 30,000 mg/L.
- For a 100x dilution, the chloride interference concentration increases from 3000 to 300,000 mg/L.

Figure 1 Chloride interference with TOC



Accuracy check

Standard solution method

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

Items to collect:

- 1000-mg/L C, TOC Standard Solution
- 200-mL volumetric flask, Class A
- 20-mL volumetric pipet, Class A and pipet filler safety bulb
- Organic-free water

¹ The test is not applicable for TOC concentrations of approximately 30 mg/L and chloride concentrations of 10,000 mg/L or higher.

1. Prepare a 100-mg/L C standard solution as follows:
 - a. Use a pipet to add 20 mL of a 1000-mg/L C standard solution into the volumetric flask.
 - b. Dilute to the mark with organic-free 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.

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 |
|-------------|-----------|-------------------------------------|--|
| TNTplus 811 | 50 mg/L C | 48.84–51.16 mg/L C | 3.5 mg/L C |

Summary of method

The total inorganic carbon (TIC) in the sample is first removed during the shaker operation. The sample is then digested to oxidize the total organic carbon (TOC) in the sample to carbon dioxide (CO₂). The CO₂ from the digested sample goes through the membrane in the double cap to the indicator vial and causes the indicator solution to change color. The color of the indicator solution is measured by the spectrophotometer. The measurement wavelength is 435 nm.

Consumables and replacement items

Required reagents

| Description | Quantity/test | Unit | Item no. |
|---|---------------|--------|----------|
| Total Organic Carbon Reagent Set, HR, TNTplus | 1 | 25/pkg | TNT811 |

Required apparatus

| 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, adjustable volume, 1.0–10.0 mL | 1 | each | 1970010 |
| Pipet tips, for 1.0–5.0 mL pipet | 1 | 75/pkg | BBP068 |
| Pipet tips, for 1.0–10.0 mL pipet | 1 | 50/pkg | 2558996 |
| Test tube rack | 1 | each | 1864100 |
| Flask, volumetric, Class A, 100-mL | 1 | each | 2636642 |
| TOC-X5 shaker | 1 | each | LQV148.99.00002 |
| Wipes, disposable | 1 | 280/pkg | 2097000 |

Recommended standards

| Description | Unit | Item no. |
|--|-------|----------|
| TOC Standard Solution Ampule (KHP Standard, 1000-mg/L C) | 5/pkg | 2791505 |

Optional reagents and apparatus

| Description | Unit | Item no. |
|---|--------|----------|
| Reactor adapter sleeves, 16 mm to 13 mm diameter, for TNTplus vials | 5/pkg | 2895805 |
| Ampule Breaker, 2-mL PourRite [®] Ampules | each | 2484600 |
| Flask, volumetric, Class A, 200 mL | each | 1457445 |
| Pipet, volumetric Class A, 20 mL | each | 1451520 |
| Pipet filler, safety bulb | each | 1465100 |
| Potassium Acid Phthalate (KHP), ACS | 500 g | 31534 |
| Water, organic-free | 500 mL | 2641549 |



FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – Call toll-free 800-227-4224
Outside the U.S.A. – Contact the HACH office or distributor serving you.
On the Worldwide Web – www.hach.com; E-mail – techhelp@hach.com

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932