

its salt

In the presence of an excess of potassium and fluoride ions, sodium reacts exothermically with aluminum to produce NaK₂AlF₆ (elpasolite). This reaction is the cornerstone of a new method for determining sodium in foodstuffs using thermometric titration. Not only is it rapid, straightforward, and reliable, but the method can also be automated which makes it ideal for routine checks – even within the process itself.

Absorbing large quantities of sodium in the diet – through table salt, for example – can have a negative impact on health. Numerous jurisdictions have responded to this issue by ruling that food packaging must specify sodium content.

That was then: The trials and tribulations of titration

To date, titrimetric sodium has commonly been determined using an indirect method that determines not the sodium, but in fact the counterion chloride using argentometric titration. In the end, it is the stoichiometry of sodium chloride that is used to deduce the sodium content. There is one flaw in this method, however, which is that not all sodium in food occurs with chloride as a counterion; as a result, it tends to underestimate the actual sodium concentration.

Sajó¹ was the first to use the exothermic precipitation of elpasolite for the purpose of sodium determination. This enthalpimetric method is based on determining the temperature difference in the sample, which arises during the following reaction:

$$Na^{+} + 2K^{+} + AI^{3+} + 6F^{-}$$
 NaK₂AIF₆

The temperature rise in the sample correlates to the quantity of sodium. Obtaining an accurate result requires exceptionally precise calorimetric measurements, but these have to be performed by highly skilled laboratory staff and are simply too cumbersome to be included in routine analysis.

This is now: The same chemistry, a new method

The new, thermometric sodium determination method, published in the Journal of Agricultural Chemistry and Environment, uses the same titration reaction described by Sajó. For the sensor, it also uses a thermometer, which is located in the titration vessel; however, the temperature measurement only serves to determine the endpoint. As the reaction that takes place is exothermic, the temperature rises, and the point at which the temperature curve bends marks the endpoint. Ultimately, the sodium quantity is calculated on the basis of the amount of titrant consumed up to the endpoint and the reaction stoichiometry. This makes it a much simpler method to execute – and more reliable as a result.

Procedure details

Titration is performed using a solution that contains aluminum and potassium in a ratio of 1:2.2. In contrast to the stoichiometric ratio of the titration reaction, potassium is present at a 10% surplus level. Aluminum is the limiting reagent, which means that the titer determination takes place against an aluminum standard. An excess of fluoride, which is added to the titrant in the form of NH₄F or NH₄F·HF, shifts the reaction equilibrium to the right.

The sample preparation stage is usually straightforward, assuming it is needed at all. It is only necessary to ensure that the matrix does not prevent the analyte from reacting with the titrant, that the sample is sufficiently mobile, and that the sample does not contain any substances that might distort the stoichiometric endpoint determination.



An example: Instant noodles

Figure 1 shows the titration curve for a sodium determination in instant noodles. The noodles are first broken up in an electric mixer until they achieve the consistency of coarse flour. A few minutes' incubation in trichloroacetic acid (TCA) releases sodium from the matrix and the sample is then homogenized. The subsequent addition of acetone prevents the starch granules from swelling, thus ensuring that miscibility is maintained. Achieving precise and reproducible results is a matter of minutes with the 859 Titrotherm.

Conclusion

Determining the sodium content of food by thermometric titration provides accurate results, involves very little work, and can be fully automated – making it the perfect choice for routine analysis applications. Ready-to-use procedures are already available for a whole host of foodstuffs. The original publication can be downloaded free of charge from the Journal of Agricultural Chemistry and Environment website.

Reference literature

[1] Sajó, I. (1969) Magyar Kemiai Folyoirat 75, 1–3

Source: Smith, T. and C. Haider (2014) J. Agric. Chem. Environ. 3(1B) 20–25

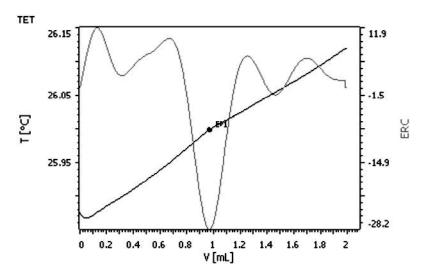


Figure 1. Thermometric titration of sodium in instant noodles. The bend in the temperature curve (black), which can be identified more clearly as the minimum of the second derivative (gray), marks the endpoint.