Redox Titration

# Purpose

To determine the concentration of a sodium thiosulphate ($Na\_{2}S\_{2}O\_{3}$) by a redox titration with the $I\_{2} $generated in a reaction with $KIO\_{3}$using the starch-iodine complex as the indicator.

# Introduction

In a reaction with the thiosulphate ion ($S\_{2}O\_{3}^{2-}$), iodine ($I\_{2}$) is reduced to iodide ($I^{-}$) and the thiosulphate is oxidized to the tetrathionate ion ($S\_{4}O\_{6}^{2-}$). Iodine is only slightly soluble in water, but in the presence of excess iodide ion, it forms the soluble tri-iodide ion ($I\_{3}^{-}$) that is used in redox titrations: $I\_{2} + I^{-} \rightarrow I\_{3}^{-}$. The actual reaction that occurs in the redox titration is then between the tri-iodide ion and the thiosulphate ion.

In this experiment, the thiosulphate is titrated against a known volume of a standard iodate in the presence of excess iodide. The endpoint is signaled by the disappearance of a blue color, due to a starch indicator, when enough thiosulfate has been added to consume the iodine.

# Pre-Lab Questions

1. Write balanced net ionic equations for the reaction of:
2. Iodate ion ($IO\_{3}^{-}$) with iodide in an acid solution to form $I\_{3}^{-}$

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| --- | --- |
| **Step** | **Process** |
| Unbalanced equation 🡪 | $$IO\_{3}^{-} + I^{-} \rightarrow I\_{3}^{-}$$ |
| Balance atoms other than $O$ and $H$ 🡪 | $$IO\_{3}^{-} + 8 I^{-} \rightarrow 3 I\_{3}^{-}$$ |
| Balance $O$ by adding $H\_{2}O$ 🡪 | $$IO\_{3}^{-} + 8 I^{-} \rightarrow 3 I\_{3}^{-}+3 H\_{2}O$$ |
| Balance $H$ by adding $H^{+}$ions 🡪 | $$IO\_{3}^{-} + 8 I^{-} + 6 H^{+} \rightarrow 3 I\_{3}^{-}+3 H\_{2}O$$ |
| Final Equation 🡪 | $$IO\_{3}^{-} + 8 I^{-} + 6 H^{+} \rightarrow 3 I\_{3}^{-}+3 H\_{2}O$$ |

1. $I\_{3}^{-}$ and the thiosulfate ion to form the iodide ion and tetrathionate ion

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| --- | --- |
| **Step** | **Process** |
| Unbalanced Equation 🡪 | $$S\_{2}O\_{3}^{2-} + I\_{3 }^{-}\rightarrow I^{-} + S\_{4}O\_{6}^{2-}$$ |
| Balance atoms other than $O$ and $H$ 🡪 | $$2 S\_{2}O\_{3}^{2-} + I\_{3}^{-} \rightarrow 3 I^{-} + S\_{4}O\_{6}^{2-}$$ |
| Balance $O$ by adding $H\_{2}O$ 🡪 |  |
| Balance $H$ by adding $H^{+}$ions 🡪 |  |
| Final Equation 🡪 | $$2 S\_{2}O\_{3}^{2-} + I\_{3}^{-} \rightarrow 3 I^{-} + S\_{4}O\_{6}^{2-}$$ |

1. Calculate the concentration of an iodate solution that contains 1.9853 g of $KIO\_{3}$ in a 1000 mL volumetric flask.

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| --- | --- |
| **Steps** | **Process** |
| Find the number of moles of $KIO\_{3}$ are in 1.9853 g. | $$n\_{KIO\_{3}}=\frac{1.9853 g}{214 g/mol}$$$$n\_{KIO\_{3}}≅0.00928 mol$$ |
| Calculate a molar ratio of iodate ions in $KIO\_{3}$. |  $ K^{+}$ $IO\_{3}^{-}$$$\frac{1}{0.00928 mol} = \frac{1}{x}$$$$0.00928 mol = x$$ |
| Use the formula $Concentration=\frac{Mol}{Volume (L)}$ to calculate the concentration. | $$C=\frac{n}{V}$$$$C = \frac{0.00928 mol}{1 L}$$$$C = 9.28 × 10^{-3} mol/L$$ |

1. Calculate the molar concentration of a thiosulfate solution given that 25.00 mL of 0.0195 mol/L $KIO\_{3}$ solution in a flask containing 2.00 g of KI and 10 mL of 0.500 mol/L $H\_{2}SO\_{4} $required 34.81 mL of thiosulfate solution to reach the starch endpoint.

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| **Reaction Between** $IO\_{3}^{-}$ **and** $ I^{-}$ |  |
| $$IO\_{3}^{-} + 8 I^{-} + 6 H^{+} \rightarrow 3 I\_{3}^{-}+3 H\_{2}O$$Mol of $IO\_{3}^{-}$ reacted = 0.00049 molNumber of mol of $I\_{3}^{-}$ released = $3 ×0.00049 mol=0.00147 mol$ | $$C\_{KIO\_{3}}=\frac{n\_{KIO\_{3}}}{V\_{KIO\_{3}}}$$$$0.0195 M=\frac{n\_{KIO\_{3}}}{0.025L}$$$$0.00049 mol=n\_{KIO\_{3}}$$ |
| **Reaction Between** $ I^{-}$ **and** $S\_{2}O\_{3}^{2-}$ |  |
| $$2 S\_{2}O\_{3}^{2-} + I\_{3}^{-} \rightarrow 3 I^{-} + S\_{4}O\_{6}^{2-}$$1 mol of $I\_{3}^{-}$ reacts with 2 mol of $S\_{2}O\_{3}^{2-}$ 🡪 $2 ×0.00147 mol=0.00294 mol$Concentration of $S\_{2}O\_{3}^{2-}$ solution 🡪$0.08 mol/L$ | $$C = \frac{n}{V}$$$$C = \frac{0.00294 mol}{0.0348 L}$$$$C ≅ 0.08 mol/L$$ |

# Definitions

|  |  |
| --- | --- |
| Titration | The precise addition of a solution in a burette into a measured volume of a sample solution |
| Titrant | The solution in a burette during a titration  |
| End Point | The point in a titration at which a sharp change in a measurable and characteristic property occurs (usually a color change) |
| Equivalence Point | The measured quantity of a titrant recorded at the point at which chemically equivalent amounts have reacted |
| Burette | A graduated tube of glassware that has a stopcock at its bottom endIt is used to dispense precise volumes of liquid reagents |

# Materials

|  |  |  |  |
| --- | --- | --- | --- |
| KIO3 (aq)\_\_\_\_\_\_ M | Erlenmeyer flasks | Na2S2O3 solution | starch solution |
| Beakers | graduated cylinders | 0.5 M H2SO4 solution |
| wash bottle | burets and clamps | solid KI |
| distilled water | balance | retort stand |

# Procedure

1. Assemble the equipment.
2. Pipette 25.0 mL of the standard $KIO\_{3}$ solution into a flask. Add 2.000 g of solid KI and 10 mL of 0.500 mol/L $H\_{2}SO\_{4} $ to the flask
3. Properly fill a burette with the thiosulfate solution.
4. Titrate with the thiosulfate until the solution has lost its reddish-brown color and has become orange.
5. Add 2 mL of starch indicator and complete the titration.
6. Note the initial and final burette readings to at least one decimal place.
7. Repeat the titrations at least twice more until the concentration of the thiosulfate agrees to within 10%.

# Observations

Concentration of $KIO\_{3}$ \_4.305g/2L 🡪0.01 mol/L\_

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| --- | --- | --- | --- | --- |
| $$Na\_{2}S\_{2}O\_{3}$$ | Rough | Trial #1 | Trial #2 | Trial #3 |
| Final Burette Reading (mL) | 19.0 | 21.0 | 40.8 | 46.6 |
| Initial Burette Reading (mL) | 0.0 | 0.0 | 21.0 | 25.0 |
| Volume Used (mL) | 19.0 | 21.0 | 19.8 | 21.6 |

# Calculations

1. What is the concentration of $KIO\_{3}$?

The concentration of $KIO\_{3}$was 0.01M

1. Write the balanced equation for the reaction between iodate and iodide ions (see pre-lab)

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| --- | --- |
| **Step** | **Process** |
| Unbalanced equation 🡪 | $$IO\_{3}^{-} + I^{-} \rightarrow I\_{3}^{-}$$ |
| Balance atoms other than $O$ and $H$ 🡪 | $$IO\_{3}^{-} + 8 I^{-} \rightarrow 3 I\_{3}^{-}$$ |
| Balance $O$ by adding $H\_{2}O$ 🡪 | $$IO\_{3}^{-} + 8 I^{-} \rightarrow 3 I\_{3}^{-}+3 H\_{2}O$$ |
| Balance $H$ by adding $H^{+}$ions 🡪 | $$IO\_{3}^{-} + 8 I^{-} + 6 H^{+} \rightarrow 3 I\_{3}^{-}+3 H\_{2}O$$ |
| Final Equation 🡪 | $$IO\_{3}^{-} + 8 I^{-} + 6 H^{+} \rightarrow 3 I\_{3}^{-}+3 H\_{2}O$$ |

1. Calculate the moles of iodate used in each titration and the moles of $I\_{3}^{-}$ produced in each reaction with the iodate ion.

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| --- | --- |
| $$C\_{IO\_{3}}=\frac{n\_{IO\_{3}}}{V\_{KIO\_{3}}}$$$$0.01 M=\frac{n\_{IO\_{3}}}{0.025 L}$$$$0.00025 mol= n\_{IO\_{3}}$$ | $$IO\_{3}^{-} + 8 I^{-} + 6 H^{+} \rightarrow 3 I\_{3}^{-}+3 H\_{2}O$$For every 1 $IO\_{3}^{-}$ ion, 3 $I\_{3}^{-} $are produced.Therefore,$$3×0.00025 mol of IO\_{3}^{-}=0.00075mol of I\_{3}^{-} produced $$ |

1. Write the equation for the reaction of the tri-iodide ion and the thiosulfate ion (see pre-lab)

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| --- | --- |
| **Step** | **Process** |
| Unbalanced Equation 🡪 | $$S\_{2}O\_{3}^{2-} + I\_{3 }^{-}\rightarrow I^{-} + S\_{4}O\_{6}^{2-}$$ |
| Balance atoms other than $O$ and $H$ 🡪 | $$2 S\_{2}O\_{3}^{2-} + I\_{3}^{-} \rightarrow 3 I^{-} + S\_{4}O\_{6}^{2-}$$ |
| Balance $O$ by adding $H\_{2}O$ 🡪 |  |
| Balance $H$ by adding $H^{+}$ions 🡪 |  |
| Final Equation 🡪 | $$2 S\_{2}O\_{3}^{2-} + I\_{3}^{-} \rightarrow 3 I^{-} + S\_{4}O\_{6}^{2-}$$ |

1. Calculate the moles of thiosulfate in each titration and the concentration of the thiosulfate solution.

$$2 S\_{2}O\_{3}^{2-} + I\_{3}^{-} \rightarrow 3 I^{-} + S\_{4}O\_{6}^{2-}$$

1 mol of $I\_{3}^{-}$ reacts with 2 mol of $S\_{2}O\_{3}^{2-}$ 🡪 0.00075 mol of $I\_{3}^{-} × 2 = 0.0015 mol of S\_{2}O\_{3}^{2-} $

|  |  |  |  |
| --- | --- | --- | --- |
| Trial # | Moles of $S\_{2}O\_{3}^{2-}$ (mol) | Volume of $S\_{2}O\_{3}^{2-}$ used (L) | Concentration of $S\_{2}O\_{3}^{2-}$ (mol/L) |
| Rough | 0.0015 | 0.0190 | 0.093 |
| 1 | 0.0015 | 0.0210 | 0.084 |
| 2 | 0.0015 | 0.0198 | 0.089 |
| 3 | 0.0015 | 0.0216 | 0.082 |

# Conclusion

The purpose of the lab was to standardize a thiosulphate solution by redox titration with a standard solution of iodate ions. Throughout this lab, the molarity of the $S\_{2}O\_{3}^{2-}$ and $IO\_{3}^{-}$ ions was calculated using, but not limited by, different volumes and half-reactions. Using the given balanced equations, it was fairly simple to standardize a thiosulphate solution and then to use that solution to determine both the degree and the magnitude (in number of moles) of the presence of iodate ions within the titrated solution. On average, the equivalence point was reached at after the addition of about 21mL of sodium thiosulphate, when the titrated solution changed from possessing a color of dark-blue/black to a clear solution, indicating the arrival of the end point. In this experiment, it was observed that there is always a relationship between the reactants of a chemical reaction. Due to these relationships, calculating the different characteristics of compounds is made much simpler, as it becomes a task of applying and using basic chemistry formulas.