Aluminum

DOC316.53.01003

Eriochrome Cyanine R Method[1]

Method 8326

0.006 to 0.250 mg/L Al^{3+}

Powder Pillows

Scope and application: For water.

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

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
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402
DR 5000 DR 3900	The fill line is toward the user.	

Before starting

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.
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 automatically with the reagent blank adjust option.
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

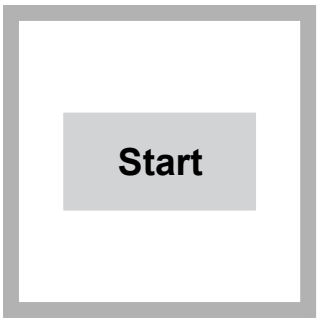
Description	Quantity
ECR Reagent Powder Pillow	1
ECR Masking Reagent Solution	1 drop
Hexamethylenetetramine Buffer Reagent Powder Pillow	1
Mixing cylinder, graduated, 25 mL, glass stopper	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

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

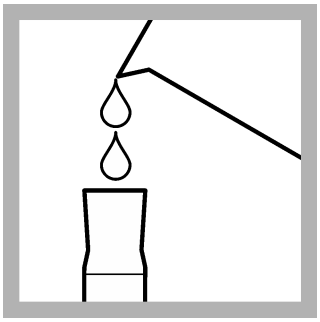
Sample collection and storage

- 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 2.9–4.9 with 12 N potassium hydroxide solution and/or 1 N potassium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

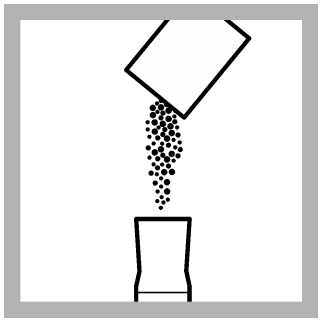
Test procedure



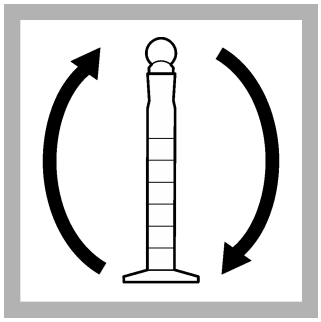
1. Start program **9 Aluminum ECR**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.



2. Prepare the sample: Fill a mixing cylinder to the 20-mL line with sample.

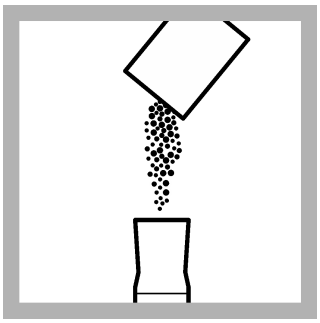


3. Add the contents of one ECR Reagent Powder Pillow for 20-mL samples.

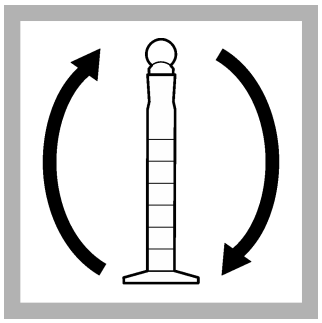


4. Put the stopper on the mixing cylinder. Invert the mixing cylinder several times to fully dissolve the powder.
Undissolved reagent causes inconsistent results.

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



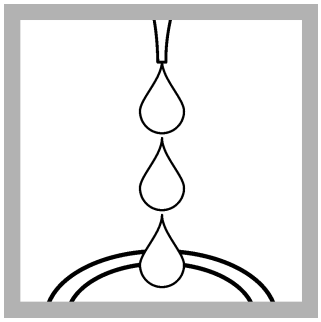
6. After the timer expires, add one Hexamethylene-tetramine Buffer Reagent powder pillow.



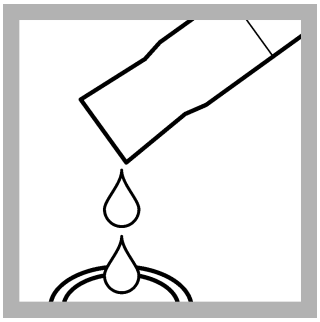
7. Put the stopper on the mixing cylinder. Invert the mixing cylinder several times to mix.
The solution color becomes red-orange if aluminum is in the sample.

8. Pour 10 mL of the solution from the mixing cylinder into the sample cell.

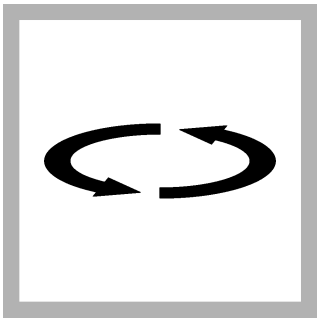
9. Add 1 drop of ECR Masking Reagent Solution into a clean square sample cell.



10. Prepare the blank:

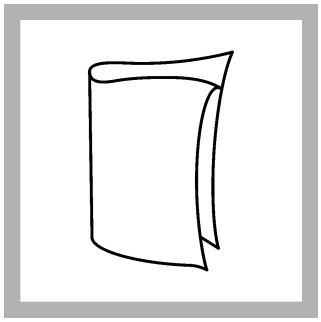


11. Pour 10 mL of the solution from the mixing cylinder into the sample cell.

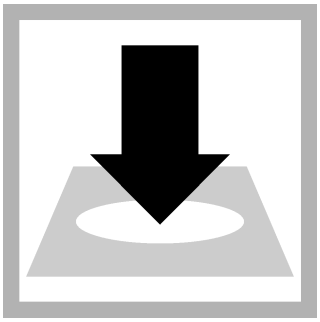


12. Swirl to mix.

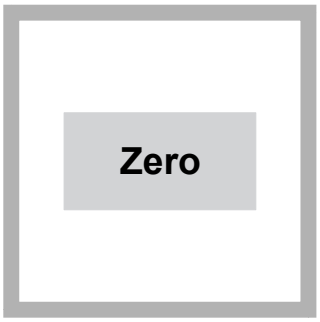
13. Start the instrument timer. A 5-minute reaction time starts.



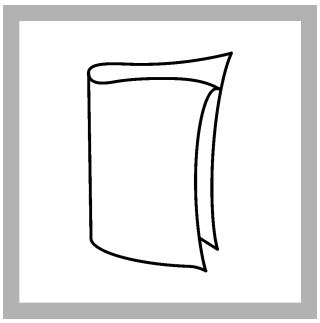
14. When the timer expires, clean the blank sample cell.



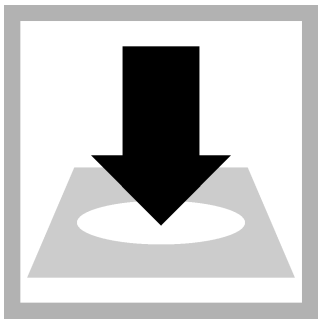
15. Insert the blank into the cell holder.



16. Push **ZERO**. The display shows 0.000 mg/L Al^{3+}. Some instrument models show a non-zero value.



17. Clean the prepared sample cell.



18. Within 5 minutes after the timer expires, insert the prepared sample into the cell holder.

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

Interferences

Table 2 shows the ions that were individually examined to the given concentrations and do not cause interference. No cumulative effects or influences of other ions were found.

Table 2 Interfering substances

Interfering substance	Interference level
Acidity	More than 62 mg/L as $CaCO_3$
Alkalinity	More than 750 mg/L as $CaCO_3$
Ca^{2+}	More than 1000 mg/L as $CaCO_3$
Cl^-	More than 1000 mg/L as Cl^-

Table 2 Interfering substances (continued)

Interfering substance	Interference level
Cr^{6+}	0.2 mg/L (error is –5% of reading)
Cu^{2+}	2 mg/L (error is –5% of reading)
Fe^{2+}	More than 4 mg/L (error is positive and equals mg/L Fe^{2+} x 0.0075)
Fe^{3+}	More than 4 mg/L (error is positive and equals mg/L Fe^{3+} x 0.0075)
F^-	Refer to Correct for fluoride interference on page 5.
Hexametaphosphate	0.1 mg/L as PO_4^{3-} (error is –5% of reading)
Mg^{2+}	More than 1000 mg/L as $CaCO_3$
Mn^{2+}	More than 10 mg/L
NO_2^-	More than 5 mg/L
NO_3^-	More than 20 mg/L
pH	2.9–4.9 or 7.5–11.5. A sample pH of approximately 4.9–7.5 causes dissolved aluminum to partially convert to colloidal and insoluble forms. This method measures many of those forms of aluminum without pH adjustment.
PO_4^{3-}(ortho)	4 mg/L (error is –5% of reading)
Polyphosphate	Use the pretreatment steps that follow to decrease the polyphosphate interference. Refer to Decrease polyphosphate interference on page 4.
SO_4^{2-}	More than 1000 mg/L
Zn^{2+}	More than 10 mg/L

Decrease polyphosphate interference

Complete the pretreatment steps that follow to decrease the polyphosphate interference in the sample.

Items to collect:

- Graduated mixing cylinder, 50-mL
- Erlenmeyer flask, 125-mL
- Magnetic stir bar
- Hot plate/stirrer
- Bromphenol Blue Indicator Solution
- Hydrochloric Acid, 6 N
- Potassium Hydroxide Standard Solution, 12.0 N
- Sulfuric Acid Standard Solution, 5.25 N
- Deionized water

1. Add a magnetic stir bar to a 125-mL Erlenmeyer flask. Rinse the Erlenmeyer flask, the magnetic stir bar and the 50-mL graduated mixing cylinder with 6 N Hydrochloric Acid. This removes aluminum contaminants.
   Note: *If a reagent blank is used, rinse two Erlenmeyer flasks.*
2. To prepare the reagent blank, measure 50 mL of deionized water into the 125-mL Erlenmeyer flask with the graduated mixing cylinder. Only do this step when a reagent is replaced—even if the new reagent has the same lot number. After the pretreated sample is analyzed, subtract the aluminum concentration of the reagent blank.
3. Measure 50 mL of sample into the 125-mL Erlenmeyer flask with the graduated cylinder. Use a small quantity of deionized water to rinse the cylinder contents in the flask.
4. Add 4.0 mL of 5.25 N Sulfuric Acid Standard Solution.

5. Use a combination hot plate/stirrer to boil and stir the sample for at least 30 minutes. Add deionized water as necessary to keep a sample volume of 20–40 mL. Do not boil dry.
6. Let the temperature of the solution decrease to almost room temperature.
7. Add 2 drops of Bromphenol Blue Indicator Solution.
8. Add 1.5 mL of 12.0 N Potassium Hydroxide Standard Solution with the calibrated, plastic dropper that is supplied.
9. Swirl to mix. The color will be yellow or green, but not purple. If the color is purple, do step 1 again with additional 1 mL N Sulfuric Acid Standard Solution from step 4.
10. While swirling the flask, add 1.0 N Potassium Hydroxide Solution, one drop at a time, until the solution is a dirty green color.
11. Pour the solution into the graduated cylinder. Rinse the flask contents into the graduated cylinder with deionized water until the total volume is 50 mL.
12. Use this solution as the prepared sample in the test procedure.

Correct for fluoride interference

Refer to Table 3 to correct for fluoride interference. Use interpolation to find intermediate values. Do not use correction graphs or charts found in other publications.
Example: If the fluoride concentration is known to be 1.00 mg/L F^- and the ECR method gives a reading of 0.060 mg/L aluminum, then the correct aluminum concentration is 0.183 mg/L.

Table 3 Fluoride concentration (mg/L)

mg/L	0	0.2	0.4	0.6	0.8	1.0	1.2	1.4	1.6	1.8	2.0
0	0	0	0	0	0	0	0	0	0	0	0
0.010	0.010	0.019	0.030	0.040	0.052	0.068	0.081	0.094	0.105	0.117	0.131
0.020	0.020	0.032	0.046	0.061	0.077	0.099	0.117	0.137	0.152	0.173	0.193
0.030	0.030	0.045	0.061	0.077	0.098	0.124	0.146	0.166	0.188	0.214	0.243
0.040	0.040	0.058	0.076	0.093	0.120	0.147	0.174	0.192	0.222	—	—
0.050	0.050	0.068	0.087	0.109	0.135	0.165	0.188	0.217	—	—	—
0.060	0.060	0.079	0.100	0.123	0.153	0.183	0.210	0.241	—	—	—
0.070	0.070	0.090	0.113	0.137	0.168	0.201	0.230	—	—	—	—
0.080	0.080	0.102	0.125	0.152	0.184	0.219	—	—	—	—	—
0.090	0.090	0.133	0.138	0.166	0.200	0.237	—	—	—	—	—
0.100	0.100	0.124	0.150	0.180	0.215	—	—	—	—	—	—
0.120	0.120	0.146	0.176	0.209	0.246	—	—	—	—	—	—
0.140	0.140	0.196	0.201	0.238	—	—	—	—	—	—	—
0.160	0.160	0.191	0.226	—	—	—	—	—	—	—	—
0.180	0.180	0.213	—	—	—	—	—	—	—	—	—
0.200	0.200	0.235	—	—	—	—	—	—	—	—	—
0.220	0.220	—	—	—	—	—	—	—	—	—	—
0.240	0.240	**True Aluminum Concentration (mg/L) Al**									

Accuracy check

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
- 1000-mL volumetric flask, Class A
- 1.0-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water

1. Prepare a 0.100-mg/L aluminum standard solution as follows:
   a. Use a pipet to add 1.00 mL of a 100-mg/L aluminum standard solution into the volumetric flask. *(Alternate preparation: Use a pipet to add 2.0 mL of a 50-mg/L aluminum standard solution into the 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 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 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
9	0.100 mg/L Al^{3+}	0.091–0.109 mg/L Al^{3+}	0.002 mg/L Al^{3+}

Summary of Method

Eriochrome Cyanine R reacts with aluminum in a sample to show an orange-red color. The intensity of color is proportional to the aluminum concentration. The measurement wavelength is 535 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Aluminum Reagent Set (100 Tests), includes:	—	—	2603700
ECR Reagent Powder Pillows	1	100/pkg	2603849
Hexamethylenetetramine Buffer Reagent Powder Pillows	1	100/pkg	2603999
ECR Masking Reagent Solution	1 drop	25 mL SCDB	2380123

Required apparatus

Description	Quantity/test	Unit	Item no.
Mixing cylinder, graduated, 25 mL, glass stopper	1	each	189640

Recommended standards

Description	Unit	Item no.
Aluminum Standard Solution, 10-mL Voluette® Ampule, 50 mg/L as Al	16/pkg	1479210
Aluminum Standard Solution, 100-mg/L as Al^{3+}	100 mL	1417442

Optional reagents and apparatus

Description	Unit	Item no.
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Bromphenol Blue Indicator Solution	100 mL MDB	1455232
Mixing cylinder, graduated, 50 mL	each	2088641
Flask, volumetric, Class A, 1000 mL glass	each	1457453
Hydrochloric Acid Solution, 6.0 N (1:1)	500 mL	88449
Nitric Acid Solution, 1:1	500 mL	254049
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet, volumetric, Class A, 1.00 mL	each	1451535
Pipet filler, safety bulb	each	1465100
Potassium Hydroxide Solution, 1.0 N	50 mL SCDB	2314426
Potassium Hydroxide Solution, 12 N	100 mL MDB	23032
Sulfuric Acid Standard Solution, 5.25 N	100 mL	244932
Water, deionized	4 L	27256

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