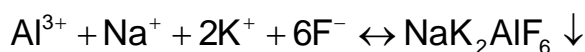


Thermo. Titr. Application Note No. H-087

Title: Determination of Hydrofluoric Acid by Aluminium Titration

Scope: Determination of hydrofluoric acid in mixed acid etchant solutions.

Principle: Titration with standard $\text{Al}(\text{NO}_3)_3$ solution in the presence of Na^+ and K^+ at $\sim\text{pH}4.5$ to give an exothermic reaction, forming insoluble NaK_2AlF_6 .



The $\text{Al}(\text{NO}_3)_3$ is standardized against A.R. anhydrous NaF.

Reagents: *Titrant: 1mol/L $\text{Al}(\text{NO}_3)_3$ solution.*

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

5 mol/L NaOH solution.

0.1% phenolphthalein solution in ethanol

Method:	<i>Basic Experimental Parameters:</i>	
	Titrant delivery rate (mL/min.)	2
	No. of exothermic endpoints	1
	Data smoothing factor (DSF)	45
	Stirring speed (802 stirrer)	10
	Wait period before titration commences(secs.)	60
	<i>Sample preparation:</i>	
	Weigh an amount of mixed acid polyetch etching solution into a titration vessel, and make to approximately 30mL with D.I. water. Mount on the 859 Titrotherm titration head, and commence stirring with the 802 stirrer manually (using Setup>Stirrer at a speed of 3. Add 2 drops of phenolphthalein solution, then through a port on the titration head, neutralize the solution dropwise with 5 mol/L NaOH solution until the appearance of a faint pink colour. Add 5mL of conditioning reagent/buffer, and start the titration sequence.	

Example:	Synthetic etch solution
	%HF w/w = 1.78±0.01 (n=8)

Calculations:
$\text{HF}\% = \frac{((\text{Titre, mL} - \text{blank, mL}) \times \text{Al}(\text{NO}_3)_3 \text{ mol/L} \times 20.00634 \times 6 \times 100)}{(\text{aliquot, g} \times 1000)}$

Thermometric Titration Plot:

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

