

## Extended Essay in Chemistry

### The effect of a magnetic field on radical reactions

RQ: What is the effect of a 60 Hz magnetic field on the free radical luminol reaction?

Word Count: 3990

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## Introduction

Luminol, (5-amino-2,3-dihydrophthalazine-1,4-dione), is an organic molecule with many uses in a laboratory environment, which typically take advantage of its chemiluminescent properties. This luminescence is promoted by the free radical oxidation of luminol with a strong oxidant, such as hydrogen peroxide ( $\text{H}_2\text{O}_2$ ), and catalyzed by potassium ferricyanide ( $[\text{Fe}(\text{CN})_6]^{3-}$ ). Luminol can be used in criminal forensics, as it illuminates upon contact with blood, and as a radical detector in biochemical research (Barni et al., 2007). The unpaired electron has an unpaired spin state and therefore a magnetic moment which theoretically interacts within a magnetic field (Carbello et al., 2023). There has been extensive literature on the effects of electromagnetic fields on radical reactions, the majority of are focused in the area of biochemistry due to concerns about the effects of electromagnetic fields on human health. These are varying in reliability and credibility, however, most still report some effect of magnetic fields on radical reactions (Khan et al, 2014). However, there has been comparatively little literature on the effects of magnetic fields on luminol reactions in a laboratory. It is interesting to note, however, that in experiments where the intensity of the luminol reactions is used to determine free radical yield, there is little to no discussion on the effect of the magnetic field on the luminol mechanism itself. Thus, it is unclear if a reported change in the luminescence of a reaction in a magnetic field is due to a change in the actual studied chemical mechanism, or the luminol radical reaction mechanism used for detection. This paper will thus investigate the effect of a super low frequency magnetic field (60 Hz) on the luminescence of the luminol radical oxidation by asking the question: What is the effect of a 60 Hz magnetic field on the free radical luminol reaction?

## Background Information

### Magnetic field

A magnetic field must be constructed to observe the effects of a magnetic field on luminol free radical oxidation reactions. A uniform magnetic field is most simply achieved using a Helmholtz coil, first developed by Hermann Von Helmholtz (Purcell, 1989). These are two identical coils of wire, in this case circular, that are separated by the radius of the coil. Given a perfectly thin wire, these in theory create a uniform magnetic field right at the centre of the two coils, as demonstrated in Figure 1.

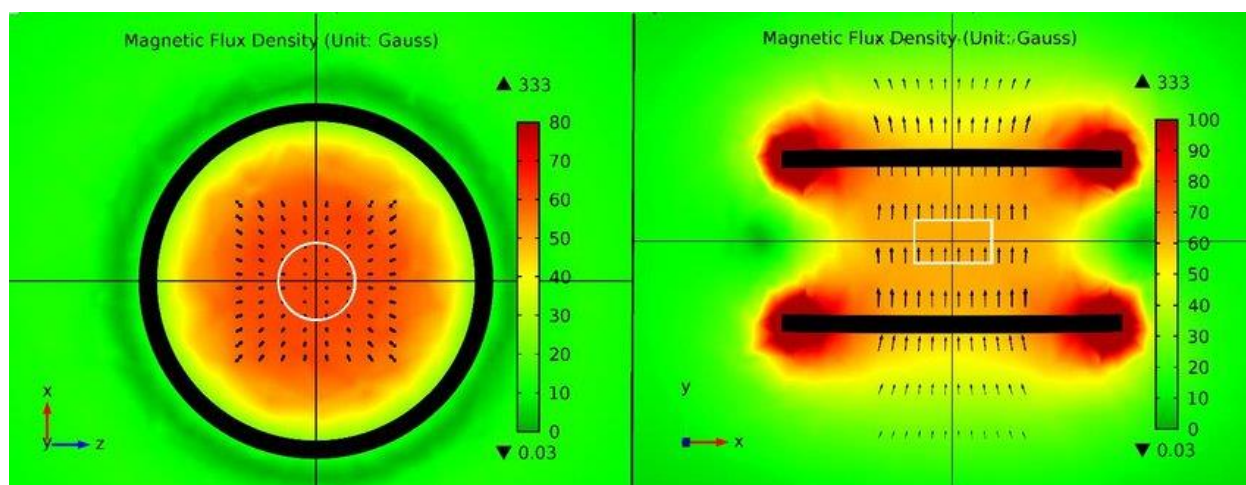


Figure 1 Example magnetic field in a Helmholtz coil (Sundarnath et al, 2020). The arrows show the field direction and the colours show the magnetic flux density. It can be seen that inside the centre of the coil, the magnetic flux is relatively constant, which removes variability.

Alternating current (AC) was used at the standard 60 Hz, or in the Super Low Frequency (SLF) range. The alternating current power supply was 12 volts, with a maximum current of 0.5 amps. The coil structure was 3D printed with a nozzle that had an overall precision of  $\pm 1$  mm. The coils were then wrapped with 50 metres each of 18 AWG (American Wire Gauge) enamel-coated copper wire. There were approximately 110 turns per coil. Each coil was then connected to a 20-ohm resistor, to ensure compliance with the amperage limit of the available power

supply of 0.5 amps. The coil's radius, which was equal to the distance between the midpoints of each coil, was 70 mm. The field strength is given by the following equation from Wolfram Alpha (*Wolfram Formula Repository*, n.d.), where  $B$  is the magnetic flux density in Tesla,  $N$  is the number of turns in one coil, and  $r$  is the radius of the coil, which is also the distance between the two coils.  $\mu_0$  is the permeability of free space, which has a value of approximately  $1.25 \times 10^{-6} \text{ m kg s}^{-2} \text{ A}^{-2}$  (Tatum, 2023).

$$B = \frac{N \cdot I \cdot \left(\frac{8}{5\sqrt{5}} \mu_0\right)}{r}$$

$$B = \frac{110 \cdot 0.5 \cdot \left(\frac{8}{5\sqrt{5}} 1.25 \times 10^{-6}\right)}{0.07}$$

$$B = 0.0007 \text{ T}$$

$$B = 0.7 \text{ mT}$$

As the coils were not perfectly thin, the midpoint was used as the measurement distance. The uncertainty of the midpoint was in this case negligible as the magnetic flux value only had one significant figure and the uncertainty would have been much smaller than that.

Importantly, any surrounding magnetic fields can be considered insignificant. The experiment was performed in a classroom without any large magnetic fields in proximity and the power supplies were placed far enough away to minimize interaction. In any case, those magnetic fields would be very small in comparison to the field generated by the coil. The earth's magnetic field strength near the location of the experiment performed was around 50  $\mu\text{T}$  (*U.S. Department of Commerce*, n.d.), many orders of magnitude smaller the field created by the

Helmholtz coil and can thus be disregarded given the low level of precision attained in the experiment.

### Radical theory

Free radicals are molecules with an unpaired electron. These electrons, being unpaired, contribute to giving the molecule an overall net magnetic dipole. In a homolytic radical split, the molecules can recombine quickly (in around  $10^{-10}$  seconds) as they would, by definition, have opposite electron spins (Timmel et al., 1998). If one spin flips (i.e. goes from up to down or vice versa), then both electrons would have the same quantum spin number. Due to the Pauli exclusion principle, they would not be able to recombine with each other, as they would be sharing the same electron domain, and thus the molecules have a longer life span ( $10^{-5}$  seconds). The longer lifespan allows for further possibility of a propagation reaction with a molecule rather than a termination reaction with the original atom (Timmel et al., 1998).

Radicals can only recombine when they encounter each other, but such encounters are not exactly randomly distributed. The cage effect plays a significant role in this, as it is also responsible for spin polarization. A primary cage is consisted of molecules of the medium which hold the two radicals together in direct contact (*Chemically Induced Magnetic Polarization*, 1977). In the cage, it has a high probability of recombining with the other radical to create the original molecule, or the “precursor” molecule, and a low probability of combining with any other reagents. In the case of luminol, this happens most of the time, as it has a relatively low quantum yield of approximately 0.01 (Li et al., 1993). Quantum yield is a way of expressing the number of photons emitted per molecule, which is the desired yield of the luminol. It is typically expressed as a decimal from 0 to 1, with 0 being no quantum yield and 1 being 100% quantum

yield. If the radicals do not immediately recombine, and leave the cage, they are called escape products. In order to increase or decrease quantum yield, the number of escape products must be increased or decreased respectively. The number of escape products, and hence the quantum yield, is affected in many ways, such as by increasing the concentration of one reagent, by changing the nature of a reagent, among many others. In this case, the relevant mechanism is the S-T (singlet triplet) transition (Molin, 1984).

Electron pair states can be classified as either singlet, doublet, or triplet. Singlet pairs have opposite spins, the doublet state is an unpaired electron, and the triplet pair has two electrons with the same spin (*Chemically Induced Magnetic Polarization*, 1977). There are 3 different triplet states, +1, -1, and 0, or the ground state. Singlet pairs form stable products, so therefore recombination is much more likely. Triplet RP (radical pair) recombination is assumed impossible unless the triplet state is the ground state. Molecule spin alignment remains constant during decomposition, so the spin state is formed in the precursor molecule. When the separation between RP molecules becomes  $10^1$  Angstroms the S and T energies become degenerate (*Chemically Induced Magnetic Polarization*, 1977).

Spin-orbit interactions are essentially what cause S-T transitions in molecules, but not in free RPs. This is due to the increased distance between RPs and thus the non-overlapping nature of their electron orbitals, whereas in molecules, the electron spins are affected by strong exchange interactions (Molin, 1984). S-T RP transitions are most effective where this can be ignored, and each radical electron spin precesses independently in the magnetic field. In a way, it can oscillate between the two states, whereas for spin relaxation, the electron pair loses its correlation (Molin, 1984). The triplet ground state is not the same as the singlet ground state;

in the singlet ground state the angular momentum vectors of equal magnitude and pointing in opposite directions, whereas in the triplet ground state the angular momentum vectors are on the same side (i.e., their vertical components cancel but the horizontal components are additive) (Muus et al., 1977). They are only separated by being out of phase (180 degrees) from each other, so if a physical factor can affect the precession phase, then there can be an S-T 0 transition, but no transitions to the unity ( $\pm 1$ ) triplet states. These can only occur if one of the electrons flips upon the quantization axis (Muus et al., 1977).

Unlike the previous mechanism which did not factor in nuclear magnetic moments, another mechanism, the hyperfine (hf) mechanism is reliant on that. This phenomenon occurs at relatively lower field strengths of  $10^{-2}$  to  $10^{-3}$  Tesla (Molin, 1984). The spin precession of the nuclei and the unpaired electron are independent of each other. The hf field can do S-T transitions to all 3 triplet states in low external fields. In high external fields the total field is close to coincidental with the external field, so the vector sum of the electron spins remains unchanged (Molin, 1984). With the low field being of different strength than the external field, the spin precession can be different, and the spin can flip about the quantization axis.

Kinetic isotopic effects are most effective in the lighter atoms, with mass differences leading to differences in their activation energies, whereas magnetic effects are created only in RPs due to different nuclear magnetic properties (Muus et al., 1977). In heavier elements, magnetic effects exceed kinetic effects. Higher field strengths only allow S-T zero transitions, and lower field strengths allow transitions to any of the three triplet sublevels. Thus, the recombination reaction is 3x more efficient at high fields than low fields (Muus et al., 1977).



This all only works if the radical lifetimes and the S-T transition mixing times are on the same order of magnitude, either radical pair lifetimes must be increased (ways of doing this include increasing the viscosity, having a relatively large initial separation between radical ions, or doing this in a biological setting which is impossible in this case). The obvious alternative would be to shorten the S-T transition times, which is done by increasing the hf constant (picking different chemicals) or by increasing the external field (Muus et al., 1977).

Thus, the external magnetic field promotes the S-T transition. With an increase in triplet state electron pairs, recombination becomes significantly less likely, and thus the overall yield of the reaction should decrease in the presence of a low strength magnetic field (Triebl et al., 1993).

#### Free radical reaction of luminol

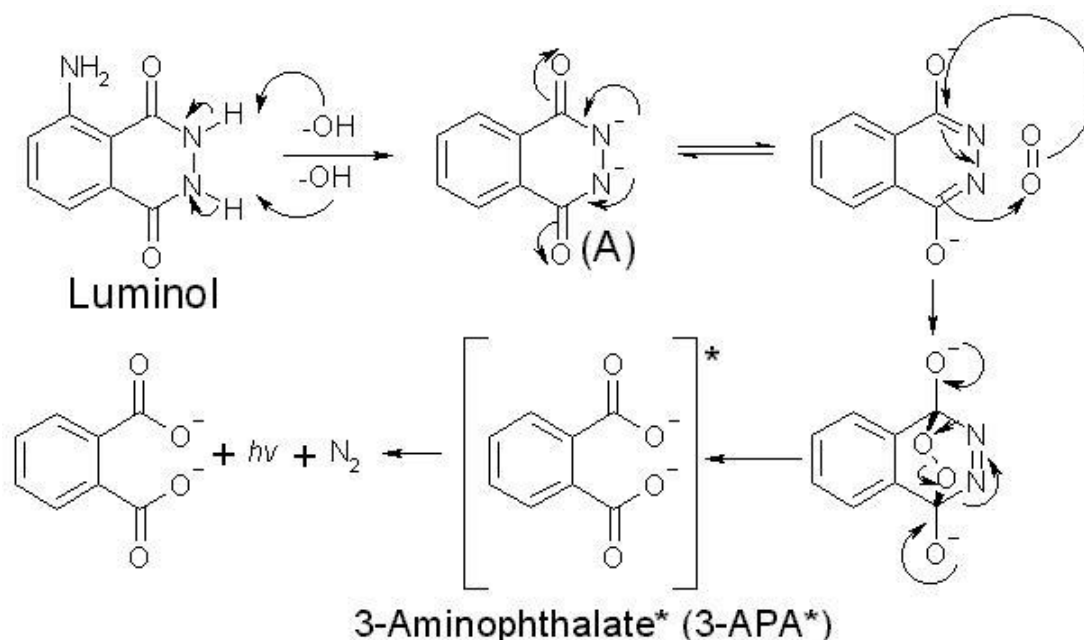


Figure 2 The luminol reaction mechanism (University of Manitoba CRYSTAL / Links, n.d.)

Figure 2 shows the luminol reaction mechanism. The surrounding hydroxide ions (from the sodium hydroxide solvent) dissociate the molecule to create a dianion (Lee & Selinger, 1972). This is then reacted with hydrogen peroxide to create unstable 3-Aminophthalate. As this returns to the ground state, it releases energy in the form of a photon (Lee & Selinger, 1972). This is a pseudo first order reaction, as the emission of a photon happens significantly faster than the radical reaction with the hydrogen peroxide, which is the slow step (Heller et al., 1971). As the radical reaction takes place in the slow step, this allows for the magnetic field to potentially interact with the unpaired electron.

The luminescence of a chemiluminescence reaction varies with temperature. At higher temperatures, the overall reaction yield and duration increases (Macey et al., 2018). Thus, the reaction temperature was controlled, all performed at room temperature (approximately 294 K). The addition of the potassium ferricyanide catalyst would significantly increase the rate of reaction to something measurable, which does produce a brighter reaction but may overall have a lesser yield (Khan et al., 2014). Iron, as a transition metal, has a large diffuse d-electron orbitals which, when interacted with by the luminol, weakens its bonds. This lowers the overall activation energy of the reaction, which then increases the rate of the reaction but can decrease the yield of the reaction due to the interactions with the iron catalyst (Khan et al., 2014).

There is a noted divergence of theory due to the physical constraints of the experiment. Given that the highest concentration of luminol is at the beginning, it follows that the luminescence should be highest initially, and decrease as the reaction progresses. As will be shown, this not observed in this experiment. There is a noticeable peak in every graph since there had to have been some mixing time between the reactants (Macey et al., 2018). Especially

in a thinner reaction vessel such as a test tube, where the initial reactant surface area is small relative to the volume, this will have a noticeable effect on the luminosity value, since the peak shown will always be lower than the theoretical peak for the conditions. The results obtained, a peak around 0.5-1.5 seconds after the addition of luminol are consistent with the literature values (Macey et al., 2018).

### Materials and Methods

The following solutions were all kept at an approximate pH of 13 using sodium hydroxide. A mass of  $0.4000 \pm 0.0001$  grams of NaOH was added to each  $100 \pm 0.08 \text{ cm}^3$  volumetric flask.  $0.0531 \pm 0.0001$  grams of luminol was added to a  $100 \text{ cm}^3$  volumetric flask then topped up with distilled water.  $0.0988 \pm 0.0001$  grams of potassium ferricyanide was added to another  $100 \text{ cm}^3$  volumetric flask then topped up with distilled water.  $0.28 \pm 0.03 \text{ cm}^3$  of 4% hydrogen peroxide was added to last  $100 \text{ cm}^3$  volumetric flask then topped up with distilled water.

Tape was placed at the same height of each  $28 \text{ cm}^3$  test tube, which was used as a marker to ensure relatively uniform placement in the test tube tongs. Each test tube was charged with  $2.00 \pm 0.03 \text{ cm}^3$  of potassium ferricyanide. A micropipette was set to fill at  $1.00 \pm 0.03 \text{ cm}^3$ ; and using the micropipette, either  $1.00$  or  $2.00 \pm 0.03 \text{ cm}^3$  of either luminol or hydrogen peroxide solution was poured into their respective beakers, dependent on the trial run. The light sensor measurement was started, then both beakers were simultaneously poured into the test tube. This was repeated as necessary.

A labelled image of the experimental setup is shown in Figure 3 (for clarity, the image was taken with more light than under actual experimental conditions, as then it would not have been very visible). The full list of materials can be found in the appendix.

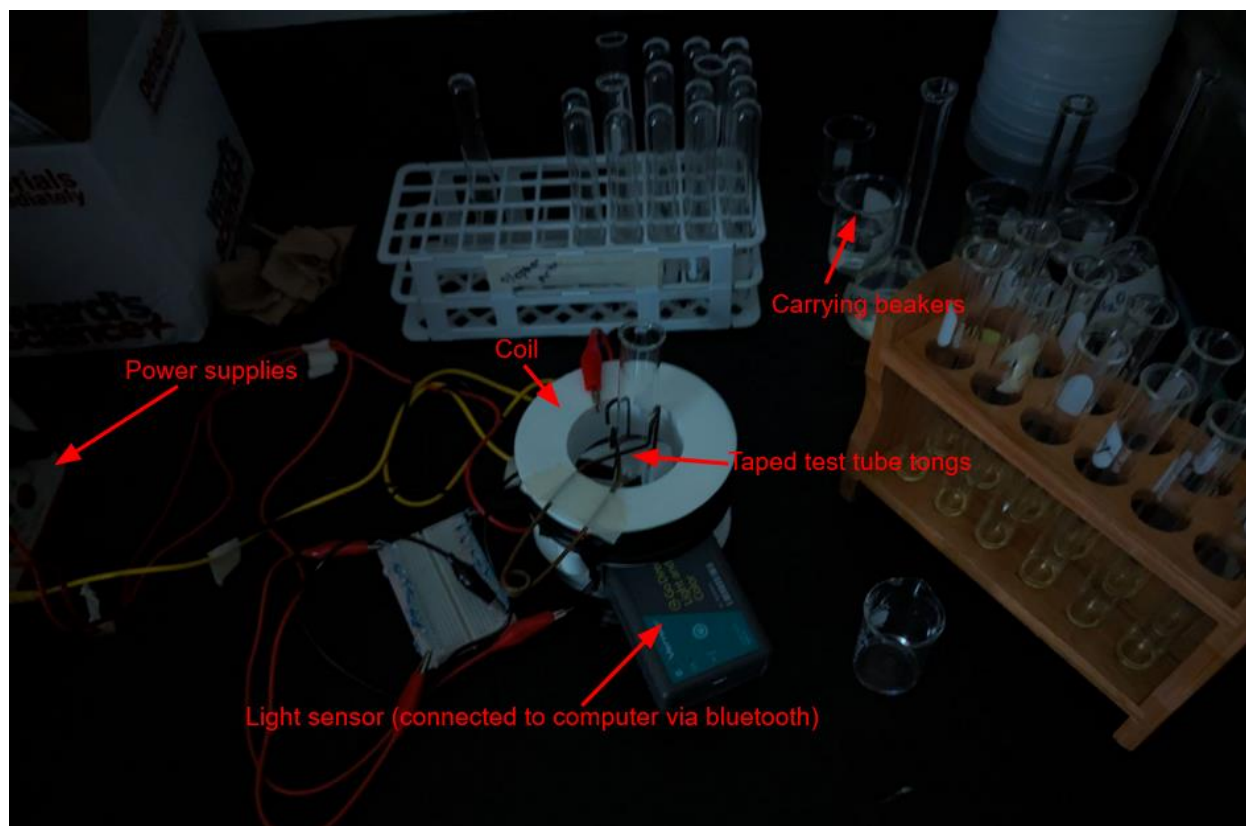


Figure 3 Experimental setup (own work). The placement of the light sensor and test tube tongs remain consistent throughout, the only equipment change was to insert a fresh test tube each time.

### Data Analysis

Quantative data was recorded on the Vernier Graphical Analysis app, which was then exported as a CSV file (table data). Once exported, trials were grouped together and then a simple average was taken between them, which was then put in a graph.

For brevity, the rest of the paper, including tables and graphs, will adopt a naming convention for the luminosity datasets. They are all named along the lines of "x.y.z.m". The x, y, and z indicate the amount of centimetres cubed of potassium ferricyanide, hydrogen peroxide, and luminol respectively in each reaction. If there is a trailing m, then that indicates that the magnetic field was on. Otherwise, the magnetic field was not on.

### 2.2.1

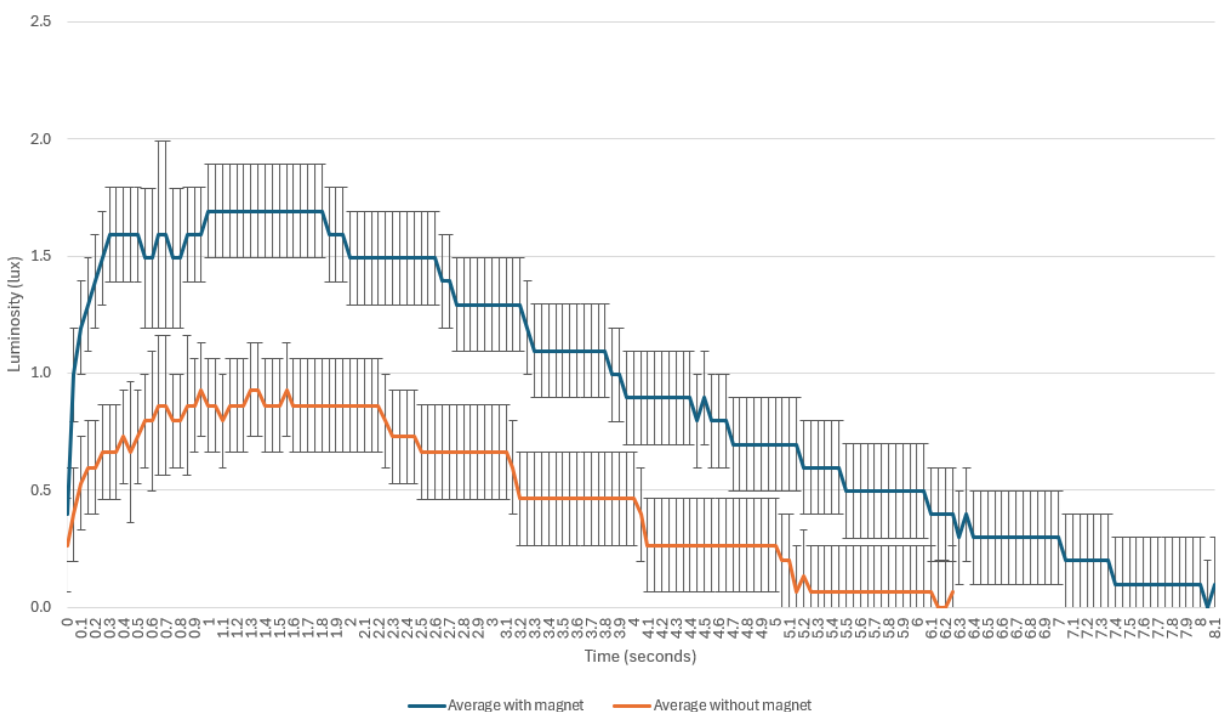


Figure 4 Example graph (own work)

The luminosity measurement was made by starting the data collection and then adding the reactants into the test tube. In order to compensate for this inconsistency in start time, the zero point was defined as the first luminescence readings. These were used to average the values, ensuring the same start time. Note that this was not necessarily the time that the reactants were added (i.e. there may have been a slight delay between addition and the luminescence values showing), but it is impossible to tell there was any delay, so it was assumed to be negligible.

The error bars were calculated by using the half-range method of statistical uncertainty, as demonstrated below. At the 0.85 second mark of the 2.2.1 dataset shown in Figure 4, the following luminosity values were read: 1.2 and 0.6 lux.

$$\text{uncertainty} = \frac{1.2 - 0.6}{2} = 0.3 \text{ lux}$$

There were 2 exceptions to this system of measuring uncertainty. If the uncertainty was below the stated 0.2 lux of the light sensor, in which case it was adjusted to be 0.2 lux. If the downwards uncertainty was greater than the value (as in the error bar would have been in the negatives), then the downwards uncertainty was truncated to match the value so that no negative luminosity values appeared on the chart, since they are not physically meaningful.

### Qualitative data

Each reaction produced light that was of a similar light blue colour. It was interesting to note that even after the light sensor stopped reading any values, there was still obviously some light being emitted from the reaction. As the reaction progressed, the intensity of the light was observed to gradually decay. A few bubbles were observed initially as the reactants were added.

### Evaluation

The data becomes significantly unreliable and unusable in the lower luminosity values. With an uncertainty of at least 0.2 lux, a value of say 0.4 lux would have at least 50% uncertainty. This cannot really be used to calculate anything. As well, it was noted that even after the light sensor stopped showing readings, there was still light clearly visible. This was also seen in a few datasets, where the value fluctuated from 0.0 to 0.2 lux. The reaction is not supposed to go back up after it hits 0.0 lux, and in any case the value of 0.2 lux has 100% uncertainty, so it cannot be used for anything either. Given the unreliability of the data at low luminosity values, total reaction yield and reaction duration would be impossible to ascertain with any degree of certainty. Thus, only the relatively low uncertainty values near the peak luminescence were used to calculate the quantum yield.

In order to calculate the quantum yield, the concentration of luminol is required. The target concentration was  $3 \times 10^{-5} \text{ mol cm}^{-3}$ . Thus,  $0.0531 \pm 0.0001$  grams of solid luminol was weighed out. This was topped up to  $100 \text{ cm}^3$  with water into a volumetric flask with a stated uncertainty of  $\pm 0.08 \text{ cm}^3$ . Using  $C = n/V$ , where  $C$  is the concentration of the luminol solution,  $n$  the amount of moles of luminol,  $V$  the volume of the solution in cubic decimetres,  $m$  the mass of the solution, and  $M$  the molar mass of luminol, the uncertainty of the concentration can be found.

$$C = n/V$$

$$C = \frac{mV}{M}$$

$$C = \frac{(0.0531 \pm 0.0001) \text{ g} \cdot (0.10000 \pm 0.00008) \text{ cm}^3}{177.1 \text{ g mol}^{-1}}$$

$$C = 2.998 \times 10^{-5} \pm 8 \times 10^{-8} \text{ mol cm}^3$$

From the equation given by Barni et al, the quantum yield can be found by dividing the number of photons emitted by the number of molecules of reactant. The light sensor measures in lux and not number of photons. It is assumed that the light generated is very similar wavelength and that the number of photons in contact with the light sensor is linearly proportional to the luminosity measured. Thus, there will be a linear relation constant  $k$ , which will be defined as the scaling constant between the number of photons emitted and the luminosity value. The actual value of  $k$  is impossible to measure with any degree of certainty, as the quantum yield of the luminol reaction has been reported to be many different values (Heller et al., 1971; Lee & Selinger, 1972; Tzani et al., 2013), so the value for the quantum yield in this experiment cannot be mapped onto a theoretical value. Also, the value of  $k$  does not really

matter, as we are just comparing relative quantum yields. In the following equation given,  $Q$  is the quantum yield,  $n$  is the number of moles, and  $I$  is the luminosity in lux.

$$Q = \frac{kl}{n}$$

In order to find the number of molecules, the  $n = CV$  relationship was again used. The volume in this case was the volume of luminol added that was micro pipetted for each trial. The micropipette has a stated uncertainty of  $\pm 0.03 \text{ cm}^3$ .

$$n_{1 \text{ cm}^3 \text{ luminol}} = (2.998 \times 10^{-5}) \cdot (0.00100) \text{ mol}$$

$$n_{1 \text{ cm}^3 \text{ luminol}} = (2.998 \times 10^{-8}) \text{ mol}$$

The uncertainty is calculated below.

$$\Delta n_{1 \text{ cm}^3 \text{ luminol}} = \left( \frac{8 \times 10^{-8}}{2.998 \times 10^{-5}} + \frac{0.00003}{0.00100} \right) \cdot (2.998 \times 10^{-8}) \text{ mol}$$

$$\Delta n_{1 \text{ cm}^3 \text{ luminol}} = 0.09 \times 10^{-8} \text{ mol}$$

An example of the data analysis is shown. The luminosity value used will be the peak value of the dataset, if there is more than one than the one with the greater uncertainty is used. The dataset being used is indicated by a subscript on the  $Q$ , following the same naming convention.

$$Q_{2.2.1} = \frac{kl}{n}$$

$$Q_{2.2.1} = \frac{k \cdot (1.7 \pm 0.2)}{(2.99 \times 10^{-8} \pm 9 \times 10^{-10})}$$



$$Q_{2.2.1} = \frac{k \cdot (1.7 + / - 0.2)}{(2.99 \times 10^{-8} \pm 9 \times 10^{-10})}$$

$$Q_{2.2.1} = k \cdot (5.7 \times 10^7) \pm (8 \times 10^6)$$

Dataset	2.2.1	2.2.1.m	2.2.2	2.2.2.m	2.1.2	2.1.2.m
Quantum yield value	$3.0 \times 10^7$	$5.7 \times 10^7$	$3.2 \times 10^7$	$3 \times 10^7$	$4.0 \times 10^7$	$5 \times 10^7$
Uncertainty	$7 \times 10^6$	$8 \times 10^6$	$4 \times 10^6$	$1 \times 10^7$	$5 \times 10^6$	$1 \times 10^7$

*Table 1 Quantum yield values (own work). The significant figures do not match since some uncertainties were in another order of magnitude.*

Thus, it can be seen that the experimental quantum yield values actually are shown to increase in the presence of a magnetic field. This is in significant disagreement with the values in many other experiments, and especially given the large uncertainty, should probably be disregarded.

### Sources of error

One source of error would be the way the reactants were added. They were poured into the test tube from a height, as compared to the standard procedure of using a continuous flow device to ensure equal contact time. The timing of the pours were aimed to be kept as close as possible, but there was still definitely error involved. As well, in other studies where larger reactant vessels were used, it was reported that the mixing time and contact area of the reactants did have a small but noticeable effect on the luminescence values (Macey et al., 2018). In this case, the reactants were not necessarily mixed, but given the nature of their addition, especially with the bubbles seen, there was error induced in the luminescence values.

This error was not quantifiable but would have contributed to lowering the luminescence value. In any case, this error would have affected both the magnet on and magnet off trials.

Creating a more concentrated solution would have reduced the uncertainty significantly. Not only would this have decreased the percent uncertainty surrounding the number of moles, but it would have also increased the luminescence, decreasing the percent uncertainty in the recorded luminosity. If this experiment were to be repeated at this concentration, then using a more sensitive light meter would also allow for a calculation of the total reaction yield using integration (by integrating the function to find the total luminosity of the reaction), as well as seeing the effect of the magnetic field on the length of the reaction.

One potential source of error that was ruled out was the effect of the magnetic field on the light sensor. The mechanism of the light sensor, being electronic and wirelessly connected to a computer, was completely unaffected by the magnetic field. This was shown by having two constant light trials, during which the same light was shone on the sensor in the same position. There was no noticeable difference measured by the sensor between the magnet on and magnet off trials.

## Conclusion

This paper measured the effect of a magnetic field on the free radical luminol reaction. While the results proved to have too great of an uncertainty and deviation from commonly accepted values to actually mean anything, there are still many interesting areas of further research in this area of magnetochemistry. This experiment could be repeated more accurately in many ways. The exact experiment can be repeated with a better setup, with a controlled

microflow device in order to maintain constant contact between the reagents. Then, the same analysis can be performed. If measuring the quantum yield is not relevant to a further study, then the luminescence values can be bypassed altogether and instead a radical quencher can be added in order to directly measure the actual yield or the rate of the reaction using some sort of spectroscopy. Another interesting area of study would be with a magnetic field in the order of magnitude around 5 Tesla and beyond. Little research has been done in this area, mostly due to the difficulty (but not impossibility) of achieving such strong magnetic fields in a laboratory setting. This is interesting though due to the reportedly different mechanisms and interactions that would be prevalent at this field strength. This may have a different effect on luminol reactions than that previously shown.

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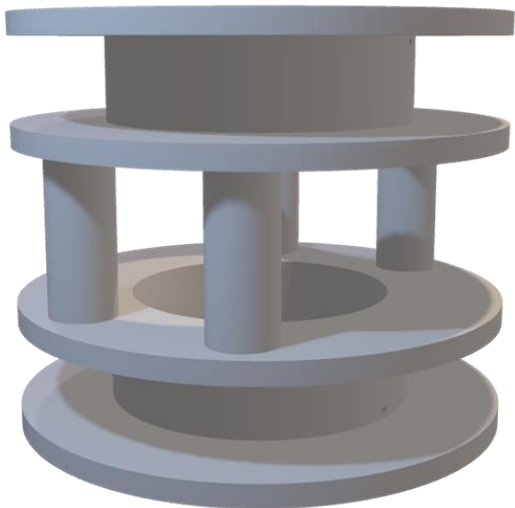
## Appendices

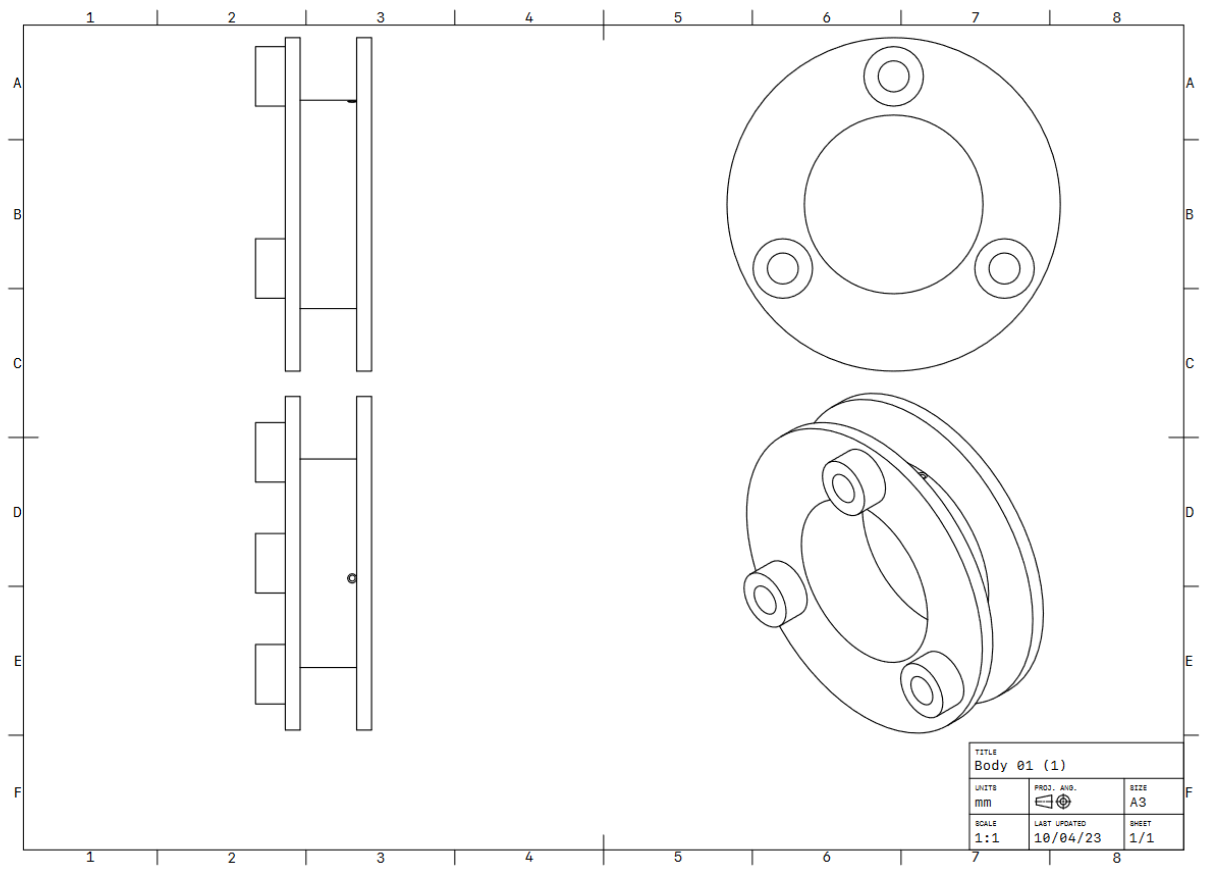
### Materials list

- 100 cm<sup>3</sup> volumetric flask (x3)
- Micropipette
- Micropipette tips (x3)
- Test tubes
- Test tube rack
- Test tube tongs
- Tape
- 100 cm<sup>3</sup> beaker (x6)
- 100 m 18 AWG copper wire
- 3D printed Helmholtz coil (diagram shown below)
- Banana-clip to alligator clip test lead (x4)
- Alligator-alligator clip test lead (x2)
- 12 V power supply (x2)
- 20-ohm resistor (x2)
- Vernier GoDirect light sensor and connected device
- Distilled water
- Sodium Hydroxide
- Luminol
- Hydrogen peroxide
- Potassium ferricyanide



## Helmholtz coil model





In this STL, the side view of the coil is on the left. There is a small hole to allow for the wire to protrude to be connected to the power supply.