

# Application

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## Determination of Calcium in jelly drinks



# Application

## Use

This method is used for the quantitative determination of Calcium in jelly drinks using the complexometric titration method with a Calcium Ion selective electrode.

## Appliances

- Titrator: TL 7000
- Basic device
- Magnetic stirrer TM 235
- 10 or 20 mL Exchange unit WA 10/WA20, with amber glass bottle for the titrant, complete
- Stirrer with heating function, balance (2 decimals or better, for the standardization 4 decimals)
- 10 ml volumetric pipette or similar

## Electrodes

- Electrode: Ca-60 combination electrode (with a cable L 1 A) or Ca 1100 A
- Reference electrode (only for Ca 1100 A): B 2920+ with cable L 1 N

## Reagents

- Titrant: EGTA 0.05 mol/l
- Ammonium chloride/ammonia buffer pH = 10
- NaOH 10 mol/l (for the EGTA titrant)
- CaCO<sub>3</sub> titrimetric standard
- HCl 1 mol/l
- Distilled/DI water

## Description and Examples

### Preparation of ammonium chloride/ammonia buffer solution pH= 10

54 g Ammonium chloride for analysis are dissolved in 200 ml DI water. To this solution is added 350 ml of 25% Ammonia solution "Analytical grade". Then it is filled up with DI water to 1 liter

### Preparation of the EGTA Titrant and standardisation

EGTA (ethylene glycol tetra acetic acid 0.05 mol/l is not available as a ready to use titrant. Weigh in 19,3 g of EGTA (e.g. Fluka 03779 or Merck 108435 Titriplex® VI) in into a beaker and approx. 200 ml dist. or DI water are added and the EGTA is suspended under stirring. Then NaOH 10 mol/l is added until everything has dissolved completely. After cooling down, the solution is transferred quantitatively to a 1000 mL volumetric flask with dist. or DI water, filled up to the mark and mixed.

The standardisation of the titrant is carried with CaCO<sub>3</sub> titrimetric standard (available e.g. from Merck or Sigma Aldrich). Weigh in about 0.5 g of the titrimetric standard in a 100 ml volumetric flask (note the exact amount of the weight e.g. 0.5043 g). Add about 20 ml distilled or DI water and shake it a little bit. Add then 12 ml HCl 1 mol/l and wait until all CO<sub>2</sub> is completely degassed. Fill up to the 100 mark with distilled/DI water.

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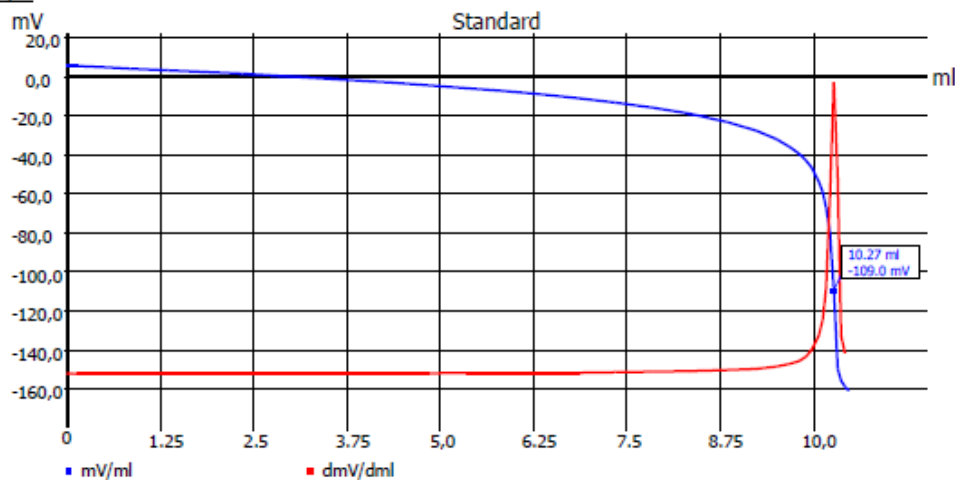
From this standard solution take exactly 10.00 ml (volumetric pipette) and pipette it into a 150 ml beaker. Add 80 ml dist./DI water and 5 ml buffer solution pH 10.

Use the method Titer EGTA (It is possible to use the default method "Titer EDTA" inside the TL 7000/7750 titrator and rename it into Titer EGTA). Please change also the decimals of the unit from 4 to 5.

Enter as sample weight the 1/10 of the weight of the CaCO<sub>3</sub> standard. In our case it was then 0.05043 g. As the end of the titration the result is calculated in mol/l. The result is stored automatically in the WA exchange unit.

### GLP documentation

#### Titration graph



#### Method data

Method name:	Titer EGTA	Titration duration:	4 m 18 s
End date:	19.12.13	End time:	17:39:25

#### Titration data

Sample ID:	Standard	Weight:	0.05043 g
Start mV:	5.8 mV	End mV:	-160.8 mV

EQ:	10.272 ml / -109.0 mV	Titer:	0.04905 mol/l
Mean value:	---	RSD:	---

#### Calculation formula

Titer:	$(W \cdot F2) / ((EQ1 - B) \cdot M \cdot F1) \rightarrow WA$	Mol (M):	100.09000
Weight (W):	0.05043 g (m)	Factor 2 (F2):	1000.0000
Blank value (B):	0.0000 ml	Factor 1 (F1):	1.0000
Statistics:	1 from 3		

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Method: Titer EGTA:

## Method data overall view

Method name:	Titer EGTA	Created at:	12/19/13 17:34:59
Method type:	Automatic titration	Last modification:	12/19/13 17:34:59
Measured value:	mV		
Titration mode:	Dynamic	Documentation:	GLP
Dynamic:	Flat		
Measuring speed / drift:	User-defined:	minimum holding time:	05 s
		maximum holding time:	15 s
		Measuring time:	03 s
		Drift:	05 mV/min
Initial waiting time:	0 s		
Titration direction:	Decrease		
Pretitration:	Off		
End value:	Off		
EQ:	On		
Slope value:	Flat	Value:	120

## Dosing parameter

Dosing speed:	100.00 %	Filling speed:	30 s
Maximum dosing volume:	20.00 ml		

## Unit values

Unit size:	20ml
Unit ID:	10039014
Reagent:	EGTA
Batch ID:	keine
Concentration [mol/l]:	0.04910
Determined at:	12/18/13 3:36:00
Expire date:	01/01/13
Opened/compounded:	01/01/00
Test according ISO 8655:	01/01/00
Last modification:	12/17/13 19:36:03

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### Sample titration

The most important step is to get a homogenous sample. We used the complete content of one jelly juice bin to get a homogenous sample.



As you can see the sample is complexly homogenous. We stirred the sample at a temperature of 35-40 ° C with a stirring speed of 750-1000 rpm. After 15 minutes the sample is homogenous:

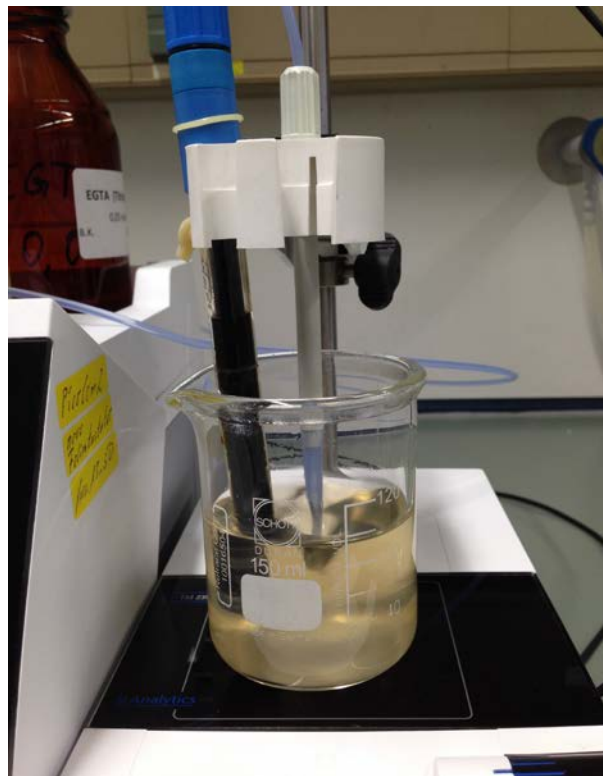


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We used a 2 decimal balance to weigh in the sample. We weighed in the sample into a 150 ml glass beaker, add about 80 ml dist./DI water and about 5 ml buffer solution pH 5.



Place the beaker on the magnetic stirrer of the titrator, start stirring and start the method Calcium. The start stirring time is 120 seconds to dilute/homogenize the sample completely in the water/buffer solution:



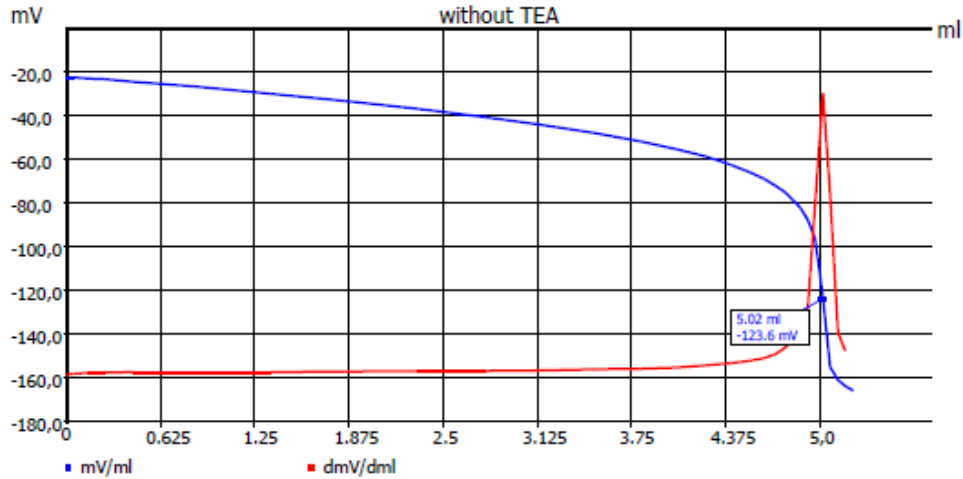


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Sample result and method:

### GLP documentation

#### Titration graph



#### Method data

Method name:	Calcium	Titration duration:	5 m 13 s
End date:	17.12.13	End time:	18:25:11

#### Titration data

Sample ID:	without TEA	Weight:	15.43400 g
Start mV:	-22.5 mV	End mV:	-165.7 mV

EQ:	5.023 ml / -123.6 mV	Calcium mg/100g:	64.0
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#### Calculation formula

Calcium mg/100g:	$(EQ1-B) \cdot T \cdot M \cdot F1 / (W \cdot F2)$	Mol (M):	40.08000
Blank value (B):	0.0000 ml	Titre (T):	0.04910000 (a)
Factor 1 (F1):	100.0000	Weight (W):	15.43400 g (m)
Factor 2 (F2):	1.0000	Statistics:	Off

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Method:

## Method data overall view

Method name:	Calcium	Created at:	12/17/13 18:19:46
Method type:	Automatic titration	Last modification:	12/17/13 18:19:46
Measured value:	mV		
Titration mode:	Dynamic	Documentation:	GLP
Dynamic:	Flat		
Measuring speed / drift:	User-defined:	minimum holding time:	07 s
		maximum holding time:	15 s
		Measuring time:	04 s
		Drift:	03 mV/min
Initial waiting time:	0 s		
Titration direction:	Decrease		
Pretitration:	Off		
End value:	Off		
EQ:	On		
Slope value:	Flat	Value:	120

## Dosing parameter

Dosing speed:	100.00 %	Filling speed:	30 s
Maximum dosing volume:	20.00 ml		

## Unit values

Unit size:	20ml
Unit ID:	10039014
Reagent:	EGTA
Batch ID:	keine
Concentration [mol/l]:	0.04910
Determined at:	12/18/13 1:22:00
Expire date:	01/01/13
Opened/compounded:	01/01/00
Test according ISO 8655:	01/01/00
Last modification:	12/17/13 17:47:16

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### Hints

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

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