Fluoride

DOC316.53.01507

USEPA SPADNS 2 Method[1]

Method 10225

0.1–2.50 mg/L F^-

TNTplus 878

Scope and application: For water, wastewater and seawater. USEPA accepted for reporting for drinking and wastewater analyses (distillation required)[2].

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater, 4500-F B & D.*
[2] Procedure is equivalent to USEPA Method 340.1 for drinking 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 1–11.
The sample temperature must be 20 °C (68 °F) for accurate results.
The recommended temperature for reagent storage is 15–25 °C (59–77 °F).
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

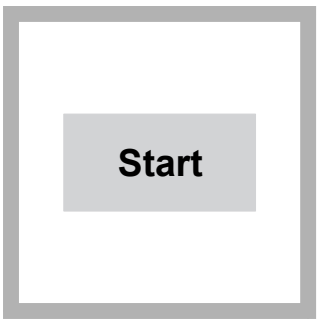
Description	Quantity
Fluoride TNTplus Reagent Set	1
Pipet, adjustable volume, 1.0–5.0 mL	1
Pipet tips, for 1.0–5.0 mL pipet	1

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

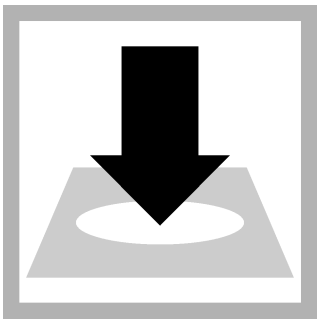
Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- To preserve samples for later analysis, keep the samples at or below 6 °C (43 °F) for up to 28 days.
- Let the sample temperature increase to room temperature before analysis.

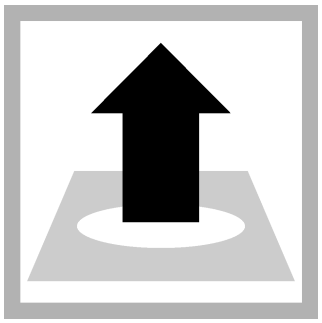
Test procedure



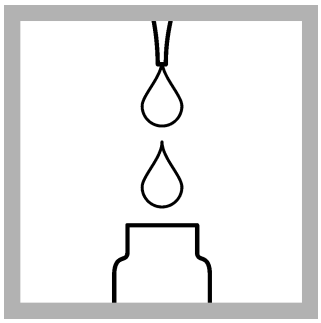
1. DR 1900 only: Select program 878. Refer to Before starting on page 1.



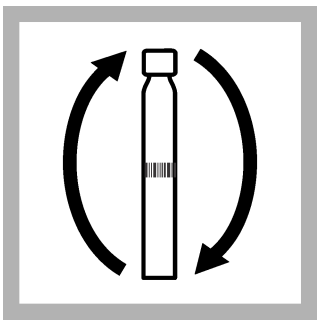
2. Insert the vial into the cell holder. DR1900 only: Push **READ 1**.



3. Remove the vial from the cell holder.

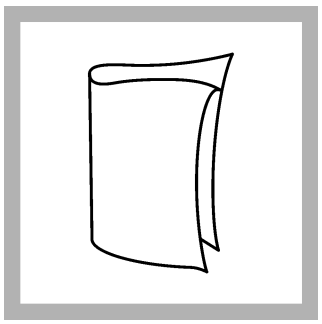


4. Use a pipet to add 3.0 mL of sample to the test vial.

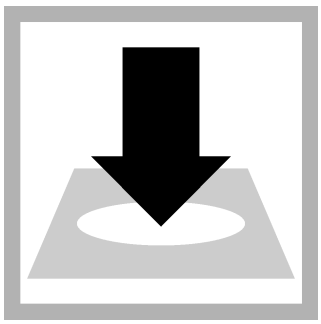


5. Tighten the cap on the vial and invert the vial 2–3 times.

6. Start the reaction time of 1 minute.



7. Clean the vial.



8. Insert the vial into the cell holder. DR1900 only: Push **READ 2**.
Results show in mg/L F^-.

Interferences

Interfering substance	Interference level
Alkalinity (as $CaCO_3$)	At 5000 mg/L, it causes a –0.1 mg/L F^- error.
Aluminum	At 0.1 mg/L, it causes a –0.1 mg/L F^- error. To find whether there is an aluminum interference, read the concentration 1 minute after reagent addition, then again after 15 minutes. An appreciable increase in concentration suggests aluminum interference. To remove the effect of up to 3.0 mg/L aluminum, wait 2 hours, then take the final reading.
Chloride	At 7000 mg/L, it causes a +0.1 mg/L F^- error.
Chlorine	SPADNS 2 Reagent contains enough non-toxic reductant to remove interference of up to 5 mg/L chlorine. For higher chlorine levels: **1.** Dilute the sample with deionized water by a factor that will lower the chlorine concentration to below 5 mg/L. **2.** Use the test procedure to measure the fluoride concentration. **3.** Multiply the result by the dilution factor to get mg/L fluoride.
Iron, ferric	At 10 mg/L, it causes a –0.1 mg/L F^- error.
Phosphate, ortho	At 16 mg/L, it causes a +0.1 mg/L F^- error.

Interfering substance	Interference level
Sodium hexametaphosphate	At 1.0 mg/L, it causes a +0.1 mg/L F^- error.
Sulfate	At 200 mg/L, it causes a +0.1 mg/L F^- error.

Distillation

To eliminate most interferences, distill the sample, then use the distilled sample in the test procedure.

Prerequisite—prepare the distillation solution:

1. Measure 60 mL of deionized water into a 250-mL, glass Erlenmeyer flask.
2. With constant stirring, add 120 mL of concentrated sulfuric acid. **Caution: The mixture will become very hot. Put the flask in an ice bath to decrease the temperature of the solution.**

Distillation procedure:

1. Set up the distillation apparatus for general purpose distillation. Refer to the Distillation Apparatus manual for proper assembly.
2. Set up a 125-mL Erlenmeyer flask to collect the distillate.
3. Turn on the water and adjust to maintain a steady flow through the condenser.
4. Use a 100-mL graduated cylinder to add 100 mL of sample into the distillation flask.
5. Add a magnetic stir bar and 5 glass beads.
6. Set the stirrer power to on. Set the stir control to 5.
7. Use a 250-mL graduated cylinder to carefully add 150 mL of distillation solution into the flask.

   Note: *For samples with large amounts of chloride, add 5 mg of silver sulfate to the sample for every mg/L of chloride in the sample.*
8. With the thermometer inserted, set the heat control to 10. The yellow pilot lamp is an indication that the heater is on.
9. When the temperature is 180 °C (356 °F) or when 100 mL of distillate has been collected, turn the still off (takes about 1 hour).
10. Dilute the distillate to a volume of 100 mL, if necessary. Use the diluted distillate in the test procedure.

Accuracy check

Standard solution method

Use the standard solution method to validate the test procedure, the reagents (if applicable) and the instrument.

Items to collect:

- Standard solution within the test range

1. Use the test procedure to measure the concentration of the standard solution.
2. Compare the expected result to the actual result.

Summary of Method

The SPADNS 2 Method for fluoride determination involves the reaction of fluoride with a red zirconium-dye solution. The fluoride combines with part of the zirconium to form a colorless complex that bleaches the red color in an amount proportional to the fluoride concentration. This method is equivalent to the EPA method for NPDES and NPDWR reporting purposes when the samples have been distilled. Seawater and wastewater samples require distillation The measurement wavelength is 588 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Fluoride TNTplus Reagent Set	1	25/pkg	TNT878

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
Thermometer	1	each	2635700
Light shield, DR 3900	1	each	LZV849
Light shield, DR 3800, DR 2800, DR 2700	1	each	LZV646

Recommended standards

Description	Unit	Item no.
Fluoride Standard Solution, 0.2-mg/L F^-	500 mL	40502
Fluoride Standard Solution, 0.5-mg/L F^-	500 mL	40505
Fluoride Standard Solution, 0.8-mg/L F^-	500 mL	40508
Fluoride Standard Solution, 1.0-mg/L F^-	1000 mL	29153
Fluoride Standard Solution, 1.0-mg/L F^-	500 mL	29149
Fluoride Standard Solution, 1.2-mg/L F^-	500 mL	40512
Fluoride Standard Solution, 1.5-mg/L F^-	500 mL	40515
Fluoride Standard Solution, 2.0-mg/L F^-	500 mL	40520
Fluoride Standard Solution, 100-mg/L F^-	500 mL	23249
Drinking Water Standard, Mixed Parameter, Inorganic for F^-, NO_3–N, PO_4^{3-}, SO_4^{2-}	500 mL	2833049

Distillation reagents and apparatus

Description	Unit	Item no.
Graduated cylinder, 100-mL	each	50842
Graduated cylinder, 250-mL	each	50846
Distillation apparatus set, general purpose	each	2265300
Distillation heater and support for apparatus set, 115 VAC option	each	2274400
Distillation heater and support for apparatus set, 230 VAC option	each	2274402
Flask, Erlenmeyer, 125-mL	each	2089743
Flask, Erlenmeyer, 250-mL	each	50546
Glass beads	100/pkg	259600
Stir bar, magnetic	each	1076416
Sulfuric Acid, ACS	500 mL	97949

Optional reagents and apparatus

Description	Unit	Item no.
Silver Sulfate	113 g	33414
Balance, analytical, 80 g x 0.1 mg 100–240 VAC	each	2936701
Paper, for weighing, 100 x 100 mm	500/pkg	1473885

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