



Ideometric Titration of Vitamin C

(According to standard NF.EN.ISO 660-1999)

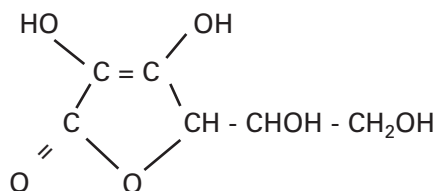
Introduction

Vitamin C (ascorbic acid or its sodium salt) is naturally present in fresh fruit juices or vegetables. It is also used in some pharmaceutical products. The food industry makes use of vitamin C as an anti-oxidation additive in cooked pork meats or canned products to avoid oxygen action (E300 for ascorbic acid or E301 for the sodium salt according to European regulations).

Summary

Many redox titrations can be used for Vitamin C determination. In some cases, depending on the media, iodometric back titration is possible, for example in fresh lemon juice or preservatives for food products.

Vitamin C formula



The redox titration is a two-step reaction.

First step

Oxidation of Vitamin C with excess iodine (I₂).

The redox reaction involves the two -OH groups according to:



Second step

The iodine excess is then determined by sodium thiosulphate according to:



As results are generally expressed in mg/l, % or mg/kg, note that the molar weight of the Vitamin C is 176.3 g/mol corresponding to the global formula C₆H₈O₆. The molar weight of sodium ascorbate (C₆H₇O₆Na) is 198.3 g/mol.

Electrode and reagents

Electrode for pre-set end point titration

M241Pt2-8 Metal electrode, double platinum (part no. E32M002) or M231Pt2 Metal electrode, double platinum (part no. E32M001) with adapter part no. A94P801

Electrode for inflection detection

MC3051Pt-9 Metal electrode combined, platinum (part

no. E31M003) with CL114 cable (part no. A94L114)

Reagents

0.05 mol/l iodine solution:

Dissolve 40 g of potassium iodide in 50 ml of distilled water then add 12.69 g of analytical grade iodine, wait for complete dissolution and complete to 1.000 ml with a volumetric flask.

Store the solution in a brown glass bottle.

0.1 mol/l thiosulphate solution:

Dissolve 24.8181 g of Na₂S₂O₃ · 5H₂O in 500 ml of freshly distilled water (or freshly boiled and cooled deionised water) and 2 or 3 drops of CHCl₃ (or 0.4 g of NaOH) and complete to 1000 ml using a volumetric flask.

Wait for one day and filter the solution if necessary (precipitation of sulphur can occur).

Stock the solution in a brown glass flask.

These two solutions are commercially available.

Sodium acetate buffer solution

Dissolve 85 g of sodium acetate (CH₃COONa) in water, add 60 ml of glacial acetic acid and dilute to 1000 ml with freshly distilled water. This solution contains approximately 1 mole of CH₃COOH and 1 mole of CH₃COONa per litre.

Freshly distilled water.

End Point Titration settings

Burette volume:	25 ml (for iodine solution)
Burette volume:	10 ml (for thiosulphate solution)
Stirring speed:	400 rpm
Working mode:	mV i>0
Current:	DC
Current value:	5 µA
Start timer:	10 seconds
Maximum volume:	10 ml
Direction:	Increasing mV
Minimum speed:	0.2 ml/min
Maximum speed:	2.00 ml/min
Back Titration:	Automatic (see Back titration note)
Excess reagent:	I ₂ 0.05M
Excess volume:	15 ml.
Number of end points:	1

End point:	200 mV
Proportional band:	190 mV
End point delay:	5 seconds
Sample unit:	g or ml
Sample amount:	Depending on the product (see below)
Result:	%
Molar weight:	198 g/mol
Excess:	2Smp + 2Exc
Reaction:	1Exc + 2Titr (see Result note)

Inflection Detection Settings Continuous IP mode

Burette volume:	25 ml (for iodine solution)
Burette volume:	10 ml (for thiosulphate solution)
Stirring speed:	400 rpm
Working mode:	mV
Start timer:	10 seconds
Maximum volume:	10 ml
Stop point:	150 mV
Direction:	decreasing mV
Minimum speed:	0.2 ml/min
Maximum speed:	4.00 ml/min
Smoothing parameter:	5
Back Titration:	Automatic (see Back titration note)
Excess reagent:	I ₂ 0.05M
Excess volume:	15 ml
Inflection No 1	
Minimum ordinate:	230 mV
Maximum ordinate:	350 mV
Sample unit:	g or ml
Sample amount:	Depending on the product (see below)
Result:	%
Molar weight:	198 g/mol
Excess:	2Smp + 2Exc
Reaction:	1Exc + 2Titr (see note Result)

Procedure

Using a biburette Titration Manager, fill burette 1 with thiosulphate solution and burette 2 with iodine solution.

With a monoburette Titration Manager, fill the burette with thiosulphate solution, and add the iodine solution manually.

Connect the electrode.

Add 10 ml of sodium acetate buffer solution and, if necessary, freshly distilled water to the sample. Dip electrode and delivery tip in the solution and run the titration.

Procedure

The general formula for result expression is:

$$C_{\text{smp}} = 1/K_{\text{smp}} * (C_{\text{exc}} * V_{\text{exc}} - (C_{\text{titr}} * V_{\text{titr}}/2))$$

- $1/K_{\text{smp}}$ is a function of the sample amount and unit
- C_{exc} concentration of the iodine solution in mol/l
- V_{exc} added volume of iodine solution in ml
- C_{titr} concentration of titrant solution in mol/l (thiosulphate solution)
- V_{titr} used volume of thiosulphate solution in ml

Using the above-mentioned coefficients for excess and reaction, the Titration Manager takes into account this formula for result calculation.

For 3 determinations with a food preservative

This is a mixture of sodium chloride, sodium acetate and vitamin C

Mean: 6.55%

Working Range

Using an iodine solution with a concentration of 0.05 mol/l, 1 ml of the iodine solution reacts with 0.05 mmole corresponding to 8.81 mg of Vitamin C. Take this into account to determine the approximate amount of iodine solution necessary for the reaction and add an excess corresponding to approximately 50% of this volume.

For low concentrations, it is possible to use 0.005 mol/l iodine solution and 0.01 mol/l thiosulphate solution.

Notes

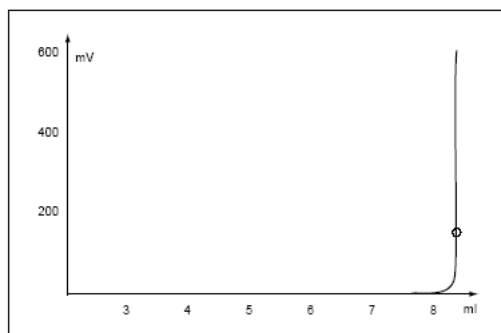
Back titration

Set "AUTOMATIC" using a biburette Titration Manager. With a monoburette system, set "MANUAL" and enter excess reagent concentration and volume. Other settings are unchanged.

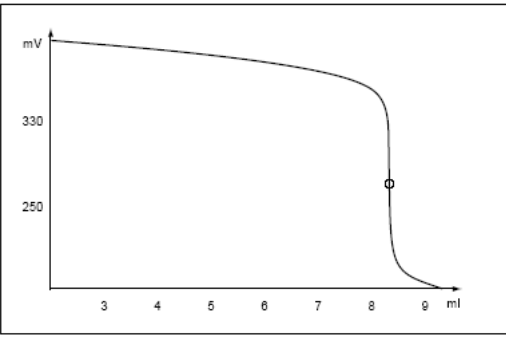
Result

Note that, in back titration, the Titration Manager asks for the SAME COEFFICIENTS for sample and titrant. It is therefore compulsory to set the titration system as indicated.

Curves



End point titration



Continuous IP mode