REDOX TITRATION

PURPOSE

To determine the concentration of a sodium thiosulphate ($Na_2S_2O_3$) by a redox titration with the I_2 generated in a reaction with KIO_3using the starch-iodine complex as the indicator.

INTRODUCTION

In a reaction with the thiosulphate ion ($S_2O_3^{2-}$), iodine (I_2) is reduced to iodide (I^-) and the thiosulphate is oxidized to the tetrathionate ion ($S_4O_6^{2-}$). 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:
   a) Iodate ion (IO_3^-) with iodide in an acid solution to form I_3^-

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_2O →	$IO_3^- + 8\,I^- \rightarrow 3\,I_3^- + 3\,H_2O$
Balance H by adding H^+ions →	$IO_3^- + 8\,I^- + 6\,H^+ \rightarrow 3\,I_3^- + 3\,H_2O$
Final Equation →	$IO_3^- + 8\,I^- + 6\,H^+ \rightarrow 3\,I_3^- + 3\,H_2O$

   b) I_3^- and the thiosulfate ion to form the iodide ion and tetrathionate ion

Step	**Process**
Unbalanced Equation →	$S_2O_3^{2-} + I_3^- \rightarrow I^- + S_4O_6^{2-}$
Balance atoms other than O and H →	$2\,S_2O_3^{2-} + I_3^- \rightarrow 3\,I^- + S_4O_6^{2-}$
Balance O by adding H_2O →	↓
Balance H by adding H^+ions →	
Final Equation →	$2\,S_2O_3^{2-} + I_3^- \rightarrow 3\,I^- + S_4O_6^{2-}$

2. Calculate the concentration of an iodate solution that contains 1.9853 g of KIO_3 in a 1000 mL volumetric flask.

Steps	**Process**
Find the number of moles of KIO_3 are in 1.9853 g.	$n_{KIO_3} = \frac{1.9853\ g}{214\ g/mol}$ $n_{KIO_3} \cong 0.00928\ mol$
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.	$C = \frac{n}{V}$ $C = \frac{0.00928\ mol}{1\ L}$ $C = 9.28 \times 10^{-3}\ mol/L$

3. 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_2SO_4 required 34.81 mL of thiosulfate solution to reach the starch endpoint.

Reaction Between IO_3^- and I^-

$IO_3^- + 8\ I^- + 6\ H^+ \rightarrow 3\ I_3^- + 3\ H_2O$
Mol of IO_3^- reacted = 0.00049 mol
Number of mol of I_3^- released = 3 $\times 0.00049\ mol = 0.00147\ mol$

$$C_{KIO_3} = \frac{n_{KIO_3}}{V_{KIO_3}}$$

$$0.0195\ M = \frac{n_{KIO_3}}{0.025L}$$

$$0.00049\ mol = n_{KIO_3}$$

Reaction Between I^- and $S_2O_3^{2-}$

$2\ S_2O_3^{2-} + I_3^- \rightarrow 3\ I^- + S_4O_6^{2-}$
1 mol of I_3^- reacts with 2 mol of $S_2O_3^{2-}$ → $2 \times 0.00147\ mol = 0.00294\ mol$
Concentration of $S_2O_3^{2-}$ solution → $0.08\ mol/L$

$$C = \frac{n}{V}$$

$$C = \frac{0.00294\ mol}{0.0348\ L}$$

$$C \cong 0.08\ mol/L$$

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 end It is used to dispense precise volumes of liquid reagents

MATERIALS

KIO_3 (aq)______ M	Erlenmeyer flasks	$Na_2S_2O_3$ solution	starch solution
Beakers	graduated cylinders	0.5 M H_2SO_4 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_2SO_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

$Na_2S_2O_3$	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

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

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_2O →	$IO_3^- + 8\,I^- \rightarrow 3\,I_3^- + 3\,H_2O$
Balance H by adding H^+ions →	$IO_3^- + 8\,I^- + 6\,H^+ \rightarrow 3\,I_3^- + 3\,H_2O$
Final Equation →	$IO_3^- + 8\,I^- + 6\,H^+ \rightarrow 3\,I_3^- + 3\,H_2O$

3. Calculate the moles of iodate used in each titration and the moles of I_3^- produced in each reaction with the iodate ion.

$C_{IO_3} = \frac{n_{IO_3}}{V_{KIO_3}}$ $0.01\,M = \frac{n_{IO_3}}{0.025\,L}$ $0.00025\,mol = n_{IO_3}$	$IO_3^- + 8\,I^- + 6\,H^+ \rightarrow 3\,I_3^- + 3\,H_2O$ 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

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

Step	**Process**
Unbalanced Equation →	$S_2O_3^{2-} + I_3^- \rightarrow I^- + S_4O_6^{2-}$
Balance atoms other than O and H →	$2\,S_2O_3^{2-} + I_3^- \rightarrow 3\,I^- + S_4O_6^{2-}$
Balance O by adding H_2O →	↓
Balance H by adding H^+ions →	
Final Equation →	$2\,S_2O_3^{2-} + I_3^- \rightarrow 3\,I^- + S_4O_6^{2-}$

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

$2\ S_2O_3^{2-} + I_3^- \rightarrow 3\ I^- + S_4O_6^{2-}$

1 mol of I_3^- reacts with 2 mol of $S_2O_3^{2-}$ → 0.00075 mol of $I_3^- \times 2 = \mathbf{0.0015\ mol\ of}\ S_2O_3^{2-}$

Trial #	Moles of $S_2O_3^{2-}$ (mol)	Volume of $S_2O_3^{2-}$ used (L)	Concentration of $S_2O_3^{2-}$ (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