Aluminum

DOC316.53.01441

Aluminon Method[1]

Method 8012

0.02 to 0.80 mg/L Al^{3+}

Powder Pillows

Scope and application: For water and wastewater.

[1] Adapted from Standard Methods for the Examination of Water and Wastewater.

Test preparation

Before starting

To make sure that all forms of the metal are measured, digest the sample with heat and acid. Use the mild or vigorous digestion. Refer to the *Water Analysis Guide* for more information.
Clean all glassware with 6.0 N (1:1) hydrochloric acid, then fully rinse with deionized water to remove contaminants.
The sample temperature must be 20–25 °C (68–77 °F) for accurate results.
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.
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.
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
AluVer® 3[1] Aluminum Reagent Powder Pillow	1
Ascorbic Acid Powder Pillow	1
Bleaching 3 Reagent Powder Pillow	1
Mixing cylinder, graduated, 50-mL, with glass stopper	1
Sample cells, 25-mm (10 mL)	2

Refer to Consumables and replacement items on page 6 for order information.

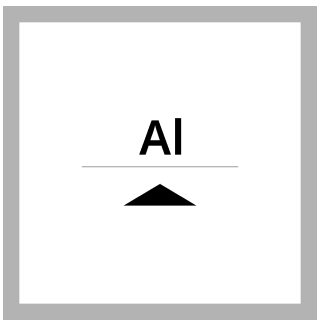
[1] AluVer is a registered trademark of Hach Company.

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Sample collection and storage

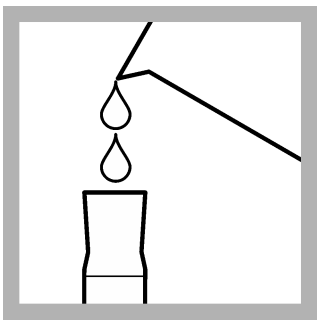
- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (approximately 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep the preserved samples at room temperature for a maximum of 6 months.
- Before analysis, adjust the pH to 3.5–4.5 with 5 N sodium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

Powder pillow procedure

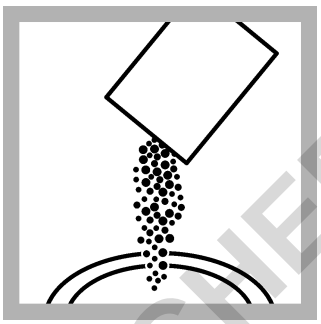


1. Set the instrument to aluminum (Al).

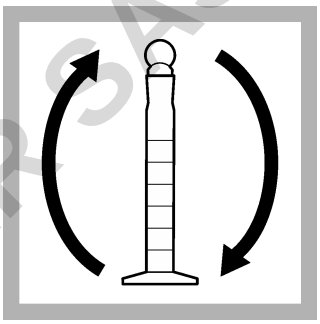
For DR300, push the up arrow button. For PCII, push the menu button, checkmark button, then the menu button again.



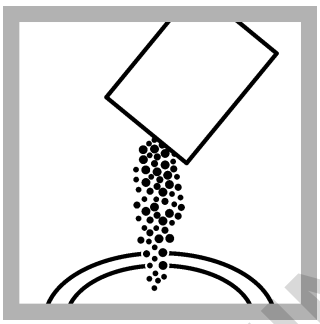
2. Fill a mixing cylinder to the 50-mL mark with sample.



3. Add the contents of one Ascorbic Acid Powder Pillow.



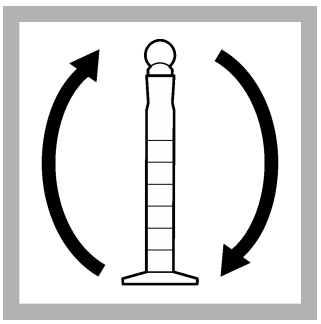
4. Put the stopper on the mixing cylinder. Invert the mixing cylinder several times to dissolve the powder.



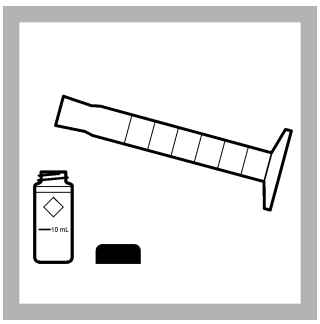
5. Add one AluVer 3 Aluminum Reagent Powder Pillow.
An orange to orange-red color will show if aluminum is in the sample.



6. Set and start a timer for 1 minute. A 1-minute reaction time starts.

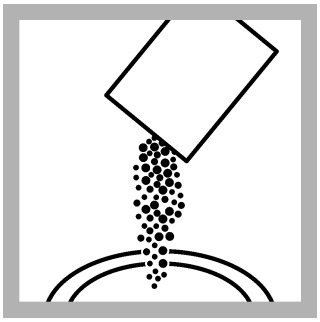


7. Invert the cylinder repeatedly during the reaction time. Undissolved powder will cause inconsistent results.



8. Prepare the blank: Fill a sample cell to the 10-mL mark with reacted sample.

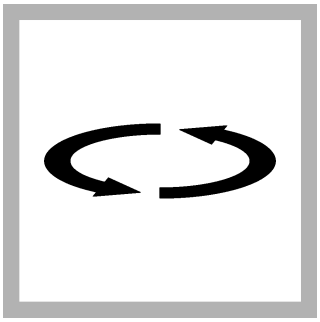
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9. Add one Bleaching 3 Reagent Powder Pillow to the blank.



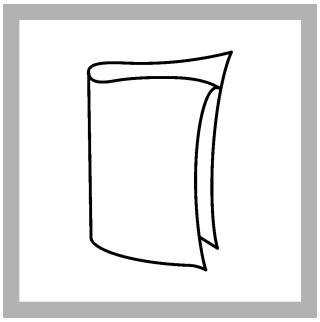
10. Set and start a timer for 30 seconds. A 30-second reaction time starts.



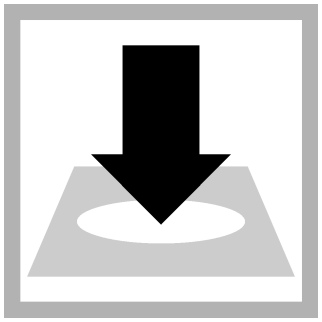
11. Swirl the sample cell vigorously. The solution will show a light to medium orange.



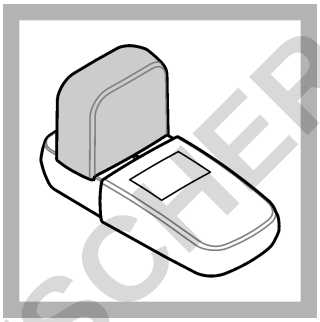
12. Set and start a timer for 15 minutes. A 15-minute reaction time starts.



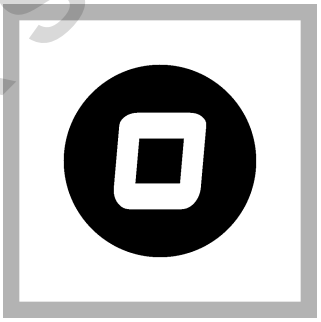
13. When the timer expires, clean the blank sample cell. Complete the remaining steps within 5 minutes.



14. Insert the blank into the cell holder. Point the diamond mark on the sample cell toward the keypad.



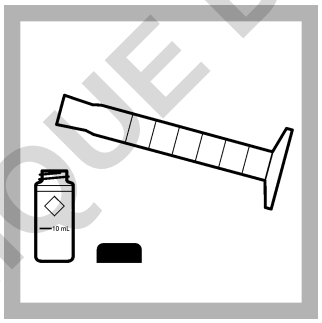
15. Install the instrument cap over the cell holder.



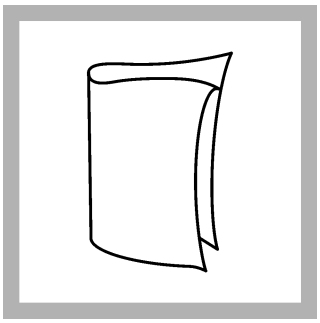
16. Push **ZERO**. The display shows “0.00”.



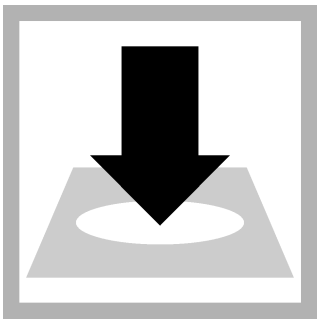
17. Remove the sample cell from the cell holder.



18. Prepare the sample: Fill a sample cell to the 10-mL mark with reacted sample from the cylinder.

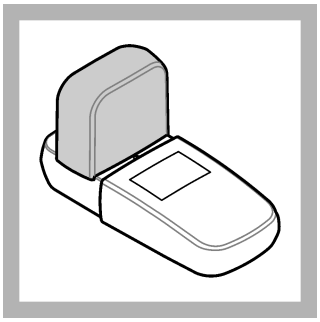


19. Clean the prepared sample cell.



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

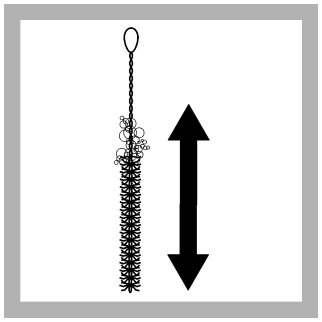
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21. Install the instrument cap over the cell holder.



22. Push **READ**. Results show in mg/L aluminum.



23. Immediately clean the graduated cylinder and sample cells with soapy water and a brush. Rinse with deionized water.

Interferences

Interfering substance	Interference level
Acidity	More than 300 mg/L as $CaCO_3$. Pre-treat samples that have more than 300 mg/L acidity as $CaCO_3$ as follows: **1.** Add 1 drop of m-Nitrophenol Indicator Solution to 50 mL of fresh sample. **2.** Add 1 drop of 5.0 N Sodium Hydroxide Standard Solution. Put the stopper on the cylinder. Invert to mix. Repeat as often as necessary until the color changes from colorless to yellow. **3.** Add 1 drop of 5.25 N Sulfuric Acid Standard Solution to change the solution from yellow to colorless. Use this treated sample in the test procedure.
Alkalinity	1000 mg/L as $CaCO_3$. Pre-treat samples that have higher alkalinity concentrations as follows: **1.** Add 1 drop of m-Nitrophenol Indicator Solution to 50 mL of fresh sample. A yellow color indicates excessive alkalinity. **2.** Add 1 drop of 5.25 N Sulfuric Acid Standard Solution. Put the stopper on the cylinder. Invert to mix. If the yellow color continues, repeat until the sample becomes colorless. Use this treated sample in the test procedure.
Fluoride	Interferes at all levels. Refer to Fluoride interference on page 4.
Iron	More than 20 mg/L
Phosphate	More than 50 mg/L
Polyphosphate	Polyphosphate interferes at all levels and causes negative errors and must not be present. The sample must be pre-treated with acid hydrolysis before the test is started to convert polyphosphate to orthophosphate. Use the Phosphorus, Acid Hydrolyzable Digestion procedure.

Fluoride interference

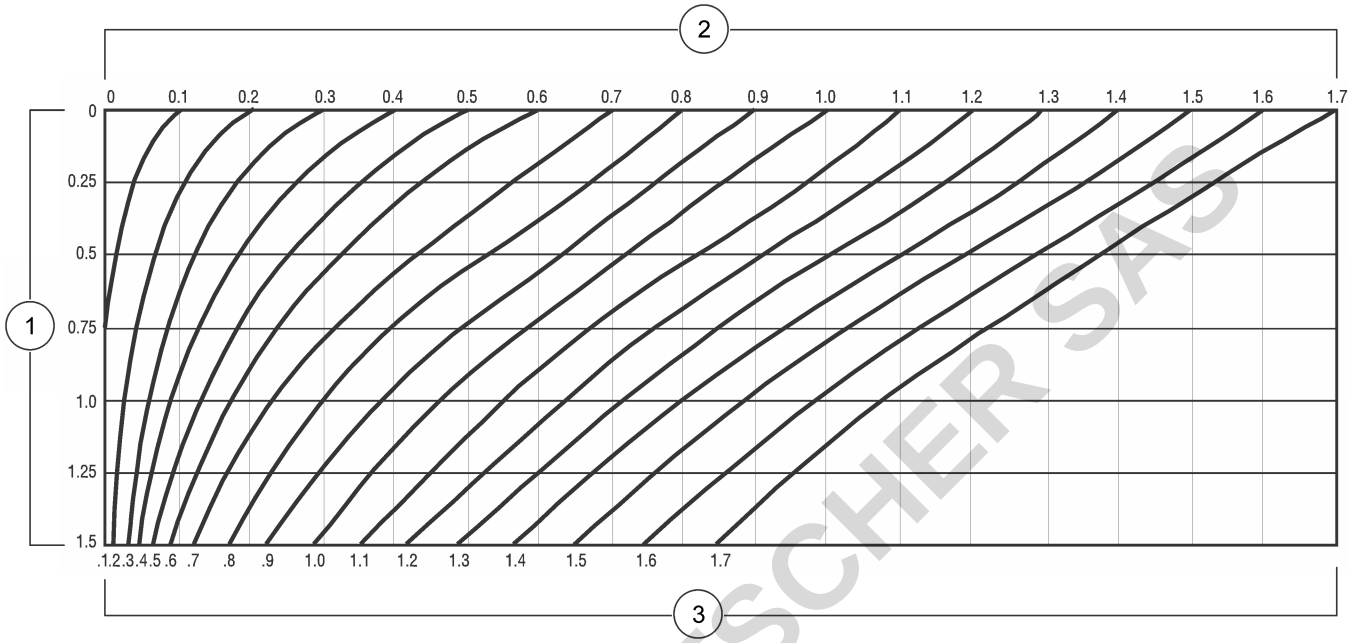
Fluoride forms complexes with aluminum and interferes at all levels. If the fluoride concentration of the sample is known, use Figure 1 to find the actual aluminum concentration.
To use the fluoride interference graph:

1. On the top of the graph, find the aluminum result from the test procedure.
2. On the left side of the graph, find the fluoride concentration of the sample.
3. Go along the grid lines to find the point on the graph where the values intersect.
4. Go along the curved lines on either side of the intersect point to find the actual aluminum concentration on the bottom of the graph.
   Example: The aluminum test result is 0.7 mg/L Al^{3+} and the fluoride concentration is 1 mg/L F^-. The 0.7 mg/L Al^{3+} grid line intersects with the 1 mg/L F^- grid line between

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the 1.2 and 1.3 mg/L Al^{3+} curves. In this example, the actual aluminum concentration is 1.27 mg/L.

Figure 1 Fluoride interference graph



1 y-axis: mg/L F^- in the sample	**2** x-axis, top: instrument result	**3** x-axis, bottom: actual mg/L Al^{3+}

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:

- 50-mg/L Aluminum Voluette® Ampule Standard
- Ampule breaker
- Pipet, TenSette®, 0.1–1.0 mL and tips
- Mixing cylinders (3)

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 50-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. The expected aluminum concentration increase is 0.1 mg/L for each 0.1 mL of standard that is added.

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- 100-mg/L Aluminum Standard Solution
- 250-mL volumetric flask, Class A
- 1.00-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water

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1. Prepare a 0.4 mg/L aluminum standard solution as follows:
   a. Use a pipet to add 1.00 mL of 100-mg/L aluminum standard solution into the volumetric flask. *(Alternate preparation: Use a pipet to add 0.8 mL of a 50-mg/L aluminum standard solution into a 100-mL volumetric flask.)*
   b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
2. Use the test procedure to measure the concentration of the prepared standard solution.
3. Compare the expected result to the actual result.

   Note: *The factory calibration can be adjusted slightly with the standard calibration adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are small variations in the reagents or instruments.*

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a DR300 and a Pocket Colorimeter II during ideal test conditions. Users can get different results under different test conditions.

Precision (95% confidence interval)
0.40 ± 0.02 mg/L Al

Summary of method

Aluminon indicator combines with aluminum in the sample to form a red-orange color. The intensity of color is proportional to the aluminum concentration. Ascorbic acid is added before the AluVer 3 reagent to remove iron interference. To establish a reagent blank, the sample is divided after the addition of the AluVer 3. Bleaching 3 Reagent is then added to one-half of the divided sample to bleach out the color of the aluminum aluminon complex. The AluVer 3 Aluminum Reagent, packaged in powder form, shows exceptional stability and is applicable for fresh water applications.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Aluminum Reagent Set, includes:	—	100 tests	2242000
AluVer® 3 Aluminum Reagent[2] Powder Pillow	1	100/pkg	1429099
Ascorbic Acid Powder Pillow	1	100/pkg	1457799
Bleaching 3 Reagent Powder Pillow	1	100/pkg	1429449

Required apparatus

Description	Quantity/Test	Unit	Item no.
Mixing cylinder, graduated, 50 mL, with glass stopper	1	each	189641
Sample cells, 10-mL round, 25 mm x 60 mm	2	6/pkg	2427606

Recommended standards and apparatus

Description	Unit	Item no.
Aluminum Standard Solution, 10-mL Voluette® Ampule, 50 mg/L as Al	16/pkg	1479210
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800

[2] AluVer is a registered trademark of Hach Company.

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Optional reagents and apparatus

Description	Unit	Item no.
Aluminum Standard Solution, 100-mg/L as Al^{3+}	100 mL	1417442
Aluminum Standard Solution, 10-mg/L as Al^{3+}	100 mL	2305842
Flask, volumetric, Class A, 100 mL, glass	each	1457442
Flask, volumetric, Class A, 250 mL	each	1457446
Hydrochloric Acid Solution, 6 N (1:1)	500 mL	88449
Liqui-Nox Phosphate-free detergent	946 mL	2088153
m-Nitrophenol Indicator Solution	100 mL	247632
Nitric Acid Solution, 1:1	500 mL	254049
Paper, pH, 0–14 pH range	100/pkg	2601300
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Sodium Hydroxide Solution, 5 N	50 mL	245026
Sulfuric Acid Standard Solution, 5.25 N	100 mL	244932
Test tube brush	each	69000
Thermometer, –10 to 110 °C	each	187701
Water, deionized	500 mL	27249

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12/2018, Edition 2