Lead

DOC316.53.01459

LeadTrak™ Fast Column Extraction Method

Method 8317

5 to 150 µg/L Pb

Scope and application: For drinking water.

Test preparation

Before starting

Clean all glassware with 1:1 nitric acid, then rinse with deionized water to remove contaminants. Refer to Prepare the apparatus and the sample on page 2.
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.
Refer to Sample collection on page 2 for the sampling requirements for “first-draw” analysis.
Reagents cause stains on the sample cells. Rinse the sample cells with 1:1 nitric acid or pPb-1 Acid Preservative Reagent and then deionized water.
The Extractor plunger may be used for more than one test. Rinse the Extractor plunger with water that contains no lead between uses.
Always do tests in sample cells. Do not put the instrument in the sample or pour the sample into the cell holder.
Make sure that the sample cells are clean and there are no scratches where the light passes through them.
Rinse the sample cell and cap with the sample three times before the sample cell is filled.
Make sure that there are no fingerprints or liquid on the external surface of the sample cells. Wipe with a lint-free cloth before measurement.
Cold waters can cause condensation on the sample cell or bubbles in the sample cell during color development. Examine the sample cell for condensation or bubbles. Remove condensation with a lint-free cloth. Invert the sample cell to remove bubbles.
Install the instrument cap over the cell holder before ZERO or READ is pushed.
After the test, immediately empty and rinse the sample cell. Rinse the sample cell and cap three times with deionized water.
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
LeadTrak™ Reagent Set	1
Beaker, polypropylene, 250 mL	1
Cylinder, graduated polypropylene, 25 mL	1
Cylinder, graduated polypropylene, 100 mL	1
Dropper, 0.5 and 1.0 mL marks	1
Sample cell, 25-mm (10 mL)	1
Water, deionized	varies

Refer to Consumables and replacement items on page 7 for order information.

Sample collection

- Collect samples from household pipes (point-of-use) or from water sources.
- Each sample type typically requires different sampling procedures. Consult with the applicable regulatory agency for more information about specific sampling requirements.

Collect samples from household sources

If sampling for lead contamination in household pipes for point-of-use drinking water, complete the steps that follow:

1. Make sure that the sampling pipes have had no flow for a minimum of 6 hours.
2. Add 10 mL of pPb-1 Acid Preservative to a 1-L bottle.
3. Open the sampling tap and collect exactly the first liter of water into the bottle that contains the acid preservative.
4. Put the cap on the sample. Invert several times to mix.
5. After 2 minutes, the sample is ready for analysis. Do not do steps 3 to 5 in the test procedure. Use 100 mL of this preserved sample directly in step 6.

Collect samples from other sources

If sampling for lead contamination from drinking water sources (e.g., well water, water from main supply lines, etc.), complete the steps that follow:

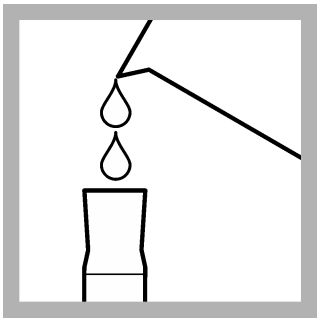
1. Add 10 mL of pPb-1 Acid Preservative to a 1-L bottle.
2. Let the tap water flow for 3–5 minutes or until the water temperature is stable for 3 minutes.
3. Collect exactly 1 liter of water into the bottle that contains the acid preservative.
4. Put the cap on the sample. Invert several times to mix.
5. After 2 minutes, the sample is ready for analysis. Do not do steps 3 to 5 in the test procedure. Use 100 mL of this preserved sample directly in step 6.
6. For a representative sample, collect at least 1 L of the sample. If less than 1 L is collected, use 1 mL of pPb-1 Acid Preservative per 100 mL of sample.
7. If nitric acid is substituted for pPb-1 as a preservative or if the sample is digested, the buffering capacity of the pPb-2 Fixer Solution can be exceeded. Adjust the sample pH to 6.7–7.1 pH with 5 N Sodium Hydroxide after step 7.

Prepare the apparatus and the sample

Since lead is very common in the environment, it is necessary to be careful to prevent sample contamination. For the most accurate test results, use the information that follows to prepare the apparatus and the sample:

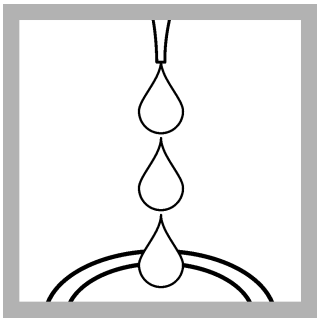
- To rinse an apparatus or dilute a sample, it is necessary to use lead-free water. Use distilled or deionized water. If the water is from a commercial source, review the label to make sure that the lead concentration is zero. If the lead concentration is unknown, determine the lead concentration with the test procedure.
- Plastic or glass sample containers and lids can be checked for contamination. Rinse the containers and lids with 1 mL of pPb-1 Acid Preservative Reagent. Add 100-mL of lead-free water to the cleaned container. Analyze this solution with the test procedure after 24 hours.
- To rinse glassware, use a small amount of dilute lead-free Nitric Acid or pPb-1 Acid Preservative Reagent. Then, rinse with lead-free water.
- Rinse the pPb-5 Indicator from the glass sample cells with a few drops of pPb-1 Acid Preservative Reagent or a small amount of dilute lead-free Nitric Acid.
- Acidify solutions that contain lead with Nitric Acid or pPb-1 Acid Preservative Reagent to below pH 2 to prevent adsorption of lead on the container walls. Refer to Sample collection on page 2.

LeadTrak fast column extraction



1. Fill a plastic graduated cylinder with 100 mL of the sample.

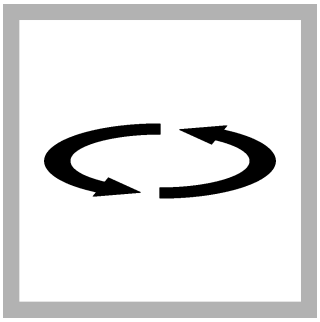
2.
Pour the measured sample into the supplied 125-mL plastic sampling bottle.



3. With a plastic 1 mL dropper, add 1.0 mL of pPb-1 Acid Preservative Solution to the sample.

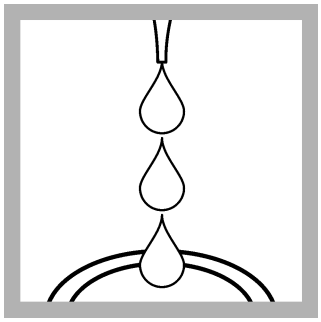
Note: *If the sample was preserved with pPb-1 Acid Preservative at a ratio of 1.0 mL per mL sample, then do not do steps* 3–6.

Note: *Samples preserved with Nitric Acid require steps* 3–6.

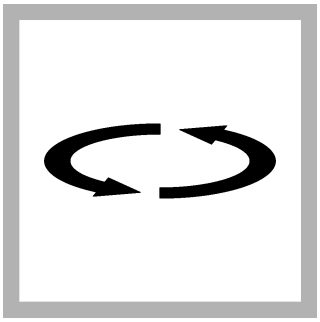


4. Swirl to mix.

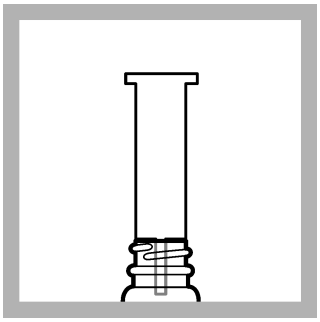
5. Set and start a timer for 2 minutes. A 2-minute reaction time starts.



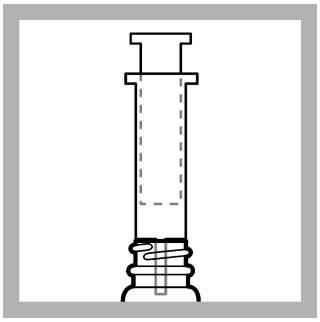
6. When the timer expires, use a second 1-mL plastic dropper to add 2.0 mL of pPb-2 Fixer Solution.



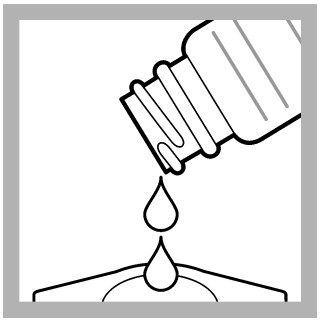
7. Swirl to mix.
Field samples that use nitric acid for preservation or for digested field samples can be more acidic than the buffer capacity of the Fixer Solution. After this step, measure the pH. Make necessary adjustments to the sample with 5 N Sodium Hydroxide to be pH 6.7–7.1. Do not do the next step until the pH is adjusted.



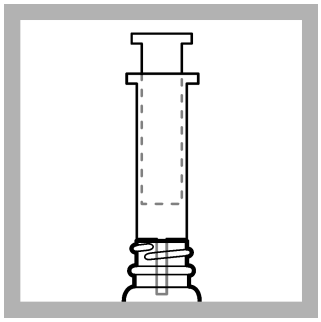
8. Put a new Fast Column Extractor on top of the second 125-mL sampling bottle. A new extractor is necessary for each test procedure.



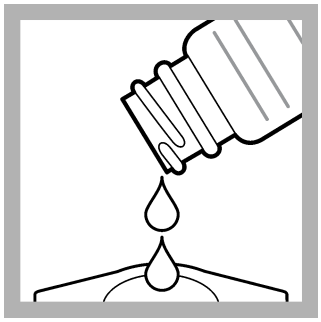
9. Soak the cotton plug with deionized water, then compress it with the plunger. Make sure that the cotton plug fits snugly against the inner wall of the column. Remove the plunger.
If the cotton plug moves up the column, push it back to the bottom with a clean, blunt rod.



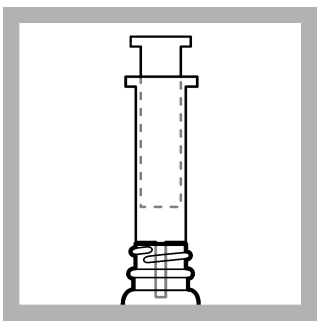
10. Slowly pour the prepared sample into the center of the Column Extractor. Wait for the sample to flow through.
It is expected that the sample solution flows slowly (2 drops per second) through the column. Keep the level of the sample solution just above the cotton plug.



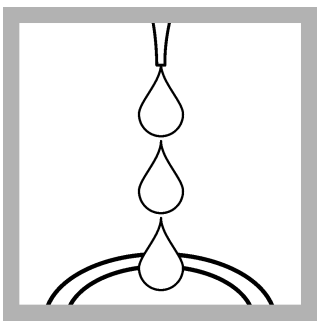
11. After the flow stops completely, fully compress the absorbent pad in the Extractor with the plunger. Safely discard the contents of the bottle.
Make sure that the absorbent pad stays at the bottom of the Extractor when the plunger is removed. If the cotton plug moves up the column, push it back to the bottom with a clean, blunt rod.



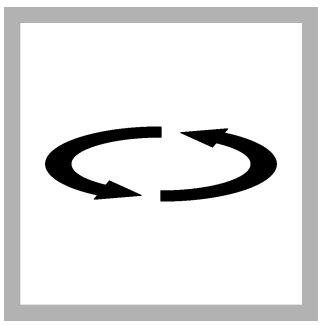
12. Put the extractor on top of the round poly mixing bottle. Measure 25 mL of pPb-3 Eluant Solution with the measuring vial and pour into the extractor. Keep the level of the eluent solution just above the absorbent pad.



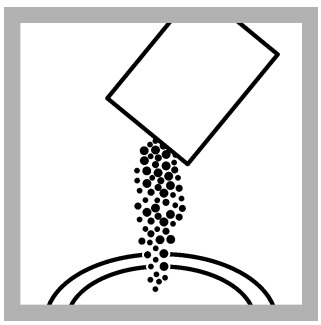
13. Let the Eluant Solution drip slowly from the Extractor. When the flow stops, fully compress the absorbent pad.



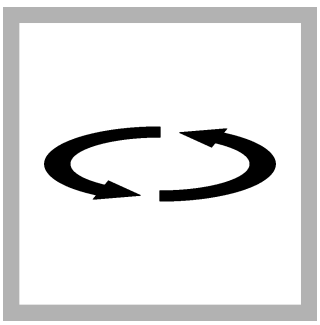
14. With a 1 mL plastic dropper, add 1.0 mL of pPb-4 Neutralizer Solution to the bottle.



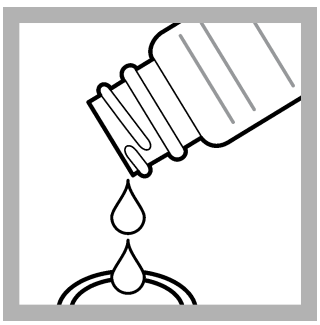
15. Swirl to mix.
Fully mix the solution, then immediately do the next step.



16. Add the contents of one pPb-5 Indicator Powder Pillow to the bottle.

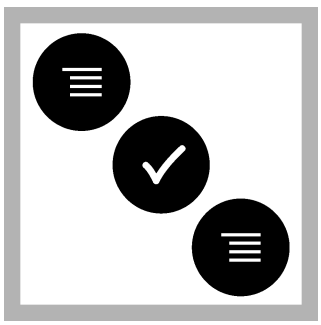


17. Swirl to fully mix. The solution color changes to brown.

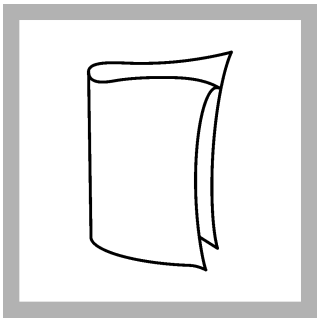


18. Fill a sample cell to the 10-mL mark with the solution.

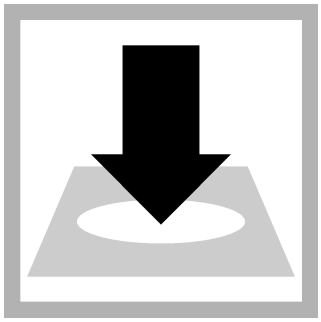
19. Set and start a timer for 2 minutes. A 2-minute reaction time starts.



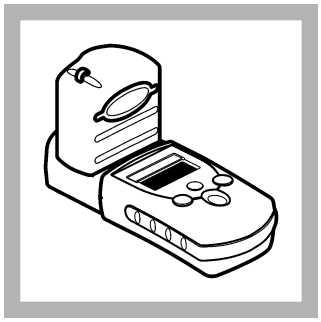
20. Set the instrument to µg/L Pb. Refer to the instrument documentation.



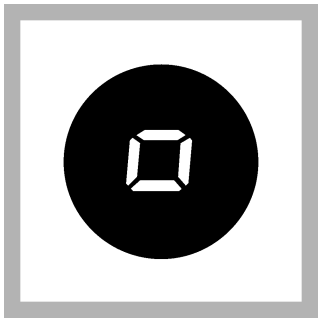
21. When the timer expires, clean the prepared sample cell.



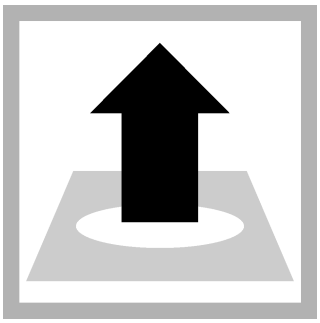
22. Insert the prepared sample into the cell holder. Point the diamond mark on the sample cell toward the keypad.



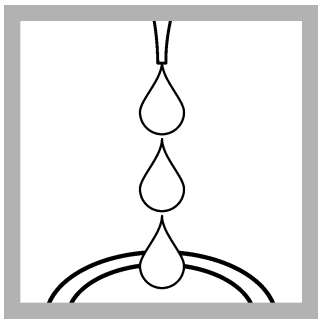
23. Install the instrument cap over the cell holder.



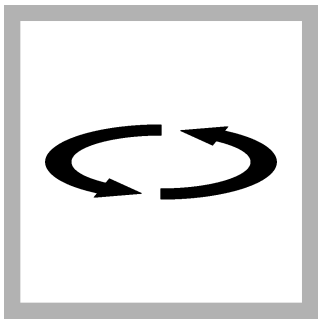
24. Push **ZERO**. The display shows “0”.



25. Remove the sample cell from the cell holder.

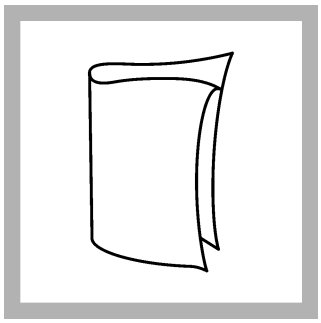


26. Add 3 drops of pPb-6 Decolorizer Solution to the sample cell.

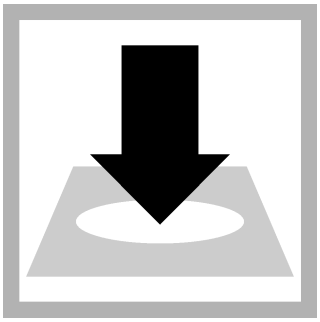


27. Vigorously swirl to mix.

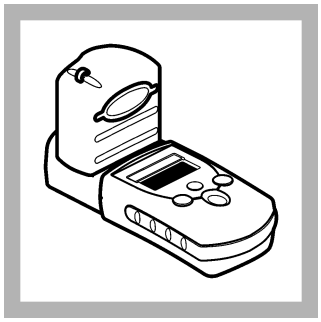
Note: *There will be little visual difference between the decolorized sample and the prepared sample.*



28. Clean the prepared sample cell.



29. Insert the prepared sample into the cell holder. Point the diamond mark on the sample cell toward the keypad.



30. Install the instrument cap over the cell holder.

31. Push READ. Results show in µg/L lead (Pb).

Interferences

Table 1 shows interference studies that were done by preparing a known lead solution of 25 µg/L as well as the potential interfering ion. The ion was said to interfere when the resulting lead concentration changed by ± 10%. Samples that contain levels more than these concentration values can be diluted 1:1 and analyzed. Multiply the value obtained by a factor of 2 to find the lead present in the original sample.

To prevent contamination, do not use black rubber stoppers, black dropper bulbs or droppers with inked graduations. Use the plastic droppers supplied in the reagent set.

Acid-wash all glassware and plasticware to prevent sample contamination, especially if the previous sample had a high lead concentration.

The extractor plunger can be reused for more than one test, but it should be rinsed with lead-free water between uses.

Table 1 Interfering substances

Interfering substance	Interference level
Aluminum, Al^{3+}	0.5 mg/L
Ammonium, NH_4^+	500 mg/L
Barium, Ba^{2+}	6 mg/L
Calcium, Ca^{2+}	500 mg/L
Chloride, Cl^-	1000 mg/L
Copper, Cu^{2+}	2 mg/L
Fluoride, F^-	10 mg/L
Iron, Fe^{2+}	2 mg/L
Magnesium, Mg^{2+}	500 mg/L
Manganese, Mn^{2+}	0.5 mg/L
Nitrate, NO_3^-	1000 mg/L
Sulfate, SO_4^{2-}	1000 mg/L
Zinc, Zn^{2+}	1 mg/L

Accuracy check

Standard additions method

Use the standard additions method to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- Lead Standard Solution, 10-mg/L (10,000-µg/L)
- Deionized water
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 100-mL portions of fresh sample. Mix well.
2. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
3. Compare the expected result to the actual result. Each 100 µL of standard added is expected to increase the lead concentration by 10 µg/L.

Standard solution method

Use one of the procedures that follow to prepare a 100-µg/L Lead Standard Solution. Refer to the instrument documentation for specific software navigation instructions.

Use the prepared 100-µg/L Lead Standard Solution instead of the sample in the test procedure.

If necessary, adjust the calibration curve with the reading from the standard solution. Set the standard adjust to on, then accept the concentration that shows. If an alternate concentration is used, enter the concentration and adjust the curve to that value.

Items to collect:

- 1000-mg/L Lead Standard Solution or 10-mg/L Lead Standard Solution as Pb

- Lead-free or deionized water
- 100-mL volumetric flask, Class A or 100-mL plastic volumetric flask
- 1.0-mL volumetric pipet, Class A
- TenSette pipet and pipet tips

1. Use a pipet to add 1.0 mL of Lead Standard, 1000-mg/L into a 100-mL volumetric flask.
2. Use a TenSette pipet to add 0.2 mL of concentrated nitric acid to the flask.
3. Dilute to the mark with deionized lead-free water.
4. Use a pipet to add 10.00 mL of the prepared solution into a 1-L plastic volumetric flask.
5. Use a pipet to add 2.0 mL of nitric acid to the flask.
6. Dilute to the mark with lead-free deionized water.
7. Prepare this solution immediately before use.

OR

1. With a TenSette Pipet, add 1.00 mL of 10-mg/L Lead Standard Solution into a 100-mL plastic volumetric flask.
2. Dilute to volume with lead-free deionized water.
3. Prepare the solution immediately before use.

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a Pocket Colorimeter II during ideal test conditions. Users can get different results under different test conditions.

Precision (95% confidence interval)
70 ± 10 µg/L Pb

Summary of method

Acid soluble lead, as Pb^{2+}, in a potable water sample is first concentrated on a Fast Column Extractor. Then, the lead is eluted from the Extractor and reacts with an indicator. A decolorizer is then added to break up the colored complex. The difference between the sample with color and the decolorized sample is directly related to the concentration of lead in the sample.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
LeadTrak™ Reagent Set	1	20/pkg	2375000

Required apparatus

Description	Quantity/test	Unit	Item no.
Bottle, sampling, 125-mL	1	6/pkg	2324333
Vial with 2-, 5-, 10-, 15-, 20, 25-mL marks	1	each	219300
Cylinder, graduated, polypropylene, 100-mL	1	each	108142
Cylinder, graduated, polypropylene, 25-mL	1	each	108140
Dropper, measuring, 0.5-mL and 1.0-mL plastic	2	20/pkg	2124720
Sample cell, 10-mL round, 25 mm x 60 mm	1	6/pkg	2427606

Recommended standards and apparatus

Description	Unit	Item no.
Flask, volumetric, polypropylene, 1000-mL	each	2099553
Flask, volumetric, polypropylene, 100-mL	each	2099542
Lead Standard Solution, 10-mg/L	25 mL	2374820
Lead Standard Solution, Voluette® Ampules, 50-mg/L as Pb2+, 10 mL	16/pkg	1426210
Lead Standard Solution, 1000-mg/L as Pb	100 mL	1279642
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	1000/pkg	2185628
Pipet, volumetric, Class A, 1.00-mL	each	1451535
Pipet filler, safety bulb	each	1465100
Pipet, volumetric, Class A, 10-mL	each	1451538
Water, deionized	4 L	27256

Optional reagents and apparatus

Description	Unit	Item no.
pPb-1 Acid Preservative Reagent	236 mL	2368531
pPb-2 Fixer Solution	43 mL	2368655
Nitric Acid, concentrated	500 mL	15249
Sodium Hydroxide Standard Solution, 5.0 N	1 L	245053
Beaker, 150-mL, polypropylene	each	108044
Beaker, 250-mL, polypropylene	each	108046

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