College of Science

UNIVERSITY OF CANTERBURY
Te Whare Wānanga o Waitaha
CHRISTCHURCH NEW ZEALAND

Determination of Iodate in Iodised Salt by Redox Titration

Safety

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

Introduction

New Zealand soil is low in iodine and hence New Zealand food is low in iodine. Until iodised salt was commonly used (starting in 1924), a large proportion of school children were reported as being affected by iodine deficiency – as high as 60% in Canterbury schools, and averaging 20 – 40% overall. In the worst cases this deficiency can lead to disorders such as goitre, and impaired physical and mental development.

In earlier times salt was "iodised" by the addition of potassium iodide; however, nowadays iodine is more commonly added in the form of potassium iodate (KIO_3). The Australia New Zealand Food Standards Code specifies that iodised salt must contain: "equivalent to no less than 25 mg/kg of iodine; and no more than 65 mg/kg of iodine".

In this method we determine the amount of iodate ($IO3^-$) in iodised salt by first reacting the iodate with added iodide (I^-), under acid conditions, to produce iodine:

$$IO_3^- + 5\ I^- + 6\ H^+ \rightarrow 3\ I_2 + 3\ H_2O$$

Then the resulting iodine is titrated with thiosulfate as follows:

$$I_2 + 2\ S_2O_3^{2-} \rightarrow 2\ I^- + S_4O_6^{2-}$$

Equipment and Materials Required

- iodised salt
- 0.002 mol L^{-1} sodium thiosulfate solution (see below for preparation)
- 1 mol L^{-1} hydrochloric acid
- 0.6 M potassium iodide solution (10 g solid KI made up to 100 mL with distilled water)
- 0.5% starch indicator solution (see below for preparation)
- 250 mL volumetric flask
- 50 mL pipette (or 20 and 10 mL pipettes)
- 250 mL conical flasks
- 10 mL measuring cylinder
- burette and stand
- distilled water

Method

1. **Preparation of 0.002 mol L^{-1} sodium thiosulfate solution:** Accurately weigh about 2.5 g of solid sodium thiosulfate (NaS_2O_3•5H2O) and dissolve in 100 mL of distilled water in a volumetric flask. (This gives a 0.1 mol L^{-1} solution). Then use a pipette to transfer 10 mL of this solution to a 500 mL volumetric flask and dilute by adding distilled water up to the mark; you will use this diluted thiosulfate solution in your titrations. The concentration of the diluted thiosulfate solution may be calculated as follows:

$$[S_2O_3^{2-}]\ (\text{mol L}^{-1}) = \text{mass of } NaS_2O_3\text{•}5H_2O \text{ used (g)} \div 248.2\ (\text{g mol}^{-1}) \div 0.1\ (\text{L}) \div 50$$

   Alternatively, the concentration of thiosulfate may be determined more accurately by titration with a standard solution of iodate or potassium permanganate (if either is available).

2. **Preparation of 0.5% starch indicator solution:** Weigh 0.25 g of soluble starch into a 100 mL conical flask or beaker and add 50 mL of distilled water. Heat solution with stirring at 79 ° C for 5 minutes, being careful not to exceed the stated temperature. Allow solution to cool to room temperature.
3. Accurately weigh about 50 g of iodised salt into a 250mL volumetric flask and add distilled water up to the mark. Shake to dissolve salt.

4. Use a pipette to transfer a 50 mL aliquot of salt solution into a 250 mL conical flask. Then add 5 mL of 1 mol L^{-1} hydrochloric acid and 5 mL of 0.6 mol L^{-1} potassium iodide solution. The solution will turn a yellow/brown colour as iodine is produced, as shown in Figure 1.
5. Titrate the solution with your 0.002 mol L^{-1} sodium thiosulfate solution until the yellow/brown colour of iodine becomes very pale (see Figure 1). Then add 1 mL of starch indicator solution, which will produce a dark blue-black coloured complex with iodine – as shown in Figure 2 – and continue your titration until this colour completely disappears (see Figure 3).
6. Repeat the titration with further aliquots of your salt solution until concordant results (titres agreeing within 0.1 mL) are obtained.

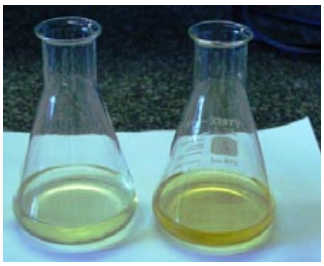

Figure 1. Right flask: yellow/brown colour of iodine formed from reaction of iodate from salt with acidic iodide solution. Left flask: pale yellow colour left when nearly all iodine has reacted with added thiosulfate during titration.

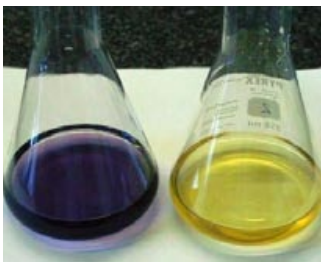

Figure 2. Right flask: yellow/brown solution containing last trace of iodine. Left flask: dark blue-black colour formed when starch indicator is added to solution containing iodine near endpoint.

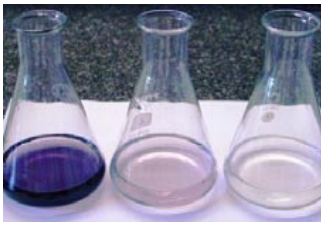

Figure 3. A series of flasks showing the colour change as the last remaining iodine (with added starch indicator) is titrated with thiosulfate. The dark blue-black colour disappears, leaving a colourless solution at the endpoint.

Calculations

1. From the redox equations above, determine the number of moles of thiosulfate required for reaction with each mole of iodate in the original salt solution.
2. Calculate the average volume of thiosulfate solution used from your concordant titres.
3. Calculate the amount, in moles, of thiosulfate reacting.
4. Calculate the amount, in moles, of iodate in the salt solution.
5. Calculate the concentration, in mol L^{-1}, of iodate in the salt solution.
6. Calculate the number of grams of iodate in the salt solution.
7. Based on the weight of the iodised salt you used to make your salt solution, calculate the iodate content of your salt, in mg of iodate (IO_3^-) per kg of salt. NB: the molecular weight of IO_3^- is 174.9 g mol^{-1}.
8. In order to see if your salt satisfies the Australia New Zealand Food Standards Code, calculate the iodine content (in mg of iodine (I) per kg of salt) from your result above as follows:

   iodine (I) content = iodate (IO_3^-) content x 126.9/174.9

Additional Notes

1. According to the specified limits for iodate in iodised salt, the volume of 0.002 mol L^{-1} sodium thiosulfate required in the above titration should lie between 5.9 mL and 15.4 mL. Therefore, a "rougher" method for quickly determining whether or not your salt sample conforms to these limits is to prepare the sample solution as above (adding hydrochloric acid and potassium iodide as described), but instead of titrating the solution simply add 1 mL of starch indicator, followed by 5.9 mL of thiosulfate solution, the blue-black colour of starch-iodine should persist. But when a further 9.5 mL of thiosulfate solution is added the colour should disappear.

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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