Chloride ISE

**Determine % Cl by Cl Meter**

**Ion Selective Electrode = ISE**

For what ion is the pH meter selective?

\[
\text{Weigh} \quad \text{Dissolve} \quad \text{ch} \quad 5\text{ml}
\]

**A.1**

\[
+ 5.0\text{ml} \quad \text{mV meter} \quad \text{[Cl}^-]\quad \% \text{Cl}
\]

**A.2**

\[
= 5.0\text{ml} \quad \text{ISA}
\]

**A.3**

\[
= 5.0\text{ml} \quad \text{ISA}
\]

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NOTES:

1. Reference material may be KCl or NaCl.
2. Reagents need not be dried.
3. ISA soln - rough - you use some soln for everything.

\[ \text{NaN}_3 \quad \text{=} \quad \text{NaNO}_3 \]
Ion selective electrode:

\[ E = E^0 + 0.059 \log [Cl^-] \]

Theory: \[ E = E^0 + 0.059 \log [Cl^-] \]

Polishing: may help

REFERENCE

SAMPLE

IONOPHORES "CARRY" Cl-

CATIONS CANNOT EXCHANGE

"PVC"
CHECKING ISE PROBE

0.45 M NaCl \( \rightarrow \) 250.0 mL (primary stock)
50.00 mL primary stock \( \rightarrow \) 100.00 mL (solution 1)

\[ \text{25.00 mL} \]

\[ \text{ISE} \]

\[ \rightarrow \text{measure mV} \]
\[ \rightarrow \text{record} \]

\[ \text{ISE} \]

\[ \rightarrow \text{measure mV again} \]
\[ \rightarrow \text{record} \]

\[ \Delta V \text{ should be 12 - 21 mV & should stabilize within a minute or two (drift } \leq 1-2 \text{ mV/min)} \]

if not - polish electrode, rinse, blot, try again
- if still no good - new probe
CALIBRATING ISE

Soln 1
25.00 mL

→ measure mV → measure mV → measure mV

→ record → record → record

+ 5.00 mL + 5.00 mL + 10.00 mL
I SE I SE I SE

+ 5.00 mL + 10.00 mL + 10.00 mL
I SE I SE I SE

→ measure mV → measure mV → measure mV

→ record → record → record

leave probe in soln
swirl, do not splash
let soln → still

model

$E = m \log[Cl^-] + b$

graph $V/mV$ vs $\log[Cl^-]$
get slope, intercept $m$
**Unknown Sample**

Examples:
- \( A : 0.1557 \, \text{g} \) sample in 25.00 \, \text{mL}
- \( B : 0.1435 \, \text{g} \)
- \( C : 0.1320 \, \text{g} \)

50.00 \, \text{mL}

Sample of A

\[ \frac{5.00}{50.00} \times \text{IS A} \]

\[ \frac{5.00}{50.00} \times \text{IS A} \]

Repeat as / B + C

For each, calculate

\[ (\circ) \, \text{log [Cl}^-\text{]} = \frac{E(\text{mV}) - b}{m} \]

\[ (\circ) \, \text{corrected [Cl}^-\text{]} = \text{[Cl}^-\text{]} \times \frac{50.00 \, \text{mL}}{5.00 \, \text{mL}} \]

\[ (\circ) \, \% \, \text{Cl}: \text{example} \quad A1 = 0.009523 \, \text{M [Cl}^-\text{]} \]

\[ \frac{0.009523 \, \text{mol Cl}^-}{L} \times \frac{0.2500 \, \text{L}}{35.547 \, \text{mol Cl}^-} \times \frac{1}{0.1557} \times 100\% = 51.28\% \]

\( \text{Cl}^- \)

\( b / b \)
To calculate the slope & intercept of the ISE calibration use Excel as follows:

1. Enter your [Cl^-] data and the mV data.
2. Calculate log [Cl^-].
3. Graph as "scatter".
4. Right-click on data points to get the menu shown.
5. Select "Add Trendline" as indicated.
(6) Select "Linear" trend line

(7) Select "Display equation on chart".

\[ y = \frac{-58.906}{I} + 15.547 \]

Your slope \( m \) = \( \frac{-58.906}{I} \) and intercept \( b \) = 15.547.
% Chloride Calculation:

For each measurement A1, A2, A3, ..., C2, C3 calculate:

(a) \( \log [Cl^-] = \frac{E_{(mv)}}{m} - b \) from calib curve

(b) corrected \([Cl^-]\)

A1 - no correction

A2 - \([Cl^-]\) corrected = \([Cl^-]\) cal - curve \times \frac{55.00 \text{ mL}}{60.00 \text{ mL}} \times \frac{60.00 \text{ mL}}{55.00 \text{ mL}}

A3 - \([Cl^-]\) corrected = \([Cl^-]\) cal - curve \times \frac{55.00 \text{ mL}}{60.00 \text{ mL}} \times \frac{60.00 \text{ mL}}{55.00 \text{ mL}}

(c) for each corrected \([Cl^-]\), calculate % Cl-

example for A1 = 0.009523 M \([Cl^-]\)

\[ \frac{0.007523 \text{ mol Cl}^-}{L} \times \frac{0.2500 \text{ L}}{35.54 \text{ g Cl}^-} \times \frac{1}{0.1000 \text{ L}} \times 100\% = 51.28\% \text{ Cl}^- \]
\[0.1557 \text{ g} \times \frac{1 \text{ mol}}{58.53 \text{ g}} \times \frac{1}{0.2500 \text{ L}} = 0.010647 \text{ M}\]

\[0.1557 \text{ g} \times \frac{1 \text{ mol}}{74.54 \text{ g}} \times \frac{1}{0.2500 \text{ L}} = 0.008384 \text{ M}\]