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 prescribing 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 epalosilite (NaK₃AlF₆).

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.

\[
\text{Al(NO}_3\text{)}_3/\text{KNO}_3 (0.5 \text{ mol/L}/1.1 \text{ mol/L})
\]

300 g Na₂SO₄ solution is weighed for two hours at 105°C and added to a 500 mL volumetric flask, dissolved in approximately 800 mL ultrapure water and made up to volume.

Complexing agent NH₄HF (300 g/L)

300 g NH₄HF are weighed into a 1000 mL volumetric flask and filled up to the mark with ultrapure water.

The titrant is a standard aluminum solution which contains an excess of potassium ions. The required excess of fluoride ions is provided by ammonium bifluoride, 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: AgNO₃ + Cl⁻ → AgCl + NO₃⁻. 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 10⁻⁴ 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.

Sodium in ketchup – Titration curve

The thermometric titration curve displays the temperature change during the addition of the Al(NO₃)₃/KNO₃ 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.

Summary

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.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Determined (n = 6)</th>
<th>RSD (%)</th>
<th>Manufacturer’s data (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ketchup</td>
<td>1.26</td>
<td>0.19</td>
<td>1.22</td>
</tr>
<tr>
<td>Bouillon soup</td>
<td>16.04</td>
<td>0.08</td>
<td>16.13</td>
</tr>
<tr>
<td>Bouillon cube</td>
<td>18.02</td>
<td>1.89</td>
<td>16.13</td>
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<tr>
<td>Gravy</td>
<td>6.34</td>
<td>0.95</td>
<td>6.67</td>
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<tr>
<td>Crackers</td>
<td>1.17</td>
<td>0.50</td>
<td>0.98</td>
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<tr>
<td>Corn chips</td>
<td>0.51</td>
<td>3.75</td>
<td>0.50</td>
</tr>
<tr>
<td>Pretzel sticks</td>
<td>1.81</td>
<td>1.02</td>
<td>1.81</td>
</tr>
</tbody>
</table>

References