Fully automatic determination of sodium in food samples – Know the "true" sodium content

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Titration

Summary

Sodium is one of the most common inorganic cations in food that is mostly added as sodium chloride. As such, it preserves or flavors food. Excess sodium, however, increases the risk of health problems such as cardiovascular diseases. An increasing health awareness and the widespread prescribed labeling of foodstuffs with nutritional information drive demand for direct sodium determination methods.

Thermometric titration is a promising method for the straightforward sodium determination in foodstuffs. Enthalpy change can be monitored as a change in temperature of the solution using a sensitive digital thermometer. The determination described relies on the exothermic precipitation of elpasolite (NaK_2AlF_6):

$$Na^+ + 2\,K^+ + Al^{3+} + 6\,F^- \rightleftharpoons NaK_2AlF_6$$

The titrant is a standard aluminum solution which contains an excess of potassium ions. The required excess of fluoride ions is provided by ammonium fluoride, which also serves to buffer the solution to an ideal pH 3. The method is robust, can be fully automated, and not least due to the highly reproducible high-frequency homogenization, copes with challenging food matrices such as ketchup, instant soups, gravies, and salty snacks. The titration is performed directly on a suspension of the food sample and is completed in less than two minutes. Relative standard deviations for ketchup, soups, gravies, and several salty snacks samples were smaller than 3.75%.

Introduction

The sodium content of foods is usually determined indirectly using a precipitation reaction with silver nitrate: $AgNO_3 + Cl^- \rightarrow AgCl\downarrow + NO_3^-$. The amount of sodium is typically calculated by assuming a 1:1 molar ratio of chloride ions to sodium ions. This is not necessarily the case when common sodium-containing food ingredients such as sodium benzoate and monosodium glutamate or chloride-containing ingredients such as potassium chloride are present in the food matrix.

Common methods of direct testing of sodium include atomic absorption spectroscopy or inductively coupled plasma spectroscopy. These techniques involve significant capital investments in equipment and infrastructure, costly ultrapure reagents and lengthy sample preparation, and system calibration.

Thermometric titration uses the rate of change in temperature of the titration solution to detect the endpoint. This method of titration is free from interfering electrochemical and solvent effects that are present in many types of titration.

In a titration, the titrant reacts with the analyte in the sample either exothermically (gives off heat) or endothermically (absorbs heat). A sensitive temperature sensor with a resolution of 10^{-5} K (Thermoprobe) measures the temperature of the titrating solution. When all of the analyte in the sample has reacted with the titrant, the temperature of the solution will change and the endpoint of the titration is revealed by an inflection in the temperature curve. The amount of analyte determined is not related to the change in temperature of the solution. Therefore, it is not necessary to use insulated titration vessels.

Typical snack food samples such as crackers, corn chips, pretzels, and other foodstuffs such as ketchup, soups, and gravies have been analyzed providing highly accurate results.

Instrumentation

- 859 Titrotherm
- 815 Robotic USB Sample Processor
- Polytron PT 1300 D (Homogenizer)

Sample preparation

- Before titration, titrant, complexing agent, and standard solution have to be prepared.

  $Al(NO_3)_3/KNO_3$ (0.5 mol/L/1.1 mol/L)
  187.57 g $Al(NO_3)_3$ and 111.22 g KNO_3 are weighed into a 1000 mL volumetric flask, dissolved in approximately 800 mL ultrapure water and made up to volume.

  Complexing agent NH_4F (400 g/L)
  400 g/L NH_4F are weighed into a 1000 mL volumetric flask and filled up to the mark with ultrapure water

  Na_2SO_4 standard solution (0.4 mol/L)
  28.408 g Na_2SO_4 that was previously dried for 2 hours at 105 °C is added to a 500 mL volumetric flask and diluted to the mark.

- Determination of the titer of the titrant:

  1…2.5 mL sodium sulfate solution, 5 mL complexing agent, and 40 mL ultrapure water are dosed into the titration vessel. This mixture is titrated with an $Al(NO_3)_3/KNO_3$ solution to the first endpoint. Using the consumption of the titrant and the sample size, the ***tiamo***™ software automatically calculates the linear regression.

- Determination of the blank:

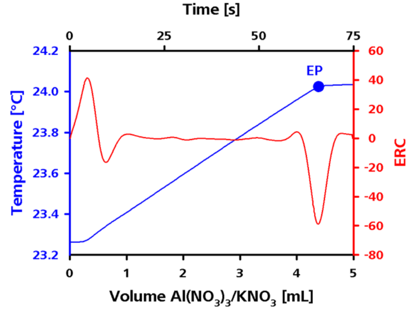
  The method blank is determined by titrating different sodium contents of the same sample. Afterwards the titrant consumption (y) is plotted against the sodium concentration (x): the y intercept of the linear regression curve is the method blank. The latter is entered along with the other method parameters in the ***tiamo***™ software.

- Before weighing 0.2…5 g sample, crush samples with a mortar.
- 5 mL complexing agent, 2 mL glacial acetic acid, and 40 mL water are automatically added
- After thorough comminution with the Polytron PT 1300 D that is mounted to the Sample Processor, the thermometric titration with the $Al(NO_3)_3/KNO_3$ solution is carried out to the first end point.

The ***tiamo***™ software automatically subtracts the blank value and calculates the sodium content.

Sodium in ketchup – Titration curve

The thermometric titration curve displays the temperature change during the addition of the $Al(NO_3)_3/KNO_3$ titrant (blue curve) to the ketchup sample. The inflection indicates the endpoint of the titration and is determined by the second derivative (red curve) of the temperature curve.



Sodium in various foodstuffs – Results

Thermometric sodium titration was tested for its applicability to various food matrices such as soups, gravy, and several salty snacks.

	Sample	Sodium content: Determined (n = 6) [%]	RSD [%]	Manufacturer´s data [%]
	Ketchup	1.26	0.19	1.22
	Bouillon soup	16.04	0.08	16.13
	Bouillon cube	18.02	1.89	16.13
	Gravy	6.34	0.95	6.67
	Crackers	1.17	0.50	0.98
	Corn chips	0.51	3.75	0.50
	Pretzel sticks	1.81	1.02	1.81

References

(1) Metrohm Application Bulletin AB-298, Automated sodium determination in various foods with 859 Titrotherm, (downloadable under http://www.metrohm.com/com/Applications).
(2) T. Smith, Metrohm Monograph: Practical thermometric titrimetry, Metrohm International Headquarters, Herisau, Switzerland, 34 pages (2006).

Download a copy of this poster from http://www.metrohm.com/com/Applications (search for 8.000.6067EN).

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