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# Procedures preparatory to setting up a luminescence pulsing system

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**Abstract**
Some key tests necessary in the setting up of a luminescence pulsing system are presented. The procedures discussed are concerned with measurement of reliable luminescence-photon counting rates whose analyses provide information about lifetimes and intensity of luminescence measured using pulsed optical stimulation. The paper presents data from a measurement system which used a time-to-amplitude converter for timing.

**Introduction**
The aim of pulsed optical stimulation is to separate in time the stimulation and emission of luminescence. Pulsed optical stimulation produces a time resolved set of signals displaying the delay between stimulation and emission of luminescence. Time-resolved spectra measured in this way may be analysed for associated lifetimes in order to improve understanding of the dynamics that underlie the emission of the luminescence.

The use of luminescence pulsing techniques has been reported previously including ones based on the use of lasers (Akselrod and McKeever, 1999; Clark et al., 1997; Markey et al., 1995; Sanderson and Clark, 1994) and on light-emitting-diodes (e.g Chithambo and Galloway, 2000). The operation of the systems varied with some laser-based systems using pulsed-lasers (Clark et al., 1997; Sanderson and Clark, 1994) and others mechanical shutters to intercept the laser light and thus provide the stimulation pulse (Markey et al., 1995). In the measurement system of Chithambo and Galloway (2000), the light-emitting-diode pulses were triggered by signals from an integrated circuit multivibrator, a 2N74221. Concerning luminescence detection, the system described by Markey et al. (1995) synchronised the operation of the mechanical shutter with the operation of a gated photon counter whereas the pulsed laser systems of Clark et al. (1997) as well as that of Sanderson and Clark (1994) used a multichannel scaler to record the luminescence-photon counting rate as a function of time.

This paper presents some key tests involved in the setting up of a luminescence pulsing system for routine use. The LED-based pulsing system of Chithambo and Galloway (2000) is used as a case study. This measurement system used a combination of a time-to-pulse-height converter and an analogue-to-digital converter for photon counting and timing of the delay between stimulation and emission of luminescence. The procedures described were performed to ensure measurement of reliable luminescence-photon counting rates whose analyses provide information about luminescence lifetimes and luminescence intensity of the material under study. The preparatory tests discussed may be of interest to others planning to set up or develop a luminescence pulsing system.

**Signal detection**
Figure 1 shows a schematic diagram of the luminescence measurement assembly. The luminescence photons are detected by a photomultiplier (EMI 9635QA). The signals from the photomultiplier are then amplified and counted in turn by the amplifier (Ortec 474) and constant fraction discriminator (Ortec 473A) respectively. The time-to-pulse height converter (TPHC) (Ortec 467) is used to determine the delay between stimulation and emission of luminescence. This is because in practice the TPHC generates a rectangular output pulse whose amplitude is linearly proportional to the time interval between the arrival of a START and STOP signal to the inputs of the converter. The output pulses from the TPHC are fed into the analogue-to-digital converter (LABEN 8213) which then furnishes the signals, in digital form, to the computer to produce a plot of luminescence-photon counts against time, a time-resolved spectrum. Luminescence time-resolved spectra are displayed with a time range equal to the selected dynamic range on the TPHC.

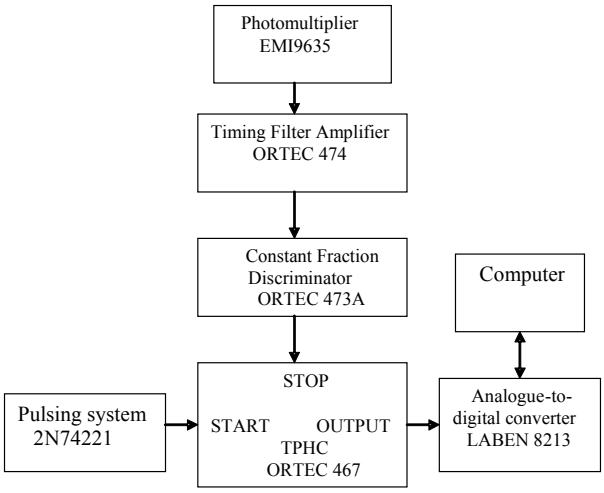


**Figure 1:** *A block diagram of the luminescence measurement assembly. The time-to-pulse-height converter (TPHC) measures the time interval between stimulation and emission of luminescence .*

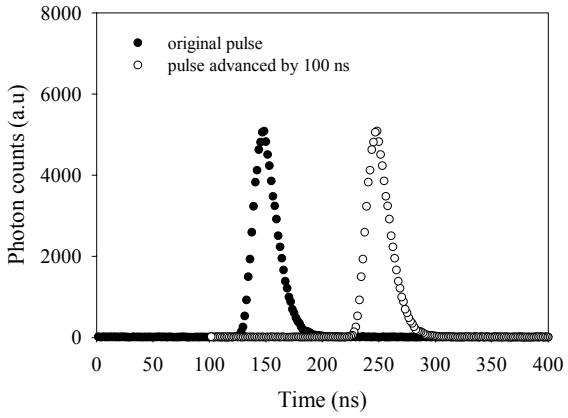


**Figure 2:** *A light-emitting-diode pulse shifted from its original position by the introduction of a 100 ns delay to the STOP signal.*

**Timing**

The time-to-pulse amplitude conversion mentioned above can only be performed if a valid START input signal (determined by threshold input voltage) is accepted within the selected time range. The START input is then disabled during this `busy' interval. The acceptance of a valid STOP pulse (again determined by threshold input voltage) indicates that a time interval has been measured and its corresponding analogue signal can then be read at the TPHC output. Thus the acceptance of a START signal is necessary to initiate a response in the TPHC. In the measurement system of Figure 1, the START signal, generated by the 2N74221 multivibrator was fed into the TPHC at a repetition rate of 11 kHz whereas the STOP signals, provided by random luminescence-photons from a quartz sample under stimulation, were obtained from the ORTEC model 473A constant fraction discriminator (Chithambo and Galloway, 2000).

In order to calibrate the time axis and hence determine the resolution of the dynamic range selected, a time-resolved spectrum of a light-emitting-diode (LED) pulse was first obtained with the photomultiplier preceded by only neutral density filters. A delay, $\Delta t$, was then introduced to the STOP pulse by connecting a calibrated delay cable between the TPHC and the constant fraction discriminator. When the system was operated again, the LED time-resolved spectrum shifted by a time $\Delta t$ from its original position. This is shown in Figure 2 for a delay of 100 ns. Since the dynamic range of the time-resolved spectrum was selected on the TPHC beforehand, calculation of the resolution of the time axis was made straightforward.

**Measurement of luminescence time-resolved spectra**

The luminescence-measurement system of Figure 1 requires that a maximum of one photon be detected for each stimulation light pulse (i.e. for each START pulse). The luminescence-photon signals provide the STOP pulses. The arrival of the first STOP pulse after a START signal will initiate timing in the TPHC. If multiple STOP pulses arrive after a START pulse, the time information recorded will be that of the first STOP pulse. The time information of subsequent pulses will not be recorded and as a result the time-resolved spectrum will be distorted, with later times being falsely low in recorded counts. It is imperative then that for undistorted time-spectra, the STOP pulse rate should be less than the START pulse rate i.e. the luminescence-photon counting rate should be less than the stimulation light pulse rate (for example see figure 3 in Chithambo and Galloway, 2000). Time-resolved spectra with an inherent distortion can lead to apparent but spurious lifetime values.

**Assessment of counting rates**

In order to establish `safe' levels of counting rates for measurement of time-resolved spectra, the set-up shown in Figure 1 was used to compare the rate of STOP pulses measured simultaneously by a scaler counter and by the TPHC. The number of STOP pulses measured by the TPHC in a given time was determined by integrating the measured time-resolved spectrum over its entire 256 channels. The

rate of STOP pulses was changed over time by simply measuring time-resolved spectra repeatedly from a sample. As the luminescence from the sample decreased in time, so did the rate of STOP pulses. However, the frequency of the START pulse was kept constant at 11 kHz. Figure 3 compares counting rates recorded by the scaler with rates determined from time-resolved spectra for stimulation using green LEDs (Nichia NSPG 500). In the absence of any dead-time in the TPHC, these counts would be equal. However, because there is a finite dead-time in the TPHC, the counts integrated from time-resolved spectra are lower than counts recorded by the scaler. The non-linearity from about 8000 counts $s^{-1}$ (y-axis) occurs because the next and later STOP pulses are being missed by the TPHC. Time-resolved spectra in this region would be extremely distorted as counts would be registered only in the first 2 or 3 channels of the analogue-to-digital converter. Lifetimes calculated from such time-resolved spectra are inevitably spurious. A `safe' working region will therefore be where the count rates integrated from time-resolved spectra are linearly proportional to count rates from the scaler, for instance the region at less than 5000 counts $s^{-1}$ (y-axis).

A further experiment was conducted to see if the performance of the system depends on the rate at which signals are presented to it. This was done by changing the LED current from 10 to 30 mA thereby increasing the light output. Figure 4 shows that, so long as the count rate stays within the "safe" working region, as indicated by the rate of scaler pulses, the performance of the system does not depend on the intensity of the light. This means that time-resolved spectra measured at different light intensities can be compared directly. Beyond the "safe" region, this is not necessarily true as can be seen in Figure 4. The scatter of data points in time-resolved spectra due to variation of intensity is reflected in the errors in lifetimes derived from it (Bailiff, 2000).

The relationship between spurious lifetimes (i.e. 'spurious' according to the criteria discussed above) and counting rates as investigated by Chithambo and Galloway (2000) implies that when measuring a lifetime, the counting rates should be chosen such that the possible spurious lifetime is much longer than the lifetime being measured. It should be noted that even though incidents of spurious lifetimes are more likely in a set up using a TPHC in which 'dead time' effects are inherent, changes in lifetime with counting rates have been reported even for multichannel scalers with multiple-STOP-signal capability (Galloway, 2002). The tests discussed in this paper are therefore necessary in any system intended for measurement of luminescence lifetimes.

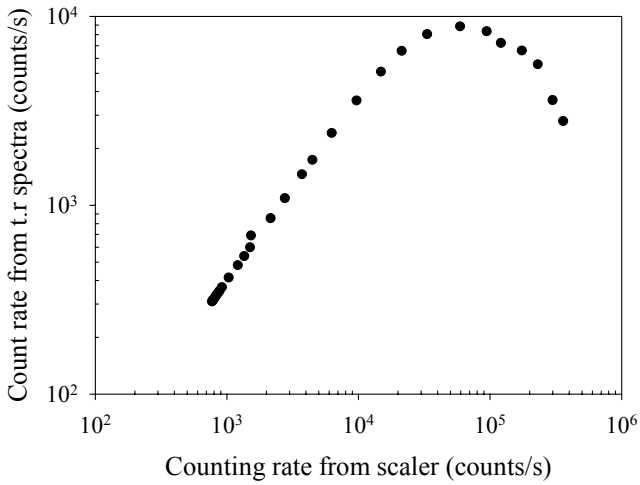


**Figure 3:** *A comparison of counting rates recorded by a scaler counter with rates determined from time-resolved (t.r.) spectra.*

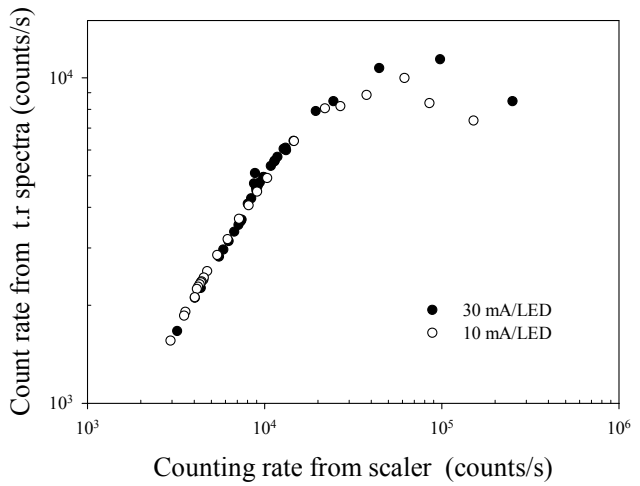


**Figure 4:** *A comparison of counting rates obtained at 10 and 30 mA per LED.*

**Conclusions**

Some key tests involved in setting up a luminescence pulsing system for measurement of luminescence lifetimes have been presented. The discussion used an LED-based pulsing system in which a time-to-amplitude converter is used for timing. The preparatory tests discussed may be of interest to others planning to set up or develop systems for measurement of time-resolved spectra.

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**Reviewer**

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