

Esterification Method¹

Method 10240

50 to 2500 mg/L CH₃COOH (Acetic Acid)

TNTplus™ 872

Scope and application: For digested sludges, activated sludges, process water and food products.

¹ Adapted from The Analyst, 87, 949 (1962).



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 3–9.

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).

Filter samples that are turbid through a 0.45-µm filter.

Centrifuge digested sludge samples at 6000 rpm for 10 minutes. The time to get the digested sludge water should be as short as possible (< 15 minutes) to prevent degradation of the volatile acids.

Keep the time for sample preparation and analysis the same from sample to sample for consistent results.

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

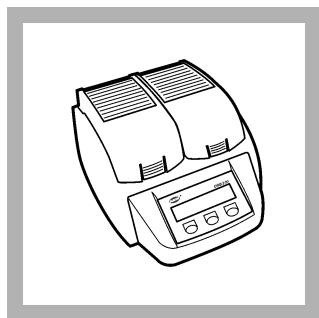
Description	Quantity
Volatile Acids TNTplus Reagent Set	1
DRB200 reactor with 13-mm wells	1
Pipet, adjustable volume, 1.0–5.0 mL	1
Pipet, adjustable volume, 0.2–1.0 mL	1
Pipet tips	1
Test tube rack	1

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

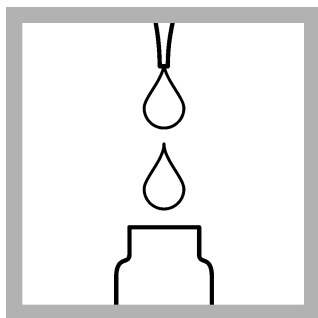
Sample collection

- Collect samples in clean glass or plastic bottles with tight-fitting caps. Completely fill the bottle and immediately tighten the cap.
- Prevent agitation of the sample or exposure to air.
- Analyze the samples as soon as possible for best results.
- If immediate analysis is not possible, keep the samples at or below 6 °C (43 °F) for a maximum of 24 hours.
- Let the sample temperature increase to room temperature before analysis.

Test procedure



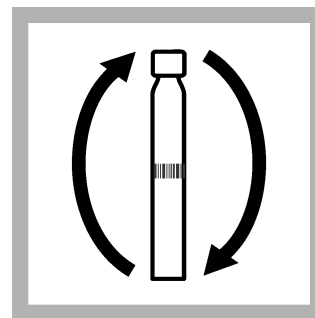
1. Set the DRB200 reactor power to on. Set the temperature to 100 °C.



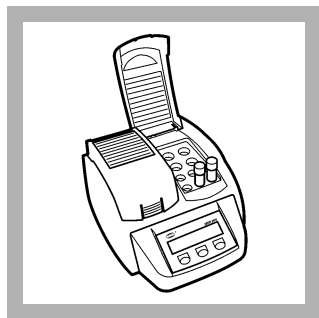
2. Use a pipet to add 0.4 mL of Solution A to the test vial.



3. Use a pipet to add 0.4 mL of sample to the test vial.



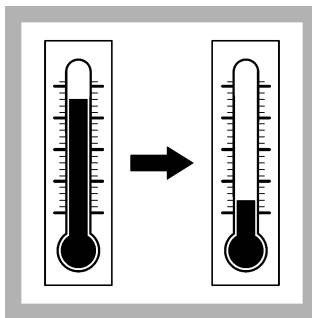
4. Tighten the cap on the vial and invert the vial 2–3 times.



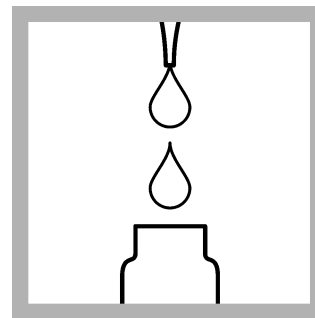
5. Insert the vial in the preheated DRB200 reactor. Close the lid.



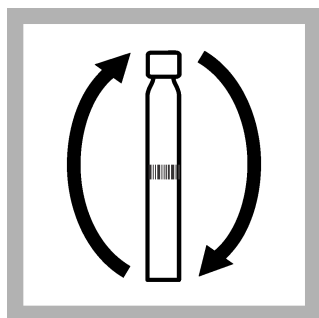
6. Start the reaction time of 10 minutes.



7. When the timer expires, carefully remove the vial from the reactor. Set the vial in a test tube rack. Let the temperature of the vial decrease to room temperature.



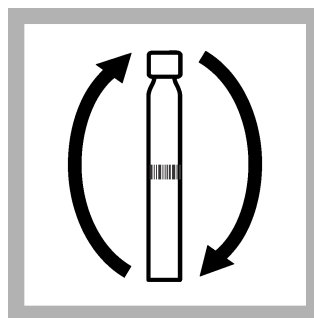
8. Use a pipet to add 0.4 mL of Solution B to the test vial.



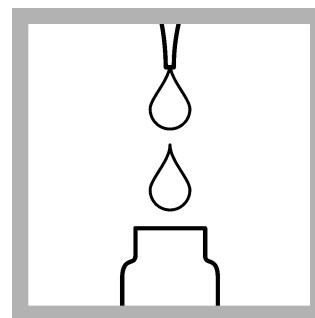
9. Tighten the cap on the vial and invert the vial 2–3 times.



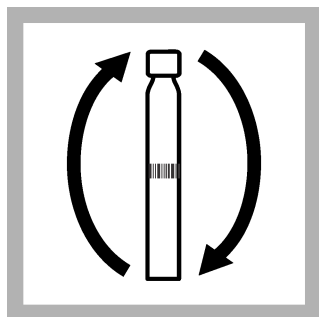
10. Use a pipet to add 0.4 mL of Solution C to the test vial.



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



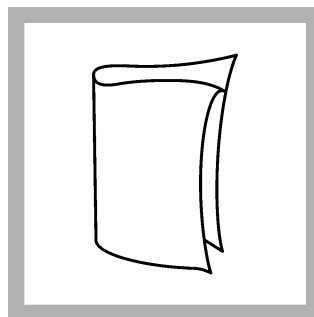
12. Use a pipet to add 2.0 mL of Solution D to the test vial.



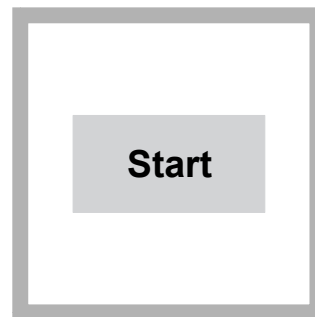
13. Tighten the cap on the vial and invert the vial 2–3 times.



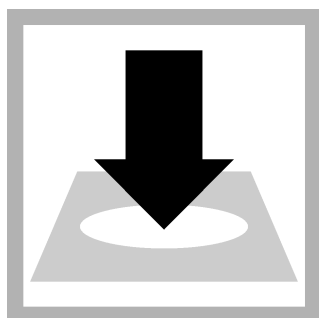
14. Start the reaction time of 3 minutes.



15. When the timer expires, clean the vial.



16. DR 1900 only: Select program 872. Refer to [Before starting](#) on page 1.



17. Insert the vial into the cell holder. DR 1900 only: Push **READ**.
Results show in mg/L CH_3COOH (Acetic Acid).

Interferences

[Table 2](#) shows that the ions were individually examined to the given concentrations and do not cause interference. No cumulative effects or influences of other ions were found.

Table 2 Interfering substances

Interfering substance	Interference level
Acetaldehyde, CH_3CHO	50 mg/L
Acetone, $\text{CO}(\text{CH}_3)_2$	50 mg/L
Aluminum, Al^{3+}	50 mg/L
Ammonium, NH_4^+	250 mg/L
Cadmium, Cd^{2+}	50 mg/L

Table 2 Interfering substances (continued)

Interfering substance	Interference level
Calcium, Ca^{2+}	1000 mg/L
Carbonate, CO_3^{2-}	50 mg/L
Chloride, Cl^-	2000 mg/L
Chromium, Cr^{3+}	50 mg/L
Chromium, Cr^{6+}	5 mg/L
Cobalt, Co^{2+}	50 mg/L
Copper, Cu^{2+}	50 mg/L
Formaldehyde (CH_2O)	50 mg/L
Iodide, I^-	50 mg/L
Iron, Fe^{2+} , Fe^{3+}	5 mg/L
Lead, Pb^{2+}	50 mg/L
Magnesium, Mg^{2+}	1000 mg/L
Manganese, Mn^{2+}	50 mg/L
Molybdenum, Mo^{2+}	50 mg/L
Nickel, Ni^{2+}	10 mg/L
Nitrite, NO_2^-	50 mg/L
Potassium, K^+	1000 mg/L
Silica, SiO_2	50 mg/L
Sodium, Na^+	1000 mg/L
Sulfate, SO_4^{2-}	2000 mg/L
Sulfide, S^{2-}	50 mg/L
Sulfite, SO_3^{2-}	50 mg/L
Tin, Sn^{2+}	50 mg/L
Zinc, Zn^{2+}	25 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:

- Volatile Acids Voluette® Ampule Standard Solution, 62,500-mg/L as acetic acid
 - 500-mL volumetric flask, Class A
 - Pipet, adjustable volume, 1.0-5.0 mL and pipet tips
 - Ampule breaker, 10-mL Voluette
 - Deionized water
1. Prepare a 500-mg/L volatile acids standard solution as follows:
 - a. Use a pipet to add 4.0 mL of a 62,500 mg/L volatile acids 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 slight variations in the reagents or instruments.

Summary of Method

Volatile acids react with diols in an acidic environment to form fatty acid esters. These esters are reduced by iron (III) salts to form red complexes. The measurement wavelength is 497 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Volatile Acids TNTplus Reagent Set	1	25/pkg	TNT872

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 tips, for 1.0–5.0 mL pipet	1	75/pkg	BBP068
Pipet, adjustable volume, 0.2–1.0 mL	1	each	BBP078
Pipet tips, for 0.2–1.0 mL pipet	2	100/pkg	BBP079
Test tube rack	1	each	1864100
Light shield, DR 3800, DR 2800, DR 2700	1	each	LZV646
Light shield, DR 3900	1	each	LZV849

Recommended standards

Description	Unit	Item no.
Volatile Acids Standard Solution, 10-mL Voluette [®] Ampule, 62,500-mg/L as HOAC	16/pkg	1427010

Optional reagents and apparatus

Description	Unit	Item no.
Ampule Breaker, 10-mL Voluette [®] Ampules	each	2196800
Filter membrane, 0.45-micron, 25-mm	100/pkg	2514101
Filter holder, 25-mm, for Luer-type syringe	each	246800
Flask, volumetric, Class A, 500-mL glass	each	1457449
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
Syringe, 10-cc, Luer-Lock tip	each	2202400
Test tube rack, polyethylene, for 13-mm OD vials, 90 holes	each	2497900
Water, deionized	4 L	27256



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.

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