Polarographic determination

Instruments

- VA instrument
  capable of operating a mercury electrode and supporting DP mode

Electrode

WE	Multi-Mode Electrode pro Mercury drop capillary	6.1246.120 6.1226.030
AE	Separate Pt rod electrode	6.0343.000
RE	Ag/AgCl reference electrode c(KCl) = 3 mol/L Electrolyte vessel filled with c(KCl) = 3 mol/L	6.0728.020 6.1245.010

Reagents

- Sodium acetate anhydrous, CH_3COONa, for analysis, CAS 127-09-3
  or
  Sodium acetate trihydrate, $CH_3COONa \cdot 3H_2O$, for analysis, CAS 6131-90-4
- Acetic acid, w(CH_3COOH) = 100%, for analysis, CAS 64-19-7
- L-ascorbic acid (vitamin C), $C_6H_8O_6$, for analysis, CAS 50-81-7
- Ultrapure water, resistivity >18 MΩ·cm (25 °C), type I grade (ASTM D1193)

Solutions

Acetate buffer pH = 4.6	8.2 g sodium acetate anhydrous or 13.61 g sodium acetate trihydrate is dissolved in ultrapure water. 6 mL acetic acid is added and the solution is made up to 100 mL with ultrapure water.

Standard

Ascorbic acid standard solution 50 mg	β(Ascorbic acid) = 1 g/L 500 mg L-ascorbic acid is dissolved in degased ultrapure water and made up to 50 mL. This solution has to be freshly prepared every day.

Sample preparation

- Drinks, fruits and vegetable juices can be analyzed directly
- For tablets and other vitamin preparations, a diluted solution in degased ultrapure water is first prepared, an aliquot of which is then used for the determination.
- Foods, stimulants and animal feeds are extracted using appropriate procedures.

Analysis

Measuring solution

10 mL ultrapure water

1 mL acetate buffer pH 4.6

0.5 mL sample

10 mL ultrapure water, 1 mL acetate buffer pH = 4.6 and 0.5 mL sample are pipetted into a polarographic vessel. After degasing the measuring solution with nitrogen for 300 s a DP polarogram is recorded using the parameters given below.

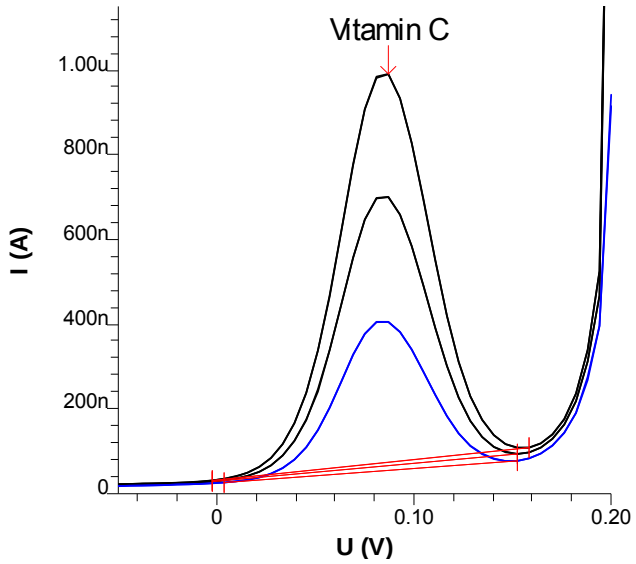
The concentration of ascorbic acid is quantified by two additions of ascorbic acid standard solution β(ascorbic acid) = 1 g/L.

Parameters

Determination	
No. of additions	2
No. of replications	2
Voltammetric	
Electrode	DME
Measuring mode	DP – Differential Pulse
Stirring speed	2000 min^{-1}
Hydrodynamic measurement	No
Sweep	
Equilibration time	10 s
Start potential	-0.05 V
End potential	0.2 V
Pulse amplitude	0.05 V
Pulse time	0.04 s
Potential step	0.06 V
Potential step time	0.6 s
Sweep rate	0.01 V/s
Substance + calibration	

Calibration	Standard addition
Name	Vitamin C
Peak potential	0.1 V
Tolerance	0.05 V
Baseline	Linear Automatic

Example determination

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Vitamin C
c =  243.506 mg/L
+/-    0.999 mg/L (0.41%)
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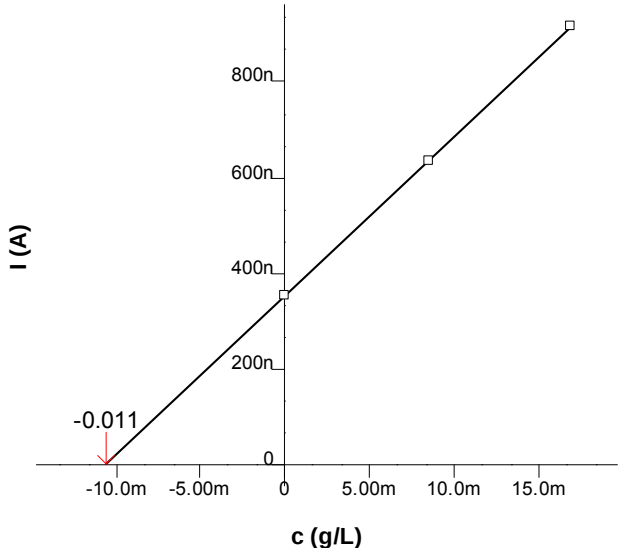

Fig. 4: Polarogram and calibration curve of a determination of ascorbic acid in orange juice.

Comments

- Ascorbic acid is sensitive against oxygen and light. Therefore it is recommended to degas the deionized water for the preparation of the standard solution and keep the standard in the dark. The standard solution should only be used on the day of preparation.
- Larger quantities of chloride ions (e.g. in sauerkraut) interfere with the polarographic determination of ascorbic acid. They are removed from the sample solution by precipitation with silver nitrate and subsequent filtration.

References

- D. Amin
  Application of differential pulse polarography to the assay of ascorbic acid
  Microchem. J., 28, 1983, 174 – 179.
- S. Kozar, A. Bujak, J. Eder-Trifunovic, G. Kniewald
  Determination of L-ascorbic acid in fresh and processed fruit and vegetables by differential pulse polarography
  Fresenius Z. Anal. Chem., 329, 1988, 760 – 763.

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