College of Science

UNIVERSITY OF CANTERBURY
Te Whare Wānanga o Waitaha
CHRISTCHURCH NEW ZEALAND

Determination of Vitamin C Concentration by Titration

(Redox Titration Using Iodine Solution)

Safety

Lab coats, safety glasses and enclosed footwear must be worn at all times in the laboratory.

Introduction

This method determines the vitamin C concentration in a solution by a redox titration using iodine. Vitamin C, more properly called ascorbic acid, is an essential antioxidant needed by the human body (see additional notes). As the iodine is added during the titration, the ascorbic acid is oxidised to dehydroascorbic acid, while the iodine is reduced to iodide ions.

$$\text{ascorbic acid} + I_2 \rightarrow 2\,I^- + \text{dehydroascorbic acid}$$

Due to this reaction, the iodine formed is immediately reduced to iodide as long as there is any ascorbic acid present. Once all the ascorbic acid has been oxidised, the excess iodine is free to react with the starch indicator, forming the blue-black starch-iodine complex. This is the endpoint of the titration.

The method is suitable for use with vitamin C tablets, fresh or packaged fruit juices and solid fruits and vegetables.

NB: This method is more straight forward than the alternative method using potassium iodate, but as the potassium iodate solution is more stable than the iodine as a primary standard, the alternative method is more reliable.

Equipment Needed

burette and stand

100 mL or 200 mL volumetric flask

20 mL pipette

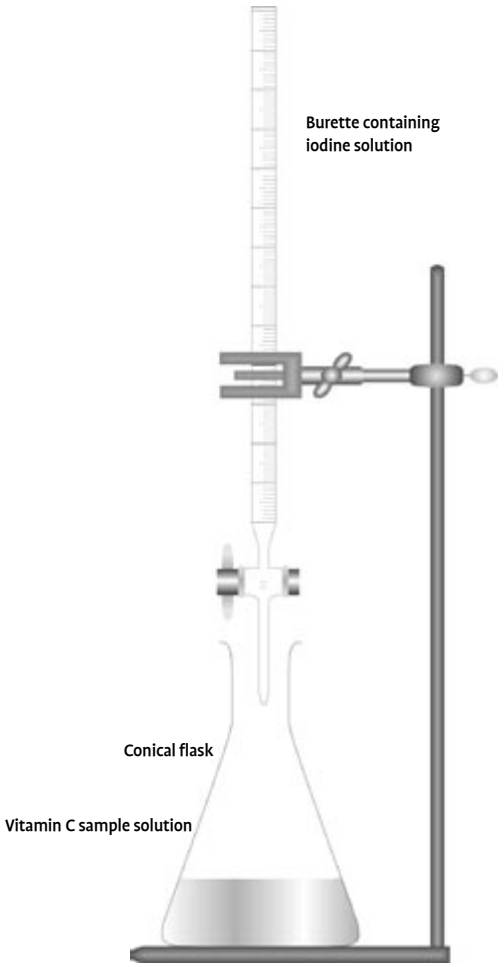
10 mL and 100 mL measuring cylinders

250 mL conical flasks

Solutions Needed

Iodine solution: (0.005 mol L^{-1}). Weigh 2 g of potassium iodide into a 100 mL beaker. Weigh 1.3 g of iodine and add it into the same beaker. Add a few mL of distilled water and swirl for a few minutes until iodine is dissolved. Transfer iodine solution to a 1 L volumetric flask, making sure to rinse all traces of solution into the volumetric flask using distilled water. Make the solution up to the 1 L mark with distilled water.

Starch indicator solution: (0.5%). Weigh 0.25 g of soluble starch and add it to 50 mL of near boiling water in a 100 mL conical flask. Stir to dissolve and cool before using.



Method

Sample Preparation

For vitamin C tablets: Dissolve a single tablet in 200 mL of distilled water (in a volumetric flask if possible).

For fresh fruit juice: Strain the juice through cheesecloth to remove seeds and pulp which may block pipettes.

For packaged fruit juice: This may also need to be strained through cheesecloth if it contains a lot of pulp or seeds.

For fruits and vegetables: Cut a 100 g sample into small pieces and grind in a mortar and pestle. Add 10 mL portions of distilled water several times while grinding the sample, each time decanting off the liquid extract into a 100 mL volumetric flask. Finally, strain the ground fruit/vegetable pulp through cheesecloth, rinsing the pulp with a few 10 mL portions of water and collecting all filtrate and washings in the volumetric flask. Make the extracted solution up to 100 mL with distilled water.

Alternatively the 100 g sample of fruit or vegetable may be blended in a food processor together with about 50 mL of distilled water. After blending, strain the pulp through cheesecloth, washing it with a few 10mL portions of distilled water, and make the extracted solution up to 100 mL in a volumetric flask.

Titration

1. Pipette a 20 mL aliquot of the sample solution into a 250 mL conical flask and add about 150 mL of distilled water and 1 mL of starch indicator solution.
2. Titrate the sample with 0.005 mol L^{-1} iodine solution. The endpoint of the titration is identified as the first permanent trace of a dark blue-black colour due to the starch-iodine complex.
3. Repeat the titration with further aliquots of sample solution until you obtain concordant results (titres agreeing within 0.1 mL).

Calculations

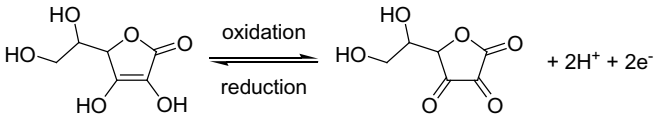
1. Calculate the average volume of iodine solution used from your concordant titres.
2. Calculate the moles of iodine reacting.
3. Using the equation of the titration (below) determine the number of moles of ascorbic acid reacting.

$$\text{ascorbic acid} + I_2 \rightarrow 2\,I^- + \text{dehydroascorbic acid}$$

4. Calculate the concentration in mol L^{-1} of ascorbic acid in the solution obtained from fruit/vegetable/juice. Also, calculate the concentration, in mg/100mL or mg/100g of ascorbic acid, in the sample of fruit/vegetable/juice.

Additional Notes

1. Iodine stains both skin and clothing so proper care is advised. If staining does occur, alcohol may remove skin stains and cleaners are available for fabric stains.
2. Vitamin C, or ascorbic acid, is a water soluble antioxidant that plays a vital role in protecting the body from infection and disease. It is not synthesised by the human body and therefore must be acquired from dietary sources – primarily fruits and vegetables. The chemical structure and antioxidant (reducing) action of ascorbic acid are illustrated in the redox half equation below:
3. The concentration of the prepared iodine solution can be more accurately determined by titration with a standard solution of ascorbic acid or a standard solution of potassium thiosulfate using a starch indicator. This should be done if possible as iodine solutions can be unstable.



ascorbic acid dehydroascorbic acid

4. The average titre volume should ideally be in the range of 10 – 30 mL. If the titre required for a 20 mL aliquot of sample solution is well outside this range then a larger or smaller aliquot volume should be chosen. If the volume of the titre is too low, dilute the standard. If the titre volume is too high, dilute the sample.
5. Ascorbic acid is susceptible to oxidation by atmospheric oxygen over time. For this reason, the samples should be prepared immediately before the titrations. However, if the samples have to be prepared several hours earlier, oxidation can be minimised by the addition of a small amount of oxalic acid (eg 1 g oxalic acid per 100 mL of sample solution).
6. Identification of the endpoint in this titration is significantly affected by the colouration of the sample solution used. If the solutions are colourless or are pale in colour, there is no problem identifying

Figure 1 Vitamin C tablet. Left photo: before endpoint, added iodine reacts with ascorbic acid leaving the solution colourless. Centre photo: At the titration endpoint all the ascorbic acid has reacted and the excess iodine reacts with the starch indicator to give a pale blue colour. Right photo: If addition of iodine is continued after the endpoint, further iodine-starch complex is formed. NB: in each of these images a flask showing the pale blue colour of the endpoint is shown for comparison.

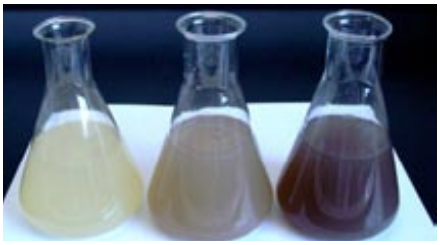


Figure 2 Commercial fruit juice. Left flask: before the endpoint, the colour of the solution reflects the pale yellow colour of the fruit juice. Centre flask: At the titration endpoint all the ascorbic acid has reacted and any excess iodine reacts with the starch indicator to form a dark blue-black complex. In this case the result is a darkening of the solution's colour from yellow to brown-grey. Right flask: This illustrates the effect of adding just a mL or two more of iodine solution after the endpoint is reached, resulting in the formation of further iodine-starch complex.

Figure 3 Freshly squeezed orange juice. Left flask: before the endpoint, the colour of the solution reflects the bright orange colour of fresh orange juice and is unaffected by addition of iodine. Centre flask: Once all the ascorbic acid has been oxidised, a slight excess of added iodine complexes with the starch indicator, giving the solution a green colour in this case. This is the endpoint of the titration. Right flask: If further iodine solution were to be added, the solution's green colour would become darker as shown.

Figure 4 Red capsicum. Left flask: Before the endpoint, the solution retains its original colour. Centre flask: Once all the ascorbic acid has been oxidised, a slight excess of added iodine forms a dark complex with starch indicator giving a purple colour. This is the titration endpoint. Right flask: If a further mL or two of iodine were to be added after the endpoint, the solution would develop the dark purple colour shown here.

the endpoint. For strongly coloured juices there can be a problem with the endpoint and it is advised to carry out a "rough" titration in order to become familiar with any distinct colour change which occurs at the endpoint, (it may just be a darkening of the colour) This will also help by establishing an approximate volume of iodine solution required.

7. The above method may be used to carry out a number of interesting investigations regarding the concentration of vitamin C in various foods and drinks:
   - Vitamin C content of different types of fruits vegetables/juices.
   - Vitamin C content of different types/brands of vitamin tablets.
   - Vitamin C content of tablets or food/drink in the presence and absence of added oxalic acid or metal ions over various periods of time.
   - Vitamin C content of food/drink before and after subjection to cooking conditions.
   - Vitamin C content of fruits/vegetables at different stages of ripeness.

Contact Us

If you have any questions or comments relating to this experiment, please contact us. Please note that this service is for senior school chemistry students in New Zealand only. We regret we are unable to respond to queries from overseas.

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