Application

SI Analytics

a **xylem** brand

<div style="border:2px solid #1a7aa8;">

Determination of NaCl in Cheese Samples

</div>

Application

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Appliances

- Titrator: TL 7750
- Burette: WA 50
- Stirrer: TM 235 & heating stirrer

Reagents

- Titrant: Ammoniumthiocynanate (NH_4SCN) Solution 0,1mol/L (Merck)
- Silver nitrate ($AgNO_3$) Solution 0,1mol/L (Merck)
- Nitric Acid (HNO_3) concentrated (65%)
- Distilled Water
- Ferric ammonium sulfate indicator ($NH_4Fe(SO_4)_2$)
- Standard: Silver nitrate solution with known titer

Use

Visual indirect titration method of sodium chloride in different cheese samples like Cottage Cheese or Feta

Description

Preparing the Indicator:

Solve ferric ammonium sulfate in 10mL distilled water as long as the solution is saturated. Stir about 10min to make sure that part of the salt will not dissolve. The solution can be light brown. Give droplet for droplet small amounts of bevor boiled and cooled HNO3 into the solution until the solution turns colorless. Filtrate the solution to get off the non-dissolved salt

Determination of titer:

For titer determination dose 10mL $AgNO_3$ Solution (Note: with defined concentration) in a 100mL glass beaker and solve in 60mL distilled water. Place the burette tip and the electrode into the solution and start the titration method which stops at equivalence point.

Calculation:

$$\text{Titer} = \frac{\text{Volume }(AgNO_3) * \text{ concentration }(AgNO_3)}{\text{Volume }(NH_4SCN) * \text{ concentration }(NH_4SCN)}$$
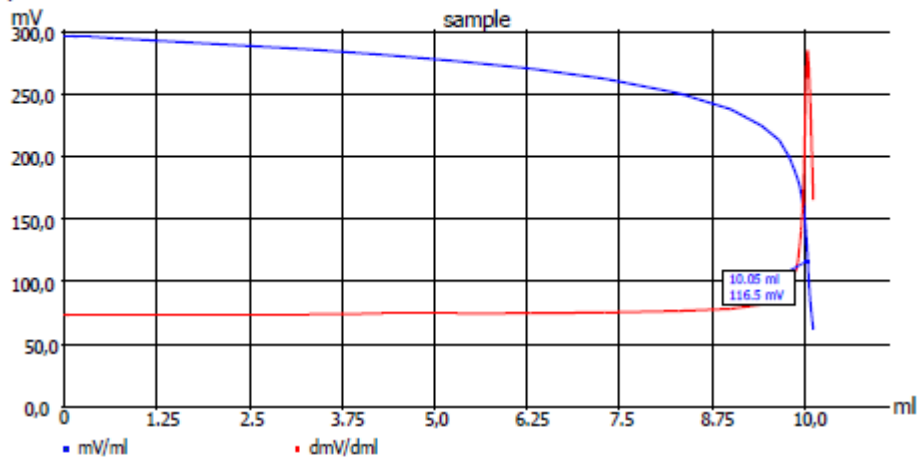
Example:

$$\frac{10,00\text{mL} * 0,1003}{10,05\text{mL} * 0,1000} = 0,998$$

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GLP documentation

Titration graph

Method data

Method name:	Titre AgNO3	Titration duration:	2 m 51 s
End date:	14.04.15	End time:	10:49:11

Titration data

Sample ID:	sample	Weight:	10.00000 g
Start mV:	296.7 mV	End mV:	61.5 mV
EQ:	10.046 ml / 116.5 mV		
Mean value:	---		

Sample measurement:

Titrate blank value:
For indirect titration it is necessary to use a blank value. For this only the titrant solutions without sample are used. Pipet 25 mL $AgNO_3$ into a 150mL glass breaker and add 5mL indicator. Fill up to 80-90 mL with distilled water and titrate with $NH_4Fe(SO_4)_2$ until the EQ is reached. Note the consumption and repeat this measurement two times.

Preparation:

If necessary use a homogenizator for better extraction of NaCl and for consistent homogenization. Use the sample weight by taking care to the following table:

Percentage of salt	Proposed sample weight
<0.1 %	>10 g
0.1 – 1 %	1 – 10 g
1 – 10 %	0,1 – 2 g

Application

Weigh the sample into a 250mL glass beaker. Dose 25mL AgNO$_3$ Solution and mix the cheese sample complete with the silver nitrate solution. Add 25mL conc.HNO$_3$ into the mixture. The solution becomes yellow. Boil as long as it needs to discolor the solution (10 – 30 min.) and cool down to room Temperature. For faster cooling use an ice bath. Add 100mL distilled water and 5 mL Indicator and titrate from light yellow to light red/brown.
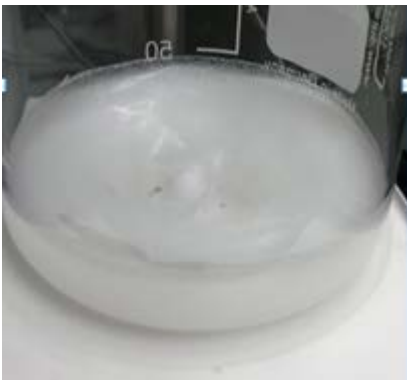
Calculation % Salt in Cheese:

$$\% \text{ Salt} = \frac{(B-V)* M * F1}{W * F2}$$

V: ml consumption at changing point [mL]
B: Blank value   [mL]
M: Molar mass (58.44) [g/mol]
F1:   0.0993
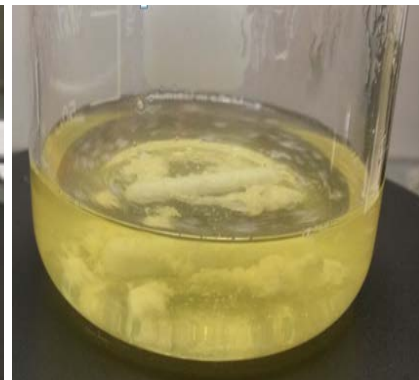W: sample weight (Standard NaCl) [g]
F2:   10

Example

$$\frac{(25{,}238 - 21{,}380)* 0{,}0993 *58{,}44}{2{,}693 * 10} = 0{,}831\%$$

1 Cheese with 25mL AgNO3

2 Cheese with AgNO3 and HNO3
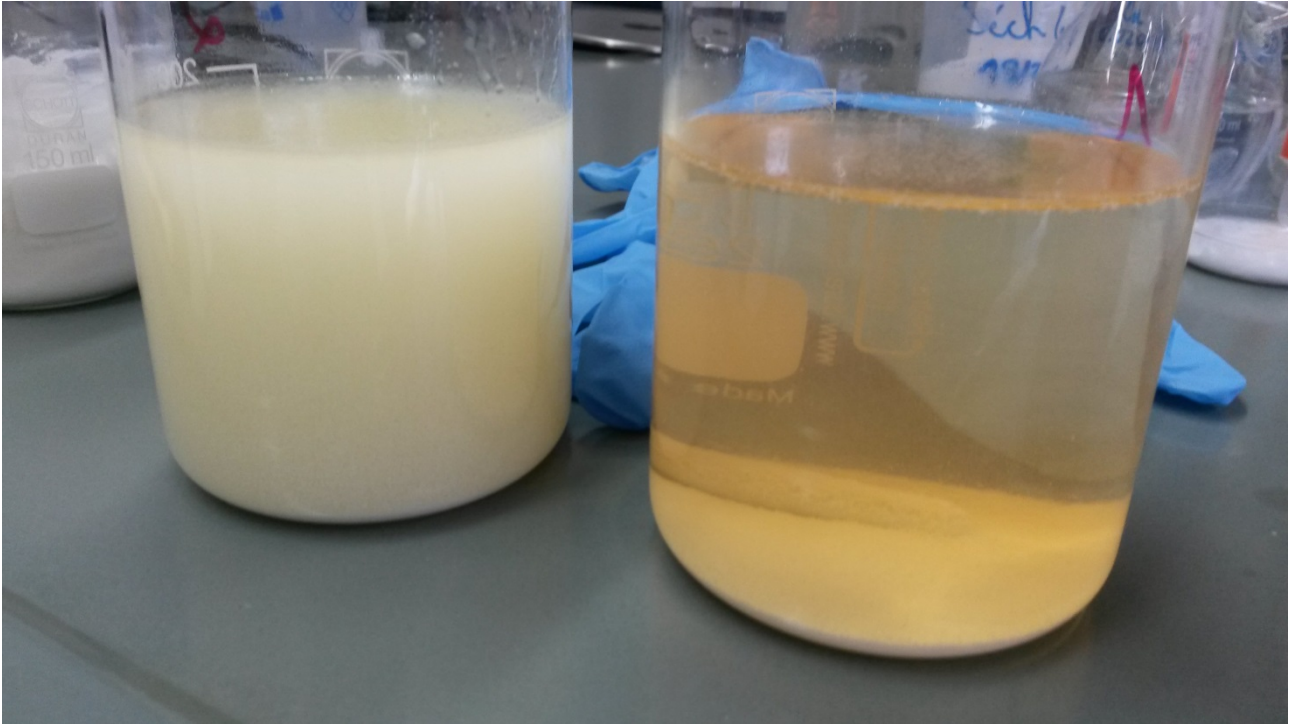

3 Cheese with AgNO3 and HNO3 boiled

4 Start of Titration

5 Endpoint

6 Reference color compared with endpoint of titration

Examples / Results

Weight Sample in g	total Consumption NH$_4$SCN in mL	Blank value in mL	Consumption NH4SCN in mL	NaCl in %
2,693	21,380	25,238	3,858	0,831
2,334	21,820	25,238	3,418	0,850
2,742	21,230	25,238	4,008	0,848
4,171	19,380	25,238	5,858	0,815
4,219	19,270	25,238	5,968	0,821
4,701	19,210	25,238	6,028	0,744
5,868	16,815	25,238	8,423	0,833
6,413	16,090	25,238	9,148	0,828
6,111	16,540	25,238	8,698	0,826

Application

Method

Method data overall view

Method name:	AOAC Method 25mL	Created at:	04/08/15 10:41:30
Method type:	Manuel titration	Last modification:	04/08/15 10:41:30
Measured value:	pH	Damping settings:	None

Dosing parameter

Dosing speed:	100.00 % (100.00 ml/min)
Filling speed:	30 s
Maximum dosing volume:	50.00 ml

Unit values

Unit size:	50ml
Unit ID:	10045175

Reagent:

Batch ID:	no entry
Concentration [mol/l]:	0.09930
Determined at:	03/30/15 18:45:31
Expire date:	--
Opened/compounded:	03/26/15
Test according ISO 8655:	--
Last modification:	03/30/15 11:46:23

Hints

It is possible that side reactions can indicate a wrong equivalence point. The excess of thiocyanate at the endpoint can react with the indicator to Fe (SCN)$_3$

This Fe (SCN)$_3$ can react as following: $AgCl + Fe(SCN)_3 \rightarrow Ag(SCN) + FeCl_3$

This effect that the color disappear and the endpoint is not exact. One possibility to handle is to filtrate the AgCl before the titration starts.

SI Analytics GmbH		Phone:	+49 (0) 6131 / 66 – 5062
Hattenbergstr. 10			+49 (0) 6131 / 66 – 5118
55122 Mainz		Fax:	+49 (0) 6131 / 66 – 5001
Germany		E-Mail:	titration@si-analytics.com
Homepage:		www.si-analytics.com	