CHAPTER - 3
EXPERIMENTAL DETAILS

Thermally Stimulated Luminescence (TSL) and Optically Stimulated Luminescence (OSL) have wide applications in dating and dosimetry apart from many other areas of research. Now a days, an advance version of thermoluminescence and optically stimulated luminescence recording systems are available. The unit consists of a sample holder cum heater, temperature controller unit, stimulation source (for OSL study) and light detection system etc. The details of TL/OSL system used for present investigations are explained systematically in this chapter.

3.1 Heating System

In TL dating and retrospective dosimetry using natural material, it is important to heat samples at constant rate in order to get a temperature-resolved glow curve for identification of peak temperature (electron trap). Linear heating is normally performed using a low-mass heater strip made of high resistance alloys e.g. nickel and kanthal (kanthal contains Chromium-23 %, Iron-72%, Aluminium-3% and Cobalt-2%) and feeding a controlled current through the ohmic heating element. Feedback control of the temperature is achieved using a thermocouple (e.g. Cr/Al) spot welded to the heater strip **(Fig-3A)**.

Normally, heating is controlled by an electronic ramp that can generate various pre-heat functions and linear heating rates (e.g. 0.1-30°C/ Sec). The maximum temperature normally used for quartz and feldspar dating is 500°C but for special investigations of deep traps effects temperature up to 700°C have been used.[1] Another heating systems are used for readout of conventional solid TL dosimeters in radiation protection. The dosimeters can also be lifted into a stream of hot nitrogen (300°C-400°C) and TL signal released during the resulting non-linear heating.[2] A CO_2 laser

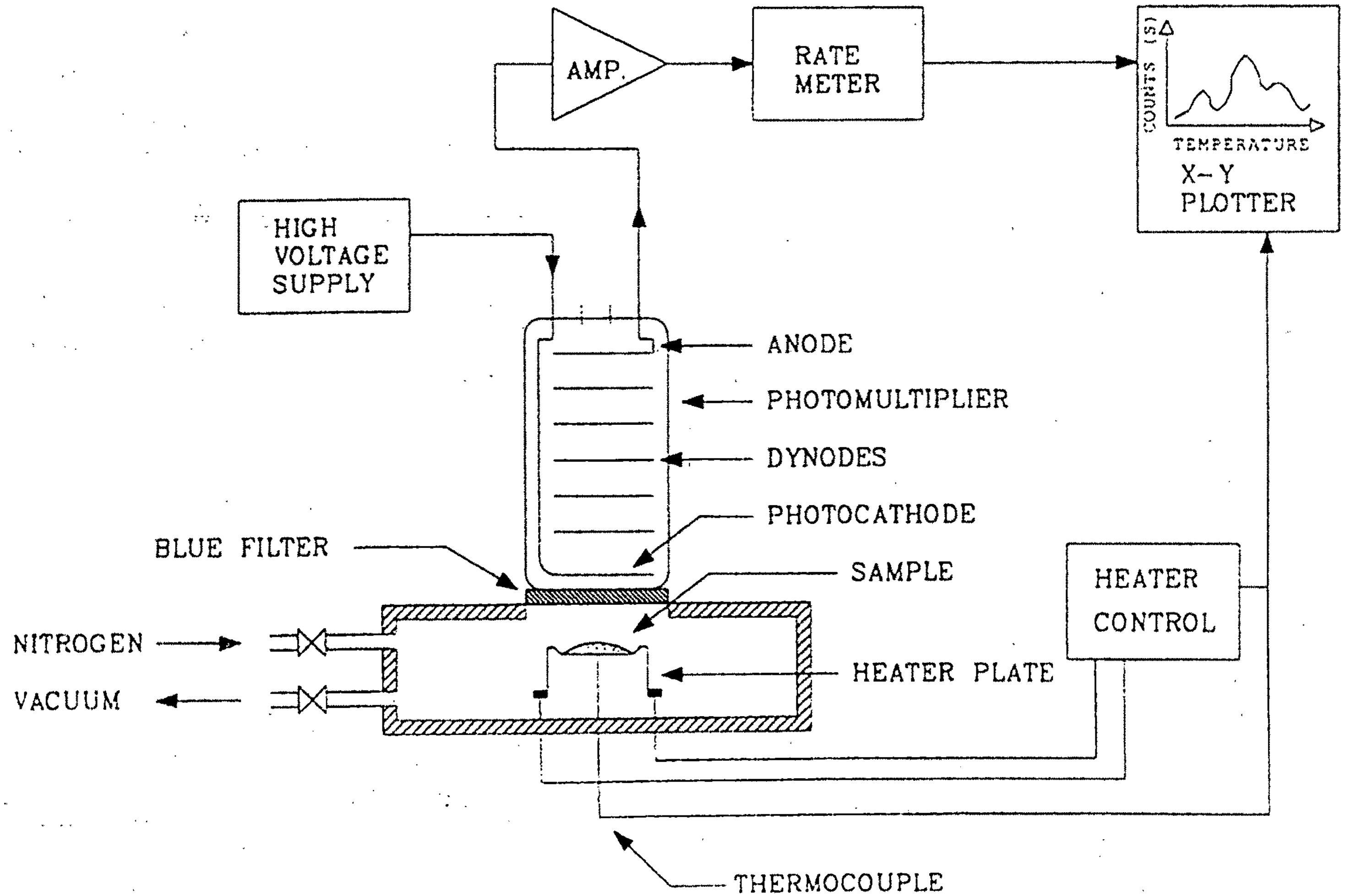


Fig-3A: A schematic diagram of a modern TL reader system.

beam has also been provided for the non-linear heating of solid TL dosimeters.[3]

3.2 Optical Filters

A limiting factor in TL measurements is the thermal background signal arising from the heating element and sample during heating to high temperature i.e. black body radiation. In order to distinguish low TL signals it is important to use blue filters in combination with heat-absorption filters to suppress the thermal background signal. Typical blue filters used in TL dating routines are Corning 7-59 and Schott UG-11 filters, and an efficient heat-absorbing filter is Pilkington HA-3. It should be noted that the Schott UG-11 filter has a near-infrared transmission window, which is the reason it cannot be used alone for either TL or infrared stimulated luminescence. An additional filter is needed to suppress the blackbody radiation e.g. Schott-BG-38 or BG-39 filters. The transmission characteristics of these filters are shown in **Fig-3B** and **Fig-3C**. All the TL/OSL measurements reported in present work are carried out with the help of RISO automated TL/OSL system (model TL/OSL-DA-15). The entire experimental setup along with the data acquisition system is shown in the photograph.

3.3 RISO Automated TL/OSL Systems

The automated RISO TL/OSL reader was first introduced in 1988.[4] Since then RISO has continuously upgraded its reader and today it is one of the world's best high sensitive, high precision luminescence (both TL as well as OSL) reader. RISO TL/OSL systems have been used worldwide for measuring TL/OSL dating, retrospective dosimetry, environmental dosimetry and material characterization. In response to the Optically

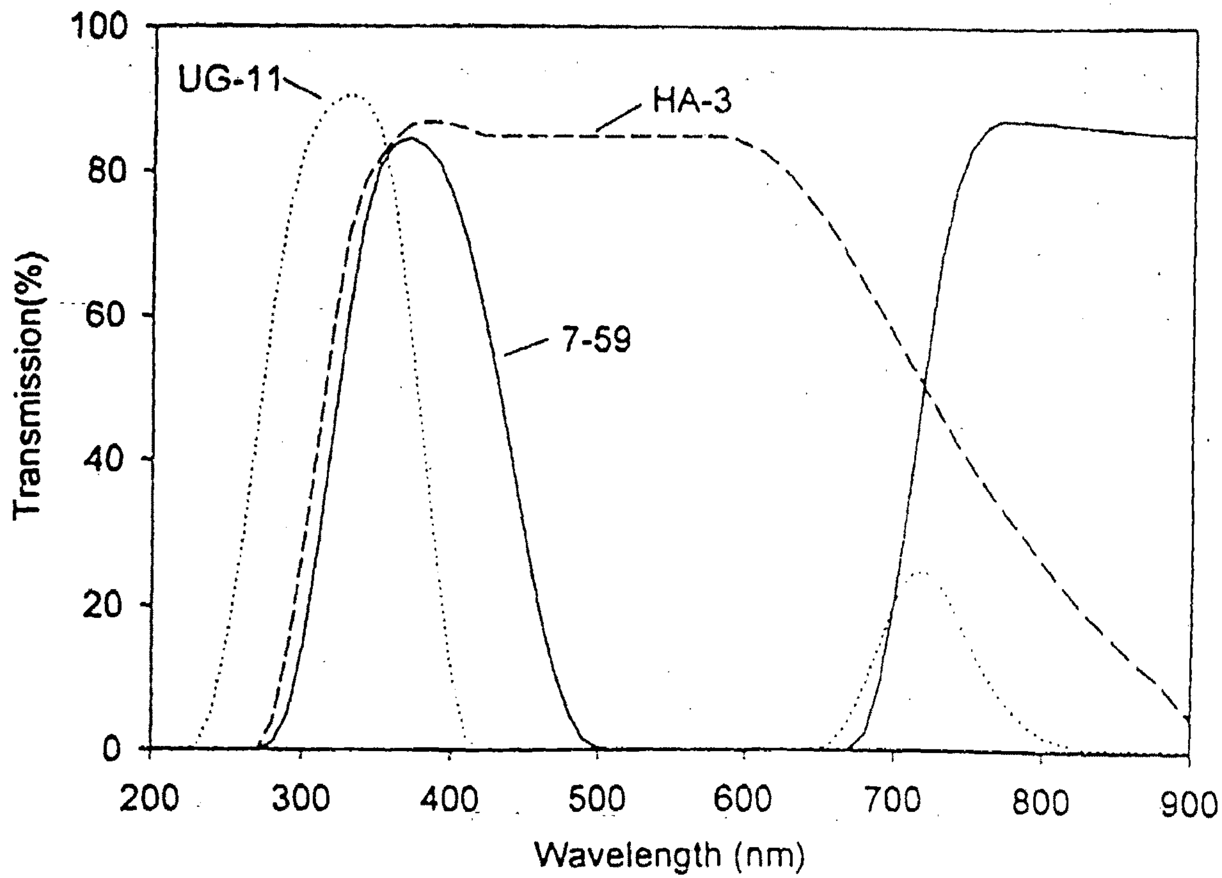


Fig-3B: Transmission characteristics of Corning 7-59, Schott UG-11 and Pilkington HA-3 filters.

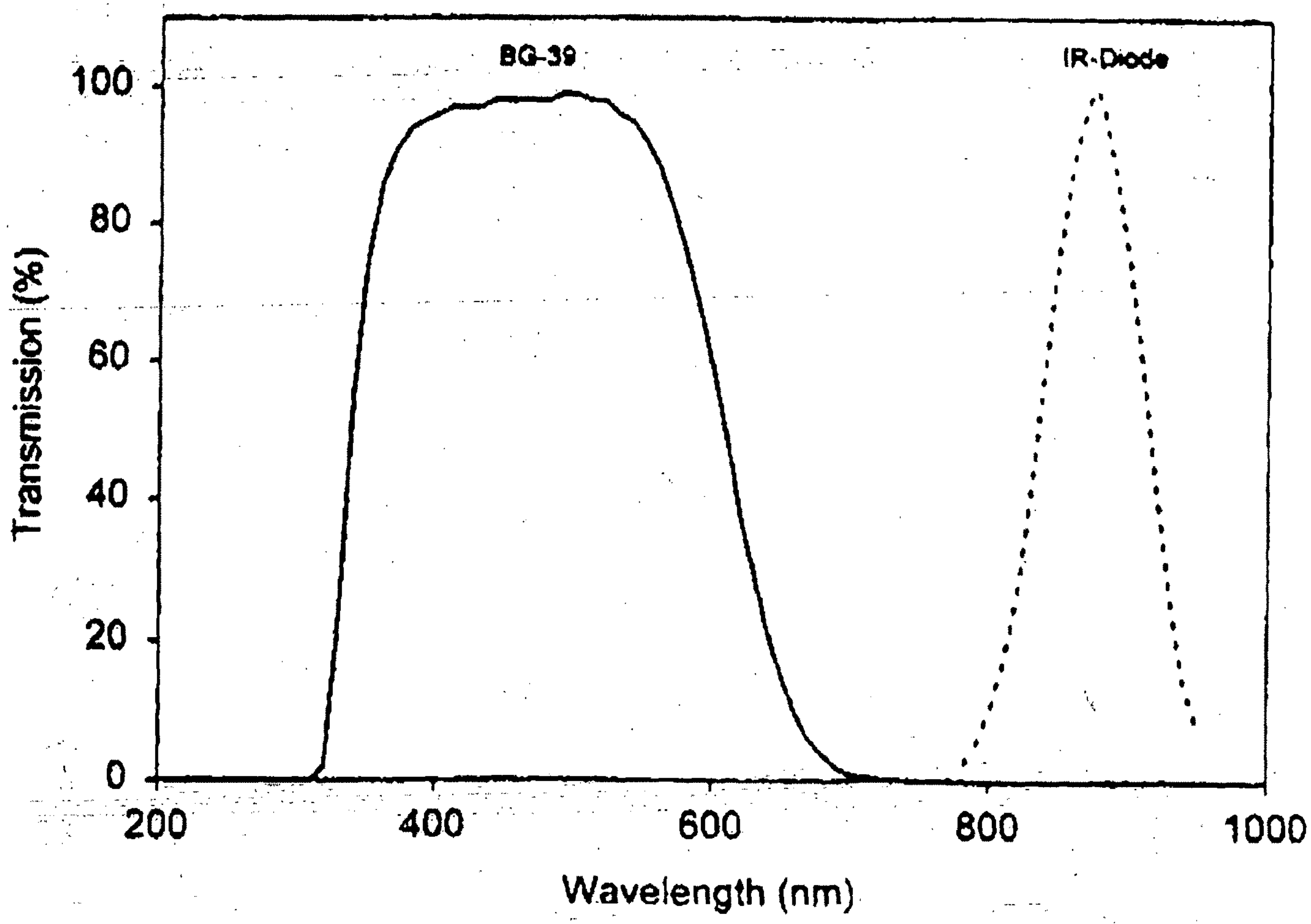
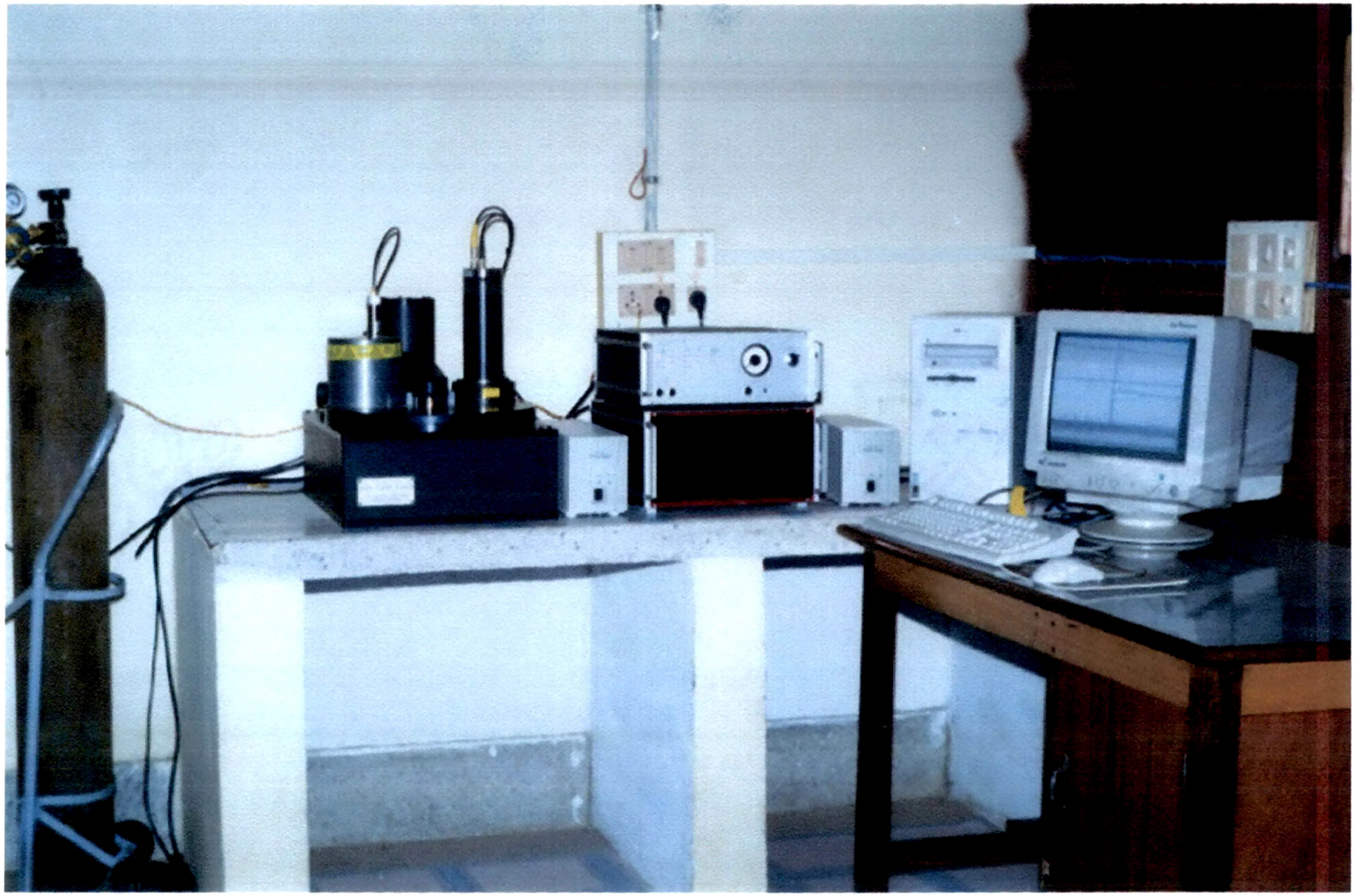


Fig-3C: Transmission characteristics of Schott BG-39 and IR LED type TEMPT 484 filters.



A photograph of the RISO OSL/TL system

Stimulated Luminescence (OSL) developed by Huntley et al.,[5] and Hutt[6] RISO introduced OSL attachment to their TL reader.[7]

[A] Hardware of Riso TL/OSL (model TL/OSL-DA-15)

In addition to basic hardware components of a TL/OSL reader described above, the new RISO TL/OSL model has many hardware and software enhancements. The hardware enhancements are shown in **Fig-3D.**

Mini Computer

All direct hardware control of the new automated TL/OSL reader has been incorporated into a "Mini-Sys" computer. A PC based "Mini-Sys" unit is interfaced with the instrument. The "Mini-Sys" connects to the host computer via an RS-232. Serial cable eliminates the need for an expansion board in the host PC. The "Mini-Sys" computer is responsible for maintaining proper timing, sample positioning, data acquisition, and error checking, etc. The "Mini-Sys" computer is equipped with a two-lined digital next display, which shows the current system status, and the command, which is currently being executed (e.g. "Recording OSL"). This display also reports failure messages such as thermal failure, OSL lamp failure or the receipt of invalid commands.

Sample Turntable

There are two modes for speed of the turntable to reduce processing time. If a sample is moved to the next position then the turntable turns at the normal speed. If, however, the turntable must advance several positions, it is accelerated to a high speed for most of the move and decelerated to slow speed before stopping. By recording a CCD camera image of the sample, repositioning the turntable several times, recording another CCD image and finally comparing the two images, it is confirmed that the acceleration and decelerations are performed gently enough to leave small grains undisturbed.

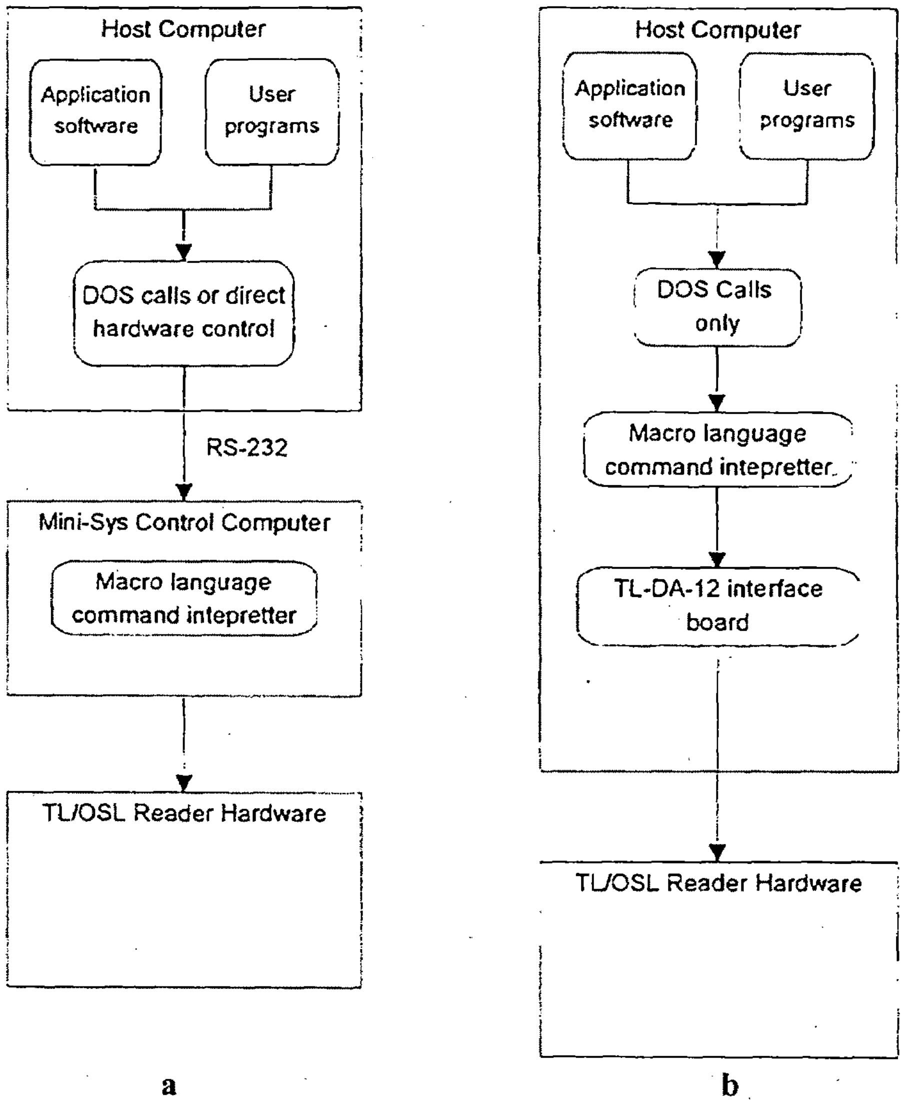


Fig-3D: A schematic diagram showing the hardware enhancements in the new RISO TL/OSL reader.
(a) New system (b) Old system.

In the event when very fine powders are used the sample distribution may be slightly affected by the high-speed turntable. For this reason, there is option for the ability to easily enable/disable the high-speed feature. The sample capacity for this new system is 48 samples. But all the older turntables of lower capacity are fully interchangeable with each other and as well as with this new one. The new, high capacity turntables may also be used in older system.

Detachable Beta Source

A completely new Sr^{90}/Y^{90} beta irradiator attachment was developed in which the source is mounted in a rotating, stainless steel wheel. With this design it allows the source to be positioned much closer to the sample than was previously possible, thus increasing the dose rate by more than a factor of two. Also, the external radiation level has been reduced. Further, the dose received by samples under the closed irradiator has been reduced dramatically. A 0.15mm window interface between the source unit and vacuum chamber has significantly improved vacuum conditions and also allows the irradiator to be removed easily in order to make very low dose measurements. An external visual indicator/interlock is added to the irradiator unit, which glows red when the irradiator is on, and green when it is off. The interlock prevents the operation of the irradiator when the system lid is open. The leakage measurement shows a reduction by a factor of approximately 33. The leakage represents a very small percentage dose when compared to the source strength. For example, the 30 minutes leakage dose of 1.47μGy is only 0.2% of the dose received with the shutter open for only 1 sec (73mGy), Dose rate is 0.084 Gy/sec.

External Equipment Interface

There is option for an additional expansion board to the "Mini-Sys", which provides user with access to 16 digital input channels, and 8 output relays. By sending appropriate command to the "Mini-Sys" it can be instructed to activate one or more relays or wait for a specific input before proceeding. This feature adds to the ability to interface the reader with other laboratory equipment such as lasers and flash lamps for illumination. Also, it is possible to use external equipment to trigger the "Mini-Sys" to start data acquisition.

[B] Software of RISO TL/OSL (Model TL/OSL-DA-15).

In this new model of TL/OSL reader "Windows based programme" are incorporated instead of "Dos Based Programme". Although it has poor 'HELP' features. It is incorporated with abundant good manuals both for Hardware and Software as well as Cautions and Low Level Commands.

The "window based programme" which are used to run the RISO TL/OSL reader through the "Mini-Sys" slave computer can be described under three main routines like CONTROL, SEQ.EDITOR and VIEW.

CONTROL (Center.exe) is meant for controlling the "Mini-Sys" (TLMSLL.CMD file). In the TLMSLL.CMD file each of the high level commands which one can insert into the spreadsheet of the sequence editor is defined as a set of "Mini-Sys" low-level commands. It is used to test the reader, to change from position 24 to position 36 or 48 position wheels and to reset the hour counter for the halogen lamp. It is enables N_2 gas value ON or OFF, β-irradiator ON or OFF etc. When the CONTROL program is started, sometime is left for it to connect and received data from the "Mini-Sys".

Sequence Editor

A sequence is like a spreadsheet; it is a rectangular grid of columns and rows. The basic unit of a sequence is a cell, in which a single command is stored. Columns are labeled from left to right, beginning with "samples" the continuing with "Run 1", "Run 2" ..."Run-n". Rows are numbered drawn from "Set-1" to "Set-n". The cells in a sequence is filled in by choosing: Edit from the Edit Menu Pressing-F2 double click on the cell with the lift mouse button or by choosing Edit from the context menu. The context menu appears whenever the right mouse button is pressed.

The SEQUENCE EDITOR is used to create any desired measurement sequence. Sequences can be stored under individual sequence names and recalled to be used (or modified) for feature measurements. All about the operation of the SEQUENCE EDITOR can be learnt like that of the basic window application through preferably using 'RISO Manuals' (since online help facility is poor).

Modes, which are available in the SEQUENCE EDITOR

TL with heat ramps from 0.01°C/sec to 20°C/sec up to the maximum temperature of 700°C, with 'Pre-Heat' and 'Background' subtraction. This system has following important features.

- OSL with IR, Blue or Green stimulation at room temperature or elevated temperatures; the stimulation time range is 0.1 sec to > 4000 sec.
- IR LASER POWER is software controlled from 0 to 100 %. Maxi 400 MW at 832 nm.
- Blue LEDs with software-controlled power from 0 to 100 %. Max 50 MW at 470 nm.
- RAMPED OSL (IR Laser and Blue LEDs), the power is ramped from start power (0-10%) to end power (0-100%) during a pre-selected time; this can also be done at an elevated temperature.
- TOL (Thermo-Optical Luminescence).
- POSL (Pulsed Optically Stimulated Luminescence).
- Low Level PROGRAMMING, which gives complete freedom in design of sequence.

- IRRADIATION (beta or alpha irradiation). Time range from 1 sec to 31 yrs.
- ILLUMINATION with IR, Green or none time range from 0.1 sec to > 4000sec. Pre-Heat of samples, temperature range from room temperature to 700°C. Time range from 1 sec to 999999 sec.
- PAUSE is possibility of a time delay. Range 1 sec to 999999 sec.

View (wtl.exe) is used to display and print curves, make integrations and print data values from the *bin files obtained by running a sequence. A single curve or a group of curves can be "dragged" from one *bin file to another, or they can be "Copied" and "Pasted" into a spreadsheet program.

Analyst is a windows based program designed to allow one to view, edit and analyses luminescence data collected using a RISO automated TL/OSL reader. It offers a range of facilities, including equivalent dose determination. Additionally, since it is impossible for any analytical program to cater for every possibility it allows one to export the data in a variety of format so that it can be transferred to other programs, including spreadsheet. The basic file structure used by 'Analyst' is the BIN file. The important point to note at this stage is that by using this data format each luminescence measurement is stored in a 'record' that contains not only the numerical data from the luminescence measurement itself, but a wide range of additional information as well. This information can be grouped into three categories: sample characteristics, measurements conditions and analytical data. Some of this information is automatically placed into the record when it is recorded by the TL/OSL system, some is set only if the user chooses to set it by using the 'Run Information' box in the sequence editor, while other parameters can only by set during processing 'Analysts' allows one to view, edit and use all of these parameters.

3.4 Sample Preparation

For present investigation, laboratory grown Synthetic Quartz (SQ) crystal was used, which was supplied by Center Glass and Ceramic Research Institute, Jadhavpur, Kolkata. The production of synthetic quartz was done using by the technique of hydrothermal growth of single crystals of quartz in high-pressure autoclaves at elevated temperature.[8]

This type of laboratory grown synthetic quartz was crushed using agate mortar and pestle and prepared fine powder with three different grain sizes such as (i) 120-140mesh (ii) 230-270mesh and (iii) 270-325mesh which were used for TL/OSL study in the present investigations.

3.5 Thermal Annealing Treatment

Thermal annealing for the specimen was carried out in the muffle furnace. The laboratory muffle furnace has temperature range up to 1200°C and the size of chamber for sample heating was 22cm × 10cm × 10cm. The temperature was maintained with ±1°C accuracy using a temperature controller, which supplied required current to the furnace. Power supply of 230V was provided to the furnace. A silica crucible containing a powdered form of virgin specimens was kept in the furnace at required annealing temperature for desired time. After completion of annealing duration the specimens were rapidly air-quenched to room temperature by withdrawing the silica crucible on to a ceramic block. Such material or specimens are called "annealed and quenched" or "thermally pre-treated specimen".

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