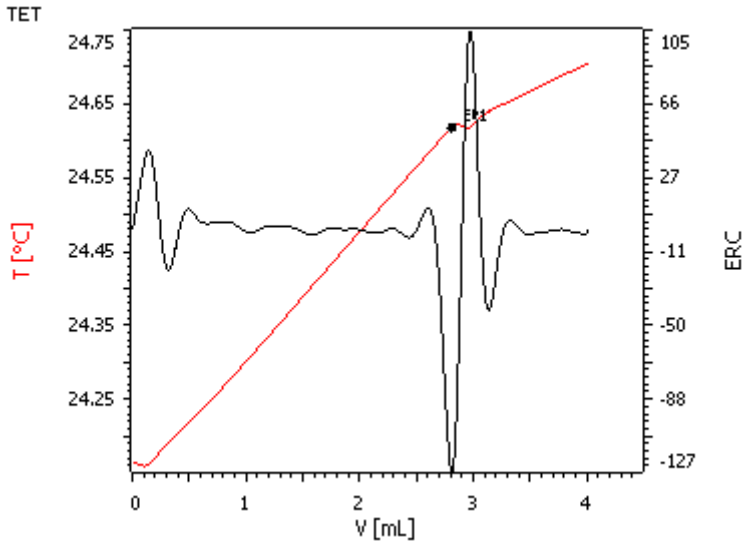
Titration Application Note H–124

Determination of sodium in margarine manufacture



This Application Note describes the determination of the total sodium content of precursor solutions used in margarine manufacture. These solutions are mixed with food fats and oils to make margarine, and may contain sodium chloride and other salts of sodium and potassium, some of which may be added in the form of emulsifiers, stabilizers, antioxidants, vitamins, coloring agents, and flavorings. It is more efficient and cost-effective for manufacturers to analyze (and, if need be, correct) the sodium content of these solutions rather than in the finished product.

Traditionally, argentometric titrations have been used to estimate the sodium content of these solutions by analyzing for chloride, and assuming all sodium is derived from sodium chloride. Clearly, argentometric titration does not account for other sources of sodium present in the solution, and the use of potassium chloride as a partial replacement for sodium chloride in some formulations leads to a further source of error. The direct titration of sodium by thermometric endpoint titration (TET) accounts for all sodium present and is not subject to interference from potassium.

Metrohm

Method description

Principle

Sodium ion reacts exothermically with aluminium ions in the presence of potassium and fluoride ions to form insoluble NaK_2AlF_6 («elpasolite»). Aluminium must be in the Al^{3+} ionic form.

The reaction may be used for the quantitative determination of total sodium in various foodstuffs.

Samples

Randomly selected samples of aqueous margarine precursor solutions were provided by a food manufacturer.

Sample preparation

The samples required no special preparation.

Configuration

Basic equipment list for automated titration

814 USB Sample Processor	2.814.0030
859 Titrotherm	2.859.0010
Sample rack 24 × 75 mL	6.2041.340
Thermoprobe HF resistant	6.9011.040
Sample beaker 75 mL	6.1459.400
802 Rod Stirrer	2.802.0010
Stirring propeller 104 mm	6.1909.020
2 × 800 Dosino	2.800.0010
1 × Dosing unit 10 mL	6.3032.210
1 × ETFE cylinder unit 10 mL 1 × Dosing unit 5 mL	6.1566.150 6.3032.150
tiamo™	6.6056.222

** Acidified solutions of fluoride ion are used in this determination*

Solutions

Titrant	$c(Al(NO_3)_3)$ = 0.5 mol/L aluminium nitrate solution prepared in a solution of $c(KNO_3)$ = 1.1 mol/L potassium nitrate
	$c(NH_4F)$ = 40% (w/v) ammonium fluoride in deionized water.
	c(HCl) = ~1 mol/L hydrochloric acid (does not require standardization
	c(NaCl) = 0.1 mol/L sodium chloride, for standardization of the $c(Al(NO_3)_3)$ = 0.5 mol/L aluminium nitrate solution

Analysis of samples

Between 0.5 and 2.5 g of sample is weighed into a clean, dry titration vessel to obtain a titration volume at the endpoint of between approximately 1 to 2.5 mL of titrant. The amount required may vary according to the formulation and may require some initial experimentation to obtain optimal sample masses for different solution formulations. The solution is diluted with 25 mL of c(HCl) = ~1 mol/L. Alternatively, 2 mL concentrated HCl and 25 mL deionized water may be used to dilute and condition the sample.

The suspension is then titrated with standardized $c(Al(NO_3)_3)$ = 0.5 mol/L solution after automated addition of 5 mL $c(NH_4F)$ = 40% (w/v) solution.

Initial determination of reagent blank

The blank determination is only required for the initial setup for analysis of this type of food product.

Five separate titrations are performed on a sample regarded as typical by the analyst. For example, for a solution where the sodium content is approximately 1% (w/w), samples masses ranging from ~1 to ~2.5 g in roughly equal increments may be used.

A regression analysis is performed on the results, with the x-axis denoting sample mass in g, and y-axis titration endpoint volumes in mL.

Standardization of titrant

Aliquots of 5, 10, 15, 20, and 25 mL of 0.1 mol/L c(NaCl) are pipetted into titration vessels and diluted with deionized water to bring the volume to ~25 mL.

To each vessel, 1 mL of concentrated HCl is added. The solutions are titrated under the same conditions as for the samples.

A regression analysis is performed, with the amount of NaCl titrated (as mmol) plotted on the x-axis, and the volume of $c(Al(NO_3)_3)$ = 0.5 mol/L in mL plotted on the

Metrohm

Method description

y-axis. The molarity of the Al^{3+} solution is calculated from the reciprocal of the gradient. A dedicated *tiamo*™ program has been created to automatically perform this standardization.

Parameters

Basic experimental parameters

Titrant dose rate (mL/min)	2
ERC EP1 (exothermic)	–8
Data smoothing («filter factor»)	47
Stirring speed (802 Rod Stirrer)	15
Evaluation start (mL)	0.7
Damping until (mL)	0.7

Calculations

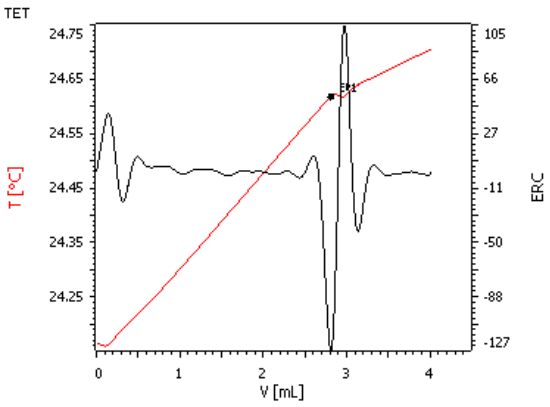
% Na (w/w) = ((EP1 – blank) × C001 × C002 × 0.1)/C00

EP1 = endpoint in mL
blank = titration blank , mL
C00 = sample mass in g
C001 = concentration of titrant in mol/L
C002 = atomic mass of Na (22.98977 g/mol)

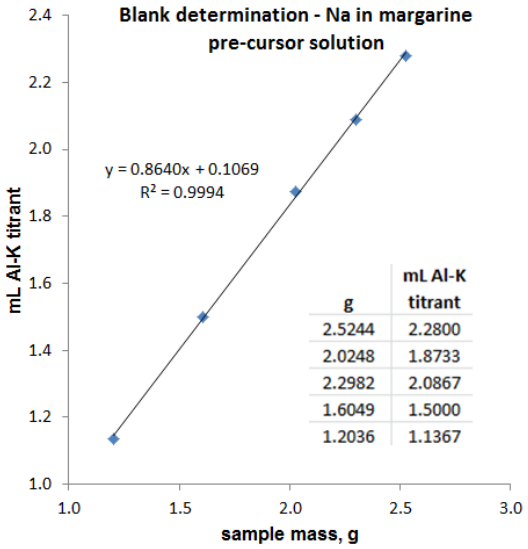
Results

Sample ID	% Na (w/w)
1	2.96, 2.96
2	0.94, 0.94
3	1.06 ± 0.01 (n = 5)
4	0.97, 0.97
5	0.97, 0,97
6	1.02 ± 0.01 (n = 8)

Titration plot



Blank determination



Blank = y – intercept = 0.1069 mL

Metrohm