

EDINBURGH INSTRUMENTS

APPLICATIONS

FLS1000



FLS1000 A NEW ERA IN

PHOTOLUMINESCENCE

Faster Measurements

New Software

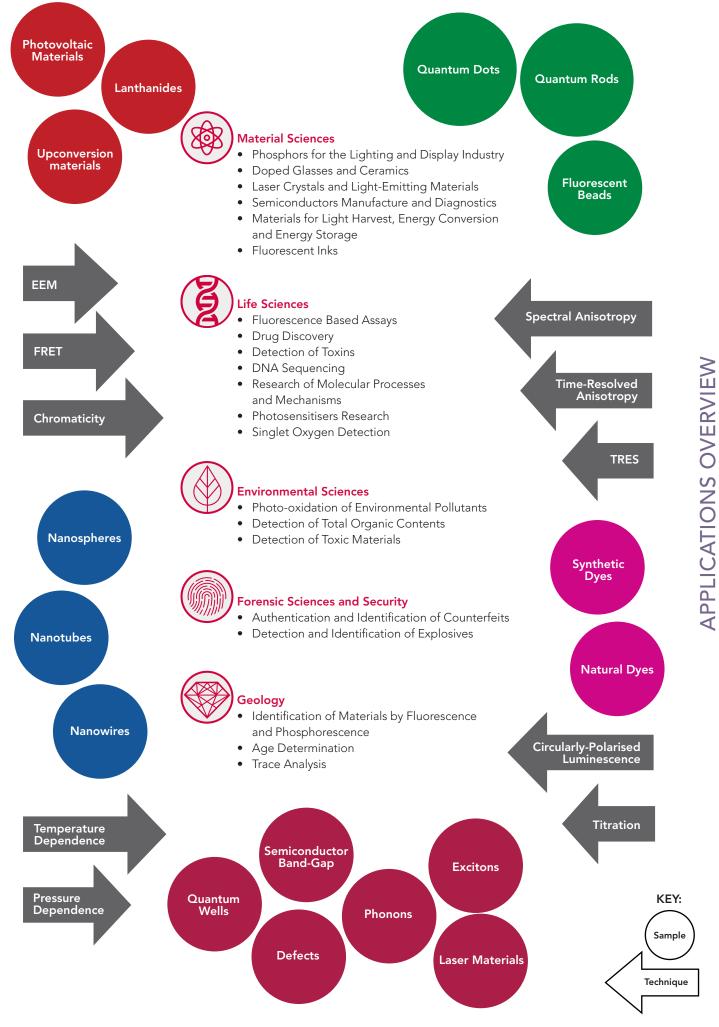


The FLS1000 is a state-of-the-art, modular photoluminescence spectrometer for the most demanding applications in Photophysics, Photochemistry, Material Sciences and Life Sciences.

The instrument sets the standards in both steady state and time-resolved spectroscopy: The system demonstrates unmatched sensitivity and can be configured for spectral measurements from the ultraviolet to the mid-infrared spectral range, and for lifetime measurements spanning time resolutions over 12 orders of magnitude from picoseconds to seconds.

STEADY STATE AND TIME-RESOLVED PHOTOLUMINESCENCE

- > Modular construction for maximum flexibility and upgradability
- > Industry leading sensitivity specification >35,000:1 (SQRT Method)
- Unrivalled spectral coverage from the deep UV to the MIR, 185 nm up to 5,500 nm
- Unmatched monochromator performance with "plug and play" triple-grating turrets, integrated filter wheel, 325 mm focal length and excellent stray light rejection
- Multiple light sources, detectors, single or double monochromators available
- Intuitive Fluoracle[®] software for all steady state and time-resolved measurements with standard and advanced data analysis options



Request application notes or test measurements by contacting applications@edinst.com

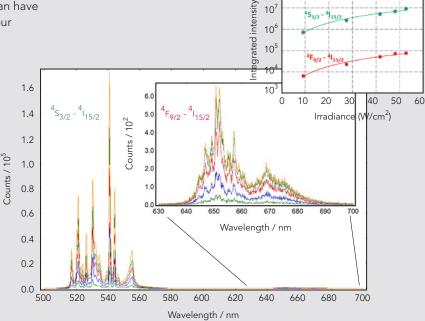


PHOTOLUMINESCENCE OF LANTHANIDES - UPCONVERSION

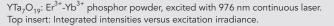
Lanthanide materials have wide applications in the lighting industry, as laser gain media, in solar cells, as fluorescent labels, and in the life science industry as sensor materials.

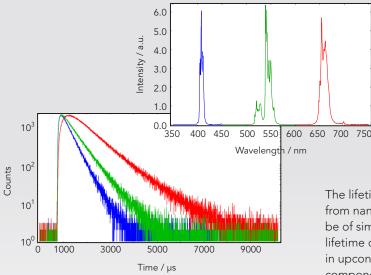
The electronic structure of lanthanide materials causes bright emission that often can have narrow-band structure and can favour non-linear upconversion processes.

The FLS1000 offers exceptional high spectral resolution, provides stray light suppression to discriminate between weak emission and scatter from the white powder sample, and has a high dynamic range to detect spectral bands that can have dramatically different amplitude. Upgraded with a 980 nm continuous laser and thanks to a built-in computercontrolled continuous attenuator, the FLS1000 is ideal for upconversion power density experiments.



10⁸





 $NaYF_4:Er^{3+}-Yb^{3+}$ phosphor powder, excited with a 976 nm microsecond pulsed laser and measured with MCS. Insert: emission spectra of the three bands chosen

for lifetime measurements.

The lifetimes of the excited lanthanide states can vary from nanoseconds to seconds. The decay kinetics may be of simple exponential nature or can exhibit complex lifetime distributions. The individual emission bands in upconversion materials often show rising components too.

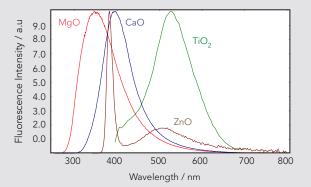
The FLS1000 can operate diode and solid state lasers in pulsed mode.

The graph contains lifetime measurements of the three main emission bands of the upconverted signal.

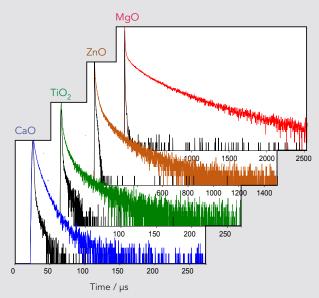
EMISSION OF SEMICONDUCTOR MATERIALS AT ROOM TEMPERATURE

A large variety of inorganic materials show semiconductor properties. The emission from these materials at ambient temperatures can be strong or extremely weak, depending on the physics of inter-band recombination, the presence of defects and impurities.

Spectral and time-resolved photoluminescence properties of these materials reveal fundamental information about their structure and can also be used for guality control and process monitoring.

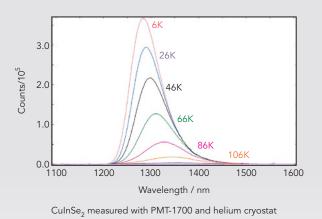


Often these materials come as white powders and a clear separation of the potentially weak emission from unwanted scatter may be challenging. Double monochromators or lifetime measurements are effective in discriminating from scattering artefacts.



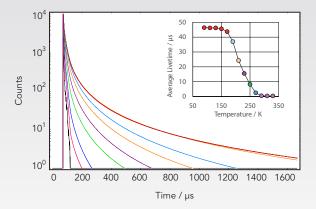
Spectral and lifetime measurements of some popular oxides at room temperature

EMISSION OF SEMICONDUCTOR MATERIALS AT CRYOGENIC TEMPERATURES



The $CulnSe_2$ graph shows a spectral temperature map as measured with a helium cryostat.

The GaN graph shows the results of a lifetime temperature map measured with a nitrogen cryostat, with all decay measurements analysed using the stretched exponential function of the advanced lifetime analysis package. Many semiconductor materials show very weak emission at room temperature. Different cryostat options are offered to enable measurements down to <4 K. Liquid nitrogen, liquid helium or closed cycle helium cryostats can be directly integrated into the FLS1000 sample chamber, without additional fibre optics. Many of the cryostat options are fully controlled by the Fluoracle software, enabling automated spectral and lifetime temperature maps to be performed.

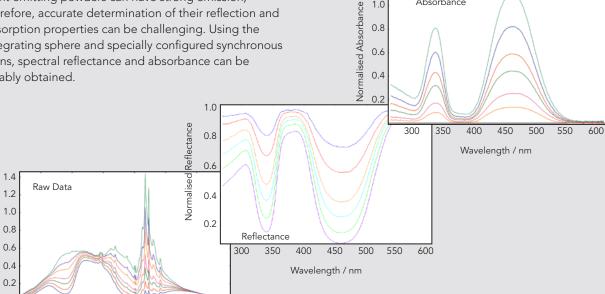


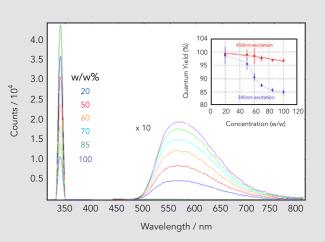
GaN thin film lifetime measurements aquired with liquid nitrogen cryostat in MCS mode. The graph shows the fitted curves after advanced lifetime analysis.

emiconducto <u>Ba</u>nd-Gap



Light emitting powders can have strong emission; therefore, accurate determination of their reflection and absorption properties can be challenging. Using the integrating sphere and specially configured synchronous scans, spectral reflectance and absorbance can be reliably obtained.





450

Wavelength / nm

500

550

600

YAG:Ce phosphor powder in BaSO₄ at various concentrations.

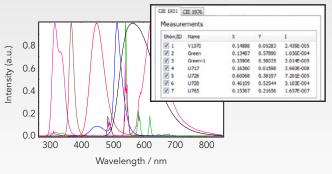
Absorbance

1.0

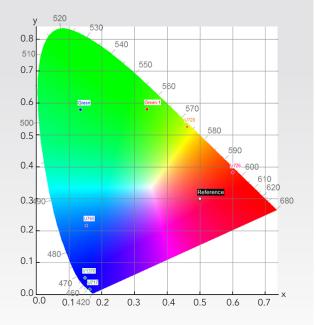
Measurements of absolute photoluminescence quantum yield require high precision in the measurement of both weak and strong signals, clear and established measurements procedures, and accurate correction factors for the whole light path including the integrating sphere, all provided by the FLS1000.

The high dynamic range of our cooled detectors, our easy-to-operate measurement wizard, and the FLS1000 sphere as an integrated part of the sample chamber, will enable you to perform reliable measurements of absolute photoluminescence quantum yields.

The Fluoracle software's chromaticity wizard provides colour coordinates and luminous intensity values. Fluoracle can display CIE 1931 and CIE 1976 colour coordinates. You can set reference values and compare a large number of spectral scans in those coordinates.



Various luminescent phosphor powders measured with integrating sphere.



Counts / 10⁶

300

350

400

anthanide

CARBON NANOTUBES

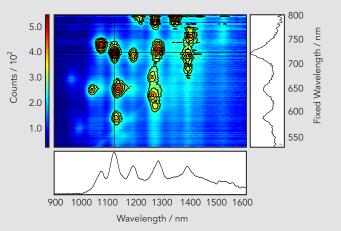
Nanoscale materials such as quantum dots, carbon nanotubes, nanoparticles and nanostructures exhibit strong spatial confinement upon photo-excitation and tuneable emission. This makes them excellent candidates in a multitude of applications from photonic devices to sensing and biology. The structural characteristics of single-walled and multiwalled carbon nanotubes can be identified from excitation-emission mapping, as shown in the graph.

The mapping function in the Fluoracle software, together with the instrument's unsurpassed resolution and NIR spectral coverage, make the FLS1000 an excellent tool to study these materials.

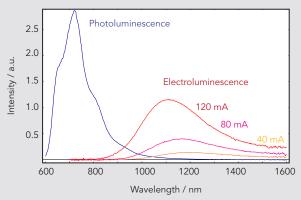
POLYMER SOLAR CELL MATERIALS

As well as their use in organic light emitting devices (OLEDs), organic polymer materials are also potential candidates as easy to operate and inexpensive photovoltaic solar cell materials.

The FLS1000 features an optional electroluminescence sample holder that can be easily exchanged with the standard photoluminescence holder. Both photoluminescence and electroluminescence can be used as diagnostic tools for the characterisation of the electronic band-gap and the charge-transfer states in these organic photovoltaic cells.



Single-walled carbon nanotubes detected with NIR-PMT

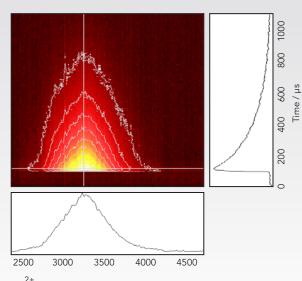


Wavelength / nm Photoluminescence and electroluminescence for three different currents

of a P3HT:PCBM thin film device, $\lambda_{_{exc}}$ = 550 nm for photoluminescence.

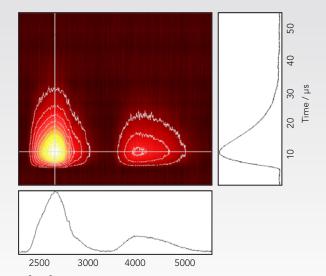
MID-IR LASER MATERIALS

The emission of novel laser materials up to $5.5 \ \mu m$ can be measured using InSb detectors. To discriminate effectively from black body radiation time-resolved



 Co^{2+} doped ZnSe crystal, measured with Nd:YAG pumped OPO and InSb detector, λ_{exc} = 808 nm.

measurements are advantageous, although phase sensitive (lock-in) technology is also available.



 ${\rm Cr}^{2+}{\rm Fe}^{2+}$ doped ZnSe crystal, measured with Nd:YAG laser and InSb detector, $\lambda_{exc}{=}$ 532 nm.

Nanotube

Photovoltai

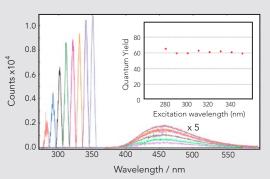
Materials



ABSOLUTE QUANTUM YIELD MEASUREMENTS OF ORGANIC DYES

The absolute method for fluorescence quantum yield (QY) measurements is now more widely used than the relative method. It does not require a QY standard and readily applicable to liquids, films and powders and can be extended into the near-infrared spectral range.

The figure shows the independence of the fluorescence QY from the wavelength of excitation for a standard organic dye. The areas of absorption for eight different excitation wavelengths and the corresponding emission spectra, scaled by a factor of 5, are shown. The inset shows the calculated quantum yields.



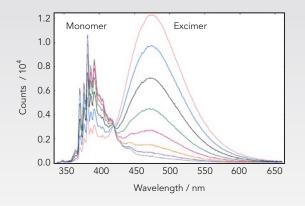
Quinine bisulphate in perchloric acid, measured using the integrating sphere.

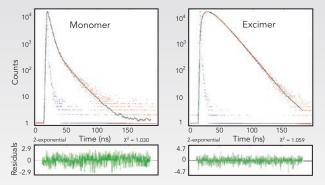


Integrating sphere accessory for the FLS1000.

MONOMER-EXCIMER EQUILIBRIUM AND KINETICS

The monomer or excimer states of organic molecules can be easily distinguished by the balance of their fluorescence. Care should be taken to the inner filter effect due to their high extinction coefficient. The emission spectrum of pyrene in cyclohexane is shown as a function of pyrene concentration. At the lowest concentrations only the monomer bands in the wavelength range 370 nm – 400 nm are observed, while at the highest concentrations the excimer peak at ~480 nm dominates. While many fluorescence lifetime measurements can be described by single or multiple exponential decays, often formation or growth kinetics can be observed too. The example below shows monomer-excimer kinetics of pyrene, with the monomer showing a double exponential decay, while the excimer displays an exponential rise, followed by an exponential decay. Rise and decay times are often linked. Global analysis of the lifetime data with linked parameters is possible using the advanced data analysis package. The global fitting, results in a double exponential with $\tau_{\rm mon}$ = 9.3 ns and $\tau_{\rm excimer}$ = 15.4 ns.





Pyrene in cyclohexane at different sample concentrations, monomer and excimer spectra excited at 335 nm.

Lifetime measurements of pyrene monomers (λ_{em} = 395 nm) and excimers (λ_{em} = 465 nm), excited at 335 nm using the nF920 nanosecond flashlamp.

SOLVENT RELAXATION DYNAMICS OF ORGANIC DYES

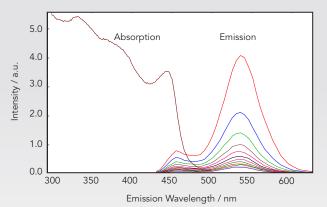
Fluorescence kinetics can be complex for homogeneous dye solutions in polar solvents, even if rotational diffusion is eliminated with magic angle conditions.

The measurement shows 16 fluorescence decays in a polar-viscous solvent of glycerol containing one fluorophore species, with multi-exponential decays at shorter wavelengths and rise-decay kinetics at longer wavelengths.

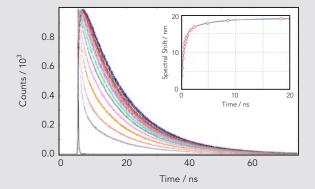
The TRES mapping option in Fluoracle enables quick data acquisition, while the FAST package allows performing complex analysis. Using globally linked 4-exponential decay analysis, decay associated spectra (DAS) can be generated and the peak position of the spectra be determined. The shift in the peak position is shown in the insert graph and clearly shows a 2-exponential reorientation dynamic of the fluorophore.

QUANTUM DOTS AND QUANTUM RODS

Quantum dots are nanometer sized semiconductor material structures that exhibit strong and specific fluorescence emission. The emission wavelength can be tuned by altering the size or the structure. These materials have widespread applications in modern displays, light emitting diodes, diode lasers, solar cell materials, as fluorescent inks and as fluorescent labels in biochemistry. Coated with biofunctional groups, quantum dots are being used as fluorescent labels in biomedical imaging and drug screening.



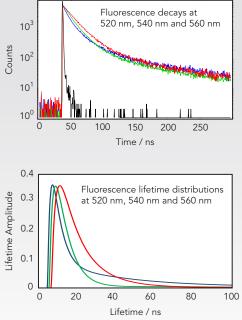
CdSe quantum rods spectra after TRES data slicing (left). Lifetime measurements and results of distribution analysis (right). Fluorescence decay measurements were excited with EPL-405.



3-amino-N-methylphthalimide in glycerol, measured with EPL-375 and TCSPC. inset: spectral shift of the calculated emission spectrum.

Compared to their spherical counterparts, quantum rods display less overlap between absorption and emission band. This reduces reabsorption and self-quenching and has therefore benefits over spherical structures.

Switching between spectral and lifetime measurements is easy in the FLS1000, enabling the user to study all aspects of their sample in a short period of time. The graphs show spectral data and TRES lifetime measurements obtained for quantum rods. Three lifetime measurements were analysed with lifetime distribution analysis to correlate distributions of lifetimes to distributions of particle size and shape parameters.





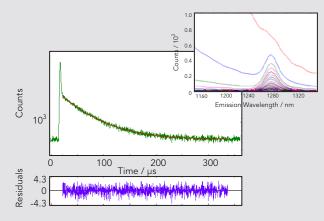




EMISSION OF SINGLET OXYGEN

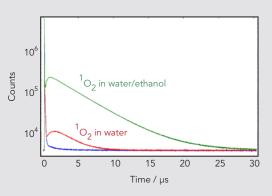
Time-resolved singlet oxygen measurements are challenging as the emission at ~1270 nm is very weak. The emission lifetime is solvent dependent and becomes comparatively short in aqueous solutions.

The figures below shows that time-resolved singlet oxygen measurements are possible with the standard microsecond flashlamp as excitation source.



Singlet oxygen generated by [Ru(bpy)₃]Cl₂ in oxygen saturated D₂O, excited with μ F2 and detected with PMT-1700. Inset: Background removed ¹O₂ emission after TRES data slicing. By measuring time-resolved spectra the singlet oxygen emission can be discriminated from the background.

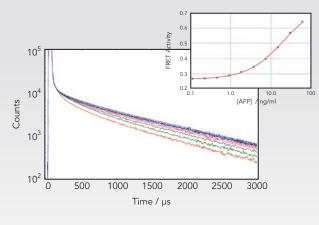
The figure below shows a time-resolved singlet oxygen emission measurement with exceptionally high temporal resolution. An instrument configured with a nanosecond laser source and MCS detection is the best choice for this type of measurement. A wide variety of ns pulsed lasers can be integrated with the FLS1000.



Singlet oxygen generated by chlorine e6 in water/ethanol, pure water, and instrumental response function. Samples were excited with DPSS laser at λ_{ex} = 355 nm, detection by PMT-1700.

FRET-BASED IMMUNOASSAYS

Time-resolved measurements of FRET in the millisecond time scale can provide more accurate data than other FRET techniques as background emission from the assay occurring in nanoseconds can be removed by detector gating. Other residual background in the microsecond time scale is eliminated during data analysis.



Europium cryptate lifetime measurements in an enhanced sensitivity immunoassay, measured on a 96-well plate within the plate reader. Excitation by μ F2 and emission detected by gated photomultiplier (PMT-900GT).

The example shows the change in the fluorescence decay kinetics of the donor europium cryptate, quenched by a nanosecond emitting acceptor. The fraction of FRET quenched donors is proportional to the concentration of the antigen, in this case Human Alfa Fetoprotein.

The high sensitivity of this time-resolved FRET measurement in this homogeneous immunoassay allows for antigen concentrations that are significantly lower than the cancer marker threshold. Fast measurements on multiple samples are possible thanks to the plate reader accessory for the FLS1000.

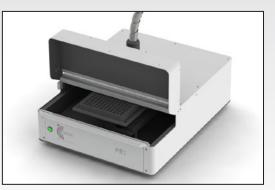


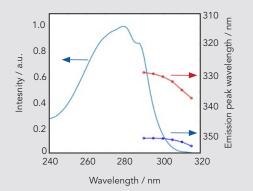
Plate reader accessory for the FLS1000.

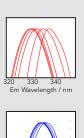
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RED-EDGE EXCITATION OF PROTEINS

Red-edge excitation fluorescence spectroscopy is a powerful method to study structure and dynamics of biological membranes, micelles and proteins. In protein research, this method takes advantage of the possibility to selectively excite a small fraction of specifically located tryptophan species at the red edge of the tryptophan absorption. The shift in the positions of the emission maximum as a function of excitation wavelength yields information about polarity and dynamics of tryptophan microenvironment and thus can reveal valuable information about the status of the protein under investigation.

The FLS1000 with its outstanding sensitivity and its automated measurement functionality is an excellent tool for red-edge excitation fluorescence spectroscopy; even for very low concentrations of the protein under investigation.

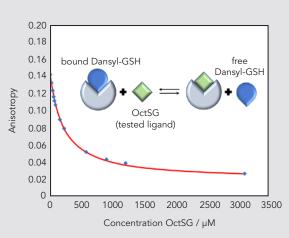






Excitation spectrum of tryptophan and fluorescence emission spectral shift functions of a crystalline eye lens protein dissolved in phosphate buffer saline (folded protein - red) and 7M urea (denatured protein - blue).

ANISOTROPY BASED LIGAND-BINDING ASSAYS

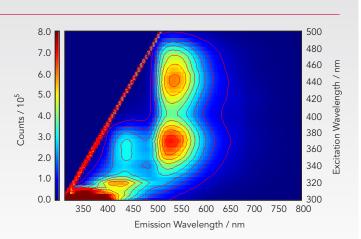


Calibration curve for the OctSG binding assay, using Dansyl as fluorescent label, $\lambda_{exc}^{}=$ 340 nm, $\lambda_{em}^{}=$ 550 nm.

A fluorophore bound to a ligand shows higher rotational mobility when free in solution, compared to when the ligand-fluorophore pair is bound to larger proteins. This change in rotational mobility can be monitored by fluorescence anisotropy measurements.

Some ligand binding assays use this anisotropy method. The example competition assay shown here, probes the concentration of the unknown ligand OctSG, using a potassium efflux system protein as a binding side and the fluorophore Dansyl as the fluorescent probe.

The FLS1000 can be configured in T-geometry for simultaneous data acquisition from two emission channels. In this experiment, the fluorescence anisotropy was measured using a T-geometry system to simultaneously record the vertical and horizontal component of the fluorescence emission.



Milk sample, diluted in water, $\Delta \lambda_{exc} = \Delta \lambda_{em} = 3$ nm.





FOOD SAMPLE MAPPING

Excitation-Emission Maps (EEMs) provide a "fingerprint" of complex fluorescent samples, containing many components. This fingerprinting technique is used in the food and drug industry to monitor content and identify potential toxic contaminants.

The map to the right shows an EEM of a sample of diluted milk. Although milk is a highly scattering sample, the double monochromators in the FLS1000 provide reliable EEMs with the absence of scattering effects. d Sam

Edinburgh Instruments has been at the forefront of research, development and manufacture of state-of-the-art photoluminescence instrumentation for over 50 years. During this time a worldwide reputation for quality and innovation has been established. Edinburgh Instruments continue to push product development of affordable, reliable and high quality instrumentation that can be tailored and upgraded to evolve with the science and technology.

WORLDWIDE CUSTOMER SUPPORT

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