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Application Note
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Determination of Calcium in Jelly Drinks

FOOD & BEVERAGE SERIES

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

Instrument	
Titrator	TL 7000
	Magnetic Stirrer TM 235
	10 or 20 mL Exchange unit WA 10/ WA20, with amber glass bottle for the titrant, complete
	10 mL volumetric pipette or similar

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

Reagents	
Titrant	EGTA 0.05 mol/l
Buffer Solution	Ammonium chloride/ammonia buffer pH = 10
	NaOH 10 mol/l (for the EGTA titrant)
Titrimetric standard	$CaCO_3$
	HCl 1 mol/l
	Distilled/DI water

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Description and Examples

Preparation of ammonium chloride/ammonia buffer solution pH= 10

Dissolve 56 g of ammonium chloride in 200 ml of DI water for anlaysis. To this solution add 350 ml of 25% Ammonia solution "Analytical grade", then fill with DI water to 1L.

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 19.3g of EGTA (eg.g Fluka 03779) into a beaker and add approximately 200 ml of distilled or DI water, then 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 distilled or DI water, filled up to the mark and mixed.

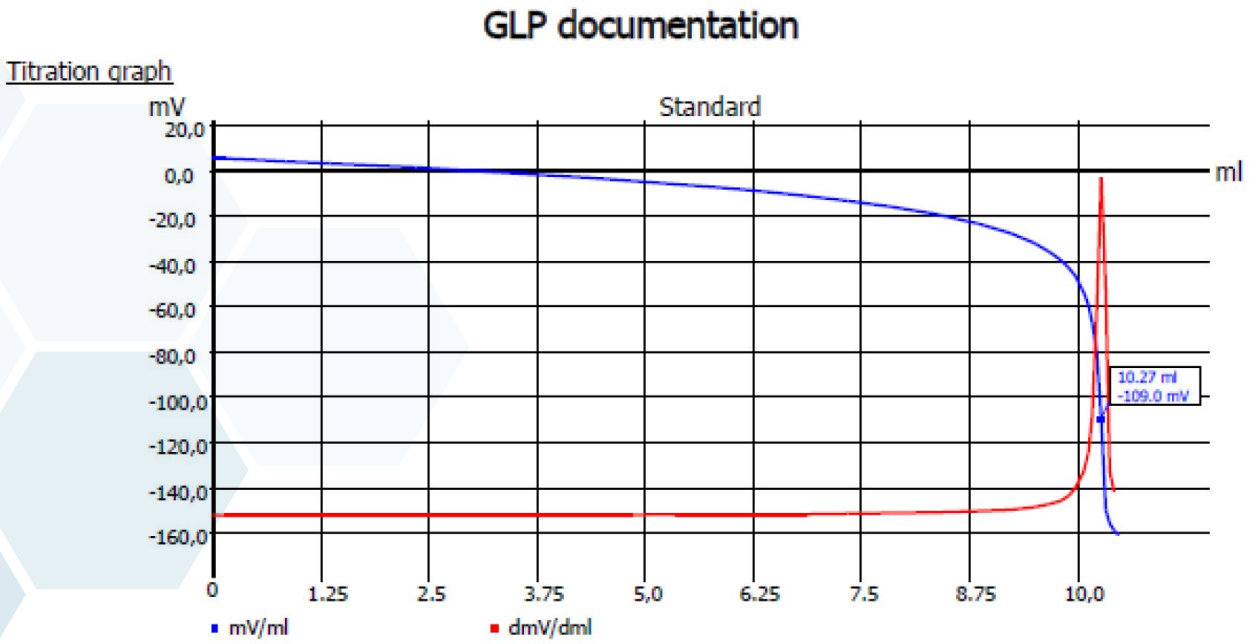
The standardisation of the titrant is carried with $CaCO_3$ titrimetric standard (e-g. from Merck or Sigma Aldrich). Weigh in about 0.5 g of the titrimetric standard in a 100 ml volumetric flask (note the exact amount of the weight e.g. 0.5043 g). Add about 20 ml distilled or DI water and shake it a little bit. Add then 12 ml HCl 1 mol/l and wait until all CO2 is completey degassed. Fill up to the 100 mark with distilled/DI water.

From this standard solution take exactly 10.00 ml (volumetric pipette) and pipette it into a 150 ml beaker. Add 80 ml of distilled 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 $CaCO_3$ 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.

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*F2)/((EQ1-B)*M*F1) -> 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		

Method Data Overview

Method Name	Titer EGTA
Method type:	Automatic Titration
Measured Value	mV
Titration Mode:	Dynamic
Dynamic	Flat

Created at:	12/19/13 17:34:59
Last Modification:	12/19/13 17:34:59
Documentation:	GLP

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 %
Maximum Dosing Volume	20.00 mL

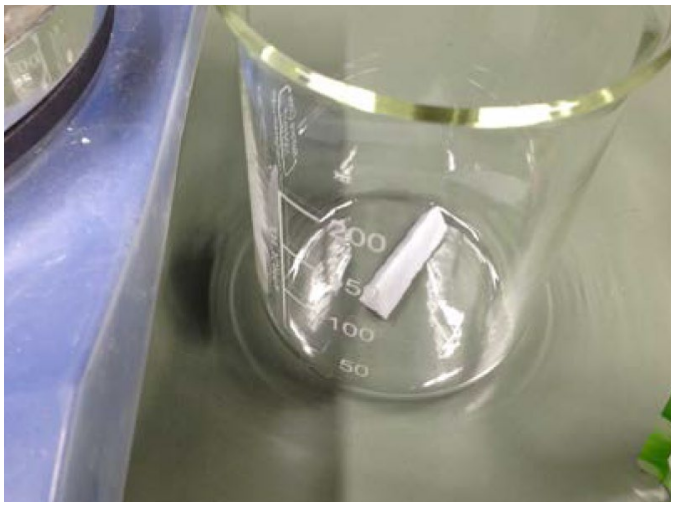
Filling Speed:	30s

Unit Values

Unit Size:	20 mL
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

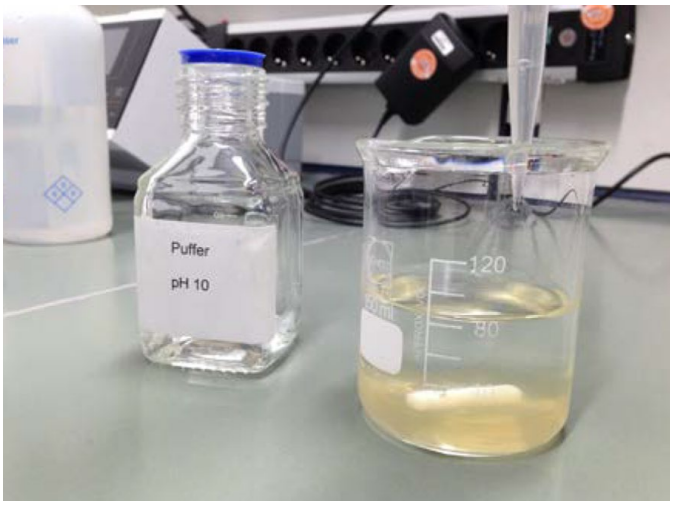
Sample titration

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



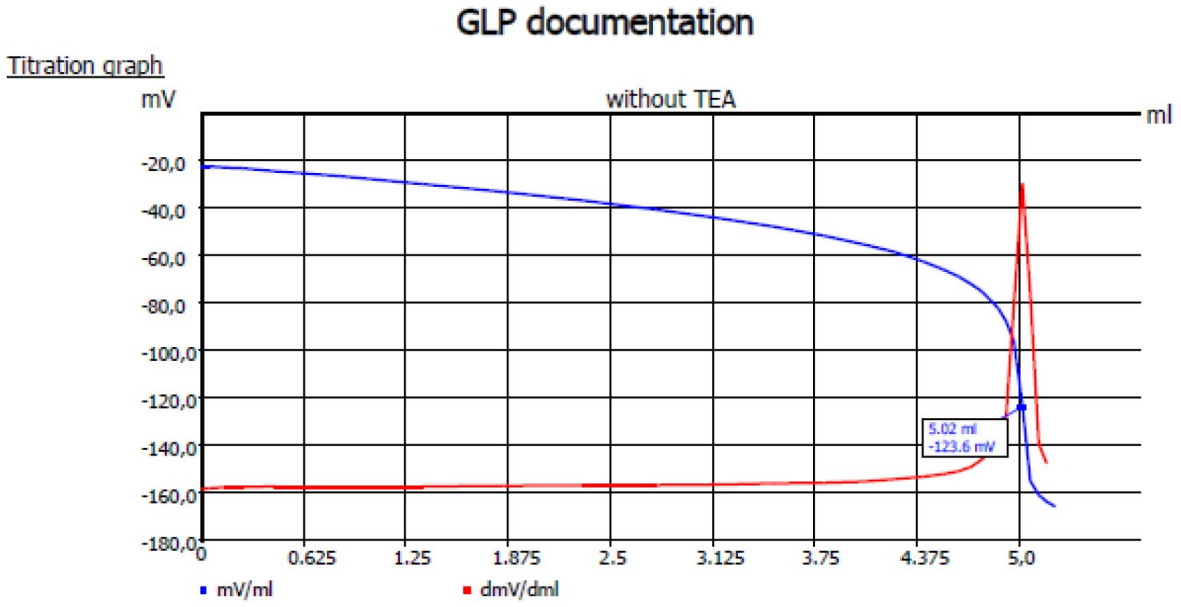
As you can see the sample is complexly homogenous. We stirred the sample at a temperature of 35 - 40 °C with a stirring speed of 750 - 1000 rpm. After 15 minutes the sample is homogenous.

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/homogeize the sample completely in the water/buffer solution:

Sample Result and Method



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

Calculation Formula			
Calcium mg/100g:	(EQ1- B)*T*M*F1/(W*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

Method Data Overview

Method Name	Calcium
Method type:	Automatic titration
Measured Value	mV
Titration Mode:	Dynamic
Dynamic	Flat

Created at:	12/17/13 18:19:46
Last Modification:	12/17/13 18:19:46
Documentation:	GLP

Measuring Speed / Drift	User-defined	Minimum holding time Maximum holding time Measuring time Drift	07 s 15 s 04 s 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 %
Maximum Dosing Volume	20.00 mL

Filling Speed:	30 s

Unit Values

Unit Size:	20 mL
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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