The illumination intensity dependence on the rate behaviour of charge recombination after hole injection was tested for two Ru-based donor acceptor style chromophores adsorbed on a nickel(II)oxide surface, using transient absorption flash photolysis. The complexes were shown to exhibit similar behaviour over the intensity range tested, through applying exponential fits to the kinetic traces obtained.
To Aradhna
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INTRODUCTION

Most of the energy generated for society today comes from fossil fuels.\textsuperscript{[1]} It is generally accepted by the scientific community that this is unsustainable.\textsuperscript{[1]} As the demand for energy increases \textsuperscript{[1,3]}, optimising alternative, clean and renewable ways of generating energy becomes increasingly important. Nature’s most abundant energy source is the sun.\textsuperscript{[2]} It is therefore clear that the concept of harvesting and storing energy directly from sunlight shows immense potential.

One way to capture energy from sunlight is to make use of the photovoltaic effect present in semiconductors. This effect occurs when light hits a semiconductor’s surface; electrons are excited to higher energy states, creating a charge separation between the negatively charged electrons in the excited states and the positively charged electron holes left in the ground states. Photovoltaic devices (solar cells) take advantage of these charge carriers by inducing movement to opposite electrodes, thereby generating an electric current. Many conventional solid state solar cells (SCs) create an electric field, through putting n-doped and p-doped semiconductors next to each other, creating a so called p-n junction. Doping is the process by which atoms in the semiconductor crystal are replaced with atoms containing more conduction band electrons (n-type) or valence band holes (p-type), thus increasing the concentration of conducting charge carriers in the material (see section 2). The field created leads the excited electrons through the n-type semiconductor and the positively charged hole through the p-type material.

Solid state solar cells based on crystalline silicon (c-Si) are the most commonly used form of solar cells,\textsuperscript{[9]} with conventional c-Si SCs now reaching efficiencies of over 20%.\textsuperscript{[15]} However, these devices employ relatively thick layers (µm)\textsuperscript{[14]} of crystalline silicon, which needs to be extensively purified (a very costly procedure).\textsuperscript{[14]} Supplements to conventional c-Si SCs are therefore being thoroughly investigated.\textsuperscript{[3,4]}

Dye sensitised solar cells (DSSCs) have been of great interest to researchers since Grätzel and O’Regan introduced an efficient solar cell constructed with a ruthenium-based sensitiser adsorbed on a nanoporous titanium dioxide surface in 1991.\textsuperscript{[5]} The sensitiser absorbs sunlight, and injects an electron into conducting states of the titanium dioxide. The oxidised sensitiser is then reduced by a redox couple in solution, which then transports the electron to a counter electrode. This type of dye sensitised solar cell is referred to as n-DSSC and the processes in these have been thoroughly studied and optimised since Grätzel’s and O’Regan’s breakthrough in 1991. Though extensively investigated, much research is, to this day, directed towards broadening the understanding of mechanisms occurring in these devices.\textsuperscript{[3]}
A concept that has become subject of interest to researchers more recently is creating p-type analogues to n-DSSCs.[4] In these p-DSSCs, the sensitiser accepts an electron from the semiconductor after being excited by a photon, thereby effectively injecting a positive charge carrier into the material. The reduced sensitiser is regenerated (oxidised) similarly to the sensitiser in n-DSSCs by a redox-couple in solution. The most common semiconductor used for p-DSSCs today is nickel(II)oxide.[4]

P-DSSCs offer exciting new possibilities. Improving the photovoltage generated by these devices would allow for building tandem cells, in which the photoanode would correspond to an n-DSSC system and the photocathode would correspond to a p-DSSC arrangement.[6] p-type cells also inspire the concept of photogenerated H₂ production on a semiconductor surface, by allowing the reduced sensitiser molecules to transfer electrons to co-adsorbed proton reducing catalysts. [7]

Despite significant improvements, p-DSSCs still lag behind their n-type correspondents in efficiency and research on these devices is still in its infancy. There is still much to be done to improve our understanding of the processes present in these cells. To understand the shortcomings of p-DSSCs, we must first consider what determines the efficiency of a photovoltaic device.

![Figure 1: Voltage as a function of photocurrent density in a hypothetical solar cell. The fill factor is the ratio of the areas of the small rectangle and the larger rectangle. This illustrates how increasing the fill factor results in a higher power output.](image-url)
1. EFFICIENCY OF SOLAR CELLS

The energy conversion efficiency of a solar cell ($\eta$) is given by the ratio of generated power ($P_{\text{max}}$) and the power of the incoming light ($P_{\text{in}}$):

\[
\eta = \frac{P_{\text{max}}}{P_{\text{in}}}
\]

The power obtained by the cell is the product of the current density and the voltage generated. As the voltage changes with the current obtained, it is useful to introduce the concept of the fill factor ($ff$). Figure 1 shows the voltage as a function of photocurrent density in a hypothetical solar cell. The voltage obtained with negligible photocurrent is denoted $V_{\text{oc}}$ (open circuit voltage), and the current density obtained with negligible resistance over the cell is denoted $J_{\text{sc}}$ (shot circuit current). $P_{\text{max}}$ denotes the point where maximal power is generated. We define the fill factor as:

\[
ff = \frac{P_{\text{max}}}{V_{\text{oc}} \cdot J_{\text{sc}}}
\]

It can be seen that minimising the decrease of voltage with increasing current will maximise the power generated by the cell. The fill factor can be thought of as a measure of how much the dependence of voltage on photocurrent looks like a rectangle, and can reach values between 0 and 1. It is clear that maximising the fill factor will lead to maximising the cell’s power output. Combining equations a and b gives us the expression:

\[
\eta = \frac{ff \cdot V_{\text{oc}} \cdot J_{\text{sc}}}{P_{\text{in}}}
\]

Increasing any of the terms in the numerator will increase the efficiency of the solar cell.

It should be mentioned that the efficiency of a conventional solar cell is strongly limited by the properties of the materials it consists of. Efficiencies approaching 100% are therefore not possible. Since no solar cell can absorb over the whole solar spectrum, spectral losses account for a big part of the lost efficiency.\textsuperscript{[13]} The theoretical efficiency limit for (single junction) silicon SCs was first estimated by Shockley and Queisser to be around 30%.\textsuperscript{[12]} Similarly, the efficiency of DSSCs is limited by spectral losses. Making use of n-type and p-type cells as photoanode and photocathode, respectively, in a tandem structure would allow for a wider range of light from the solar spectrum to be absorbed. In terms of efficiency as described above, this would allow for a higher voltage to be generated, since the voltage in the tandem cell would be the sum of the voltage produced by each of the photoelectrodes separately.
2. THEORY OF p-DSSCS

2.1 The Structure

To understand the structure and the processes present in p-type DSSCs it is useful to introduce band theory to the system. This helps describe the properties of a semiconductor in terms of energy levels. The structure of a semiconductor allows a broad distribution of electronic energy levels, and instead of considering isolated energy states, it becomes more suitable to consider the density of electronic states in the material for given energies. In semiconductors, the highest occupied states form an energy band, referred to as the valence band. The lowest unoccupied states form another band, called the conduction band. The bands are separated by an energy gap, the band gap. The valence band and the conduction band can be seen as the bulk equivalent of the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) respectively in individual molecules.

An important concept that needs to be mentioned when introducing band theory is that of the Fermi-level; the total chemical potential of electrons (and electron holes) in the semiconductor. In terms of distribution, this can be seen as a statistical energy level with a 50% probability of being occupied by an electron at any time. When holes are injected into the semiconductor, the concentration of charge carriers in the semiconductor shifts from equilibrium, and the distribution of electrons and holes can no longer be described with the same energy level. A so called quasi-Fermi level for each charge carrier is then assigned to describe the distribution of the electrons and electron holes (not denoted in figure 2).

Figure 2 shows the energetics of a sensitiser adsorbed on a semiconductor surface in a p-DSSC arrangement, and the important processes present in the cell.

![Figure 2: Illustration of the energetics in a p-DSSC. Each of the relevant processes is illustrated with an arrow.](image)
The processes, as denoted in the scheme, are:

1. **Excitation of the sensitisier**: The sensitisier adsorbed on the semiconductor surface absorbs a photon, delocalising an electron to an electronically excited state.

2. **Hole injection into the valence band of the semiconductor**: The semiconductor transfers an electron to the excited sensitisier molecule creating a positively charged hole in the valence band.

3. **Charge recombination between the reduced dye and the injected hole in the valence band**: The reduced sensitisier transfers an electron back to the semiconductor, recombining it with the positive charge in the valence band.

4. **Regeneration of the reduced sensitisier with the redox couple in the electrolyte**: The redox mediator oxidises the reduced sensitisier.

5. **Interception of the hole in the valence band by the redox couple in the electrolyte**: The redox mediator in the electrolyte transfers an electron to the semiconductor, combining it with a positive charge in the valence band.

6. **Electron transfer from the redox mediator to the counter electrode**: The reduced mediator transfers an electron to the counter electrode (CE) of the cell.

Of the processes listed above, 3 and 5 are undesirable. Fast interception and recombination (severely) limit the efficiency of current p-DSSCs.\(^4\) In order to improve the performance of these devices, we must understand what causes these processes, and how they affect the energy output.

### 2.2 The Limiting Factors

As mentioned above, p-DSSC efficiencies are considerably smaller than their n-type counterpart’s. The small generated currents, fill factors and low photovoltages limit the present cells’ efficiencies.\(^4\)

The low fill factor is mainly attributed to a high “dark current” in the semiconductor, caused by interception of the holes by the electrolyte. This reaction is dependent on the voltage. It can be seen as causing a current in the wrong direction, as it competes with the current in the electric circuit created by the cell. The most successful approach to reduce this effect has been through modifying the sensitisier to block the electrolyte from the semiconductor surface, as reported by Nattestad. However, despite the decreased dark current, the fill factor obtained was still low (ff=0.35).\(^4\)
The low current is, among other things, a consequence of the low mobility of electron holes in the valence band of the semiconductors used. This directly limits the efficiency of the p-DSSC. The low mobility also leads to charge recombination reactions being able to compete more efficiently with the diffusion of the holes.

The Voc is limited by the difference in potential between the quasi-Fermi level above the valence band, and the potential of the redox shuttle. This puts a theoretical maximum to the Voc, restricted by the materials of which it is constructed. Current NiO based p-DSSCs do not reach a high enough potential difference with their redox mediators to promise efficiencies matching their n-type correspondents. [4] Furthermore, the voltage is reduced by the interception and recombination reactions.

![Figure 3: The sensitisers used in the study.](image)

[The complexes are \([\text{Ru(dcb)}_2\text{NMI-Phen}]^{2+}\) with the carboxylic groups in the 3,3’ (left picture, denoted J04) and 4,4’ (right picture, denoted LD2). These complexes adsorb to the NiO – surface with their carboxylic groups. When reduced, the negative charge is localised on the phen-NMI group. [10] ]

In order for the solar cell to generate a current the reduced sensitisier must be oxidised by the redox mediator within the charge separated life time. In other words, the regeneration reaction must efficiently compete with charge recombination of the hole and the electron in the reduced dye. Until recently, p-type solar cells were plagued with ultrafast charge recombination on the sub-nanosecond scale. [4] Significant improvements have been made through designing donor-acceptor style sensitisers, which localise the electron as far as possible from the semiconductor surface. [10] These show that at least part of the recombination can be slowed down to the microsecond timescale. This was proposed to depend on the geometry of the adsorbed sensitisers. Despite recent progress, the fast charge recombination is still a problem in p-DSSCs and needs to be further investigated.
2.3 The aim: Illumination intensity dependence study

The purpose of this study is to gain additional information on the recombination process by investigating if, and how, the kinetics of the decay of the reduced state (through recombination) change when varying the incoming light intensity. Increasing the intensity of the incoming light will increase the quantity of the excited sensitisers complexes, which will lead to increased hole injection into the semiconductor, possibly changing its chemical properties (for example the energy of the quasi-Fermi-level for electron holes). As a p-DSSC is a very complex system, this, among many other possible mechanisms, could lead to a change in the decay rate of the reduced state. The experiments were conducted in the hope to gain additional information on the present system.

Two sensitisers (shown in figure 3) adsorbed on a nickel(II)oxide surface were investigated. These systems have been shown to exhibit long charge separated life times, making the recombination process suitable for study with flash photolysis (as described below).\textsuperscript{[10]} The sensitisers adsorbed on a NiO surface have known absorbance maxima near 460nm.\textsuperscript{[10]} The reduced states of the sensitisers formed after hole injection show maximal absorbance near 560nm.\textsuperscript{[10]} Through exciting the samples at 460nm, and monitoring the decay of the reduced state at 560nm, the kinetic behaviour of the recombination process can be studied.
3. EXPERIMENTAL AND TECHNIQUES

3.1 The device

The system investigated in these experiments isolates the recombination reaction from the solar cell. The films consist only of a nanoporous nickel(II)oxide layer, sensitised by a ruthenium based dye on conductive FTO (Fluorine-doped tin oxide) glass in a lithium perchlorate solution. It should be noted that the lithium perchlorate solution does not contain a redox couple, and that its purpose is only to stabilise the negatively charged reduced state of the dye. Figure 3 shows the sensitisers (denoted J04 and LD2 respectively) used in the experiments.

![Figure 3: Showing the sensitisers used in the experiments.]

3.2 Flash photolysis

The decay of the reduced state was studied with transient absorbance spectroscopy, using flashphotolysis. Figure 4 shows a typical setup for this technique. A Nd-Yag laser tuned to a specific wavelength with the OPO is used to excite the sample in the sample chamber. The sample is then flashed by a high pressure Xenon lamp and the change in absorbance of a certain wavelength (set by the monochromator) is monitored by the PMT over time after the laser flash.

All samples were excited with light at 460nm. Steady state spectra were taken to determine the absorbance of the adsorbed sensitisier at this wavelength. Before each experiment, the energy of the laser beam was measured. The average energy per shot and its standard deviation were calculated. As the beam diameter could not be measured accurately, and due to the inhomogeneous light distribution over the beam, the exact energy of the fraction of the laser beam exciting the sample cannot be known. However, the experiments allow for comparison between measurements conducted with high and low intensity settings, respectively (see section 4).

The intensities were lowered by placing partially transparent filters in front of the sample. Additionally, a filter absorbing below 400nm was placed between the Xenon lamp and the sample,
and a filter absorbing below 495nm was placed between the sample and the PMT to avoid detecting scatter from the laser. The monochromator was set to 560nm with a bandwidth of 5nm, as the reduced state was previously shown to have an absorbance maximum in this region.\textsuperscript{[10]} All measurements were made averaging multiple laser shots firing at 1 Hz (shot per second).

### 3.3 Sample preparation

The NiO films were prepared through spreading a NiCl\textsubscript{2} solution in 99.5% ethanol on FTO glass, using the doctor blading technique. The films were then heated in a furnace to 450° C for 30 minutes and allowed to cool down to room temperature. The glass was then cut and the film removed from the edges, leaving a surface of 0.8cm *1.6cm. The film was sensitised in a saturated solution of the sensitiser in ethanol. The cells were sealed with a pre-drilled cover glass containing two holes using thermoplastic surlyn. 100mM Lithium perchlorate in propylene carbonate was injected through the holes, which were then sealed with thermoplastic surlyn and a microscope cover slip. Table 1 shows preparation conditions and properties of each sample.

<table>
<thead>
<tr>
<th>Sample</th>
<th>J1</th>
<th>J2</th>
<th>J3</th>
<th>J4</th>
<th>L1</th>
<th>L2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dye</td>
<td>J04</td>
<td>J04</td>
<td>J04</td>
<td>J04</td>
<td>LD2</td>
<td>LD2</td>
</tr>
<tr>
<td>Loading Time</td>
<td>24 h</td>
<td>72 h</td>
<td>72 h</td>
<td>50 h</td>
<td>50 h</td>
<td>50 h</td>
</tr>
<tr>
<td>Steady State Absorbance At 460nm</td>
<td>0.18</td>
<td>NA</td>
<td>NA</td>
<td>0.20</td>
<td>0.24</td>
<td>0.14</td>
</tr>
</tbody>
</table>

\textit{Table 1: Properties of each sample used in the experiments.}
4. RESULTS AND DISCUSSION

Tables 1 and 2 show the conditions under which each kinetic trace was taken in detail. The graphical data is added in Appendix I.

A first order decay mechanism (as for example oxidation of the reduced sensitiser ion in solution can be expected to be) follows exponential decay of the form:

\[
[M] = [M]_0 \exp(-\frac{t}{\tau})
\]

Where \([M]\) denotes the concentration of the compound, \([M]_0\) denotes the initial concentration, \(t\) denotes the time after the start of the reaction, and \(\tau\) denotes the compound’s lifetime. Similarly (as the absorbance of a compound is proportional to its concentration according to Beer’s law) a transient absorbance signal would take the form:

\[
S = A \exp\left(-\frac{t-t_0}{\tau}\right)
\]

Where \(S\) is the signal strength, \(A\) is a constant governing the intensity of the signal and \(t_0\) is the time-offset marking the start of the decay.

As the oxidation of the reduced sensitiser doesn’t take place “freely” in solution, but on the nickel oxide surface, it doesn’t follow simple monoexponential decay. This can be attributed to many factors, such as (among others) the different ways of adsorption to the surface, and the many states of (and above) the nickel oxide valence band. Recombination to each of these states should in theory have a different lifetime. To model the kinetic behaviour, it is possible to fit the empirical data to a sum of exponentials of the form:

\[
S = A_1 \exp\left(-\frac{t-t_0}{\tau_1}\right) + A_2 \exp\left(-\frac{t-t_0}{\tau_2}\right) + ...
\]

The reaction is thereby separated into empirical phases, each given a separate lifetime. The constants denoted \(A_i\) can in this case be seen as weighing constants, as the respective ratios between these determine the contribution of each phase to the total signal. Note that each component does not represent an actual transition to an energy state in the conduction band. The fit is merely employed to make comparison between samples possible, and the components are not ascribed deeper meaning.

In order to compare the signals obtained with low and high illumination intensities, respectively, the signals were fitted to sums of exponentials as described above. As the measurements taken do not contain the whole decay signal, an additional fitting parameter accounting for the \(y\)-offset (not
present in table 2) had to be introduced. Table 2 gives the fitting constants obtained for each measurement. The functions are also graphically represented in appendix I.

As seen in the table 2, the samples prepared with the sensitisier J04 were not traced over longer timescales than 10µs. This was due to poor signal quality obtained in the measurements.

Looking at the first set of data monitoring the reduced state decay of J04 over 4µs, we can see that each fit follows a similar pattern. The decay can be described as the sum of a fast phase, with a life time ranging from 0.12-0.21 µs and a weight of roughly 30%, and a slower phase contributing roughly 70%. Even though the fits show a slight variation in the coefficients, due to the low resolution of the data, the kinetic behaviour of the decay cannot be shown to be significantly different.

The kinetic trace of J04 over 10µs also shows similar rates among measurements taken with both high and low illumination intensity. Furthermore, the second set of data showing the decay of LD2 over 100µs exhibits similar behaviour between low and high intensity settings. Variation in the lifetime of the long phases when changing light intensity can be seen, but this is ascribed less meaning, as the trace still does not capture the whole decay signal and a y-offset has to be employed in the fit. The traces and their similarities are illustrated in figures 5-7.

![Figure 5: The kinetic traces of sample L1 (LD2) over 100µs at low (blue -) and high (red +) intensities, normalised.](image-url)
Figure 6: The kinetic traces of sample J4 (J04) over 10µs at low (blue -) and high (red +) intensities, normalised.

Figure 7: The kinetic traces of sample J1 (J04) over 4µs at low (blue -) and high (red +) intensities, normalised.

As only a fraction of the laser beam hits the sample, and because the beam is not uniform, it is not possible to know the exact irradiance of the light exciting the sample. In order to optimise the overlap of the laser pump and the probe beam, the sample holder had to be moved when changing samples, thereby possibly changing which part of the laser beam hit the sample. Additionally, the angle orientation of the samples (though attempted to be kept at approximately 45 degrees from both the pump and the probe beam in every experiment) might have varied. This, among many other factors (such as varying film thickness, differing dye loading efficiencies and variations in the concentration of the stabilising electrolyte) makes comparison between different samples difficult. However, the measurements of the same samples under different intensity settings can confidently be compared; the only parameter changed was the power of the laser beam.

It should be noted that the energy per laser shot does not directly correspond to the energy exciting the sample. However, when the mean power of the beam is decreased by a constant factor, it can be assumed that the fraction of the beam hitting the sample exhibits the same behaviour.
Furthermore, during most of the measurements, the energy per laser shot showed significant instability, which can be seen by the high standard deviation calculated for the means. The relatively high resolution of the signal, despite high variation in excitation intensity during the measurements on LD2 might be further indication that the kinetic behaviour of the decay does not change significantly when exciting with intensities in the studied range.

Due to technical problems of the laser system limiting the maximum intensity of the pump pulse, and insufficient dye adsorption on the NiO films the illumination intensity dependence of the decay could not be studied over a broader range in these measurements.

Table 2: Fitting parameters for fits on the kinetic traces. Each trace was fitted to a double exponential. A1 and A2 denote the weight of each exponential (normalised to 1). Tau1 and tau2 are the respective lifetimes. The average illumination power given by the laser, and the standard deviation of the power are also shown.

<table>
<thead>
<tr>
<th>Sensitiser</th>
<th>J04</th>
<th>LD2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scale</td>
<td>4 µs</td>
<td>10 µs</td>
</tr>
<tr>
<td>Sample</td>
<td>J1</td>
<td>J2</td>
</tr>
<tr>
<td>High Intensity Measurements:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fitting coefficients: A1</td>
<td>0.36</td>
<td>0.29</td>
</tr>
<tr>
<td>tau1</td>
<td>0.21 µs</td>
<td>0.16 µs</td>
</tr>
<tr>
<td>A2</td>
<td>0.64</td>
<td>0.71</td>
</tr>
<tr>
<td>tau2</td>
<td>1.9 µs</td>
<td>2.5 µs</td>
</tr>
<tr>
<td>Average Power:</td>
<td>12.3 mW</td>
<td>12.3 mW</td>
</tr>
<tr>
<td>Standard deviation</td>
<td>1.46 mW</td>
<td>1.46 mW</td>
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<tr>
<td>Low Intensity Measurements:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fitting coefficients: A1</td>
<td>0.28</td>
<td>0.32</td>
</tr>
<tr>
<td>tau1</td>
<td>0.12 µs</td>
<td>0.23 µs</td>
</tr>
<tr>
<td>A2</td>
<td>0.72</td>
<td>0.68</td>
</tr>
<tr>
<td>tau2</td>
<td>6.9 µs</td>
<td>4.5 µs</td>
</tr>
<tr>
<td>Average power:</td>
<td>3.97 mW</td>
<td>3.97 mW</td>
</tr>
<tr>
<td>standard deviation</td>
<td>0.874 mW</td>
<td>0.874 mW</td>
</tr>
</tbody>
</table>

15
5. CONCLUSIONS

Despite the noisy data obtained, laser flash photolysis is a promising way of investigating this system. The data obtained does not suggest any intensity dependent mechanism changing the kinetics of the recombination reaction in the range investigated. It could however be of interest to repeat these experiments studying a wider range of illumination intensities.

It should also be noted that complete devices will have a redox mediator present in the electrolyte, further changing the kinetics of the processes in the cell. The redox shuttle was left out during these experiments, in order to be able to isolate the recombination process with transient absorbance spectroscopy. Monitoring a cell containing a working redox couple the same way would not give as much information on the recombination reaction, as regeneration and intermission reactions are also present.

However, illumination intensity dependent mechanisms in a p-DSSC could also involve the redox couple in the electrolyte. Investigating other properties, such as the Voc, in a complete cell, while varying illumination intensity could give additional information on the behaviour of the recombination reaction (as well as other mechanisms in the cell).

Similar studies have been conducted on n-DSSCs, resulting in a model describing the dependence of the Voc on the illumination intensity in terms of recombination rates for electrons in the conduction band and occupying trap states in the titanium dioxide bandgap (note that the dominant recombination mechanism in n-DSSCs is the interception of injected electrons with the redox mediator). [11]

Additional information could also be gained by testing the temperature dependence of the reactions present in the cell.
APPENDIX I

The data obtained in the experiments and graphical illustrations of the fits are presented here. For further information and fitting coefficients, see Table 2. The traces on the left were taken under high intensity settings, and the traces on the right show the data obtained with lowered illumination intensity. The x scale denotes the time in microseconds. The y scale is given in arbitrary units.

Sample J1

Sample J2

Sample J3
Sample J4

Sample L1

Sample L2

High intensity

Low intensity
REFERENCES


