

## Radiation Instruments

# Product Catalogue



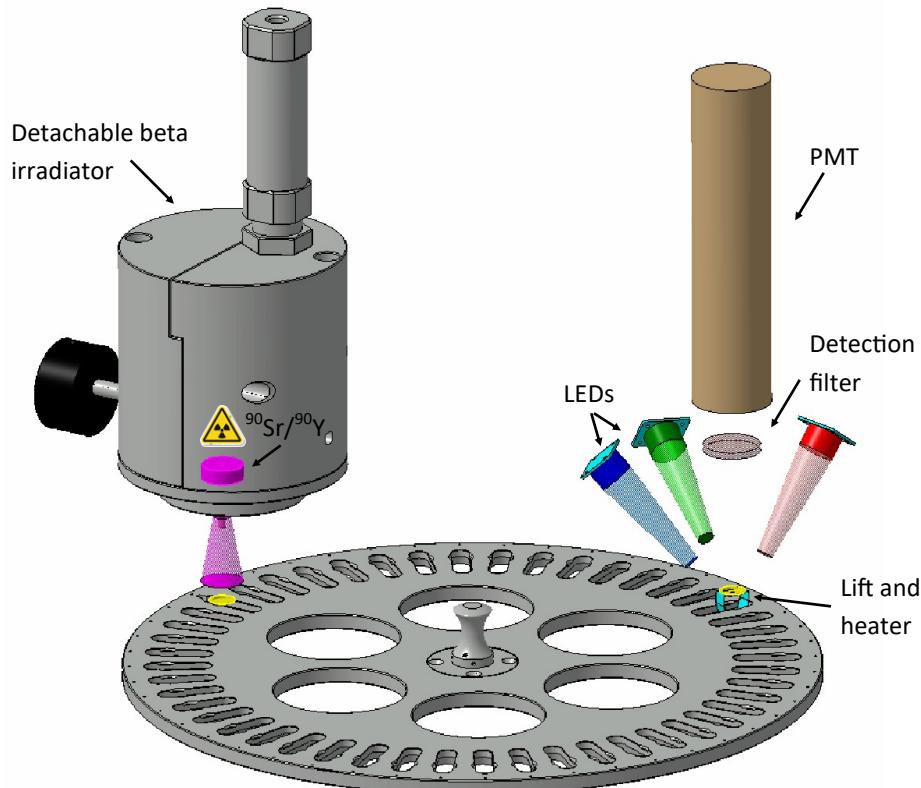
- Risø TL/OSL Reader
- Ultra-low-level Beta GM Multicounter
- Risø gamma spectrometry system

# 100. The Risø TL/OSL Reader

## The Instrument at a glance

The Risø TL/OSL Reader enables automated measurements of Thermoluminescence (TL) and Optically Stimulated Luminescence (OSL) signals. As the measurement system is highly sensitive and includes a reference radiation source, it is widely used for determining radiation doses in natural and artificial materials with applications in geological and archaeological dating, forensic and accident dosimetry, and radiation protection. Several state-of-the-art attachments to the TL/OSL reader allow investigations into the luminescence physics of different phosphors/dosimeters.

The fully automated Risø TL reader (model TL-DA-8; Bøtter-Jensen and Bundgaard, 1978) was the first of its kind in the world and became commercially available in 1983 (Bøtter-Jensen et al., 1983). In 1991, the platform was expanded to enable OSL measurements. Since then the Risø TL/OSL Reader has undergone continuous development to include new features and instrument attachments which can be retrospectively fitted to previous models. At present, more than 500 units have been delivered to outstanding research laboratories all over the world. The key features of our instruments are documented in peer-reviewed journal papers, and their continued development is driven by our own research needs, and those identified by the scientific community.



Schematic drawing of the standard Risø TL/OSL reader

The Risø TL/OSL Reader is an advanced automated measurement platform for the measurement of both TL and OSL. The standard TL/OSL Reader consists of a vacuum tight measurement chamber, a blue/UV sensitive photomultiplier tube (PMT), various detection filters, a thermal stimulation system, an optical stimulation system and complete control and analysis software. These individual units are described in the following.

# 100. The Risø TL/OSL Reader

## Overview of item 100

The following items are included when ordering the Risø TL/OSL Reader [item 100]:

- Automated 48-position light-tight measurement platform with vacuum chamber (0.2 mbar)
- Vacuum sensing system with automatic switching on reaching the pre-define pressure, vacuum gauge, and combined vacuum/nitrogen solenoid valves
- Blue/UV sensitive PMT [item 102]
- Detection filters [item 103]:
  - \* 5 mm Hoya U-340 [item 704]
  - \* 2.5 mm Hoya U-340 [item 705]
  - \* 2 mm Schott BG-39 [item 707]
  - \* 3 mm Schott BG-3 [item 709]
- Automated detection filter changer [item 104]
- Thermal stimulation system [item 105]
- Optical stimulation unit [item 106] equipped with
  - \* Blue (470 nm) LEDs,
  - \* Green (525 nm) LEDs
  - \* IR (850 nm) LEDs
- Controller [item 107]
- Risø software suite [item 108]
- 1 sample carousel for 48 discs [item 722]
- 1 sample carousel for 48 cups [item 723]
- 100 stainless steel sample cups [item 713]
- 100 stainless steel sample discs [item 715]



TL/OSL Reader [item 100] equipped with the following options: Beta irradiation unit [item 201], Automated detector changer [item 501], Red sensitive PMT [item 302] and the EMCCD camera [item 304].

### Notes:

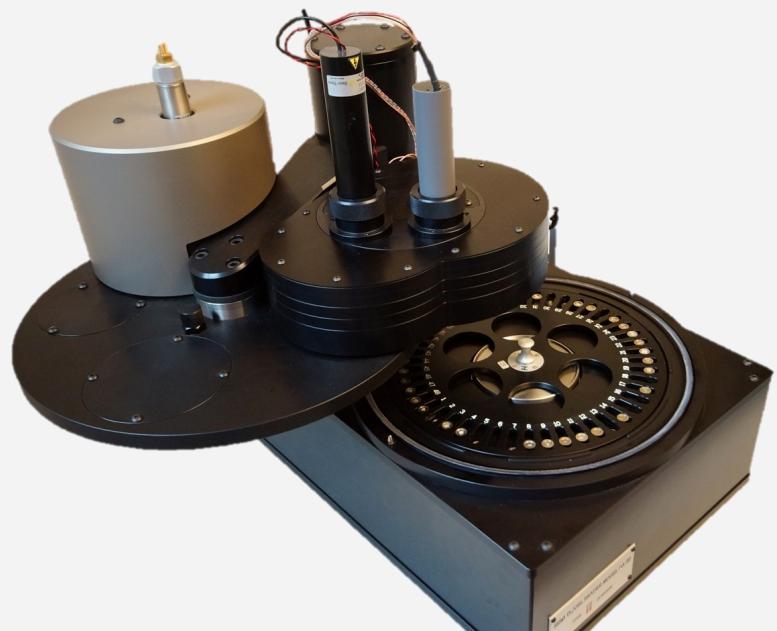
- i) The Risø Reader is run using a standard PC, which is not included in item 100. It can be supplied by the customer or purchased through DTU Physics [item 711]
- ii) In order to obtain vacuum conditions a separate vacuum pump must be used. Such a pump can be supplied by the user or purchased through DTU Physics [item 725]
- iii) Connection between a vacuum pump and the Reader requires the vacuum accessories [item 726]

# 100. The Risø TL/OSL Reader

## 101. Measurement Chamber

Samples are loaded onto an exchangeable sample carrousel accommodating up to 48 individual samples. The sample carrousel is placed in the sample chamber which can be programmed to be evacuated or have an inert gas atmosphere maintained by adjustable inert -gas flow.

- Automated 48-position light-tight measurement platform with vacuum chamber (0.2 mbar)
- Two exchangeable sample carrousels (each designed to hold up to 48 samples) for sample discs or cups
- Vacuum sensing system with automatic switching on reaching the pre -define pressure, vacuum gauge, and combined vacuum/nitrogen solenoid valves
- 100 stainless steel sample cups ( $\phi=11.7$  mm)
- 100 stainless steel sample discs ( $\phi=9.7$  mm)

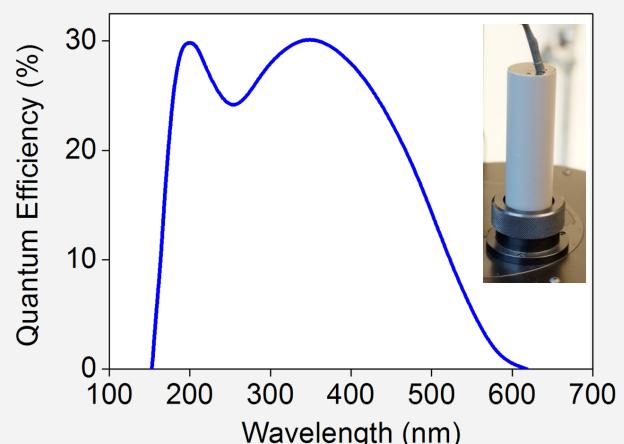


The Reader shown with the lid open and a sample carrousel in place.

## 102. Detection Unit

The standard Risø TL/OSL Reader is equipped with a Blue/UV sensitive Electron Tube PMT, which has maximum detection efficiency between 200 and 400 nm, making it particularly suitable for detection of luminescence from both quartz and feldspar.

- Electron Tube PDM9107Q-AP-TTL-03 with quartz window
- Spectral range: 160-630 nm
- 25 mm diameter biakali photocathode
- Dark count typically <50 cps at 20 °C
- Dead time: ~30 ns
- Max. count rate (with dead time correction) >20 Mcps



The quantum efficiency of the photomultiplier tube 9107Q as a function of photon wavelength and energy.

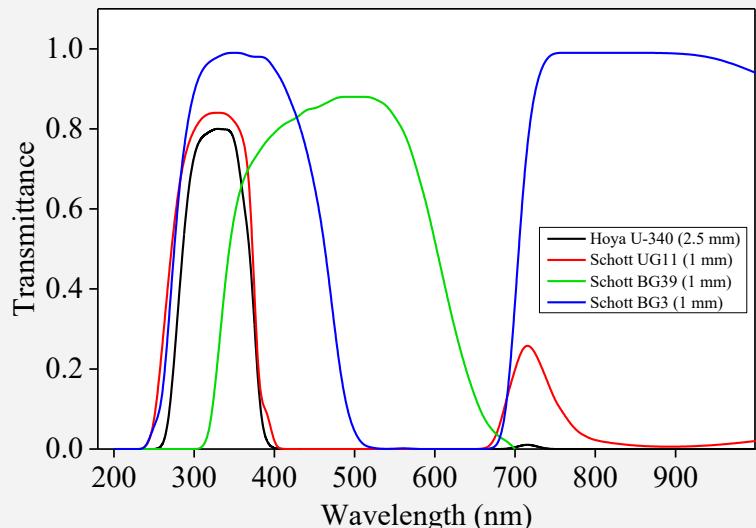
# 100. The Risø TL/OSL Reader

## 103. Detection Filters

Detection filters define the spectral range of the measured luminescence and prevent scattered stimulation light from reaching the detector.

The detection filters supplied with the Risø TL/Readers are particularly suited for the measurement of OSL from quartz and K-rich feldspar. The diameter of these filters is 25 mm (1 inch).

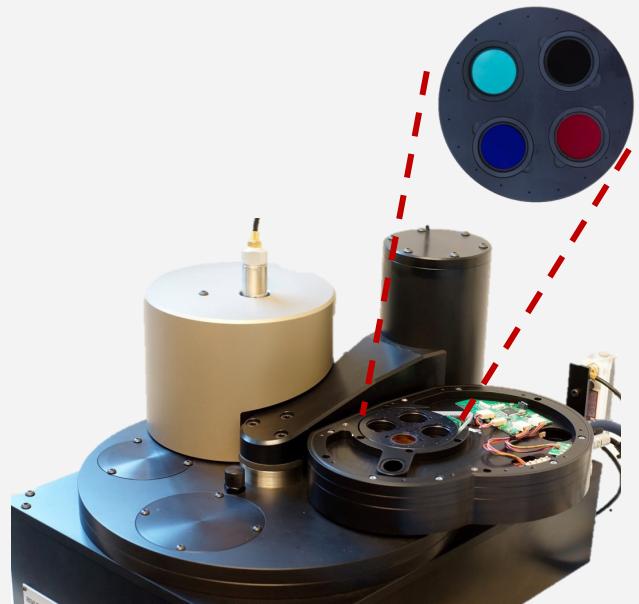
- 5 mm Hoya U-340
- 2.5 mm Hoya U-340
- 2 mm Schott BG-39
- 3 mm Schott BG-3
- Neutral density filter



Spectral characteristics of the supplied detection filters.

## 104. Automated detection filter changer

The automated detection filter changer (Lapp et al., 2015) is an integrated part of the Detection and Stimulation Head (DASH) which is modularised into four layers: Optical stimulation unit (bottom layer), filter changer (middle two layers) and detector changer (top layer). The filter changer enables automatic selection of detection filters during a measurement sequence. The detection filter changer consists of two filter changer wheels; each accommodating up to 4 detection filters and thus enabling the use of up to 16 filter combinations in a given measurement sequence. The detection filter changer is designed to accept individual filters (or stack of filters) up to 7.5 mm thick with a diameter of 25 mm (1 inch).



The detection filter changer occupies the two middle layers of the DASH. In this picture the detector changer (optional) has been removed for better illustration.

# 100. The Risø TL/OSL Reader

## 105. Thermal stimulation system

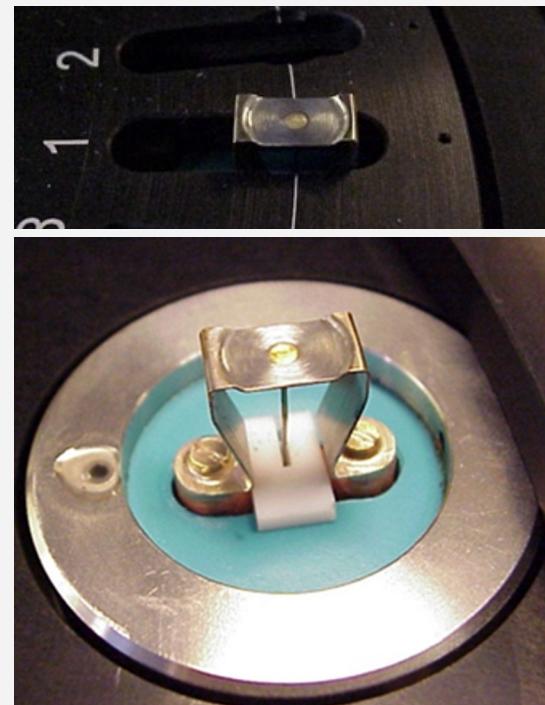
The heating element and lift mechanism is located directly underneath the photomultiplier tube. The heating element has two functions: 1) it heats the sample and 2) it lifts the sample into the measurement position. The heater strip is made of low-mass high resistance alloy, which is shaped with a depression to provide good heat transmission to the sample and to lift it securely and reproducibly into the measurement position.

Heating is accomplished by feeding a controlled current through the heating element. Feedback control of the temperature employs a Cromel-Alumel thermocouple (0.5 mm) mounted underneath the heater strip. The thermocouple is fixed to the heater element using a gold rivet.

Heating is provided by a continuous non-switching fixed frequency sine wave generator. The heating system is able to heat samples to 700 °C at constant heating rates from 0.1 to 10 °C / s. To minimise thermal lag between sample and heater strip heating rates above 5 °C / s are usually not employed.

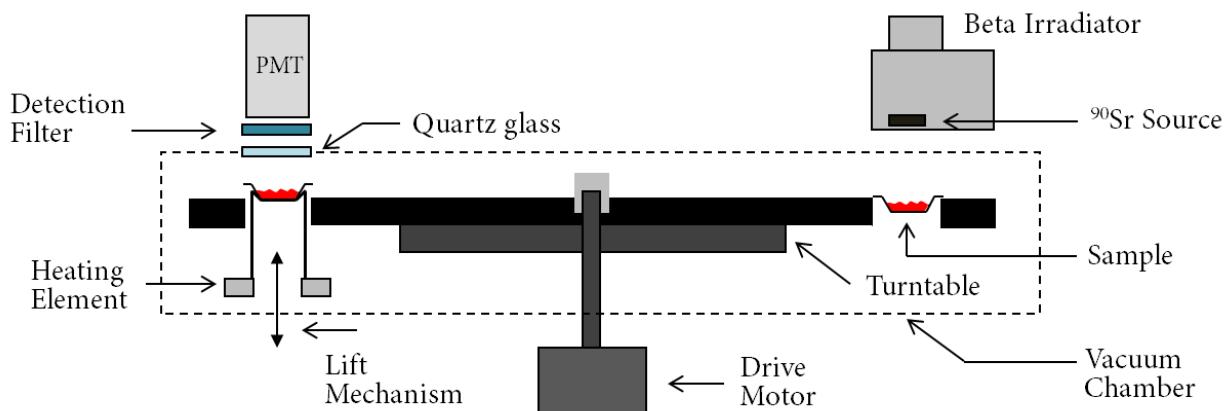
The heating strip can be cooled by a nitrogen flow, which also protects the heating system from oxidation at high temperatures.

Software corrects for systematic deviations (primarily related to electronic non-linearity) between set temperature and actual temperature of the heating element. The calibration for each heating system is unique. After calibration the systematic deviations are all within 0.25 °C of the Set temperature.



Heating/lift unit

- Heating/lift unit enabling heating of individual samples up to 700 °C
- Linear heating rates from 0.1 to 10 °C/s.



Schematic cross-section the Risø TL reader (from Bøtter-Jensen, 1988)

# 100. The Risø TL/OSL Reader

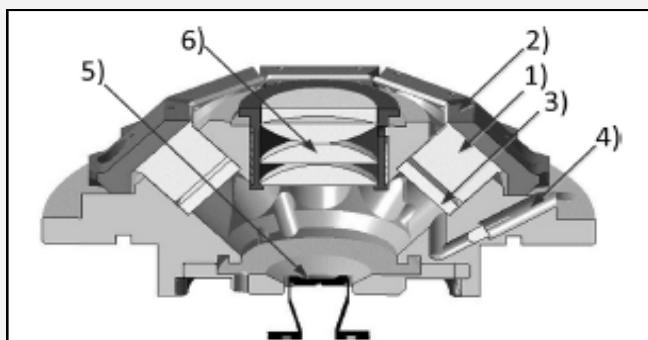
## 106. Optical stimulation unit

Optical stimulation is achieved using LEDs with high condensing lenses. LEDs are chosen for optical stimulation mainly because of their long lifetime and stability. The standard LED configuration is given in the table:

Colour	Wavelength (nm)	Bandwidth (nm)	Power density (mW/cm <sup>2</sup> )
Blue	470	20	>80
Green	525	30	>40
IR	850	33	>300

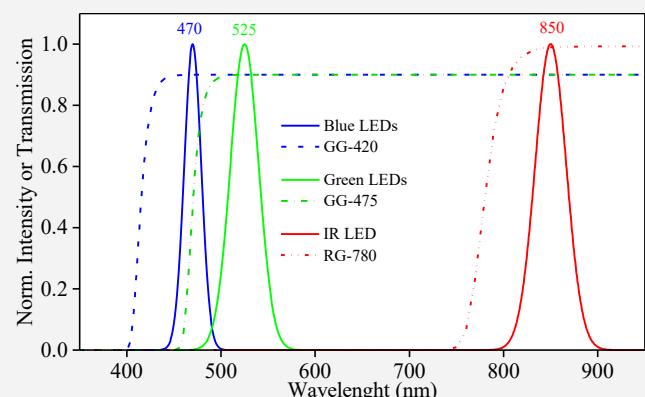
Other light source combinations available upon request.

The stimulation unit includes 7 locations for LEDs and one through-hole to allow the entry of the single grain laser beam (see item 401) or another external stimulation light source (e.g. item 403 or a user-supplied light source).



Cross section of the automated DASH base unit. 1) Stimulation LED with reflector, 2) LED cooler 3) stimulation filter, 4) feed-back photodiode, 5) sample heater, 6) collimating optics (Lapp et al., 2015).

Long-pass filters are used in front of the individual LEDs to minimise the amount of directly scattered stimulation light from reaching the light detection system.



Emission characteristics of the stimulation LEDs and transmission characteristics of the long-pass filters used in front of the individual LEDs.

The 8 stimulation locations are evenly distributed around the periphery of a circle centred on the sample location. The beams from the individual LEDs have an incident angle of 45° at the sample. The LEDs are mounted in close thermal contact with the aluminium framework of the DASH to provide good thermal contact with the entire reader and so ensure long LED lifetime and stability. Three of the LED positions (normally one blue, one green and one IR) have a feed-back photodiode mounted for regulation of the power during stimulation. All selections/operations are soft-

ware controlled, and can be user-selected in a measurement sequence.

The LED modules can be operated in

- Continuous wave (CW) mode [0-100%]
- Linear modulated (LM) mode [0-100%]
- Pulsed (POSL) mode [on/off times from 5 µs with 0.1 µs resolution]. Note that POSL measurements require the optional Pulsed OSL attachment.

# 100. The Risø TL/OSL Reader

## 107. Controller

All direct hardware control of the reader is performed by the Controller, which is responsible for maintaining proper timing, sample positioning, data acquisition, error checking, etc. The Controller is equipped with a two-line text display, which shows the current system status and the command which is currently being executed. This display also reports failure messages such as thermal failure and the receipt of invalid commands.



The Controller: controls all hardware

## 108. The Risø software suite

The Risø reader is supported by full-featured suite of software for controlling the hardware, automating the measurement sequence and analysing the recorded data. Main Risø programs:

- Sequence Editor
- Analyst
- Viewer+
- PTanalyse
- Control program
- XRFanalyse
- RLanalyse
- Viewer
- CorrSGbin
- DTcorrChange



These programs are described in more detail in what follows.

### Sequence Editor

The Sequence Editor PC program allows easy creation and execution of automatic TL/OSL measurement sequences in a convenient spreadsheet-like environment. Each cell can hold a single command that can be executed for any position on the sample wheel. Luminescence measurement methods directly supported by commands include: TL, OSL, LM-OSL, POSL, SG OSL, SG LM-OSL, TOL and RL.

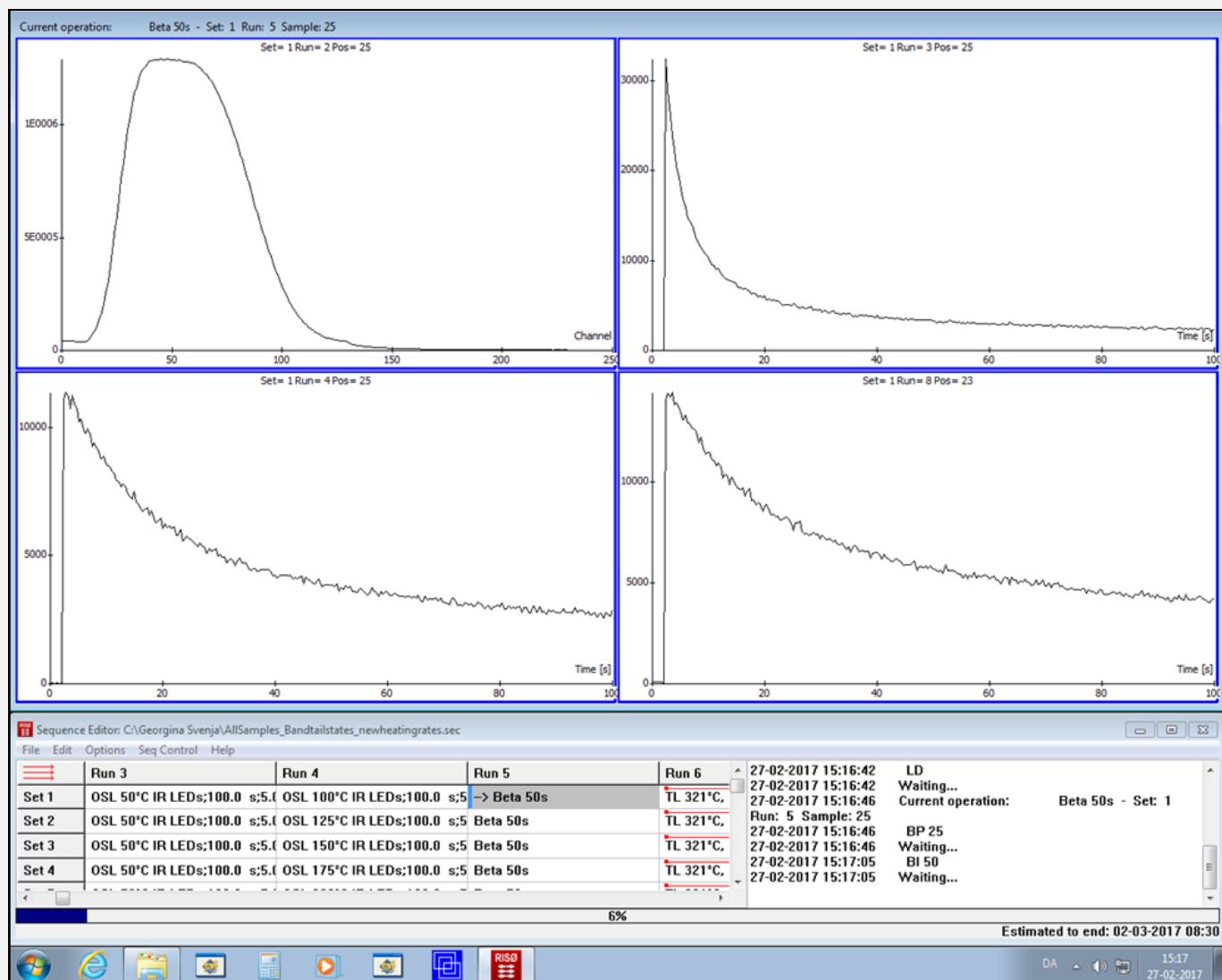
The commands are highly configurable with many parameters, but dialog boxes dedicated to each type of command provide help to the user in the form of input validation, context-sensitive help and graphical representation of the settings.

# 100. The Risø TL/OSL Reader

## 108. The Risø software suite continued

The execution of a sequence is done in cooperation with software embedded in the Controller box. The results of running a sequence are collected in a file on the PC in the standard BINX format. A BINX file consists of a list of records, one record for each

data acquisition command in the sequence file. A record typically contains a curve recorded from the PMT. The BINX file is the input to the analysis software.



Screenshot showing the Sequence Editor while it is running a measurement. The window is divided into three sections. The top section is a graphical representation of the last four recorded measurements. When a new measurement is undertaken the data are shown live. The left bottom section shows part of the measurement sequence and the command currently being carried out is highlighted (in this case irradiation with the beta source for 50 s). The right bottom section shows the information being stored in the log file in the form of the low-level commands executed by the Controller.

# 100. The Risø TL/OSL Reader

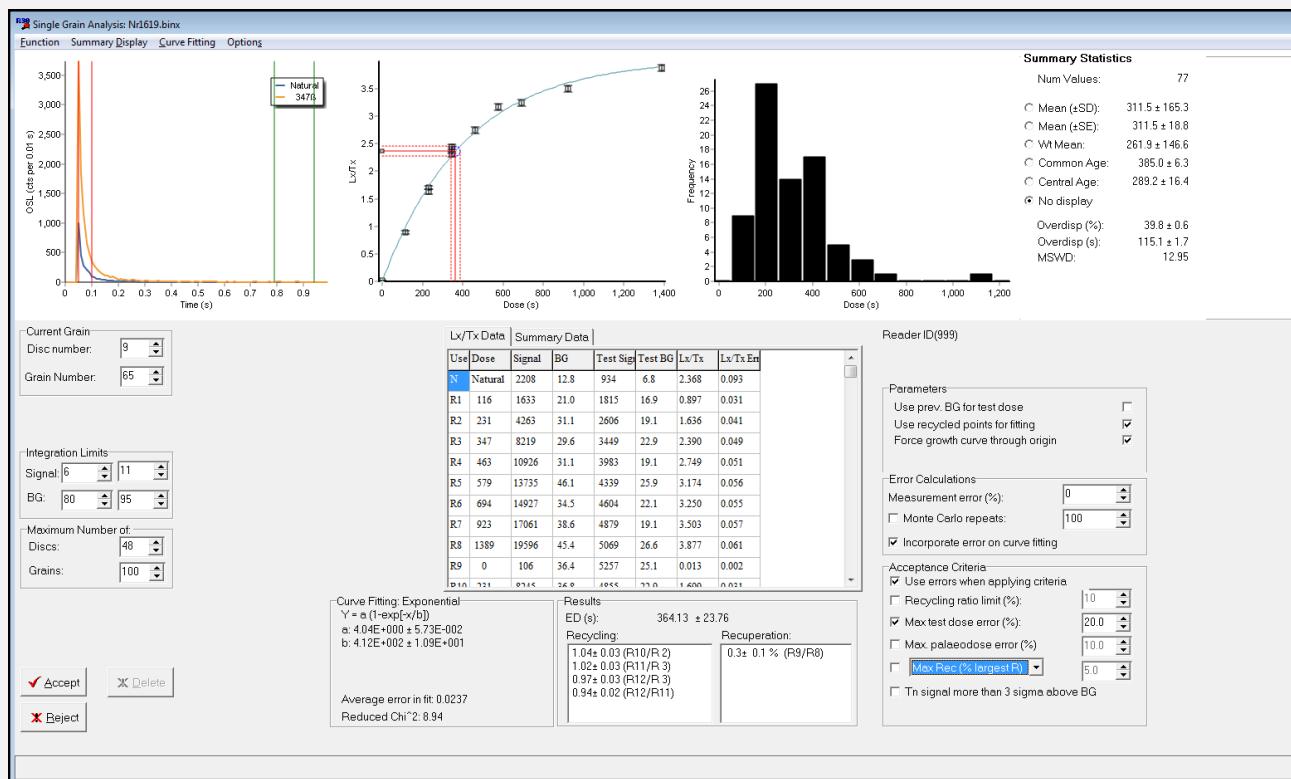
## 108. The Risø software suite continued

### Analyst

The central component in the analysis of measurements is the Analyst PC program which is developed by Prof. Geoff Duller of Aberystwyth University. It is at all times kept up-to-date with the rest of the Risø software by supporting all of the fields in the shared BINX file format. For example, it also supports the newer attachments like the DASH, EMCCD camera and spectrometer.

Analyst is too feature-rich to describe fully here but some of its main functions are preparing and

selecting BINX file records for analysis, viewing data as recorded, data processing (normalisation, background subtraction and channel integration), equivalent dose determination with multiple aliquot regenerative or additive dose procedures and single aliquot or single grain regenerative procedure.



Screenshot of single-grain quartz data analysis of a sedimentary sample.

# 100. The Risø TL/OSL Reader

## 108. The Risø software suite continued

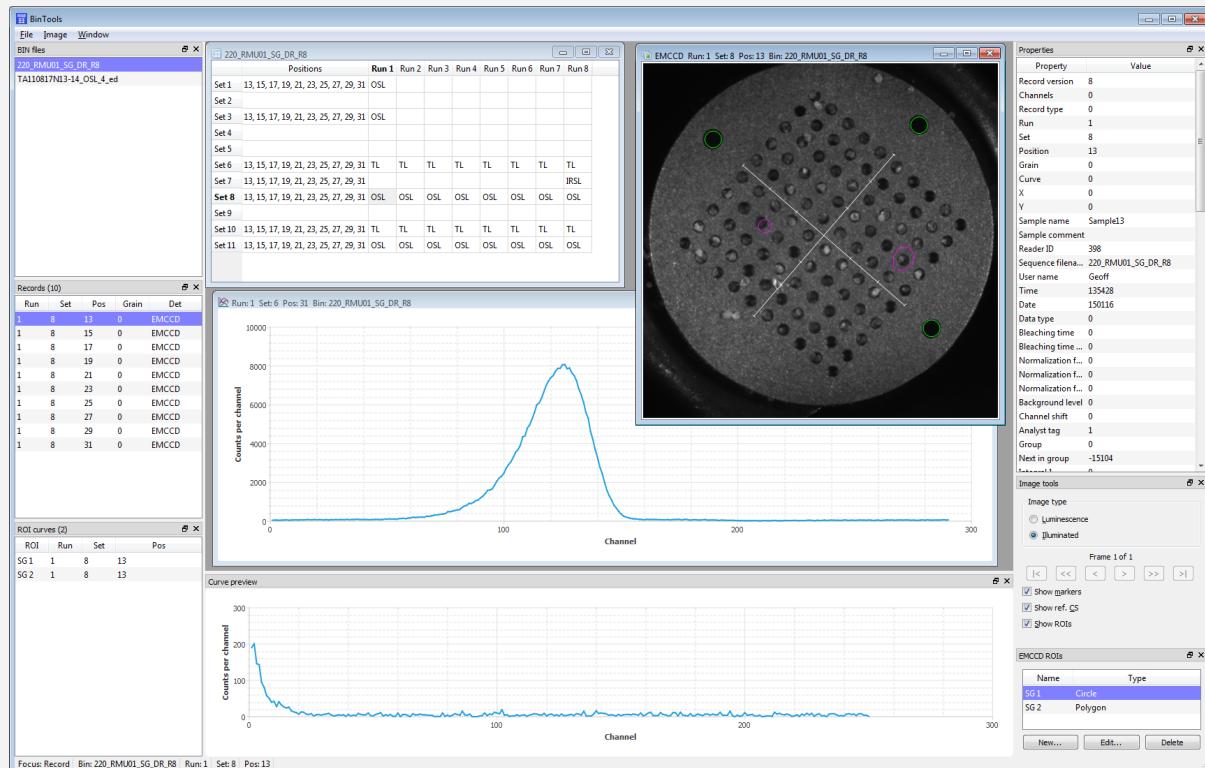
Some of the attachments produce data in formats that cannot be used directly by Analyst. Two programs are supplied for viewing and processing the raw data from these attachments, before further analysis is performed in other software, like Analyst, R Luminescence or Excel.

### Viewer+

The Viewer+ PC program is the most recent member of the Risø software suite and is designed to be the future platform for all the specific data processing and analysis that is not covered by Analyst. Presently, Viewer+ handles images from the EMCCD camera and spectra from the spectrometer attachment.

The basic tasks of Viewer+ are to open and read the image files and let the user navigate, view, plot and export the data. Specialised pre-processing, like computer vision algorithms for

cosmic ray removal and sample disc marker hole detection, is also the responsibility of Viewer+. But the main use is for extraction of curves from spatially or spectrally resolved regions of interest. These ROI curves are saved in the standard BINX file format, so they can be further analysed in e.g. Analyst.



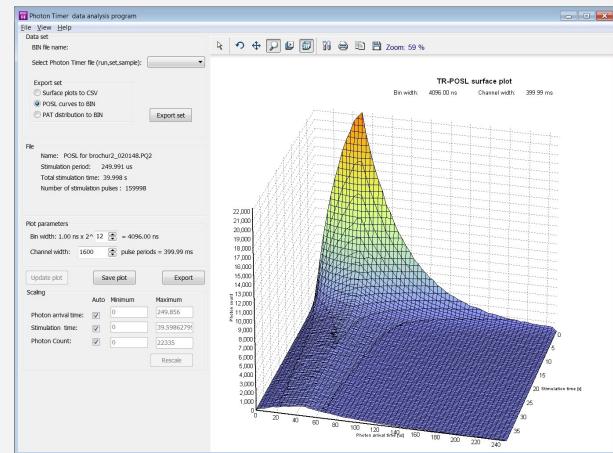
Screenshot showing the Viewer+ program while a Region of Interest (ROI) is being defined for an EMCCD image

# 100. The Risø TL/OSL Reader

## 108. The Risø software suite continued

### PTanalyse

The PTanalyse is used to process the files output by the photon timer attachment. Due to the very high resolution of the photon timer, and the correspondingly large data amounts produced, it is necessary to bin the data before they can be plotted in a 3D surface plot or exported to a CSV file. The POSL curve feature of PTanalyse allows you to calculate decay curves for arbitrary intervals within the pulse stimulation period. Another feature can visualise how the photon arrival time distribution has changed during the measurement.



Screenshot from the photon timer data analysis program, *PTanalyse*

### Miscellaneous software

#### XRFanalyse

XRFanalyse is a PC program used to analyse the spectra from the XRF attachment. The main analysis result is ternary diagram showing the composition of feldspar samples.

#### RLanalyse

RLanalyse is a PC program used to perform a specific fitting of two decay curves from the RL attachment. It calculates how many seconds a natural decay curve should be shifted to align with a section of a bleached decay curve.

#### Viewer

The Viewer is the original program for viewing and analysing BINX files. It has now been superseded by Analyst.

#### CorrSGbin

The CorrSGbin utility is used to correct BINX files containing single-grain (SG) OSL measurements for potential beta source non-uniformity.

#### DTcorrChange

DTcorrChange is a simple utility to add or remove dead time correction from all the PMT curves in a BINX file.

# 100. The Risø TL/OSL Reader

## 108. The Risø software suite continued

### System software

The software embedded in the Risø Controller has a documented interface (called the low-level interface) that makes it valuable in its own right. For example, you can control the Risø reader from LabView or another programming language that allows communication on the serial interface. High-level commands in the Sequence Editor are also built on the low-level commands of the Controller. This gives the end-user complete flexibility for customising existing commands or building entirely new commands for the Sequence Editor.

### Control program

The Control PC program uses the low-level commands of the Controller to operate the reader hardware. It is mainly used during installation or troubleshooting, but it can also be useful for end-users to perform simple and isolated tests of the different parts of the reader. This can be operations such as raising and lowering the sample lift, setting the sample wheel position, changing filter settings in the DASH and turning stimulation lights on and off.

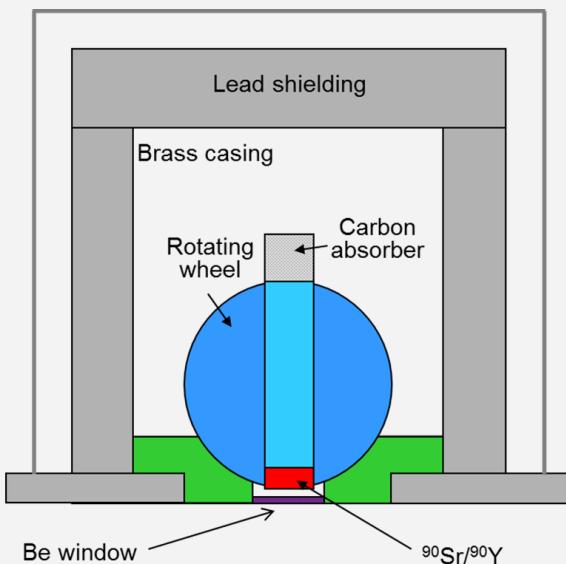


# 200. Optional Irradiation sources

Samples can be irradiated *in situ* using optional software-controlled irradiation sources: Sr-90 beta, Am-241 alpha and X-ray (50 kV, 1 mA, 50 W). Radioactive sources are delivered directly from the source manufacturer.

## Beta irradiation

A detachable beta irradiator is located above the sample carousel. The irradiator is made of brass (outer diameter 10 cm) and is surrounded by 20 mm of lead on the sides, and 40 mm on the top. Furthermore, an aluminium safety helmet (outer diameter 222 mm) covers the entire irradiator and lead shielding. This irradiator accommodates a  $^{90}\text{Sr}/^{90}\text{Y}$  beta source, which emits beta particles with a maximum energy of 2.27 MeV. The half life is 28 years. The radioactive source is mounted into a rotating, aluminium wheel, which is pneumatically activated. The source is placed inside the irradiator, directly followed by a 20 mm thick aluminium spacer, a 20 mm thick lead spacer, a spring washer, and finally a 25 mm thick aluminium spacer. When the source is *off* (default position) it is pointing upwards directly at a 10 mm Carbon absorber. When the source is *on* (activated position) it is pointing downwards towards the measurement chamber. A 0.125 mm beryllium window is located between the irradiator and the measurement chamber to act as vacuum interface for the measurement chamber.

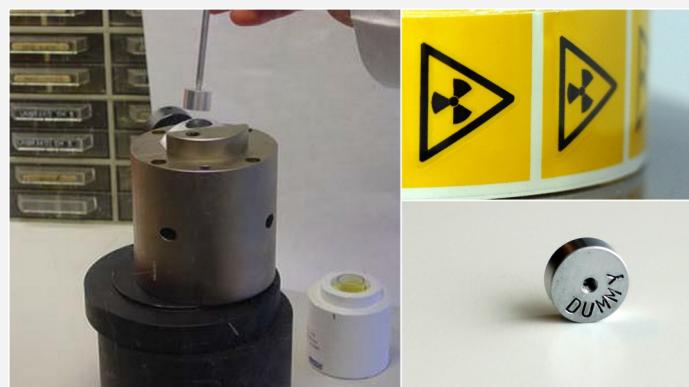


Schematic diagram of the cross section of the beta irradiator. The  $^{90}\text{Sr}/^{90}\text{Y}$  source is placed in a rotating aluminium wheel, which is pneumatically activated. The source is shown in the *on* (irradiating) position. When the source is *off* the wheel is rotated 180°, so that the source points directly at the carbon absorber. Redrawn from Markey et al. (1997).

## 201. Beta irradiation unit (1.48 GBq)

- Radioisotope: Sr-90 (see item 207)
- Nominal activity: 1.48 GBq ( $\pm 20\%$ )
- Type: Ceramic source (SICB20231)
- Dose rate:  $\sim 0.10$  Gy/s (in quartz on stainless steel discs)
- Detachable beta irradiator with a beryllium foil end window (see item 207)

The radioactive source is loaded into the detachable beta irradiator on-site.



# 200. Optional Irradiation sources

## 202. Beta irradiation unit (37 MBq)

- Radioisotope: Sr-90 (see item 208)
- Nominal activity: 37 MBq ( $\pm 30\%$ )
- Type: Ceramic source
- Dose rate:  $\sim 2.5$  mGy/s (in quartz on stainless steel discs)
- Detachable beta irradiator with a beryllium foil end window (see item 207)

The radioactive source is loaded into the detachable beta irradiator on-site.

## 203. Beta irradiation unit (2.96 GBq)

- Radioisotope: Sr-90 (see item 209)
- Nominal activity: 2.96 GBq ( $\pm 30\%$ )
- Type: Ceramic source (SICB18447)
- Dose rate:  $\sim 0.25$  Gy/s (in quartz on stainless steel discs)
- Detachable beta irradiator with a beryllium foil end window (see item 207)

The radioactive source is loaded into the detachable beta irradiator on-site.

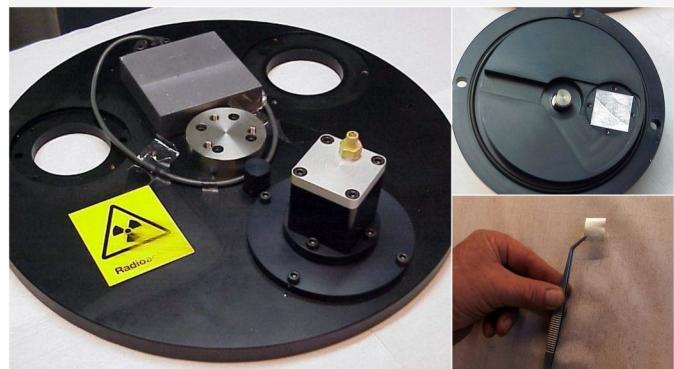


## 204. Alpha irradiation unit (10.7 MBq)

The alpha irradiator accommodates a 10.7 MBq (290  $\mu$ Ci) Am-241 foil source. Am-241 is a mixed alpha/gamma emitter. The dominating alpha energy is 5.49 MeV (85.1%) and the dominating gamma energy is 59 keV.

The source is mounted behind a pneumatically controlled shutter. The alpha irradiator option is integrated with the system lid and a sealed shaft allows operation of the irradiator under vacuum.

- Radioisotope: Am-241
- Nominal activity: 10.7 MBq ( $\pm 15\%$ )
- Type: foil source type (AMMB7616)
- Detachable alpha irradiator including pneumatic control valves



Left: Alpha irradiator mounted on a free-standing lid. Top right: Alpha irradiator seen from below showing the mounted foil alpha source . Bottom right: Foil alpha source.

The radioactive source is loaded into the detachable alpha irradiator on-site. Due to the short range of alpha particles from Am-241 the measurement chamber must be evacuated prior to irradiation. Vacuum control is part of the Risø TL/OSL Reader, but a vacuum pump must be ordered separately.

# 200. Optional Irradiation sources

## 205. X-ray generator

Highly uniform and reproducible *in situ* irradiation can be performed using this 50 kV X-ray source. A mechanical shutter incorporated into the collimator prevents sample irradiation before the X-ray output has stabilised (Andersen et al., 2003).

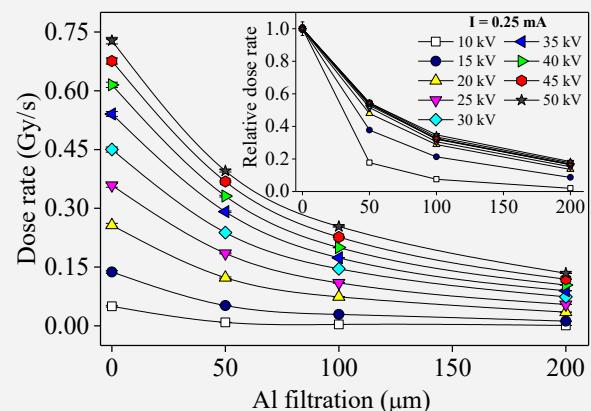


Left: The X-ray tube during testing at DTU Physics. Top: The Varian VF-50 X-ray tube before assembly. Bottom: The X-ray collimator block with mechanical shutter.

- Shielded, air-cooled Varian VF-50J (W) industrial X-ray tube with Wolfram target
- Spellman high-voltage power-supply (a modified version of the 50 kV, 50 W XRM50P50 model with filament preheat and 2 mA maximum current)
- Stainless steel mechanical shutter (7 mm thick)
- 35 mm long brass collimator (internal diameter 10 mm) with a 50 µm Al end window at the exit
- Shielding, cooling, and interlock features
- Control electronics and low-voltage power supplies.
- Dose rate: adjustable up to ~2 Gy/s (in quartz on stainless steel discs using a exchangeable 50 µm thick Al filter in front of the X-ray tube)

The observed dose-rate sample dependence in

sedimentary quartz (Thomsen et al., 2006) can be removed by hardening the X-ray spectrum, e.g. by increasing the thickness of the Al foil at the end window exit. The effect of the thickness of the Al on the dose rate is shown below.



Dose rate as a function of Al filtration thickness. The inset shows the relative dose rate as a function of Al thickness, i.e. the dose rate has been normalised to the unfiltered dose rate (from Thomsen et al., 2006).

## 206. Detachable beta irradiator without shielding

Designed to house a single Sr-90 source (see items 208 -210). A beta source can be mounted into a pneumatically activated rotating, aluminium wheel.



Brass beta irradiator

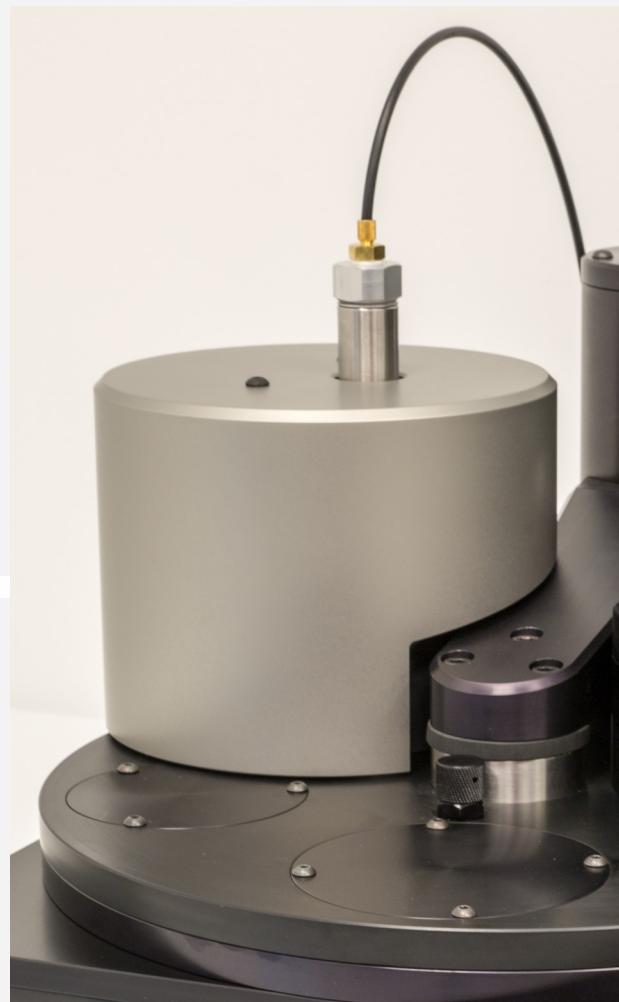
- Brass beta irradiator (outer diameter 10 cm)
- 125 µm thick beryllium end window foil

# 200. Optional Irradiation sources

## 207. Detachable beta irradiator with shielding

Designed to house a single Sr-90 source (see item 208-210). A beta source can be mounted into a pneumatically activated rotating, aluminium wheel.

- Brass beta irradiator (outer diameter 10 cm)
- 125 µm thick beryllium end window foil
- Lead (Pb) shielding (20 mm on the sides and 40 mm on the top)
- An aluminium safety helmet (outer diameter 222 mm) covering the entire irradiator and lead shielding



## 208. Sr-90 beta source (1.48 GBq)

- Radioisotope: Sr-90
- Nominal activity: 1.48 GBq ( $\pm 20\%$ )
- Type: Ceramic source (SICB20231)
- Dose rate:  $\sim 0.10$  Gy/s (in quartz on stainless steel discs)

Excluding the detachable beta irradiator (item 207)



## 209. Sr-90 beta source (37 MBq)

- Radioisotope: Sr-90
- Nominal activity: 37 MBq ( $\pm 30\%$ )
- Type: Ceramic source
- Dose rate:  $\sim 2.5$  mGy/s (in quartz on stainless steel discs)

Excluding the detachable beta irradiator (item 207)

## 210. Sr-90 beta source (2.96 GBq)

- Radioisotope: Sr-90
- Nominal activity: 2.96 GBq ( $\pm 30\%$ )
- Type: Ceramic source (SICB18447)
- Dose rate:  $\sim 0.25$  Gy/s (in quartz on stainless steel discs)

Excluding the detachable beta irradiator (item 207)

# 300. Optional luminescence detectors

## 301. Blue/UV sensitive PMT (160-630 nm)

Blue/UV sensitive Electron Tube PMT, with maximum detection efficiency between 200 and 400 nm (see item 102).

## 302. Red sensitive PMT detection module (300-720 nm)

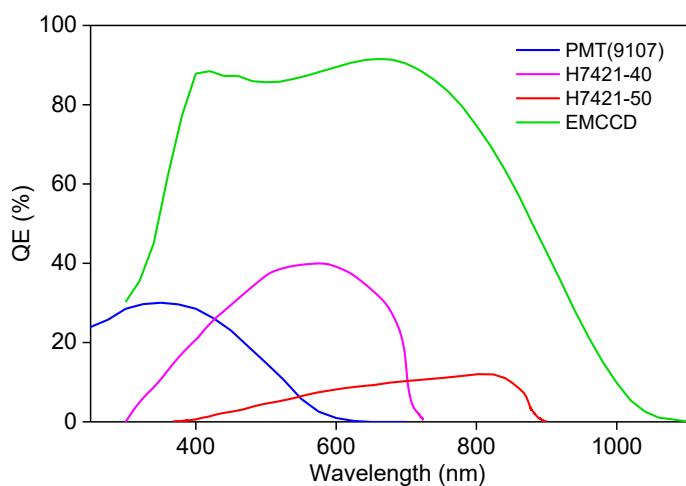
- A Hamamatsu H7421-40 Photon counting head thermoelectrically cooled to 0 °C
- Spectral range: 300-720 nm
- Quantum efficiency: 40% at peak wavelength
- Power supply and temperature controller (built into the Controller)
- Optical interface to the PMT module based on fused silica lenses
- The measurement sample area has  $\phi = 5$  mm

## 303. Red sensitive PMT detection module (380-890 nm)

- A Hamamatsu H7421-50 Photon counting head thermoelectrically cooled to 0 °C
- Spectral range: 380-890 nm
- Quantum efficiency: 12% at peak wavelength
- Power supply and temperature controller (built into the Controller)
- Optical interface to the PMT module based on fused silica lenses
- The measurement sample area has  $\phi = 5$  mm



The standard UV/Blue sensitive PMT (item 102) mounted for luminescence detection simultaneously with the red sensitive PMT detection module (item 302). The detector changer (item 501) is a prerequisite when several detectors are mounted simultaneously.

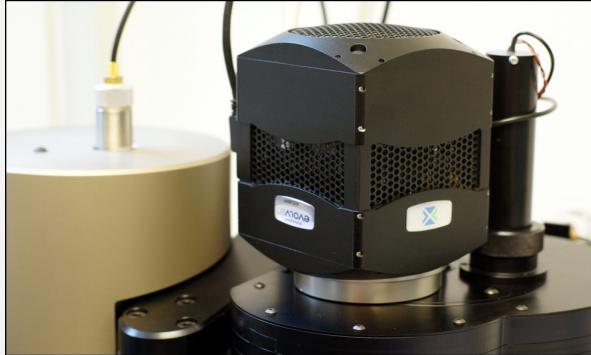


Quantum efficiencies for items 301 (PMT(9107)), 302 (H7421-40), 303 (H7421-50) and 304 (EMCCD) as a function of wavelength.

# 300. Optional luminescence detectors

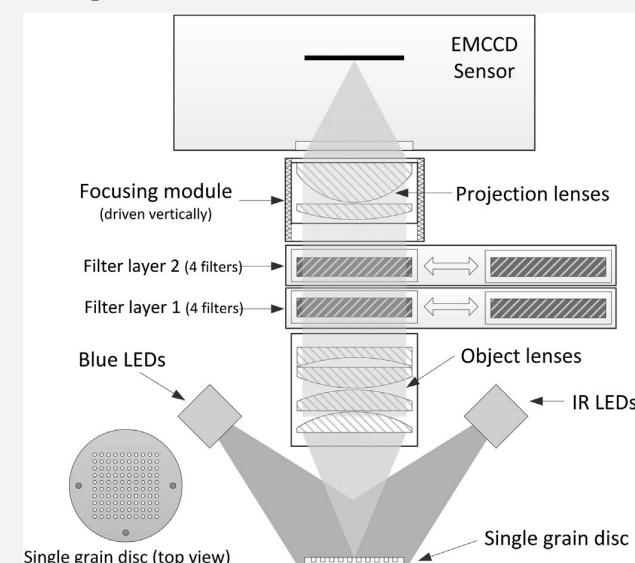
## 304. EMCCD imaging attachment

Enables routine luminescence mapping.



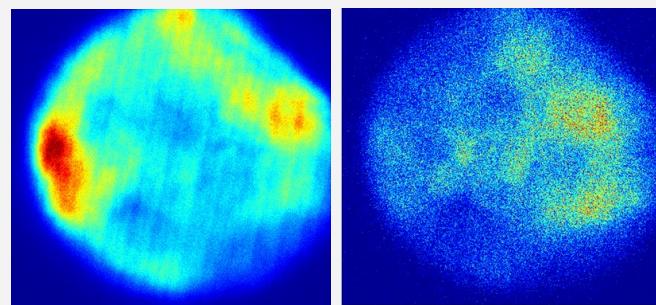
High sensitivity EMCCD imaging attachment

The detection optics is based on fused silica lenses with anti-reflection coatings, providing high UV-visible transparency, large numerical aperture (0.35) and magnification of ~0.8. One aspheric lens is used to enhance the image quality. Because the location of the focal plane is dependent on the measured wavelength, the projection lenses of the optics are mounted on a motorized focussing unit, software-controlled by the measurement sequence. The motorized focussing unit is calibrated by three LEDs, emitting at 470 nm, 525 nm and 870 nm. Sample thickness or location also affects the location of the focal plane.

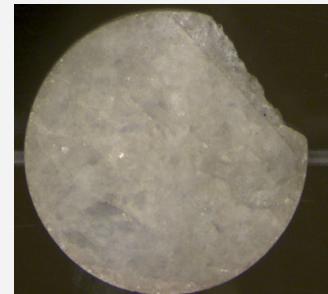


Schematic diagram showing the components of the luminescence imaging system mounted on a Risø reader (from Kook et al., 2015)

The automatic focusing module utilizes a low-noise piezoelectric linear motor capable of providing a much-improved focusing precision down to 0.5  $\mu\text{m}$ . The improved focusing module also includes a mo-



Top: EMCCD images of a quartzite rock slice (optical image shown to the right). Detection: U-340/UG11. Left: TL (90-120 °C) image. Right: Blue OSL (0-4 s) image.

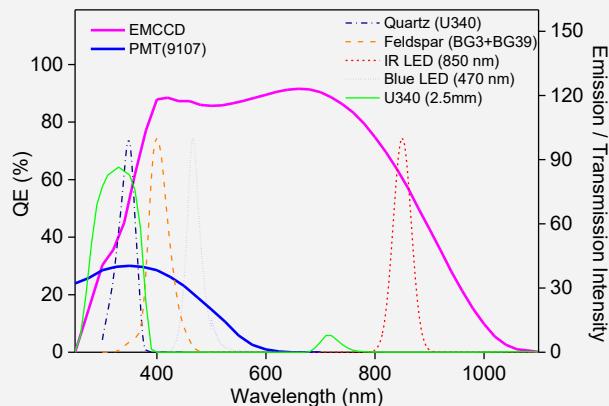


torized aperture adjustment function to meet different requirements during measuring samples with ultra-low signal to very high signal.

Images are captured by a Peltier cooled (-80 °C) Evolve EMCCD camera (Photometrics); this uses frame transfer and electron multiplier gain (up to 1000 times). The imaging area (512×512, 16 mm pixel) of the chip is 8.2×8.2 mm, and so the object size is 10.2×10.2 mm due to the reduction of the optics. The camera is connected to the host computer by IEEE-1394 (FireWire) for highspeed communication and is triggered by a signal from the Risø controller for synchronization with stimulation light. Read-out time for all pixels is 29.5 ms, but frame-transfer operation effectively reduces this considerably. In this mode, the image array is exposed to the signal for the desired period, and then the entire image is rapidly shifted (0.7  $\mu\text{s}$  vertical shift time) to the storage array. While the masked storage array is being read, the image array integrates charge for the

# 300. Optional luminescence detectors

## 304. EMCCD imaging attachment continued



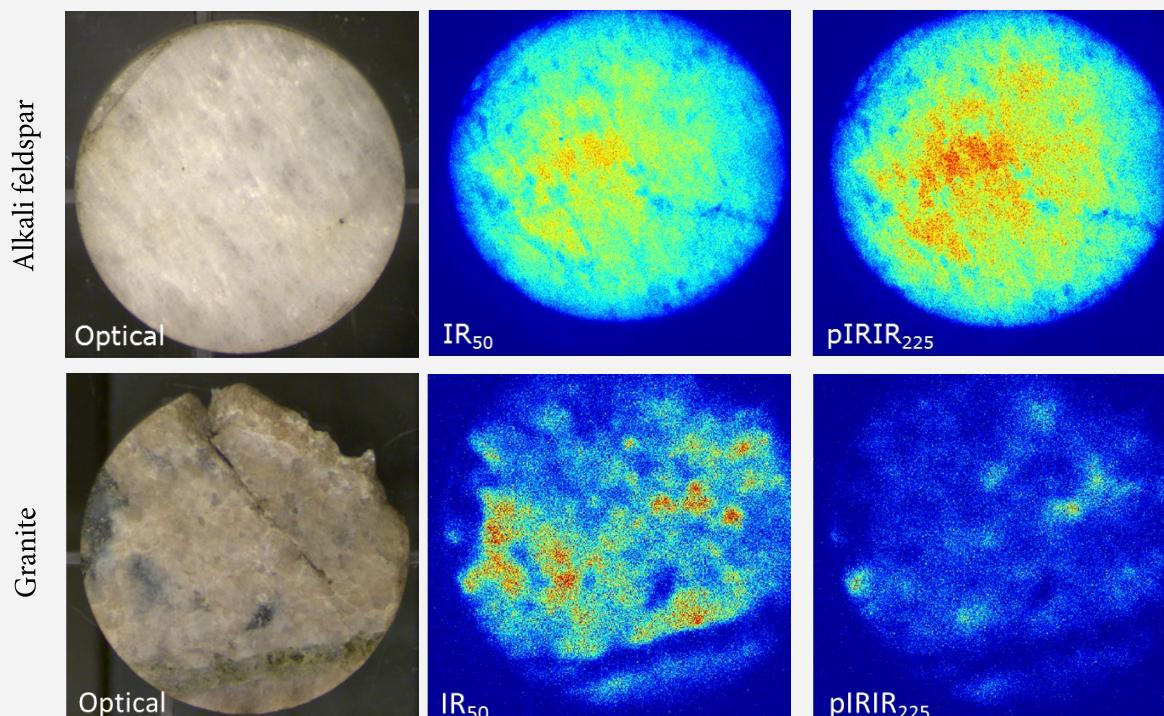
Comparison of EMCCD and PM tube response, LED emission and the transmission of the U340 detection filter. All curves should be read on the right hand axis except that of EMCCD and the photomultiplier tube, which are read on the quantum efficiency axis.

next image, providing a minimum dead time of ~400  $\mu$ s between frames.

The EMCCD has a broadband (UV to Near IR, UV-enhanced) window and a high quantum efficiency (QE) compared to a PM tube.

- EMCCD camera: Photometrics Evolve 512
- Peltier cooled, -80°C
- Frame rate: up to 30 fps
- Fused silica optics with broadband (UV-NIR) transmission
- Automated focussing and iris
- Data acquisition controlled by the Sequence Editor
- Data Analysis in Viewer+ (see item 109)
- 50 single-grain discs ( $\phi=300 \mu\text{m}$ , see item 713)
- 3 mm Hoya U-340 detection filter
- 2 mm Schott coated UG11 detection filter (IR blocking)
- 2 mm Schott BG 39 detection filter
- PC

The detector changer (see item 501) is a prerequisite for the EMCCD option.



Optical and EMCCD images of rock slices of alkali feldspar and granite measured using BG3 and BG39. The luminescence images were obtained after a dose of ~120 Gy and summation of the first 4 s of IRSL.

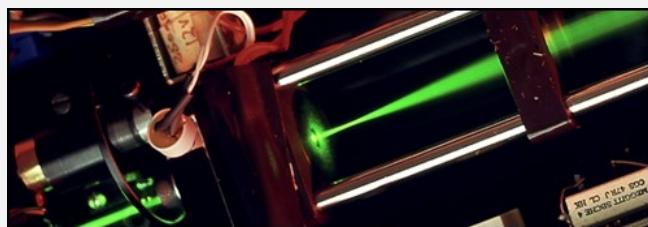
# 400. Optional light sources

The standard Risø TL/OSL Reader contains blue (470 nm), green (525 nm) and IR (850 nm) LEDs (item 106). Below additional available optical stimulation sources are described

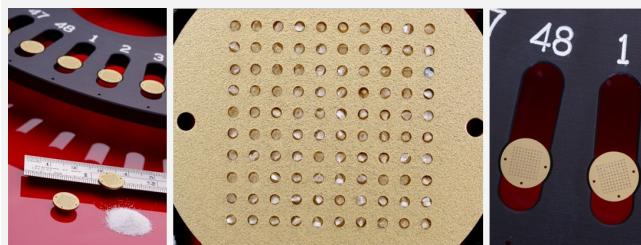
## 401. Single grain OSL attachment

Enables routine OSL measurements of individual sand-sized grains using a focused green laser (Bøtter-Jensen et al., 2003).

Individual grains are loaded in special aluminium discs containing 100 sample holes on a 10 by 10 grid with 600 µm spacing between hole centres. The diameter and depth of the individual sample hole is 300 µm (see item 713, other depths and diameters of the sample holes are available, see items 714-717).



Irradiation and heating is performed simultaneously on all 100 grains, whereas the OSL signal can be measured separately from individual grains using a focused laser (beam diameter on the sample disc is < 20 µm). This laser spot is steered to each of the grain holes in turn and switched on. The focused laser enables a high energy fluence rate and reduces the risk of optical cross-talk by ensuring that the entire spot enters the 300 µm diameter hole. Only a small part of the grain will be stimulated directly by the laser beam, but internal reflection within the grain hole is assumed to provide a uniform illumination of the grain.



Anodized, aluminum single grain discs. Do not heat above 500 °C.

The OSL is detected by the standard Blue/UV sensitive photomultiplier tube (item 102) and appropriate detection filters, e.g. Hoya U-340.

Three lenses are used to focus the laser beam. The laser spot is steered by two orthogonal mirrors and can be positioned arbitrarily on the sample disc. The mirrors are moved by two motor driven stages equipped with position encoders. The mirror in the x-direction is placed at an angle of 45° to the direction of the laser and the y-mirror at an angle of 22.5° to obtain an angle of incidence on the sample disc of 45°.

- Green (532 nm) 10 mW Nd:YVO<sub>4</sub> diode-pumped solid-state laser
- An X-Y scanning device using movable mirrors mounted on software controlled encoded motorised linear stages.
- 50 single-grain discs with hole depth and width of 300 µm



Single grain OSL attachment mounted on the Risø TL/OSL Reader.

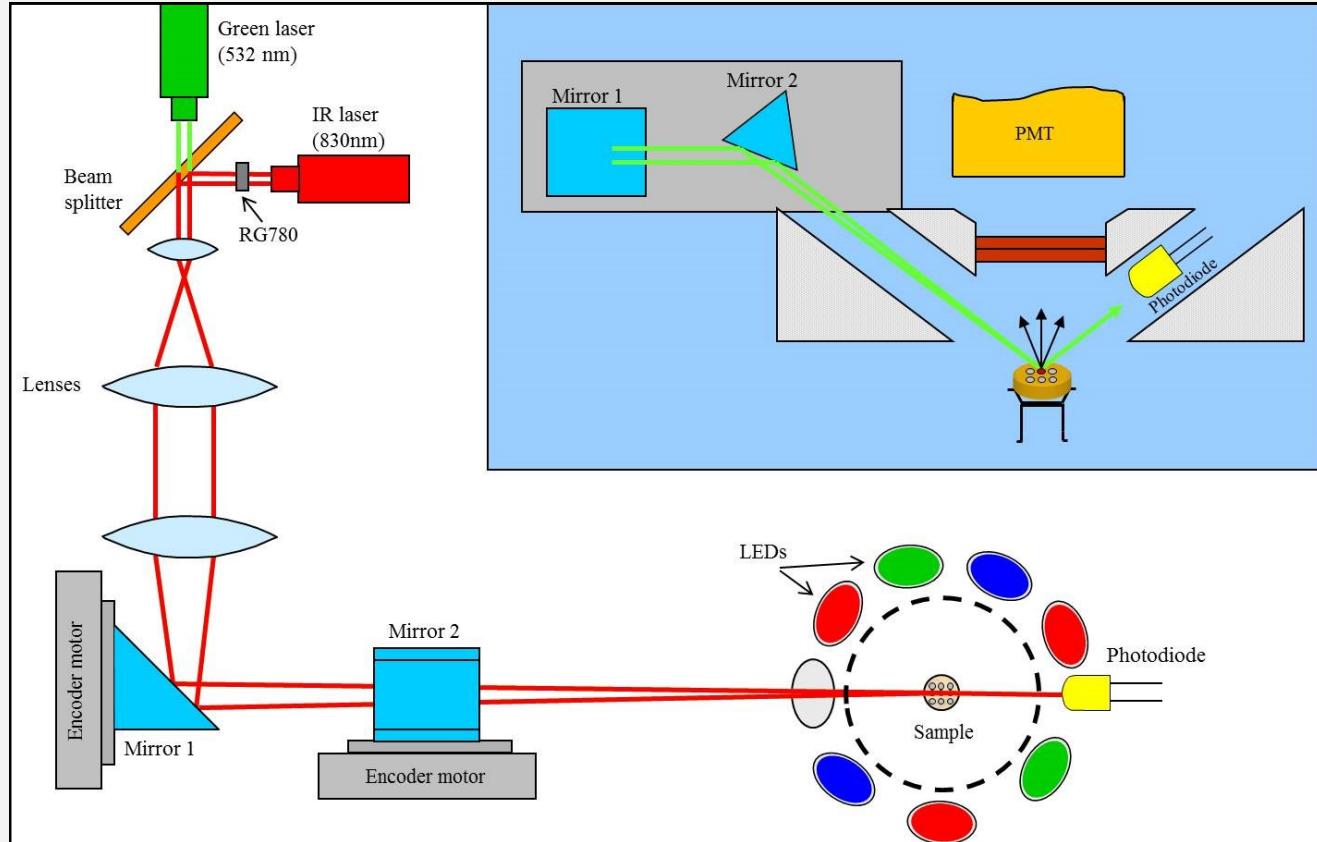
# 400. Optional light sources

## 402. Dual laser single grain OSL attachment

Enables routine OSL measurements of individual sand-sized grains using either a focused green or an infrared (IR) laser. A beam splitter enables the use of the same optics to focus the IR laser beam onto the sample disc as used for the green laser.

Individual grains are loaded in special aluminium discs containing 100 holes, 300 µm deep by 300 µm in diameter, on a 10 by 10 grid with 600 µm spacing between hole centres (other depths and diameters available upon request). Each grain hole is stimulated individually using one of the two focussed laser. A Schott RG 780 filter is placed directly in front of the IR laser to cut a small resonance emission at 415 nm.

- Green (532 nm) 10 mW Nd:YVO<sub>4</sub> diode-pumped solid-state laser
- IR (830 nm) 140 mW TTL modulated laser
- An X-Y scanning device using movable mirrors mounted on software controlled encoded motorised linear stages.
- 50 single-grain discs with hole depth and width of 300 µm
- 3 mm RG-780 longpass filter (mounted directly in front of the IR laser)



Schematic diagram of the dual laser single grain OSL attachment. Optical stimulation is achieved using a laser beam focused by three lenses. The position of the laser spot on the sample is controlled by moving two mirrors. Single grain OSL attachment seen from above. Inset: cross-section of the single grain OSL attachment. Adapted from Duller et al., 1999.

# 400. Optional light sources

## 403. Violet stimulation attachment

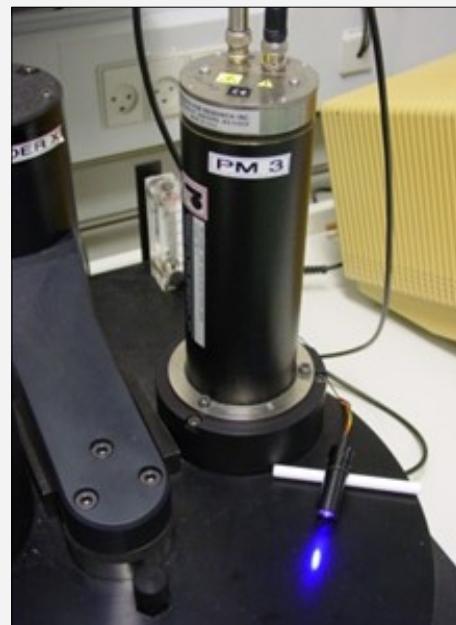
Probing deep traps require stimulation with high energy photons (e.g. >2.6 eV), which is challenging because of a general lack of powerful light sources in this energy range, and a risk of overlap between stimulation and detection windows (Jain et al., 2009).

Violet stimulation is achieved using a 405 nm laser module and the external port in the optical stimulation unit. The laser is focusable and is used with a circular diameter of 4 mm. It operates at 8 V–12 V DC and 80 mA current.

- Violet (405 nm) 100 mW laser module
- ITOS GG395 (3 mm) stimulation filter
- AHF F49-402, ET bandpass 402/15 nm stimulation filter (mounted directly in front of the laser)
- Semrock Brightline FF01-340/26 detection filter

The electronics for driving the amplifier is built into the controller. Due to physical constraints the violet stimulation attachment cannot be used simultaneously

with the of the single grain systems (see items 401 and 402).

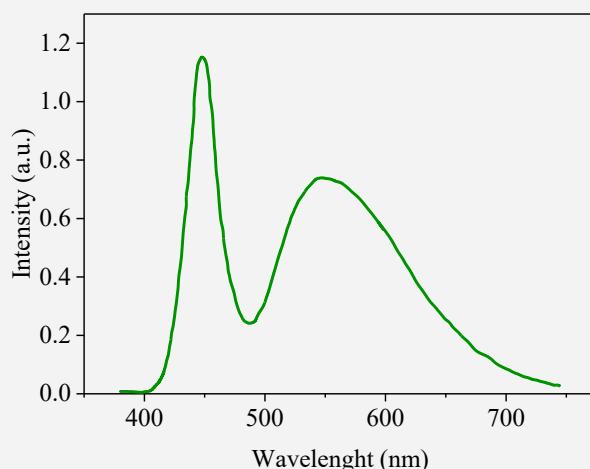


The violet laser

## 404. Bleaching facility – External broadband source

Powerful external broadband LED stimulation system for fast bleaching of aliquots (no data acquisition)

- FlexiLux 4000 broadband LED system
- Spectral range: 400–750 nm
- Optical power: 65 W
- Colour temperature: 5,800 K
- Liquid light guide
- Software control (on/off)



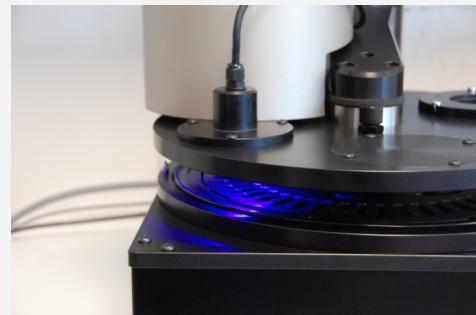
Emission spectrum of the external broadband source

# 400. Optional light sources

## 405. Bleaching facility – Violet LED module

A powerful Violet LED system for fast bleaching of aliquots (no data acquisition)

- Peak wavelength: 400-405 nm
- FWHM: ~ 20 nm
- Optical power: 1000 mW
- Software control (on/off)

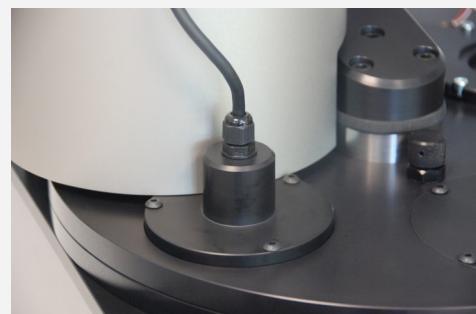


The violet bleaching facility

## 406. Bleaching facility – UV LED module

A powerful UV LED system for fast bleaching of aliquots (no data acquisition)

- Peak wavelength: 385-390 nm
- FWHM: ~ 12 nm
- Optical power: 1100 mW
- Software control (on/off)



The UV bleaching facility



Example of how a user-supplied external light source can be coupled to the Reader using the empty position in the DASH.

The stimulation unit in DASH (item 106) includes 7 locations for LEDs and one through-hole to allow the entry of the single grain laser beam (see item 401) or another external stimulation light source (e.g. item 403 or a user-supplied light source). The picture on the right shows how a user-supplied external light source can be coupled to the Reader.

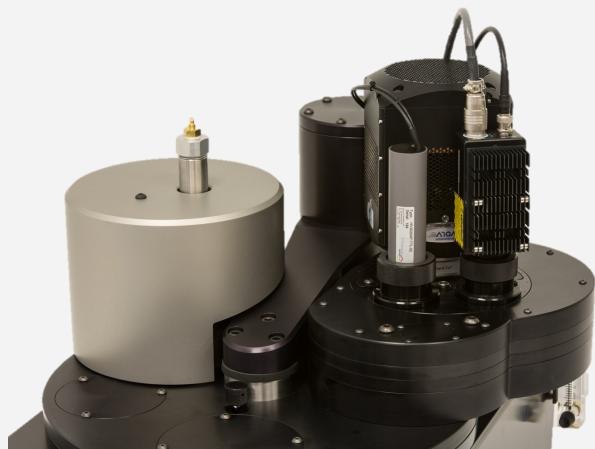
# 500. Optional attachments

The Risø TL/OSL Reader can be equipped with a range of attachments described below. Most attachments can be retrospectively fitted to previous versions of the Risø TL/OSL Reader.

## 501. Automated detector changer

The automated detector changer enables automatic selection of detector (Lapp et al., 2015). The detector changer accommodates up to three different detectors with one position dedicated to the EMCCD attachment (see item 304). All detectors use a common collimating lens system placed in the base unit.

- Three detector positions
- Software controlled detector change during a measurement sequence
- Fused-silica lens system



The detector changer is the top layer of the Detection and Stimulation Head (DASH) shown to the right. Here the detector changer is equipped with a standard blue/UV PMT (item 301), the red sensitive H7421-40 PMT (item 302) and the EMCCD camera (item 304).

## 502. High sensitivity emission spectrometer

Enables measurement of both TL and OSL emission luminescence spectra (Prasad et al., 2016)

- Andor DU-888U3-CS0-UVB EMCCD Camera, Back-illuminated 1024×1024 with standard AR coating and additional lumogen coating
- SR-193I-A Shamrock 193 imaging spectrograph base unit, 193 mm focal length, F/3.6 aperture
- 193i ruled gratings a 300 l/mm (500 nm blaze), and a 150 l/mm (500 nm) blaze giving wavelength regions of e.g. 300-700 nm and 300-850 nm (centre wavelength adjustable), mounted on a motorised dual grating turret



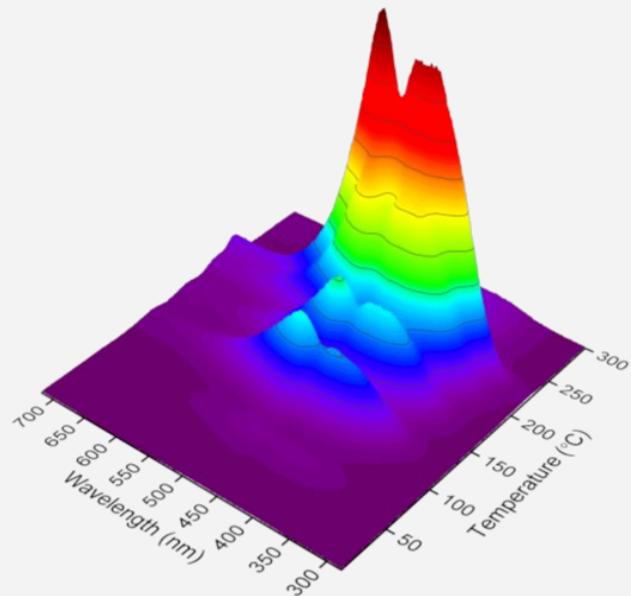
The Risø emission spectrometer attachment and the Risø TL/OSL Reader

# 500. Optional attachments

## 502. High sensitivity emission spectrometer continued

- Specially designed optical 3.1 mm circular fibre bundle with 114, 200 µm UV-VIS-NIR fibres, with a NA = 0.22
- Optical interface to the reader (mounted on the Automated detector changer)
- Reference Mercury/Argon calibration light source with optical fibre interface to the Reader for wavelength calibration
- The Sequence Editor controls the spectrometer when acquiring TL and/or OSL spectrometer data. The spectral data may be read with Andor Solis software and Risø Viewer+ software
- PC

The Automated detector changer (see item 501) is a prerequisite for the spectrometer attachment.



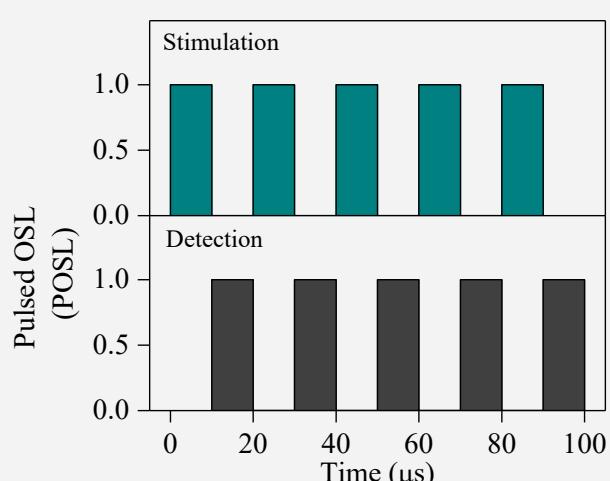
TL spectra from a LiF pellet.

## 503. Pulsed OSL

Combined blue, IR and green LED pulsed optical stimulation unit. The unit generates pulsed regulated power for the stimulation LEDs and gates the OSL signal according to specified parameters. The parameters for the pulses are set from the Sequence Editor program (Lapp et al., 2009).

The use of the Pulsed OSL attachment does not require the TCSPC attachment (item 504). The Pulsed OSL attachment can be ordered separately.

The unit is e.g. suitable for separating quartz from feldspar OSL signals in feldspar contaminated samples, because the life time of the feldspar OSL signal is significantly faster than that of quartz. Thus by pulsing the diodes and only detecting when most of the feldspar signal has decayed away, we can separate quartz and feldspar signals from each other in a mixed sample (e.g. Denby et al., 2006).

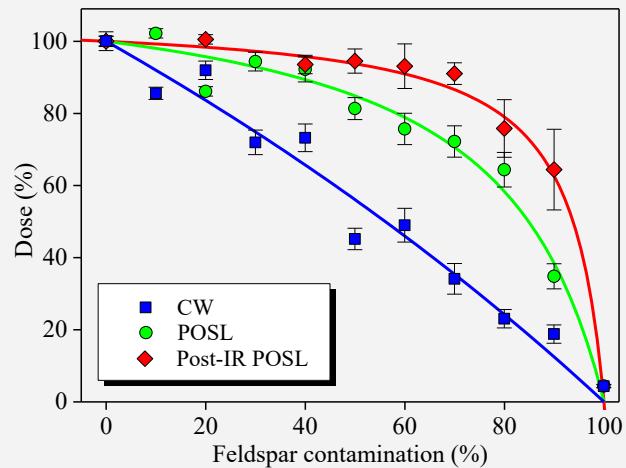


Schematic drawing showing stimulation and detection timing for on/off times of 10/10 µs.

# 500. Optional attachments

## 503. Pulsed OSL continued

The figure to the right, shows the results from a laboratory experiment in which dosed quartz (23 Gy) and undosed feldspar (0 Gy) were mixed together in various proportions. The mixed samples were measured in CW as well as POSL mode using the conventional blue LEDs and Hoya U-340 detection filters. In the presence of 20% of feldspar contamination the measured dose is within 5% of the true dose if only blue light POSL stimulation is used. If a bleach using the IR LEDs is used before POSL blue light stimulation then the measured dose is within 5% of the true dose when the feldspar contamination is as high as 40%.



Normalised dose as a function of feldspar contamination (by mass) in laboratory mixed quartz/feldspar samples. Results from CW, POSL and post-IR POSL are shown (Thomsen et al., 2006)

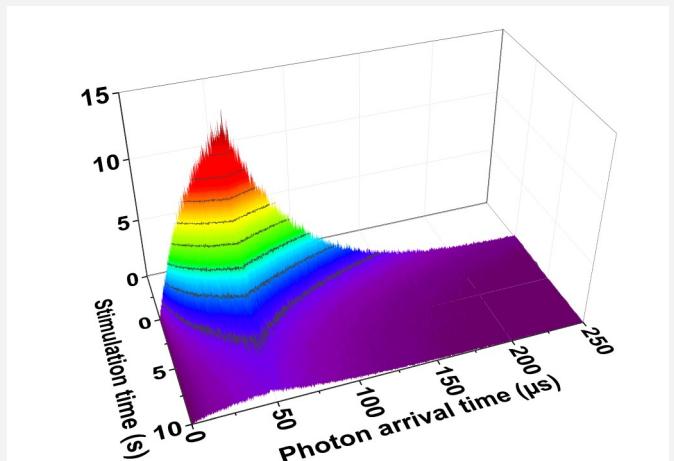
## 504. The Risø Time-Correlated Single Photon Counting (TCSPC)

All individual photons can be time-stamped using this TCSPC attachment based on the PicoQuant Time harp 260 board.

The TCSPC attachment is a PC plug-in board including software for doing Time-Resolved Pulsed OSL (TR-POSL) using the Pulsed OSL attachment. The board is mounted in a PCI-Express slot of a standard desktop PC which is also supplied (without monitor). The Pulsed OSL plug-in board furthermore will be equipped with an interface for the PC Photon Timer board. All detected photons are time-stamped with respect to the start of the preceding pulse. The time-stamp resolution is 0.25 ns.

Data acquisition is controlled by the Sequence Editor program, and the software program ‘PTanalyse’ is used for analysing the Photon Timer data, e.g. to determine photon arrival time distribution curves, OSL decay curves with different parameters settings and to export data for further analysis (Lapp et al., 2009).

- TCSPC Plug-in board
- PC



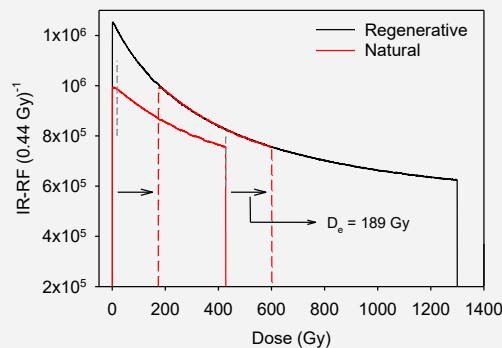
TR-POSL surface plot of a quartz sample with on-/off-time of 50/200  $\mu$ s.

# 500. Optional attachments

## 505. Radioluminescence

Radioluminescence (RL) from a sample can be measured during beta irradiation, using a light guide and a choice of PM tubes covering 180-890 nm or the high sensitivity spectrometer (Lapp et al., 2012).

A beta irradiator is modified to facilitate detection of luminescence emitted during irradiation (RL). This modification reduces the dose rate to the sample to about half of the dose rate achievable using the standard beta irradiation unit.



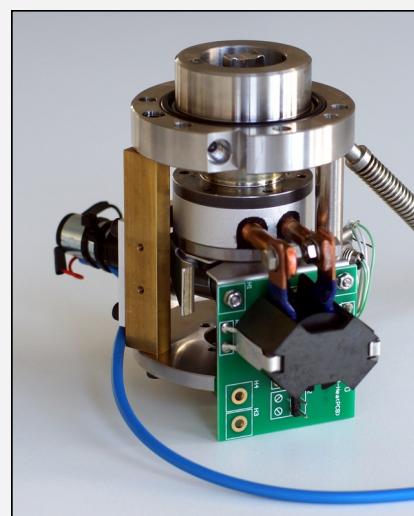
Principle of a fit of the additive dose response curve (solid red curve) onto the regenerative dose response curve (black) using *RLanalyse*; the horizontal displacement gives the  $D_c$  value. The vertical dashed grey lines indicate the region of the additive dose curve that was used for the fit. The best fit is shown using a dashed red line. (Buylaert et al., 2012)

- A Hamamatsu H7421-50 Photon counting head with spectral response 380 -890 nm, thermoelectrically cooled (see item 303)
- Lumatec liquid light guide (Transmission range of 350-2000 nm)
- Detection filter holder and Chroma D 900/100 interference filter (bandpass: 850-945 FWHM)
- Electronics for switching between counter input from RL PMT and standard PMT (built into the Controller)
- Power supply for the Hamamatsu H7421-50 Photon counting head and thermoelectric cooler (built into the Controller)
- A powerful UV LED (395 nm, 1000 mW) placed in a special bleaching position that may be used to bleach the samples during a sequence (see item 406)
- Dedicated software, *RLanalyse*, is supplied for analysing RL data.

## 506. Extra heating and lifting module

This module is mounted underneath the beta irradiator/Radioluminescence attachment and enables irradiation at elevated temperatures (from room temperature to 700 °C). The extra lift and heating module makes it possible to heat the sample during irradiation. The heating and lifting module is similar to the heating and lifting module in the standard measuring position (see item 105).

- Heating/lift unit enabling heating of individual samples up to 700 °C
- 30 kHz non-switching sine wave
- Type K thermocouple (Chromel-Alumel)
- cooling by a continuous gaseous flow
- linear heating rates from 0.1 to 10 °C/s.



The heating and lifting module.

# 500. Optional attachments

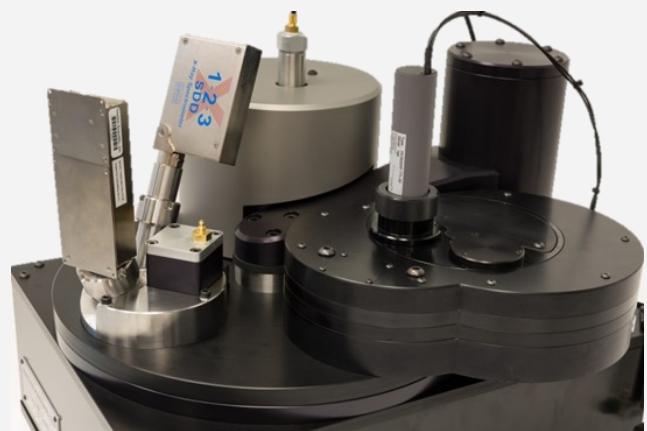
## 507. X-ray Fluorescence (XRF)

The relative proportion of K-/Na-/Ca-feldspar, and the fraction of quartz in each of the up to 48 samples on a measurement wheel can be measured using this XRF attachment which is based on an Amptek Spectrometer and an Amptek mini X-ray tube.

- Amptek X-123 SDD Complete X-Ray Spectrometer
  - \* Silicon Drift Diode (SDD)
  - \* Detector Area: 25 mm<sup>2</sup>  
(collimator area 17 mm<sup>2</sup>)
  - \* Detector Thickness: 500 µm
  - \* Detector Window: C1
  - \* 1.5" Detector Extension
  - \* Thermoelectric Cooler: 2-Stage  
(85° ΔT<sub>max</sub>)
  - \* Internal Multilayer Collimator)
- Amptek MINI-X Miniature X-Ray Tube
  - \* 50 kV/80 µA
  - \* Gold (Au) Transmission Target
  - \* High Voltage Power Supply
  - \* Beryllium end window
  - \* USB Controller
  - \* Molybdenum (Mo) sample cups  
(200 pcs)
  - \* Reference samples for making and maintaining the K-/Na-/Ca-feldspar calibration
  - \* Shielding and interlock features

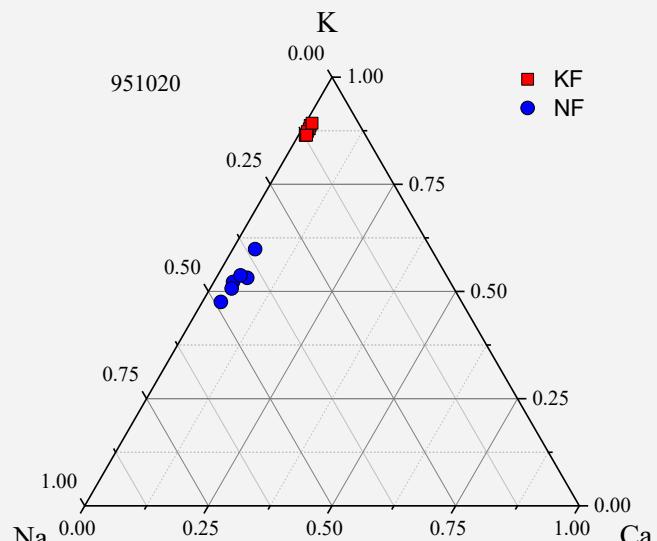
### Software:

A dedicated PC program for analysis of XRF data and converting this to position in the ternary diagram of K-/Na-/Ca-feldspar and giving estimated quartz contamination will be supplied. The standard data acquisition software "Sequence Editor" will include support for making data acquisition from the XRF unit.



XRF-attachment used in Guralnik et al., 2015, Porat et al., 2015

XRF measurements require vacuum (vacuum pump and vacuum accessories not included). The XRF spectrometer occupies the same position as the alpha irradiator (item 204), so the two units cannot be mounted simultaneously. The XRF and the single grain attachments (items 401-402) cannot be mounted simultaneously.



Ternary diagram showing the results for K-feldspar rich ( $\rho < 2.58 \text{ g cm}^{-3}$ ) and Na-rich extracts ( $2.58 \text{ g cm}^{-3} < \rho < 2.63 \text{ g cm}^{-3}$ ) measured with the Riso XRF-attachment. Six multi-grain aliquots were measured per extract. The 180-250 µm feldspar grains were extracted using conventional sample preparation techniques (HCl, H<sub>2</sub>O<sub>2</sub>, HF, heavy liquids) from sediment from Greenland.

# 500. Optional attachments

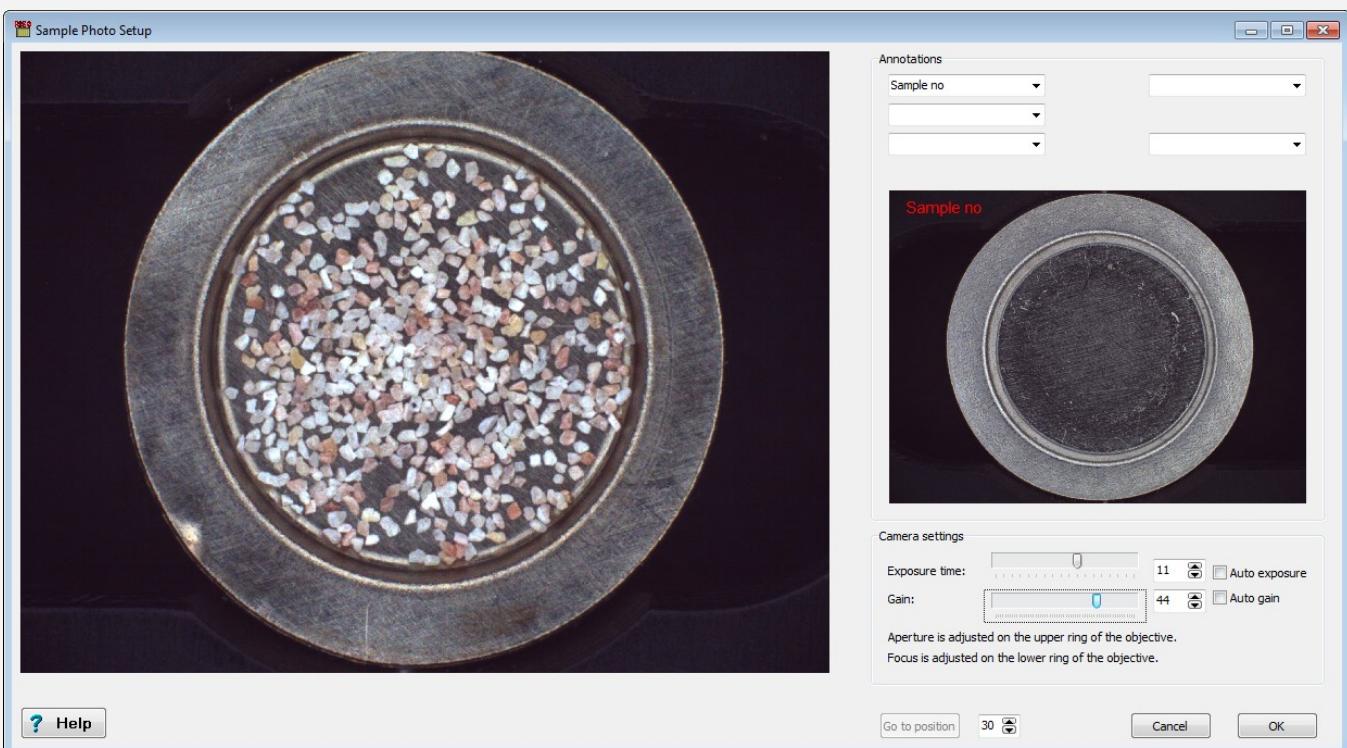
## 508. Sample camera

An optical image of a sample on the measurement wheel can be collected as part of the luminescence measurement sequence allowing evaluation of the number and spatial location of grains on a sample disc.

- The Imaging Source 1/2.5" Micron CMOS sensor, 2952×1944 (5 mega pixel)
- Computar f=25 mm, F1.8, ultra-low distortion lens
- Vacuum and light-tight mechanical fixture holding a diffuse light illumination system to ensure proper white light illumination of the samples that are photographed
- Driver for the illumination light (built into the Controller)
- The “Sequence Editor” allows collection of optical pictures during measurement sequences



Sample camera attachment mounted in front of the beta irradiator



Screenshot from the Sequence Editor showing an optical image taken using the sample camera attachment of a sample cup with sedimentary feldspar

# 500. Optional attachments

## 509. Single Detector IRPL Attachment

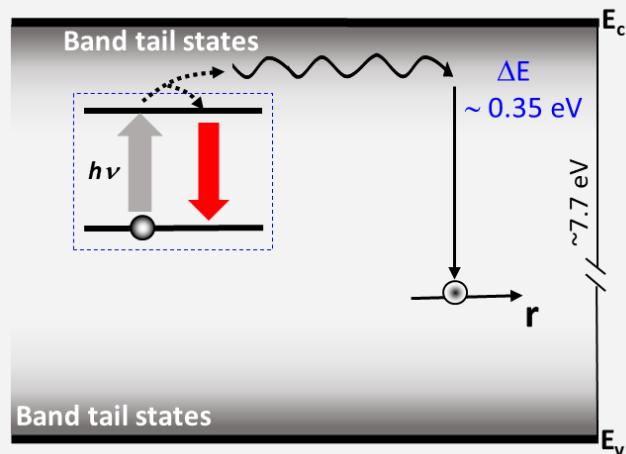
Enables routine Infrared photoluminescence (IRPL) measurements.

IRPL is a Stokes shifted emission resulting from the relaxation of the excited state of the principal (dosimetric) trap in Feldspar. Prasad et al. (2017) discovered an IRPL emission peak centered at ~955 nm using an 885 nm laser excitation and Kumar et al. (2018) discovered a second IRPL emission peak at ~880 nm using a shorter wavelength laser excitation at 830 nm.

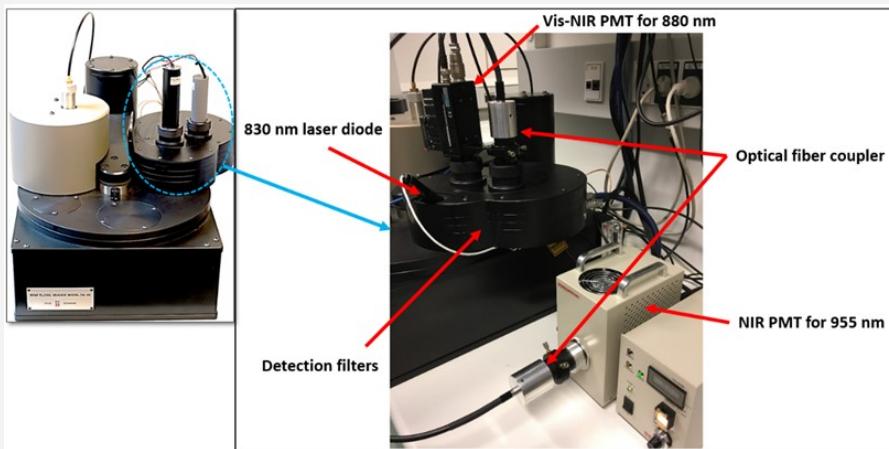
The Single Detector IRPL attachment uses an external 830 nm laser light source and a red sensitive PMT (Hamamatsu H7421-50) for detecting the 880 nm (1.41 eV) IRPL emission. The laser can be operated in either CW or pulsed mode using a control signal from DASH. The laser beam passes through a ground-glass diffuser to provide uniform illumination at the sample position. The PMT module is mounted directly on the DASH detector changer. A lens focuses the collimated IRPL beam from the sample to an effective measurement area. Pulsed IRPL measurement ensures a low background count rate by allowing the rejection of breakthrough from excitation light. It is possible to select a combination of detector and filters to measure IRSL and IRPL.

- Excitation: IR (830 nm) 140 mW TTL modulated laser and a diffuser
- Detection: Red sensitive Hamamatsu PMT - H7421-50 (380-890 nm)
- Detection filters: BP880+LP850 for the 880 nm IRPL emission

The *Automated detector changer* (item 501) and *Pulsed OSL* (item 503) are both prerequisite for the *Single Detector IRPL Attachment*.



Band diagram showing infrared stimulated luminescence (IRSL) and infrared photoluminescence (IRPL) emission processes from the principal trap. From Jain et al. (in prep.)



IRPL attachment to Risø TL/OSL reader (right figure). The standard DASH (left figure) is modified to include a 830 nm laser and a Vis-NIR PMT for detection at 880 nm. This picture also shows how a NIR PMT for 955 nm detection is included (see item 510 for further description).

# 500. Optional attachments

## 510. Dual Detector IRPL Attachment

Enables routine Infrared photoluminescence (IRPL) measurements.

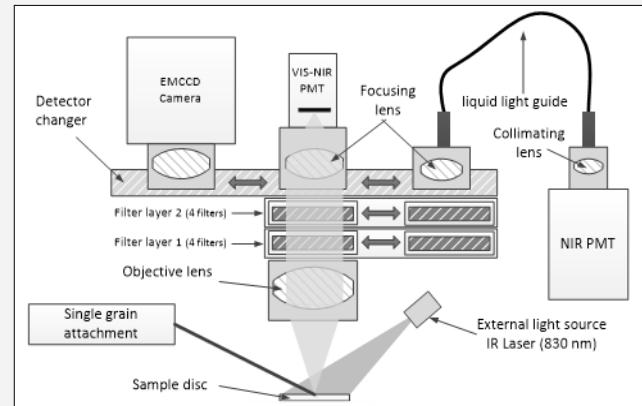
The dual detector IRPL measurement system uses an 830 nm external laser light source and two red/Near-IR sensitive photomultiplier tubes (PMTs) for detecting emissions at 880 nm (1.41 eV) and 955 nm (1.3 eV), respectively.

The 880 nm IRPL detection is the same as that described in item 509. For the detection of the 955 nm IRPL emission, the Hamamatsu H10330C-25, NIR-PMT detector is used; the detection module consists of the detector and the unit controller. The module is placed beside the Risø reader.

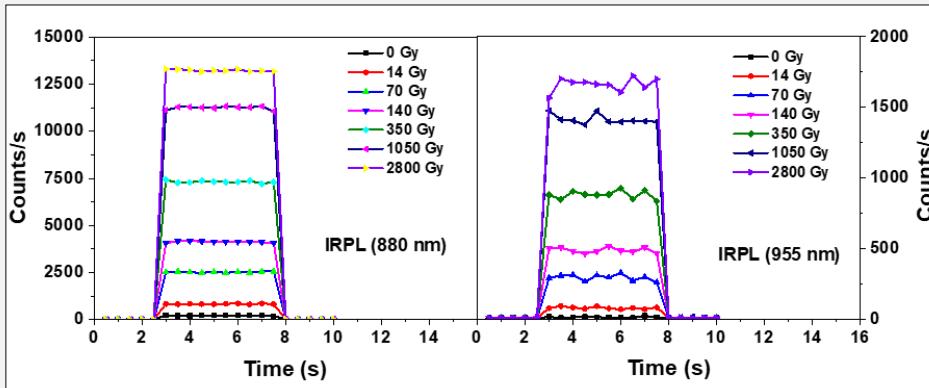
A 5 mm Ø liquid light guide (Lumatec Liquid Light Guide series 2000; transmission range of 350-2000 nm) is used to couple the DASH detector position with the NIR-PMT. The IRPL at the end of the liquid light guide is divergent (uncollimated); therefore, a lens is placed between the light guide and the detector to collect the IRPL photons impinging on the photocathode (18 mm effective Ø) of the PMT.

- Excitation: IR (830 nm) 140 mW TTL modulated laser and diffuser
- Detection: Hamamatsu, H7421-50 (380-890 nm)  
Hamamatsu H10330C-25 (950-1200 nm)
- Detection filters: BP880+LP850 for the 880 nm IRPL emission  
BP950 + LP925 for 955 nm IRPL emission

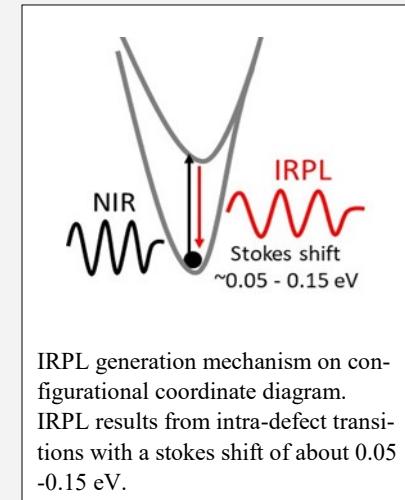
The *Automated detector changer* (item 501) and *Pulsed OSL* (item 503) are both prerequisite for the *Dual Detector IRPL attachment*.



Schematic diagram of the dual detector IRPL attachment mounted on the Risø TL/OSL Reader.



IRPL 880 nm (1.41 eV) and 955 nm (1.30 eV) emission signals in response to increasing regeneration dose. From Jain et al. (in prep.)



IRPL generation mechanism on configurational coordinate diagram.  
IRPL results from intra-defect transitions with a stokes shift of about 0.05 - 0.15 eV.

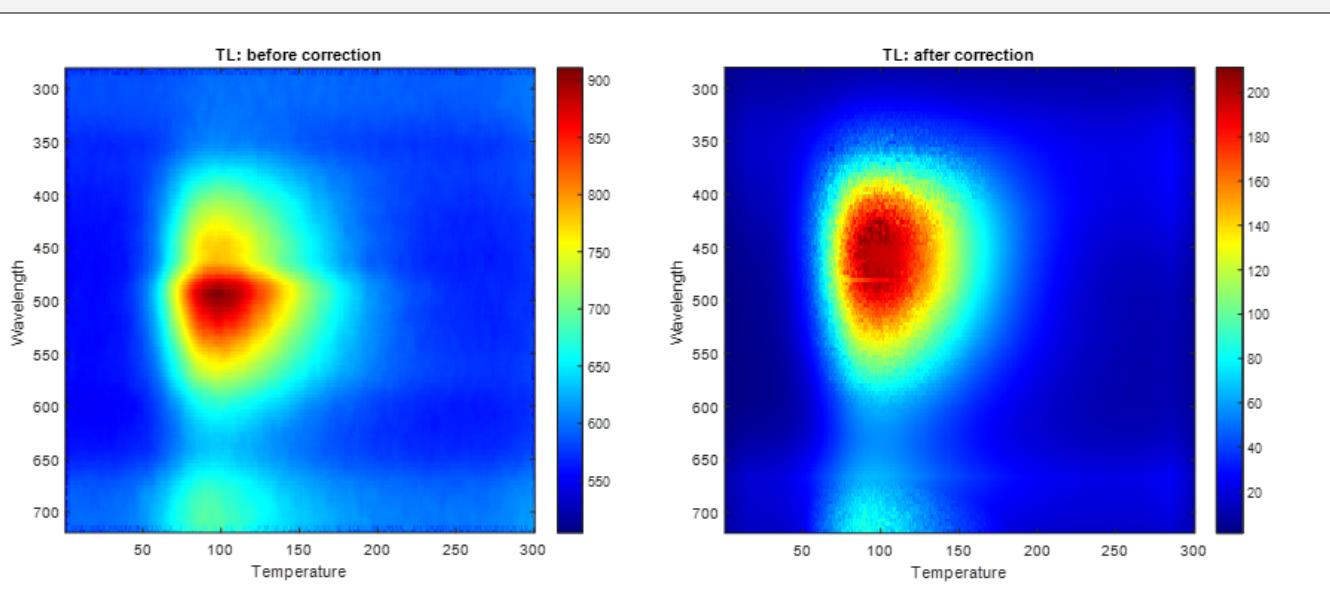
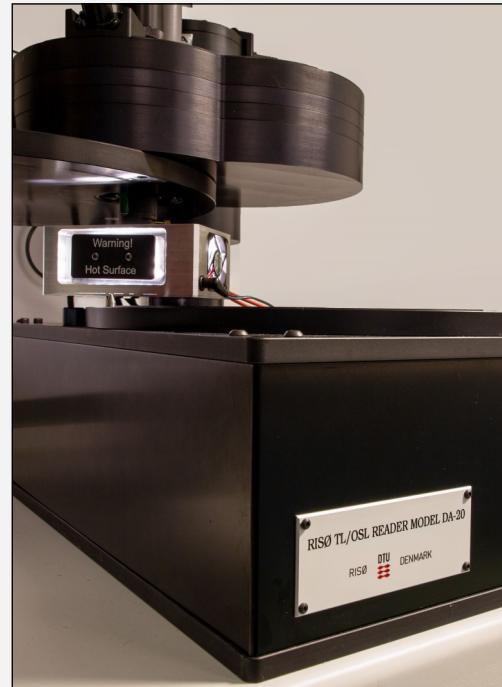
# 500. Optional attachments

## 511. Risø Spectrometer Calibration Attachment

In order to obtain emission spectra from the Risø Spectrometer (item 502), it is important that the instrument is calibrated to measure relative signal intensities at different wavelengths accurately in order to avoid distortion of the peak shape. The Risø Spectrometer Calibration Attachment enables relative efficiency correction for the different Risø spectrometer configurations, i.e. TL, RL, OSL and PL spectral measurements. The different configurations need to be calibrated individually because of differences in filters, optical fibre, grating, detector, etc. The figures below shows the effect of efficiency calibration on a TL spectrum.

The Risø Spectrometer Calibration Attachment uses a standard halogen lamp (CL2) and power supply (610 Current Stabilised Lamp Power Supply) from Bentham Instruments. The modified adaptor includes a light source, so the lamp can be attached to the Risø Reader directly.

- Grit-blasted quartz tungsten halogen lamp, G6.35 base
- Nominal lamp power and voltage: 100W, 12V
- Correlated colour temperature (typ.): 3200 K
- Calibration wavelength range: 250-1700 nm



# 600. Installation and Training

## 601./602. On location installation

Two-day installation, testing and commissioning by a DTU Physics representative on location at your laboratory. The availability of this option will depend on Danish authorities' recommendations for travel.



Unpacking the Reader

## 603. Training

DTU Physics and Aarhus University (through the Nordic Centre for Luminescence Research) jointly offer a two week course and training for one person in retrospective dosimetry using optically stimulated luminescence. The course will take place at DTU Physics's facilities at Risø, Roskilde, Denmark.

The course covers basic OSL theory and introduces the participants to the two main aspects of retrospective dosimetry: dose and dose-rate determination. The course consists of a series of lectures covering theory as well as practical exercises.

The participant will gain hands-on experience in operating the Risø TL/OSL reader; including installation and maintenance.

At the end of the two week course the participants should confidently be able to determine a luminescence age.

### Course Outline

#### **Basics of luminescence and OSL dating**

Choice of dosimeter material, mineral separation and dose response

#### **Installation and use of the Risø TL/OSL Reader**

Detailed description of how to install the reader, use of hardware and software, and instrument maintenance.

#### **Dose estimation**

The Single Aliquot Regenerative-Dose (SAR) protocol

#### **Origins and determination of dose rate**

Gamma spectroscopy and beta counting

#### **Age calculation**

Includes uncertainty analysis

#### **Dating of young sediments**

Problems and case studies

#### **Dating of old sediments**

Problems and case studies (age limits etc.)

#### **Rock surface dating**

Theory and case studies

Course fee includes course compendium, refreshments and lunches but not accommodation and transportation to and from Denmark.



Sampling for luminescence dating



DTU Physics, Risø Campus, Roskilde, Denmark

# 700. Accessories

## Calibration quartz

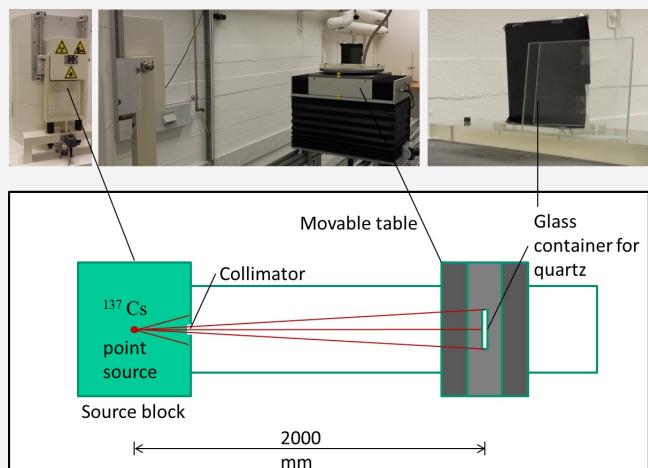
Sedimentary quartz sieved to 180 to 250 µm or 4-11 µm with usual initial preparation. Annealed at 700°C for one hour, given a ~2 kGy gamma dose and then annealed at 450°C for one hour. Stored in the dark prior to use. Packed into glass flat pack 100×100 mm, 1.9 mm wall thickness, 1 mm spacing between walls. Irradiated in the dark, normal to plane of pack (Hansen et al., 2015).

Dose in air =  $5.000 \pm 0.005$  Gy

*Attenuation in pack:* Assuming 0.3 mm of glass required to produce secondary electron equilibrium, remaining wall thickness (1.6 mm) attenuates the beam. For normal irradiation, assume half mean path length through packed quartz is 0.5 mm. This also attenuates beam.

Using 0.0770 cm<sup>2</sup>/g for quartz/glass mass attenuation coefficient at 662 keV, and density of 2.66 g/cm<sup>3</sup> (glass) and 2.0 g/cm<sup>3</sup> (packed quartz), gives overall attenuation factor of  $0.960 \pm 0.010$  (estimated error).

*Calculated dose to quartz:* Ratio of mass absorption coefficient of quartz to that of air is 1.0008 at 662 keV. Dose in quartz is thus  $5.000 \times 1.0008 \times 0.960$  Gy, i.e.  $4.81 \pm 0.07$  Gy.



## 701. Coarse grain calibration quartz

1 bag of calibration quartz, ~0.25 g, 4.81 Gy, 180-250 µm

Also included is an undosed portion of the sample which can be used for dose recovery testing.

## 702. Fine grain calibration quartz

1 bag of calibration quartz, 0.25 g, 4.81 Gy, 4-11 µm

Also included is an undosed portion of the sample which can be used for dose recovery testing.



Other calibration materials (e.g. solid quartz slices 1 mm thick, 9.7 mm diameter) are under development.

DTU Physics can, in some circumstances, also arrange custom irradiation of non-standard materials/geometries. Please enquire at [osl@ntech.dtu.dk](mailto:osl@ntech.dtu.dk) for further details.

# 700. Accessories

## Detection filters

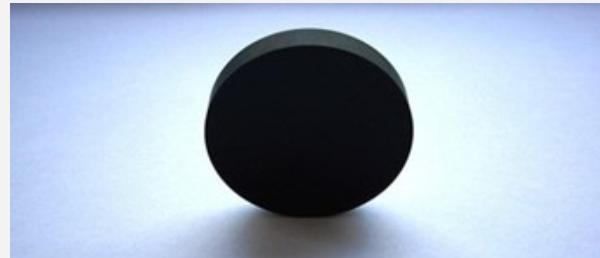
The intensity of the stimulation light is  $\sim 10^{18}$  orders of magnitude larger than the emitted luminescence. In order to be able to measure the emitted luminescence, detection filters must be used to prevent scattered stimulation light from reaching the PMT, and the spectral stimulation and detection windows must be well separated.

### 703-705. U-340 detection filter

Band pass filter transmitting in the ultraviolet. Transmission centred on 340 nm (FWHM  $\sim 70$  nm).

- 703    t7.5 mm     $\phi=45$  mm    (classic OSL head)
- 704    t5 mm       $\phi=25$  mm    (DASH)
- 705    t2.5 mm      $\phi=25$  mm    (DASH)

Quartz OSL is often detected using the Hoya U-340 filter. Transmittance curve is given in item 103.

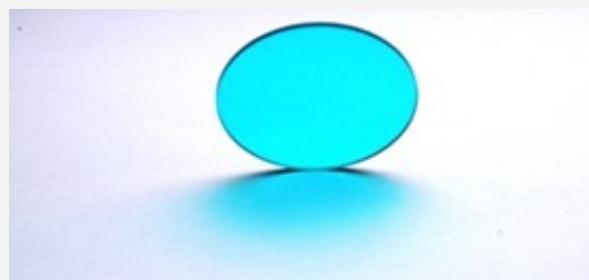


### 706-707. Schott BG-39 detection filter

Ionomically coloured glass, Band pass filter centred on  $\sim 500$  nm (FWHM  $\sim 260$  nm)

- 706    t2 mm       $\phi=45$  mm    (classic OSL head)
- 707    t2 mm       $\phi=25$  mm    (DASH)

Quartz TL is often detected using the BG-39. Transmittance curve is given in item 103

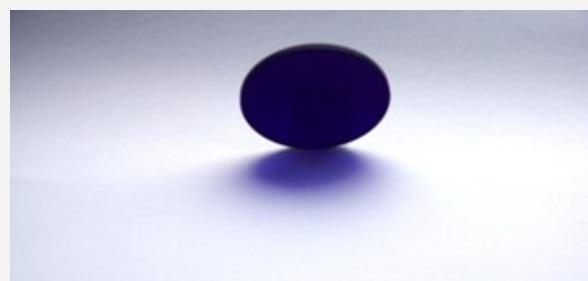


### 708-709. Schott BG-3 detection filter

Ionomically coloured glass, Band pass filter centred on  $\sim 350$  nm (FWHM  $\sim 190$  nm)

- 708    t3 mm       $\phi=45$  mm    (classic OSL head)
- 709    t3 mm       $\phi=25$  mm    (DASH)

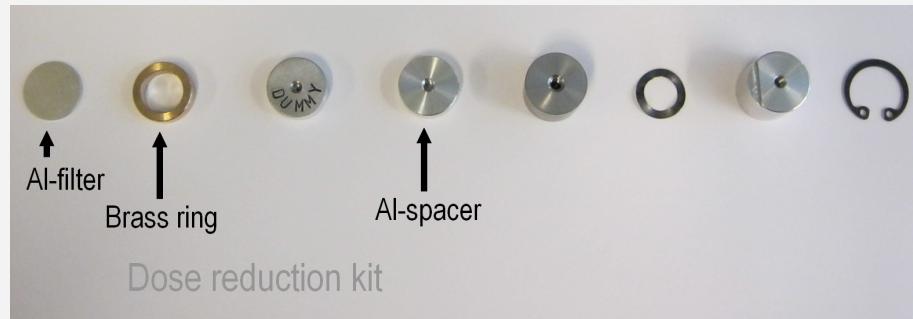
Feldspar IRSL is often detected using the BG-3 in combination with BG39. Transmittance curve is given in item 103



# 700. Accessories

## 710. Dose reduction kit

The dose reduction kit reduces the dose rate of the Sr-90 beta source by a factor of ~10. The kit consists of rings and spacers and is inserted into the source module before the beta source.



## 711. PC and monitor

Personal computer with Windows and special application software installed, monitor and UK keyboard

Note: The Risø Reader is run using a standard PC, which is not automatically included in the Reader configuration, except when explicitly mentioned. It can be supplied by the customer or purchased through DTU Physics.

## 712. Piezoelectric ultrasonic cleaner

Ultrasonic scaler for cleaning of single grain discs in order to release trapped grains from sample holes.

Output tip vibration frequency:  $30 \pm 3$  kHz

Output half-excursion force: <2N

Output primary tip vibration excursion:  $\leq 100\mu\text{m}$

Holder for 24 single grain discs is provided



# 700. Accessories

## Sample holders

Samples are either mounted on 9.7 mm diameter flat discs (stainless steel or aluminium) using silicone oil as an adhesive or poured (as loose grains) into sample cups ( $\phi=11.7$  mm; stainless steel or aluminium). Sample holders are loaded onto an exchangeable sample carousel (items 718, 719) that can accommodate up to 48 sample holders.

### 713. Stainless steel sample cups

11.7 mm diameter stainless steel sample cups

### 714. Aluminium sample cups

11.7 mm diameter aluminium sample cups

### 715. Stainless steel sample discs

9.7 mm diameter stainless steel sample discs

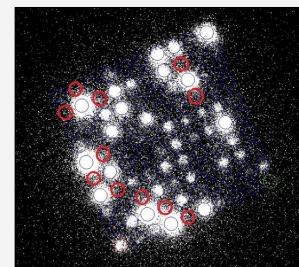
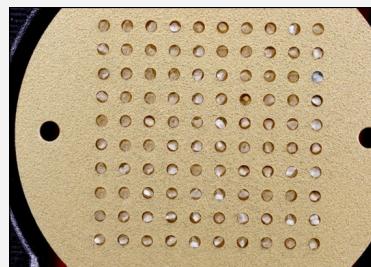
### 716. Aluminium sample discs

9.7 mm diameter aluminium sample discs



## 717-721. Single grain discs

The aluminium sample discs designed for mounting single grains are 1 mm thick and have a diameter of 9.7 mm (i.e. same surface area as the conventional sample discs). The individual grains are placed in 100 holes drilled into the surface of the sample discs. These holes are  $x \mu\text{m}$  deep by  $x \mu\text{m}$  in diameter ( $x=100, 150, 200, 250$  or  $300$ ) on a 10 by 10 grid with  $600 \mu\text{m}$  spacing between hole centres.

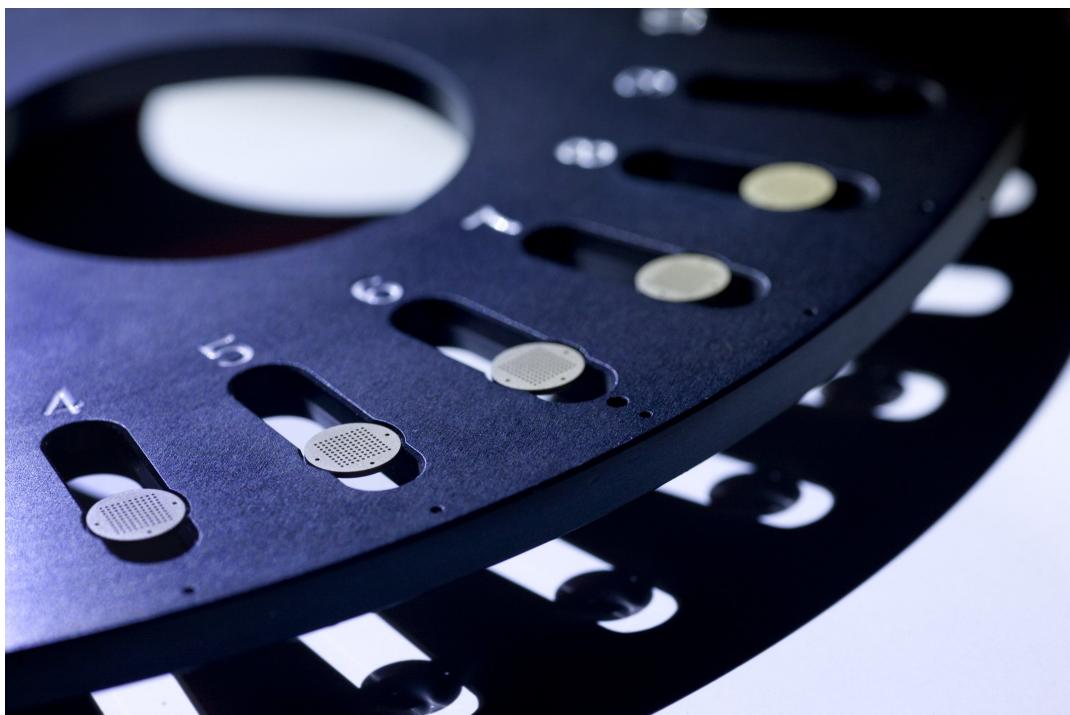


Standard single grain discs with depth and diameter of  $300 \mu\text{m}$ . Picture on the right shows blue stimulated quartz OSL recorded with the EMCCD option (item 304; Thomsen et al., 2015).

# 700. Accessories

## Sample carrousel

Samples are loaded onto an exchangeable sample carrousel ( $\varnothing=306$  mm) that can accommodate up to 48 samples. Two types of sample carrousels are available: one for flat sample discs ( $\varnothing=9.7$  mm) and one for sample cups ( $\varnothing=11.7$  mm)



## 722. Disc carrousel

Exchangeable sample carrousel for flat 9.7 mm sample discs (items 715-721)

## 723. Cup carrousel

Exchangeable sample carrousel for 11.7 mm sample cups (items 713, 714)

## 724. Spray masks

When mounting sample grains using e.g. silicone oil as an adhesive (e.g. to ensure that the grains are presented in a monolayer), the spot size should be controlled.

Set containing one base plate and two masks with holes with diameters of 2 and 8 mm, respectively.



# 700. Accessories

## 725. Vacuum pump

Leybold TRIVAC® B Rotary Vane Vacuum Pump D 4 B with an AR 4-8 Exhaust filter. The Risø TL/OSL reader enables measurements of both TL and OSL in vacuum (down to 0.2 mbar), but a vacuum pump is not included in the standard configuration. If the Risø TL/OSL reader is equipped with an alpha source or XRF attachment, the measurement chamber should be evacuated before alpha irradiation/XRF measurement is undertaken.



## 726. Vacuum accessories

Relay box and vacuum extension tube (1 m)

The Relay box is necessary for automatic software control of atmosphere in sample chamber as is the flexible steel vacuum extension tube which is used to connect the vacuum pump to the Risø TL/OSL Reader



## 727. Risø Safelight

### Safelight for luminescence preparation and measurement laboratories

Many luminescence minerals are sensitive to light, and it may be important that any inherent signal is preserved during sample processing and loading into the reader. The Risø Safelight is designed to meet this objective, particularly when used with quartz and feldspar. It is centered on 594 nm, a wavelength chosen to maximize human eye sensitivity, and at the same time reduce the effect on the trapped charge giving rise to quartz fast-component OSL and feldspar IRSL.

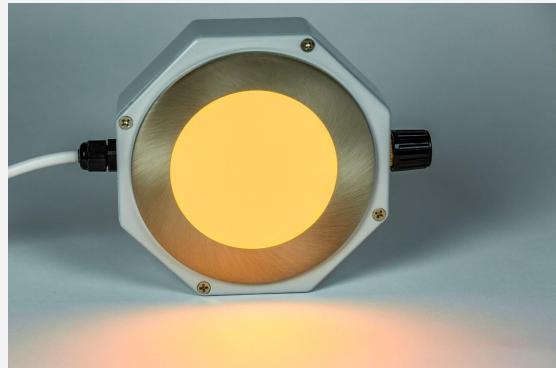


# 700. Accessories

## 727. Risø Safelight continued

### Specifications:

- Wavelength: 594 nm (-9 nm, +6 nm)
- Output power: from < 0.3  $\mu\text{W}/\text{cm}^2$  up to > 6  $\mu\text{W}/\text{cm}^2$  at 0.5 m, user adjustable.
- Input voltage: 100-240 V AC. The unit must be connected to earth/ground.
- Input power max: 3W with min adj. 0.3W



Note: The safelight is approved and marked under the relevant CE (Conformité Européenne) standard, but this only gives certification for use as a light source within Europe. Use of the safelight outside this region is not expected by the manufacturer, and takes place at the user's own risk.

### Mounting and connection:

The number of lamps required depends mainly on the desired uniformity of illumination. According to Sohbati et al. (2017, ISSN:1871-1014) the maximum power density at 594 nm for a 48 hour exposure of quartz or feldspar should be ~0.2  $\mu\text{W}/\text{cm}^2$ , if undesirable bleaching of the luminescence signal is to be kept below 1%.

In our experience, satisfactorily uniform room illumination (with lamps mounted on a 2 m high ceiling) can be achieved with 1 lamp per  $\sim 10 \text{ m}^2$  of floor area. For illumination of a bench top (where more uniform lighting may be required) we have found 1 lamp per meter of bench at a height of 1 m is satisfactory.

The safelights are designed to be mounted flat (e.g. on ceiling) using the flat metal bracket provided. Electrical connection is through a standard IEC C14 socket fitted on a 1 m long flying lead (note this expects an earth/ground wire).

Important: The safelight is not designed to be resistant to laboratory chemicals. If used for fume cupboard illumination they must be mounted outside the active volume, and illuminate through a plastic/glass window.

### Measuring power density:

Each safelight is fitted with a user-adjustable control with which the output can be adjusted up to 100% of full power. The correct power setting is dependent on the position of the lamp, and the length of time samples are likely to be exposed (discussed in Sohbati et al., 2017; ISSN:1871-1014). We recommend the use of a suitable power meter, calibrated at 594 nm (e.g. Thorlabs PM160) to ensure that the desired maximum power density of ~0.2  $\mu\text{W}/\text{cm}^2$  at the sample position is not exceeded.

# 800. Environmental dose rate instruments

## 801. Ultra-low-level Beta GM Multicounter

The Risø ultra-low-level beta multi-counter is intended for counting of very low activity samples. It is based on five flow-through Geiger-Müller pancake detectors (each with a detection efficiency of ~45%) and a common guard counter. The guard counter reduces the cosmic-ray background using anti-coincidence techniques. The system can count up to five samples simultaneously.

The low activity materials used to make the detectors (electrolytic copper and plastic), together with the anti-coincidence cosmic ray guard detector, give a background of <0.2 cpm when operated inside 10 cm of lead shielding.

Optimum high voltage is set automatically by the dedicated software, which also controls counting times and provides statistical analysis. Calibration and automated high voltage setting employs five  $^{99}\text{Tc}$  <125 Bq sources of known activity (provided).

A lift mechanism minimizes the sample-to-window distance to obtain optimal efficiency. Samples are lifted into counting position using a manual slide; they can be up to 7 mm thick and freestanding or mounted on nylon sample supports.

Typical applications include analysis of environmental samples for contamination, measurement of  $^{234}\text{Th}$  in marine sediments and  $^{40}\text{K}$  in feldspar extracts.



The beta counter is shown above with two sample slides (green and black) and one slide lift (red), but without the lead shield and electronics box. The anti-coincidence shield lies under the copper plate. Six samples are shown mounted on the slides for counting, and five  $^{99}\text{Tc}$  standards are in the separate box (yellow lid). External dimensions: 100 × 50 × 250 mm (counter), 250 × 65 × 250 mm (control box; not shown).

### 5-sample GM gas-flow multicounter unit

5-sample gas-flow multicounter unit, each counter window with a diameter of 25 mm and a density < 1 mg/cm<sup>2</sup>, with common guard counter system, sample slide for five samples and lift mechanism, valve for fine adjustment of gas flow as well as bubble chamber for gas flow control. Module size is 50×100×300 mm which fits into a lead shielding made of standard lead bricks .

### Electronics

Electronic system comprising six complete counter channels (five samples and one guard) that measure simultaneously. Each counter channel incorporates preamplifier, discriminator, anticoincidence gate, pulse amplifier and six decade scaler/timer. LED indicators are provided for all counter channels for visual control. Electronics further include low and high voltage supplies and USB interface (to allow control by portable PC).

# 800. Environmental dose rate instruments

## 801. Ultra-low-level Beta GM Multicounter continued

### Software

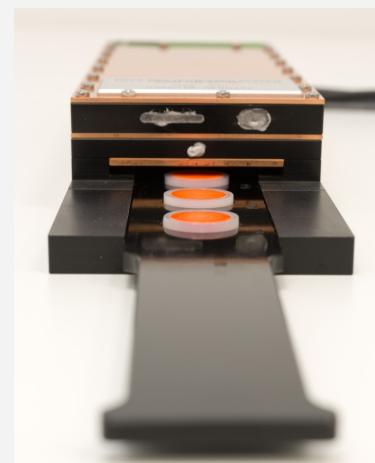
WINDOWS software to support USB interface to provide automatic high voltage plateau control (pulse height analysing system), master clock/timer system and display of measurement results including calculated counting statistics on the computer screen. The software is compatible with the IAEA standard desktop software environment.

### Sample holders

100 pieces of nylon sample holders (disc and ring) made according to Health and Safety Laboratory procedures manual HASL-300 USAEC.

### Calibration

5 pieces of absolute calibrated Tc-99 sources (<125 Bq) for stability testing.



Beta counter with sample slide partially inserted.

## 802. Lead shielding for the ultra-low-level Beta GM Multicounter

Complete 100 mm thick lead shielding made up of low contaminated lead bricks.

Note that two specifically machined lead-fittings for the ends of the counter is included in item 801 and do not need to be ordered separately.



Beta counter is mounted in a lead castle. A sample slide is partially inserted into the counter.

## 803. Sample holders for the ultra-low-level Beta GM Multicounter

Nylon sample holders (disc and ring) made according to Health and Safety Laboratory procedures manual HASL-300 USAEC.



Sample holder for the beta counter

# 800. Environmental dose rate instruments

## 810. Risø gamma spectrometry system

The Risø scintillation gamma spectrometry system is intended for routine analysis of natural radionuclide activity concentrations for use in dosimetry calculations in luminescence age determination.

The system includes detector, dedicated analytical software and calibration standards.

This simple scintillator-based spectrometry system is a useful laboratory method for accurate and precise determination of burial dose rates at a significantly lower cost than high resolution gamma spectrometry. This, combined with the large (and so more representative) sample size, makes it a strong competitor to other analytical methods used in OSL dating.



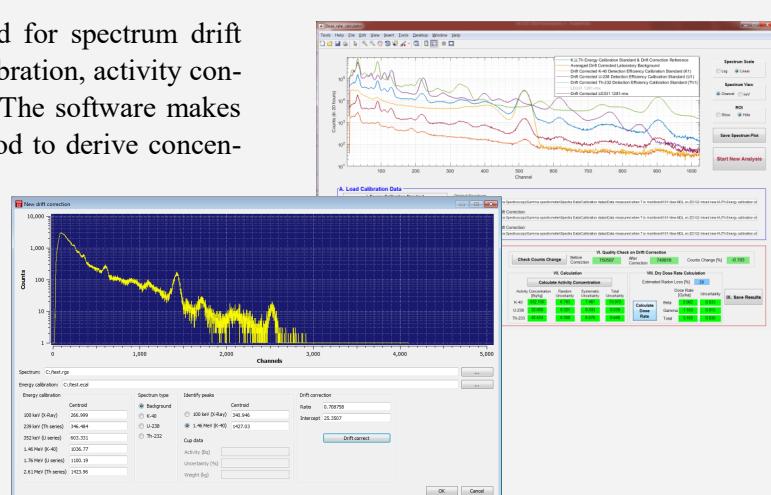
The picture shows the detector placed in the holder with a sample in place for counting.

### Detector

The system is based on a 3"×3" NaI(Tl) crystal connected to a digital tube base consisting of a digital pulse processor, a charge sensitive preamplifier, a multichannel analyser (MCA) and power supplies. The detector and MCA are all contained within a 10 cm thick lead shielding.

### Analysis software

The Risø *DoseRateAnalyzer* software is used for spectrum drift corrections,  $^{40}\text{K}$ ,  $^{238}\text{U}$  and  $^{232}\text{Th}$  efficiency calibration, activity concentration analysis and dose rate calculation. The software makes use of the full spectrum analysis (FSA) method to derive concentration activities. This approach uses statistical information available in the entire spectrum, and so is considerably more sensitive than the more usual 3-window approach. The minimum detectable activity concentrations, for cups containing ~250 g sample, are 5.8 Bq/kg ( $^{40}\text{K}$ ), 0.47 Bq/kg ( $^{238}\text{U}$ ) and 0.37 Bq/kg ( $^{232}\text{Th}$ ).



# 800. Environmental dose rate instruments

## 810. Risø gamma spectrometry system continued

### Calibration standards

A set of 11 wax impregnated calibration standards are supplied with the system, including 3 sets of individual  $^{40}\text{K}$ ,  $^{238}\text{U}$  and  $^{232}\text{Th}$  standards (9 cups in total), a mixed K, U, Th sample and a background sample. These are all cast in a cup-shaped geometry using the same aluminium mould (also supplied). When inverted, these cups fits over the top of the NaI(Tl) detector.

Uranium and thorium standards are prepared by diluting certified reference material BL-5 ( $7.09 \pm 0.03\%$  U, NRCAN-1<sup>1</sup>) or OKA-2 ( $2.893 \pm 0.058\%$  Th, NRCAN-2<sup>2</sup>) in low activity quartz sand, and then mixing with high viscosity wax (Bottle wax, blend 14944, British Wax Refining Company) to give individual parent activities of  $\sim 800$  Bq/cup. For potassium standards, analytical grade  $\text{K}_2\text{SO}_4$  (14.20 Bq/g assuming stoichiometry, purity given as 100.4%) is mixed directly with the wax to give  $\sim 2,700$  Bq/standard. Following a strict protocol, these standards have been carefully prepared in separate batches to achieve a dispersion as low as 1%, so the user can include accurate estimates of uncertainties in their uncertainty budgets.

The mixed KUTH sample (prepared by diluting BL-5 and OKA-2 in  $\text{K}_2\text{SO}_4$  to give a sample cup containing  $\sim 2700$  Bq  $^{40}\text{K}$  and  $\sim 70$  Bq  $^{238}\text{U}$  and  $^{232}\text{Th}$ ) is used as a standard reference for drift correction of all spectra from unknown samples as well as the calibration standards.

The background sample cup is cast with pure wax and is included to ensure that the background is measured in the same manner as the calibration standards.

### Specifications

- 3"×3" NaI(Tl) detector with digital MCA, resolution (FWHM) 6.3% at 661 keV ( $^{137}\text{Cs}$ )
- Plastic detector holder to support the detector
- *Risø DoseRateAnalyzer* software
- Aluminium casting mould
- Calibration standards
- 3  $^{40}\text{K}$  standards
- 3  $^{238}\text{U}$  standards
- 3  $^{232}\text{Th}$  standards
- 1 KUTH mixture as spectrum correction standard
- 1 background sample



# 800. Environmental dose rate instruments

## 811. Risø aluminium casting mould

Aluminium mould for casting samples cups. This mould is identical to the mould used for casting the calibration standards supplied with the Risø gamma spectrometry system.

When users prepare their own samples, it is highly desirable to use a casting mould with the same shape and dimensions as that of the K, U Th efficiency calibration standards in order to minimise potential uncertainties arising from differences in shape and dimension between sample and calibration standards.

Samples should be ground to <200 µm and mixed with high viscosity wax at a typical mass ratio of 1:2 (wax:sample), to give a typical sample weight of 250-300 g.

Note: one mould is supplied as standard with the Risø gamma spectrometry system. Users can order more moulds if they wish to prepare samples in batches.



## 812. Lead shield for the Risø gamma spectrometry system

Complete 100 mm thick lead shield made up of low contamination lead bricks. The lead shield is supplied with a sliding lid for ease of access to the measurement chamber when loading/unloading samples.

Note:

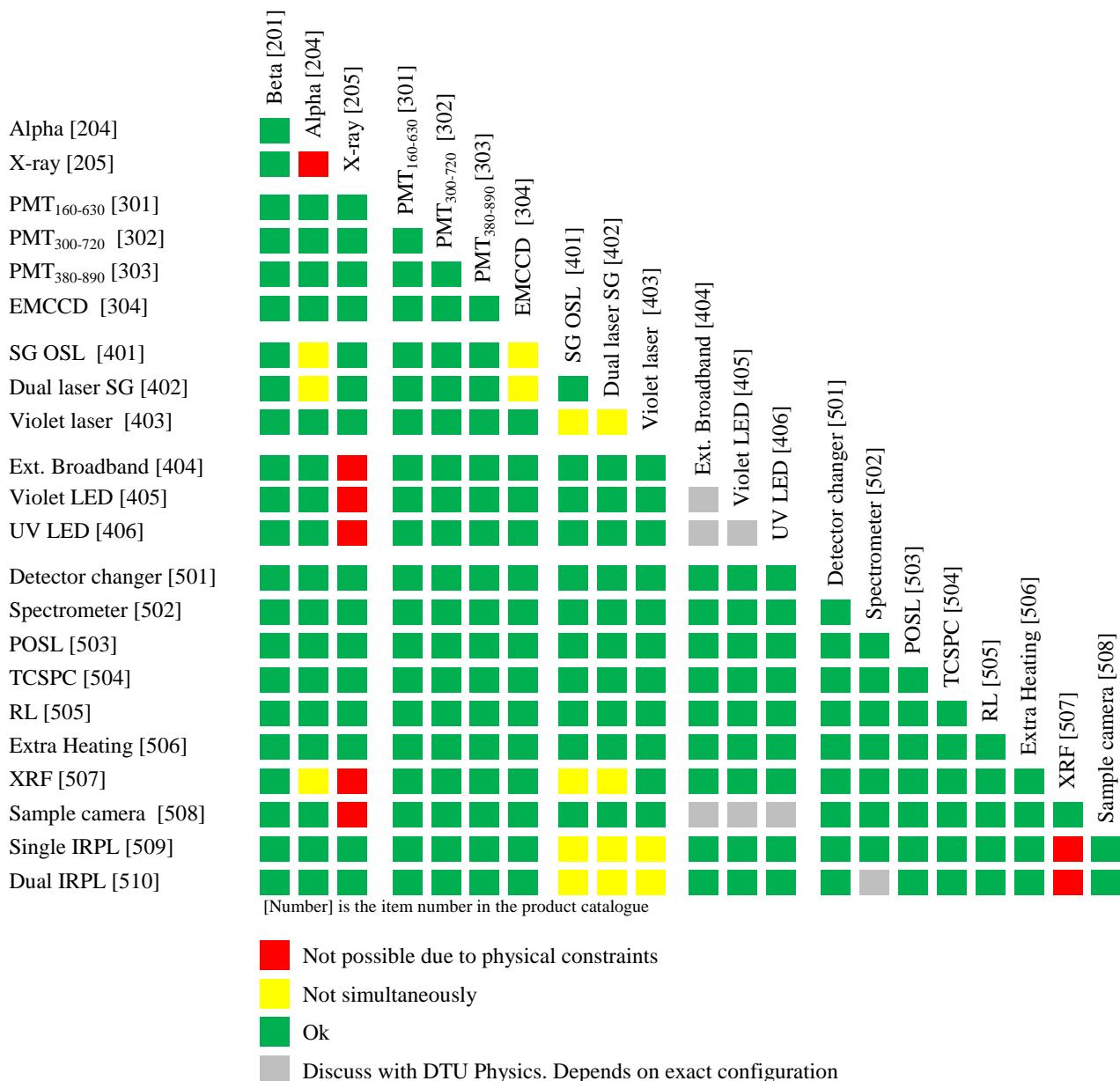
1. The sliding lid will be shipped separately from the lead shield itself.
2. One lead brick has a special slot to allow a USB cable to pass through the lead shield. Please follow the supplied lead shield assembly instruction carefully when positioning this special lead brick in the lead shield.



The picture shows the double lead shield assembly at DTU Physics. Note: Only a single lead shield with a matching sliding lid is supplied

# Combination chart

Guide to potential combinations of options for the Risø TL/OSL Reader



Note that this chart assumes no more than two options on a Risø TL/OSL Reader. In most cases more will be possible