Aluminum in Brine

DOC316.53.01565

Ternary ECR Method

0 to 100 µg/L as Al

Scope and application: For ultrapure saturated NaCl feed brine used in the Chlor-alkali industry. Not for water or a non-saturated brine solution.

Test preparation

Before starting

This test is for the DR3000 or higher, but can be used with other narrow bandpass spectrophotometers with a sample pathlength of at least 20 mm.
Use glassware for this test. Do not use plastic ware.
Soak or rinse glassware in warm hydrochloric acid, 6.0 N (1:1). Rinse with demineralized water that contains no aluminum.
Analyze one purified brine sample and at least one of the aluminum brine standards that is prepared in Prepare a calibration curve on page 2 frequently. Use the test results to make sure that the reagents are good and the calibration is satisfactory.
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

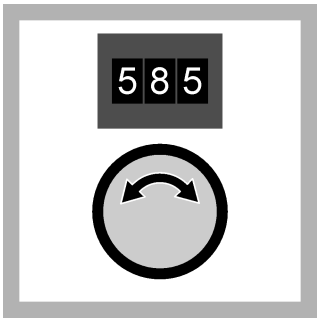
Description	Quantity
Aluminum Standard Solution, 10 mg/L	25 mL
Ammonium Fluoride Solution, 30%	4 drops
Masking Reagent for Aluminum in Brine	9.0 mL
ECR Reagent Solution for Aluminum in Brine	18.0 mL
Buffer Reagent for Aluminum in Brine	9.0 mL
Tensette pipet, 0.1 to 1.0 mL	1
Graduated cylinder, glass, 25 mL	9
Flask, volumetric, class A, 50 mL	1
Sample cells, 1-inch square glass, matched pair, 25 mL	1

Refer to Required reagents and apparatus on page 7 for order information.

Prepare a calibration curve

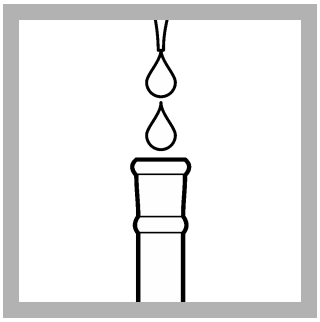
Prepare a new calibration curve for each type of brine analyzed and for each lot of reagents.

- The aluminum brine standards prepared in step 8 are stable for approximately 1 hour.
- At step 11, the carbonates in the brine will be released as carbon dioxide (CO_2) gas when the masking reagent is added. The gas will increase the pressure in the cylinders. Use caution when the cylinder cap is removed.
- At step 12, when the ECR reagent solution mixes with the acidic brine, a slight precipitation of the ternary complex can occur. Precipitation does not occur when the brine pH is adjusted to pH 5.5 to 6.5 with the buffer reagent. Formation of the ternary complex can be very slow if the sequence of the reagent addition is changed.

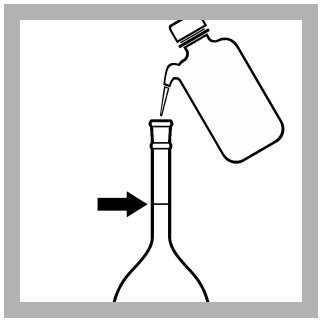


1. On the DR3000 or higher, set the wavelength to 585.0 nm.

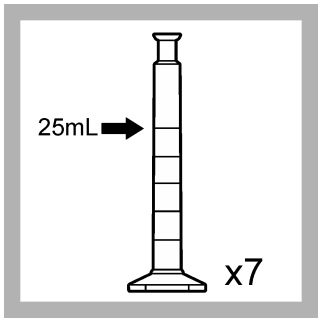
On the newer spectrophotometers (DR2800-DR3900 and DR5000-DR6000), make a single wavelength program and enter the wavelength.



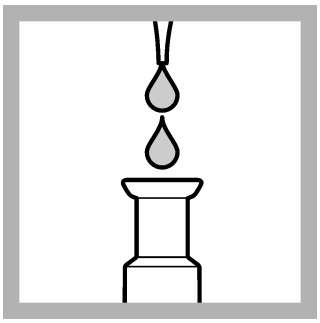
2. 5.0-mg/L aluminum standard: Use a pipet to add 25.00 mL of the 10.0-mg/L aluminum standard solution to a clean, 50-mL, class A volumetric flask.



3. Add aluminum-free, deionized water to the volumetric flask to the 50-mL mark. Mix well.

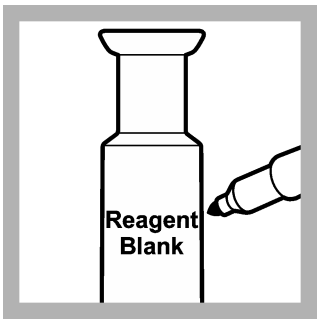


4. Cylinders of brine (7x): Add 25-mL of purified brine to seven, clean, 25-mL graduated mixing cylinders.

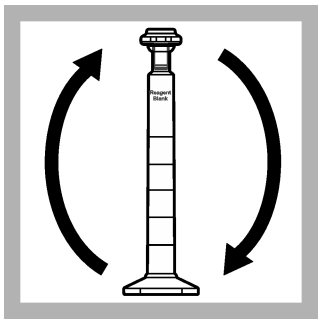


5. Reagent blank: Add 2 drops of Ammonium Fluoride Solution, 30% to one of the graduated cylinders.

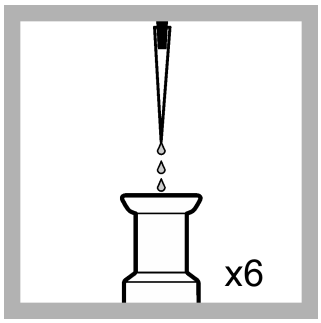
The ammonium fluoride will complex the aluminum and set a zero reference point.



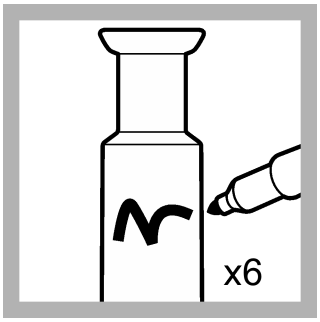
6. Write "Reagent Blank" on the cylinder.



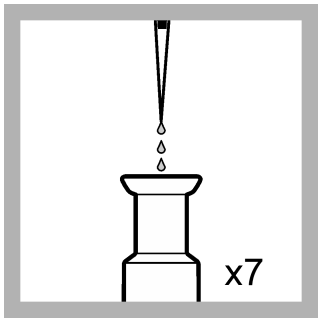
7. Insert a stopper into the reagent blank cylinder. Invert to mix.



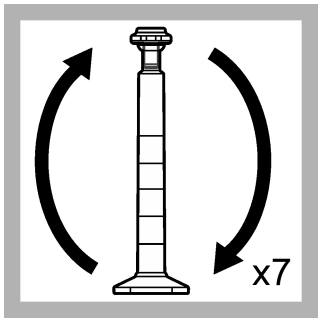
8. Aluminum brine standards (0, 20, 40, 60, 80 and 100 µg/L): Use the Tensette pipet to add aliquots of 0.0, 0.1, 0.2, 0.3, 0.4 and 0.5 mL of the prepared 5.0-mg/L aluminum standard to the six remaining cylinders.



9. Write the applicable µg/L Al value for the brine standards on each cylinder (0, 20, 40, 60, 80 and 100 µg/L).

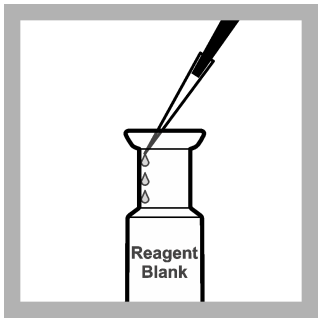


10. Use a pipet to add 1.0 mL of Masking Reagent for Aluminum in Brine to the reagent blank and the six aluminum brine standards.



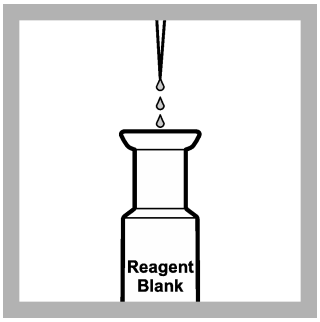
11. Insert a stopper into each cylinder. Invert to mix.

The masking reagent adjusts the sample pH to less than 4.

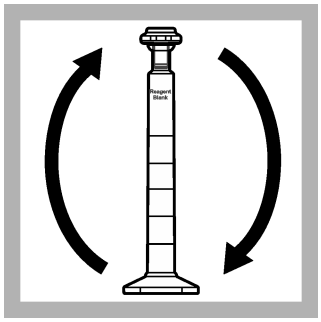


12. Use a pipet to add 2.00 mL of ECR Reagent Solution to the reagent blank.

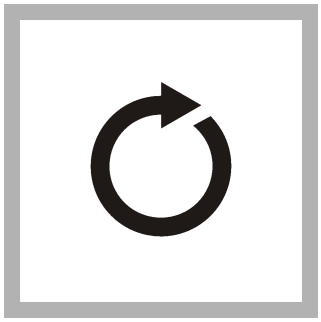
Touch the pipet to the inner surface of the cylinder wall. Let the reagent flow down the surface of the cylinder. Let the reagent float on top of the brine solution.



13. Within 1 minute, use a pipet to add 1.0 mL of Buffer Reagent for Aluminum in Brine to the reagent blank.

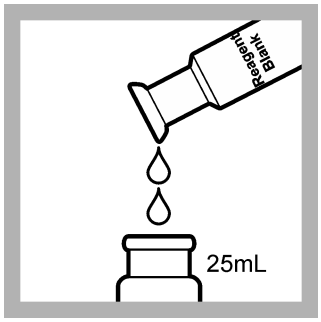


14. Insert a stopper into the reagent blank cylinder. Invert to mix.

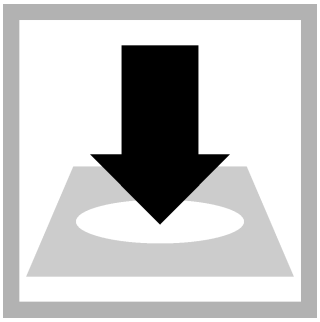


15. Do steps 12 to 14 again to each of the six aluminum brine standards.

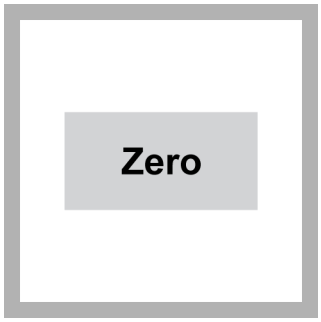
Make sure to measure the reagent blank and then the aluminum brine standards after 3 minutes but no more than 15 minutes.



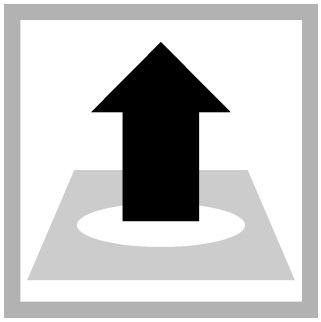
16. Measure the reagent blank: Add approximately 25-mL of the reagent blank to a clean sample cell.



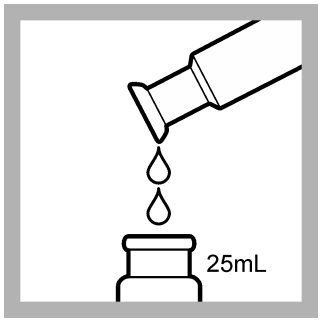
17. Put the sample cell into the cell compartment. Close the sample compartment cover.



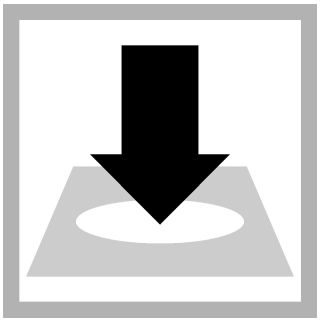
18. Push **ZERO ABS**.



19. Remove the sample cell.



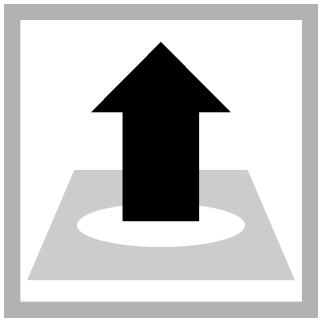
20. Measure the aluminum brine standards: Add approximately 25-mL of an aluminum brine sample to a clean sample cell.



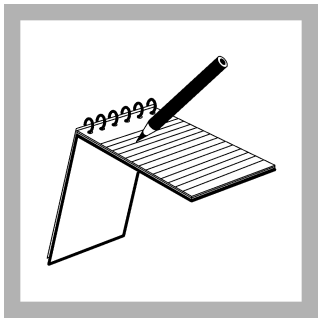
21. Put the sample cell into the cell compartment. Close the sample compartment cover.



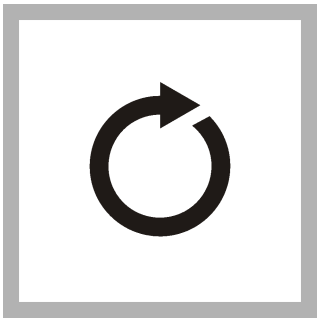
22. Push **READ**.



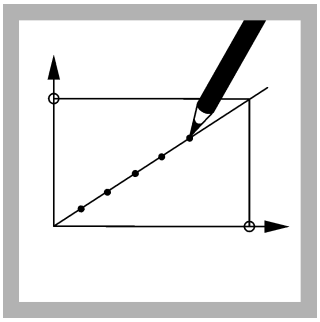
23. Remove the sample cell.



24. Record the absorbance of the aluminum brine standard.



25. Do steps 20 to 24 again to measure each aluminum brine standard.

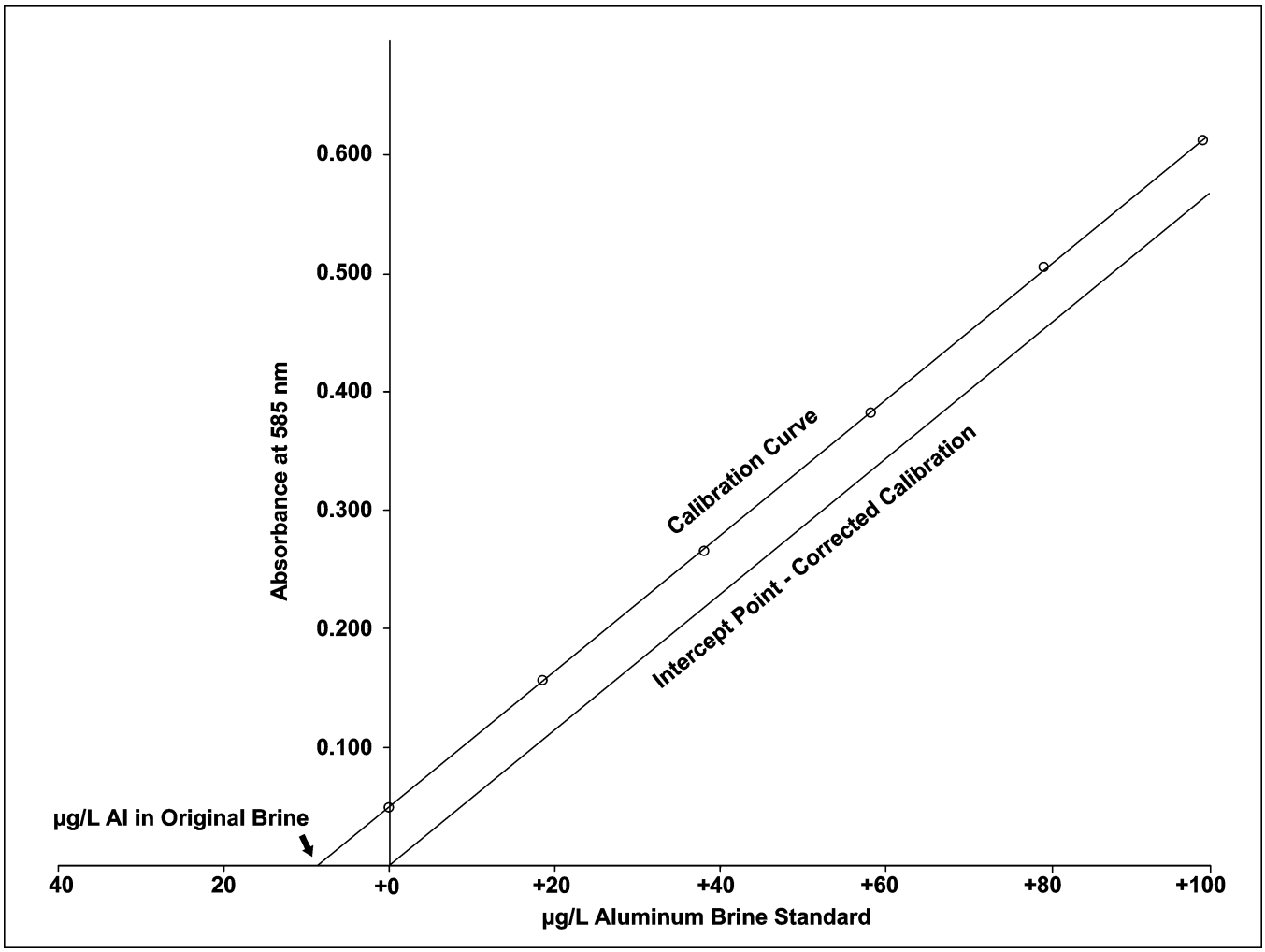


26. Do the steps in Identify the calibration curve on page 4.

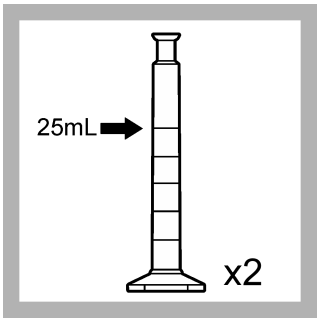
Identify the calibration curve

1. Record the absorbance of each calibration brine standard (0, 20, 40, 60, 80 and 100 µg/L) on a graph similar to Figure 1.
2. Record the soluble µg/L Al of the original brine on the graph (the negative intercept).
3. Make a line from the negative intercept through the five points on the graph (the calibration curve).
4. Subtract the absorbance of the 0 µg/L aluminum brine standard from the absorbance values of the five aluminun brine standards (20, 40, 60, 80 and 100 µg/L).
5. Record the adjusted absorbance for each of the five calibration brine standards on the graph.
6. Make a line through the five points on the graph (the intercept point-corrected calibration).
7. Save the five points of the intercept point-corrected calibration as a single wavelength calibration on the DR3000 or higher. Refer to the instrument user manual for instructions.

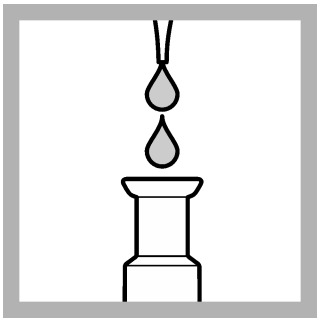
Figure 1 Example calibration graph



Test procedure

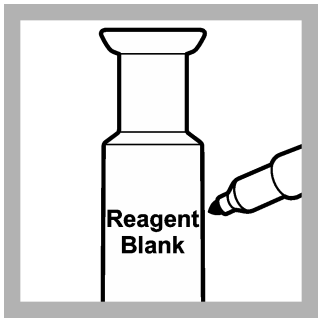


1. Cylinders of brine (2x): Add 25-mL of purified brine to two, clean, 25-mL graduated mixing cylinders.

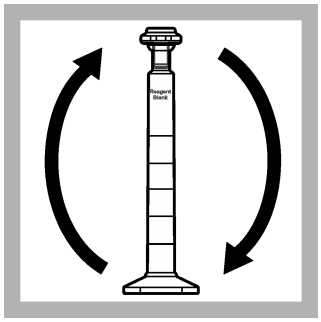


2. Reagent blank: Add 2 drops of Ammonium Fluoride Solution, 30% to one of the graduated cylinders.

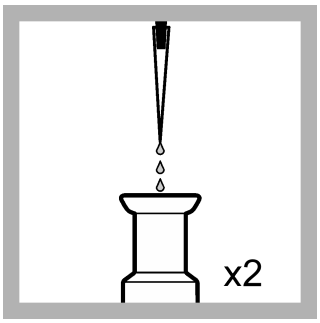
The ammonium fluoride will complex the aluminum and set a zero reference point.



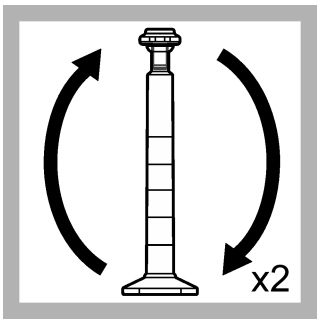
3. Write "Reagent Blank" on the cylinder.



4. Insert a stopper into the reagent blank cylinder. Invert to mix.

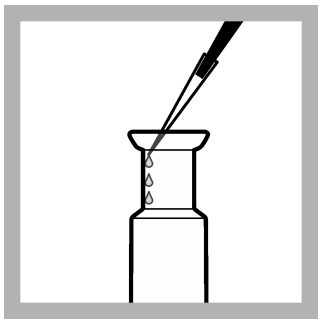


5. Use a pipet to add 1.0 mL of Masking Reagent for Aluminum in Brine to each cylinder.



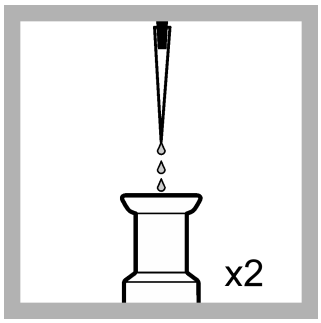
6. Insert a stopper into each cylinder. Invert to mix.

The masking reagent adjusts the sample pH to less than 4.

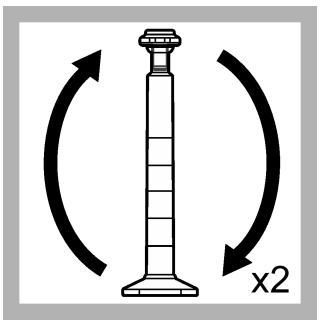


7. Use a pipet to add 2.00 mL of ECR Reagent Solution to each cylinder.

Touch the pipet to the inner surface of the cylinder wall. Let the reagent flow down the surface of the cylinder. Let the reagent float on top of the brine solution.

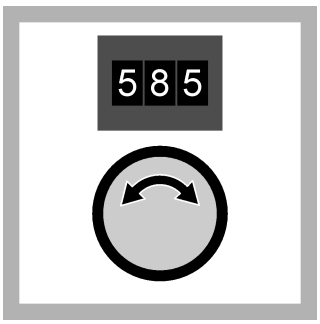


8. Within 1 minute, use a pipet to add 1.0 mL of Buffer Reagent for Aluminum in Brine to each cylinder.



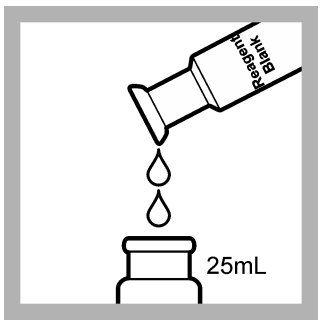
9. Insert a stopper into each cylinder. Invert to mix.

Make sure to measure the reagent blank and then the other brine solution after 3 minutes but no more than 15 minutes.

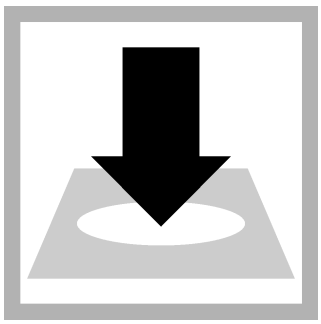


10. On the DR3000 or higher, set the wavelength to 585.0 nm.

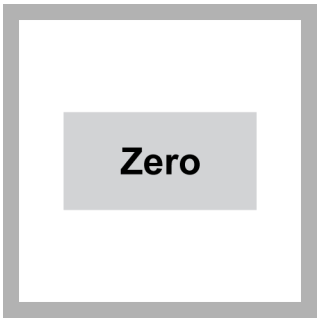
On the newer spectrophotometers, select the single wavelength program for 585 nm.



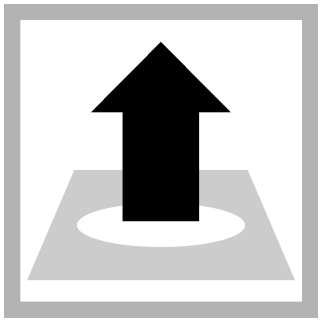
11. Reagent blank: Add approximately 25-mL of the reagent blank to one of two clean, matched sample cells.



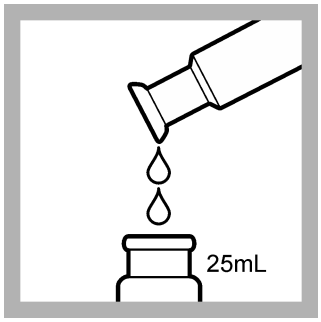
12. Put the sample cell into the cell compartment. Close the sample compartment cover.



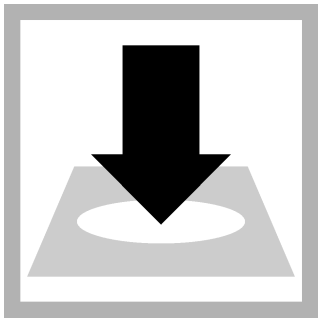
13. Push **ZERO ABS**.



14. Remove the sample cell.



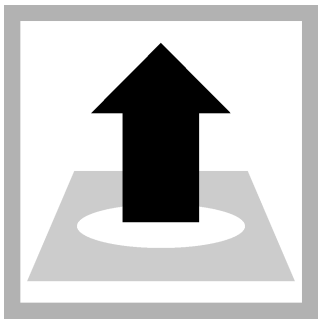
15. Prepared solution: Add approximately 25-mL of the solution from the other cylinder into the other clean, matched sample cell.



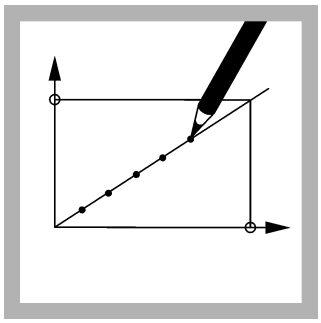
16. Put the sample cell into the cell compartment. Close the sample compartment cover.



17. Push **READ**.



18. Remove the sample cell.



19. Record the absorbance on the calibration graph to determine the µg/L aluminum.

On the newer spectrophotometers, the mg/L Al measurement shows on the display if the calibration curve was added to the single wavelength program for 585 nm.

Interferences

Iron interference is 1.0 µg/L Al at 250 µg/L Fe^{+3} and 11.0 µg/L Al at 1000 µg/L Fe^{+3}. Phosphate interferes above 5.0 mg/L PO_4^{3-}.

Summary of Method

The ternary ECR method is based on a formation of a ternary complex between aluminum, eriochrome cyanine R and a cationic polymer. The ternary chemistry is 90% more sensitive than the conventional ECR method. pH, reagent blank and chemical interferences have less of an effect on the result. The measurement wavelength is 585 nm.

Required reagents and apparatus

Description	Unit	Item no.
Masking Reagent for Aluminum in Brine	100 mL	2310542
ECR Reagent Solution for Aluminum in Brine	200 mL	2310629
Buffer Reagent for Aluminum in Brine	100 mL	2310742
Ammonium Fluoride Solution, 30%	50 mL SCDB	2305726

Required reagents and apparatus (continued)

Description	Unit	Item no.
Graduated cylinder, glass, 25 mL	each	2088640
Flask, volumetric, class A, 50 mL	each	1457441
Aluminum Standard Solution, 10 mg/L	100 mL	2305842

Optional reagents and apparatus

Description	Unit	Item no.
Sample cells, 1-inch square glass, matched pair, 25 mL	each	1993500
TenSette pipet, 0.1 to 1.0 mL, includes: 100 pipet tips	each	1970001
Pipet tips for TenSette pipet (1970001)	50/pkg	2185696
Pipet, volumetric, class A, 2 mL	each	1451536
Pipet, volumetric, class A, 25 mL	each	1451540
Hydrochloric acid, 6.0 N (1:1)	500 mL	88449

FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – **Call toll-free 800-227-4224**
Outside the U.S.A. – **Contact the HACH office or distributor serving you.**
On the Worldwide Web – **www.hach.com**; E-mail – **techhelp@hach.com**

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932

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