

Research Question: Which is the most accurate method (redox titration with sodium thiosulfate solution, and reaction with hydrogen peroxide) of determining the concentration of sodium hypochlorite in household bleach?

International Baccalaureate Chemistry Internal Assessment

Word Count - 2999

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Design

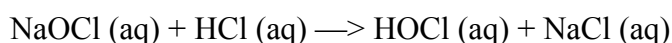
Research Focus

In my investigation, I looked at different methods that can be used to determine concentration of sodium hypochlorite in household bleach, and how this varies the calculated concentration.

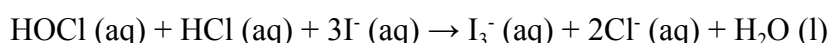
Background Information

Sodium hypochlorite has the formula NaOCl, and is most commonly used as a bleaching agent and so is in household bleach. According to the website of the Domestos Thick Bleach¹ that I used, the concentration of sodium hypochlorite in the bottle was 5.0% by mass.

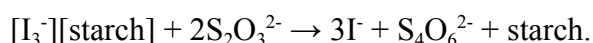
The chemistry involved in the redox titration consists of the hydrochloric acid reacting with sodium hypochlorite to form hypochlorous acid:



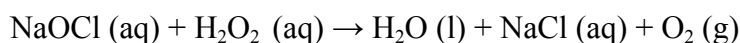
And then, the hypochlorous acid reacts with iodide ions when the solution is acidic:



The triiodide combines with the starch and forms a dark blue triiodide complex. Finally, the starch-triiodide is titrated by sodium thiosulfate to form a white solution of iodide, dithionite and starch.



The chemistry involved in the gas collection reaction goes like this. It involves a reaction between NaOCl and H₂O₂. The hydrogen peroxide behaves as a reducing agent with the chlorate ions as the oxidising agent.



¹ Domestos Original Thick Bleach, Sainsbury's (Accessed 25 June 2024)

Development of methods

I performed some preliminary research to find a redox titration method² for bleach and sodium thiosulfate solution on the web. As it suggested, I used 0.26mol dm^{-3} sodium thiosulfate solution in my burette, and personally decided to use a 1% concentration of starch solution. I attempted a few trial redox titrations, none of which showed any colour change as I was expecting to observe, so I altered the method so that I added all the sodium thiosulfate at one time, instead of adding some, then adding the starch solution, and then adding more. I added the starch solution, and then began the redox titration. This was because I assumed that the iodine in the potassium iodide solution must have already reacted with the small added volume of sodium thiosulfate solution and so nothing was happening when I added the starch solution. I repeated the redox titration a number of times after this methodological alteration, however this still was not showing any results, and so there must have been something else wrong with my experiment. I checked each individual variable was correct, and it was, so then I checked the bleach and found that it was out of date. I attempted with a new bleach, and the redox titration worked instantly.

My other method I also found in my research³ consisted of reacting bleach with hydrogen peroxide in order to produce oxygen gas, which I first collected in a water bath with a measuring cylinder. I realised that I needed to subtract 10cm^3 of gas from the result, as when I added 10cm^3 of hydrogen peroxide to the solution, it would inevitably displace 10cm^3 of air that was already in the conical flask. I then realised that the suitability of this method was flawed, as the oxygen gas, which is soluble in water, would dissolve on its way into the water bath and the measuring cylinder, so I would collect less gas than was actually produced. In order to circumvent this, I decided to collect the oxygen gas with a gas syringe, however this meant that I had to very quickly add the hydrogen peroxide solution and then place the bung on the conical flask, as, unlike with the measuring cylinder method, I could not add and collect simultaneously.

Materials and equipment

To measure out my volumes, and when creating solutions, I used a mixture of volumetric pipettes and measuring cylinders. This meant that there was significant variation in the volumes and concentrations, only when using measuring cylinders, as volumetric pipettes are very precise with an uncertainty of $\pm 0.060\text{cm}^3$ for the 25cm^3 pipette, and $\pm 0.050\text{cm}^3$ for the 5cm^3 pipette. The measuring cylinders had an uncertainty of $\pm 0.2\text{cm}^3$ for the 10cm^3 cylinder, $\pm 0.5\text{cm}^3$ for the 25cm^3 cylinder, and $\pm 1\text{cm}^3$ for the 100cm^3 cylinder. The burette had an uncertainty of $\pm 0.05\text{cm}^3$. The measuring cylinder and gas syringe both had an uncertainty of $\pm 1\text{cm}^3$.

² *The determination of hypochlorite in bleach, The City University of New York, 2010*

³ *Estimating the concentration of bleach, Royal Society of Chemistry*

Methods and Variables

Titration method

- Fill a burette with 0.26mol dm^{-3} sodium thiosulfate solution, after rinsing it with distilled water to remove any pre-existing solutions left in the burette.
- Allow a few millilitres of solution to run through the burette to remove any trapped air.
- Record the initial volume of sodium thiosulfate in the burette.
- Then perform a 10-fold dilution of bleach. Use a volumetric pipette to gather 25cm^3 of bleach solution into a 250cm^3 volumetric flask. Fill the rest with distilled water, and shake the solution. This makes the bleach one tenth of the original concentration.
- Use the volumetric pipette to add 25cm^3 of the diluted solution into a 250ml conical flask, and then add roughly 15cm^3 of distilled water.
- Add approximately 20cm^3 of 10% potassium iodide solution to the conical flask.
- Add roughly 20cm^3 of hydrochloric acid solution to the conical flask, and then 2cm^3 of 1% starch solution, which should turn the solution a blue/black colour.
- Begin the redox titration by adding the sodium thiosulfate solution dropwise until you notice a colour change to white.
- Record the final volume of sodium thiosulfate in the burette and calculate the difference in volume.
- Repeat eight times, with a total of three different bleach solutions created in the volumetric flask.

Table 1 - Variable Table for the above method

Variable	Variable Type	Values (Control variables)
Volume of sodium thiosulfate used (cm^3)	Dependent	N/A
Volume and concentration of bleach (cm^3 , mass%)	Control	25cm^3 , 0.5%
Volume and concentration of potassium iodide solution (cm^3 , mass%)	Control	20cm^3 , 10%
Volume and concentration of hydrochloric acid solution (cm^3 , mol dm^{-3})	Control	20cm^3 , 1mol dm^{-3}
Volume and concentration of starch solution (cm^3 , mass%)	Control	2cm^3 , 1%

Gas Syringe Method

After adjustments were made, my second method worked in this way.

- Use a volumetric pipette to measure 5cm^3 of bleach into a conical flask, and then add 25cm^3 of distilled water to the conical flask and swirl it.
- Quickly, add 10cm^3 of 20% hydrogen peroxide to the conical flask, then attach the bung, which is also attached to a gas syringe, to the conical flask.
- Swirl and collect the gas produced in the gas syringe.
- Record the volume of gas displaced, and subtract 10cm^3 from the volume to compensate for the gas displaced when the hydrogen peroxide was added.
- Repeat seven times.

Table 2 - Variable Table for the above method

Variable	Variable Type	Values (Control variables)
Volume of oxygen gas displaced (cm^3)	Dependent	N/A
Volume and concentration of bleach (cm^3 , mass%)	Control	30cm^3 (diluted), 16.7%
Volume and concentration of hydrogen peroxide solution (cm^3 , mass%)	Control	10cm^3 , 20%

Measuring Cylinder Method

This method is very similar to the method above, I simply used a different means of collecting the oxygen gas.

- Use a volumetric pipette to measure 5cm^3 of bleach into a conical flask, and then add 25cm^3 of distilled water to the conical flask and swirl it.
- Fill a trough with water and submerge a 100cm^3 measuring cylinder with water and invert it under water in a clamp.
- Attach a delivery tube from the conical flask to the submerged measuring cylinder.
- Measure 10cm^3 of 20% hydrogen peroxide solution into a syringe that is directly attached to the bung that is on the conical flask.
- Add the hydrogen peroxide solution and swirl the contents around and collect the gas produced in the measuring cylinder.
- Record the volume of gas displaced, and subtract 10cm^3 from the volume to compensate for the gas displaced when the hydrogen peroxide was added.
- Repeat seven times.

Table 3 - Variable Table for the above method

Variable	Variable Type	Values (Control variables)
Volume of oxygen gas displaced (cm ³)	Dependent	N/A
Volume and concentration of bleach (cm ³ , mass%)	Control	30cm ³ (diluted), 16.7%
Volume and concentration of hydrogen peroxide solution (cm ³ , mass%)	Control	10cm ³ , 20%

Safety and environmental considerations

My safety and environmental considerations consisted of being careful around harmful substances like bleach, hydrogen peroxide, sodium thiosulfate and potassium iodide. When washing away equipment, I made sure to wash it away with water running simultaneously, so the substances would be more diluted and not damage the drains as significantly. I also made sure that I was always wearing goggles and a lab coat, to protect my eyes, my skin, and my clothes.

Results and Analysis**Observations**

In the redox titration I observed a colour change from blue/black to white, and in the reaction methods, I observed small amounts of fizzing and bubbling when the hydrogen peroxide solution was added.

Raw data, sample calculations, processed data & uncertainties

In order to adequately assess these methods, I need to convert the literature mass% concentration of sodium hypochlorite to mol dm⁻³, as these are the standard units for concentration in Chemistry.

To do this, I take the mass% value of 5% of sodium hypochlorite (NaOCl) in 1 dm³ of bleach, and calculate the mass using the density of the bleach⁴.

$$\text{density} = \text{mass} \div \text{volume}$$

$$1082 \text{ g dm}^{-3} = \text{mass} \div 1 \text{ dm}^3$$

$$\text{mass} = 1082 \text{ g}$$

Now I find 5% by mass.

$$0.05 \times 1082 = 54.1 \text{ g}$$

With the mass, I can find the amount.

$$\text{amount} = \text{mass} \div \text{molar mass}$$

$$54.1 \div (22.99 + 16 + 35.45) = 0.7268 \text{ mol (4s.f.)}$$

⁴ Unilever (2010)

With the mass and hence the amount, I can then find the concentration in mol dm^{-3} .

$$\text{amount} = \text{concentration} \times \text{volume}$$

$$0.7268 \div 1 = 0.7268 \text{ mol dm}^{-3}$$

This is the value for concentration that I will compare my results with.

Table 4 - Raw Data from Redox Titration Method

Titration	0.26M sodium thiosulfate solution (cm^3) initial	0.26M sodium thiosulfate solution (cm^3) final	Difference (cm^3)
1	4.95	7.00	2.05
2	7.00	9.15	2.15
3	9.15	11.20	2.05
4	11.20	13.55	2.35
5	13.70	15.50	1.80
6	15.50	17.20	1.70
7	17.20	19.20	2.00
8	19.25	21.40	2.15

In order to work out the concentration of sodium hypochlorite from the redox titration method, I had to use a different series of calculations.

Let's use Titration 1 as an example.

$$\text{amount} = \text{concentration} \times \text{volume}$$

$$0.26 \text{ mol dm}^{-3} \times (2.05 \div 1000) \text{ dm}^3 = 0.000533 \text{ mol of sodium thiosulfate.}$$

For every two moles of sodium thiosulfate, one mole of sodium hypochlorite reacts, so the value must be halved.

$$0.000533 \text{ mol} \div 2 = 0.0002665 \text{ mol of sodium hypochlorite.}$$

This is then divided by the volume of bleach solution used to calculate the concentration of sodium hypochlorite in mol dm^{-3} .

$$0.0002665 \text{ mol} \div 0.025 \text{ dm}^3 = 0.0107 \text{ mol dm}^{-3} \text{ (3s.f.)}$$

I repeated this for all values.

I now use the total uncertainty, which is calculated from the sum of the resolutions of the different apparatus I used, ($\pm 0.97 \text{ cm}^3$) and convert it to a percentage of the total volume of the solutions in the conical flask ($25 + 15 + 20 + 20 + 2 + \text{volume of sodium thiosulfate used}$) cm^3 . Then multiply this value by the calculated concentration for each given test to work out the numeric uncertainty. This will be used for the error bar intervals on my bar graphs.

Let's continue with Titration 1 as an example!

$$0.97 \div (25 + 15 + 20 + 20 + 2 + 2.05) = 0.0115... \%$$

$$0.0115... \% \times 0.0107 = \pm 0.000123 \text{ mol dm}^{-3}$$

I repeated this for all values.

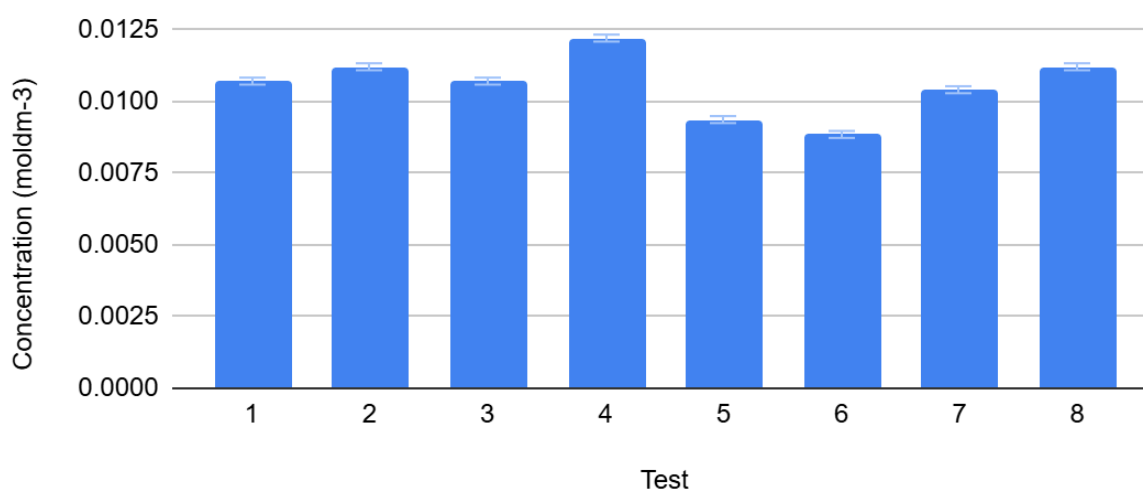
Table 5 - Calculated concentration for each test using redox titration, and uncertainty.

Titration	Difference (cm ³)	Concentration (mol dm ⁻³) (3s.f.)	Concentration Uncertainty (mol dm ⁻³) (3s.f.)
1	2.05	0.0107	±0.000123
2	2.15	0.0112	±0.000129
3	2.05	0.0107	±0.000123
4	2.35	0.0122	±0.000140
5	1.80	0.00936	±0.000108
6	1.70	0.00884	±0.000102
7	2.00	0.0104	±0.000120
8	2.15	0.0112	±0.000129

Aiming to plot the above on a column graph, I needed to plot error bars, though I couldn't use my actual value for each individual point as the software required a standardised value, so I found the average value for uncertainty (±0.000122) and used that.

Figure 1 - Column graph showing concentration of NaOCl through Redox Titration, with error bars.

Concentration NaOCl by Titration



From this set of results, I can say that my results were precise and consistent. I will now calculate a mean value by summing all values and dividing by the number of tests (8).

$$0.0107 + 0.0112 + 0.0107 + 0.0122 + 0.00936 + 0.00884 + 0.0104 + 0.0112 = 0.0846$$

$$0.0846 \div 8 = 0.010575 = 0.0106 \text{mol dm}^{-3} \text{ (3s.f.)}$$

Combined with the uncertainty, this gives the bounds:

$$0.0105 \text{mol dm}^{-3} \leq \text{mean} \leq 0.0107 \text{mol dm}^{-3}$$

Table 6 - Raw Data from Gas Syringe Method

Gas Syringe	Oxygen gas collected (cm ³) $\pm 2\text{cm}^3$
1	11.5
2	14.5
3	16.5
4	14.0
5	17.0
6	23.0
7	13.0

The calculations to calculate concentrations in both gas collection methods were the same. Let's use Gas Syringe Test 7 as an example!

I first took the volume of oxygen gas collected in cm³ and divided it by 22700 cm³ mol⁻¹ which is the molar volume of an ideal gas at Standard Temperature and Pressure.

$$13/22700 = 5.726872247 \times 10^{-4} \text{mol}$$

Because the number of moles of oxygen gas has a 1:1 ratio to the number of moles of sodium hypochlorite - as I could tell from my equation

(NaOCl (aq) + H₂O₂ (aq) → H₂O (l) + NaCl (aq) + O₂ (g)), I simply used this value for sodium hypochlorite.

This means that there was $5.726872247 \times 10^{-4} \text{mol}$ of sodium hypochlorite in 5 cm³ of bleach, so I can work out the concentration.

$$\text{concentration} = \text{amount} \div \text{volume}$$

$$(5.726872247 \times 10^{-4}) \text{mol} \div 0.005 \text{dm}^3 = 0.1145374449 \text{mol dm}^{-3}$$

$$= 0.115 \text{mol dm}^{-3} \text{ (3s.f.)}$$

I repeated this for all values.

I now use the total uncertainty ($\pm 2\text{cm}^3$), and calculate the numeric uncertainty for each test using the same method as in the Titration.

Using Test 7 as an example...

$$2\text{cm}^3 \div 40\text{cm}^3 = 0.05 = 5\%$$

$$0.05 \times 0.115 = \pm 0.00575\text{mol dm}^{-3}$$

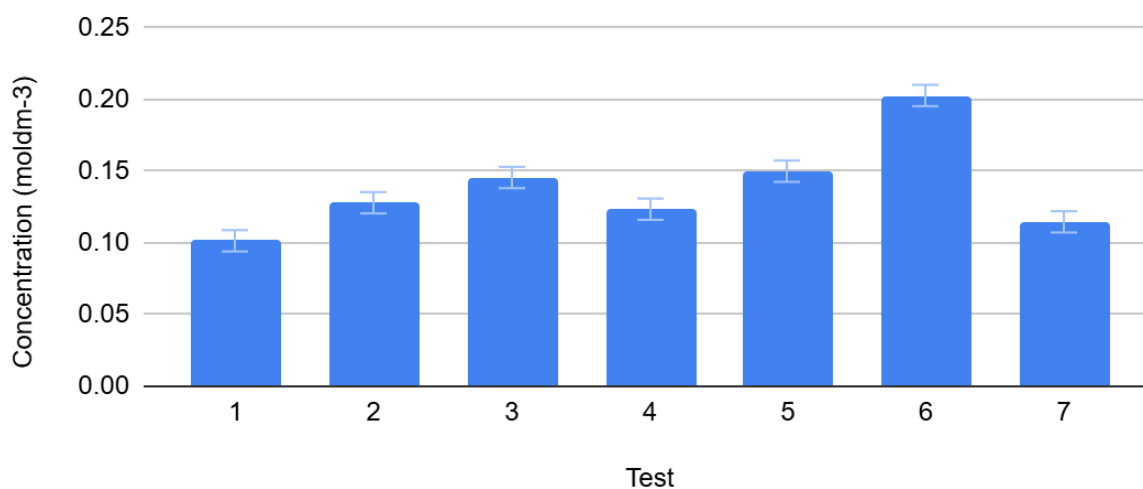
Table 7 - Calculated concentration for each test using Gas Syringe Method, and uncertainty.

Gas Syringe	Oxygen gas collected ($\pm 2\text{cm}^3$)	Concentration (mol dm^{-3}) (3s.f.)	Concentration Uncertainty (mol dm^{-3}) (3s.f.)
1	11.5	0.101	± 0.0088
2	14.5	0.128	± 0.0064
3	16.5	0.145	± 0.00725
4	14	0.123	± 0.00615
5	17	0.150	± 0.0075
6	23	0.203	± 0.01015
7	13	0.115	± 0.00575

Again, aiming to plot the error bars I needed a universal value for my uncertainty, so I calculated a mean value (± 0.00743)

Figure 2 - Column chart showing concentration of NaOCl through Gas Syringe Method, with error bars.

Concentration NaOCl by Gas Syringe



From these results, it is clear that collection by gas syringe gives quite reliably consistent values. I can deduct that Test 6 was an anomaly, as it is significantly higher than all other values, and does not overlap error bars with any other test, and so can be discounted for the mean. I work out the mean by adding all of the concentrations together and dividing by the number of tests used (6).

$$0.101 + 0.128 + 0.145 + 0.123 + 0.150 + 0.115 = 0.762$$

$$0.7621 \div 6 = 0.127\text{mol dm}^{-3} = 0.127\text{mol dm}^{-3} (3\text{s.f.}) \pm 0.00743\text{mol dm}^{-3}$$

Combined with the uncertainty, this gives the bounds:

$$0.120\text{mol dm}^{-3} \leq \text{mean} \leq 0.134\text{mol dm}^{-3}$$

Table 8 - Raw Data from Measuring Cylinder Method

Measuring Cylinder	Oxygen gas collected (cm ³) $\pm 0.2\text{cm}^3$
1	4.0
2	13.0
3	14.0
4	5.0
5	12.0
6	7.0
7	1.0

As I previously stated, the **calculation** for both gas collection methods is the same. So, here are the results for the measuring cylinders.

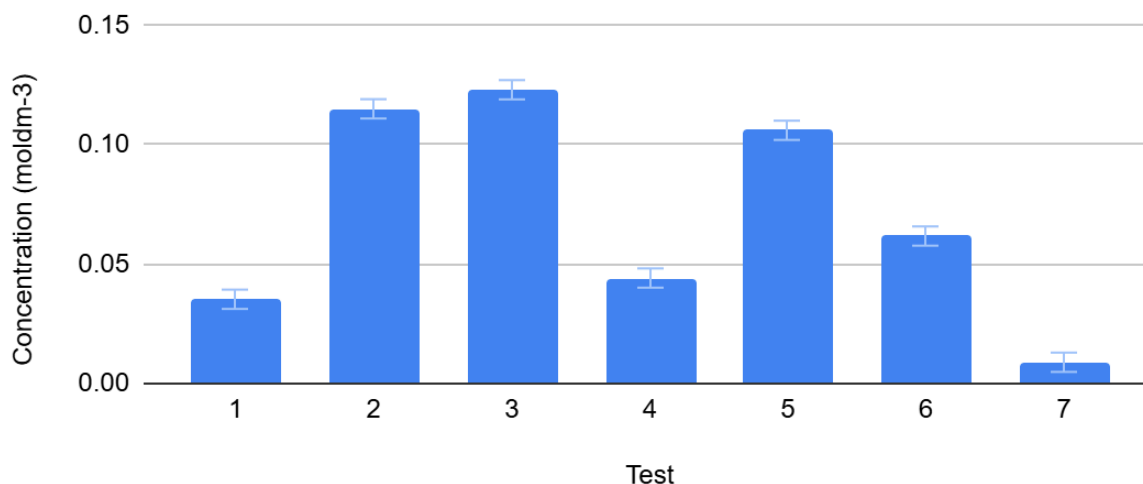
Table 9 - Calculated concentration for each test using Measuring Cylinder, and uncertainty.

Gas Volume	Oxygen gas collected ($\pm 0.5\text{cm}^3$)	Concentration (mol dm ⁻³) (3s.f.)	Concentration Uncertainty (mol dm ⁻³) (3s.f.)
1	4	0.0352	± 0.00176
2	13	0.115	± 0.00575
3	14	0.123	± 0.00615
4	5	0.0441	± 0.00221
5	12	0.106	± 0.0053
6	7	0.0617	± 0.00309
7	1	0.00881	± 0.000441

Given Test 7's tiny uncertainty value, I will discount it when calculating the average uncertainty which will be used for my error bars (± 0.00404).

Figure 3 - Concentration of NaOCl through Measuring Cylinder, with error bars.

Concentration NaOCl by Measuring Cylinder



From these results, I can tell that the measuring cylinder method was less reliable for me to obtain consistent values, as there is a significant range between them. It is difficult to deduct anomalies, as the values have a large range and so all values will be used for the mean.

$$0.0352 + 0.115 + 0.123 + 0.0441 + 0.106 + 0.0617 + 0.00881 = 0.485$$

$$0.485 \div 7 = 0.080333333 \text{ mol dm}^{-3} = 0.0693 \text{ mol dm}^{-3} \text{ (3s.f.)}$$

Combined with the uncertainty, this gives the bounds:

$$0.0653 \text{ mol dm}^{-3} \leq \text{mean} \leq 0.0733 \text{ mol dm}^{-3}$$

Conclusion

Results Table

Method	Final value
Redox Titration	$0.0105 \text{ mol dm}^{-3} \leq \text{mean} \leq 0.0107 \text{ mol dm}^{-3}$
Gas Syringe	$0.120 \text{ mol dm}^{-3} \leq \text{mean} \leq 0.134 \text{ mol dm}^{-3}$
Measuring Cylinder	$0.0653 \text{ mol dm}^{-3} \leq \text{mean} \leq 0.0733 \text{ mol dm}^{-3}$

From my investigation, I can conclude that whilst the redox titration gave me the most consistent results, these were furthest from the expected value, which I calculated from the literature value to be $0.7268 \text{ mol dm}^{-3}$. I am inclined to believe that the gas syringe collection method was most effective, as the results were fairly consistent, and closest to the literature value. I can firmly say that the worst method for determining the concentration of sodium hypochlorite was the measuring cylinder gas collection method. I can conclude with little confidence that there truly is $0.7268 \text{ mol dm}^{-3}$ sodium hypochlorite in the bleach solution, as it is not contained within the error intervals for any of my results, though I am not confident in my results either.

My most consistent set of results, the redox titration, is hindered by the fact that it is very far from the literature value, which suggests that there is likely systematic and random error in my results. There is also little value in the fact that it is impossible that the true value lies within the range of my means for all three sets of data, if the given value of 0.7268mol dm^{-3} is accurate. It is difficult to determine solely that my results are accurate, as there are many factors playing into this, specifically the uncertainty. As I am dealing with such small values, this makes it more difficult to judge whether the inaccuracy is a result of random or systematic error. There is a strong indication to suggest systematic error in all three results, due to their values being far lower than expected. All three results are also likely affected by random error in my experiments, although this is definitely less significant than the systematic error. On the whole, I think that the Redox Titration was the best method for identifying a repeatable value for the concentration, because the Titration uses precise measurements, which make it easy to repeat and obtain consistent results with small values. Yet the Gas Syringe method was most accurate, and closest (though still far off) to the literature value, as it naturally is a well controlled objective experiment.

Evaluation

There are many possible sources of error in this experiment, for each different method that I used to obtain my results.

For the gas syringe method, a possible source of error is the time it took me between adding the hydrogen peroxide solution to the conical flask, and fixing the bung on top which is connected to the gas syringe. This is a source of systematic error as the reaction will begin as soon as the hydrogen peroxide solution mixes with the bleach solution, and so will start producing oxygen gas, which would escape the conical flask, and so I must add the bung quickly to ensure the oxygen gas is trapped in the gas syringe instead of released into the atmosphere. I think that this has little significance over my results, as I was naturally quite fast, and a significant amount of the reaction only occurred after swirling the solutions together. Alternatively, to minimise systematic error, I could have used a rubber seal with an injection to add the hydrogen peroxide solution without losing oxygen gas to the surroundings. The mean value I obtained from the gas syringe method was far lower than the literary value of 0.7268mol dm^{-3} , and is not within the value's uncertainty and so this can be largely attributed to systematic errors, and random errors were likely largely insignificant.

Moving over to the measuring cylinder method, a significant source of error for this experiment was the number of moving parts, with the clamp, the water, and the delivery tube, as this gave many opportunities for error and for the gas to be dispersed into places that I didn't intend it to. In order to mitigate this systematic error, I could have had someone help me to guide the delivery tube in carefully, rather than having to do it by myself. Another source of error in this experiment, is the fact that I was collecting oxygen gas in water, in which it is soluble, so it is plausible to assume that a significant amount of the oxygen would have dissolved in the water, not allowing it to collect in the measuring cylinder. However, in conjunction with this, this method created another error which could offset the solubility issue, which is that bubbling in the water could increase volume due to the production of

water vapour. It would have been very difficult to reduce this source of error at all because of the nature of the method. These errors likely explain the sporadic results I obtained through the use of this method, which gave me a mean value that is significantly lower than the literature value, and does not contain the true value within its error intervals, which is an indication of systematic error.

With the redox titration method, I think a significant source of error was the subjectivity of the experiment, in relation to the point of completion, especially because I was dealing with such small values, and so each drop makes a significant difference. To combat this, I could have removed a small portion of my result, every time, to try and obtain a more accurate result, though this would likely have had its own issues in obtaining the same portion repeatedly, and so it was helpful to make sure that I was the one doing the experiment and determining the end-point of the titration.

To improve all of my experiments, I could have used volumetric pipettes to measure out my reactants, which would have decreased the margins of uncertainty, and thus given more suggestion of systematic or random error. I also could have investigated the purity of my reactants, as this too will have affected my results. Specifically for the redox titration, I could have used a colorimeter to identify a clear point of colour change and completion, which would have helped with the consistency of my results.

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