



# LEXSYG SMART — A LUMINESCENCE DETECTION SYSTEM FOR DOSIMETRY, MATERIAL RESEARCH AND DATING APPLICATION

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**Abstract:** Following the luminescence system *lexsyg research*, which was designed for research, the luminescence reader *lexsyg smart* for the application of luminescence detection was developed by Freiberg Instruments. It is suited for routine measurements of luminescence (thermoluminescence, photoluminescence, photon-stimulated, optically stimulated and infrared stimulated luminescence) for a wide range of materials because of the availability of several stimulation sources. The possibility for user definition and change of most parameters provides a great deal of flexibility and also allows research applications. While detection is limited to a single unit and sample storage to 40 positions, the *lexsyg smart* is much faster in aliquot transportation compared to the *lexsyg research*, and allows fast mass measurements in luminescence dating, retrospective and personal dosimetry, etc. Cross talk of optical stimulation is absent and cross-irradiation is negligible from the single radioactive source ( $\alpha$ ,  $\beta$  or x-ray) because of a disconnected sample storage wheel from the measurement chamber, which has a small volume and therefore gas consumption is small. Thermoluminescence measurements and pre-heatings are possible with a versatile heater, which can be programmed for linear/non-linear heating at varying rates and durations for an almost unlimited number of steps. Optical excitation for up to three wavelength bands (violet, blue, green, yellow, infrared) is provided from high power LEDs or laser diodes, with an optional filter wheel to vary detection wavelength bands according to the material specific requirements. Either can be programmed to change at almost any time within measurement sequences.

**Keywords:** luminescence equipment, dosimetry, luminescence dating, PL, TL-OSL, PSL.

## 1. INTRODUCTION

Luminescence detection in dosimetric application, especially in dating application, often requires the fast and efficient measurement of large numbers of sub-samples (aliquots), or the number of dosimeters to be measured in environmental/personal dosimetry is high. However, such

applications often require some degree of flexibility because of the use of a variety of dosimetric materials with different properties, requiring different parameters and measurement setups, like detection wavelength. Additionally, various ways to stimulate the luminescence for thermoluminescence (TL), photoluminescence (PL) photon-stimulated (PSL), optically stimulated (OSL), infrared stimulated luminescence (IRSL) measurements are required. While some luminescence detection systems are aimed towards research (e.g. Lapp *et al.*, 2015; Richter *et al.*, 2013), and thus being of rather large dimensions, others (e.g. Bortolot, 2000; Bortolot and Carrievau, 1982;

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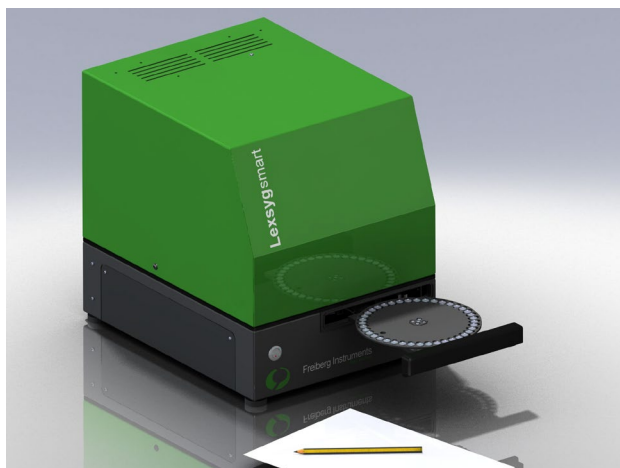


Fig. 2. The *lexsyg smart* luminescence system.

### Concept of the *lexsyg smart* luminescence reader

Likewise the *lexsyg research* (Richter *et al.*, 2013), the general concept of the *lexsyg smart* follows the separation of sample aliquot storage from the measurement chamber in order to eliminate cross bleaching effects during optical stimulation/bleaching and minimize cross-irradiation (Richter *et al.*, 2012; 2013). Due to the reduction in the distance of sample to detection, the sensitivity of the *lexsyg smart* is almost twice compared to the *lexsyg research*.

### Mechanical concept

Separation is achieved by storing the aliquots awaiting measurement on a 40 position wheel for cups/discs holding the material to be measured (Fig. 3). Physical dimensions of cups/discs to be used follow the standard sizes of  $10 \pm 0.0 - 0.2$  mm diameter, a typical thickness of 0.5 mm and a total allocation of maximum height (including sample) of 2.5 mm. The removable wheel containing the discs/cups is placed in a tray, which allows quick change of the sample wheel for efficiency. For example another wheel can be loaded during measurement allowing rapid continuation and quasi continuous use of the reader. Furthermore, it is possible to add, remove or change discs/cups during measurement when no transportation in or out of the sample wheel is taking place, which is useful in case of mistakes or the need to add additional material or aliquots and allows uninterrupted use.

The disc/cup is lifted by a piston from its position in the sample wheel and slid under the optional irradiation source or into the measurement chamber on to the heating plate (Fig. 3), similar to other approaches (e.g. Bortolot, 2000; Bortolot and Carriveau, 1982). In order to minimize gas consumption, the measurement chamber ( $\sim 10$  ml) is closed before any stimulation by lowering the upper part of the chamber (lid) by means of an eccentric drive mechanism, thus entirely separating the operation

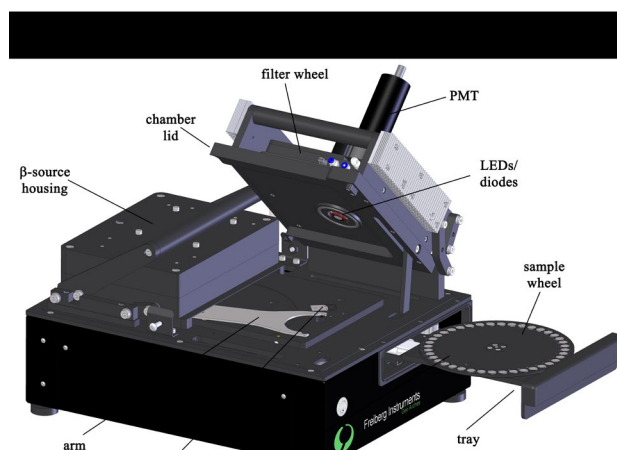


Fig. 3. The *lexsyg smart* system with the cover removed and chamber lid lifted for maintenance. The sample aliquots are stored on a sample wheel separate from the measurement chamber and moved to the measurement (and optional irradiation) position by an arm.

from the sample/disc reservoir (wheel). The measurement chamber containing the heating element is sealed with a quartz window (Sico SQ1) and can be evacuated as well as flooded with inert gas. With the exception of the optional filter wheel (pneumatically driven), lifting of the chamber lid and opening the source shutter, all movements are realised by electrical stepping motors.

### Irradiation sources

The *lexsyg smart* can be optionally equipped with a single irradiation source (Fig. 3), which use is indicated by a red LED for radiation safety purposes. Due to the safe construction, shipment of the source in place is possible, and therefore, in principal, no specialized installation of the radioactive source is required.

For alpha irradiation in air a  $^{241}\text{Am}$  (30 MBq) source can be used and two types of  $\beta$ -sources ( $^{90}\text{Sr}$ - $^{90}\text{Y}$ ) are available. One with an activity of  $\sim 1.85$  GBq delivers approximately  $0.13 \text{ Gy s}^{-1}$  to coarse grain (90–160  $\mu\text{m}$ ) quartz, while a weaker  $\beta$ -source ( $\sim 1.6$  GBq) provides a very homogeneous irradiation field (Richter *et al.*, 2012) at the expense of a dose rate of  $\sim 0.06 \text{ Gy s}^{-1}$ . As with the *lexsyg research* it possible to employ the Bremsstrahlung of the  $\beta$ -source for a much lower dose-rate irradiation by placing the sample for a defined time (software driven) underneath the closed source. For dosimetry purposes weaker  $^{90}\text{Sr}$ - $^{90}\text{Y}$ -sources delivering  $\sim 0.01 \text{ Gy s}^{-1}$  and  $0.001 \text{ Gy s}^{-1}$  can be used. Alternatively, a full housed x-ray irradiator based on tungsten (Varian VF-50) is available which includes a fully integrated dose rate life-time monitoring system. This equipment has the advantage of zero radiation when not in use and, depending on the chosen absorber, a useful variable dose-rate between  $\sim 0.01$  and  $\sim 2.1 \text{ Gy s}^{-1}$  (Richter *et al.*, in preparation).

## Luminescence stimulation

Luminescence can be stimulated in the *lexsyg smart* by means of heating or exposure to light. For thermal luminescence stimulation (TL) a metal plated ceramic contact heater reaching up to maximum 500°C (optional 710°C) is implemented for homogenous sample heating with repeatability and accuracy, which has also been shown by Lomax *et al.* (2014). The heater can be operated at rates between 0.1 and 20.0 K s<sup>-1</sup> with temperature calibration for each individual heater by pyrometer measurements of the surface of sample discs/cups. This allows for a precise temperature control, with the temperature lag (Kitis *et al.*, 2015) therefore being minimized and the temperature gradient within the sample itself presumably becoming dominant. Cooling rates can be user defined and are achieved with a pump. While inert gas certainly should be used for elevated temperature procedures, the equipment can be run with compressed air as well.

Optical stimulation in the *lexsyg smart* can be achieved by two (optionally three) different wavelengths integrated into a stimulation unit, consisting of a total of 15 positions for LEDs or laser diodes (usually 5 for each wavelength), which are identical to the stimulation sources in the *lexsyg research*. Each individual LED or diode is monitored by a photodiode and a feedback loop ensures constant light power output (Fig. 4), with deviations of <0.2%, as has also been shown in Lomax *et al.* (2014). This ensures the same light output from all LEDs/diodes and compensates for individual differences or fluctuations, rather than from an entire array consisting of several LEDs, thus also compensating for individual aging of each LED/diode. The efficiency of the feedback loop to provide excellent reproducibility of the stimulation is also shown for a green laser source used to measure the OSL signal from  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>:C exposed and meas-

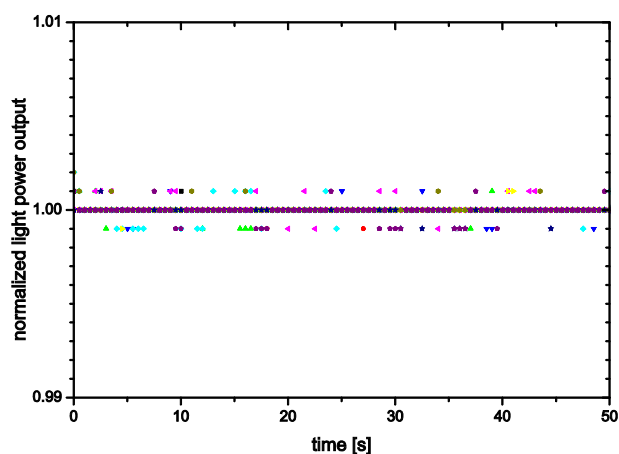


Fig. 4. Normalized light power output measured with a photodiode at 0.1 mW resolution for 10 individual blue LEDs (458 nm) set to 100 mW. The LED output was held constant through the photodiode feedback loop via PID controller for the power regulation of each individual LED.

ured to the identical dose 100 times, showing an excellent reproducibility and variation of < 1%, which includes also the variation caused by the material (Fig. 5). In combination with the alignment, setup and by the use of lenses a homogeneous illumination of the area of interest ( $\varnothing = 6\text{--}8$  mm) by the LEDs is ensured (Fig. 6). Thus, a maximum power stability, homogeneity of stimulation light on the sample and optical power stability is provided for the stimulation wavelengths listed in Table 1. In addition each LED/diode is equipped with appropriate filters to provide, if possible, narrow stimulation wavelength bands with maximized power (Fig. 7).

These setups allow OSL measurements in continuous wave (CW-OSL) as well as linear modulated (LM-OSL) mode at room and elevated temperatures for user specified power and duration, where the power (in mW cm<sup>-2</sup> s<sup>-1</sup>) at the start and at the end can be defined in LM-OSL.

## Luminescence detection

It is well known that luminescence emissions at different wavelengths might correspond to different physical properties and the need for restriction of the detection wavelength window in luminescence application (Fig. 1) has been shown, e.g. by spectral analysis (e.g. Huntley *et al.*, 1988; Krbetschek *et al.*, 1997; Rendell *et al.*, 1993; Townsend, 1994). The *lexsyg smart* can hold filters of 1 inch diameter with a total thickness up to 7.5 mm in a manual quick changer. However, full use of the potential of the equipment and flexibility is only achieved with an optional filter changer which is operated through the LexStudio software and allows change in detection wavelength bands during measurement sequences. This pneumatically driven filter wheel consists of 6 positions (one inch filters with a total maximum thickness of 7.5 mm) each providing the possibility for changing the detection

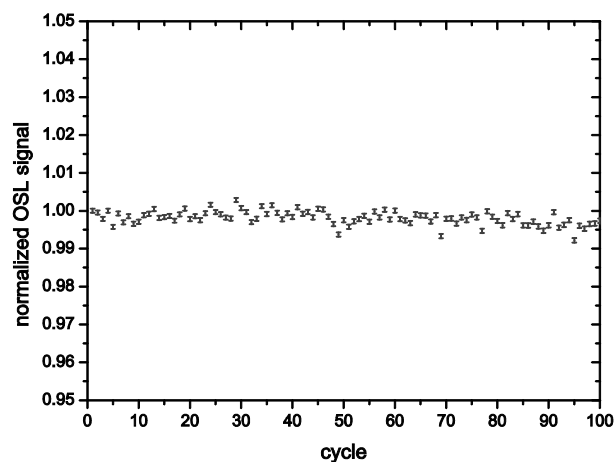
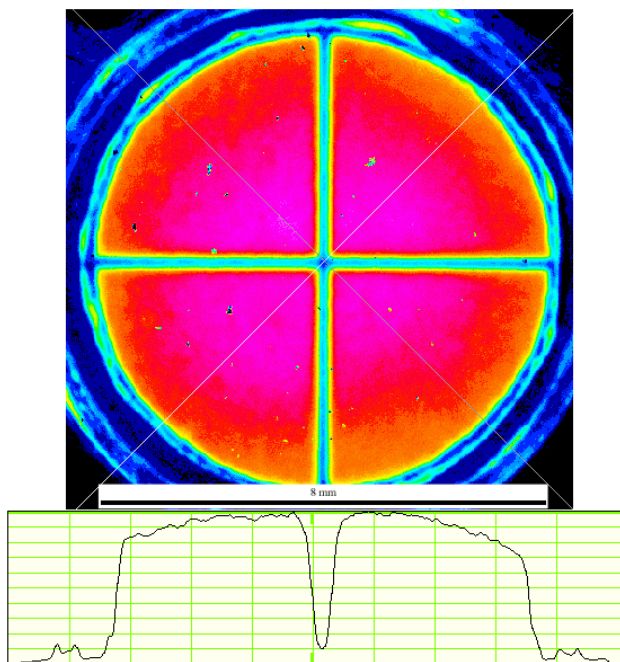


Fig. 5. First second of OSL-Signal (background 200–300 s subtracted) for a single  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>:C crystal of 100 cycles of identical  $\beta$ -dose exposure, measured with a green laser and normalised to first cycle.

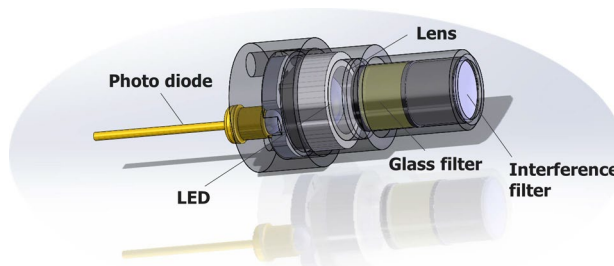


colour	type	wavelength (nm)	~ maximum power on sample (mW cm <sup>-2</sup> )	filter
blue	LED	458 ± 5	100	GG420 (3 mm) + HC 458/10
green	LED	525 ± 20	80	OG515 (3 mm) + HC 530/43
yellow	LED	590 ± 30	50	BG 39 (2 mm)
infrared	LED	850 ± 20	300	RG 715 (3 mm)
violet	laser diode	405 ± 3	100	BG 39 (1 mm) + OD405
blue	laser diode	445 ± 3	100	GG420 (3 mm) + HC 448/20
infrared	laser diode	850 ± 3	300	RG 715 (3 mm)

**Table 1.** Colour, type and wavelength of stimulation sources with the respective delivered power (maximum limited by software and calculated for the central 8 mm diameter area) on the sample position after passing through the filters. Standard sets are marked by a grey background.



**Fig. 6.** Relative reflected light measurement showing the homogeneity of illumination by IR-LEDs (850 ± 20 nm) on a sample cup lined with white paper. Note the homogeneity over the central area of interest and the reduced reflectance in the form of a cross in the centre caused by reference lines.



**Fig. 7.** Schematic drawing of the setup of an individual LED with lens, filters and the photodiode for the feedback loop to the PID which controls the power for the LED to provide stability.

window between measurements within 0.3 to 3.0 s. **Table 2** lists standard sets of filters for luminescence application with some selected materials. Filter combinations were optimized for best blocking of stimulation light (OSL) and/or thermal noise (TL, black-body radiation), while still providing maximum transmission in the desired wavelength range.

The *lexsyg smart* can be equipped with a single detector. For most applications standard PMTs are sufficient and the choice depends largely on the detection window and desired efficiency. Most commonly the bi-alkaline photomultiplier tube from Hamamatsu (H7360-02) with a range from 300–650 nm (27% quantum efficiency at 400 nm) is suitable, while an extension into UV detection is achievable with the EMI (9235QB), providing a detec-

use	filter combination	stimulation	material
Violet OSL – 340 nm	U340 + HC 340/26	Laser diodes (405 nm)	quartz
Blue OSL – 340 nm	U340 + HC 340/26	LED (458 nm)	quartz, Al <sub>2</sub> O <sub>3</sub> , BeO
Blue OSL – 365 nm	U340 + BP 365/50	LED (458 nm)	quartz, Al <sub>2</sub> O <sub>3</sub> , BeO
Green OSL – 365 nm	BG 3 + BP 365/50	LED (525 nm)	quartz, Al <sub>2</sub> O <sub>3</sub>
Yellow OSL – 330 nm	BG 3 + HC 340/26	LED (590 nm)	quartz, Al <sub>2</sub> O <sub>3</sub>
Yellow OSL – 365 nm	BG 3 + BP 365/50	LED (590 nm)	quartz, Al <sub>2</sub> O <sub>3</sub>
Yellow OSL – 410 nm	BG 3 + HC 414/46	LED (590 nm)	feldspar, Al <sub>2</sub> O <sub>3</sub>
IRSL – 410 nm	BG 39 + HC 414/46	LED / Laser diodes (850 nm)	feldspar
IRSL – 565 nm	BG 39 + HC 575/25	LED / Laser diodes (850 nm)	feldspar
IRSL wideband blue	BG 39 + BG25 + KG 3	LED / Laser diodes (850 nm)	feldspar
TL wideband blue	BG 39 + BG25 + KG 3	heating element	flint, quartz, LiF
TL – 330 nm	U340 + HC 340/26	heating element	quartz
TL – 380 nm	BG 3 + BP 365/50	heating element	quartz
TL – 410 nm	BG 39 + HC 414/46	heating element	feldspar
TL – 565 nm	BG 39 + HC 575/25	heating element	feldspar, quartz

**Table 2.** Suggested filter sets for some selected materials with standard sets on a grey background.

tion range between 160 and 630 nm with 30% quantum efficiency. The beneficial use of a red-enhanced UV-VIS-PMT for more efficient detection of luminescence emission at e.g. 620 nm for TL of quartz has been shown in a recent study (Richter *et al.*, 2015), employing the Hamamatsu H7421-40 thermoelectric/air cooled GaAsP photocathode PMT with a sensitivity between 300 and 720 nm and a quantum efficiency of 40% at 580 nm. Infrared luminescence detection can be achieved with a thermoelectric/air cooled ( $-10^{\circ}\text{C}$ ) GaAs photocathode PMT Hamamatsu H7421-50, providing a general sensitivity between 380 and 890 nm, with a peak of 12% quantum efficiency at 800 nm. In principle, any detection system can be mounted on a *lexsyg smart*.

#### 4. SOFTWARE LEXSTUDIO AND LEXEVA

The software *LexStudio* for operating the *lexsyg smart* allows easy programming of individual as well as standard measurement sequences. The graphical user interface combines clear arrangement with usability and provides sequence editing, device controlling and live viewing of measurements, within a user account management system. Parameter selection ranges are software restricted for safe operation of the *lexsyg smart*, while pre-sets can be defined by the user. Such are notably the choice of detection window for the selected stimulation type/wavelength in order to prevent overexposure of the detection system. Heating rates, stage duration times, maximums temperatures as well as cooling temperatures, whether for preheats, TL or elevated temperature meas-

urements can be set. Channel integration times, delayed stimulation or detection start times as well as their durations can be changed or predefined.

The generation of cyclic measurements, e.g. SAR-protocol (Murray and Wintle, 2000) can be tedious and is prone to error especially for novices. A SAR Plug-in (SARPI) helps with the quick generation of standard SAR sequences which are based on the user defined pre-sets of an approximation of the expected palaeodose, test-dose, preheat/cutheat temperature, read temperature and stimulation time (Fig. 8). Other user defined parameters, which are less frequently changed, like preheat/cutheat durations, stimulation powers, heating rates, channel width, cooling temperatures, *etc.* can be set in a parameter table (advanced options) depending on user authorization level. The irradiation times for regeneration dose points are expressed as percentage of the expected palaeodose and the number of regeneration dose points can be freely set, while including an obligatory recycling point. Optionally, an IR-depletion test can be automatically implemented for the last dose point to be measured and the software provides feedback if IR-depletion and recuperation points were omitted. To further aid in SAR sequence generation the general setup of measurements is visualized as a graph showing the dose points set on a theoretical dose response curve according to a user set simple mathematical model and estimated  $D_0$ . The various dose points are colour coded for visual verification of the principal correctness of the sequence (Fig. 8). Obviously choices for the stimulation wavelength and the detection window have to be made as well. The SARPI feature then auto-

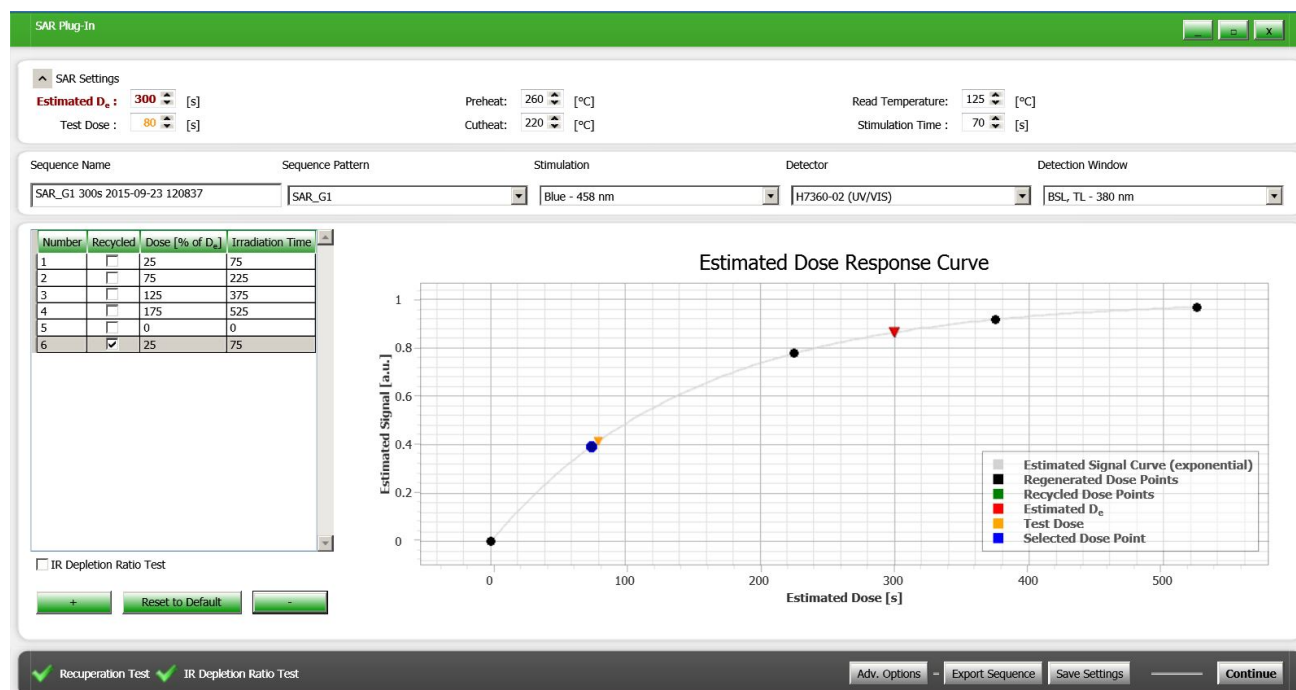


Fig. 8. Screenshot of SARPI, a *LexStudio* software plugin for the simple generation of standard SAR sequences for *lexsyg* devices.

matically generates a SAR cycle according the user defined parameters. The resulting SAR sequence based on such a rather standardized approach is often appropriate, but it can be modified like any other measurement sequence according further user needs.

The acquired data are stored together with the measurement parameters in a specific lexsyg format (xsyg), which includes not only the luminescence counts but also e.g. stimulation power over time, temperature during OSL measurements, *etc.* By providing such a full data set it is possibly to analyse and check the entire measurement and its parameters, as is required for physical measurements. Additional export for further data treatment is possible for ASCII and the luminescence data can be also recorded as bin/binx-format for processing in Analyst (Duller, 2015). The evaluation software LexEva provides the link to the R-package ‘luminescence’ (Kreutzer *et al.*, 2012) for data evaluation in luminescence dating application, while many other operations can be performed within LexEva (LexEva – User manual). For the analysis of artificial or other dosimeters the software TLStudio (TLStudio – User manual). is available, which was designed especially for TL-dosimetry and also reads and analyses the lexsyg generated files.

## 5. CONCLUSIONS

The *lexsyg smart* system is a versatile tool for standard mass luminescence measurements, while providing flexibility for the adaptation of measurements protocols by user interference to many measurement parameters like heating rates, maximum temperature, stimulation power and duration. It therefore also serves research in luminescence dosimetry as well as dating application. Cross-irradiation and –talk is minimized by the separation of measurement chamber and sample storage. It can be used for most dosimetric materials because it provides several stimulation possibilities. The *lexsyg smart* is capable of the excitation of luminescence by thermal, as well as violet, blue, green, yellow or IR stimulation, which can be measured by a single detector. Detection wavelength bands can be automatically changed with a filter wheel holding 6 slots to restrict luminescence detection to the appropriate wavelength band. Samples can be irradiated either by  $\alpha$ - or  $\beta$ -sources, and an x-ray generator can provide a flexible range of dose-rates.

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