

Direct ISE method

Method 10255
3.55 g/L to 35 g/L Cl⁻
Powder Pillow ISA

Scope and application: For the measurement of high concentrations (1 M) of chloride in brine solutions, produced waters and hydraulic fracturing waters.



Test preparation

Instrument-specific information

This procedure is applicable to the meters and probes that are shown in [Table 1](#). Procedures for other meters and probes can be different.

Table 1 Instrument-specific information

Meter	Probe
HQ4100 and HQ30d portable one input, multi-parameter HQ4200 and HQ40d portable two input, multi-parameter HQ4300 portable three input, multi-parameter HQ430d benchtop one input, multi-parameter HQ440d benchtop two input, multi-parameter	Intellical ISECL181 combination chloride ISE

Before starting

Refer to the meter documentation for meter settings and operation. Refer to probe documentation for probe preparation, maintenance and storage information.

Condition the probe before use. To condition the probe, put the probe in a 3.55 g/L Chloride Standard solution for a minimum of 30 minutes.

Stir the standards and samples at a slow and constant rate to prevent the formation of a vortex.

Air bubbles under the sensor tip can cause slow response or measurement errors. To remove the bubbles, carefully shake the probe.

Calibrate the probe regularly for the best measurement accuracy. Refer to [Calibration](#) on page 3.

During calibration, measure the standard solutions from lowest to highest concentration for best results.

Between measurements, rinse the probe with deionized water. Blot dry with a lint-free cloth.

Make sure that the calibration solutions and the samples are at the same temperature (± 2 °C (± 3.6 °F)) for best 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.

This procedure is specified for the HQ meters and HQd meters. The Sension+ meters can be used, but the menus and navigation will be different.

Items to collect

Description	Quantity
Chloride Ionic Strength Adjustor (ISA) Buffer Powder Pillows	varies
Sodium chloride	11.55 g
Beaker, polypropylene, 50 mL, low form	4

Items to collect (continued)

Description	Quantity
Volumetric flask, 200-mL	3
Water, deionized	varies
Stir bar, magnetic, 2.2 x 0.5 cm (7/8 x 3/16 in.)	4
Stirrer, magnetic	1
Wash bottle with deionized water	1
Lint-free cloth	1

Refer to [Consumables and replacement items](#) on page 4 for order information.

Sample collection

- Collect samples in clean glass or plastic bottles.
- If immediate analysis is not possible, keep the samples at room temperature for a maximum of 28 days.

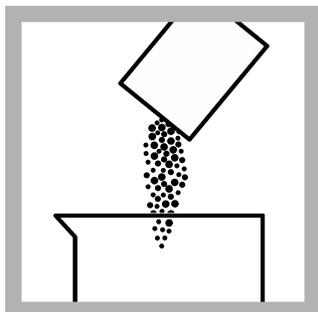
Configure the meter

Make sure that the meter is configured for calibration and measurements in g/L. Refer to the documentation for the applicable meter.

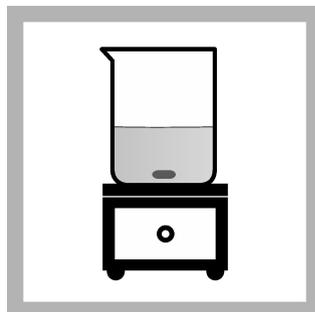
Procedure



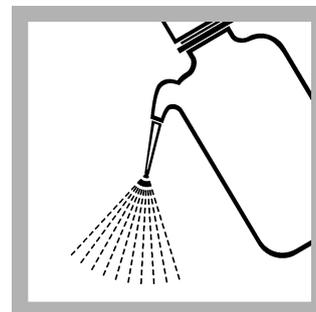
1. Add 25 mL of sample to a beaker.



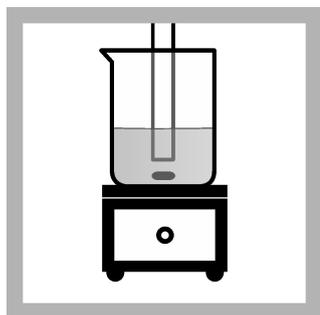
2. Add the contents of one chloride ISA powder pillow.



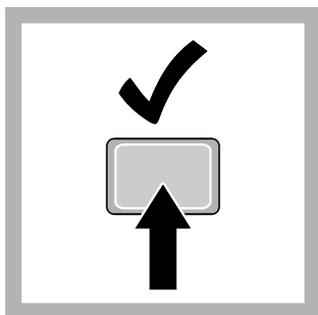
3. Add a stir bar and put the beaker on a magnetic stirrer. Stir at a moderate rate.



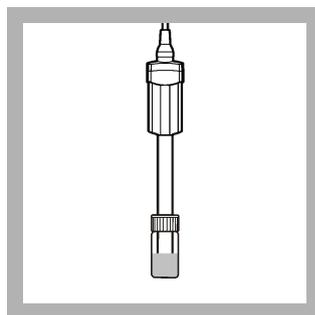
4. Rinse the probe with deionized water. Dry the probe with a lint-free cloth.



5. Put the probe in the solution. Do not let the probe touch the stir bar, bottom or sides of the container. Remove the air bubbles from under the probe tip.



6. Push **Read**. A progress bar is shown. When the measurement is stable, the lock icon is shown.



7. When measurements are done, put the probe in storage. Refer to the probe documentation.

Sample dilution

If the chloride concentration is more than 35 g/L (1 M), dilute the sample to a lower concentration. Complete the steps that follow to make a 1:10 (10-fold) dilution.

1. Measure 2.5 mL of the sample in a 25-mL graduated cylinder.
2. Dilute to the mark with deionized water. Mix well.
3. Pour the diluted sample into a beaker.
4. Use the test procedure to measure the concentration of the sample.
5. Multiply the result by 10 to get the concentration of the sample before dilution.

Calibration

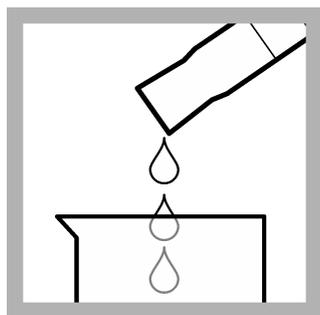
Prepare the standard solutions

Prepare the standard solutions for calibration as follows.

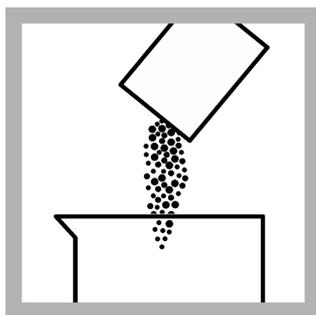
Items to collect:

- Sodium chloride (NaCl)
 - 200-mL volumetric flasks (3), Class A
 - Laboratory balance
 - Deionized water
1. Prepare a 35-g/L Chloride Standard Solution as follows:
 - a. Weigh 11.5 g of sodium chloride.
 - b. Quantitatively move the NaCl into a 200-mL volumetric flask.
 - c. Dilute to the mark with deionized water. Mix well.
 2. Prepare a 12.5-g/L Chloride Standard Solution as follows:
 - a. Move 71.43 mL (or g) of the 35-g/L Chloride Standard Solution into a 200-mL volumetric flask.
 - b. Dilute to the mark with deionized water. Mix well.
 3. Prepare a 3.55-g/L Chloride Standard Solution as follows:
 - a. Move 56.8 mL (or g) of the 12.5-g/L Chloride Standard Solution into a 200-mL volumetric flask.
 - b. Dilute to the mark with deionized water. Mix well.

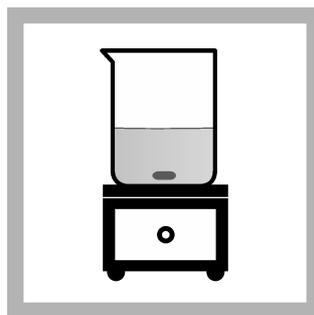
Calibration



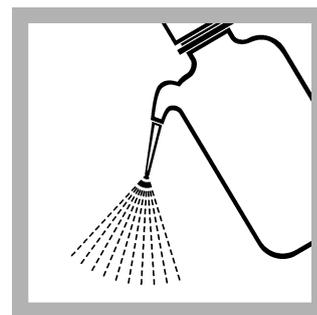
1. Add 25 mL of the lowest concentration standard solution to a beaker.



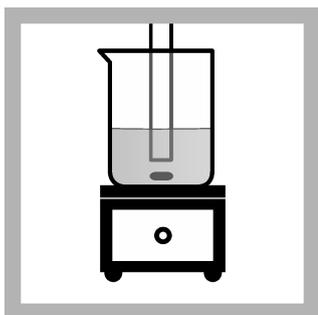
2. Add the contents of one chloride ISA powder pillow.



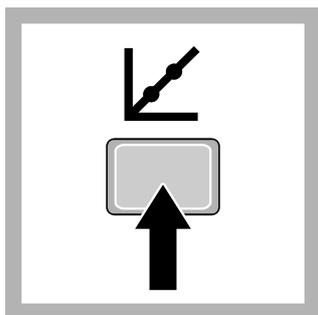
3. Add a stir bar and put the beaker on a magnetic stirrer. Stir at a moderate rate.



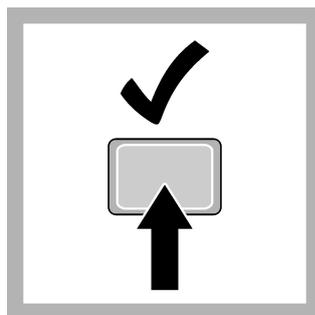
4. Rinse the probe with deionized water. Dry the probe with a lint-free cloth.



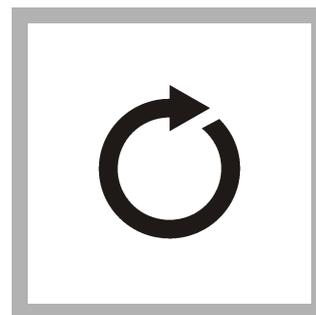
5. Put the probe in the solution. Do not let the probe touch the stir bar, bottom or sides of the container. Remove the air bubbles from under the probe tip.



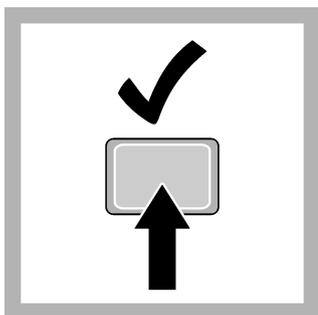
6. Push **Calibrate**. The standard solution value is shown.



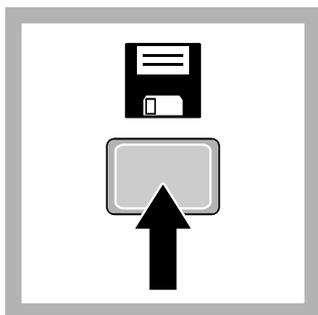
7. Push **Read**. A progress bar is shown. When the measurement is stable, the lock icon is shown.



8. Measure the remaining standard solutions.



9. Push **Done**. A calibration summary is shown when the minimum number of calibration standards are measured.



10. Push **Store** to accept the calibration.

Interferences

The sensing element reacts to chloride as well as other ions. Typically, probe response to another ion increases the potential, and causes a positive error. If Chloride ISA is added to the standards and samples, the effect of interfering ions is decreased. Refer to [Table 2](#).

Table 2 Interfering substances

Interfering substance	Interference level
Oxidizing agents such as Copper (Cu^{2+}), Iron (Fe^{2+}) and Permanganate (MnO_4^-)	Do not interfere.
Mercury	Interferes at all levels.
Ions that form insoluble salts of silver	Can form a layer of salt on the sensing surface and cause probe errors.
Strong reducing solutions	Can form a surface layer of silver.

Consumables and replacement items

HQ meters, HQd meters and probes

Description	Unit	Item no.
HQ4100 portable one input, multi-parameter meter	each	LEV015.53.4100A
HQ4200 portable two input, multi-parameter meter	each	LEV015.53.4200A

HQ meters, HQd meters and probes (continued)

Description	Unit	Item no.
HQ4300 portable three input, multi-parameter meter	each	LEV015.53.4300A
HQ430d benchtop one input, multi-parameter meter	each	HQ430D
HQ440d benchtop two input, multi-parameter meter	each	HQ440D
Intellical ISECL181 digital combination chloride ISE, 1 meter cable	each	ISECL18101
Intellical ISECL181 digital combination chloride ISE, 3 meter cable	each	ISECL18103

Recommended reagents and standards

Description	Unit	Item no.
Chloride Ionic Strength Adjustor (ISA) Buffer Powder Pillows	100/pkg	2318069
Sodium Chloride, ACS	454 g	18201H

Accessories

Description	Unit	Item no.
Beaker, polypropylene, 50 mL, low form	each	108041
Bottle, wash, 500 mL	each	62011
Graduated cylinder, polypropylene, 25 mL	each	108140
Flask, volumetric, Class A, 200 mL	each	1457445
Water, deionized	4 L	27256
Probe clips, color-coded, for IntelliCAL probes	50/pkg	5818400
Probe holder, 3 probes, for sensION+ benchtop meters	each	LZW9321.99
Probe stand, universal	each	8508850
Stir bar, magnetic, 2.2 x 0.5 cm (7/8 x 3/16 in.)	each	4531500
Stirrer, electromagnetic, 120 VAC, with electrode stand	each	4530001
Stirrer, electromagnetic, 230 VAC, with electrode stand	each	4530002



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