Lead

DOC316.53.01056

PAR Method **Method 10216**

0.1 to 2.0 mg/L Pb **TNTplus™ 850**

Scope and Application: For wastewater and process control

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 207 Instrument-specific information

Instrument	Light shield
DR 6000	—
DR 5000	—
DR 3900	LZV849
DR 3800, DR 2800	LZV646

Before starting the test:

Install the light shield if applicable (see *Instrument-specific information*).
Please read Safety Advice and Expiration Date on package.
Recommended sample pH is 3–9.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F).
Recommended reagent storage is 15–25 °C (59–77 °F).
Samples which are free from complexing agents and have a pH between 3 and 6 can be analyzed directly.
Samples with a pH between 6 and 9 must be additionally digested with Metals Prep Set TNT 890 in order to bring undissolved lead hydroxide or complex lead compounds into solution.
TNTplus methods are activated directly from the Main Menu when the sample vial is inserted into the sample cell holder.

Collect the following items :

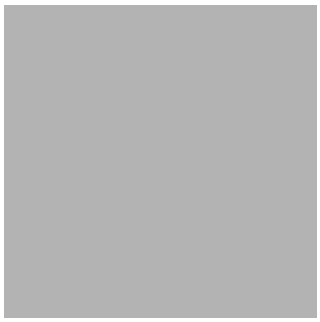
Description	Quantity
Lead TNT850 Reagent Set	1
Light Shield (see *Instrument-specific information*)	1
Pipet variable volume, 1–5 mL	1
Pipet tips for variable volume pipet	1
Pipet, volumetric 10 mL	1

Collect the following items (continued):

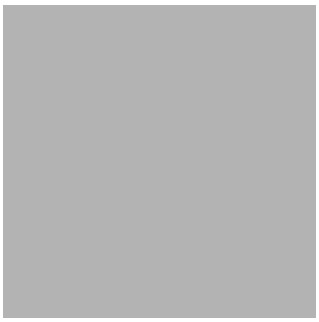
Description	Quantity
Safety pipet bulb	1
Pipet, variable volume, 0.2–1.0 mL	1
Pipet tips for variable volume pipet	1

See *Consumables and replacement items* for reorder information.

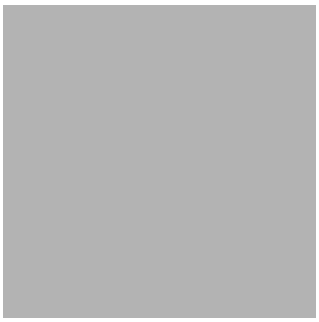
TNTplus 850



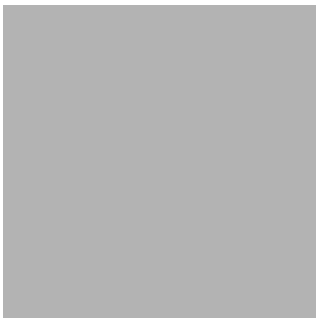
1. Pipet 10 mL of sample into the 20 mm reaction tube.



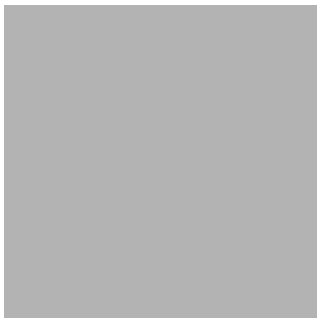
2. Add 1 level spoonful of Reagent A to the reaction tube.



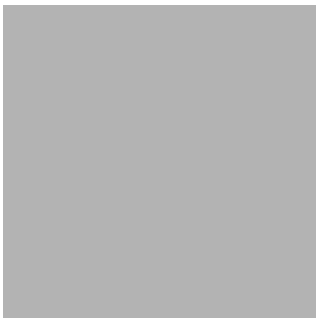
3. Cap the reaction tube and invert 2–3 times.



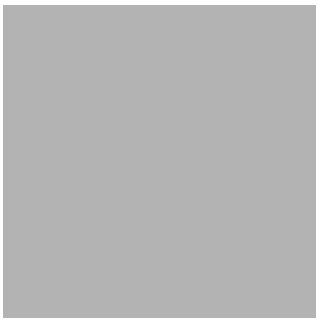
4. Wait two minutes.



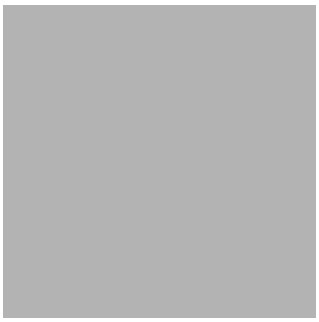
5. When the timer expires, add 1.5 mL of Solution B into a sample vial.



6. Pipet 4.0 mL of the pretreated sample from the 20 mm reaction tube prepared in step 3 into the vial.



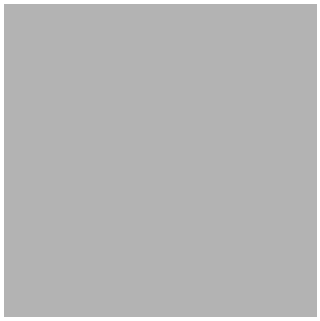
7. Cap and invert the vial 2–3 times.



8. Wait two minutes.

DR 2800: Install the Light Shield in Cell Compartment #2.

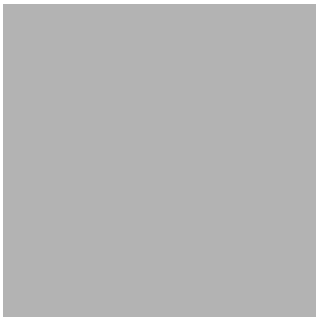
TNTplus 850 (continued)



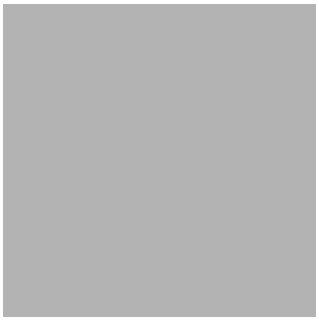
9. Thoroughly clean the outside of the vial and insert it into the sample cell holder. The instrument reads the barcode, then selects the method and sets the blank.

The instrument displays E1 when zeroing is complete.

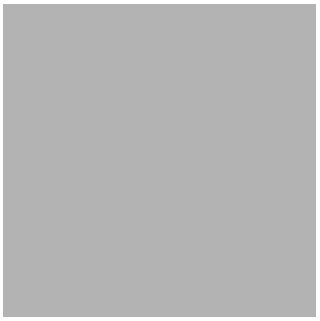
Note: *When analyzing a sample that has been digested with the Metals Prep Set (TNT 890), change the chemical form from the program options.*



10. Remove the vial and add 0.3 mL (300 µL) of Solution C into the vial.



11. Cap the vial and invert the vial 2–3 times



12. Wait one minute, then insert the prepared vial into the cell holder. The instrument reads the barcode, then reads the sample.

Results are in mg/L Pb.

Reagent blank

A reagent blank can be measured and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 12. Press **OPTIONS>MORE>REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Interferences

The ions listed in the *Interfering substances* table have been individually checked up to the given concentrations and do not cause interference. Cumulative effects and the influence of other ions have not been determined.

Measurement results can be verified using sample dilutions or standard additions.

Table 208 Interfering substances

Interfering substance	Interference level
K^+, Na^+, Ca^{2+}, Mg^{2+}, NO_3^-, Cl^-, PO_4^{3-}, CO_3^{2-}, SO_4^{2-}	500 mg/L
F^-, NH_4^+, Sr^{2+}	50 mg/L
Ag^+, Cd^{2+}, Cr^{6+}, Zn^{2+}, Cu^{2+}, Co^{2+}, Ni^{2+}	25 mg/L
Cr^{3+}, Al^{3+}, Fe^{2+}, Fe^{3+}	10 mg/L
Mn^{2+}, Hg^{2+}	5 mg/L
Sn^{2+}	0.5 mg/L

Sample collection, preservation and storage

- Collect samples in acid-washed glass or plastic containers.
- Adjust the pH to 2 or less with nitric acid (about 2 mL per liter).
- Store preserved samples up to six months at room temperature.
- Adjust the pH to between 3 and 6 with 5.0 N sodium hydroxide before analysis.
- Correct the test result for volume additions.

Accuracy check

Standard solution method
Note: *Refer to the instrument user manual for specific software navigation instructions.*

Required for accuracy check:

- 100 mg/L lead standard solution
- 100 mL volumetric flask
- Volumetric pipet

1. Prepare a 1.0 mg/L lead standard solution as follows:
   a. Pipet 1.0 mL of 100 mg/L lead standard solution into a 100 mL volumetric flask.
   b. Dilute to the mark with deionized water. Mix well.
2. Use 10 mL of this solution in place of the sample. Follow the *TNTplus 850* test procedure.

Summary of method

Lead (II) ions react at pH 9 with 4-(2-pyridylazo)-resorcinol (PAR) to form a red complex. Test results are measured at 520 nm.

Consumables and replacement items

Required reagents

Description	Quantity	Unit	Catalog number
Lead TNT 850 Reagent Set	1	25/pkg	TNT850

Required apparatus

Description	Quantity	Unit	Catalog number
Light Shield (DR 2800 only)	1	each	LZV646
Pipet, variable volume, 1-5 mL	1	each	BBP065
Pipet Tips, for BBP065 pipet	1	75/pkg	BBP068
Pipet, Volumetric 10 mL	1	each	1451538
Pipet Filler, safety bulb	1	each	1465100
Pipet, variable volume, 0.2–1.0 mL	1	each	BBP078
Pipet Tips, for BBP078 pipet	1	100/pkg	BBP079

Recommended standards

Description	Unit	Catalog number
Lead Standard Solution, 100 mg/L	100 mL	1261742
Nitric Acid, ACS	500 mL	15249
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Sodium Hydroxide Standard Solution, 5.0 N	59 mL SCDB	245026
Water, deionized	4 L	27256

Optional reagents and apparatus

Description	Unit	Catalog number
Metals Prep Set, TNTplus	50 digestions	TNT890
DRB200 digital reactor block, 9 x 16 mm vial wells, 2 x 20 mm vial wells, 115 VAC	each	LTV082.53.30001
DRB200 digital reactor block, 9 x 16 mm vial wells, 2 x 20 mm vial wells, 230 VAC	each	LTV082.52.30001
Reducing adapters for DRB200 reactor block, 16 mm to 13 mm vial wells	5/pkg	2895805

Edition 7