

Thermo. Titr. Application Note No. H-067

Title: Determination of Chloride in Bayer Process Liquor

Scope: Determination of chloride in Bayer Process liquor.

Principle: Neutralization of an aliquot of Bayer Process liquor with nitric acid, and consequent thermometric titration with 0.1 mol/L silver nitrate solution.

Reagents: *Titrant:* Standard 0.1 mol/L AgNO₃ solution
Concentrated nitric acid solution

Method: Basic Experimental Parameters:

Titrant delivery rate (mL/min.)	3
No. of exothermic endpoints	1
Data smoothing factor (DSF)	50
Stirring speed (802 stirrer)	10
Titrant pre-dose (mL) (low chloride)	0.2
Titrant pre-dose (mL) (high chloride)	0.5
Wait time after pre-dose (secs.)	30

In this exercise, a serial dilution of liquor was prepared in order to demonstrate the best precision of the method. However, where faster results are required, a direct 1mL aliquot taken by micropipette may be used.

A 10mL aliquot of liquor was diluted to 200mL in a volumetric flask with DI water. A 20mL aliquot of this solution was dispensed into a titrating vessel, 10mL of DI water added, and the contents then cautiously neutralized with 2mL concentrated HNO₃. The contents were then gently swirled to dissolve all precipitated solids.

The titration commences with an automatic pre-dose of titrant, followed by a waiting period. This allows formation of sufficient AgCl precipitate to allow the rapid deposition of AgCl during the main part of the titration, assisting in obtaining a sharp, reproducible endpoint.

Examples:
Liquors from different alumina refineries

Origin	Chloride expressed as...		
	Cl ⁻ g/L	NaCl g/L	Na ₂ CO ₃ g/L
Refinery #1	3.73±0.01 (n=7)	6.16±0.02 (n=7)	5.58±0.02 (n=7)
Refinery #2	10.26±0.01 (n=6)	16.92±0.02 (n=6)	15.34±0.02 (n=6)

Calculations:

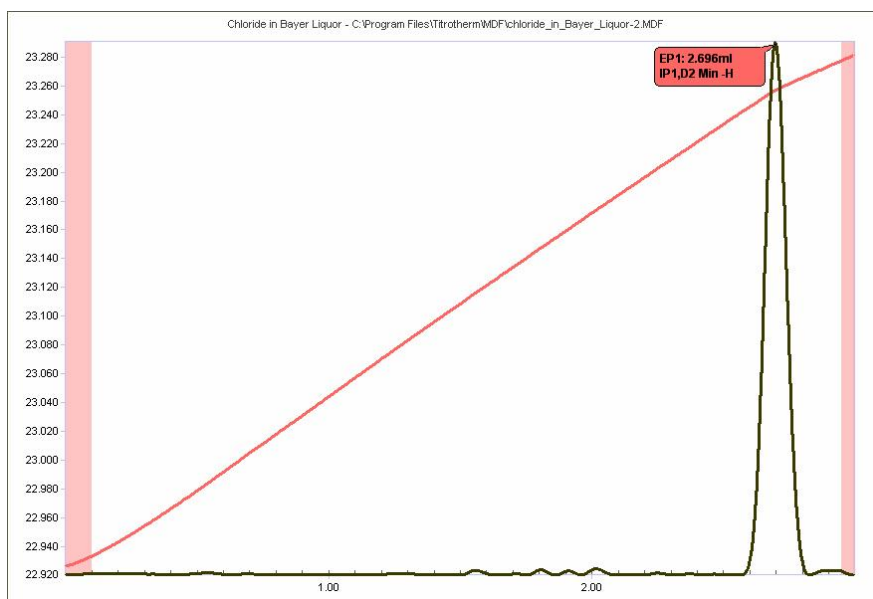
$$\text{Cl}^- \text{ g/L} = \frac{((\text{Titre, mL} - \text{blank, mL}) \times \text{AgNO}_3 \text{ mol/L} \times 35.4527)}{\text{aliquot, mL}}$$

$$\text{NaCl g/L} = \frac{((\text{Titre, mL} - \text{blank, mL}) \times \text{AgNO}_3 \text{ mol/L} \times 58.44247)}{\text{aliquot, mL}}$$

$$\text{Na}_2\text{CO}_3 \text{ g/L} = \frac{((\text{Titre, mL} - \text{blank, mL}) \times \text{AgNO}_3 \text{ mol/L} \times 105.9884)}{(\text{aliquot, mL} \times 2)}$$

Thermometric Titration Plot:
Legend:

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



Titration of chloride in Bayer liquor