

Thermo. Titr. Application Note No. H-057

Title: Standardization of Sodium Fluoride for Aluminium Titrations

Scope: Standardization of sodium fluoride titrant for determination of aluminium.

Principle: Sodium fluoride titrant is standardized with a solution of aluminium prepared from high purity metal in the presence of Na⁺ and K⁺ at ~ pH 4.5 to give an exothermic reaction, forming insoluble NaK₂AlF₆.

$$\text{Al}^{3+} + \text{Na}^+ + 2\text{K}^+ + 6\text{F}^- \leftrightarrow \text{NaK}_2\text{AlF}_6 \downarrow$$

Reagents:

Titrant: 1 mol/L NaF solution

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.

Concentrated HCl

Primary standard Al solution, 0.4 mol/L

Prepare from 99.99 % pure Al metal turnings, which have been freshly degreased, washed and dried. Transfer 0.4 mole of Al (AW=26.9815) into a 1000 mL volumetric flask containing 100 g NaOH dissolved in 500 mL DI water. Place a small funnel in the neck of the flask to catch any spray. After the metal has completely dissolved, make to volume with DI water.

Secondary standard Al solution, 0.04 mol/L:

Using a volumetric (bulb) pipette, transfer 50 mL primary standard Al solution into a 500 mL volumetric flask. Add concentrated HCl in amounts of 1 mL to first precipitate Al(OH)₃ and then dissolve this precipitate. Towards the end of the dissolution, add the concentrated HCl dropwise until the last trace of precipitate just disappears. Cool the solution, and make to volume with DI water.

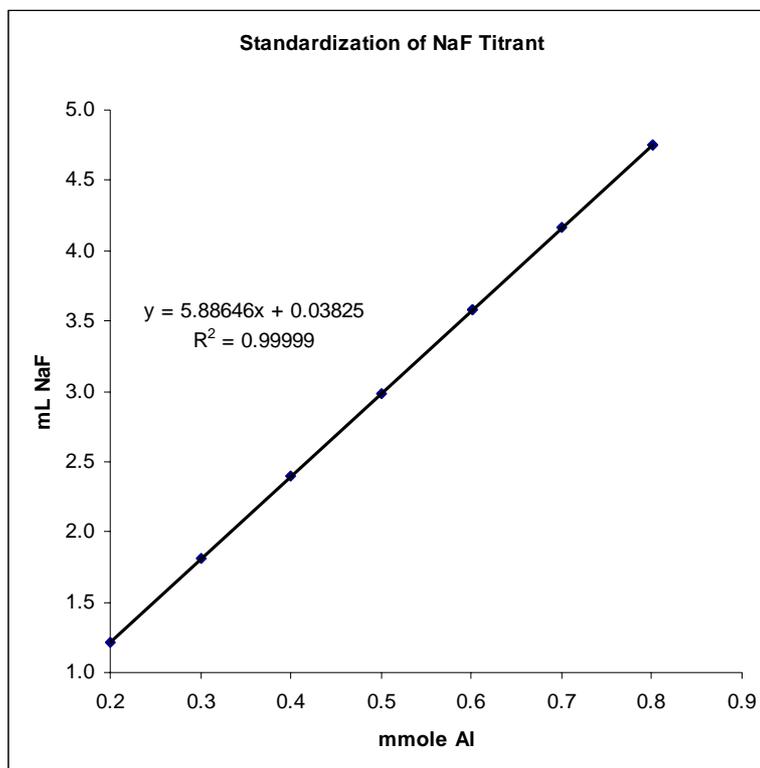
Method:	Basic Experimental Parameters:	
	Titrant delivery rate (mL/min.)	2
	No. of exothermic endpoints	1
	Data smoothing factor (DSF)	65
	Stirring speed (802 stirrer)	7
<p>Prepare and titrate a series of solutions using the acidic Al secondary standard according to the table below. Express the amounts of Al titrated as mmole, and plot against the corresponding titres of NaF solution. Note that 4 data points are regarded as a minimum. If a Dosino is used to dispense the standard Al solution, it is easy to create a calibration curve with more data points. In the example illustrated, 7 data points have been plotted.</p>		

Preparation of titration solutions:			
	<i>mL Al standard</i>	<i>mL DI water</i>	<i>mL acetate buffer</i>
	20	0	10
	15	5	10
	10	10	10
	5	15	10

**Example of calibration
curve:**

gradient = 5.88646

y-intercept ("titration
blank") = 0.03825 mL

**Calculation of titrant
molarity:**

Since 6 mole of F^- reacts with 1 mole Al^{3+} , NaF molarity =
 $6 \div \text{curve gradient} = 6 \div 5.88646 = 1.0193 \text{ mol/L}$