Determination of acidity/acid degree in milk and milk products

Application

Use

This method is used for the quantitative determination of acidity (acid degree) in milk and milk products. The acidity is calculated either as SH-, Dornic- or Therner degree. The different degree uses different concentrations of NaOH titrant.

Appliances

- Titrator: TL 6000/7000 (TL 6000/7000 M2/20) consists of
- Basic device
- Magnetic stirrer TM 235
- 20 mL Exchange unit WA 20, with nrown glass bottle for titrant complete
- And pH combination electrode A 162 DIN ID

Electrodes

- Electrode: A 162 DIN ID
- Calibration: DIN buffer pH= 4.00 and pH= 7.00

Reagents

- Titrant: sodium hydroxide solution 0.1mol/l (Therner), 0.25 (SH) or 0.11 (Dornic) mol/l
- Soda lime for carbon dioxide uptake of the reagent.
- Titer: potassium hydrogen phthalate (reference material)

Description

Calibration

The pH combination electrode is calibrated in technical buffer pH=4.00 and pH= 7.00 or in DIN buffer pH= 4.01 and pH= 6.87.
Example of the calibration documentation:

Calibration

Buffers used

pH buffer 1:	TEC_4.000	
pH buffer 2:	TEC_7.000	

Measured values

pH buffer 1:	TEC_4.000	165.6 mV / 23.4 °C
pH buffer 2:	TEC_7.000	-11.2 mV / 23.0 °C

Calibration data

Slope:	99.4 % / -58.8 mV/pH
Zero point:	pH 6.81 / -11.2 mV
Temperature:	23.4 °C (a)
Date and time:	07.03.13 / 15:04

Determination of the exact concentration of the standard solution

By carbon dioxide absorption from the air occurs in the sodium hydroxide solution of sodium bicarbonate, which changes the pH of the titrant. To prevent this, a drying tube filled with soda lime is placed on the reagent bottle. The exact concentration of the sodium hydroxide solution is determined using the standard potassium hydrogen phthalate. The potassium hydrogen phthalate is dried in the oven before the titer determination for 2 hours at 120°C and cooled in a desiccator.

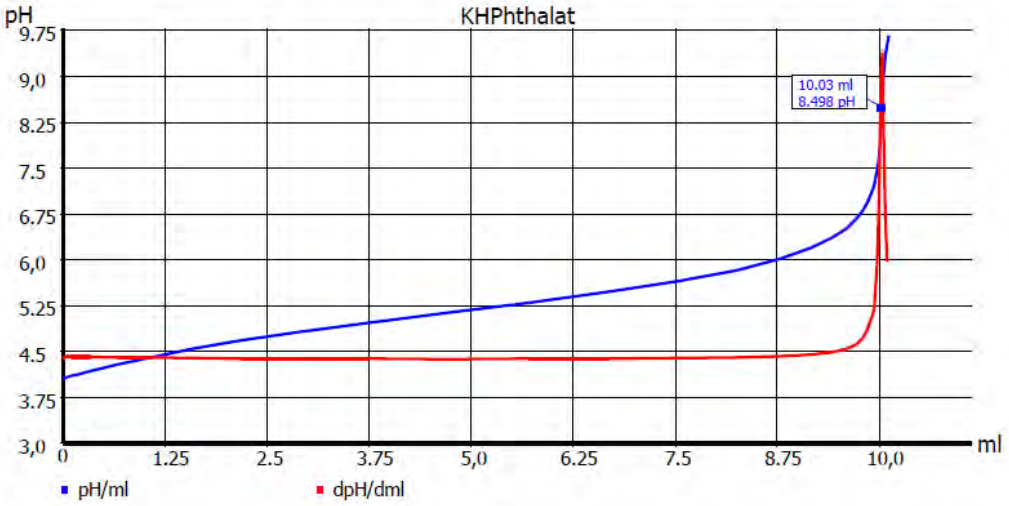
Implementation

In a 50 mL beaker, 0.2 to 0.3g potassium hydrogen phthalate were weighed accurately and dissolved in 30 mL of dist. water with stirring. It is titrated with 0.1 mol/l sodium hydroxide solution. For 0.25 mol/l NaOH you need 0.5 – 0.75 g potassium hydrogen phthalate.

Standardisation titration (result)

GLP documentation

Titration graph



Method data

Method name:	Titre NaOH	Titration duration:	2 m 15 s
End date:	08.01.13	End time:	15:46:03

Titration data

		Weight:	0.20490 g
Start pH:	pH 4.065	End pH:	pH 9.667
Start temperature:	25.0 °C (m)	End temperature:	25.0 °C (m)
Zero point:	pH 6.85 / -8.9 mV	Slope:	98.7 % / -58.4 mV/pH
EQ:	10.032 ml / pH 8.498	Titre:	0.1000 mol/l
Mean value:	---	RSD:	---

Calculation formula

Titre:	(W*F2)/((EQ1-B)*M*F1) -> WA	Mol (M):	204.22000
Weight (W):	0.2049 g (m)	Factor 2 (F2):	1000.0000
Blank value (B):	0.0000 ml	Factor 1 (F1):	1.0000
Statistics:	3		

Standardisation titration (method

Method data

Method name:	Titer NaOH	Created at:	02/15/12 15:32:03
Method type:	Automatic titration	Last modification:	02/16/12 10:14:55
Measured value:	pH	Documentation:	GLP
Titration mode:	Dynamic		
Dynamic:	average		
Measuring speed / drift:	Normal:	minimum holding time:	02 s
		maximum holding time:	15 s
		measuring time:	02 s
		drift:	20 mV/min
Initial waiting time:	0 s		
Titration direction:	Increase		
Pretitration:	Off		
End value:	10.500 pH		
EQ:	On		
slope value:	Steep	Value:	700

Dosing parameter

Dosing speed:	100 %	Filling speed:	30 s
Maximum dosing volume:	30.00 ml		

Calculation formula

Titer NaOH 0,1mol/l:	(W*F2)/((EQ1-B)*M*F1)		
Mol (M):	20.42230		
Unit:		Decimal places:	4
Weight (W):	man	Factor 2 (F2):	1000.0000
Blank value (B):	0.0000 ml	Factor 1 (F1):	1.0000

Device information

Device: TitroLine 6000
Serial number:
Software version: 07_12

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Application

Titration of the sample

Pipette accurately in 100 ml beaker either:

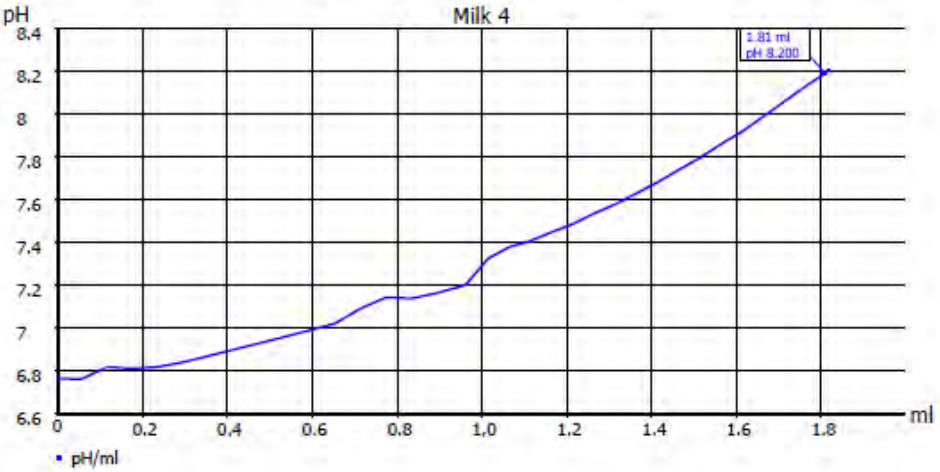
- 25 ml milk for SH degree
- 11 ml milk for Dornic degree
- 9 ml for Therner degree

Add app. 25 ml – 40 ml CO_2 free water into and mix the sample on the magnetic stirrer for a few seconds. Titrate with the suitable NaOH titrant to a fixed pH endpoint (8.2-8.7, depending on national norms).

Example

GLP documentation

Titration graph



Method data

Method name:	acidity in milk	Titration duration:	2 m 5 s
End date:	29.04.13	End time:	13:47:45

Titration data

Sample ID:	Milk 4	Weight:	24.67000 g
Start pH:	pH 6.768	End pH:	pH 8.215
Start temperature:	19.1 °C (a)	End temperature:	19.3 °C (a)
Zero point:	pH 6.82 / -10.3 mV	Slope:	98.8 % / -58.4 mV/pH
EP1:	1.806 ml / pH 8.200	Acidity:	7.321 °SH

Calculation formula

Acidity:	(EP1-B)*T*M*F1/(W*F2)	Mol (M):	1.00000
Blank value (B):	0.0000 ml	Titre (T):	1.00000000 (a)
Factor 1 (F1):	100.0000	Weight (W):	24.67000 g (m)
Factor 2 (F2):	1.0000	Statistics:	Off

Application

Method parameters: (optimized for SH degree)

Method data overall view

Method name:	acidity in milk	Created at:	04/29/13 13:24:34
Method type:	Automatic titration	Last modification:	04/29/13 13:44:18
Measured value:	pH	Damping settings:	None
Titration mode:	End pt.	Documentation:	GLP
Linear steps:	0.050 ml		
Measuring speed / drift:	Normal:	minimum holding time:	02 s
		maximum holding time:	15 s
		Measuring time:	02 s
		Drift:	20 mV/min
Initial waiting time:	0 s		
Titration direction:	Increase		
Pretitration:	Off		
Endpoint 1:	pH 8.200	delta endpoint 1:	pH 1.000
		Endpoint delay 1:	5 s
Endpoint 2:	Off		

Dosing parameter

Dosing speed:	20.00 %	Filling speed:	30 s
Maximum dosing volume:	50.00 ml		

Unit values

Unit size:	20ml
Unit ID:	00360005
Reagent:	NaOH
Batch ID:	no Charge
Concentration [mol/l]:	1.00000
Determined at:	04/29/13 20:26:19
Expire date:	01/01/13
Opened/compounded:	02/01/12
Test according ISO 8655:	01/01/00
Last modification:	04/29/13 13:26:21

Device information

Device:	TitroLine 7750
Serial number:	10018602
Software version:	1316

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Notes

The acidity can also calculate as % lactic acid instead of acid degree. In this case the calculation is:

% lactic acid: (EP1-B) * M*T*F1 /(W*F2)

EP1: ml consumption to pH endpoint
B: blank value in ml. Here = 0
M: molecular weight of lactic acid: 90.08
T: exact concentration of the NaOH in mol/l (e.g. 0.2510 mol/l
F1: 0.1 (conversion factor to %)
W: sample amount in g
F2: 1

If you have any questions on the application, you can feel free to contact us.

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