

Thermo. Titr. Application Note No. H-078

Title: Determination of Total Halides in Brines

Scope: Determination of total halides ($\text{Cl}^- + \text{Br}^- + \text{I}^-$) in sea water and similar brines. This procedure is suitable for the analysis of total halides in sea water contaminated with sodium aluminate solutions emanating from alumina refineries, and sea water which has been used for the neutralization of alumina refinery waste („red mud“) slurries. Given the small concentration of bromine and iodine in sea water, the total halide content approximates the chloride concentration.

Principle: Titration of an acidified sample of brine with standardized silver nitrate solution

Reagents: *Titrant:* 0.1 mol/L AgNO_3 solution.
Acid: 1 mol/L HNO_3 , prepared from A.R. HNO_3 and DI water

Method: Basic Experimental Parameters:

Titration delivery rate (mL/min.)	4
Delay before titration commences (sec)	3
No. of exothermic endpoints	1
Data smoothing factor (DSF)	40
Stirring speed (802 stirrer)	15

Titration: Pipette 1mL of sea water or treated brine (or volume of diluted sea water equivalent to 1mL) into a titration vessel. Acidify with 5mL 1 mol/L HNO_3 and dilute with DI water to make approximately 35mL of fluid. Titrate to a single exothermic endpoint.

Examples:		<i>Analysis of sea water collected from Moreton Bay, Queensland, Australia 25/06/2008</i>	
<p>The contaminant was a „spent liquor“ obtained from an alumina refinery.</p> <p>Samples were dispensed by Dosino to eliminate human error in pipetting.</p>	Sample	Cl ⁻ g/L	
	Sea water as collected	19.07±0.05 (n=5)	
	Sea water contaminated with 2mL/L sodium aluminate solution (filtered)	19.13±0.01 (n=5)	
	Sea water contaminated with 4mL/L sodium aluminate solution (filtered)	19.07±0.03 (n=5)	
	Sea water contaminated with 10mL/L sodium aluminate solution (filtered)	19.04±0.07 (n=5)	

Calculations:

$$\text{Cl}^- \text{ g/L} = \frac{((\text{Titre, mL} - \text{blank, mL}) \times \text{AgNO}_3 \text{ mol/L} \times 35.4527)}{\text{aliquot, mL}}$$

Thermometric Titration Plot:

Legend:
 Red = solution temperature curve
 Black = second derivative curve (for endpoints)

