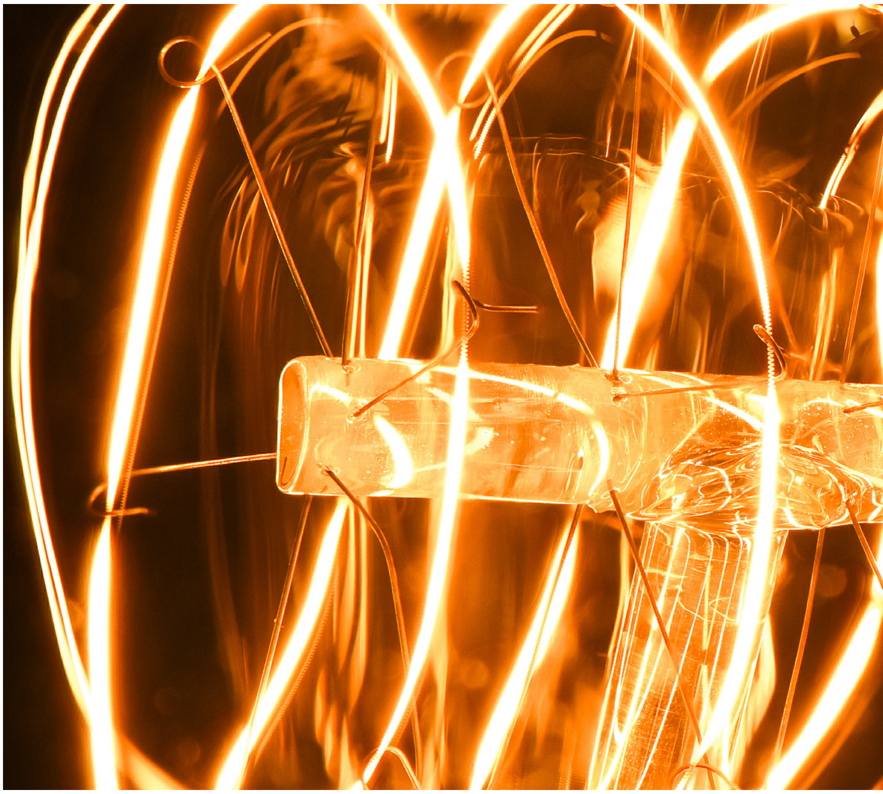

APPLICATION NOTE

Fluorescence Spectrometer

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Measurement of Delayed Fluorescence and Phosphorescence Lifetimes on the FL6500 Steady-State Fluorescence Spectrometer

Introduction

The FL6500 is a steady state fluorescence spectrometer which uses a pulsed xenon flash light source. Although classed as a steady state instrument (i.e. not a lifetime measuring instrument in terms of "prompt" singlet fluorescence), it is extremely good at measuring slower lifetimes which involve a symmetry forbidden triplet state as part of their overall excitation and emission pathway.

The FL6500 uses a pulsed xenon flashlamp as its light source. This is the latest iteration on a system that was developed by PerkinElmer in conjunction with the UK Royal Institution by the late Prof. Mike West and was used on the PerkinElmer Model 1000 fluorescence spectrometer in 1975.

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These lamps we designed in order to protect light-sensitive compounds and also have a very long service life – usually over several years. The pulsing and detection parameters are controlled entirely electronically and so it is possible to get very precise control over these parameters. In particular, it is possible to set the detection window with a time-incremented offset, such that it can measure between the lamp pulses in order to detect delayed fluorescence (as encountered with certain lanthanides such as europium, terbium, dysprosium and samarium) as well as phosphorescence. Any prompt (singlet, symmetry-allowed) fluorescence and scatter artefacts are rejected as they are only present during the period of the flash envelope.

The pulsed lamp can be controlled by the instrument's electronics in order to control and progressively increment the delay time (shown in Figure 1 as $\mathbf{t}_d$) for the onset of this detection window (gate time, $\mathbf{t}_g$).

The FL6500 is able to make a series of rapid measurements with progressively incremented delay times. This data is constructed in real time as a decay curve as shown in figure 2. For longer lived phosphors, it is possible to irradiate the sample and then follow the decay curve when the lamp is switched off.

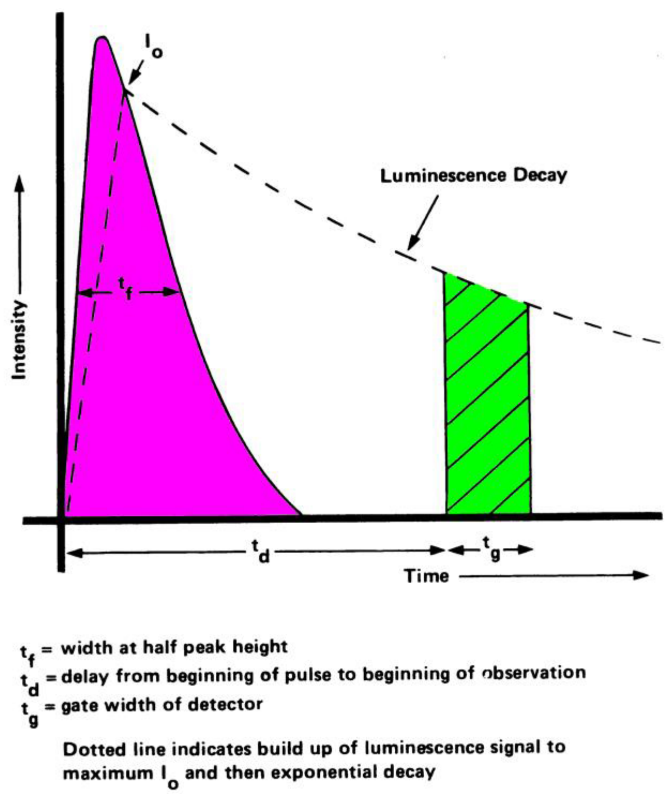

Figure 1. Offset of the detection window using a pulsed xenon lamp.

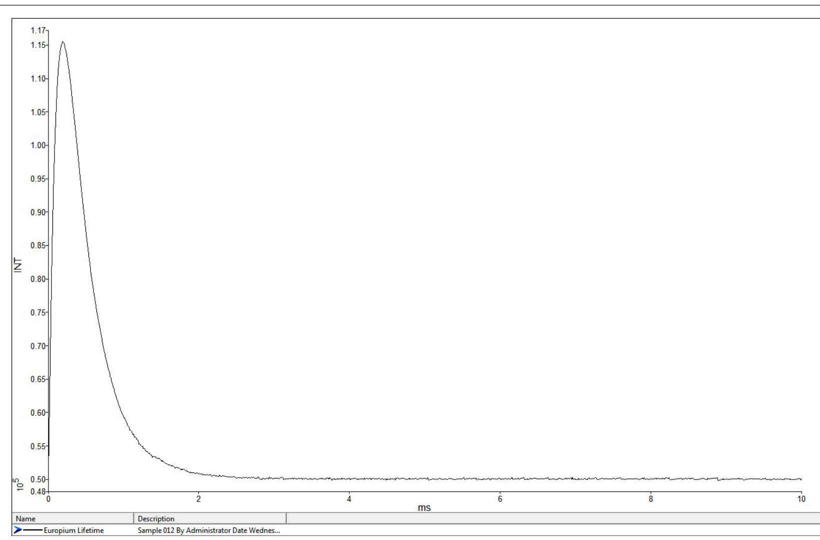

Figure 2. Measurement of the decay curve of europium.

Collecting the Lifetime Data

Block P1 (a PMMA block doped with europium) from the set of six fluorescent materials (part number 52019600) was used.

The Lifetime mode was selected and the settings are shown in Table 1.

Table 1. FL6500 Instrument Settings for the Measurement of Europium.

Instrument Settings	
Data Mode	Phosphorescence (Short)
Excitation Correction	On
Source Mode	Pulse
Flash Count	1
Flash Power (kW)	20
Frequency (Hz)	100
Excitation Wavelength (nm)	350
Excitation Slit (nm)	10
Excitation Filter	Air
Emission Wavelength (nm)	613
Emission Slit (nm)	10
Emission Filter	Air
PMT Voltage (V)	550 (Medium)
PMT Gain	X8
Emission Correction	On
Delay Time (μs)	5
Accessory Type	Single Cell

Data Analysis

The Spectrum FL software (as shown in Figure 3) has data processing algorithms for general spectral manipulation but there are some excellent third party programs which are dedicated specifically to decay analysis such as DecayFit available for download from www.fluortools.com. At the time of writing, this program is available to researchers free of charge. Data can be exported from Spectrum FL as an Excel .csv (comma separated variable) file for further manipulation.

Using the advanced exponential curve fitting tools, it is possible to generate an exponential curve fit such as the example shown in Figure 4.

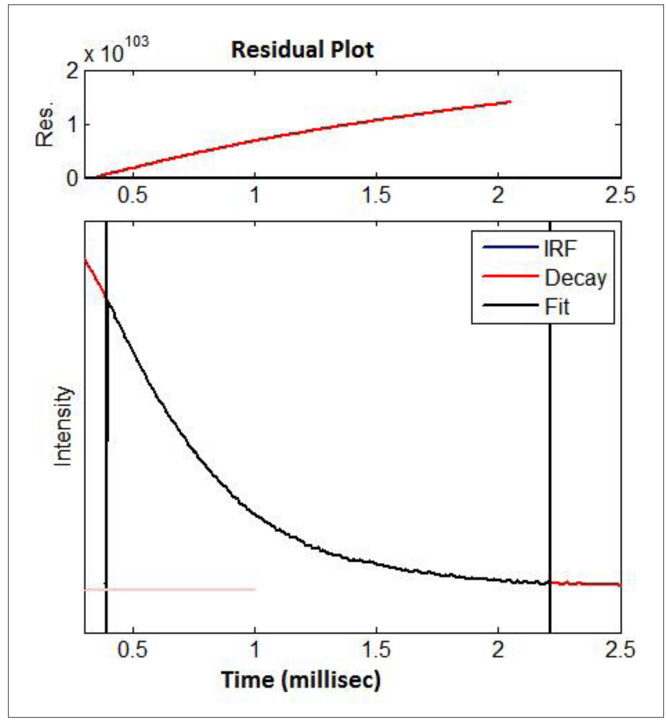

Figure 4. Double exponential plot of europium delayed fluorescence decay showing residuals.

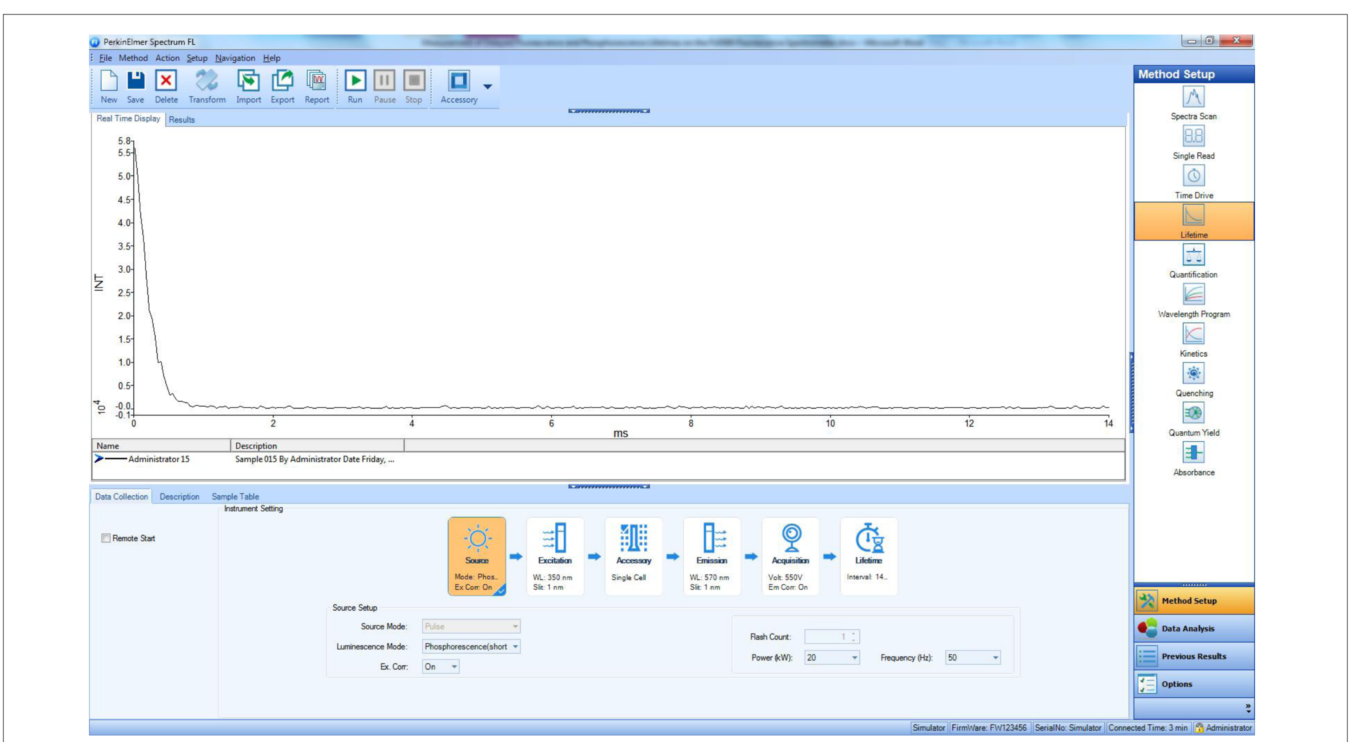

Figure 3. Spectrum FL Lifetime Module.

Delayed fluorescence and phosphorescence measurements are also possible on the FL8500 fluorescence spectrometer. This instrument uses a conventional 150W continuous xenon lamp. In this case, the phosphorescence parameters are set mechanically by the rotating chopper instead of the pulsed light source but they can give similar results to the pulsed source instrument (Figure 5).

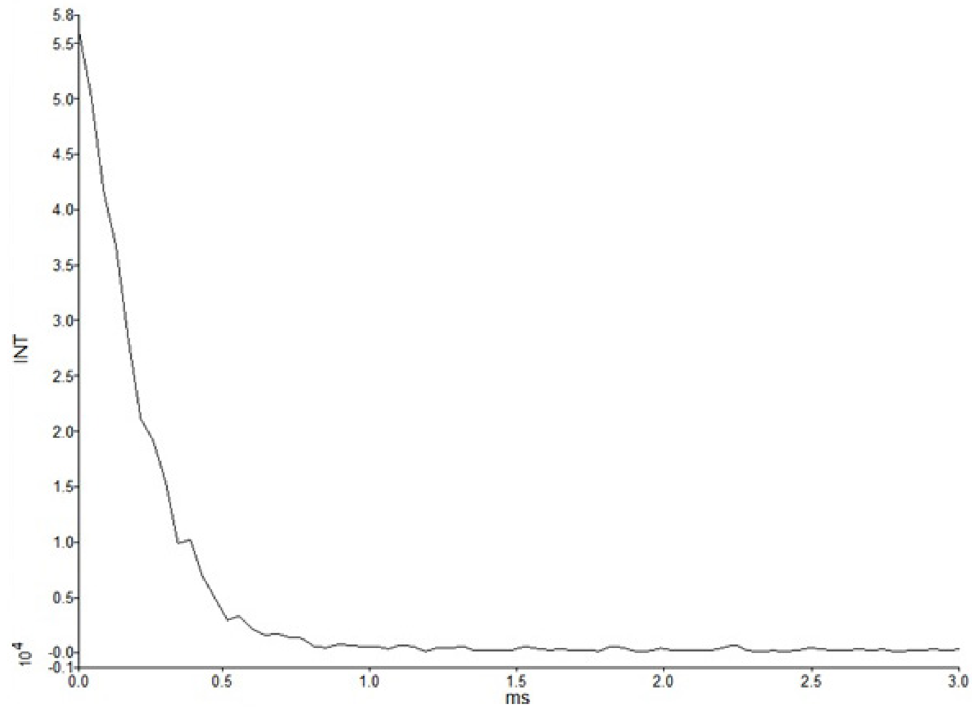

Figure 5. Delayed fluorescence Decay Curve FL8500.

Conclusion

Both the FL6500 and FL8500 fluorescence spectrometers are suitable for measuring delayed fluorescence and phosphorescence lifetimes. The pulsed xenon lamp of the FL6500 gives excellent control over the detection parameters and makes it the ideal choice for this application. For phosphorescence applications which are often conducted at low temperatures in order to reduce inter-molecular collisions which would result in energy being lost to non-radiative pathways and thus degrade the luminescence, a cryostat accessory is available as an accessory option.

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