Aluminum

DOC316.53.01002

Aluminon Method[1] — Method 8012

0.008 to 0.800 mg/L Al^{3+} (spectrophotometers) — Powder Pillows

0.01 to 0.80 mg/L Al^{3+} (colorimeters)

Scope and application: For water and wastewater.

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

Test preparation

Instrument specific information

The table in this section shows all of the instruments that have the program for this test. Table 1 shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests.

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

Table 1 Instrument-specific information for powder pillows

Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700	The fill line is to the right.	2495402
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906

Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.
The sample must be digested with heat and acid to make sure that all forms of the metal are measured. Use the mild or vigorous digestion. Refer to the Water Analysis Guide for more information.
Clean all glassware with 6.0 N (50%) hydrochloric acid, then rinse thoroughly with deionized water to remove contaminants.
The sample temperature must be between 20–25 °C (68–77 °F) for accurate results.
The Pour-Thru Cell can be used (for applicable instruments) if rinsed well with deionized water between the blank and the prepared samples.
For best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to get the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used and use any recommended personal protective equipment.
Dispose of reacted solutions according to local, state and federal regulations. Use the Safety Data Sheets for disposal information for unused reagents. Consult 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
Cylinder, graduated mixing, 50-mL, with glass stopper	1
Sample cells. For information about sample cells, adapters or light shields, refer to Instrument specific information on page 1.	2

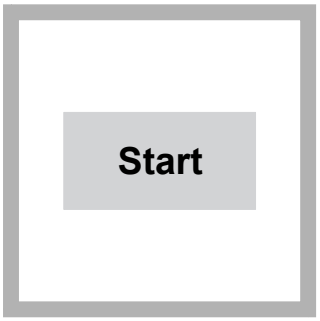
[1] AluVer is a registered trademark of Hach Company.

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

Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (about 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep preserved samples at room temperature for a maximum of 6 months.
- Before analysis, adjust the pH to 3.5–4.5 with 5.0 N sodium hydroxide standard solution.
- Correct the test result for the dilution from the volume additions.

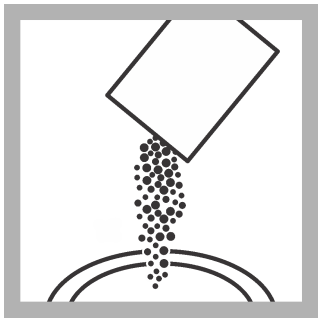
Powder pillow procedure



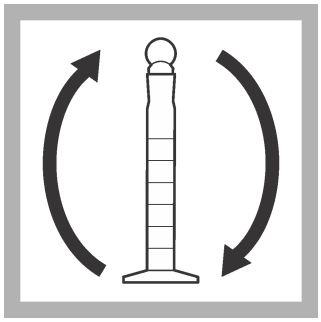
1. Start program **10 Aluminum Alumin**. For information about sample cells, adapters or light shields, refer to Instrument specific information on page 1.

Note: *Although the program name may vary between instruments, the program number does not change.*

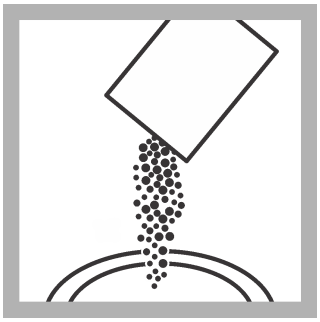
2. Fill a graduated mixing cylinder to the 50-mL mark with sample.



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

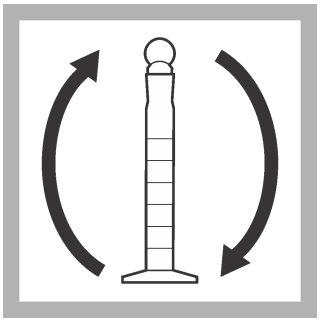


4. Close the cylinder. Invert the cylinder several times to mix.

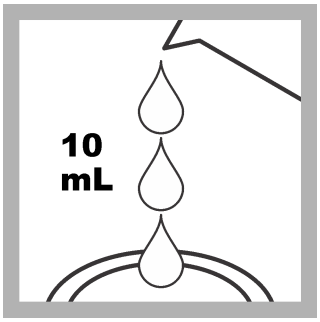


5. Add one AluVer 3 Aluminum reagent powder pillow.
An orange to orange-red color shows if aluminum is present.

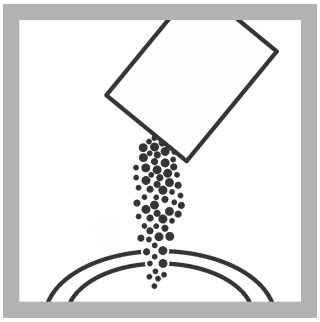
6. Start the instrument timer. A 1-minute reaction time starts.



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

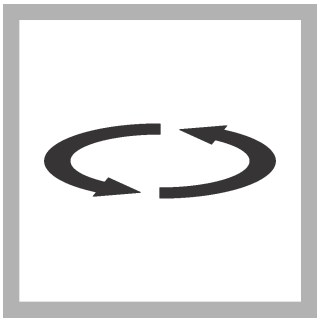


8. Prepare the blank: Pour 10 mL of the reacted sample into a sample cell.



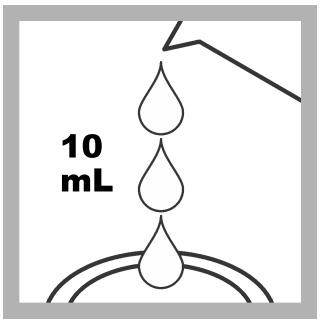
9. Add one Bleaching 3 Reagent powder pillow to the blank.

10. Start the instrument timer. A 30-second reaction time starts.

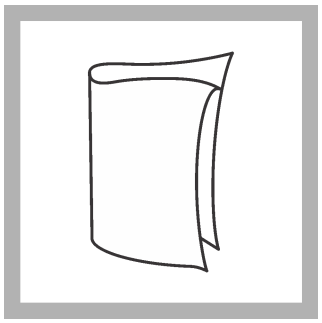


11. Swirl the sample cell vigorously. The solution will show a light to medium orange.

12. Start the instrument timer. A 15-minute reaction time starts.

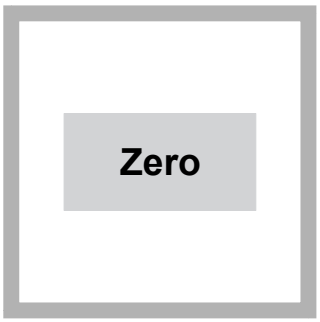


13. Prepare the sample: Pour 10 mL of solution from the cylinder into a second sample cell.

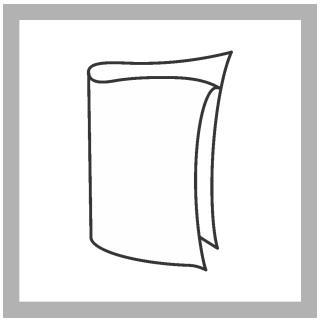


14. When the timer expires, clean the blank.

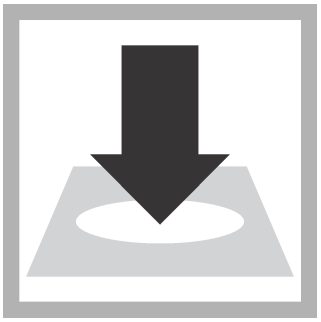
15. Insert the blank into the cell holder.



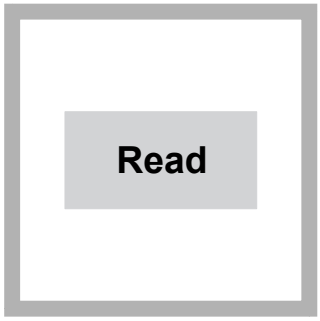
16. Push **ZERO**. The display shows 0.00 or 0.000 mg/L Al^{3+}.



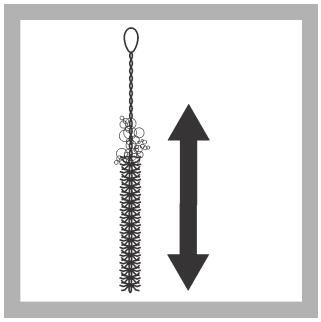
17. Clean the prepared sample.



18. Within five minutes after the timer expires, insert the prepared sample into the cell holder.



19. Push **READ**. Results show in mg/L Al^{3+}.



20. Clean the graduated cylinder and sample cells with soapy water and a brush immediately after the test. 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 one drop of m-Nitrophenol Indicator Solution to 50 mL of fresh sample. **2.** Add one 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 one 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 one drop of m-Nitrophenol Indicator Solution to 50 mL of fresh sample. A yellow color indicates excessive alkalinity. **2.** Add one 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.

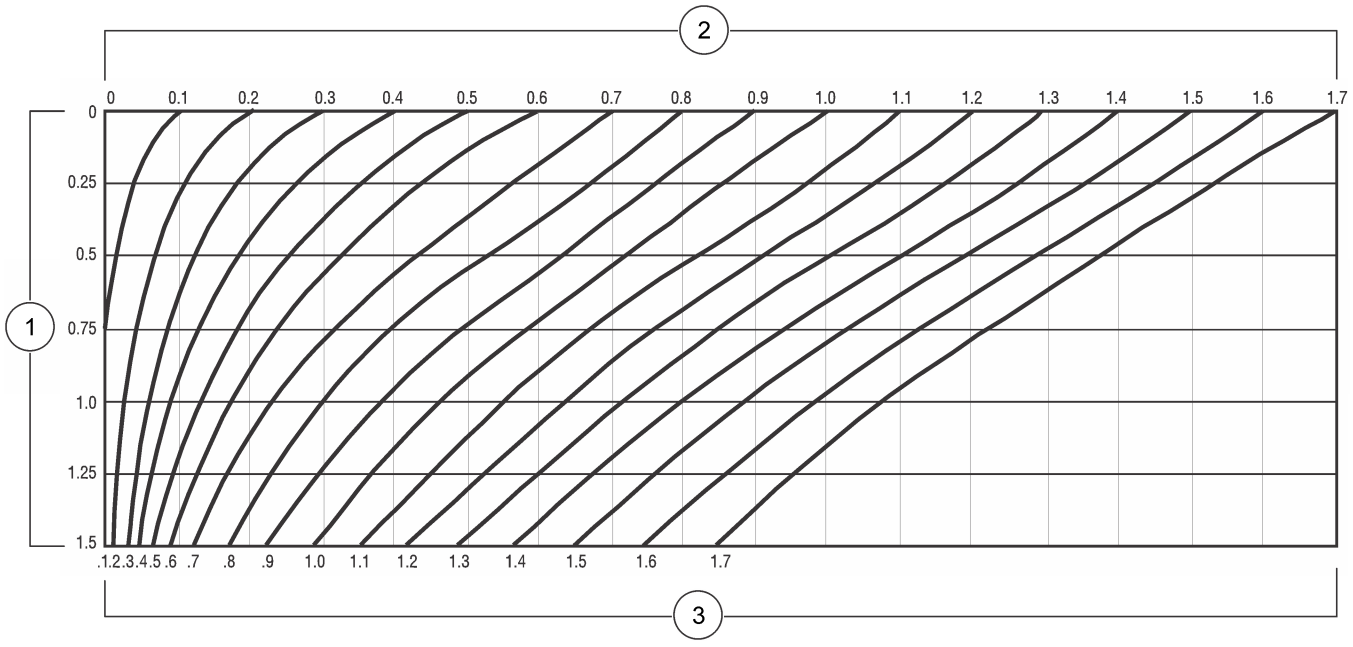
Fluoride interference

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

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 mg/L F^- in the sample	**2** mg/L Al^{3+} from test procedure	**3** Actual mg/L Al^{3+}

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:

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

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 0.3 mL of the standard solution, respectively, to three 50-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 the standard solution method to validate the test procedure, reagents and 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
- Deionized water

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 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 slight variations in the reagents or instruments.*

Method performance

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

Program	Standard	Precision (95% Confidence Interval)	Sensitivity Concentration change per 0.010 Abs change
10	0.40 mg/L Al^{3+}	0.385–0.415 mg/L Al^{3+}	0.008 mg/L Al^{3+}

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. The measurement wavelength is 522 nm for spectrophotometers or 520 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Aluminum Reagent Set, includes:	—	100 tests	2242000
AluVer® 3[1] Aluminum Reagent Powder Pillow	1	100/pkg	1429099
Ascorbic Acid Powder Pillow	1	100/pkg	1457799
Bleaching 3 Reagent Powder Pillow	1	100/pkg	1429449
Hydrochloric Acid, 6.0 N	varies	500 mL	88449
Water, deionized	varies	4 L	27256

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

Required apparatus

Description	Quantity/test	Unit	Item no.
Cylinder, graduated mixing, 50-mL, with glass stopper	1	each	189641

Recommended standards or Recommended standards 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
Aluminum Standard Solution, 10-mL Voluette® Ampule, 50-mg/L as Al	16/pkg	1479210
Ampule Breaker, Voluette® ampules	each	2196800

Optional reagents and apparatus

Description	Unit	Item no.
Flask, volumetric, Class A, 100-mL	each	1457442
Flask, volumetric, Class A, 250-mL	each	1457446
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 1970001	50/pkg	2185696
Sodium Hydroxide, 5 N	50 mL	245026
Sulfuric Acid Standard Solution, 5.25 N	100 mL	244932
Test tube brush	each	69000

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