Chapter-2

Experimental Procedures

This chapter summarizes the various experimental aspects of analysis used in the studies presented in the thesis. Chapter includes a discussion on the following aspects–

(i) Sample preparation

(ii) TL/OSL measurements

(iii) Calibration of irradiation source

(iv) Estimation of paleodose

(v) Annual dose measurement

2.1 Sample preparation

All the sediments were collected in a specially designed cylindrical tube (~20 cm long and 5 cm diameter) made of aluminum or galvanized iron. These tubes were hammered into a vertical face of a cleaned stratigraphic sequence. Inside the laboratory, these tubes were opened under subdued red light conditions and the sediment from outer (light exposed) portion of both the ends of the tube was collected and kept for estimating the concentration of radioactive elements. Samples in the middle portion of the sample pipe was sequentially pretreated with 10% HCl and 30% H_2O_2 to remove carbonates and organic material. Further separation depended on the type and the mean grain size of the

sediment. These were, quartz mineral separates (>100 micron) and polyminerallic fine grains (4-11 micron). The sample preparation for fine/coarse-grained sediment is below–

2.1.1 Coarse Grain separation technique

Chemically pretreated coarse-grained sediments were sieved (grain size 90-150 micron). Density separation using Sodium Polytungstate (ρ=2.58 g/cm^3) was carried out to separate quartz and feldspar minerals. The quartz fraction was etched with 40% HF for 80 min to remove the alpha affected skin. 12N HCl treatment for 40-50 minutes immediately followed the HF treatment to convert insoluble fluorides to soluble chlorides. Clean quartz grains extracted were then mounted on stainless steel discs (aliquot) with help of *Silkospray*™ silicon oil and their purity was tested for IRSL to detect possibilities of residual feldspar contamination. Samples with finite IRSL indicated a feldspar contaminant and such samples were re-etched for 10 minutes of HF followed again by 12N HCl.

2.1.2 Fine grain separation technique

Extraction of 4-11 micron grain size fraction involved de-flocculation of the samples with 0.1N sodium oxalate after treatment by HCl and H_2O_2. In general several treatments of Sodium oxalate were needed. At each stage samples were given vigorous ultrasonic treatment. The deflocculated sediment was put in 6 cm high column of acetone and allowed to settle for 1.5 minutes. In this period the grain size fraction of >12 micron settles down. From the suspended sample the grain fraction 4-11 micron were obtained by allowing this size to settle on typically for 15 minutes. This fraction was then dispersed either in acetone or alcohol solution and equal volumes (1 ml) of this suspension were dropped using pipette onto small flat-bottomed glass vials, each containing a polished aluminum disc (~9.65 mm diameter) at their bottom. After drying the solution in an oven (temperature < 45°C) for ~15-16hrs, the discs with thin layer typically a few micron of fine grain were taken out and transferred into sample holders.

2.2 TL/OSL Measurement

The TL/OSL measurements were made on three TL/OSL readers - (i) Daybreak 1100; (ii) Risoe TL/OSL Reader DA-15 and (iii) Risoe TL/OSL with Single grain attachment. All the systems comprised a photon counting system and a digital ramp based temperature control system (Fig.2.1). The luminescence was detected by EMI 9635 QA Photo Multiplier Tube (PMT) attached to a filter pack consisting of BG-39 + U-340 (in case of UV emission and blue stimulation OSL for quartz) or BG-39 + Cs 7-59 (in case of IRSL for feldspar/polymineralllic fine grain). For very bright samples, fused silica neutral density filters were used and the maximum photon counts rate was always kept below $5x10^5$ counts/second to avoid any pulse pile up effects. The signal from the PMT was routed through a computer interface to an IBM-PC. TL measurement was done using a heat ramp rate of 5^oC/second. All measurements of TL/OSL were done in an ultra pure Nitrogen gas atmosphere.The system stimulation window comprises, an array of blue LED's ($\lambda = 470 \pm 30$ nm) and red LED's ($\lambda = 880 \pm 80$ nm) for blue and Infra red photon stimulations of the quartz and feldspar respectively. The average power delivered from these LED's ranged from 25-45 mW/cm^2 in total. In Daybreak TL/OSL reader (model 1100) 57 aliquots can be kept at once in the machine but due to absence of inbuilt beta irradiator, it restricts its use for Single Aliquot measurement as it needs 5-6 cycles of irradiation, preheat and OSL measurement on a single aliquot. In Risoe TL-DA 15, one can put 48 aliquots at a time to analyze and due to inbuilt irradiator; all the SAR measurements were done on this system only (Botter et al., 2000; Botter and Murray, 2001)

2.3. Laboratory Irradiation and Calibration of the Beta Dose Source

Beta source dose calibration is an important part of the growth curve construction in luminescence dating. It is necessary to assign the accurate as well as precise value for it. For single aliquot analysis, a set of 6 small aliquots having 90-150 μm of gamma dosed (5 Gy) calibration Quartz (from Risoe National Laboratory, Denmark) were run for SAR during installation of the TL/OSL reader. The mean value of all the Paleodoses was taken as a source dose rate.

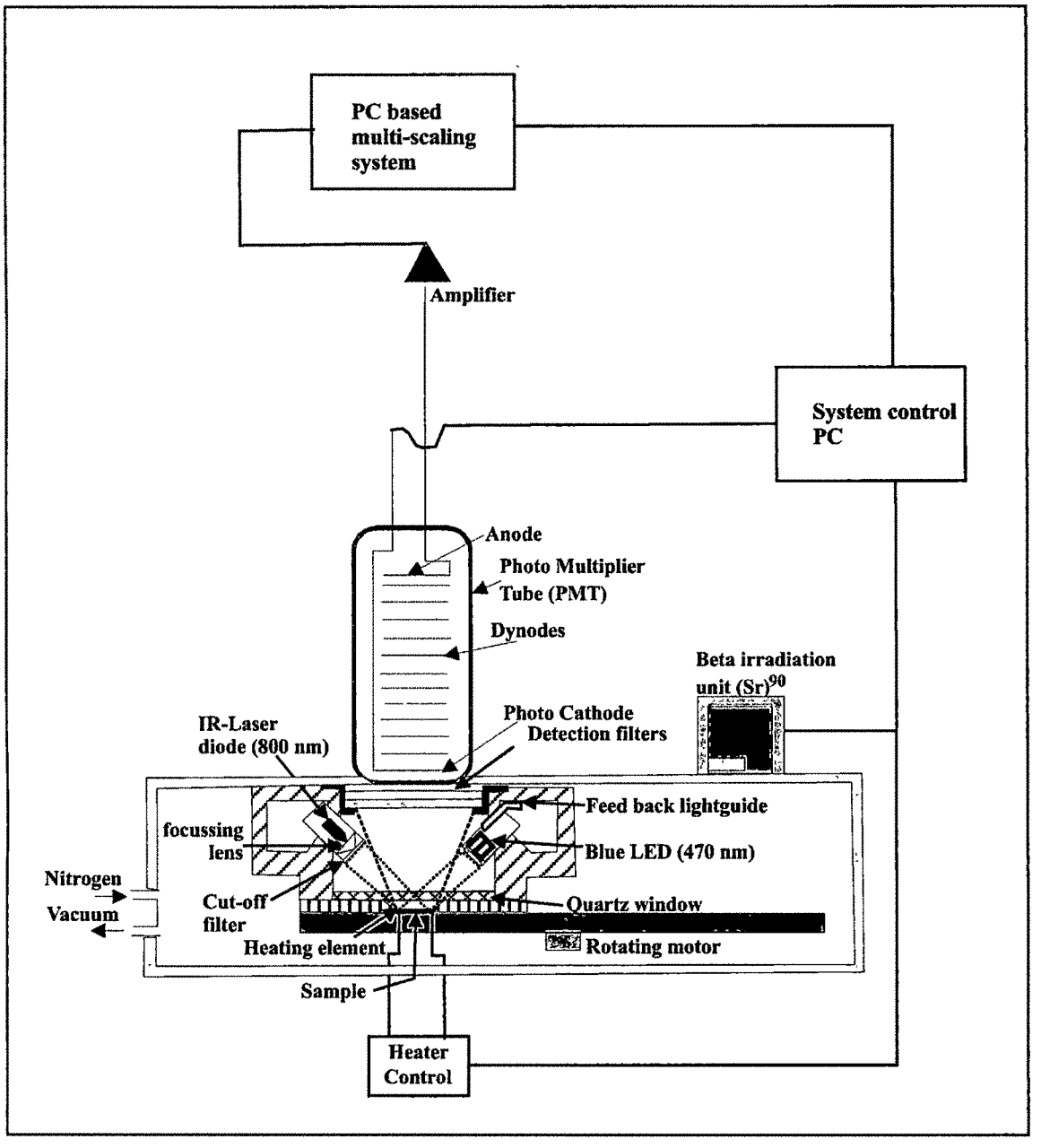

Figure 2.1. Schematic of the instrument used for the TL/OSL measurement. (Modified after Aitken, 1998; Botter Jenson et al., 2000)

Most of the experiments were done on Risoe TL/OSL reader-15, which has inbuilt beta irradiator, making convenient to do all the measurements in a single machine and thus reducing the chances of loss of any grains from aliquots during transportation. The calibration of Risoe TL/OSL reader (model TL DA-15) gave the beta source dose rate to be 7.31 Gy/minute measured in April, 2001.

Beta irradiations on some of the samples were performed by a 20 positions beta irradiator manufactured by Daybreak-Nuclear and medical systems. The source was calibrated for fine (4-11μm) and coarse grain (90-150 μm) samples using CaF_2 from Oxford and Quartz supplied by Risoe National Laboratory, Denmark. An inter-laboratory calibration was made between BARC-Mumbai and Oxford luminescence laboratory sources. The calibration of Daybreak beta irradiator was done on April 2001 that gave a dose rate of 3.65 Gy/minute and 2.44 Gy/minute for Quartz (90-150 μm) and fine grained (4-11 μm) respectively.

The Single Grain TL/OSL reader was calibrated including to check for the homogeneity of the beta dose rate on an aliquot In case of single grain analysis, the paleodose is to be calculated from a grain sitting on one of the 100 holes (300 μm diameter each) arranged in a matrix of 10 x10. Hence it was necessary to do the calibration of each hole containing grain and to ensure about the homogeneity of the beta dose source. To do so, 210-250 μm size grains of gamma dosed calibration Quartz grains were mounted on the single grain disks. Nine disks were analyzed containing 900 (100 x 9) grains. The homogeneity was checked by measuring average paleodose from each successive rectangle containing the grains on the disk (Fig. 2.2). The outermost rim contains 36 grains, second outermost contains 28 grains, middle rim contains 20 grains, second innermost rim contains 12 grains and innermost rim contains only 4 grains. The average paleodose from the each rim is tabulated in Table 2.1. The standard deviation of the data was <5% of the natural. The values of the paleodoses decreases slightly as you move from outermost rim to the innermost rim, however total change in paleodose is < ~7.5 %. These results shows that beta irradiator provide a homogeneous dose rate on the aliquot with ~7.5 % variation.

Table 2.1. Source dose rate distribution in a single grain disk from the outermost rim to the innermost rim. The data are averaged over 9 single grain discs (~100 grains in each disc).

Rim	Dose Rate (Gy/minute)
Outermost	7.0 ± 0.1
Second outermost	7.2 ± 0.1
Middle	7.4 ± 0.2
Second innermost	7.5 ± 0.2
Innermost	7.6 ± 0.4

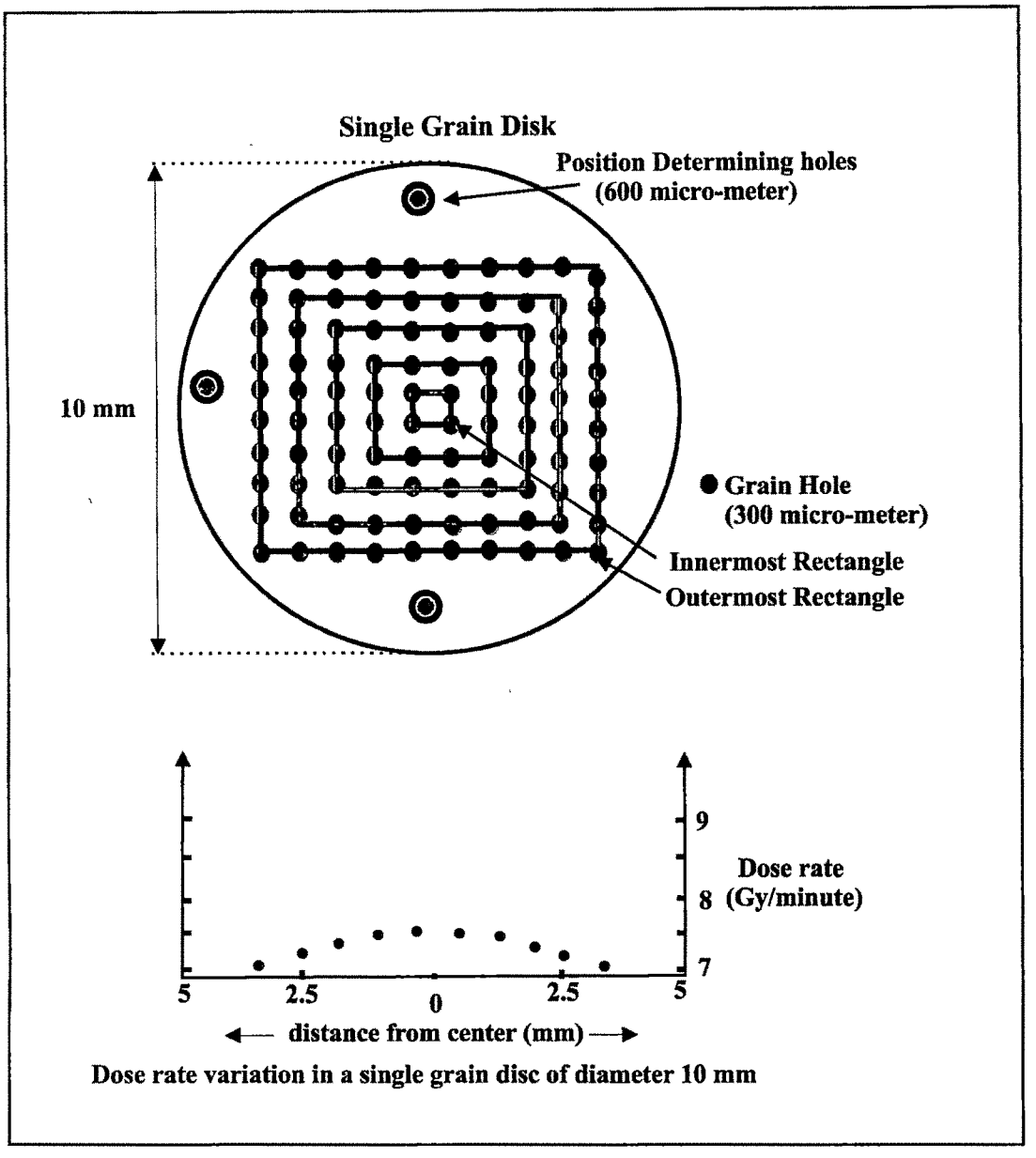

Figure 2.2. Diagram of a single grain disc. The calibration of the dose source was done using 5 Gy gamma dosed calibration quartz. The dose source strength was calculated on each rim of the disc as shown above. The source strength was found highest in the innermost rim i.e. around the center. However, values decreased systematically towards the outer rim with a maximum difference of up to 7.5 %. The error ranges from 1.5% to <5% on the dose rate values from outer to central rim.

2.4 Estimation of paleodoses

Several methods have been developed for the measurement of Palaeodose, which deal with situation of partial or total bleaching, sensitivity change and luminescence dose saturation. Totally bleached samples are most likely in wind-transported sediments (sand dunes etc.) and sediments deposited in stagnant water conditions (e.g. silt or silty-clay). The samples where partial bleaching can occur are proximally transported aeolian or fluvial sediments deposited under condition of large bed load etc. The methods can be placed in two broad categories,

(i) The Multiple aliquot Methods: (a) Multiple aliquot additive method (MAAD)
(b) Multiple aliquot regeneration method (MAR)
(c) Partial bleach method

(ii) The Single aliquot Methods: (a) Single aliquot additive method (SAAD)
(b) Single aliquot regeneration (SAR).

2.4.1 Multiple aliquot methods

This method assumes that at a grain level sample is homogeneous in respect of thermal or bleaching history. Consequently this method has been successfully applied in the case of archaeological pottery, burnt sediments (baked clay) and wind blown sediments (well exposed to the daylight) using both the TL and OSL. The multiple aliquot methods are,

2.4.1.1 Multiple Aliquot Additive Dose (MAAD)

In this method, several aliquots (typically 30-40) are used to determine the paleodose. In this, few aliquots are kept for natural measurement. A few groups of aliquots (~5-6 aliquots in each group) are given additional laboratory beta doses in increasing order (say β_1, β_2, β_3,...) such that $\beta_1<\beta_2<\beta_3<$..... and then their luminescence is read onto TL/OSL reader. The luminescence yield of the aliquots is plotted against applied dose added (β_1, β_2, β_3,...) to aliquots to construct a luminescence vs. dose growth curve (Fig. 2.3). This procedure ensures that the aliquots have identical thermal and radiation history. Extrapolation of this to dose axis provides equivalent laboratory beta

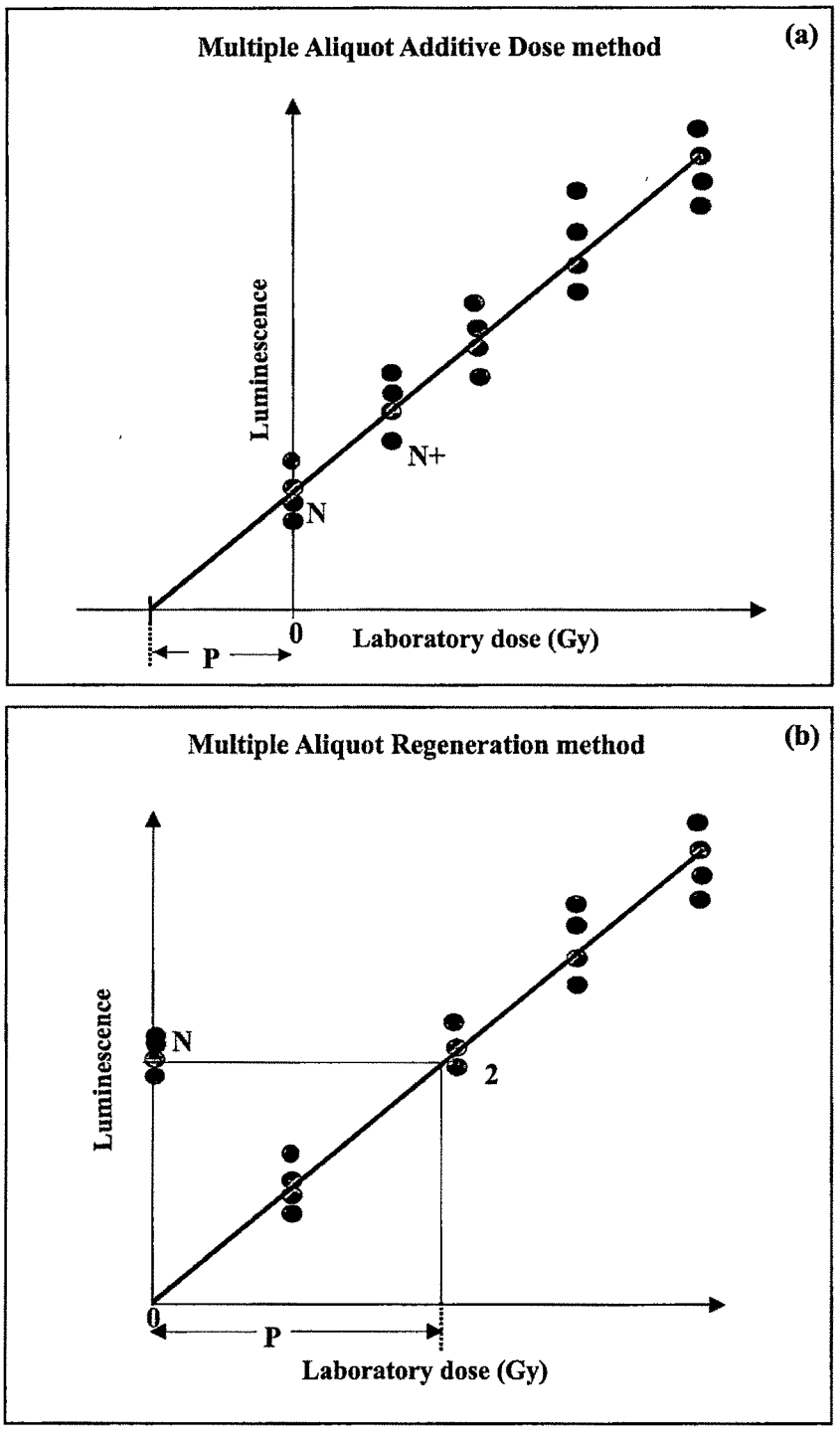

Figure 2.3. Multiple aliquot methods: (a) multiple aliqupt additive dose growth curve constructed between incremental additional lab doses and luminescence counts. Paleodose is calculated by extrapolating the growth curve onto dose axis. (b) multiple aliquot regeneration method in which paleodose is calculated by interpolating natural luminescence counts onto growth curve plotted between luminescence counts and incremental beta doses given to sun bleached aliquots.

dose that is needed to induce in a sample a luminescence equal to the natural luminescence. This is termed variously asequivalent dose or paleodose (Aitken 1985; 1998). This method while ensures against sensitivity changes, requires an apriori assessment of the nature of the growth curve. Based on monte-carlo simulations done by Felix and Singhvi (1997) provide practical recipes for construction of growth curves and extrapolations. As the construction of growth curves involves a large number of aliquots, appropriate normalization is needed. There are several normalization procedures (Aitken, 1985; 1998; Jain et al., 2003) and these are listed below–

(a) Weight normalization
(b) Post OSL residual TL normalization
(c) Zero glow normalization
(d) Natural normalization or short shine normalization
(e) Dose normalization or second glow normalization.

In present study, the samples were normalized using natural normalization (short shine normalization). In this method, the sample aliquots with their natural OSL were stimulated by blue light for 0.3 second and the OSL was measured. This duration and stimulation flux was so adjusted that the depletion of natural OSL was <1% of the total OSL signal. This intensity was used as a measure for luminescent grains and helped normalize to remove inter-aliquot variability in respect to variation in number of luminescent grains and their sensitivities from each aliquot. An assumption in this procedure is that the luminescence sensitivity in the first interval and the rest of the shine down curve are covariant, sensu-stricto. This conditionality was not met in some cases, as also, short shine photon statistics was poor making it difficult to normalize. In such cases, second glow normalization was used. In second glow normalization, after all the OSL measurement the luminescence output for a test dose is measured and used for the normalization.

2.4.1.2 Multiple Aliquot Regeneration (MAR)

In regeneration method, the natural process is replicated. Here except the set of natural, each set of disk is sun bleached and then incrementally irradiated. A regenerated growth curve is then constructed between laboratory doses and the luminescence yield.

Paleodose is then evaluated by interpolating natural signal onto the growth curve (Fig. 2.3). This method has an advantage over MAAD of interpolating the natural OSL onto the growth curve rather than extrapolation onto the dose axis that increases the chance of error due to poor fitting of the growth curve. The limitation of this is the change in luminescence sensitivity of the sample may occur during daylight bleaching prior to regeneration.

The nature of luminescence vs. dose growth curve can be complex depending upon the luminescence properties. It can ranges from linear, non linear, exponential and a combination of these. The nature of the growth curve with respect to doses and thus the selection of proper dose protocol are discussed in detail by Felix and Singhvi (1997).

2.4.1.3 The Partial Bleach Method and Linearly Modulated Optically Stimulated Luminescence (LM-OSL)

Partial bleach methods recognize and exploit the fact that the luminescence of a mineral (Quartz/Feldspar) comprises several components with different sensitivity to photo bleaching. Typically the Blue light stimulated luminescence shine down curve of the Quartz comprises three parts viz., (i) the fast component (most sensitive); (ii) the medium component and (iii) the slow component (hard to bleach). In a partially bleached sample, it is expected that only a part of the signal (fast component) was bleached and other components of the signal (medium and slow) were partially reset. (Wintle and Huntley, 1979).

In such samples the measurement of paleodose comprises several sets of aliquots. A normal additive growth curve is constructed (i.e. N, N+β, N+2β,....) as also additional additive dose growth curve using samples which record a short duration daylight exposure after beta dose (i.e. the sequence, N+$\in$sun, N+β+$\in$sun, ...). Luminescence-dose growth curve is constructed for each set of daylight exposure. The intersection of the growth curve of the daylight-exposed set of aliquots with the growth curve of unexposed set of aliquots gives the paleodose. This is called the partial bleach method. A simplified approach of this method was suggested by Singhvi and Lang (1998). In this approach, the portion of Infra Red Stimulated Luminescence shine down curve was analyzed. It was suggested that in a shine down curve, the intensity of the first interval

represents an unbleached signal and the later intervals effectively represent equivalents of daylight-bleached samples. Partial bleach growth curves are then constructed as above. Paleodose is estimated from intersection of these growth curves from the unbleached growth curve. This approach is now known as 'differential partial bleach' (Fig. 2.4).
Bulur (1996) gave the concept of measuring luminescence output with linearly increasing intensity of stimulation light. Conventional constant stimulation method provides a monotonically decreasing signal (shine down curve) whereas Linearly Modulated OSL provides the opportunity of probing the substructure of a shine down curve in a manner similar to a TL glow curve. This procedure therefore provided new possibility of analysis of different components of a signal. Overlapping OSL components considered to originate from different traps can be separated and characterized. The three components of a shine down under constant stimulation, viz., are fast, medium and slow. LM-OSL enabled identification of an additional component (Bulur and Goksu, 1999; Bulur et al., 2000).

Larsen et al. (2000) used LM-OSL to find out partial bleaching in quartz and feldspar. He took annealed quartz disk, irradiated and bleached for different time duration ranging from 0.2 second to 10,000 seconds followed by LM-OSL measurement. Then by plotting a graph between partially bleached signal and non-bleached signal, he was able to show most rapidly bleachable signal, which is not seen clearly in the constant wave OSL (CW-OSL). The key is that the LM-OSL method separates the various components through differences in the photo-ionization cross-section of the traps. These are not easily detectable with conventional CW-OSL.

In the present thesis, a combination of LM-OSL and differential partial bleach method was used to arrive at a most effective way to deal with partially bleached samples. In doing so, the integration times for signal were so computed so as to keep the net flux equal in each interval. A preheat of 240°C/10 seconds was used.

2.4.2 Single Aliquot methods – Single aliquot Regeneration (SAR)

In this method, aliquots of 5mm diameter of the sample area, were used for analysis which comprises ~500-600 grains of Quartz. Though on methodological grounds it is desirable to work with aliquots of 60-100 grains or less (Olley et al., 1998; 1999),

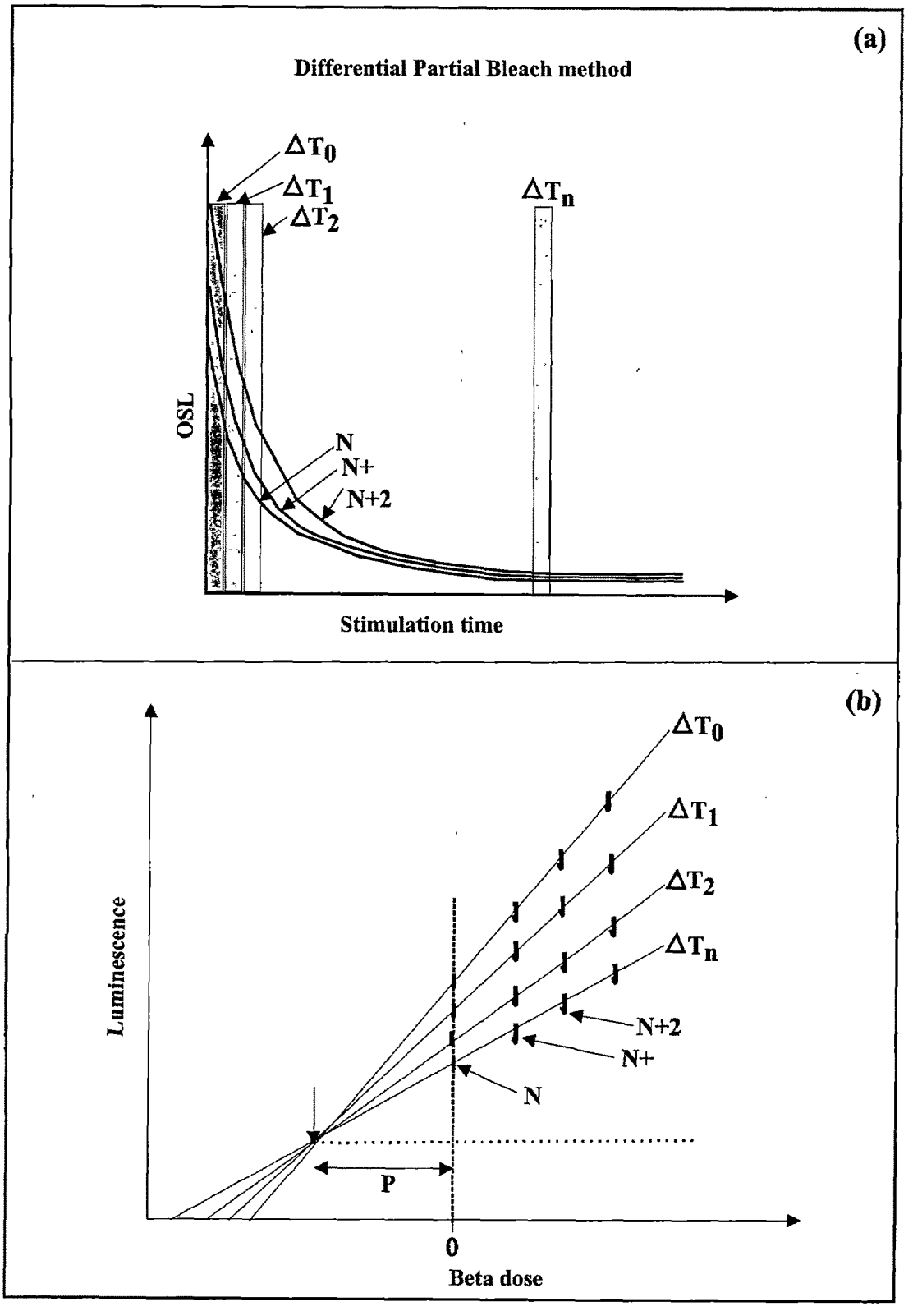

Figure 2.4. The partial bleach method: (a) shine down curve is divided for a fixed time interval and then (b) the luminecence counts from these portion of shine down is plotted against the laboratory doses. The paleodose is the intersection point of these growth curves (modified after Singhvi and Lang, 1998)

poor luminescence sensitivity of Himalayan samples necessitate use of larger number of grains. SAR protocol by Murray and Wintle (2000) was used (Fig. 2.5 and 2.6). In this method, first natural OSL (Ln) is measured after preheat of 240°C/10 seconds. Any changes in luminescence sensitivity during preheat and OSL measurement is corrected by measuring the OSL output (Tn) from a small test beta dose (10-20% of natural dose). The sample is heated to ~160°C prior to measurement of Tn. This preheat termed as cut heat. The ratio L/T provides a sensitivity corrected OSL. After this incremental regenerated laboratory doses R_1, R_2, R_3 respectively are given on same aliquot and OSL L_1, L_2 and L_3 is measured for each regeneration dose along with test dose OSL T_1, T_2, T_3 (Fig. 2.5 and 2.6). The zero dose point is measured in the similar manner but without giving any dose. In last a regeneration point is recycled by giving similar dose to monitor the validity of sensitivity corrections. A typical SAR protocol involves 12 cycles of irradiation, preheat and OSL measurement and can take up to several hours for a single aliquot. A growth curve between dose points (L_1/T_1, L_2/T_2 and L_3/T_3) and laboratory regeneration doses (R_1, R_2 and R_3) is then constructed. The natural signal (Ln/Tn) is read onto that growth curve to get the laboratory equivalent dose i.e. paleodose. In the present study initial 2 seconds of the shine down curve was taken with a background subtraction from the same curve.

2.4.3 Natural Sensitivity Corrected SAR (correction for changes in natural OSL sensitivity)

In a SAR procedure, the sensitivity change during first OSL readout is corrected by the test dose luminescence followed by the natural OSL measurement. The sensitivity after natural OSL readout does not account for possible sensitivity changes during preheat and measurement of the natural signal. Stokes and Singhvi (Under preparation) pointed out that this has been ignored in standard SAR protocol suggested by Murray and Wintle (2000) and can cause systematic errors. They proposed the use of 110°C TL peak that was correlated with OSL signal and a revised protocol was made (Fig. 2.7) (Stoneham and Stokes, 1991; Stokes, 1994a, 1994b). In the present study it was shown that OSL and TL signal correlates for that sample with respect to test dose. Using this, sensitivity corrected SAR procedure was used with encouraging results.

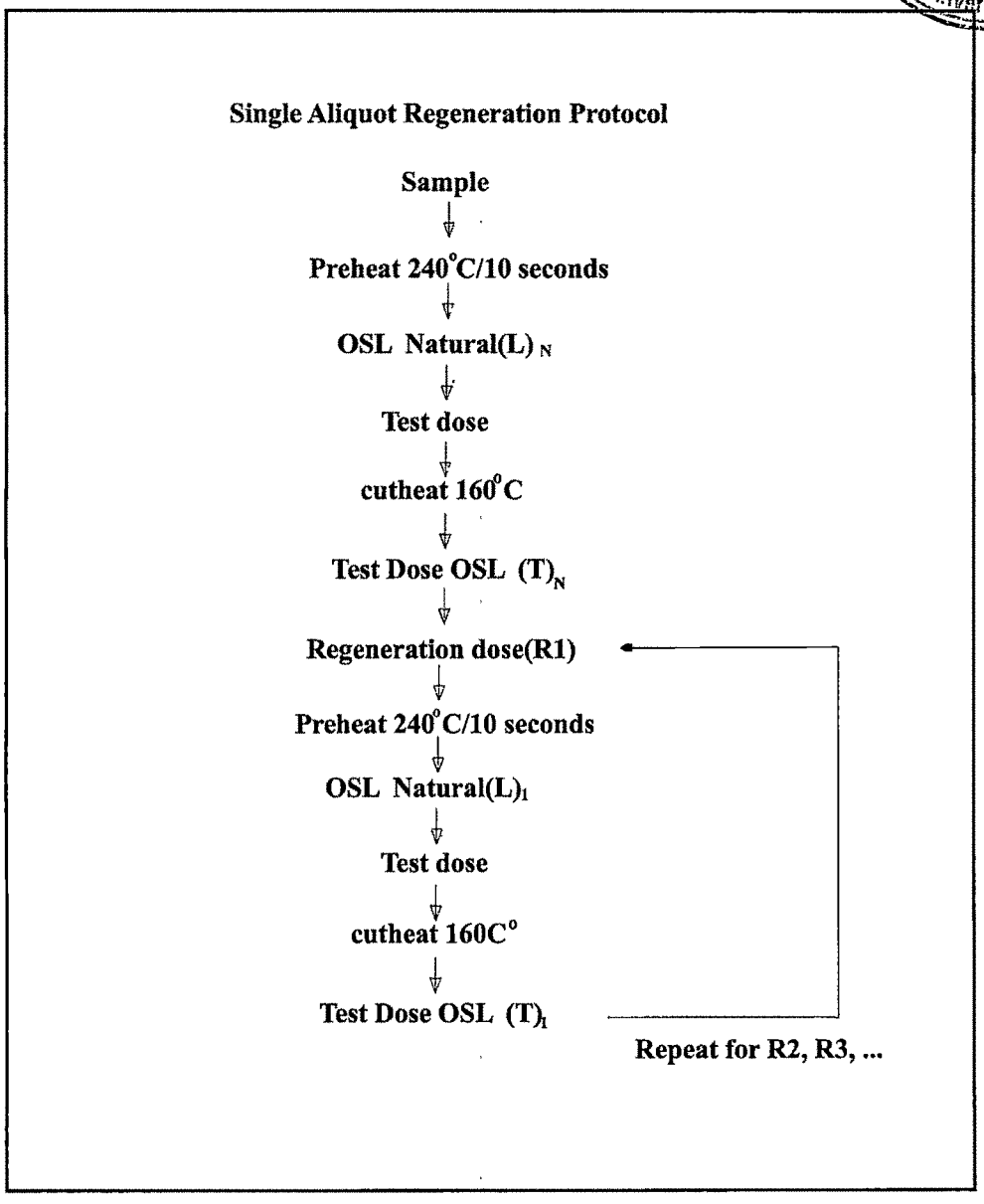

Figure 2.5. The Single Aliquot Regeneration protocol as used in dating fluvial sediment. It involved 6 cycles of irradiation, preheat and OSL measurement along with test dose measurement (after Murray and Wintle, 2000)

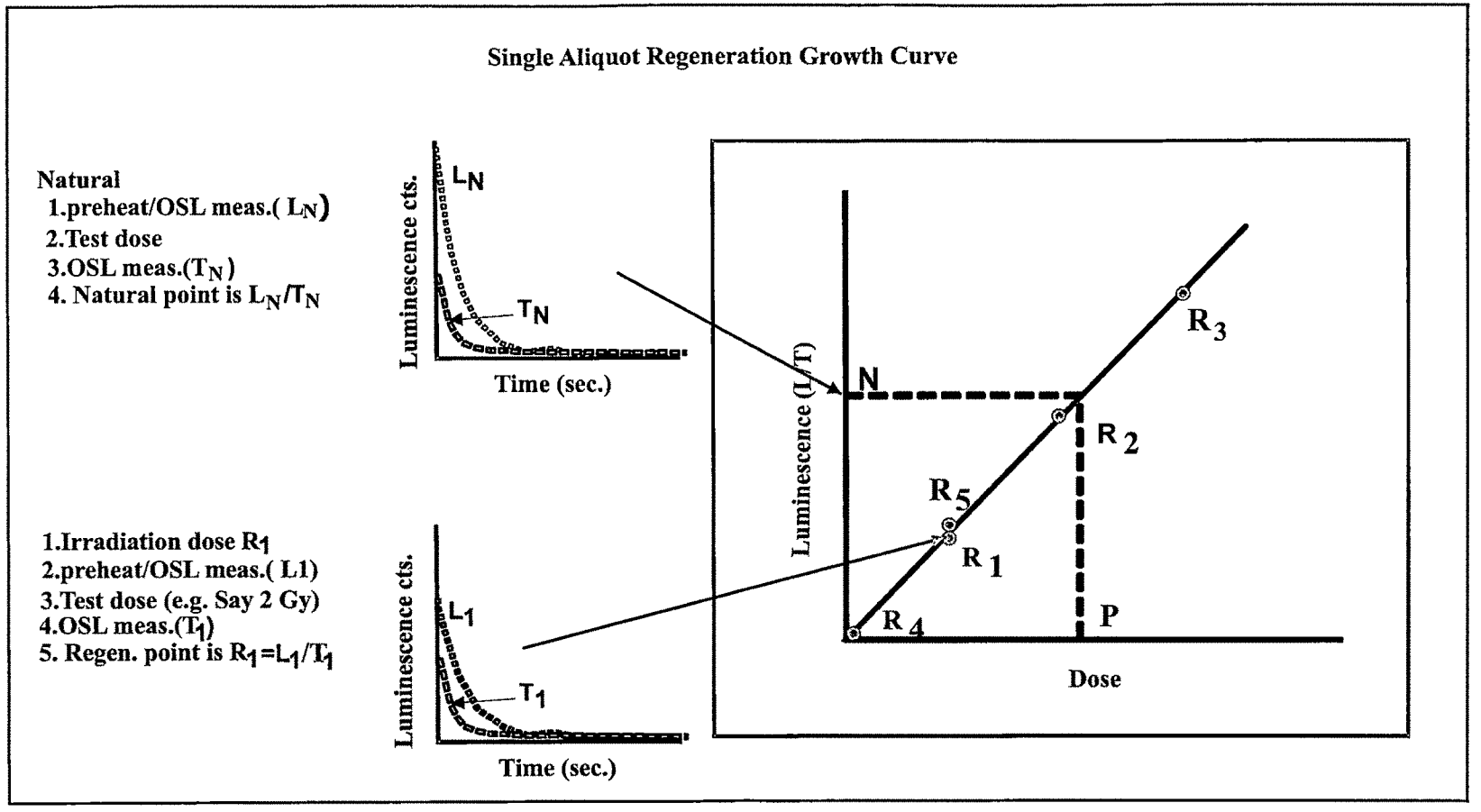

Figure 2. 6. SAR growth curve showing constructed between sensitivity corrected luminescence (L/T) and incremental dose points R1, R2 and R3. The sensitivity corrected natural luminescence (Ln/Tn) is interpolated onto that growth curve for estimating paleodose (after Murray and Wintle, 2000)

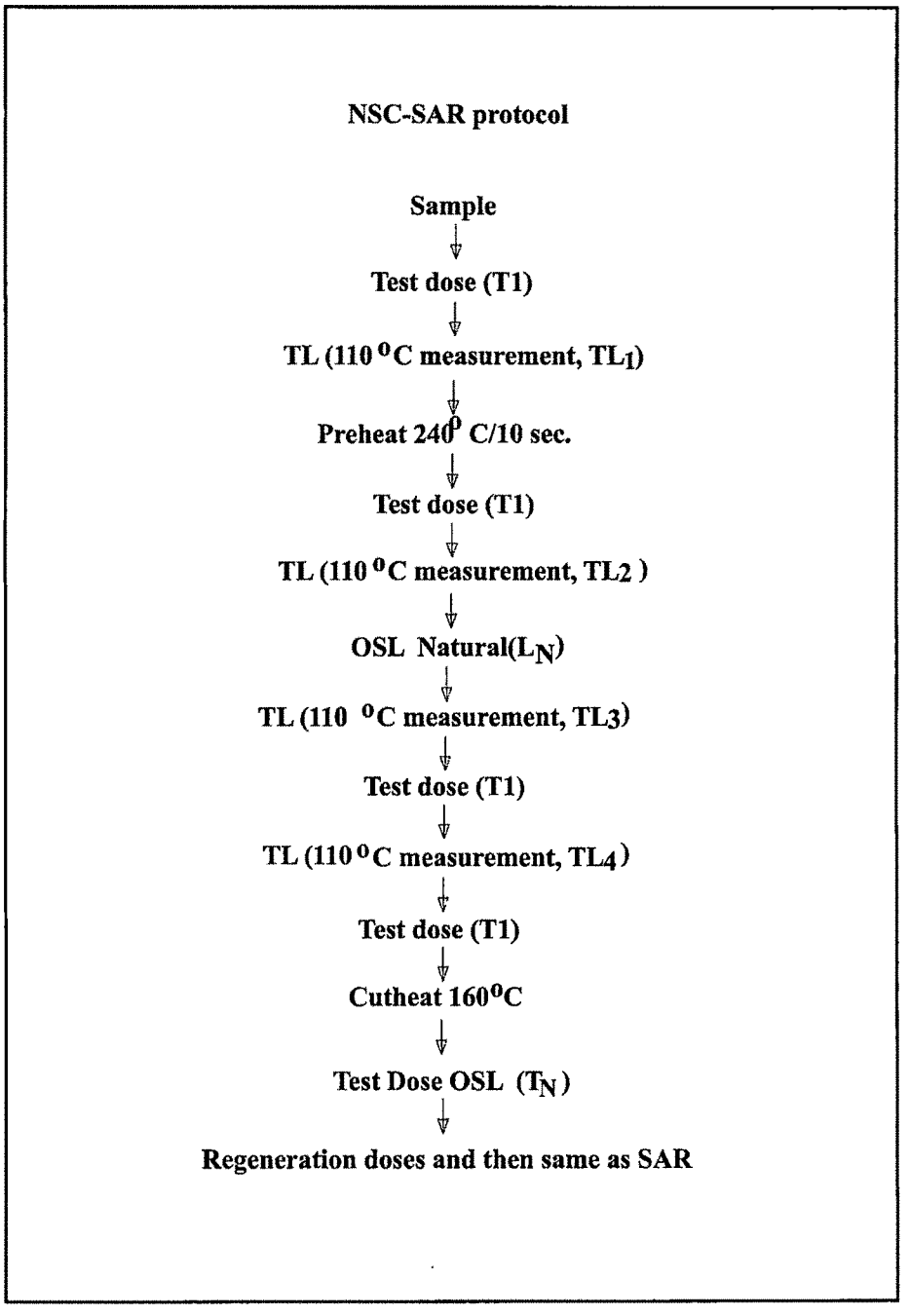

Figure 2.7. The natural sensitivity corrected Single Aliquot Regeneration protocol as suggested by Stokes and Singhvi (under preparation). It involved additional steps of irradiation and 110°C TL measurement during natural OSL measurement along with test dose.

In NSC-SAR method, additional steps of test dose and its 110°C TL peak measurement have been introduced in the standard SAR protocol to quantify the exact sensitivity changes occurred during preheat and natural OSL measurement. First a test dose (T) is administered to the natural sample followed by 110°C TL (TL_1) measurement by heating it up to 160°C. TL_1 represent the sensitivity of natural sample prior to OSL measurement. Similarly few more measurements are made as depicted in Figure 5. Measurement TL_4 represent the sensitivity of the sample after preheat and OSL measurement. In present method a term "natural correction factor" (ncf) has been introduced that is defined as the ratio of TL_1 and TL_4 and is used to correct for the sensitivity.

A plot of the sensitivity corrected regenerated OSL signal with dose enables the construction of an OSL-dose growth curve. In this procedure, natural signal is corrected using test dose signal and ncf for any sensitivity changes and then it is read out on regenerated OSL growth curve (Fig. 2.8)

2.5 Annual Dose Measurement

The annual dose was computed by measuring the elemental concentration of Uranium, Thorium and Potasium. Uranium and Thorium concentrations were measured using thick source ZnS(Ag) alpha counting (Woithe and Prescott, 1995). Potassium concentrations were measured using NaI(Tl) gamma ray spectrometry. The dose rate depends upon the average moisture content of the sediment through its antiquity, cosmic ray variation, alpha efficiency value, grain size and disequilibrium in the radioactive decay chain.

In most of the luminescence dating, a secular equilibrium in the decay chain of U and Th is assumed. This is reasonably met in most cases. Olley et al. (1996) has examined the disequilibrium in fluvial sediments of different age range and found that ^{232}Th decay chain was near to secular equilibrium in almost all kinds of fluvial sediment. This was due to very short half-life of Rn in the decay chain. Disequilibrium in the ^{238}U decay chain is evident in most of the sediment examined; however, the overall effect on dose rate is decreased by 6% for 100 micron grains and 8% for fine grain for the

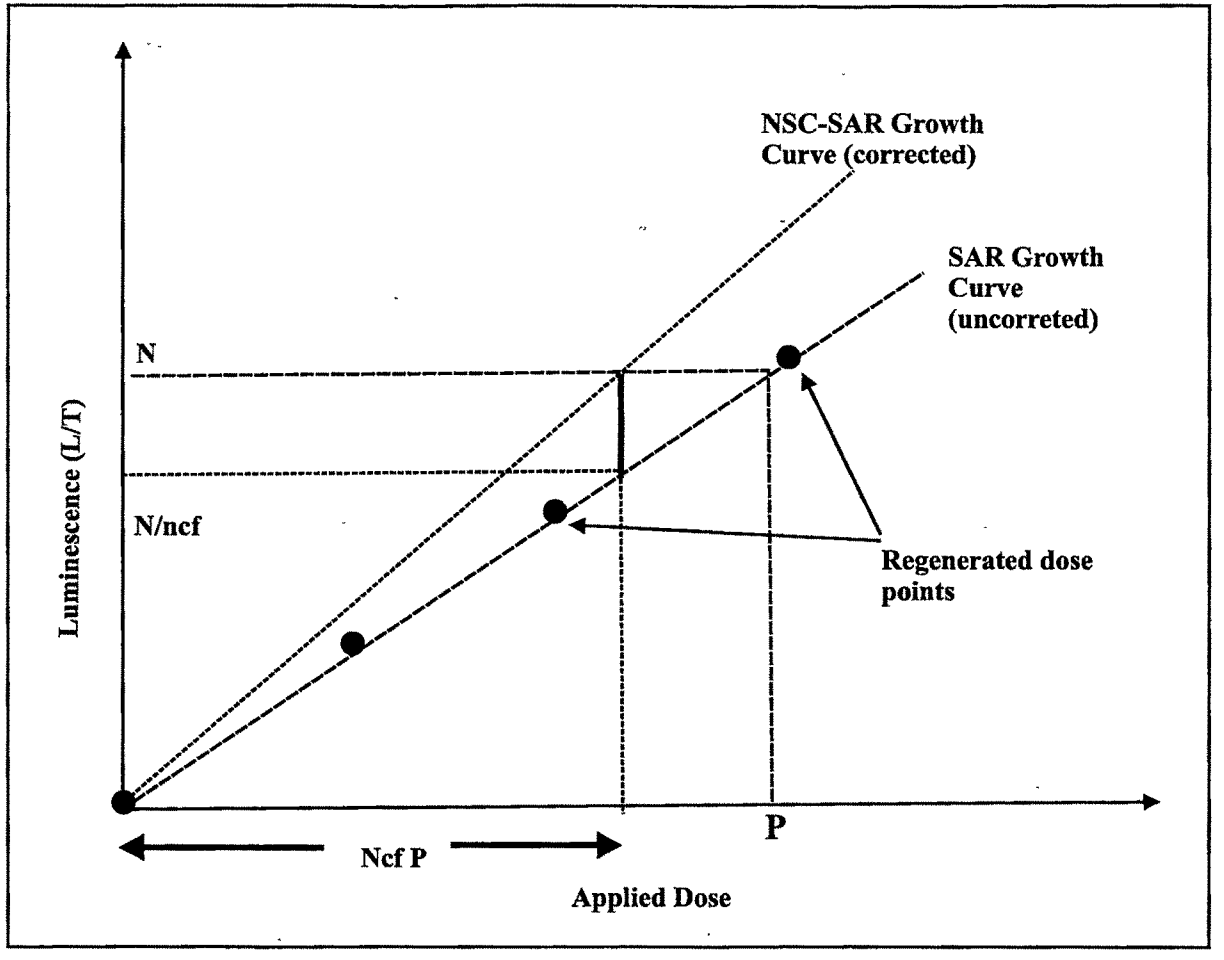

Fig. 2.8. Natural Sensitivity Corrected-SAR (NSC-SAR) growth curve. The natural luminescence is corrected using ncf and then interpolated onto the uncorrected SAR growth curve. The growth curve that will give the ncf corrected paleodose will be the virtual growth curve considered as NSC-SAR growth curve.

sediment comprising 1 ppm ^{238}U, 3 ppm ^{232}Th and 1% K. (Aitken, 1985; Dickson and Wheller, 1992; Krbetschek et al., 1994).

For measuring Uranium and Thorium concentrations, the samples were gently crushed to thickness less than 10 μm, then spread onto a ZnS(Ag) scintillator. For coarse grain, the samples were crushed to powder (<10 μm) and used for estimation of average radioactivity concentration. This is essential for determining beta dose from U and Th concentration. The counting system was calibrated using NBL Uranium standard BL-3 with 1% U and sand 105A with 10.2 ppm U. The counting threshold was set at 83.5 % efficiency to allow the efficiency of counting for the two decay chains to be nearly equal (Aitken, 1985).

Typical background count rate were ~0.2 counts/ks for a counting area of 13.85cm^2 and the typical samples alpha counts rate were ~8-10 counts/ks for the samples. Typically samples were counted over a period of 300 ks and a total count of >1000 was always achieved to obtain a counting error of <3%. All the measurements were made using a DAYBREAK 582 alpha counter.

The estimation of K was made using 3"x3" well type NaI(Tl) scintillator coupled to a standard amplifier and a multi channel analyzer. The energy window was set at 1.46 MeV. The count rate of 7.9 gram of standard (AR grade KCl) is ~60 counts per minute for the same geometry. In the comparison, only the photo peak counts significantly up to the full width at half maxima were used. Typically sample weights were ~15 gm giving a count rate of 5-7 counts/minute for sample compared to a background of 1-1.5 counts/minute. The cosmic ray dose was estimated using the prescription given by Prescott and Stephan (1982). The correction for moisture content was made according to Aitken (1985). After calculating weight percentages of the above-discussed radioactive elements, the dose rate was calculated from Table 2.2 based on the Aitken (1998).

Table 2.2 Calculation of dose rate from known concentrations of radioactive elements. (after Aitken, 1998).

Se. No.	Elements	Concentration (wt %)	Alpha dose (Gy/ka)	Beta dose (Gy/ka)	Gamma dose (Gy/ka)
1	U	1 ppm	0.231	0.145	0.113
2	Th	3 ppm	0.193	0.082	0.143
3	K	1 %	--	0.782	0.243
4	Cosmic	--	--	--	0.18

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