Methodologies for the determination of Salt (as Sodium Chloride), and elemental Sodium analyses.

Raymond Hartley
Introduction

Methodologies for the determination of Salt (as Sodium Chloride), and elemental Sodium.

• Titrimetric analyses
• Potentiometric titrators
• Atomic absorption spectroscopy
• Inductively coupled plasma spectroscopy
Salt Analyses

Salt analyses (NaCl):

• Titrmetric analyses:
  • Mohr titration, AgNO₃ using KCr indicator
  • Volhards method

• Potentiometric titrators (auto titrators)
Potentiometric titrators

•Potentiometric titration is a volumetric method which measures the potential between two electrodes.

•The basic principle of Potentiometric titration is the determination of unknown concentration of analysed solution by titration with some standard solution, where a change of indicator electrode potential is shown, and determines the equivalence point of the titration.

•Instrumental determination of the final point has several advantages over the usage of visual detection-indicator. The visual determination of the end point of titration is influenced by subjective factors, and cannot be used in coloured solutions. Potentiometric methods of determination of the equivalence point of titration can be used not only with acid-basis titrations but in precipitation, oxidation-reduction and other titrations.
# Ti Application Note No. T-4

<table>
<thead>
<tr>
<th>Title:</th>
<th>Salt content (NaCl) of meat products</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Summary:</strong></td>
<td>Determination of chloride in meat products by potentiometric titration with silver nitrate using the Ag Titrode.</td>
</tr>
<tr>
<td><strong>Sample:</strong></td>
<td>Meat products (ham, dried meat, sausages, fish etc.)</td>
</tr>
<tr>
<td><strong>Sample Preparation:</strong></td>
<td>Cut up the sample into small pieces. Weigh about 10 g sample (precision 1 mg) into a mixer. Add 190 g distilled water and mix for 1...2 min.</td>
</tr>
<tr>
<td><strong>Instruments and Accessories:</strong></td>
<td>702, 716 or 736 Titrino or 726 Titroprocessor, 6.0430.100 Ag Titrode</td>
</tr>
<tr>
<td><strong>Analysis:</strong></td>
<td>Weigh 50 g homogenisate into a beaker, add 50 mL distilled water and 2 mL c(HNO₃) = 2 mol/L and titrate with c(AgNO₃) = 0.1 mol/L.</td>
</tr>
<tr>
<td><strong>Calculation:</strong></td>
<td>% NaCl = EP1 * C01 * C02 * C03 / C00</td>
</tr>
<tr>
<td></td>
<td>EP1 = titrant consumption in mL</td>
</tr>
<tr>
<td></td>
<td>C00 = ca. 2.5 (g of original sample contained in the sample volume used for the titration)</td>
</tr>
<tr>
<td></td>
<td>C01 = 0.1 (concentration of the titrant in mol/L)</td>
</tr>
<tr>
<td></td>
<td>C02 = 58.44 (M(NaCl) in g/mol)</td>
</tr>
<tr>
<td></td>
<td>C03 = 0.1 (conversion factor for %)</td>
</tr>
<tr>
<td><strong>Remarks:</strong></td>
<td>For samples containing more than 4.5 % NaCl, a smaller aliquot of the homogenisate has to be used for the analysis.</td>
</tr>
</tbody>
</table>
Elemental Sodium Analyses

Regulation stipulates the use of:

• AA - Atomic Absorption Spectroscopy
• ICP - Inductively Coupled Plasma Spectroscopy

(MCLS, ICP-OES, microwave digestion)
Atomic Absorption Spectroscopy

• The techniques of analytical atomic absorption spectrophotometer have been developed intensively since the first studies in 1955. The method rely on the absorption of light by free dissociated atoms, produced either in a flame or a simple thermal dissociation of inorganic material.

• Most AA analyses are performed in flame atomizers. Here the sample in liquid form is reduced to a spray of fine droplets and introduced into a flame, where high temperature chemical reactions destroy any organic matter in the sample and dissociate inorganic compounds into free atoms.

• Atoms of each element absorb light at characteristic wavelengths, and by measuring the absorption at a given wavelength, the concentration of the particular element in the sample is determined.
AA – Na analyses

Lamp current: 5mA
Fuel: Propane/air-acetylene
Support: Air
Flame Stoichiometry: Oxidizing: blue cone 1,5cm

Air-acetylene flame can also be used to provide an improved signal/noise ratio.

Interferences:
Sodium is partially ionized in the air –acetylene flame. To suppress ionization, add potassium nitrate or chloride solution.
Description of ICP Optical Emission Spectrometry (ICP-OES)

• It has been 25 years since ICP optical emission spectrometer (ICP-OES) began to be widely used, and is now one of the most versatile methods of inorganic analysis.

• Its features are often compared to atomic absorption spectrometer. Compared to atomic absorption spectrometer, in which the excitation temperature of air-acetylene flame measures 2000 to 3000 K, the excitation temperature of argon ICP is 5000 to 7000 K, which efficiently excites many elements. Also, using inert gas (argon) makes oxides and nitrides harder to be generated.
ICP-OES

• Inductively coupled argon plasma optical emission spectrometers (ICP-OES) uses an Echelle optical design and a Charge Injection Device (CID) solid-state detector to provide elemental analysis.

• Most samples are liquids that are pumped through a nebuliser to produce a fine spray. The larger droplets are removed by a spray chamber and the small droplets then pass through to the plasma.

• The solvent is evaporated. The residual sample decomposed to atoms and ions that become excited and emit characteristic light which is measured, giving measurement of the concentration of each element type in the original sample.
Sample Introduction

• To generate plasma, first, argon gas is supplied to the torch coil, and high frequency electric current is applied to the work coil at the tip of the torch tube.

• Using the electromagnetic field created in the torch tube by the high frequency current, argon gas is ionized and plasma is generated. This plasma has high electron density and temperature (10000K) and this energy is used in the excitation-emission of the sample.

• Solution samples are introduced into the plasma in an atomized state through the narrow tube in the centre of the torch tube.
Instruments

• ICP-OES
Conclusion

• Salt analyses
• Elemental sodium analyses
• Caution
  • Substitutes/replacements
  • Pathogens
  • Increase hygiene surveillance
THANK YOU!