Ascorbic Acid as a Standard for Iodometric Titrations

An Analytical Experiment for General Chemistry

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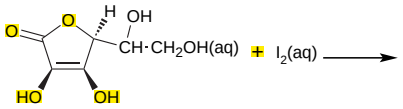
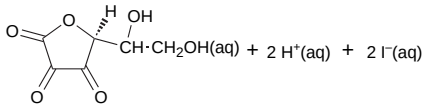
Vitamin C is a familiar name to chemistry students, and simple experiments that determine the amount of vitamin C in pharmaceutical prescriptions are important in the general chemistry laboratory (*1*, *2*). This procedure involves an ascorbic acid redox titration with iodine solution using starch as an indicator:



Ascorbic acid ($C_6H_8O_6$)



Dehydroascorbic acid ($C_6H_6O_6$)

The end point of this titration is determined by the first excess of iodine in the reaction medium that reacts with starch, forming a complex with an intense dark blue-violet color (*3*).

In conventional analytical iodometric experiments, most of the experimental work is involved with the standardization of the iodine solution. This procedure determines the iodine concentration using a solution of sodium thiosulfate ($Na_2S_2O_3$), which is itself standardized against a primary standard such as potassium dichromate or potassium iodate in an acid aqueous media (*4*). Both of these compounds produce iodine in a redox reaction that is titrated with a thiosulfate solution using a starch solution as indicator (*5*). The concentration of the thiosulfate solution is then calculated considering the number of moles of the primary standard used. From an analytical point of view this is not very accurate, because the titration errors are cumulative.

The purpose of this article is to show that ascorbic acid is an excellent compound to use for the standardization of iodine solutions. Using ascorbic acid, the standardization of iodine solutions is fast, accurate, and easy compared with the conventional methods. During a single laboratory period, the students in a general chemistry course are able to standardize an iodine solution and determine, with good precision, the amount of vitamin C in some pharmaceutical tablets.

Experimental Procedure

Glassware and Reagents

All reagents were of analytical grade or better. A buret (50 mL) and a microburet (5 mL) were used without calibration. A pipet (10 mL) was calibrated with water (5 replicates), giving (9.970 ± 0.004) mL.

Preparation of Solutions

Iodine (3.8 g) and 20 g of potassium iodide (KI) were completely dissolved in 20 mL of distilled water and the mixture was diluted to 1 L. This iodine solution (0.015 mol L^{-1}) was stored in a dark flask for further standardization.

Twenty-five grams of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) and 0.1 g of sodium carbonate (Na_2CO_3) were dissolved in distilled water and diluted to 1 L in a volumetric flask. The function of carbonate in the 0.1 mol L^{-1} thiosulfate solution is to prevent decomposition by light and bacterial action.

Starch (0.1 g) was mixed with 100 mL of distilled water and stirred until the almost complete dissolution of the starch. The mixture was then heated to boiling for 2 minutes to obtain total dissolution. The starch indicator solution was left to cool and stored in a plastic bottle.

Potassium dichromate ($K_2Cr_2O_7$) was dried for 2 h at 393 K before use. Approximately 0.20 g of the dried $K_2Cr_2O_7$ was weighed within 0.00001 g, quantitatively transferred to a conical flask, and dissolved in 50 mL of distilled water. Two grams of potassium iodide (weighed to 0.01 g) and 8 mL of concentrated HCl were added. The iodine produced in the reaction was titrated with the sodium thiosulfate solution to the appearance of a pale yellow coloration. At this point, 3 mL of the starch solution was added, and the titration was continued until the disappearance of the blue color of the iodine–starch complex.

Standardization of the Iodine Solution with the Standardized $Na_2S_2O_3$ Solution

A 10-mL aliquot of the iodine solution was transferred to a conical flask, diluted with 40 mL of distilled water, and titrated with the previously standardized sodium thiosulfate solution until the brown color changed to pale yellow. Immediately, 2 mL of the starch solution was added and the titration was continued until the intense blue color disappeared.

Standardization of Iodine Solution with Ascorbic Acid

Approximately 70 to 90 mg of analytical grade ascorbic acid (Merck), dried at 393 K for 2 h and weighed to within 0.00001 g, was quantitatively transferred to a conical flask and dissolved in 50 mL of distilled water. Two milliliters of starch solution was added, and the colorless ascorbic acid

solution was immediately titrated with the iodine solution. The titration must be performed immediately to prevent air-oxidation of the ascorbic acid (*6*). The end point was determined when a pale blue color remained with the first excess of iodine.

Determination of Vitamin C in Pharmaceutical Preparations

A pharmaceutical tablet of 2 g of vitamin C (Redoxon, manufactured by Roche) was ground in a beaker and then quantitatively transferred to a 250-mL volumetric flask and diluted to the mark with distilled water. Aliquots of the solution were taken using a calibrated pipet and titrated with the standardized iodine solution as described above.

Results and Discussion

The conventional chemical procedure to standardize iodine solutions has the disadvantage of being very time consuming, because of the two chemical reactions shown below:

$$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6I^-(aq) \rightarrow 3I_2(aq) + 2Cr^{3+}(aq) + 7H_2O$$

$$6S_2O_3^{2-}(aq) + 3I_2(aq) \rightarrow 3S_4O_6^{2-}(aq) + 6I^-(aq)$$

According to these reactions, the concentration of the $Na_2S_2O_3$ solution is calculated by taking into account that each mole of $K_2Cr_2O_7$ reacts with 6 moles of $Na_2S_2O_3$. Using this conventional procedure the average value (3 replicates) of the concentration was (0.1078 ± 0.0001) mol L^{-1}, with a standard deviation of less than 0.2 parts per thousand.

The next step was the standardization of the iodine solution with the standardized thiosulfate solution. The average value (6 replicates) of the iodine concentration found was (0.0152 ± 0.0001) mol L^{-1}, with a standard deviation of 7 parts per thousand.

The results for the standardization of the same iodine solution using untreated ascorbic acid as a standard gave an average value (7 replicates) of (0.0152 ± 0.0001) mol L^{-1} for the concentration of the iodine solution, corresponding to an error of 3 parts per thousand. This value is the same as that obtained from the standardization of the iodine solution with standardized thiosulfate, within experimental error.

Using ascorbic acid, previously dried at 393 K for 2 h, as the standard, the average value (3 replicates) of the iodine concentration was (0.0152 ± 0.0001) mol L^{-1}, the same as used without any prior treatment. Thermogravimetric analysis of this ascorbic acid showed that its water content was very low (about 0.5%) even before drying, and so the standardization of the iodine solution is not affected when this procedure is carried out with undried ascorbic acid.

As recommended in the literature (*6*), a standard for titration has to be readily available, of high purity, stable in air, of relatively high molecular mass, soluble in titration solvent, and not hygroscopic so that it can be weighed accurately. As can be seen from the experimental results in comparison with the conventional procedure for iodometric titrations, the use of ascorbic acid as a standard can be suggested for this purpose. In addition, since there are fewer steps in the overall procedure, the cumulative experimental errors are smaller with ascorbic acid than with thiosulfate.

However, there is the disadvantage that ascorbic acid is not very stable when dissolved in water, because it scavenges oxygen. Thus, care should be taken in the laboratory work, and each titration must be performed individually immediately after the dissolution of this standard.

The analysis of a tablet of vitamin C, Redoxon, was made (3 replicates).The average value was (2.091 ± 0.002) g of vitamin C per tablet with a standard deviation of less than 1 part per thousand, which is in good agreement with the value shown in the description of the medication, provided by the manufacturer.

Since iodometric titrations are widely used in many senior courses, ascorbic acid can be used as a weighable standard when the experimental procedure involves the standardization of an iodine solution.

Conclusion

The standardization of an iodine solution using ascorbic acid as the weighable standard was shown to be accurate and precise. Since the titration is colorless, a clear and defined end point is achieved using starch, with results that are identical to those obtained using the conventional analytical procedure using standardized $Na_2S_2O_3$ solution. Thus, for a general chemistry experiment, ascorbic acid can be used instead of thiosulfate to standardize the iodine solution, provided that the titrations are performed immediately after the dissolution of the ascorbic acid. Use of ascorbic acid results in a simpler and much shorter procedure that gives good accuracy and precision for the analysis of vitamin C in pharmaceutical tablets.

Literature Cited

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