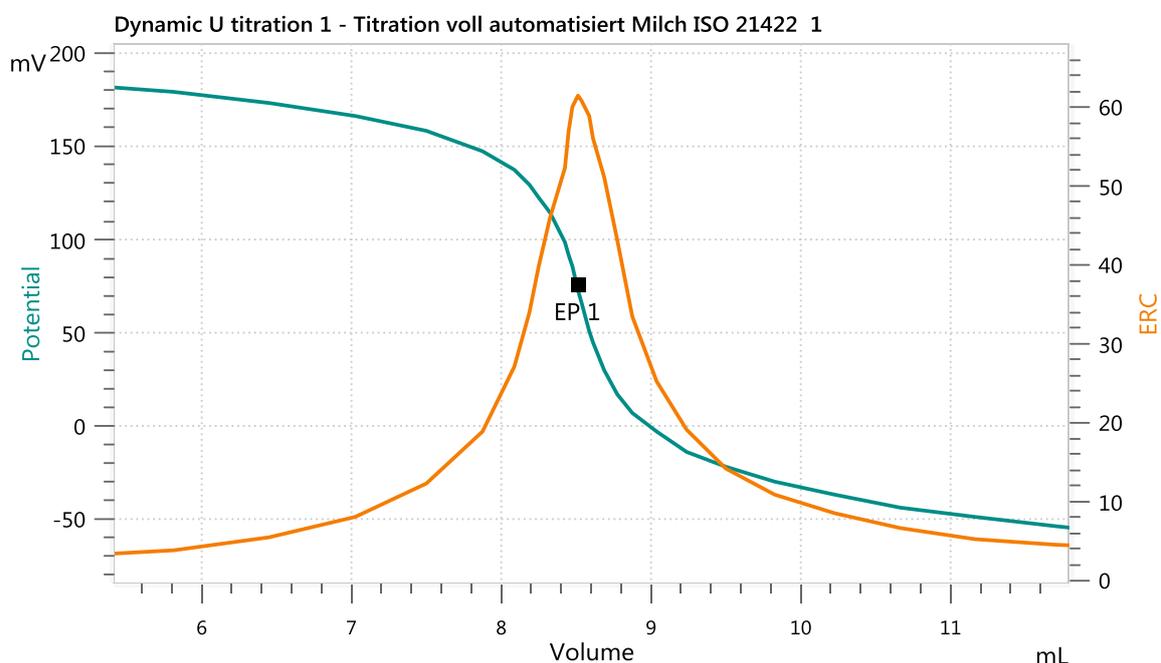


Titration Application Note T-133

Chloride in milk and milk powder

Automation saves time, increases throughput and provides a solution for high fat samples



In order to maintain product quality, the sodium chloride content in dairy products must be monitored and not exceed the limits defined by the respective public health authorities. The chloride content in food correlates with the salt content, its determination is therefore described in various norms and standards. However, preparation of such samples is time consuming, as it includes a chloride extraction with warm water. Whole milk powders in particular are difficult to handle as an inhomogeneous dispersion of fat in the titration suspension occurs.

In order to reduce the workload, increase sample throughput, and eliminate the matrix challenges posed by high fat products, this Application Note presents a fully automatic potentiometric titration of chloride with silver nitrate in milk and milk powder based on ISO 21422, IDF 242, AOAC 2015.07, AOAC 2015.08 and AOAC 2016.03

Method description

Sample

- Toddler milk powder
- Whole milk powder
- Milk
- Salted milk
- Protein shake

Sample preparation

All samples are well mixed before weighing them.

Configuration

OMNIS Sample Robot S Pick&Place	2.1010.1010
OMNIS Advanced Titrator without stirrer	2.1001.0210
OMNIS Dosing module, 2x	2.1003.0010
OMNIS Cylinder unit 50 mL	6.03001.250
OMNIS Cylinder unit 20 mL, 2x	6.03001.220
Digital measuring modules, 2x	6.02100.010
OMNIS Stand-Alone license	6.06003.010
OMNIS instruments license	6.06002.010
Heating plate with magnetic stirrer	n.a.
dAg-Titrode	6.00404.300
dProfitrode, bridge electrolyte c(KNO ₃) = 1 mol/L	6.00204.300

Solutions

Titrant	c(AgNO ₃) = 0.1 mol/L This solution should be bought from a supplier. The solution has to be protected from daylight and can be stored for up to two months.
Nitric acid	c(HNO ₃) = 4 mol/L 200 mL nitric acid is carefully added to 600 mL deionized water.
Wash solution	ϕ(Isopropanol) = 70% (v/v)

Analysis

First, 130 mL of 45 °C warm water is transferred to the sample beaker and stirred for 10 s. 5 mL c(HNO₃) = 4 mol/L is added, stirred for 5 s and the pH is measured. If it is above 1.5, an additional 5 mL c(HNO₃) = 4 mol/L is added. Then, the sample is titrated with c(AgNO₃) = 0.1 mol/L until after the endpoint. ϕ(Isopropanol) = 70% (v/v)

is used for dip rinsing as wash solution in between the different measurements in addition to dip rinsing with water.

For whole milk powder having a high fat content, 20 mL isopropanol is added and 110 mL warm water is used instead of only water.

A blank determination is performed the same way as described above, omitting the sample and using a MET U titration with a volume increment of 0.005 mL and an EP criterion of 30 mV.

Parameters

Mode	DET U
Pause	0 s
Signal drift	50.0 mV/min
Min. waiting time	0 s
Max. waiting time	26 s
Min. Volume increment	10 µL
Meas. point distance	4
Stirring rate	10
Stop volume	15 mL
Stop EP	Off
EP criterion	5
EP recognition	greatest

Results

Sample	Chloride content in mg / 100 g sample (n = 6)	s(rel) / %
Whole milk powder	832.9	0.2
Toddler formula powder milk based	293.7	0.3
Salted milk	411.8	0.2
Protein shake	88.4	2.7
Organic milk	99.3	0.5

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