



Determination of Salt in Butter (According to ISO 1738.1997)

Introduction

Salt, as sodium chloride, is present in or added to butter. The chloride determination should be performed by titration with silver nitrate (AgNO₃) as titrant.

Standard NF ISO 9297 uses a colorimetric determination of the equivalence point (with potassium chromate) but it is possible with the same titrant to make a potentiometric determination of the equivalence point.

This application uses a potentiometric titration with a silver electrode as measuring electrode and a mercury/mercurous sulphate as reference electrode. It is suitable for butters containing more than 0.03% of sodium chloride.

Principle

The silver nitrate reacts with the chloride ion according to



Then 1 mole of AgNO₃ corresponds to 1 mole of chloride.

The titrant concentration is generally 0.1M. The results are expressed as a % of salt (NaCl with a molecular weight of 58.453 g/mol).

Electrode and Reagents

M291Ag Silver Metal Electrode (part no. E34M002) connected to the Titration Manager with cable part no. A94L114.

REF601 Hg/Hg₂SO₄ Reference Electrode (part no. E21M012) connected to the Titration Manager with adapter part no. A94P807 (**see notes**).

(Other electrodes can be used such as the M295Ag Silver Metal Electrode (part no. E34M002) with adapter part no. A94P807 or the REF621 Reference Electrode (part no. E21M006) connected with the CL114 cable (part no. A94L114).

If temperature control of the solution is required: T201 Temperature Sensor (part no. E51M001)

HNO₃ 1M

Dilute 78 ml of concentrated nitric acid in 1000 ml of distilled water.

This operation is highly exothermic. Observe laboratory safety regulations.

AgNO₃ 0.1M

Dry the AgNO₃ at 105°C for 2 hours and let it cool to room temperature in a dessicator.

Using a volumetric flask, dissolve 16.9873 g of AgNO₃ in 1000 ml of distilled water. This solution is also available commercially.

Hot distilled water (approximately 60°C for electrodes and beaker cleaning).

Inflection Detection Settings

CONTINUOUS ADDITION MODE

Stirring speed:	500rpm
Stirring delay:	30 s
Burette volume:	25 ml
Maximum volume:	25 ml
	(see working range)
Stop point:	0 mV
Smoothing parameter:	5
Inflection point number:	1
Minimum speed:	0.2 ml/min
Maximum speed:	5 ml/min
Direction:	Increasing mV
Stop at last IP:	YES
Inflection 1:	
Min. ordinate:	-200 mV
	(see notes)
Max. ordinate:	-50 mV
	(see notes)
Sample unit:	g
Sample amount:	4 (see notes)
Results:	
Unit:	%
Reaction:	1 smp + 1 Titr
Molar weight:	58.453 g/mol

Procedure

Accurately weigh around 4 or 5 g of butter in the titration beaker.

Add 100 ml of hot distilled water (60°C) and mix the solution until the butter is properly mixed in.

(It is also possible to add cold water and to warm up the solution).

Add 1 ml of HNO₃ (**see notes**) .

Dip electrodes and delivery tip in the solution.

It is recommended to dip electrodes and delivery tip in a stirred solution to avoid butter deposit at the reference junction.

Run the titration with a beaker temperature of around 50 - 60°C (**see notes**).

Results

As in this case 1 molecule of titrant reacts with 1 molecule of Cl-

Generally expressed as a % of sodium chloride ion (MW = 58.453 g/mol)

$$R = V(\text{titr}) * C(\text{titr}) * 58.453 * 100 / W(\text{smp}) * 1000 \quad (1)$$

-V(titr) = total volume of titrant to reach the inflection point in ml

-C(titr) = Titrant concentration in mol/l (currently 0.1)

-W(smp) = sample weight in g

58.453 = Molar weight of sodium chloride (NaCl)

100 = To have a result for 100g of product (%)

1000 = To have a result expressed in g

Using the above inflection detection settings, the Titration Manager directly calculates the result in mg/l of chloride.

For three determinations on salted butter

Mean:	1.995%
Standard deviation:	0.0018%
Rel. standard deviation:	0.09%
(Butter with a salt content of 2%)	

For three determinations on butter (with a 10 ml burette)

Mean:	0.036%
Standard deviation:	0.0003%
Rel. standard deviation:	0.8%

Results

Using a 25 ml burette, the experimental titrant volume should be between 1 and 23 ml.

According to formula (1), 1 ml of titrant corresponds to approximately 0.145% of salt for 4 g of sample.

For a 25 ml burette, this corresponds to a working range of 0.145% to 3.33%.

Although the ISO standard specifies a low limit corresponding to a salt content of 0.1%, it is possible to measure salt content below this value using a 10 ml burette and a sample amount of about 6 g. In this case, set the maximum volume to 10 ml.

Notes

Note regarding electrodes

As the sample solution can cause the porous pin of the reference electrode to become clogged, it is advisable to use separate rather than combined electrodes. At the end of each titration, immediately rinse the electrodes with hot water.

Note regarding sample amount

Try to have a sample test representative of the raw product. If necessary, warm and mix the product before weighing.

Note regarding HNO₃ addition

This optional addition allows a more stable and reproducible starting potential.

If you do not add HNO₃ check the minimum and maximum ordinate values and correct them if necessary.

Note regarding titration temperature

According to the ISO standard, it is also possible to run the titration at room temperature. However, it is essential to always work with the same conditions. As the Hg/Hg₂SO₄ electrode is used as reference electrode, it is recommended to run the titration at a temperature below 65°C.

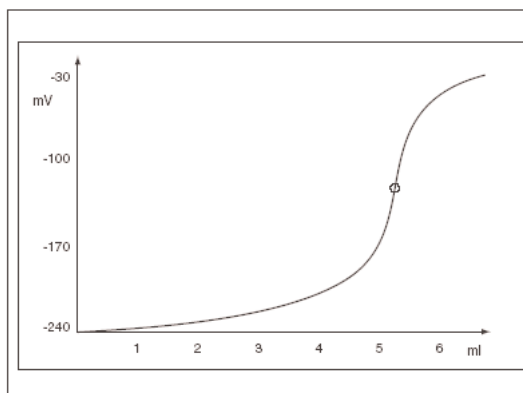
Note regarding a blank titration

Depending on the purity of the reactants, it may be necessary to run a blank titration. For this purpose just enter:

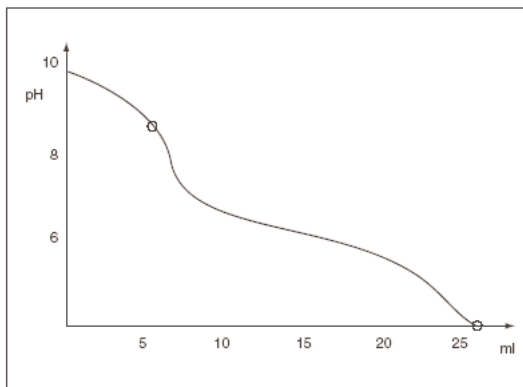
Titration with blank: YES

Other settings are unchanged. For this application blank was checked and corresponded to 0.00 ml.

Curves



Salted Butter



Butter