Salt in Food Products

DOC316.52.93096

Based on Standard ISO 1738.1997 and International Standard ISO 1841-2

1. Introduction

Salt (NaCl) is present in many foods such as meats, canned products, dried soups and dairy produce.

As NaCl is soluble in water, the most common way to determine the amount of salt is a titration with silver nitrate after sample dissolution in water.

For the determination of salt in food products, two applications are proposed depending on the type of sample (liquid or solid food), and on the sample amount unit (weight for solid food or volume for liquid food).

2. Principle

The silver nitrate reacts with chloride ion according to:

$$Ag^+ + Cl^- \rightarrow AgCl_{(precipitate)}$$

This application uses a potentiometric titration with a combined silver/reference electrode and the equivalence point is detected using the inflexion point mode.

The titration is performed in an acidic solution.

The results are expressed as % of sodium chloride or in g/L, depending on the sample type (the molar weight of NaCl is 58.443 g/mol).

3. Electrode and reagents

- **MTC306** Intellical combined metal/reference ($Ag/AgNO_3$) electrode with integrated temperature sensor.
- **Titrant: $AgNO_3$ 0.1 mol/L**
  Dry $AgNO_3$ for 2 hours at 105 °C and leave it to cool to room temperature in a desiccator. Using a volumetric flask, dissolve an accurate weight of about 16.987 g of $AgNO_3$ in 1000 mL of deionized water. Store the solution in a brown glass bottle.
- **Reagent: HNO_3 1 mol/L**
  Dilute 78 mL of concentrated nitric acid (65%) in 800 mL of deionized water and adjust the volume to 1000 mL with deionized water. This operation is highly exothermic. Observe laboratory safety regulations.
- **Standard solution for titrant calibration: NaCl 0.1 mol/L**
  Dry the pure NaCl at 105 °C and leave it to cool to room temperature in a desiccator. Dissolve an accurate weight of about 5.844 g of NaCl in 1000 mL of deionized water using a volumetric flask.
- **Deionized water**

4. Ranges and settings

4.1. Default parameters

The working procedure is described using the following parameters:

- V sample = 50 mL
- Burette volume = 10 mL

4.2. Working range

With the 10 mL burette, for an amount of solid sample between 1 and 5 g, the working range is 0.1 to 5% NaCl.

For an amount of liquid sample of 50 mL, the working range is 0.1 to 1 g/L NaCl. For liquid food, it is possible to measure sample with a higher concentration by using a smaller amount of sample and dilute it to 50 mL with deionized water.

For the equivalence point determination, it is recommended to have about 1 mL of titrant before and after the inflection point.

With 0.1 M $AgNO_3$ as titrant, 1 mL of titrant corresponds to:

0.12 g/L of Sodium chloride with 50 mL of sample
0.12% with 5 g of sample

Sample amount/titrant volume	1 mL	5 mL	9 mL
1 g	0.58% NaCl	2.92% NaCl	5.26% NaCl
5 g	0.12% NaCl	0.58% NaCl	1.05% NaCl
10 mL	0.58 g/L NaCl	2.92 g/L NaCl	5.26 g/L NaCl
30 mL	0.19 g/L NaCl	0.97 g/L NaCl	1.75 g/L NaCl
50 mL	0.12 g/L NaCl	0.58 g/L NaCl	1.05 g/L NaCl

4.3. Settings

4.3.1. Salt in liquid food

Name	Default parameter	Unit
Application		
Application name	Salt in liquid food	
Advisable syringe	10mL (Hamilton)	
Sample		
Name	Liquid Food ? [1]	
Amount	50.0	[mL]
Minimum amount	10	[mL]
Maximum amount	55	[mL]
Probe		
Recommended probe	MTC 306	
Titrant		
Name	$AgNO_3$	
Titrant concentration	0.1000	[mol/L]
Leveling		
Active	No	
Automatic addition		
Active	Yes	
Reagent name	HNO_3 1M	
Pump ID	Pump 1	
Time	3	[s]
Stirring speed	25	[%]
Manual addition		
Active	No	
Stirring		
Active	Yes	
Time	15	[s]
Stirring speed	25	[%]
IP titration		
Stirring speed	25	[%]
Predose volume	0	[mL]
Delay	0	[s]
Max volume stop point	10	[mL]
Stop on last EQP	Yes	
Result 1(R1) name	Salt	
R1 hide	No	
R1 min	0.1	[g/L]
R1 max	60	[g/L]
R1 QC min	0.1	[g/L]
R1 QC max	60	[g/L]
R1 molar weight	58.443	

4.3.2. Salt in solid food

Name	Default parameter	Unit
Application		
Application name	Salt in solid food	
Advisable syringe	10mL (Hamilton)	
Sample		
Name	Solid Food ? [1]	
Amount	5.000	[g]
Minimum amount	0	[g]
Maximum amount	10	[g]
Probe		
Recommended probe	MTC 306	
Titrant		
Name	$AgNO_3$	
Titrant concentration	0.1000	[mol/L]
Leveling		
Active	No	
Automatic addition		
Active	Yes	
Reagent name	HNO_3 1M	
Pump	Pump 1	
Time	3	[s]
Stirring speed	25	[%]
Manual addition		
Active	No	
Stirring		
Active	Yes	
Time	45	[s]
Stirring speed	25	[%]
IP titration		
Stirring speed	25	[%]
Predose volume	0	[mL]
Delay	0	[s]
Max volume stop point	10	[mL]
Stop on last EQP	True	
Result 1(R1) name	Salt	
R1 hide	No	
R1 min	0.1	[%]
R1 max	6	[%]
R1 QC min	0.1	[%]
R1 QC max	6	[%]
R1 molar weight	58.443	

[1] "?" in the name, indicates that the sample name will be automatically incremented with a number for each analysis

4.4. Modification of the settings

4.4.1. Sample homogenization duration

Depending on the sample, the stirring time must be changed before titration in order to have the appropriate salt extraction.

4.4.2. Sample dilution

The final results are calculated based on the sample volume.

When a dilution of the sample is needed, **Enter the real sample amount** in the application edit window.

4.4.3. Addition of a predose volume

The parameters are defined in order to have the best compromise between accuracy and titration time.

For higher concentrations with a high titrant volume, the titration time can be reduced with an addition of titrant (predose) at the beginning of the titration. Enter the predose volume (in mL) and the stirring time after the addition in the application edit window.

4.4.4. Reagent addition

It is possible to replace the automatic addition of nitric acid by a manual addition. This is done by deactivation and activation of the corresponding method in the application edit window.

4.4.5. Sample leveling

For the salt in food application, for a sample volume higher than 30 mL, it is possible to use a leveling method. It is deactivated by default.

To use this method, an external pump is required. All elements (probes, tubes from the titrator and the tube from the external pump) have to be well installed on the probe holder. The beaker has to contain a level of sample higher than the position of the tube of the external pump. When the beaker is attached to the probe holder, this method allows the system to automatically remove the excess sample by a defined pump working time, and always keep the same sample volume before launching the analysis.

In order to define this volume, the autoleveling calibration sequence has to be previously executed (see section **10 Appendix: Autoleveling calibration**).

When this option is active, the working time of the external pump must be set (default 30 s). The minimum working time must allow the pump to be removing air during the last few seconds of the external pump activation.

Note: *Do not forget to re-edit the sample amount with the expected value when deactivating the leveling method.*

5. Titration procedure

5.1. Sample preparation

Sample preparation varies according to the product. This procedure does not describe the procedure for the extraction.

For solid food, take the required amount of the aliquot sample and adjust the volume to 50 mL with deionized water. For a butter sample, adjust the volume to 50 mL with hot deionized water (temperature of about 60 °C).

For liquid food, pipette 50 mL of the sample or the required amount of sample corresponding to the working range and adjust the volume to 50 mL with deionized water.

5.2. Sample analysis

1. Select the application corresponding to your sample (liquid food or solid food).
2. Dip the electrodes and titrant tip in the sample.
3. Adjust the tip from the peristaltic pump to above the sample surface.
4. Press **Start**. At the beginning of the analysis, 5 mL of nitric acid 1M will be automatically added with the peristaltic pump.
5. For the solid food application, enter the real weight of the sample at the end of the analysis.
6. At the end of the titration, the result will be displayed.

6. Results

6.1. Result calculation

For liquid food, the result is directly calculated in g/L of sodium chloride.

For solid food, the result is directly calculated in % of salt (sodium chloride).

As 1 molecule of titrant reacts with 1 molecule of Cl^-:

- Result in g/L sodium Chloride = V_{titr} x C_{titr} x 58.443 / V_{smp}
- Result in percent = = V_{titr} x C_{titr} x 58.443 x 100/ (m_{smp} x 1000)

Where:

V_{titr} = Total volume of titrant to reach the inflection point in mL
C_{titr} = Titrant concentration in mol/L
V_{smp} = Sample volume in mL
m_{smp} = Sample amount used during titration in g
58.443 = Molar weight of NaCl

It is possible to express a result in g/L of Cl^- by replacing the atomic weight of the NaCl (58.443) by the atomic weight of the Cl^- (35.453) for the result calculation.

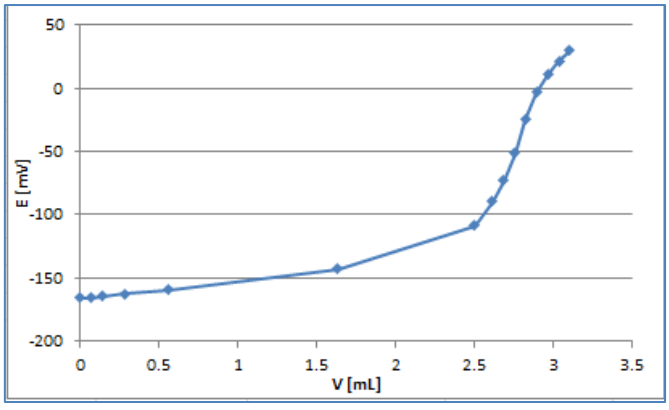
6.2. Experimental results

These results are indicative and have been obtained for a given sample for ten successive determinations. Results are given for the titration of milk, tomato puree and butter

Application	Liquid food (milk)	Solid food (tomato puree)	Solid food (butter)
Number of samples	10	10	10
Mean value	1.60 g/L	1.44%	1.64%
Standard deviation	0.010 g/L	0.002%	0.015%
Relative standard deviation	0.65%	0.15%	0.91%

6.3. Example of a titration curve

This curve has been obtained during the analysis of one of the 10 samples. Depending on the electrode used, minimum and maximum values of the potential can be different, but should be between ±300 mV for a correct detection of the equivalent point.



7. Recommendations

Between titrations, rinse the silver electrode with distilled water. Do not use abrasive strips to clean the silver rod.

If the potentials are not between -300 and +300 mV, replace the electrolyte in the reference part of the electrode.

8. Bibliography

- *Standard ISO 1738.1997: Determination of salt in butter*
- *International Standard ISO 1841-2: Meat and meat products - Determination of chloride content*

9. Appendix: Titrant calibration

Analytical grade silver nitrate can be considered as a standard, but you can also standardize the silver nitrate solution versus a NaCl solution with the same molar concentration.

1. Pipette and weigh 5.000 mL of NaCl solution (corresponding to half the capacity of the burette).
2. As the solution density can be taken as 1, if the weight is not exactly 5.000, enter the measured weight as a volume.
3. Dilute the NaCl standard to 50 mL with deionized water and add 5 mL of HNO_3 1M solution.
4. Calibrate the titrant using the titrant calibration module instead of the sample analysis.

Default settings for titrant calibration

Name	Setting	Unit
Automatic addition		
Active	Yes	
Reagent name	HNO_3 1M	
Pump	Pump 1	
Time	3	[s]
Stirring speed	25	[%]
Manual addition		
Active	No	
IP titration		
Stirring speed	25	[%]
Predose	3	[mL]
Delay	10	[s]
Max. vol. stop point	10	[mL]
Stop on last EQP	Yes	
Titer result		
Min titrant conc.	0.0900	
Max titrant conc.	0.1100	
Standard		
Standard name	NaCl	
Standard amount	5.000	[mL]
Min. amount	4	[mL]
Max. amount	6	[mL]
Concentration	0.1000	[mol/L]

10. Appendix: Autoleveling calibration

This option is **ONLY** available from the calibration menu if **Method Leveling** is set to Active (**Yes**). Refer to the documentation delivered with the external pump for a correct installation, paying particular attention to the suction tube from the pump.

1. Prepare a 0.001M NaCl solution in a 1000 mL volumetric flask, by diluting 100 mL of the standard solution NaCl 0.01M (used in the titrant calibration) with deionized water.
2. Pour a sufficient amount of the standard solution 0.01M into a beaker, allowing the external pump tube to be immersed in the liquid.
3. In the calibration menu, select **Autoleveling calibration** and then the application to use. The titration settings are the same as those used during a sample titration
4. At the end of the procedure, the result obtained is the volume remaining in the beaker after leveling. It is automatically written in the **Sample amount** field for the application editor and will be used in the next titration calculations of the application.
   This result is compared to the minimum and maximum sample amounts defined in the **Sample** section of the application editor. For this application, leveling can be used only for a sample volume higher than 30 mL (**Sample min. amount** has to be set to **30 mL**).

Settings for Autoleveling calibration

Name	Setting	Unit
Sample		
Min. amount	30	[mL]
Max. amount	55	[mL]
Method leveling		
Active	Yes	
Time	30	[s]
Autoleveling calibration		
Solution name	NaCl	
Concentration	0.01	[mol/L]