Quantitative analysis of ascorbic acid with specific titrant DCPIP

Application

Use

This method is used to determine the content of ascorbic acid (Vitamin C) with the specific titrant 2,6-Dichlorphenolindophenol (DCPIP).

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 brown glass bottle for titrant complete

Electrodes

- Electrode: Pt 62 or Pt 6280 with cable L 1 A

Reagents

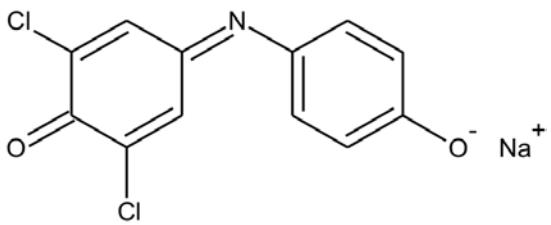
- Titration agent: 2,6-Dichlorphenolindophenol solution $(C_{12}H_7Cl_2NO_2)$ 0,001mol/L
- Standardisation ascorbic acid solution $(C_6H_8O_6)$ (prepared freshly)
- Others: oxalic acid solution $(H_2C_2O_4)$ 10% and sodium acetate solution (CH_3COONa) 10%

Description

Preparation of the 2,6-Dichlorphenolindophenol solution (0,001 mol/L)

163,1 mg 2,6-Dichlorphenolindophenol are weighed into a 400 mL beaker and filled up to 250 mL with distilled water. This solution is than stirred for 20 minutes at 50°C. After this it is filltrated into a 500 mL volumetric flask. For stabilisation 50 mg potassium hydrogen carbonate may be added. After that the flask is filled up with distilled water.

chemical structure of 2,6-Dichlorphenolindophenol:



Preparation of the sodium acetate solution

10 g sodium acetate are weighed into a 100 mL volumetric flask and filled up with distilled water.

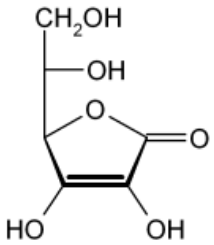
Preparation of the oxalic acid solution

10 g oxalic acid are weighed into a 100 mL volumetric flask and filled up with distilled water.

Preparation of the ascorbic acid solution

50 mg ascorbic acid (p.a.) are weighed into a 100 mL volumetric flask, dilluted in 10-20 mL oxalic acid and filled up with distilled water.

chemical structure of ascorbic acid:



Standardisation of the 2,6-Dichlorphenolindophenol

In a 50 mL beaker a mixture of 15 mL oxalic acid, 1 mL sodium acetate solution and 15 mL distilled water is made. After that 1 mL of the ascorbic acid solution is added. This solution is then titrated with the 2,6-Dichlorphenolindophenol solution.

calculation (t(DCPIP)):

$$t(DCPIP) = \frac{m(\text{ascorbic acid})}{(V(DCPIP) \cdot 0{,}176)}$$

t(DCPIP): titer of the 2,6-Dichlorphenol-indophenol solution
m(ascorbic acid): amount of ascorbic acid [mg] (here 0,5 mg)
V(DCPIP): consumption of the 2,6-Dichlorphenol-indophenol solution [mL]

The factor 0,176 is the product of the concentration of the 2,6-Dichlorphenolindophenol solution and the molar mass of ascorbic acid. 1mL of the 2,6-Dichlorphenolindophenol solution complies with 0,176 mg ascorbic acid.

Titration

In a 150 mL beaker 0,5-5 g of the sample are weighed exactly. Then 40 mL oxalic acid and 1 mL sodium acetate solution are added. The whole solution is now stirred for 5 minutes. After adding 40 mL distilled water it is titrated with the 2,6-Dichlorphenolindophenol solution.

chemical equation:

$$\underset{\text{ascorbic acid}}{C_6H_8O_6} + \underset{\text{2,6-Dichlorphenolindophenol}}{C_{12}H_7Cl_2NO_2} \rightarrow \underset{\text{dehydroascorbic acid}}{C_6H_6O_6} + C_{12}H_9Cl_2NO_2$$

calculation:

1.) result in %:

$$\frac{V(DCPIP) \cdot t(DCPIP) \cdot 0{,}176 \cdot 100}{m(\text{sample}) \cdot 1000}$$

t(DCPIP): titre of the 2,6-Dichlorphenol-indophenol solution
V(DCPIP): consumption of the 2,6-Dichlorphenol-indophenol solution [mL]
m(sample): amount of the sample [g]

factor 0,176: see above
The factor 1000 (under the fraction stroke) is the result of the conversion from g into mg. Because the result should be indicated in %, the numerator has to be multiplied with 100.

2.) result in mg/100g:

$$\frac{V(DCPIP) \cdot t(DCPIP) \cdot 0{,}176 \cdot 100}{m(\text{sample})}$$

t(DCPIP): titer of the 2,6-Dichlorphenol-indophenol solution
V(DCPIP): consumption of the 2,6-Dichlorphenol-indophenol solution [mL]
m(sample): amount of the sample [g]

factor 0,176: see above

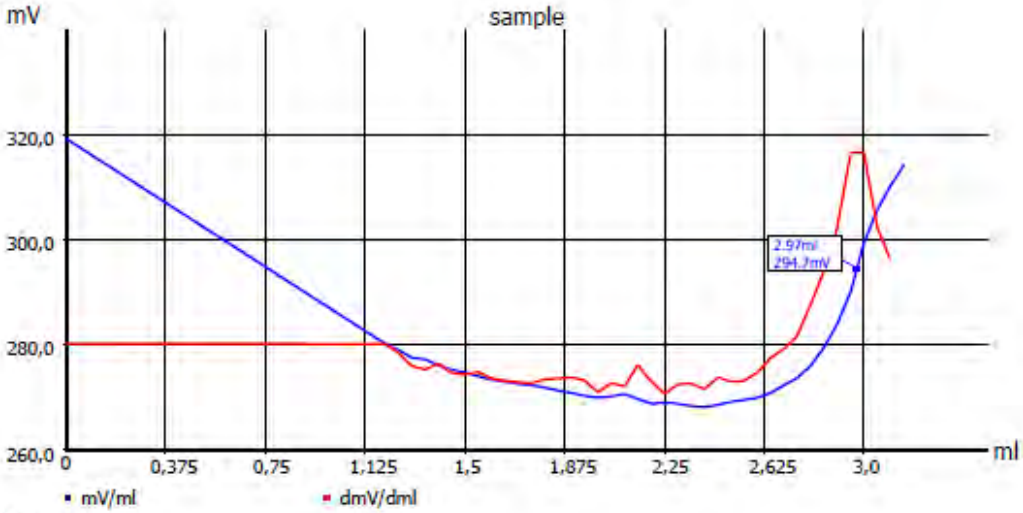
Because the result should be indicated in mg/100g, the numerator has to be multiplied with 100.

Methods

standardisation (page 1):

GLP-Dokumentation

Titrationsdiagramm



Methodendaten

Methodenname:	Titer 2,6 DCPIP	Titrationsdauer:	5 m 46 s
Enddatum:	15.02.12	Endzeit:	14:23:15

Titrationsdaten

Proben ID:	ohne	Einwaage:	0.5020 g
Start mV:	320.4 mV	End mV:	314.3 mV
EQ:	2.974 ml / 294.7 mV	c:	0.958 mmol/l

Berechnungsformel

c:	(W*F2)/((EQ1-B)*M*F1)
Mol (M):	0.17612

Einwaage (W):	man	Faktor 2 (F2):	1.0000
Blindwert (B):	0.0000 ml	Faktor 1 (F1):	1.0000

standardisation (page 2):

Method data

Method name:	Titer 2.6 DCPIP	Created at:	04/26/13 12:09:46
Method type:	Automatic titration	Last modification:	04/29/13 12:45:51
Measured value:	mV	Damping settings:	None
Titration mode:	Linear	Documentation:	GLP
Linear steps:	0.050 ml		
Measuring speed / drift:	User-defined:	minimum holding time:	05 s
		maximum holding time:	10 s
		Measuring time:	02 s
		Drift:	40 mV/min
Initial waiting time:	0 s		
Titration direction:	Increase		
Pretitration:	1.200 ml	Delay time:	10 s
End value:	Off		
EQ:	On (1)		
Slope value:	User-defined	Value:	80

Dosing parameter

Dosing speed:	100.00 %	Filling speed:	30 s
Maximum dosing volume:	6.00 ml		

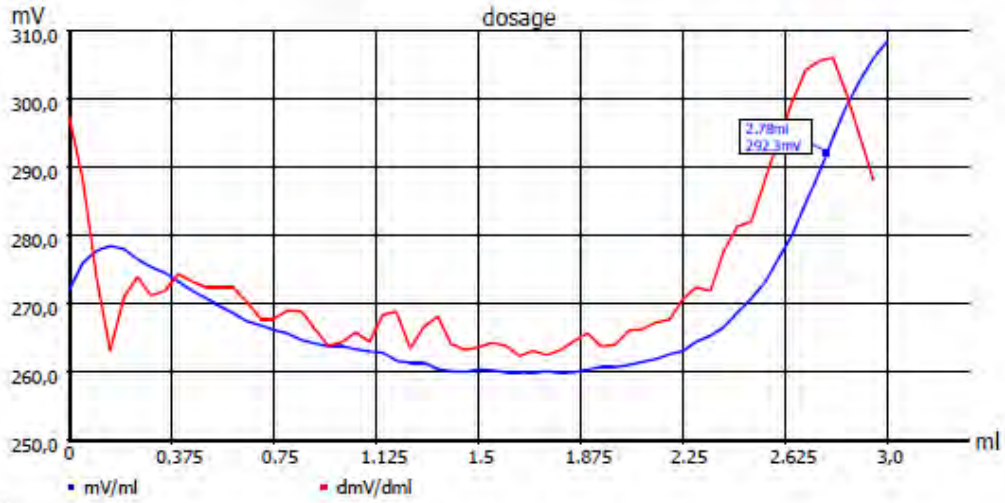
Calculation formula

Titer:	(W*F2)/((EQ1-B)*M*F1) -> WA	Mol (M):	0.17612
Unit:	mmol/l	Decimal places:	3
Weight (W):	man	Factor 2 (F2):	1.0000
Blank value (B):	M01	Factor 1 (F1):	1.0000
Statistics:	3		

sample titration (page 1):

GLP documentation

Titration graph



Method data

Method name:	Vitamin C	Titration duration:	7 m 22 s
End date:	16.02.12	End time:	14:11:50

Titration data

Sample ID:	dosage	Weight:	1.1215 g
Start mV:	272.5 mV	End mV:	308.6 mV
EQ:	2.779 ml / 292.3 mV	Vitamin C mg/100g:	41.5

Calculation formula

Vitamin C mg/100g:	(EQ1-B)*T*M*F1/(W*F2)
Mol (M):	17.60000

Blank value (B):	0.0000 ml	Titre (T):	0.95100000
Factor 1 (F1):	1.0000	Weight (W):	man
Factor 2 (F2):	1.0000		

sample titration (page 2):

Method data overall view

Method name:	Vitamin C	Created at:	02/16/12 13:54:17
Method type:	Automatic titration	Last modification:	02/16/12 13:58:25
Measured value:	mV		
Titration mode:	Linear		
Linear steps:	0.050 ml		
Measuring speed / drift:	7 s		
Initial waiting time:	0 s		
Titration direction:	Increase		
Pretitration:	Off		
End value:	Off		
EQ:	On		
slope value:	user-defined	Value:	80

Dosing parameter

Dosing speed:	100 %	Filling speed:	30 s
Maximum dosing volume:	10.00 ml		

Unit values

Unit size:	10ml
Unit ID:	10035409
Reagent:	La(NO3)3
Batch ID:	no comment
Concentration [mol/l]:	0.10000
Determined at:	09/21/11 23:15:06
Expire date:	12/31/21
Opened/compounded:	09/20/11
Test according ISO 8655:	01/01/00
Last modification:	09/21/11 16:15:13

Hints

Normally the solutions should be titrated under nitrogen. This is not necessary, if you work as quickly as possible. Additionally all solution (especially the ascorbic acid solution) should be prepared freshly.

If you have any questions concerning the application, you are welcome to contact us.

Literature

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