

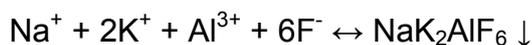
Thermo. Titr. Application Note No. H-112

Title: Determination of sodium in cheeses

Scope: Determination of total sodium in commercial cheeses

Principle: Samples of shredded, grated, or sliced cheese are dispersed with a high-speed disintegrator in a solution of trichloroacetic acid, which denatures protein and assists in the liberation of all sodium from the matrix. Toluene is added in a second step of the dispersion process to assist in the solubilization of fat.

Ammonium fluoride solution is then added, and the sodium titrated with a titrant comprising 0.5 mol/L $\text{Al}(\text{NO}_3)_3$ and 1.1 mol/L KNO_3 to an exothermic endpoint.



In this determination, ammonium fluoride (NH_4F) has been found to give sharper endpoints than ammonium bifluoride ($\text{NH}_4\text{F} \cdot \text{HF}$)

Reagents: Mixed titrant: $c(\text{Al}) = 0.5 \text{ mol/L Al}(\text{NO}_3)_3$, $c(\text{K}) = 1.1 \text{ mol/L KNO}_3$
Na standard: $c(\text{Na}) = 0.25 \text{ mol/L NaCl}$
 $c(\text{CCl}_3\text{COOH}) = 300 \text{ g/L trichloroacetic acid}$
 $c(\text{NH}_4\text{F}) = 400 \text{ g/L ammonium fluoride}$
A.R. Toluene

Method: *Basic Equipment List (for automated titration):*
2.136.0100 Polytron PT 1300D
2.859.0010 859 Titrotherm
2.814.0020 814 USB Sample Processor
2.800.0010 800 Dosinos (2 required - minimum)
6.157.5150 Dosing Unit complete, 5mL
6.303.2210 Housing for 10 mL cylinder unit
6.156.6210 ETFE cylinder unit with piston and valve disk, 10 mL
6.901.1040 Thermoprobe (fluoride resistant)
6.123.6050 Sleeve with SGJ 14/12mm
2.802.0010 Rod Stirrer
6.204.1340 Sample rack, 24 places
6.145.9400 Sample beaker 75mL (144 pieces)
6.190.9010 Propeller stirrer, 102mm
6.145.8070 Titration head 2 x SGJ14

Basic Experimental Parameters:

Titration delivery rate (mL/min)	4
No. of exothermic endpoints	1
Data smoothing factor	25
ERC (2 nd derivative)	-30
Stirring speed (802 Rod Stirrer)	15
Delay before start (s)	30

Basic Method:

Depending on total sodium content, weigh accurately between 2 and 5 g cheese sample into a titration tube (the aim is to obtain an endpoint volume approximately in the range 2–4 mL). To assist in rapid dispersion, grate block cheeses, or cut sliced cheese into approximately 5 mm squares.

Add 10 mL 300 g/L trichloroacetic acid and 10 mL DI water. Disperse for 30 seconds at 20,000 rpm until a homogenous milky fluid is obtained. Add 5 mL toluene, and disperse for another 30 seconds (this prevents build-up of cheese residue on the titration head). Wash the dispersing tool with minimum DI water (maximum 10 mL) into the titration tube.

Titrate using method “AA Sodium in oily foods.mmet”

Determination of blank.

In “Configuration”, prepare a Common Variable (CV) “blank Na by AI”.

Prepare 5 titration tubes as detailed in “basic method”, weighing accurately in approximately equal increments. For example, if a 5 g sample of cheese is found to give titration volumes of approximately 3 mL, weigh accurately amounts approximating 1, 2, 3, 4, and 5g. Titrate using method “AB Sodium in oily foods.mmet”. The blank value is automatically computed by **tiamo**TM, and updated as the CV.

Standardization of Al(NO₃)₃, KNO₃ titrant.

Prepare a Dosino with a 10 mL dosing unit with c(Na) = 0.25 mol/L NaCl solution, which has been prepared from A.R. NaCl (dried at 120 °C for 2 hours) and DI water. Using automated method “AS Al-K titrant with NaCl standardization.mmet”, prepare an automation program where 2, 4, 6, 8, and 9 mL of 0.25 mol/L NaCl solution are dispensed into successive titration tubes containing 10 mL 300 g/L trichloroacetic acid and 13, 11, 9, 7, and 6mL DI water, respectively.

After titration of the set of solutions, the molarity of the titrant is computed automatically by **tiamo**TM.

Examples:

Processed cheese purchased from a supermarket

Cheese	Na, mg/100 g	
	Label value	Found
Shredded parmesan	1060	986±3
Shredded tasty cheese	610	732±5
Light slices	600	684±3
Colby slices	668	725±9

Calculation:

$$\text{mg Na/100g} = ((\text{EP, mL} - \text{blank, mL}) * c(\text{Al}^{3+}) * \text{AW Na} * 100) / \text{sample mass, g}$$

Standardization

Standardized against 0.2501 mol/L NaCl solution

$$c(\text{Al}^{3+}) = 0.5143 \text{ mol/L}$$

$$\text{Coefficient of correlation (R}^2\text{)} = 0.9999$$

Blank determinations

Determined on shredded tasty and parmesan cheeses

	Tasty cheese	Parmesan
Blank, mL	0.0556	0.0551
Coeff. of det. (R ²)	0.9997	0.9999

Thermometric Titration Plots:

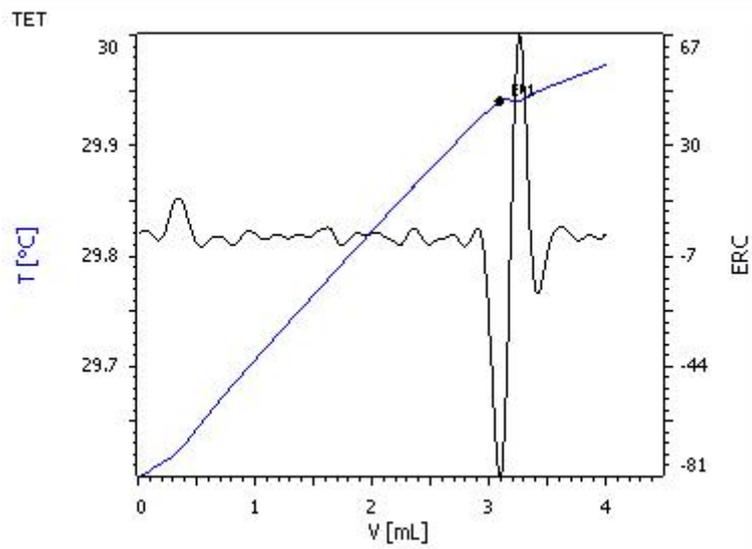


Fig. 1. Na in "light" sliced cheese.

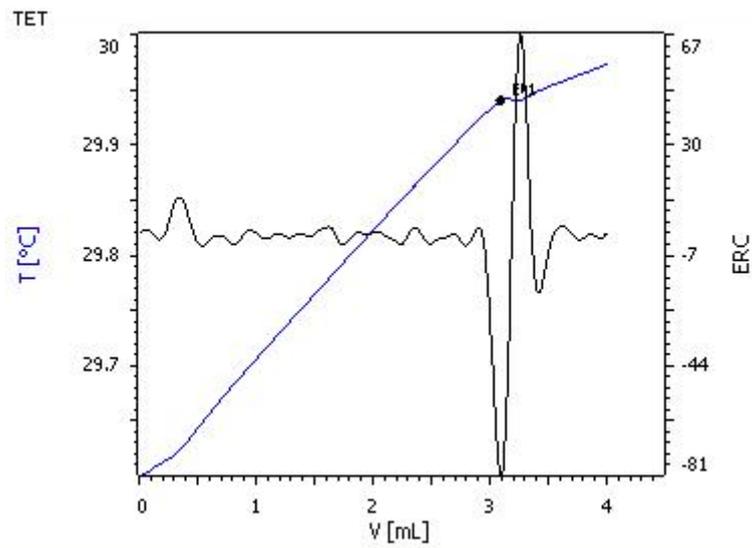


Fig. 2. Na in "Colby" sliced cheese

Legend:

Blue = solution temperature curve

Black = second derivative curve