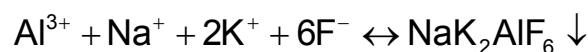


Thermo. Titr. Application Note No. H-053

Title:	Determination of Aluminium by Fluoride Titration
---------------	---------------------------------------------------------

Scope:	Determination of aluminium in acidic, basic and neutral solutions; including alum, etching solutions and aluminate solutions.
---------------	-------------------------------------------------------------------------------------------------------------------------------

Principle:	Titration with standard NaF solution in the presence of Na ⁺ and K ⁺ to give an exothermic reaction, forming insoluble NaK ₂ AlF ₆ .
-------------------	----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------



The NaF is standardized against an Al solution prepared from high purity Al metal.

Reagents:	<i>Titrant: 1 mol/L NaF solution.</i>
------------------	---------------------------------------

Conditioning reagent: Combined ionic adjustment and buffer solution

Dissolve 130.9 g anhydrous potassium acetate and 54.7 g anhydrous sodium acetate in 500 mL DI water. Add 115 mL glacial acetic acid, and make to 1 L with DI water. Alternatively, dissolve 164 g anhydrous sodium acetate and 75 g potassium chloride in 700 mL DI water, add 115 mL glacial acetic acid and make to 1 L with DI water.

10 % w/v sulfuric acid solution

Required for neutralization of basic solutions.

Method:	Basic Experimental Parameters:		
	Titrant delivery rate (mL/min.)	2	
	No. of exothermic endpoints	1	
	Data smoothing factor (DSF)	50	
	Stirring speed (802 stirrer)	6	
	Sample preparation:		
	1. <i>Basic aluminate solutions</i>		
	Take aliquots of aluminate liquor using the following table as a guide:		
	<i>Al conc. range</i> g/L	<i>Aliquot, mL</i>	<i>Suggested dilution</i>
	≤1	20	direct
	10	2	20→200 mL
	100	0.2	5→500 mL
	200	0.1	5→1000 mL
	Dispense the aliquot into a titration vessel. Place under the 802 stirrer, start the stirrer manually on speed 4 and slowly add 10 % w/v H ₂ SO ₄ solution until all precipitated hydroxyl gel has just dissolved. Add 10 mL pH 4.5 combined ionic adjustment buffer solution, and allow to cool to at least 30°C before commencing the titration. It is possible to program a set waiting period (to allow the solution to cool) before the titration commences.		
	2. <i>Aluminium salt solutions (neutral or slightly acidic).</i>		
	Prepare an aluminium salt solution of approximately 0.05 mol/L Al content. Pipette a 20 mL aliquot into a titration vessel, and add 10 mL of combined ionic adjustment buffer solution.		

Examples:

	Sample type	Al ₂ O ₃ g/L
	Bayer liquor (spent)	85.6±0.06 (n=6)
	Alum solution	4.76±0.0015 (n=5)

Calculations:

$$\text{Al g/L} = \frac{((\text{Titre, mL} - \text{blank, mL}) \times \text{NaF mol/L} \times 26.9815)}{(\text{aliquot, mL} \times 6)}$$

$$\text{Al}_2\text{O}_3 \text{ g/L} = \frac{((\text{Titre, mL} - \text{blank, mL}) \times \text{NaF mol/L} \times 101.9613)}{(\text{aliquot, mL} \times 6 \times 2)}$$

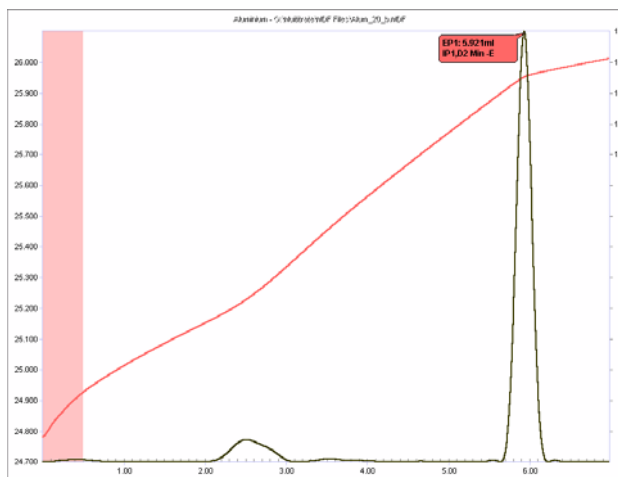
Thermometric Titration Plots:

Legend:

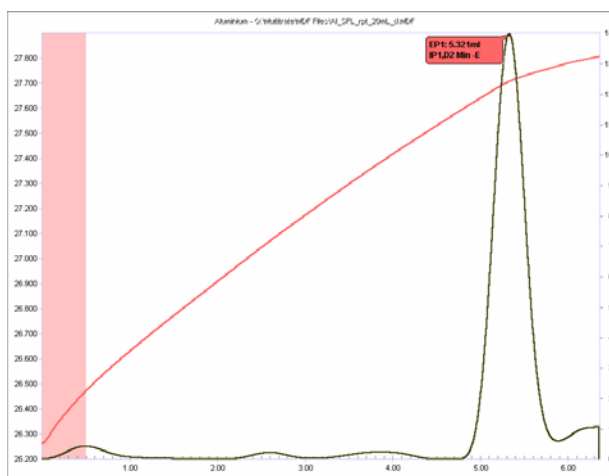
Red = solution

temperature curve

Black = second derivative curve (for endpoints)



Titration of alum solution



Titration of Bayer aluminate solution