Lead

DOC316.53.01054

LeadTrak™ Fast Column Extraction Method

Method 8317

5 to 150 µg/L

Scope and Application: For drinking water

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 212 Instrument-specific information

Instrument	Sample cell	Cell orientation
DR 6000	2495402	Fill line faces right
DR 5000	2495402	Fill line faces user
DR 3900	2495402	Fill line faces user
DR 3800, DR 2800, DR 2700	2495402	Fill line faces right

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
The sampling requirements for “first-draw” analysis are detailed in *Sample collection, preservation and storage*.
Reagents will stain the sample cells, rinse the cells with 1:1 nitric acid, LeadTrak followed by deionized water.

Collect the following items:

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
Sample Cells (see *Instrument-specific information*)	1
Support for Ring Stand	1

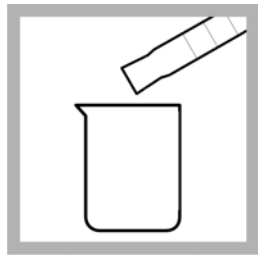
See *Consumables and replacement items* for reorder information.

LeadTrak Fast Column Extraction

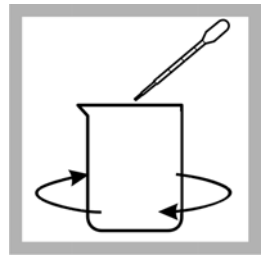
1. Select the test.

Insert an adapter if required (see *Instrument-specific information*).

Refer to the user manual for orientation.



2. Fill a 100 mL plastic graduated cylinder with 100 mL of the sample. Pour the measured sample into a 250 mL plastic beaker.



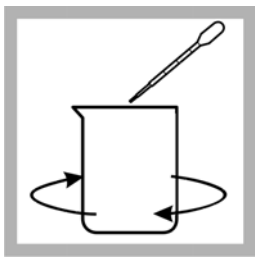
3. Using a plastic 1mL dropper, add 1.0 mL of pPb-1 Acid Preservative Solution to the sample and swirl to mix.

If the sample has been preserved previously with pPb-1 Acid Preservative at a ratio of 1.0 mL per 100 mL sample, omit steps 3 and 4.

Samples preserved with Nitric Acid require steps 3 and 4.

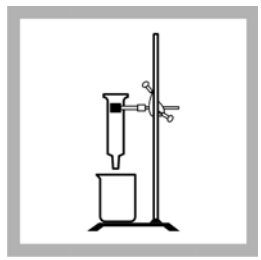
4. Start the instrument timer.

A two-minute reaction period will begin.



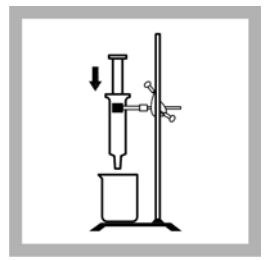
5. When the timer expires, use a second 1 mL plastic dropper to add 2.0 mL of pPb-2 Fixer Solution. Swirl to mix.

Field samples that have been preserved with nitric acid or samples that have been digested may exceed the buffer capacity of the Fixer Solution. After step 5, check the pH of these samples and adjust with 5 N Sodium Hydroxide to a pH of 6.7–7.1 before proceeding with step 6.



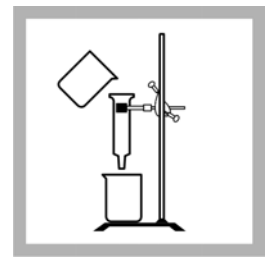
6. Mount a new Fast Column Extractor in a ring stand with a clamp. Place a 150-mL plastic beaker under the Extractor.

A Fast Column Extractor is included in the LeadTrak® Reagent Set. A new extractor is required for each test.



7. Soak the cotton plug with deionized water and compress it with the plunger. Remove the plunger. If the cotton plug moves up the column, push it back to the bottom with a clean, blunt rod.

The cotton plug should fit snugly against the inner wall of the column.

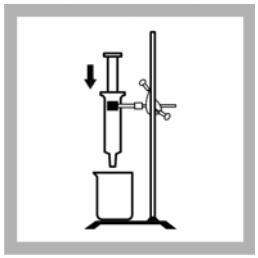


8. Pour the prepared sample slowly into the center of the Column Extractor. Wait for the sample to flow through.

The sample solution should flow relatively slowly (2 drops per second) through the column.

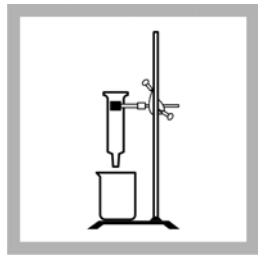
Keep the level of the sample solution just above the cotton plug.

LeadTrak Fast Column Extraction (continued)



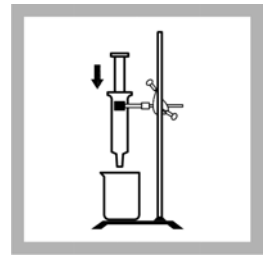
9. After the flow has stopped, fully compress the absorbent pad in the Extractor with the plunger. Discard the contents of the beaker. Slowly withdraw the plunger from the Extractor.

The absorbent pad should remain 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.



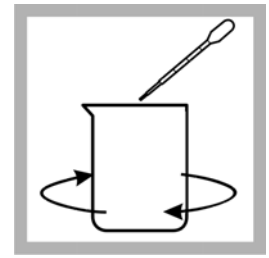
10. Place a clean, dry 150 mL beaker under the Extractor. Using a 25 mL plastic graduated cylinder, add 25 mL of pPb-3 Eluant Solution to the Extractor.

Keep the level of the eluent solution just above the absorbent pad.

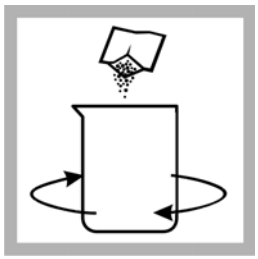


11. Allow the Eluant Solution to drip slowly from the Extractor.

After the flow has stopped, fully compress the absorbent pad.

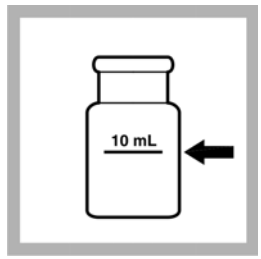


12. Using a 1 mL plastic dropper, add 1.0 mL of pPb-4 Neutralizer Solution to the beaker. Swirl thoroughly to mix and proceed immediately to step 13.



13. Add the contents of one pPb-5 Indicator Powder Pillow to the beaker and swirl thoroughly to mix.

The solution will turn brown.



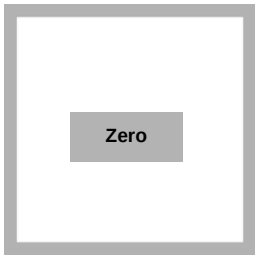
14. Pour 10 mL of solution into a sample cell.

15. Start the instrument timer.

A two-minute reaction period will begin.

16. When the timer expires, insert the sample cell.

LeadTrak Fast Column Extraction (continued)



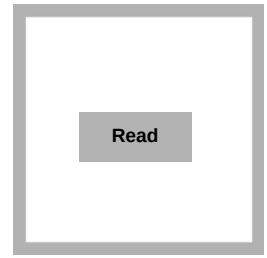
17. **ZERO** the instrument.

The display will show:

0 µg/L Pb

18. Remove the sample cell and add 3 drops of pPb-6 Decolorizer Solution to the cell. Swirl vigorously to mix

19. Insert the sample cell into the cell holder.



20. **READ** the results in µg/L Pb.

Interferences

Interference studies were conducted 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 containing levels exceeding these concentration values may be diluted 1:1 and analyzed. Multiply the value obtained by a factor of 2 to determine the lead present in the original sample.

To avoid contamination, do not use black rubber stoppers, black dropper bulbs and droppers with inked graduations. Use the plastic droppers provided in the reagent set.

Acid-wash all glassware and plasticware to prevent sample contamination, especially if the previous sample had a high lead level (see *Apparatus and sample preparation*).

The Extractor plunger may be reused for more than one test but should be rinsed with lead-free water between uses.

Table 213 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

Apparatus and sample preparation

Because lead is very common to our environment, care must be taken to prevent sample contamination. Follow these steps for greatest test accuracy:

- Lead-free water is necessary to minimize sample contamination when rinsing apparatus or diluting sample. The water may be either distilled or deionized. If the water is obtained from a grocery store, verify the lead concentration is zero from the label. If the lead concentration is uncertain, determine the lead concentration with the LeadTrak test.
- Plastic or glass sample containers and lids may be checked for contamination by rinsing with 1 mL of pPb-1 Acid Preservative Reagent*. Add 100 mL of lead-free water. After 24 hours, analyze this solution using the LeadTrak® test to confirm the absence of lead.
- Rinse glassware used in this test with a small amount of dilute lead-free 0.1 N nitric acid or pPb-1 Acid Preservative Reagent followed by rinsing with lead-free water.
- pPb-5 Indicator may be rinsed 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 containing lead with Nitric Acid or pPb-1 to below pH 2 to prevent adsorption of lead onto the container walls. See *Sample collection, preservation and storage*.

Sample collection, preservation and storage

- Samples may be collected either from household pipes (point-of-use) or from water sources.
- Preserved samples may be stored up to six months.
- Each sample type typically requires different sampling procedures. Consult with the appropriate regulatory agency for more information about specific sampling requirements.

Sampling for lead contamination in household pipes for point-of-use drinking water

- The sample should be collected after sitting in pipes with no flow for a minimum of six hours.
- Add 10 mL of pPb-1 Acid Preservative* to a one-liter bottle.
- Turn on tap and collect exactly the first liter of water in the bottle containing acid preservative.
- Cap and invert several times to mix.
- After two minutes the sample is ready for analysis. Steps 3 and 4 are skipped in the analysis procedure. Use 100 mL of this preserved sample directly in step 5.

Sampling for lead contamination from drinking water sources such as well water or water from main supply lines

- Add 10 mL of pPb-1 Acid Preservative* to a one-liter bottle.
- Turn on the tap for 3–5 minutes or until the water temperature has been stable for 3 minutes.
- Collect exactly one liter of water into the bottle containing the acid preservative.
- Cap and invert several times to mix.
- After two minutes the sample is ready for analysis. Steps 3 and 4 are skipped in the analysis procedure. Use 100 mL of this preserved sample directly in step 5.
- At least one liter should be collected to obtain a representative sample. If less than one liter is collected, use 1 mL of pPb-1 Acid Preservative per 100 mL of sample.
- If nitric acid is to be substituted for pPb-1 as a preservative or the sample is digested, the buffering capacity of the pPb-2 Fixer Solution* may be exceeded. Adjust the sample pH to 6.7–7.1 pH with 5 N Sodium Hydroxide* after step 6.

* See *Optional reagents and apparatus*.

Accuracy check

Standard additions method (sample spike)
Required for accuracy check:

- 10 mg/L (10,000 µg/L) Lead Standard Solution
- TenSette Pipet and pipet tips

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Select Options>More>Standard Additions from the instrument menu.
3. Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Open the standard solution.
5. Use the TenSette Pipet to prepare spiked samples: add 0.1 mL, 0.2 mL and 0.3 mL of standard to three 100 mL portions of fresh sample.
6. Follow the *LeadTrak Fast Column Extraction* test procedure for each of the spiked samples starting with the 0.1 mL sample spike. Measure each of the spiked samples in the instrument.
7. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

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

Required for accuracy check:

- Lead Standard Solution, 1000 mg/L or Lead Voluette® Ampule Standard Solution, 50-mg/L as Pb
- Lead-free water or Deionized water
- 100 mL Class A volumetric flask or 100-mL plastic volumetric flask
- 1.0 mL Class A volumetric pipet
- TenSette Pipet and Pipet Tips

1. Prepare a 100 µg/L Lead standard solution as follows:
   a. Pipet 1.0 mL of Lead Standard, 1000 mg/L, into a 100 mL volumetric flask.
   b. Use a Tensette Pipet to add 0.2 mL of concentrated nitric acid to the flask
   c. Dilute to the mark with lead-free deionized water.
   d. Pipet 10.00 mL of this prepared solution into a 1 liter plastic volumetric flask.
   e. Add 2.0 mL of nitric acid to the flask.
   f. Dilute to the mark with lead-free water.
   g. Prepare this solution immediately before use.

   OR

1. Prepare a 100 µg/L Lead Standard Solution as follows:
   a. Use a Tensette Pipet to add 0.2 mL from a Lead Voluette® Ampule Standard Solution, 50 mg/L as Pb into a 100 mL plastic volumetric flask.
   b. DIlute to volume with deionized water. Prepare solution immediately before use

2. Use the standard solution in place of the sample. Follow the *LeadTrak Fast Column Extraction* test procedure.
3. To adjust the calibration curve using the reading obtained with the 100-mg/L Standard Solution, select Options>More>Standard Adjust from the instrument menu.
4. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value.

Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution	Sensitivity Concentration change per 0.010 Abs change	
			Point of Curve	Concentration
283	50 µg/L Pb^{2+}	45–55 µg/L Pb^{2+}	Entire curve	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. The lead is then eluted from the Extractor and determined colorimetrically with an indicator. Test results are measured at 477 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
LeadTrak™ Reagent Set	1	20/pkg	2375000

Required apparatus

Description	Quantity	Unit	Catalog number
Beaker, polypropylene, 150 mL	2	each	108044
Beaker, polypropylene, 250 mL	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, 0.5 and 1.0 mL marks	1	20/pkg	2124720
Sample cell, 10 mL square, matched pair	2	2/pkg	2495402
Support for Ring Stand	1	each	56300

Recommended standards and apparatus

Description	Unit	Catalog number
Flask, volumetric, polypropylene, 1000 mL	each	2099553
Flask, volumetric, polypropylene, 100 mL	each	2099542
Lead Standard Solution, 1000 mg/L as Pb	100 mL	1279642
Lead Standard Solution, 50-mg/L 10 mL Voluette® Ampules	16/pkg	1426210
Lead Standard Solution, 10 mg/L	25 mL	2374820
Nitric Acid, ACS	500 mL	15249

Recommended standards and apparatus (continued)

Description	Unit	Catalog number
Pipet, TenSette®, 0.1 to 1.0 mL	each	1970001
Pipet Tips, for TenSette Pipet 1970001	50/pkg	2185696
Pipet Tips, for TenSette Pipet 1970001	1000/pkg	2185628
Pipet, volumetric, Class A, 1.00 mL	each	1451535
Pipet Filler, safety bulb	each	1465100
Pipet, volumetric, Class A, 10.00 mL	each	1451538
Water, deionized	4 L	27256

Optional reagents and apparatus

Description	Unit	Catalog number
pPb-1 Acid Preservative Reagent	236 mL	2368531
pPb-2 Fixer Solution	43 mL	2368655
Sodium Hydroxide, 5.0 N	1 L	245053

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Edition 7