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27/34

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May

Year

2012

Diploma Programme subject in which this extended essay is registered: Chemistry
(For an extended essay in the area of languages, state the language and whether it is group 1 or group 2.)

Title of the extended essay: An Investigation into the Chemiluminescence of
Luminol

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The extended essay I am submitting is my own work (apart from guidance allowed by the International Baccalaureate).

I have acknowledged each use of the words, graphics or ideas of another person, whether written, oral or visual.

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approached this topic with a lot of thought, and clearly enjoyed setting up the experiments and measuring the intensities of light emitted.

It emerged that the kinetics of luminal reactions are considerably more complex than he had anticipated. Notwithstanding, he pressed on and worked hard to collect and analyse a great deal of evidence.

discussed his research and results with a great deal of enthusiasm. He clearly enjoyed the project as a whole. ✓

This declaration must be signed by the supervisor; otherwise a grade may not be issued.

I have read the final version of the extended essay that will be submitted to the examiner.

To the best of my knowledge, the extended essay is the authentic work of the candidate.

I spent 4 hours with the candidate discussing the progress of the extended essay. ✓

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Assessment form (for examiner use only)

Candidate session number

Achievement level

Criteria	Examiner 1	maximum	Examiner 2	maximum	Examiner 3
A research question	2	2		2	
B introduction	1	2		2	
C investigation	3	4		4	
D knowledge and understanding	3	4		4	
E reasoned argument	3	4		4	
F analysis and evaluation	2	4		4	
G use of subject language	3	4		4	
H conclusion	2	2		2	
I formal presentation	3	4		4	
J abstract	2	2		2	
K holistic judgment	3	4		4	

Total out of 36

27

Signature of examiner 1: _____
(ITAL letters)

Examiner number: _____

Signature of examiner 2: _____
(ITAL letters)

Examiner number: _____

Signature of examiner 3: _____
(ITAL letters)

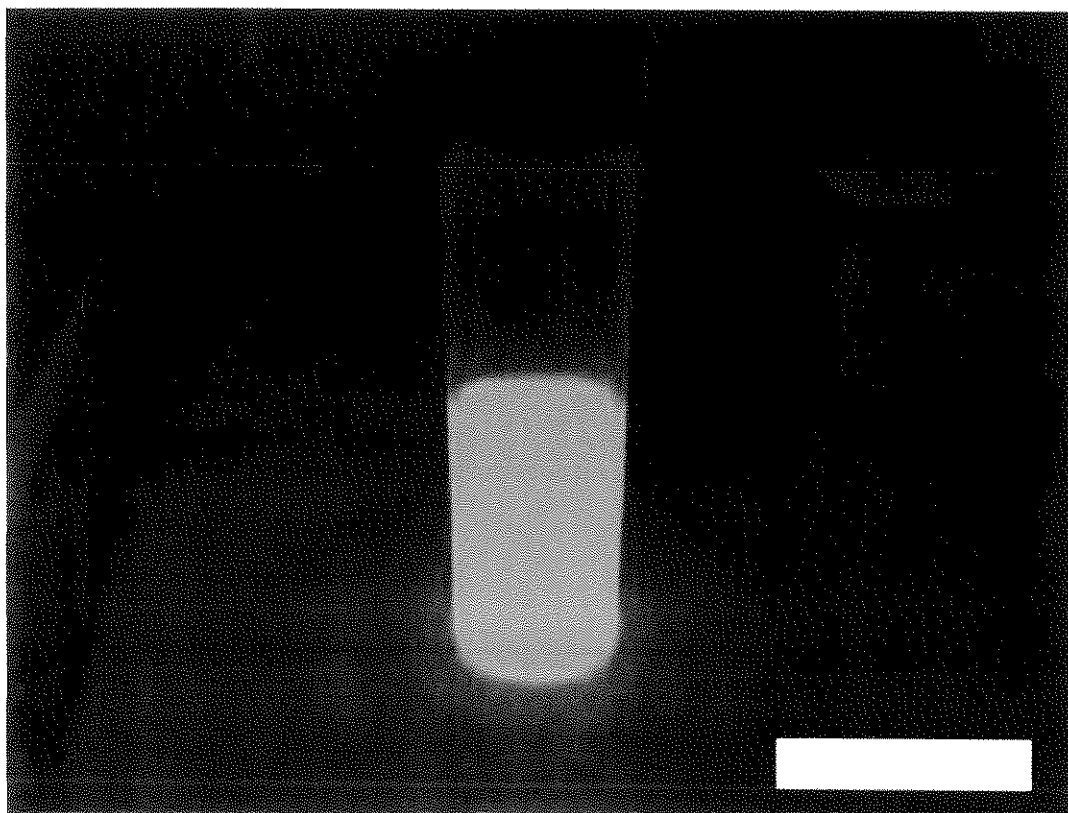
Examiner number: _____

IB Cardiff use only: B: _____

IB Cardiff use only: A: _____

Date: _____

An Investigation into the Chemiluminescence of Luminol



Name:

IB Candidate Number:

Supervisor:

Word Count: 3855 ✓

ABSTRACT

An exothermic reaction is one in which the products contain less energy than the reactants and thus energy is released. In most common exothermic reactions, this energy is lost in the form of heat. However, in chemiluminescent reactions this energy is lost in the form of light, when photons are released. The oxidation of luminol with hydrogen peroxide and sodium hypochlorite is a chemiluminescent reaction. My dependent variable was the concentration of hydrogen peroxide in the reactants and so my research question was "How does varying the concentration of hydrogen peroxide affect the light emitted during the oxidation of luminol (5-Amino-2,3-dihydro-1,4-phthalazinedione) using hydrogen peroxide and sodium hypochlorite?" My hypothesis was that increasing hydrogen peroxide concentration would increase the maximum light intensity of the reaction as well as make the reaction faster, using up more reactants and making a second reaction using the very same reactants produce less light. The experiment was carried out by adding varying volumes of hydrogen peroxide to a constant volume of luminol solution and then adding a constant volume sodium hypochlorite to this solution and measuring the light intensity of the luminescence. The opposite of my hypothesis was concluded to be true as negative correlations were found between both concentration and maximum light intensity as well as between concentration and rate of reaction. However, when no drops of hydrogen peroxide were present at all, the reaction produced the least light intensity and had the slowest reaction rate. My results have shown a role for hydrogen peroxide as a controller or limiter in this reaction in these quantities and have also increased the potential for further investigations on the same reaction. (274)

¹ Title page picture from:

http://s3.amazonaws.com/picable/2008/03/17/127709_Chemiluminescence-2_400.jpg

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INTRODUCTION

A chemiluminescent reaction is one in which an electronically excited species is produced as an intermediate, which drops to a lower state (usually the ground state) and emits 'cold light' in the process, i.e. light which is in excess of and not a result of any heat or black body radiation caused by the reactionⁱ. Luminescence alone is the emission of cold light and there are many types including chemiluminescence, electroluminescence, mechanoluminescence, bioluminescence, thermoluminescence and photoluminescence. Phosphorescence and fluorescence are both a subset of photoluminescence, and these are the two forms of luminescence most confused with chemiluminescence.ⁱⁱ

Apart from its use in glow sticks, there are many practical applications to luminescence. Bioluminescent bacteria are used for quality control in the pharmaceutical industry. Medical uses include the detection of nitric oxide in the breath of asthma patients and in forensics luminol is used to detect blood.ⁱⁱⁱ ✓

My interests in this topic first lay only in luminescence, but after finding out about all the different types, I soon reached the conclusion that chemiluminescence would be the most suitable to look at because it is appropriate to investigate in a laboratory environment and because the presence of a chemical reaction would mean the ability to easily investigate the effect of different variables by changing conditions of the reaction. ✓

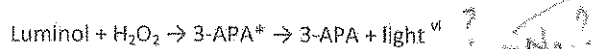
ⁱ <http://www.chm.bris.ac.uk/webprojects2002/fleming/intro.htm>

ⁱⁱ <http://en.wikipedia.org/wiki/Luminescence>

ⁱⁱⁱ Ibid.

EXPERIMENT

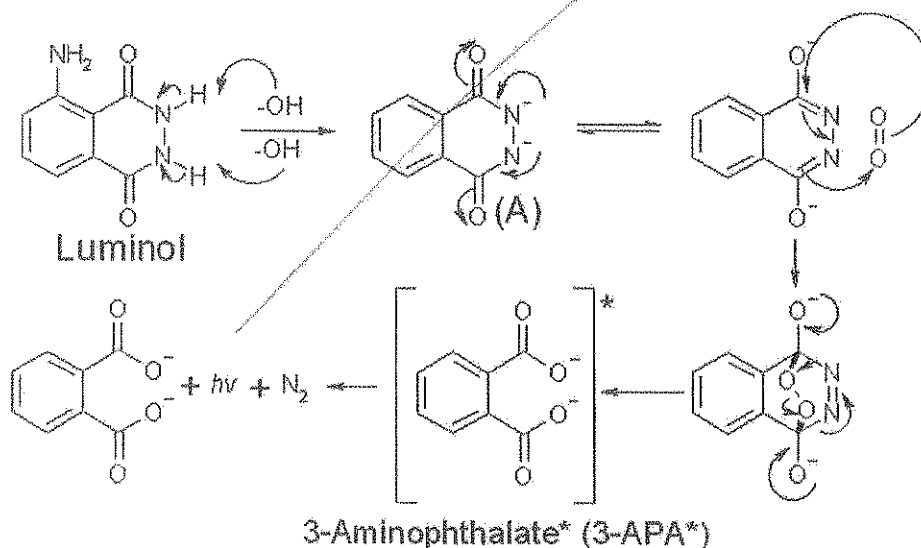
One well-known chemiluminescent reaction and the one I decided to investigate is the oxidation of luminol. In this reaction, luminol (IUPAC name: 5-Amino-2,3-dihydro-1,4-phthalazinedione) is dissolved in an alkaline solution and reacted with an oxidising agent in the presence of a catalyst. The most common oxidising agent used for this reaction is hydrogen peroxide as the O-O peroxide bond is relatively weak but releases large amounts of energy from molecular reorganisation when it is broken, making its breakage energetically favourable.^{iv} The catalyst I decided to use was bleach (NaOCl) as it is a common laboratory and household substance, as well as because sodium hypochlorite and hydrogen peroxide are commonly known to react together to release oxygen, which is what the luminol needs to react with to become oxidised^v. The equation for the reaction of luminol with hydrogen peroxide is as follows:



- 3-APA* is 3-aminophthalate in an electronically excited state
- 3-APA is 3-aminophthalate in its ground state.

*equivalent mechanism
do not agree
where is the N₂?*

The best accepted mechanism for this reaction is shown in Figure 1:



^{iv} <http://www.chm.bris.ac.uk/webprojects2002/fleming/mechanism.htm>

^v http://en.wikipedia.org/wiki/Sodium_hypochlorite

^{vi} <http://en.wikipedia.org/wiki/Chemiluminescence>

Figure 1^{vii}

There are many stages to the process. Firstly, the base converts the luminol into the resonance-stabilised dianion (A) by removing the protons attached to the nitrogen atoms, creating negative charges which then move to the oxygen atoms, producing an enolate ion. Then the oxygen produced by the oxidising agent performs a cyclic addition to create a cyclic peroxide, which directly converts to 3-APA*, a dicarboxylate anion, by releasing the two nitrogen atoms as nitrogen gas. When this compound drops to its ground state, visible light is emitted.

Luminol is a green solid powder which is soluble in most polar solvents but insoluble in water. However, when added to an alkaline solution, it dissolves and the solution acquires a green tinge. In small quantities, it remains clear. When mixed with hydrogen peroxide and bleach, the mixture glows bright blue and then turns yellow when the glow finishes. The blue glow actually consists of two different colours, caused by two different wavelengths. 3-APA* ions which are hydrogen bonded or fully protonated emit at a wavelength of 424nm whereas 3-APA* ions not bonded in this way emit at 485nm.^{viii}

RESEARCH QUESTION

"How does varying the concentration of hydrogen peroxide affect the light emitted during the oxidation of luminol (5-Amino-2,3-dihydro-1,4-phthalazinedione) using hydrogen peroxide and sodium hypochlorite?"

In this reaction, it is commonly acknowledged that hydrogen peroxide is the oxidising agent^{ix, x} and sodium hypochlorite is the chemical which causes the hydrogen peroxide to evolve oxygen^{xii} which will then react with the luminol. However, when carrying out test reactions during my background

^{vii} <http://www.chm.bris.ac.uk/webprojects2002/fleming/mechanism.htm>

^{viii} Ibid.

^{ix} <http://en.wikipedia.org/wiki/Luminol>

^x <http://qsad.bu.edu/curriculum/labs/luminol.pdf>

^{xi}

<http://www.carolina.com/category/teacher%20resources/classroom%20activities/luminol%20the%20glowing%20reaction.do>

^{xii} http://en.wikipedia.org/wiki/Sodium_hypochlorite

✓ Good Q. but not really clear why you have chosen to study the kinetics of the reaction

research, I observed that even when no hydrogen peroxide was added, chemiluminescence occurred. This made me question the roles the two chemicals had and perhaps to what extent sodium hypochlorite is also a reactant in this reaction, as it is also a strong oxidising agent. Thus my research question deals with whether or not the amount of hydrogen peroxide has an effect on the light emitted by the reaction and if so, then what kind of correlation exists. Due to the very nature by which the question was derived, it would be pertinent to include a result for when the amount of hydrogen peroxide in the reaction is 0. ✓

HYPOTHESIS

I predict that as the concentration of hydrogen peroxide added to the luminol solution is increased, the light intensity of the reaction will increase. The maximum light intensity will be greatest when the concentration of hydrogen peroxide is the greatest that I test and it will be the least when there is no hydrogen peroxide present in the reactants. I also predict that, although the light intensity will be greater, the reactants will be used up more in the same time, i.e. the rate of the reaction will be quicker. This means that although the light is brighter, it will not last as long. ✓ OK

METHOD

One of the things I spent most time on as part of my background research was trying to get the most suitable recipe for the luminol reaction as there are many different procedures to make the luminol solution, all with different amounts of different basic substances acting as buffers to provide alkaline conditions required to dissolve the luminol and to allow the reaction to work. The luminol solution I ended up using, as it produced a sufficient amount of light with hydrogen peroxide and sodium hypochlorite and was relatively easy to make, was produced as follows:

- 4g of sodium carbonate (Na_2CO_3) was dissolved in 500cm^3 of water
- 0.2g of luminol was added to this solution and dissolved
- 25g of sodium bicarbonate (NaHCO_3) and 0.2g of ammonium carbonate ($(\text{NH}_4)_2\text{CO}_3$) were added and dissolved in this solution
- The solution was then diluted to 1dm^3 by adding to a 1dm^3 volumetric flask and filling up to the mark with distilled water^{xiii}

what is the function of
 Na_2CO_3 , NaHCO_3 and $(\text{NH}_4)_2\text{CO}_3$?
explain

^{xiii} <http://www.chm.bris.ac.uk/webprojects2002/fleming/experimental.htm>

Chemistry Extended Essay

Even though it is suggested that the catalyst used with this luminol solution is potassium ferricyanide, I still used sodium hypochlorite due to the reasons mentioned above as well as the limited availability of potassium ferricyanide.

When deciding how to measure the light produced, my first consideration was that I would have to be able to block out ambient light, as the dependent variable I would be measuring was light intensity and so ideally I wanted to include in this measurement only light produced by the reaction. I decided that a fully closed box in which the reaction takes place hidden from outside light was the best way to create a dark environment. The reactants would need to be added through a hole in the top of the box as small as possible and a light meter would project inwards through a hole in the side of the box, pointing at the container.

Before beginning my investigation, I carried out a few test reactions with different amounts of reactants to get a rough idea of how much light would be produced. The blue glowing occurred as soon as the two reacting substances came into contact with each other and, more importantly, occurred only *where* the two reacting substances came into contact with each other before fading away quickly within 2 seconds or so. Therefore it became clear that for the investigation I would have to have small volumes for the reactants so that I could have small containers for the reaction to take place in – more specifically, containers with small radii. If the container has a small radius, then the total number of places the luminescence can occur within the horizontal plane of the liquid, all at different distances from the light meter, is reduced and so the experiment will be more consistent and reliable.

Even when using small volumes of reactants, I observed that there would almost always be some of the reactants left over, unless a very large amount of sodium hypochlorite was added. So I decided to react the remaining solution with the same amount of sodium hypochlorite again and, as I expected, the results of this also varied with changes in different conditions. Therefore I also decided to include in my investigation the results for the second additions of sodium hypochlorite to the reaction for all the concentrations of hydrogen peroxide.

Obviously, the factor I would be varying was the concentration of hydrogen peroxide added to the solution and I would do this by adding different volumes of hydrogen peroxide to the same volume of luminol solution. This means every other factor would have to remain constant. The constant volume

6 | would be this change in overall volume
and hence the concentration of all species
You do not mention keeping the total volume
constant. Although you only had 1-6 drops of H_2O_2

Chemistry Extended Essay

of luminol solution I used, keeping in mind that it had to be a small amount, was 5cm^3 and the container I used was a small conical flask which could hold about 15cm^3 .

With the reaction I had, I needed to acquire a suitable range of values for the volumes of hydrogen peroxide to investigate. I found out that very small volumes of hydrogen peroxide had to be used in order for there to be any difference in light intensity. This was probably due to the small volume of luminol solution (the other reactant). These volumes were under 1cm^3 and due to the absence of any equipment to accurately measure such volumes, I decided to measure hydrogen peroxide by drops of a hypodermic needle. The range of values I used was from 0 drops to 6 drops of hydrogen peroxide, added to the same 5cm^3 of luminol solution. I then added a constant volume of 1cm^3 of sodium hypochlorite and measured the light intensity of the light produced by using a light meter.

What concentration of H_2O_2 ?

The apparatus was set up as follows:

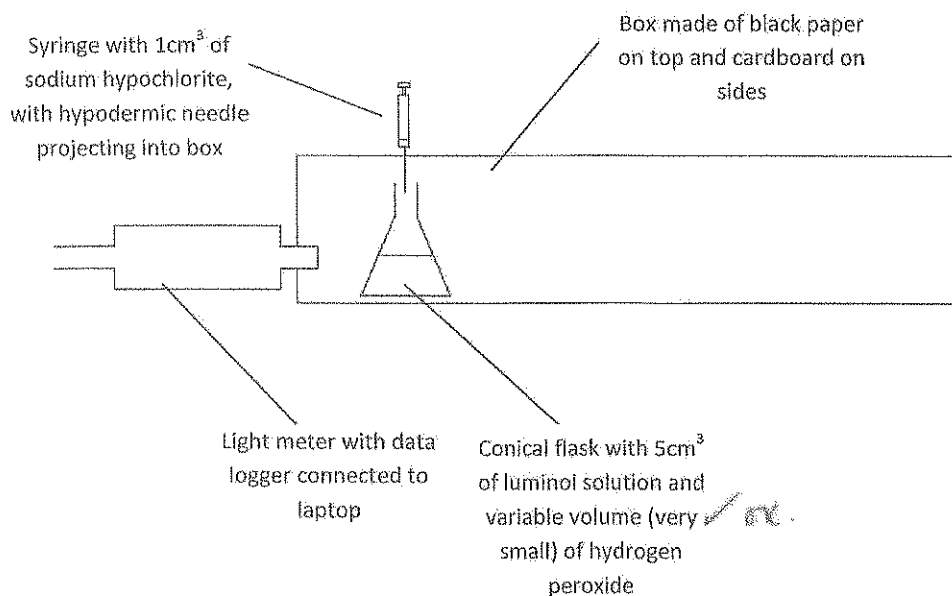
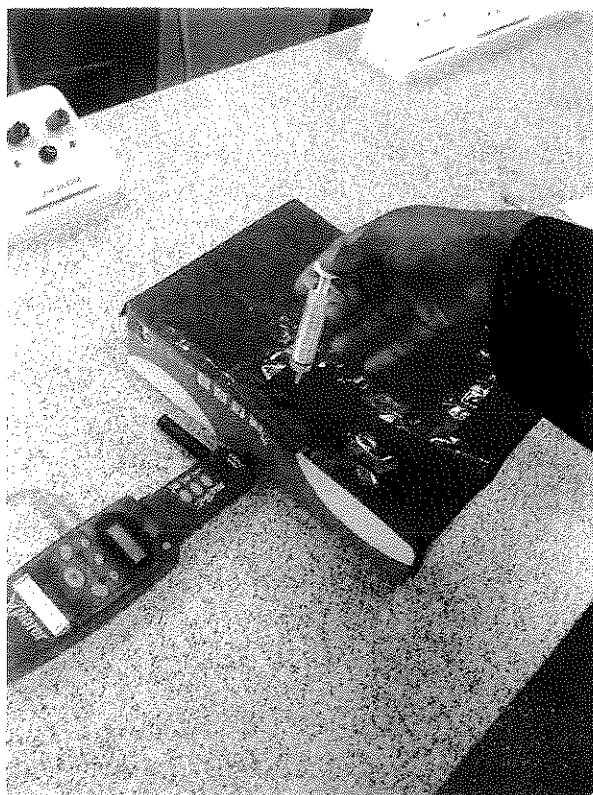
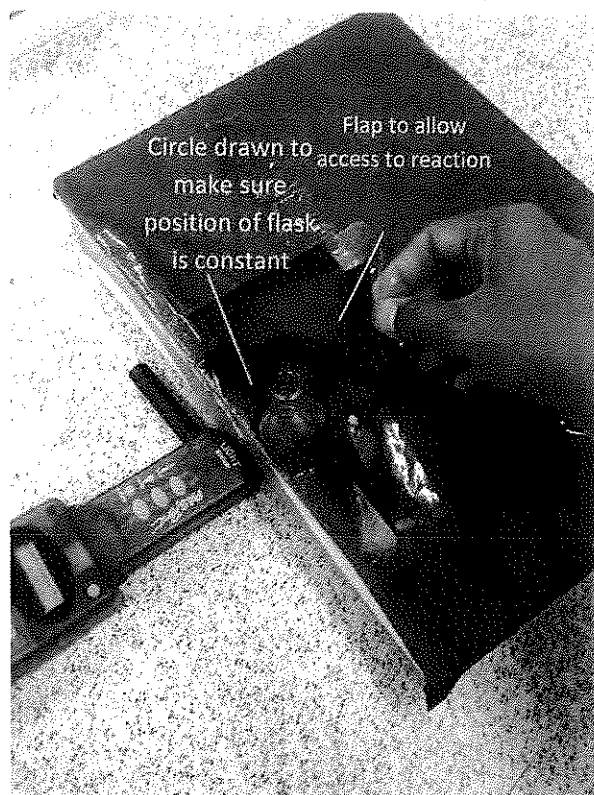


Figure 2

*no concentrations of
 NaOCl , luminol or H_2O_2
given.*



Photograph 1



Photograph 2

✓ excellent
use of images.

The apparatus consisted of a cardboard box lid with black paper taped to the top, acting as a lid. A flap was made so that I could move the flask easily to change between tests. A small circular hole was cut out in the side of the box to allow the light meter, which was connected to a laptop to digitally record results, to project inside. The paper had to be black in order to make the inside of the box as dark as possible. The adding of the sodium hypochlorite would also need to allow as less light as possible into the box. Hence a hypodermic needle was used to do this, as no light could enter through the very small hole in the paper.

Before carrying out the experiment, I placed a flask inside the box next to the light meter and drew a circle to mark its position. I then placed the conical flask in that same position for all the following tests in order to ensure that the position remained constant. This was important as the distance between the reaction and the light meter would affect the light intensity reading. I had to also make

Chemistry Extended Essay

sure the place I would add the sodium hypochlorite into the flask would remain constant and so I made a small hole using the hypodermic needle in the best position possible and then always used that hole to add the sodium hypochlorite to the flask.

✓ good amount of initiative shown and personal input.

For each run, I measured out 5cm^3 in a measuring cylinder and then poured it into the small conical flask. Then I added the correct amount of drops of hydrogen peroxide into the flask and placed it in the box, on the marked circle. To carry out the reaction, I then closed the flap, drew 1cm^3 of sodium hypochlorite in a syringe and injected it into the flask through the hypodermic needle, projecting through the paper. The digital data logger connected to the light meter would show a series of changing values for light intensity but as it was connected to a PC, a graph of light intensity against time could be obtained for each test. I carried out the test for each concentration of hydrogen peroxide twice to make the experiment more reliable.

The colour changes observed in the reaction are shown in photograph 3:



Photograph 3

RESULTS AND ANALYSIS

To record the data, I used the computer software 'DataStudio' which accompanies the light meter and data logger. The software produced a graph of light intensity against time for each run I carried out, meaning I would have many graphs each producing a peak of light intensity at the time I injected the sodium hypochlorite into the conical flask of reactants. I had to adjust the peaks so that they were all relatively in the same time position on the x-axis and I could then take the start of each peak as 0 seconds.

The light meter was set up by default so that it took a reading once every 0.5 seconds but I adjusted it to take readings as frequently as possible, which was once every 0.001 seconds, or 1000 times per second. This was done so that the results would be as accurate as possible and manual interpolation of any graphs, if ever necessary, would not be needed. The peak of maximum light intensity, which would be crucial to my conclusion, would also be more accurate.

A set of graphs showing all my results is shown in Figure 3. As I carried out each run twice, these are also included.

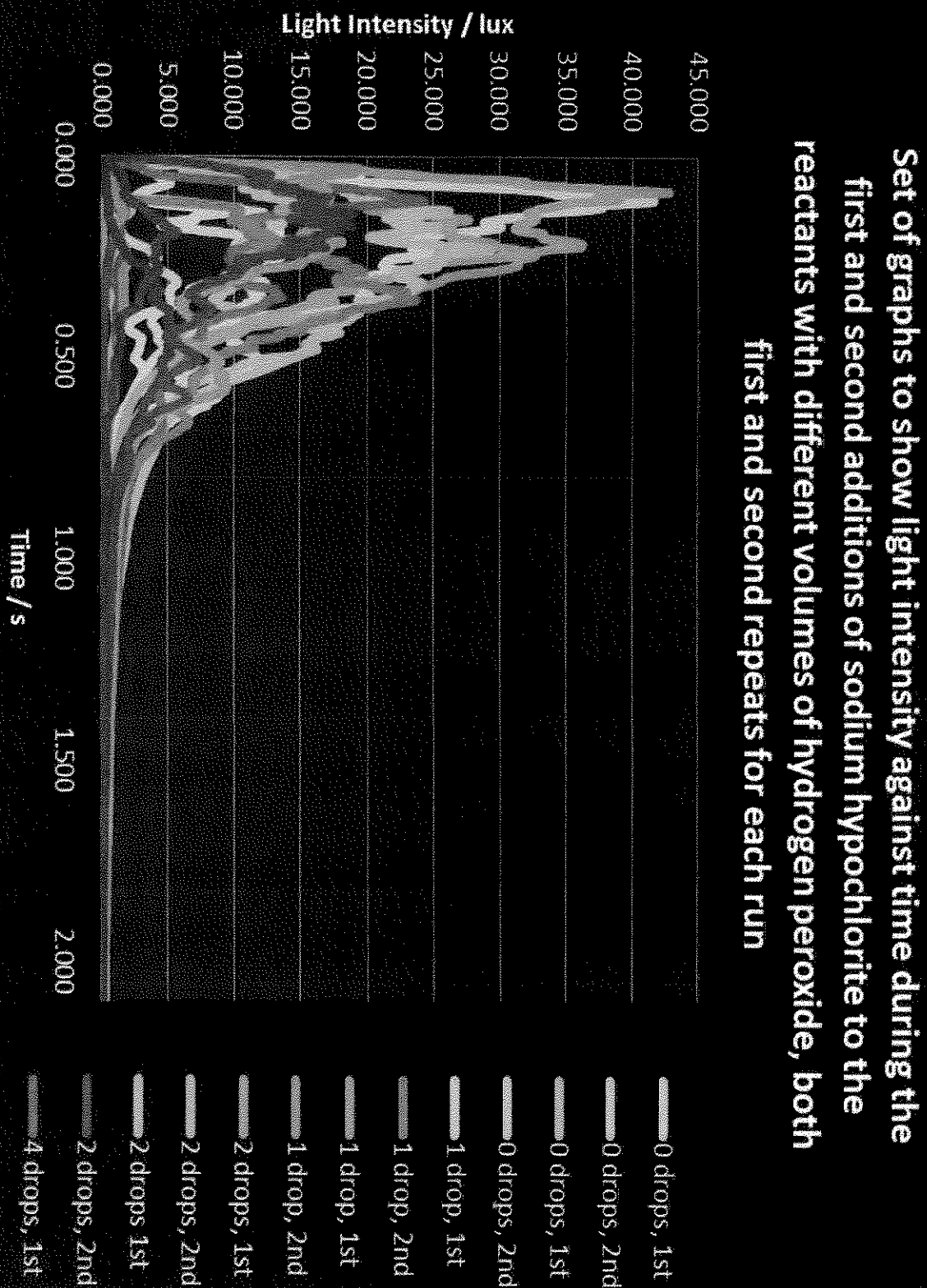


Figure 3

My results showed that the correlation between the amount of hydrogen peroxide in the reactants and the light intensity of the reaction was not as simple as I thought. Overall, it seemed that there was a negative correlation because, during the first addition of sodium hypochlorite, the more hydrogen peroxide that was added, the less the maximum light intensity was. However, the reaction with no hydrogen peroxide did not produce the greatest maximum light intensity, as would be expected. The less hydrogen peroxide used, the brighter the light is, until the point when no hydrogen peroxide is used at all. Then the light becomes dim.

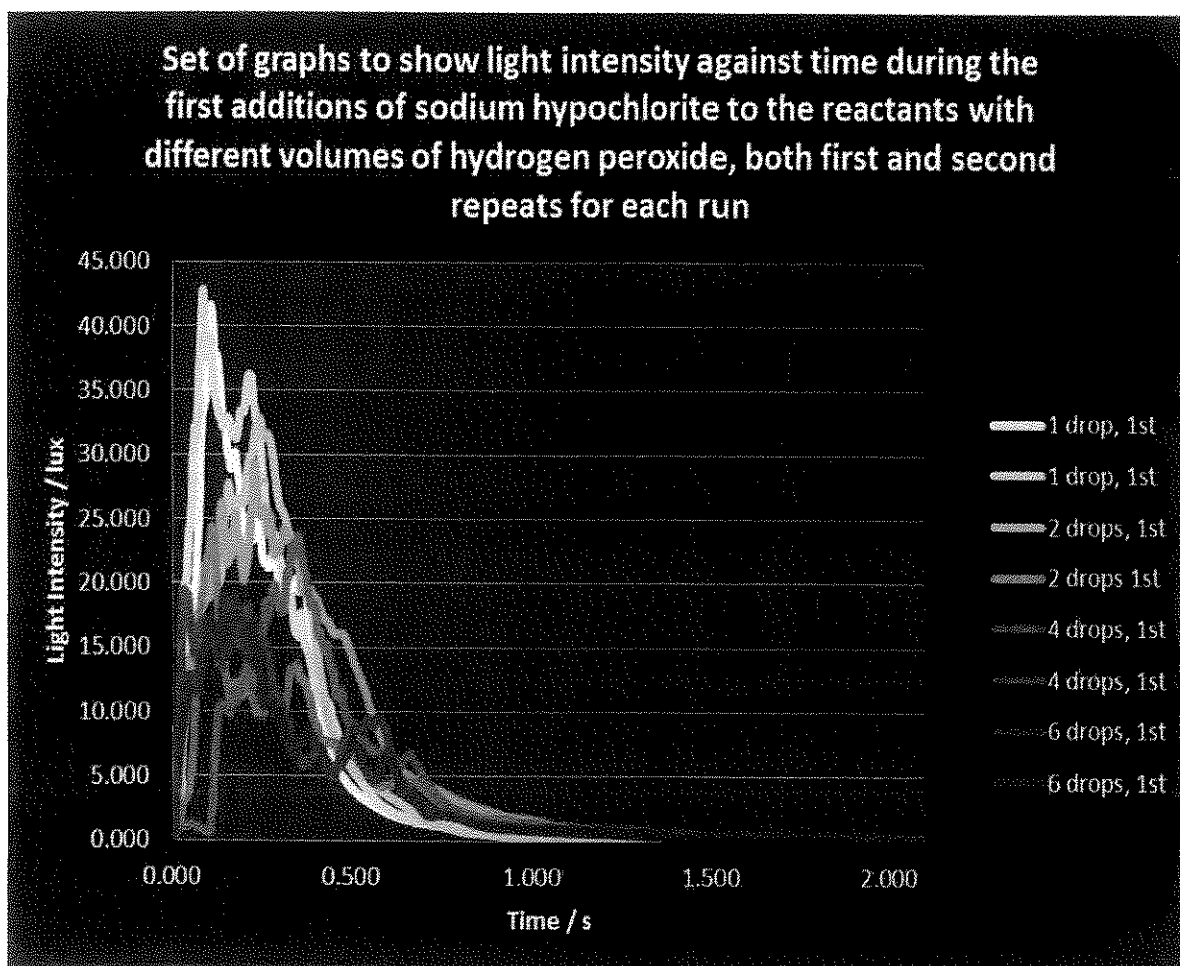


Figure 4

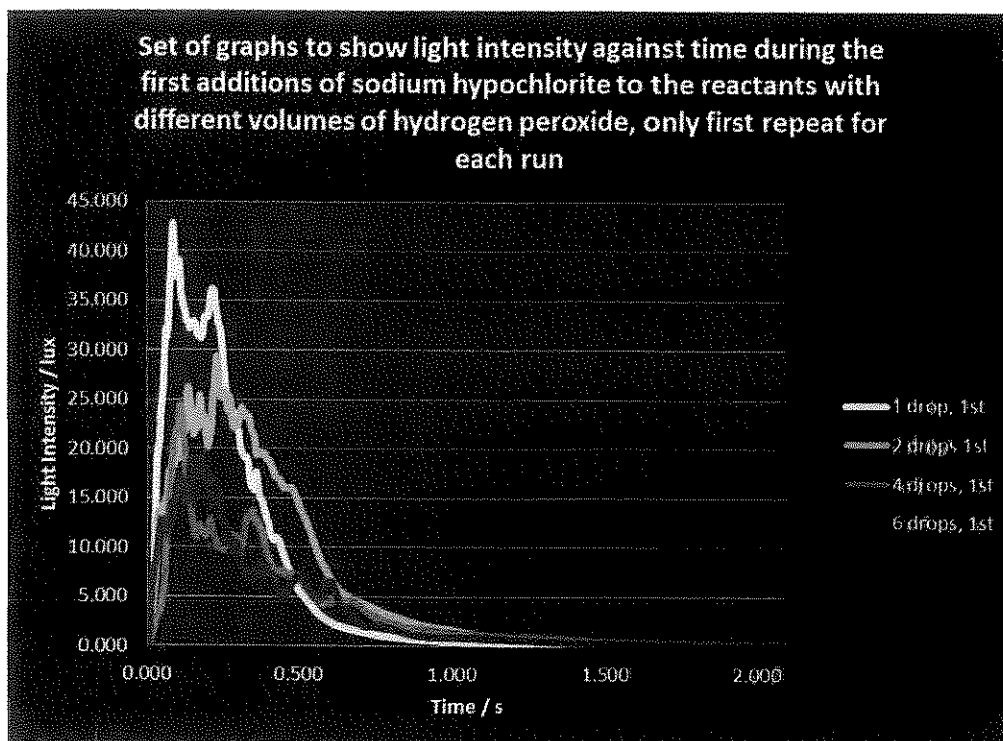


Figure 5

Figure 4 and Figure 5 show the negative correlation between the concentration of hydrogen peroxide added to the luminol solution, between 1 drop and 6 drops, and the light intensity of the reaction caused by the first addition of sodium hypochlorite. The red colour of the lines representing each concentration of hydrogen peroxide in drops has been set to get darker with increasing concentration of hydrogen peroxide, and on both graphs it is visible that the darker the line, the smaller its peak is. The graphs for 0 drops of hydrogen peroxide added have been omitted.

Figure 6

Maximum light intensities of first additions of sodium hypochlorite (raw data)

	Drops of hydrogen peroxide added to 5cm ³ of luminol solution				
	6	4	2	1	0
Repeat 1	18.99	23.72	29.35	42.79	17.95
Repeat 2	19.48	24.35	32.81	41.6	16.21
Average	19.235	24.035	31.08	42.195	17.08

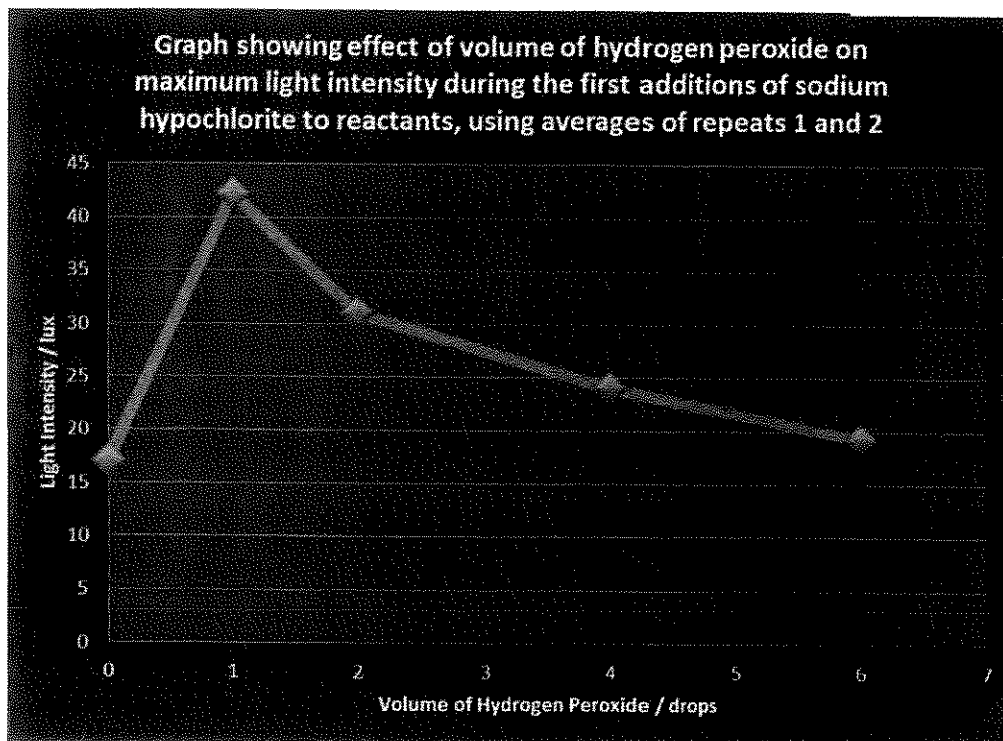


Figure 7

Figure 6 and Figure 7 show the direct relationship between concentration of hydrogen peroxide and the maximum light intensity produced by the first addition of sodium hypochlorite to the reaction, from 0 drops to 6 drops. From this we can see that the more drops of hydrogen peroxide present in the reactants, the less the maximum intensity, but if no hydrogen peroxide is present, the light intensity is even lower than if 6 drops were present. To obtain this graph, I made a table of values (Figure 6) for the maximum light intensities of repeats 1 and 2 against volume of hydrogen peroxide added, by extracting the values from the maximum points on the graphs of light intensity against time, and then took the averages of the two repeats to obtain a single set of results.

The graphs for the second addition of sodium hypochlorite to the reactants were obtained by adding the same amount of sodium hypochlorite (1cm^3) to the flask again, allowing any unreacted substances in the flask to react again. As the light intensity of this second reaction is an inverse measure of how much reactant was used up in the first reaction, and the time of reaction is the same, it can act as an inverse measure of the rate at which the reactants are used up. I found a positive correlation between the concentration of hydrogen peroxide in the luminol solution and the light intensity of the second reaction with sodium hypochlorite, and therefore a negative correlation between the concentration of hydrogen peroxide and the rate of the reaction.

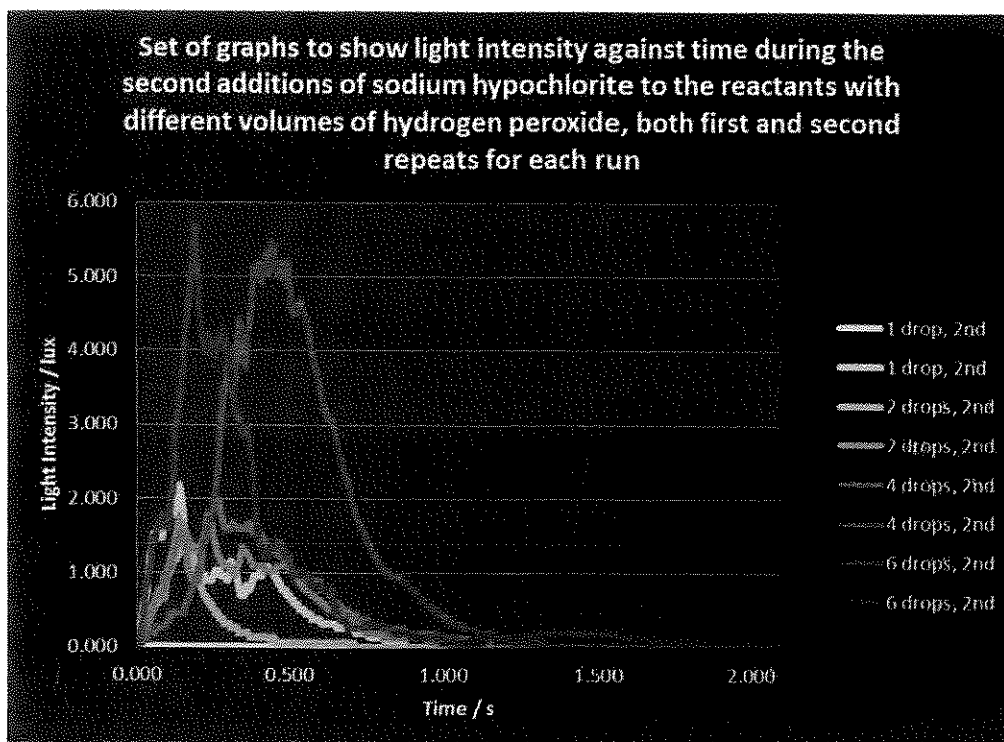


Figure 8

Figure 8 and Figure 9 show the positive correlation between the volume of hydrogen peroxide added to the luminol solution, between 1 drop and 6 drops, and the light intensity of the reaction caused by the second addition of sodium hypochlorite. In this case, the darker the line, the more drops of hydrogen peroxide it represents, and the darker the line, the higher the peak is. The graphs for 0 drops of hydrogen peroxide added have been omitted.

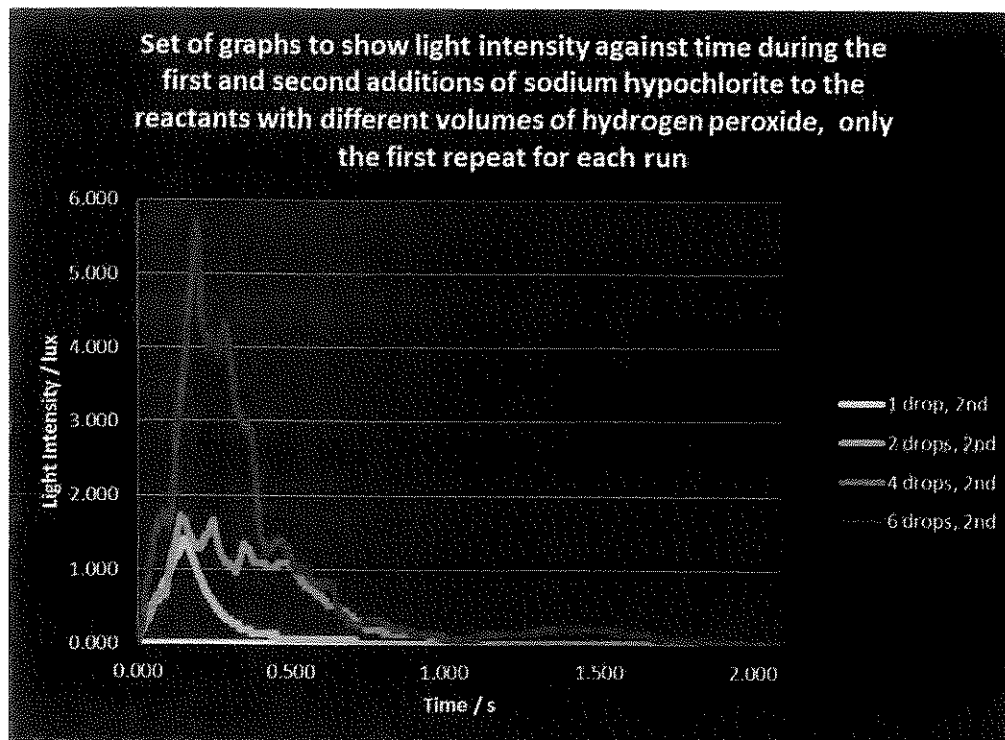


Figure 9

Figure 10

Maximum light intensities of second additions of sodium hypochlorite (raw data)

	Drops of hydrogen peroxide added to 5cm ³ of luminol solution				
	6	4	2	1	0
Repeat 1	5.7	1.99	1.41	0	7.18
Repeat 2	5.45	1.73	2.18	0	12.67
Average	5.575	1.86	1.795	0	9.925

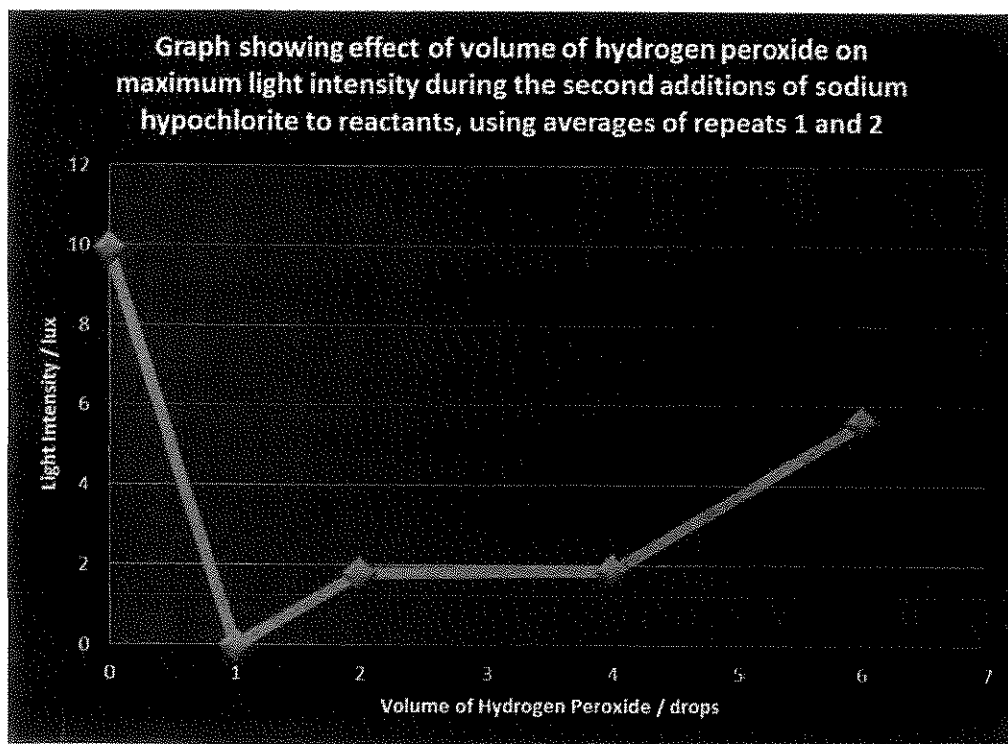


Figure 11

I obtained Figure 11 in the same way as Figure 7. The table of values for the maximum light intensities of the reactions due to the second additions of sodium hypochlorite is shown in Figure 10. Figure 10 and Figure 11 show the direct relationship between the concentration of hydrogen peroxide and the maximum light intensity produced by the second addition of sodium hypochlorite to the reaction, from 0 drops to 6 drops. From this we can see that the more drops of hydrogen peroxide present in the reactants, the more the maximum light intensity of the second addition, but if no hydrogen peroxide is present, the light intensity is even higher than if 6 drops were present. This shows that the rate of reaction decreases as the concentration of hydrogen peroxide increases unless there is no hydrogen peroxide present at all in the reactants, in which case the rate is very slow.

CONCLUSION

My original hypothesis was that as hydrogen peroxide concentration increases, the maximum light intensity of the reaction will increase. The results show that this is wrong, as Figure 7 indicates a decrease in maximum light intensity with an increase in the concentration of hydrogen peroxide in the conical flask of reactants. However, the lowest value of maximum light intensity was still produced by the reaction with no hydrogen peroxide in it.

My second hypothesis was that as hydrogen peroxide concentration increases, the amount of reactants used up during the same time, i.e. the rate of the reaction, will also increase, specifically due to the higher light intensity (that I predicted). As my first hypothesis was incorrect, my second hypothesis was also incorrect as Figure 11 indicates an increase in maximum light intensity of the second addition of sodium hypochlorite, i.e. a decrease in rate of reaction, from an increase in the concentration of hydrogen peroxide in the conical flask of reactants. However, the greatest value of light intensity which was caused by the second addition of sodium hypochlorite, and therefore the lowest reaction rate, was produced by the reaction with no hydrogen peroxide in it.

EXPLANATION

I had expected the light intensity of the reaction to increase with increasing hydrogen peroxide due to the increase in the amount of oxidising agent molecules, causing an increase in luminol molecules which react, causing more 3-APA* molecules, thus causing more light. However this assumes that the luminol is in excess and so increasing hydrogen peroxide concentration will increase the reaction. One consideration I had not thought of while making the hypothesis is that it is very likely that it is instead the hydrogen peroxide which is in excess, even with one drop from a hypodermic needle. This is because very little luminol was added to make the luminol solution in the first place, as only a very little amount is needed (all luminol solution recipes have a very small concentration of luminol)^{xiv}. This means using sodium hypochlorite will evolve far more oxygen from the hydrogen peroxide than the luminol can react with anyway, and so increasing hydrogen peroxide concentration from 1 drop does not increase light intensity but in fact decreases it. ✓ fair point

I came to the conclusion that sodium hypochlorite must also be a reactant in this reaction, working as an oxidising agent without the need for a catalyst. This explains why the reaction still works when no hydrogen peroxide is present in the reactants. This means that a very small amount of hydrogen peroxide is needed to make the solution glow much brighter. After this amount, the hydrogen

dissolved O₂?

^{xiv} <http://www.chm.bris.ac.uk/webprojects2002/fleming/experimental.htm>

peroxide merely slows down the reaction as there are more H_2O_2 molecules in the solution which are uninvolved and do not participate in any collisions and so prevent collisions of the molecules which do react.

Applications of my Findings

My result indicating that increasing hydrogen peroxide concentration decreases light intensity and decreases the rate of the reaction shows that increasing the concentration of hydrogen peroxide in the reaction could be used to control and limit the reaction. At these quantities, more hydrogen peroxide could be added if one wanted less light emission and/or the luminol to be used up less quickly so that the reaction could be carried out many times for a longer time period using the same container of reactants.

EVALUATION AND FURTHER INVESTIGATIONS

The results and conclusion to this experiment have opened up many other questions that could be investigated and answered. As I concluded that the hydrogen peroxide is in excess even at one drop, it should then follow that there would have been a positive correlation between hydrogen peroxide and light intensity up to a certain concentration as hydrogen peroxide is increased before the optimum was reached and then the hydrogen peroxide was in excess. A different way to measure the different hydrogen peroxide concentrations other than by volume, for example the ratio of number of moles of hydrogen peroxide to luminol, especially as the concentration of luminol itself in the solution is so low, would have been more suitable. Alternatively, the constant volume of luminol solution used in the reaction could be increased to make it easier and more accurate. Then the optimum ratio could have been found and compared with the ratio of the reactants in the full equation for the reaction.^{xv}

The series of values for hydrogen peroxide could have also been extended the other way to lengthen the graph in Figure 7. So far the graph stops with 6 drops. However it would be interesting to see, as the line continues, if it continues to plateau, approaching an asymptote, always above the value of maximum light intensity for 0 drops of hydrogen peroxide, or if it dips below the value for 0 drops. If it does dip below, it would be possible to then determine the number of drops of hydrogen peroxide above which it is better to use no hydrogen peroxide at all to create brighter light, by determining the precise concentration value for which the light intensity is the same as when no hydrogen peroxide is added.

^{xv} In fact, after doing more research, I learnt that a positive correlation has actually been found at extremely small volumes (in the concentration range 5×10^{-8} – 7.5×10^{-6} mol/l as shown by the following source: <http://onlinelibrary.wiley.com/doi/10.1002/bio.1170060309/abstract>). This then supports my explanation

should really be in body of text not conclusion no new material

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The great weakness of this EE is that it is non reproducible as the concentrations of the NaOCl and the H_2O_2 solutions used are nowhere stated. So the limiting/excess reagent(s) could not be verified. An interesting RA but lacks proper literature review (bibliography poor) so lacks academic credence. Does show considerable initiative and personal inquiry.