Thermo. Titr. Application Note No. H-062

Title:	**Standardization of Titrant for Direct Titration of Sodium**

***Scope*:**	Standardization of titrant for direct determination of sodium.

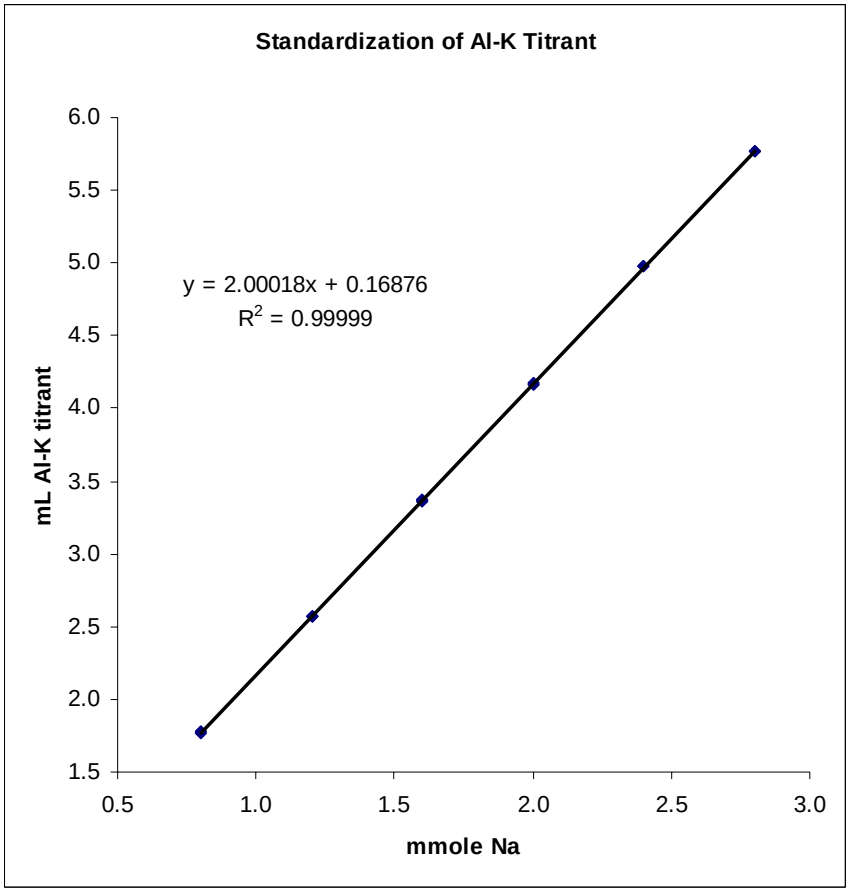
***Principle*:**	The direct titration of sodium is accomplished with a standardized solution of aluminium containing a stoichiometric excess of potassium ions in the presence of ammonium hydrogen difluoride at ~pH3; giving an exothermic reaction with the formation of insoluble NaK_2AlF_6 (elpasolite). $Al^{3+} + Na^+ + 2K^+ + 6F^- \leftrightarrow NaK_2AlF_6 \downarrow$ The titrant is standardized against a solution prepared from anhydrous sodium sulfate

Reagents:	*Titrant: Mixed 0.5mol/L $Al(NO_3)_3$, 1.1mol/L KNO_3 solution*. C*omplexing reagent: 300g/L $NH_4F.HF$* *Standard solution of sodium, 0.4mol/L:* prepared from A.R. anhydrous Na_2SO_4 (for dispensing from a Dosino)

Equipment List:	
	2.136.0010 859 Titrotherm (with 6.9011.040 Thermoprobe, fluoride resistant) 2.800.0010 Dosino 6.3032.210 Dosing unit to Dosino, 10mL 2.804.0010 804 Titration stand without stand rod 2.802.0010 Rod stirrer 6.2727.010 Intermediate SG sleeve 6.2026.010 Stand rod with base plate 6.2013.010 Clamping ring 6.1414.010 Titration vessel lid with 5 openings 6.1446.130 Stopper B14/15 (2 required) 6.1415.220 Titration vessel 20-90 mL 6.2061.010 Bottle holder for Dosinos 6.2065.000 Stacking frame for 846 Dosing Interface

Method:	*Basic Experimental Parameters:*	
	Titrant delivery rate (mL/min.)	4
	No. of exothermic endpoints	1
	Data smoothing factor (DSF)	45
	Stirring speed (802 stirrer)	9
	For highest precision, aliquots of standard sodium solution are dispensed from a second Dosino. However, the method may be adapted to the use of conventional bulb pipettes to dispense aliquots. In this case, a weaker solution of sodium sulfate should be prepared.	
	Using the second Dosino, set up a titration program to dispense (pre-dose) aliquots of 1, 1.5, 2, 2.5, 3 and 3.5mL 0.4mol/L Na_2SO_4 solution to titration vessels containing 25mL DI water and 5mL $NH_4F.HF$ reagent. Duplicate determinations for each dose volume of Na_2SO_4 solution may be performed to obtain results of higher precision.	
	Express the amount of Na_2SO_4 titrated in mmole, and plot titration results as illustrated in the calibration curve. Calculate the molarity of the Al component of the titrant from the gradient of the calibration curve.	

Calibration Curve:



Calculations:	*(refer to above calibration curve)*
	Molarity of Al component of titrant = 1÷gradient = 1÷2.00018 = 0.49996 mol/L Blank = y-intercept = 0.16876 mL

Thermometric Titration Plot:

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