Titration of Chloride in food samples according to ISO 5943

Description

The determination of the chloride content of food is done by titration with silver nitrate solution 0.1 mol/l. This determination is not always easy, since first the chloride has to be released from the sample. Sample preparation is very important for samples such as cheese, butter or similar products to capture all the chloride. The Norm ISO 5943 requires heating up to 55 ° C to dissolve the sample. However, it has been found that heating up to the boiling point gives better results for difficult samples. The result is calculated as % Chloride or % NaCl.

Instruments

Titrator	TL 5000, TL 7000 or higher
Exchangeable head	WA 10
Electrode	AgCl 62 or AgCl 62 RG
Cable	L 1 A
Stirrer	Magnetic stirrer TM 235 / TM 50
Lab accessory	Glass beaker 150 ml
	Magnetic stirrer bar 30 mm
	Homogenizer Kinematica PT1200 or similar (optional)

Reagents

1	Silver nitrate solution 0.1 mol/l
2	Nitric acid 4 mol/l
3	Polyvinylalkohol – solution 0.5% (optional)
4	Electrolyte solution L2114 (KNO_3 2 mol/l + KCl 0.001 mol/l) for AgCl 62
5	Distilled Water
6	
All reagents should be of analytical grade or better.	

Titration procedure

Reagents

The titer determination of the $AgNO_3$ solution is carried out as described in the application report "Titer determination of $AgNO_3$".

Polyvinyl alcohol - solution 0.5%
0.5 g of polyvinyl alcohol are dissolved in 100 ml of distilled water.

Cleaning of the electrode

The electrode is rinsed with distilled water. The electrolyte solution L2114 is also suitable for storage.

Sample preparation

The sample is weighed into a 150 ml beaker and made up to about 80 ml of distilled water. The mixture is heated up to boiling with vigorous stirring. If necessary, the homogenizer Kinematica PT1200 (or similar) can also be used for better comminution of the sample. After 10 min. the sample is allowed to cool down to room temperature and 0.5 ml 4mol / l HNO3 are added. After cooling, the titration is done with 0.1 mol/l with AgNO3 solution to an equivalence point. In order to prevent deposits of AgCl on the electrode, 0.5 - 1 ml of the polyvinyl alcohol solution can be added. The consumption should be about 5 - 15 ml.

The titration can be carried out with samples with chloride contents of a few ppm - 100%, but the amount of sample has to be adjusted.

Chloride content [%]	Sample [g]
< 0.1	> 10
0.1 – 1	1 – 10
1 – 10	0.1 – 2.0
10 – 50	0.05 – 0.1
50 - 100	0.05

Checking the silver electrode

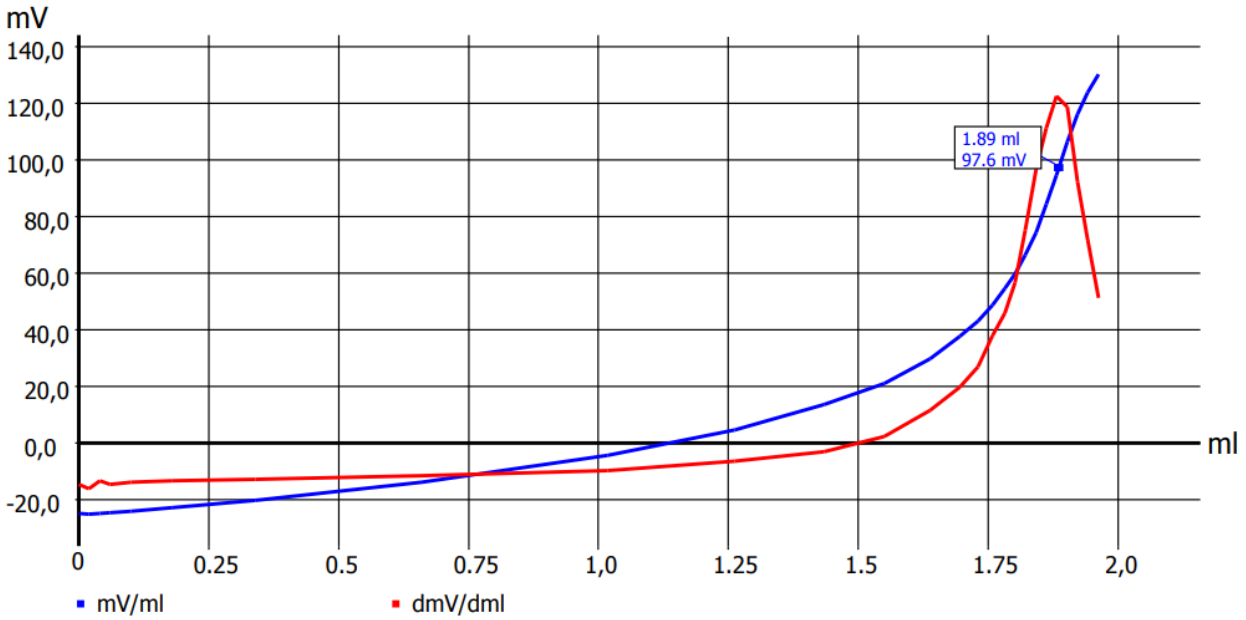
Calibration with buffers or comparable test solutions as for pH electrodes is not possible, but also not necessary. With a pH electrode, the measured voltage in mV in the pH meter/titrator is converted into pH values using the values determined during the pH calibration such as slope and zero point. In addition, there are many methods where titration to a certain pH value is required, such as the determination of total acidity in beverages.

This is not the case with chloride titration. Here, titration always takes place to an equivalence point (EQ). This means that a certain measuring potential is not important, but the change in the measuring potential during several measuring points.

To check the silver electrode we recommend to titrate a standard such as NaCl and to compare the resulting titration curve with a stored titration curve of a standard at the beginning of use. The potentials should be in the same order of magnitude as at the beginning. More important is the appearance of the curve. It should not be noisy or jagged.

Titration parameter

Sample titration



Default method	Chloride %		
Method type	Automatic titration		
Modus	Dynamic		
Measured value	mV		
Measuring speed / drift	User defined	Minimum holding time	3 s
		Maximum holding time	15 s
		Measuring time	3 s
		Drift	10 mV/min
Initial waiting time	0 s		
Dynamic	steep	Max step size	1.0 ml
		Slope max ml	15
		Min. step size	0.02 ml
		Slope min. ml	230
Damping	none	Titration direction	increase
Pretitration	off	Delay time	0 s
End value	off		
EQ	On (1)	Slope value	400
Max. titration volume	50 ml		
Dosing speed	100%	Filling speed	30 s

For some samples it may happen that the titration curve is very flat and the titrator does not stop the titration at the EQ. In this case, the slope value for the EQ should be reduced to 200.

Calculation:

$$Result\ [\%] = \frac{(EQ1 - B) * T * M * F1}{W * F2}$$

B	0	Blank value
EQ1		Consumption of titrant at first Equivalence point
T	WA	Actual concentration of the titrant
M	35.45	Molecular weight
W	man	sample weight in g
F1	0.1	Conversion factor
F2	1	Conversion factor

If the calculation value is not % chloride, but % NaCl, then M is set to the molar mass of NaCl 58.44 g/mol.

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