Analysis of Hypochlorite in Bleach (Redox titrations are cool!)

This experiment allows you to determine the percentage (w/v) of active ingredient, **sodium hypochlorite** (NaOCl) in common commercial bleaches. We won't do it directly, but we will use some cool chemistry and we will be able (or should be able) to determine percentages pretty accurately.

THE CHEMISTRY: (for more a more detailed description see your lab manual)

$$OCl_{(aq)} + 2 H_{(aq)}^{+} + 2 I_{(aq)}^{-} \rightarrow I_{2(aq)}^{-} + Cl_{(aq)}^{-} + H_2O_{(l)}$$
^[1]

$$2 S_2 O_3^{2-} + I_2 \rightarrow 2 I^- + S_4 O_6^{2-}$$
^[2]

$$I_{2(aq)} + I_{(aq)} \rightarrow I_{3}_{(aq)}$$
 (occurs rapidly and completely) [3]

So when I⁻ is in excess:

$$2 S_2 O_3^{2-}_{(aq)} + I_3^{-}_{(aq)} \rightarrow 3 I_{(aq)}^{-} + S_4 O_6^{2-}_{(aq)}$$
^[4]

$$I_3^{-}_{(aq)}$$
 + starch \rightarrow deep-blue complex (also occurs rapidly) [5]

Our problem: The analysis of OCl⁻ by titration with $S_2O_3^{2-}$ requires that we know the concentration of the $S_2O_3^{2-}$ solution. Unfortunately, **sodium thiosulfate** (Na₂S₂O₃) is not a primary standard, so we must standardize the sodium thiosulfate solution. A similar series of reactions is used for the standardization, only **potassium iodate** (KIO₃) is used to oxidize **iodide** (**I**⁻) to **iodine** (**I**₂):

$$IO_{3^{-}(aq)} + 5I_{(aq)} + 6H_{(aq)} \rightarrow 3I_{2^{-}(aq)} + 3H_{2}O_{(l)}$$
 [6]

So we'll titrate a known amount of KIO_3 (the limiting reagent), in the presence of excess of I⁻, with our $Na_2S_2O_3$ solution. Using the stoichiometry of reaction 6, we can calculate the I_2 generated (theoretically) and then using the stoichiometry of reaction 2 (this gives us moles $Na_2S_2O_3$ used in each titration) and the titration volumes we can calculate $[Na_2S_2O_3]$.

THE EXPERIMENT:

1) Standardize $S_2O_3^{2-}_{(aq)} \Rightarrow$ Solves problem.

The stoichiometry is important:

1 mole $IO_3^{-} = 3$ mole I_2 [6] 1 mole $I_2 = 2$ mole $S_2O_3^{2-}$ [2]

So after titrations are completed (volumes), you can find $[S_2O_3^{2-}]$. You need at least 3 titration volumes within 0.05 mL.

2) Determine % NaOCl in bleach (w/v)

You need at least 3 titrations within 0.05 mL.

 $[S_2O_3^{2-}]$ x titration vol. of $S_2O_3^{2-}$ = mole $S_2O_3^{2-}$

1 mole $S_2O_3^{2-} = 1/2$ mole I_2 [2] 1 mole $I_2 = 1$ mole OCl⁻ (= mole NaOCl) [1]

Convert to mass NaOCl (g) (MW NaOCl = 74.44 g/mole) Find (g/sample vol.) x 100%

sample vol. = $0.10 \text{ mL} \Rightarrow 5.00 \text{ mL}$ of original bleach solution/250.0 mL, so, stock bleach solution is 0.02 (or 1/50) of original bleach solution. For each titration $\Rightarrow 5.00 \text{ mL} \times 0.02$ (or 5.00 mL/50) = 0.10 mL

SAMPLE CALCULATIONS:

1) Standardization of $Na_2S_2O_3$:

 $(0.2000 \text{ g KIO}_3)(1 \text{ mole KIO}_3/214.00 \text{ g/mole}) = 9.346 \text{ x } 10^4 \text{ mole KIO}_3$

You made up KIO₃ stock solution in a 100.0 mL volumetric flask and for each titration you used 5.00 mL of your stock solution

So.... for each titration you used 1/20 of your stock solution:

 KIO_3 used in each titration = 9.346 x 10⁻⁴ mole $KIO_3/20$

 KIO_3 used in each titration = 4.67 x 10⁻⁵ mole KIO_3

1 mole KIO₃ = 3 mole $I_2 \Rightarrow 1.40 \times 10^{-4} \text{ mole } I_2$ 1 mole $I_2 = 2 \text{ mole } \text{Na}_2\text{S}_2\text{O}_3 \Rightarrow 2.80 \times 10^{-4} \text{ mole } \text{Na}_2\text{S}_2\text{O}_3$ e.g. For a titration volume = 5.61 mL (from experimental data):

 2.80×10^{-4} mole Na₂S₂O₃/ 0.00561 L = 0.0500 M Na₂S₂O₃

2) Analysis of Bleach (Clorox)

e.g. For a titration volume = 3.44 mL (from experimental data)

 $(0.00344 \text{ L Na}_2\text{S}_2\text{O}_3)(.0500 \text{ mole/L Na}_2\text{S}_2\text{O}_3) = 1.72 \text{ x } 10^{-4} \text{ mole Na}_2\text{S}_2\text{O}_3$

1 mole Na₂S₂O₃ = 1/2 mole I₂ \Rightarrow 8.68 x 10⁻⁵ mole I₂ 1 mole I₂ = 1 mole mole NaOCl \Rightarrow 8.68 x 10⁻⁵ mole NaOCl

mass NaOCl = $(8.68 \times 10^{-5} \text{ mole NaOCl})(74.44 \text{ g/mole}) = 0.00640 \text{ g NaOCl}$ % NaOCl (w/v) = $(0.00640 \text{ g NaOCl}/0.10 \text{ mL bleach}) \times 100\% = 6.40 \%$ NaOCl

RMD (ppt) = (mean deviation/mean) x 1000