Thermo. Titr. Application Note No. H-061

Title:	**Direct Titration of Sodium**

***Scope*:**	Determination of sodium in salts, process solutions and foods.

***Principle*:**	Titration 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 (see AN H-062)

Reagents:	*Titrant: Mixed 0.5mol/L $Al(NO_3)_3$, 1.1mol/L KNO_3 solution*. *Complexing reagent: 300g/L $NH_4F.HF$*

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 – 15 (depending on solution viscosity – see below)

Sample preparation – basic procedure: Take a measured quantity of sample such that the amount of sodium to be titrated is approximately 1.5 – 2mmole Na (equivalent to a titrant volume of approximately 3 – 4mL). Add 5mL $NH_4F.HF$ solution, and sufficient DI water to give a volume of approximately 30mL prior to titration. In the case of viscous samples, a starting volume of approximately 35-40mL is recommended. Stirrer speed should be increased for viscous samples and when larger starting volumes are used.

Where semi-solid samples such as ketchup, sauces and soups are to be analyzed, the sample should be stirred for 60 seconds prior to commencing the titration; to ensure proper dispersion and extraction of the sodium content. This can be programmed into the method.

Sample	*Sample Mass/volume*	*Stirrer speed*
Semi-solid foods (e.g., ketchup, sauces)	5 – 10g depending on Na content	10 - 12
Sodium tripolyphosphate, technical grade*	0.16g	
Sodium lauryl ether sulfate, technical grade (SLES)*	0.5g	

**In the case of the sodium salts, solutions were prepared and dosed from a Dosino. This was done for the purpose of preparation of this application note, so that titration errors in isolation from possible sample preparation errors could be studied. However, it is recommended that in the case of highly viscous samples such as SLES, a dilute solution should be prepared beforehand, due to their slowness in dispersion and dissolution.*

Where the sodium content is such that the titrant consumption is less than approximately 1mL, analytical precision can be increased by employing a standard addition technique. In this case, a second Dosino is charged with standard sodium sulfate solution. The titration program is modified to pre-dose a volume of standard solution to give a titrant consumption of approximately 3mL. This is the "blank" volume, and is subtracted from the volume of the subsequent sample titration which also contains the same volume of standard.

Examples:

Sample	*Result (as Na, units specified)*
Tomato ketchup	908±4 mg/100g, n=7
Sodium tripolyphosphate, technical grade	28.55±0.06% w/w, n=7
Sodium lauryl ether sulfate, technical grade (SLES)	4.10±0.04% w/w, n=7

Calculations:

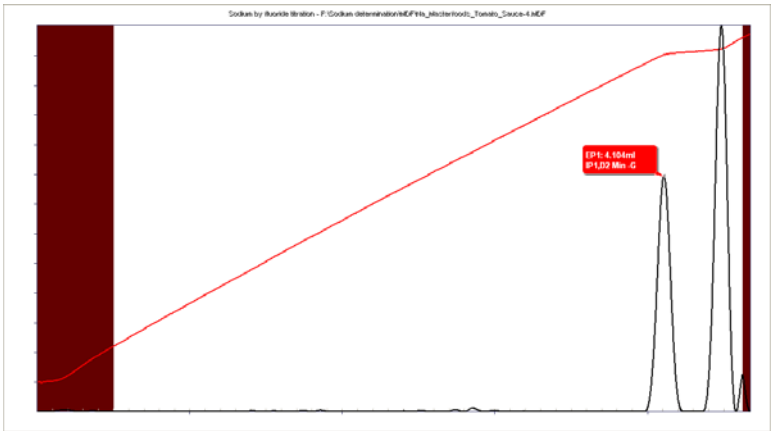
$$\text{Na g/L} = \frac{((\text{Titre, mL} - \text{blank, mL}) \times \text{Al mol/L} \times 22.98977)}{(\text{sample volume, mL})}$$

$$\text{Na\%} = \frac{((\text{Titre, mL} - \text{blank, mL}) \times \text{Al mol/L} \times 22.98977 \times 100)}{(\text{sample mass g} \times 1000)}$$

$$\text{Na mg/100g} = \frac{((\text{Titre, mL} - \text{blank, mL}) \times \text{Al mol/L} \times 22.98977 \times 100)}{(\text{sample mass g})}$$

Thermometric Titration Plots:

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

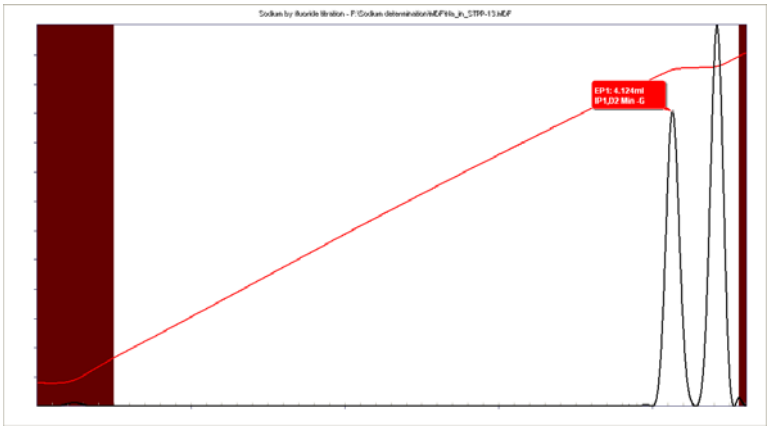


Sodium in tomato ketchup

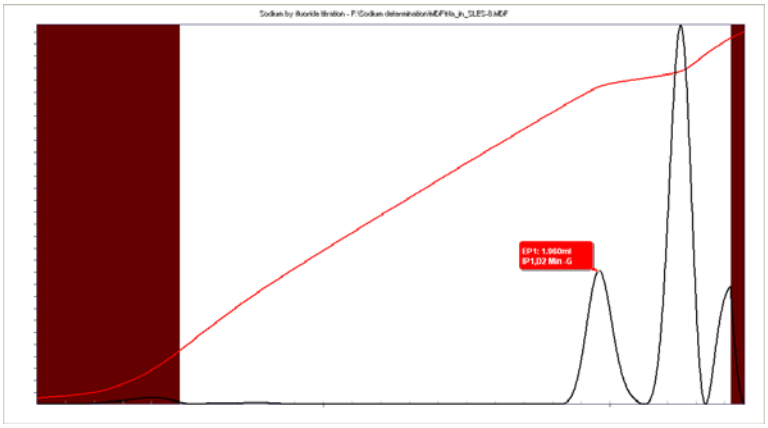
Notes on progress of titration:

(reference to red solution temperature curve).

After commencement of the titration, the solution temperature is stable until nuclei of NaK_2AlF_6 are formed.The precipitation then proceeds exothermically to the endpoint. The endpoint is marked by a change in gradient to a „plateau“. The temperature then starts to rise again with the formation of K_3AlF_6.



Sodium in sodium tripolyphosphate



Sodium in sodium lauryl ether sulfate