

USEPA<sup>1</sup> 4-Aminoantipyrine Method<sup>2</sup>

Method 10266

5 to 40 mg/L, 20 to 150 mg/L

TNTplus 868

**Scope and application:** For wastewater, seawater, drinking water, surface water and process water. For the exhaust air (after absorption) and exhaust air condensates that form during the manufacture and processing of benzene, petroleum products, glass and mineral fibres, hardboard, coke, oil shale, hazardous waste, town gas, coal and brown coal products, tar, asphalt and bitumen.

<sup>1</sup> USEPA procedure is equivalent to USEPA method 420.1 for wastewater.

<sup>2</sup> 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 2–10.

The sample temperature must be 20–25 °C (68–77 °F) for accurate results.

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

Distill all the samples and the standards<sup>1</sup> with a Micro Dist distillation block. Refer to [Distillation procedure](#) on page 2.

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.

<sup>1</sup> Always distill the standards with the samples. For the most accurate results, complete a user calibration with distilled standards.

## Items to collect

Description	Quantity
Phenols TNTplus Reagent Set	1
Pipet, adjustable volume, 1.0–5.0 mL	1
Pipet, adjustable volume, 0.2–1.0 mL	1
Pipet tips	1

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

## Sample collection

- Analyze the samples as soon as possible for best results.
- Collect samples in clean glass bottles.
- Rinse the sample bottle several times with the sample to be collected.
- Collect a sufficient quantity of sample to get a representative sample, for replicate analysis (if necessary) and for minimum waste disposal.
- At the time of collection, adjust the sample pH to 2 or less with sulfuric acid.
- If prompt analysis is not possible, keep the sample at or below 6 °C (43 °F) for a maximum of 28 days.

## Distillation procedure

Items to collect:

- Micro Dist distillation block
- Micro Dist tubes, user-fill
- Pipet, 1.0–10.0 mL
- Pipet tips

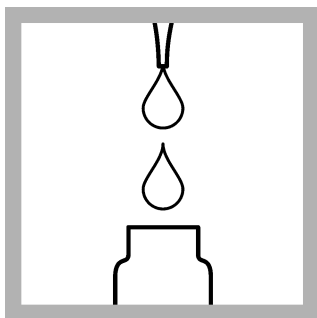
Distill all samples and standards with a Micro Dist distillation block. Refer to the Micro Dist documentation for the distillation procedure and [Table 2](#). Always distill the standards with the samples. For the most accurate results, complete a user calibration with distilled standards.

Adjust the sample and the standard to approximately a pH of 4 with 1 M NaOH or 10% H<sub>2</sub>SO<sub>4</sub> before distillation.

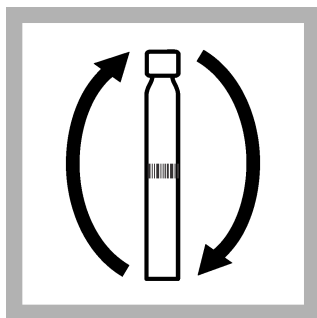
**Table 2 Micro Dist information for phenols**

Specification	Value
Block temperature	130 °C
Sample or standard solution	6.0 mL
Distillation time	90 minutes

## Test procedure



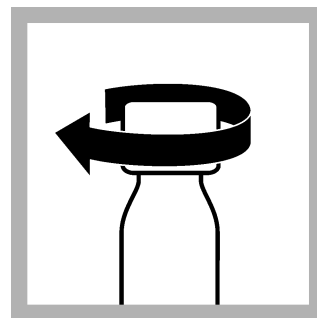
1. Add the sample volume that is specified for the test range to the test vial. Refer to [Sample volumes](#) on page 3.



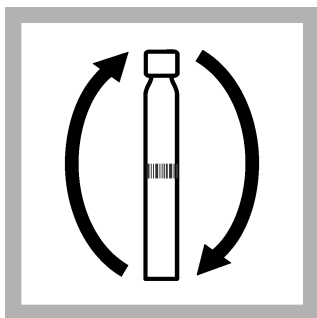
2. Tighten the cap on the vial and invert the vial 2–3 times.



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



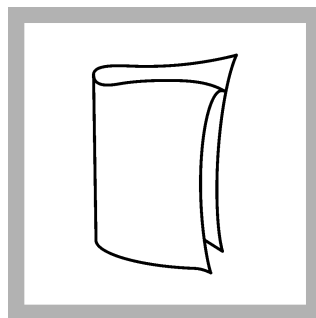
4. Put an orange DosiCap B on the vial.



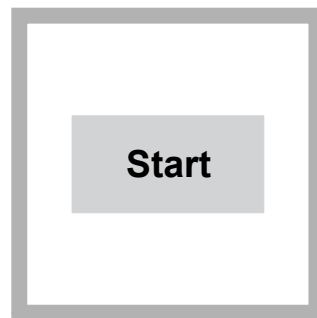
5. Tighten the cap on the vial and invert the vial 2–3 times.



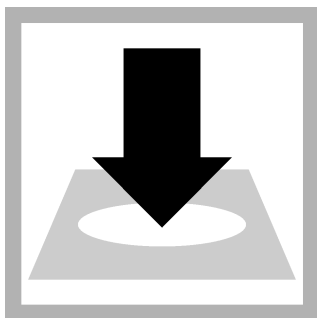
6. Start the reaction time of 1 minute.



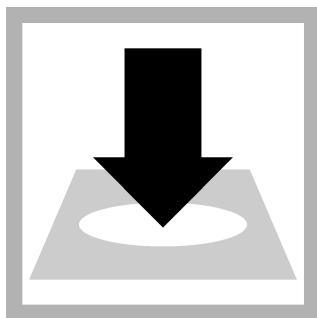
7. Clean the vial.



8. DR 1900 only: Select program 868. Refer to [Before starting](#) on page 1.



9. Insert the Zero vial into the cell holder. DR 1900 only: Push **ZERO**. The instrument zero is set.



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

## Sample volumes

Table 3 Sample volumes and ranges

Range	Volume
5 to 40 mg/L	2.0 mL
20 to 150 mg/L	0.4 mL

## Interferences

Table 4 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.

- Larger quantities of cobalt, iron(III), chromium(III) and sulfide interfere (high-bias results).
- Higher volume percentages of water-soluble organic solvents interfere (high-bias results or low-bias results with different phenols types).
- High concentrations of strong oxidizing and reducing agents in the sample interfere with the reaction process. Remove strong oxidizing and reducing agents from the sample before analysis.
- Other substances that combine with 4-aminoantipyrine (e.g., naphthols and aromatic amines) are also analyzed, which results in a higher phenol concentration.
- Verify the measurement results with sample dilutions or standard additions.

**Table 4 Non-interfering substances**

Substance	Test concentration	Substance	Test concentration
Cl <sup>-</sup>	1700 mg/L	Sn <sup>2+</sup>	50 mg/L
Na <sup>+</sup>	1500 mg/L	Fe <sup>2+</sup>	50 mg/L
K <sup>+</sup>	1500 mg/L	Zn <sup>2+</sup>	50 mg/L
NH <sub>4</sub> <sup>+</sup>	1500 mg/L	Hg <sup>2+</sup>	50 mg/L
Ca <sup>2+</sup>	1500 mg/L	Cd <sup>2+</sup>	50 mg/L
Cu <sup>2+</sup>	300 mg/L	Ni <sup>2+</sup>	50 mg/L
SO <sub>4</sub> <sup>2-</sup>	200 mg/L	Ag <sup>+</sup>	50 mg/L
NO <sub>3</sub> <sup>-</sup>	100 mg/L	Co <sup>2+</sup>	20 mg/L
SO <sub>3</sub> <sup>2-</sup>	50 mg/L	Fe <sup>3+</sup>	10 mg/L
NO <sub>2</sub> <sup>-</sup>	50 mg/L	S <sup>2-</sup>	10 mg/L
CN <sup>-</sup>	50 mg/L	CH <sub>2</sub> O	10 mg/L
I <sup>-</sup>	50 mg/L	Cr <sup>3+</sup>	2 mg/L
CH <sub>3</sub> COO <sup>-</sup>	50 mg/L	H <sub>2</sub> O <sub>2</sub>	1 mg/L
Al <sup>3+</sup>	50 mg/L	CH <sub>3</sub> OH	5 volume %
Pb <sup>2+</sup>	50 mg/L	C <sub>2</sub> H <sub>5</sub> OH	5 volume %
Mn <sup>2+</sup>	50 mg/L	(CH <sub>3</sub> ) <sub>2</sub> CO	5 volume %
Cr <sup>6+</sup>	50 mg/L		

## Accuracy check

### Standard solution method

Items to collect:

- Phenol, ACS
- 1000-mL volumetric flasks (2), Class A
- 500-mL volumetric flask, Class A
- 10-mL volumetric pipet, Class A, with safety bulb
- Deionized water

1. Prepare a 1000-mg/L phenol stock solution as follows:
  - a. Weigh 1.000 g of phenol.
  - b. Add the phenol to a 1000-mL volumetric flask.

- c. Dilute to the mark with freshly boiled and cooled deionized water. Mix well to dissolve.
2. Prepare a 10-mg/L working phenol standard solution as follows:
  - a. Use a pipet to add 10 mL of the stock phenol solution to a 1000-mL volumetric flask.
  - b. Dilute to the mark with deionized water. Mix well.
3. Prepare a 0.200-mg/L standard solution as follows:
  - a. Use a pipet to add 10 mL of the 10-mg/L working solution to a 500-mL volumetric flask.
  - b. Dilute to the mark with deionized water. Mix well.
4. Use the test procedure to measure the concentration of the prepared standard solution.
5. Compare the expected result to the actual result.

## Summary of Method

When an oxidizing agent is in the sample, ortho- and meta-substituted phenols form colored complexes with 4-aminoantipyrine (AAP). The measurement wavelength is 510 nm.

## Consumables and replacement items

### Required reagents

Description	Quantity/Test	Unit	Item no.
Phenol TNTplus Reagent Set	1	25/pkg	TNT868

### 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
Pipet, adjustable volume, 0.2–1.0 mL	1	each	BBP078
Pipet tips, for 0.2–1.0 mL pipet	2	100/pkg	BBP079
Light shield, DR 3900	1	each	LZV849
Light shield, DR 3800, DR 2800, DR 2700	1	each	LZV646

### Micro Dist apparatus

Description	Quantity/test	Unit	Item no.
Micro Dist <sup>®</sup> distillation block, 100 VAC	1	each	A17102
OR			
DRB200 Reactor Block	1	each	DRB20004
DRB200 adapter sleeves, Micro Dist	1	8/pkg	LZT144
Micro Dist <sup>®</sup> tubes, Phenolics-1, pre-filled	varies	21/pkg	A17002
Micro Dist <sup>®</sup> tubes, user-fill	varies	50/pkg	A17517
Micro Dist <sup>®</sup> tubes, user-fill	varies	100/pkg	A17117

## Recommended standards

Description	Unit	Item no.
Phenol, ACS	113 g	75814

## Optional reagents and apparatus

Description	Unit	Item no.
Sulfuric Acid Standard Solution, 19.2 N	100 mL	203832
Sulfuric Acid Standard Solution, 19.2 N	500 mL	203849
Paper, pH, 0–14 pH range	100/pkg	2601300
Thermometer, non-mercury, –10 to +225 °C	each	2635700
Flask, volumetric, Class A, 500 mL, glass	each	1457449
Flask, volumetric, Class A, 1000 mL glass	each	1457453



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