Lead

DOC316.53.01054

LeadTrak™ Fast Column Extraction Method

Method 8317

5 to 150 µg/L Pb

Scope and application: For drinking water.

Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows sample cell and orientation requirements for specific instruments.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information

Instrument	Sample cell orientation	Sample cell
DR6000 DR3800 DR2800 DR2700 DR1900	The fill line is to the right.	2495402 
DR5000 DR3900	The fill line is toward the user.	

Before starting

For more accurate results, determine the reagent blank value for each new lot of reagent. Do the test procedure and use deionized water instead of the sample. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.
Refer to Sample collection on page 2 for the sampling requirements for “first-draw” analysis.
Reagents stain the sample cells. Rinse the cells first with 1:1 nitric acid, and then rinse 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, 150 mL	2
Beaker, polypropylene, 250 mL	1
Clamp, 2-prong extension, with clamp holder	1
Cylinder, graduated polypropylene, 25 mL	1
Cylinder, graduated polypropylene, 100 mL	1
Dropper, 0.5 and 1.0 mL marks	1

Items to collect (continued)

Description	Quantity
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1
Support for ring stand	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.
- Keep the preserved samples at room temperature for a maximum of 6 months.
- 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:

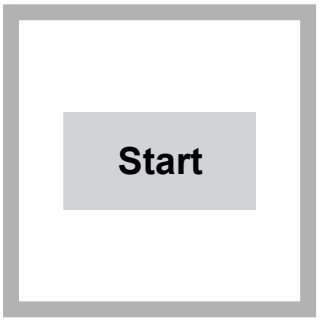
1. Make sure that the sampling pipes have had no flow for a minimum of 6 hours.
2. Add 10 mL of pPb-1 Acid Preservative to a 1-L bottle.
3. Open the sampling tap and collect exactly the first liter of water into the bottle that contains the acid preservative.
4. Put the cap on the sample. Invert several 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

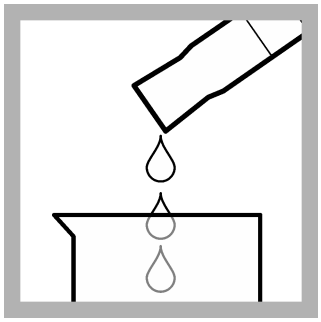
If sampling for lead contamination from drinking water sources (e.g., well water, water from main supply lines, etc.), complete the steps that follow:

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.

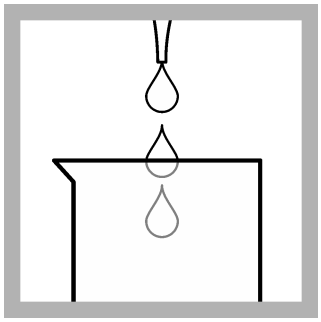
LeadTrak fast column extraction



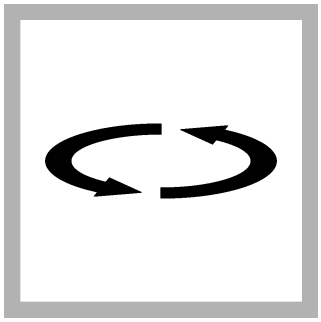
1. Start program **283 Lead, LeadTrak**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.



2. Fill a plastic graduated cylinder with 100 mL of the sample, then pour the measured sample into a clean 250-mL plastic beaker.
If the sample was preserved with pPb-1 Acid Preservative at a ratio of 1.0 mL per 100-mL sample, then ignore steps 3 and 4. Samples preserved with Nitric Acid require steps 3 and 4.

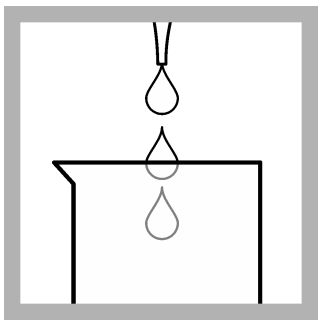


3. With a plastic 1 mL dropper, add 1.0 mL of pPb-1 Acid Preservative Solution to the sample.

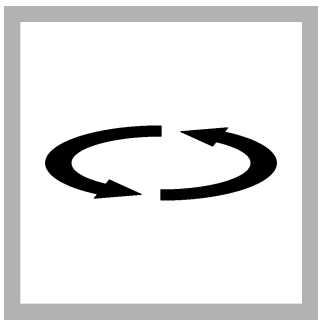


4. Swirl to mix.

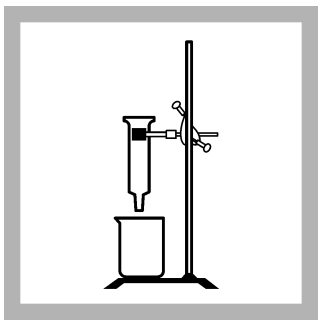
5. Start the instrument timer. A 2-minute reaction time starts.



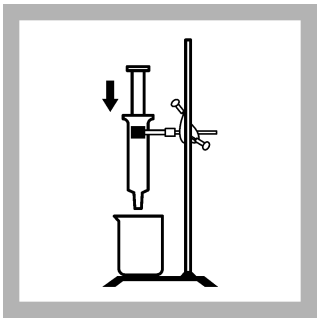
6. When the timer expires, use a second 1-mL plastic dropper to add 2.0 mL of pPb-2 Fixer Solution.



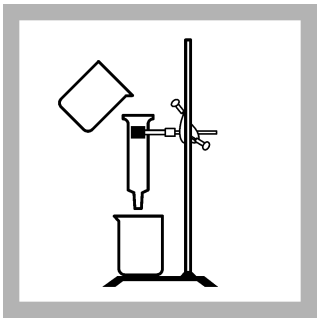
7. Swirl to mix.
Field samples that are preserved with nitric acid for preservation or that are digested can exceed the buffer capacity of the Fixer Solution. After this step, measure the pH. Before the next step, adjust the sample pH with 5 N Sodium Hydroxide to 6.7–7.1.



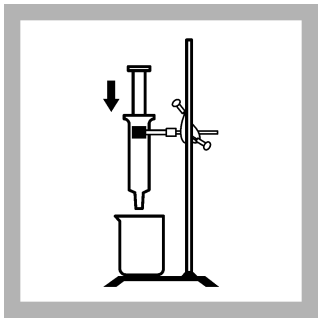
8. Install a new Fast Column Extractor in a ring stand with a clamp. Put a 150-mL plastic beaker under the extractor. The extractor is in the LeadTrak® Reagent Set . A new extractor is necessary for each test procedure.



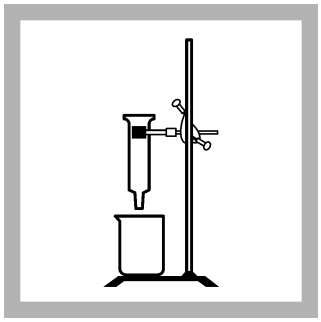
9. Soak the cotton plug in the extractor with deionized water, then compress it with the plunger. If the cotton plug moves up the column, push it back to the bottom with a clean, blunt rod. Make sure that the cotton plug fits snugly against the inner wall of the column. Remove the plunger.



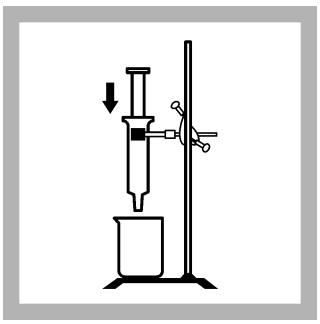
10. Slowly pour the prepared sample into the center of the extractor. Wait for the sample to flow through.
The sample solution must flow slowly (2 drops per second) through the extractor. Keep the level of the sample solution just above the cotton plug.



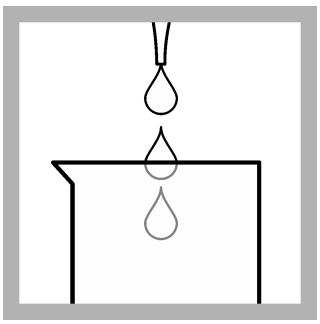
11. After the flow stops, fully compress the absorbent pad in the Extractor with the plunger. Safely discard the contents of the beaker. Slowly remove the plunger from the extractor.
Make sure that the absorbent pad stays at the bottom of the Extractor when the plunger is removed. If the cotton plug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



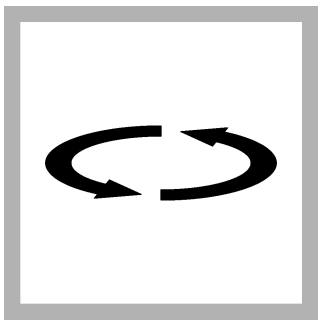
12. Put a clean, dry 150 mL beaker under the Extractor. Use a 25-mL plastic graduated cylinder to add 25-mL of pPb-3 Eluant Solution to the Extractor. Keep the level of the eluent solution just above the absorbent pad.



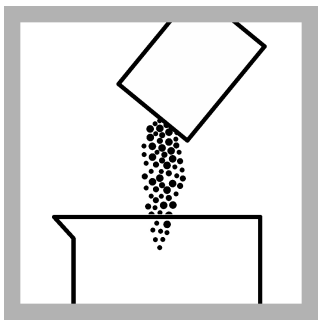
13. Let the Eluant Solution drip slowly from the extractor. When the flow stops, use the plunger to fully compress the absorbent pad.



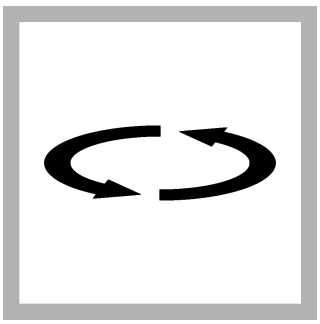
14. With a 1 mL plastic dropper, add 1.0 mL of pPb-4 Neutralizer Solution to the beaker.



15. Swirl to mix.
Fully mix the solution. Immediately continue to the next step.



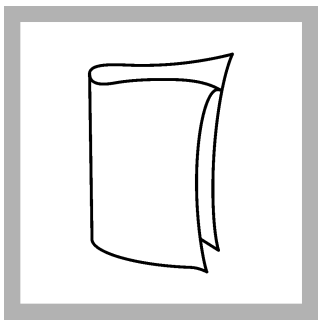
16. Add the contents of one pPb-5 Indicator Powder Pillow to the beaker.



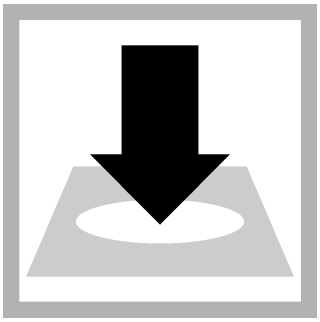
17. Swirl to fully mix. The solution becomes brown.

18. Pour 10 mL of the solution into a sample cell.

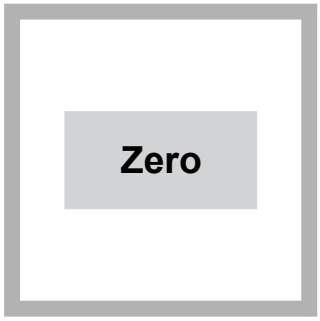
19. Start the instrument timer. The reaction time starts.



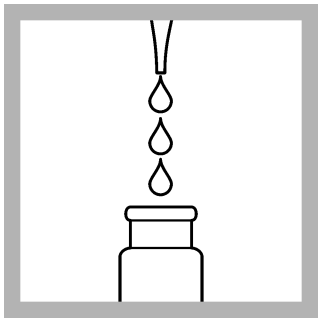
20. Clean the prepared sample cell.



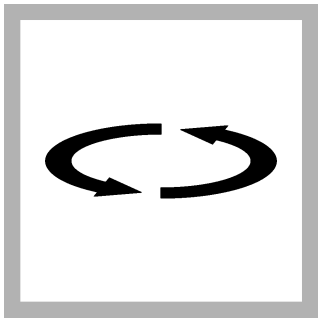
21. Insert the prepared sample into the cell holder.



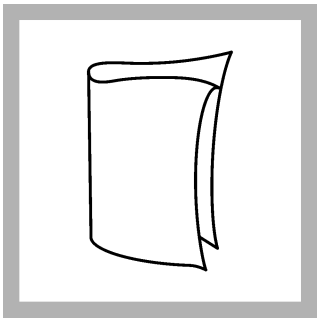
22. Push **ZERO**. The display shows 0 µg/L Pb.



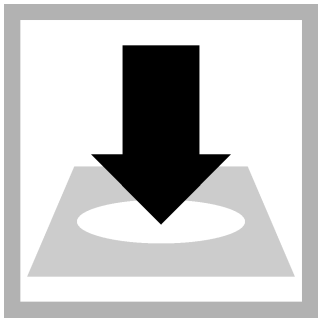
23. Remove the sample cell, then add 3 drops of pPb-6 Decolorizer Solution to the sample cell.



24. Vigorously swirl to mix.



25. Clean the prepared sample cell.



26. Insert the prepared sample into the cell holder.

27. Push **READ**. Results show in µg/L Pb.

Interferences

Table 2 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 2 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

Table 2 Interfering substances (continued)

Interfering substance	Interference level
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

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.

Accuracy check

Standard additions method (sample spike)

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

Items to collect:

- 10-mg/L (10,000-µg/L) Lead Standard Solution
- Deionized water
- Pipet, TenSette® with pipet tips

1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
2. Go to the Standard Additions option in the instrument menu.
3. Select the values for standard concentration, sample volume and spike volumes.
4. Open the standard solution.
5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3mL of the standard solution, respectively, to three 100-mL portions of fresh sample. Mix well.
6. 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.
7. Select **Graph** to compare the expected results to the actual results.

   Note: *If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.*

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 spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% confidence interval)	Sensitivity Concentration change per 0.010 Abs change
283	50 µg/L Pb^{2+}	45–55 µg/L Pb^{2+}	4 µg/L Pb^{2+}

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 determined colorimetrically with an indicator. The measurement wavelength is 477 nm.

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.
Beaker, 150-mL, polypropylene	2	each	108044
Beaker, 250-mL, polypropylene	1	each	108046
Clamp, 2-prong extension	1	each	2114500
Clamp holder	1	each	32600
Cylinder, graduated, polypropylene, 25-mL	1	each	108140
Cylinder, graduated, polypropylene, 100 mL	1	each	108142
Dropper, measuring, 0.5 mL and 1.0 mL, plastic	2	20/pkg	2124720
Sample cells, 10-mL square, matched pair	2	2/pkg	2495402
Support, Ring Stand, 5-inch x 8-inch base	1	each	56300

Recommended standards and apparatus

Description	Unit	Item no.
Flask, volumetric, polypropylene, 100 mL	each	2099542
Lead Standard Solution, 1000 mg/L as Pb	100 mL	1279642
Lead Standard Solution, 10 mg/L	25 mL	2374820
Nitric Acid, concentrated	500 mL	15249
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
Sodium Hydroxide Standard Solution, 5.0 N	1 L	245053

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