Iron, TNTplus

DOC316.53.01249

Phenanthroline Method[1]

Method 10229

0.2 to 6.0 mg/L Fe

TNTplus™ 858

Scope and application: For drinking water and wastewater. Digestion can be necessary to determine total iron.

[1] Adapted from Standard Methods for the Examination of Water and 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.
The recommended sample pH is 3–10.
The recommended temperature for samples and reagents is 15–25 °C (59–77 °F).
The recommended temperature for reagent storage is 2–8 °C (35–46 °F).
To make sure that all forms of the metal are measured, digest the sample with heat and acid. Use the Metals Prep Set TNTplus 890 to digest the sample.
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

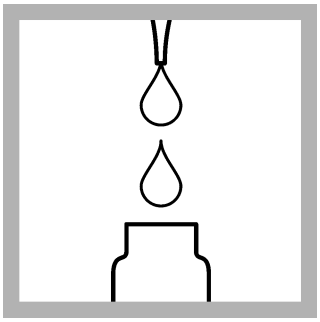
Description	Quantity
Iron TNTplus Reagent Set	1
Pipet, adjustable volume, 1.0–5.0 mL	1
Pipet tips, for 1.0–5.0 mL pipet	1

Refer to Consumables and replacement items on page 4 for order information.

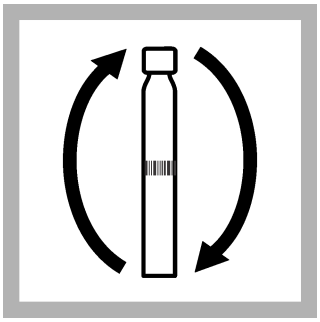
Sample collection and storage

- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (approximately 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- If only dissolved iron is to be determined, filter the sample before the acid addition.
- Keep the preserved samples at room temperature for a maximum of 6 months.
- Before analysis, adjust the pH to 3–5 with 5 N sodium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

Test procedure

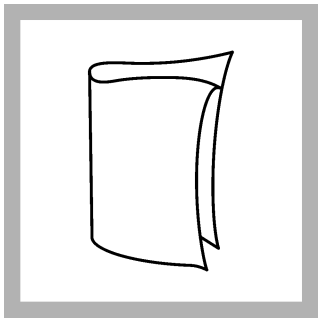


1. Use a pipet to add 2.0 mL of sample to the test vial.

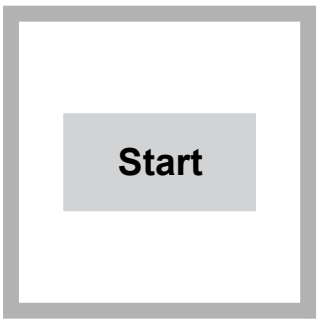


2. Tighten the cap on the vial and invert until completely mixed.

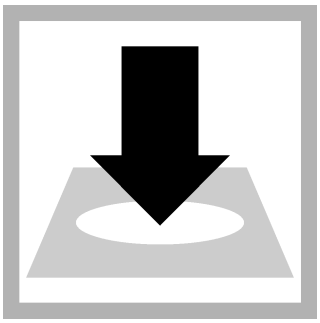
3. Start the reaction time of 15 minutes.



4. When the timer expires, clean the vial.



5. DR 1900 only: Select program 858. Refer to Before starting on page 1.



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

Sample blank

Samples with color or turbidity can cause high test results. Samples without color or turbidity do not have a sample blank. To adjust for color or turbidity, use the steps that follow to find the sample blank.

1. Complete the test procedure. Pour the sample into a TNT919 vial. After the TNT858 results are displayed, place the vial into the cell holder.
2. The instrument automatically calculates the sample blank and displays the corrected sample concentration by subtracting the sample background color and/or turbidity.
   Note: *As an alternative, samples with only turbidity can be filtered through a membrane filter, then analyzed.*

Reagent blank correction

For the best results, measure the reagent blank value for each new lot of reagent. 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. Measure the reagent blank value when a new lot of reagent is used.

1. Use deionized water as the sample in the test procedure to measure the reagent blank value.
2. Set the reagent blank function to on. The measured reagent blank value is shown.
3. Accept the blank value. The reagent blank value is then subtracted from all results until the reagent blank function is set to off or a different method is selected.
   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

Ions that do not cause an interference to the maximum tested concentrations are shown in Table 2. Combinations of ions were not tested.

Table 2 Interfering substances

Interfering substance	Interference level
Cd^{2+}	No effect at 70 mg/L.
Ca^{2+}	No effect at 500 mg/L.
CO_3^{2-}	No effect at 50 mg/L.
Cl^-	No effect at 1000 mg/L.
Cr^{3+}, Cr^{6+}	No effect at 50 mg/L.
Co^{2+}	No effect at 50 mg/L.
Color	Can cause high results. To make a correction for the interference, measure a Sample blank.
Cu^{2+}	No effect at 10 mg/L. Higher concentrations cause high results.
Pb^{2+}	No effect at 50 mg/L.
Hg^{2+}	No effect at 50 mg/L.
Ni^{2+}	No effect at 25 mg/L. Higher concentrations cause high results.
NO_3^-	No effect at 50 mg/L.
Potassium, K^+	No effect at 500 mg/L.
Sodium, Na^+	No effect at 500 mg/L.

Accuracy check

Standard solution method

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

Items to collect:

- 100-mg/L Iron Standard Solution
- 100-mL volumetric flask, Class A
- Pipet, adjustable volume, 1.0–5.0 mL and pipet tips
- Deionized water

1. Prepare a 2.00-mg/L iron standard solution as follows:
   a. Use a pipet to add 2.0 mL of a 100-mg/L iron 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.*

Summary of Method

Ferrous iron (Fe^{2+}) forms an orange-red complex with the 1,10-phenanthroline indicator in the reagent. Any ferric iron (Fe^{3+}) in the water sample is reduced to ferrous iron (Fe^{2+}) by ascorbic acid before the complex is formed. The measurement wavelength is 515 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Iron TNTplus Reagent Set	1	25/pkg	TNT858

Required apparatus

Description	Quantity/test	Unit	Item no.
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.
Iron Standard Solution, 100-mg/L Fe	100 mL	1417542
Iron Standard Solution, 1-mg/L Fe	500 mL	13949

Optional reagents and apparatus

Description	Unit	Item no.
DRB 200 Reactor, 115 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	each	DRB20001
DRB 200 Reactor, 230 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	each	DRB20005
Flask, volumetric, Class A, 100 mL, glass	each	1457442
Metals Prep Set TNTplus	50/pkg	TNT890
Nitric Acid, concentrated	500 mL	15249
Sample blank vials for TNTplus methods	5/pkg	TNT919
Sampling bottle with cap, low density polyethylene, 500-mL	12/pkg	2087079
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Water, deionized	4 L	27256

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