THERMOLUMINESCENCE DATING OF SEDIMENTS

by

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ABSTRACT

I have attempted to develop a method of dating peat and silt materials using the R-Γ technique of A.G.Wintle and D.J.Huntley. The R-Γ method was applied to minerals extracted from peat and silt samples of known radio-carbon (C-14) ages ranging from 0-70 ka to obtain their equivalent doses (ED's). No significant dependence of the ED on the grain size of the minerals from the samples of zero age was observed. The spectral distribution of the thermoluminescence (TL) of the samples and the results of anomalous fading tests on magnetic, non-magnetic and quartz fractions of several samples are reported. The in-situ gamma dose rates at two sample sites measured with CaF_2 :Dy phosphor dosimeters are in agreement with the results obtained from alpha scintillation counting and K analysis. Dose rates are corrected for the presence of organic matter as well as water in the samples.

Peat samples yield apparent TL ages that agree well with known ages while some silt materials show anomalous results. Not being 'zeroed' at the time of deposition or the dose rate variability at sample sites are suggested as possible reasons for the latter. The preliminary results show that the R-Γ method has much promise in obtaining reliable ages of sediments in the future.

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I. THERMOLUMINESCENCE DATING

1.1 Introduction

Thermoluminescence (TL) techniques are established in dating pottery from archaeological sites (Aitken, 1974; Fleming, 1979), but dating sediments by TL is still in its infancy. This thesis describes an attempt of studying the applicability of a TL technique for dating certain types of land sediments. A brief overview of TL and TL dating will first be presented.

1.2 Thermoluminescence

Thermoluminescence is the light emitted by a crystalline or glassy material when it is heated after being exposed to ionizing radiation. A quantitative understanding of the phenomenon can be brought about with the aid of the band model of solids. Exposure to ionizing radiation produces free electrons and holes in the crystal; electrons are released from the valence band to the conduction band leaving holes in the valence band (Fig.1.1-a). All crystalline materials have a great many structural imperfections, for instance the occurance of negative or positive ion vacancies or the presence of impurity atoms in the lattice; they are either negative or positive charge deficit sites. These defects or 'traps' as they are usually called, are metastable states in which electrons or holes can be captured and retained for significant times. An electron or a hole trapped at such a site could be visualized as a system similar to a hydrogen atom like bound state which has its own quantized energy levels. Some of the free electrons and holes produced by ionizing radiation get trapped in these metastable levels instead of recombining. For a given type of traps the mean life time τ of such metastable levels depend on the 'trap depth' E and the absolute temperature T and is given by,

$$\tau = s \exp(E/kT) \qquad (1.1)$$

where s is a constant, k is the Boltzmann's constant (Curie, 1963). So the deeper the trap (i.e, larger E), the longer the life time.

An electron or a hole may eject from a trap by absorption of a photon of sufficient energy or by acquiring the necessary activation energy (E) from the phonon interaction with the surroundings. In TL this activation energy is supplied by heating the material. The ejected electron can wander through the crystal and recombine with a trapped hole giving out visible light (Fig.1.1-b). A released hole may undergo a similar process. The liberated hole can migrate via the valence band until it recombines with a trapped electron and luminesces (Fig.1.1-c). Since the two processes are similar, one can discuss TL in terms of either electrons or holes without loss of generality.

The luminescence spectra depend on the type of recombination centre but not on its depth below the conduction band. It is a characteristics of the ions forming these centres. For instance, the TL spectra of doped alkali halides with known luminescent centres (or activators) are characterisics of these centres (Fig.1.1-d).

As a crystal is heated the probability of releasing a particular distribution of trapped electrons is increased and at a certain temperature there is virtual certainty of their release. Hence the light emitted (TL) will start weak, go through a maximum, and decrease again to zero. The graph of TL as a function of time or temperature is called a glow curve.

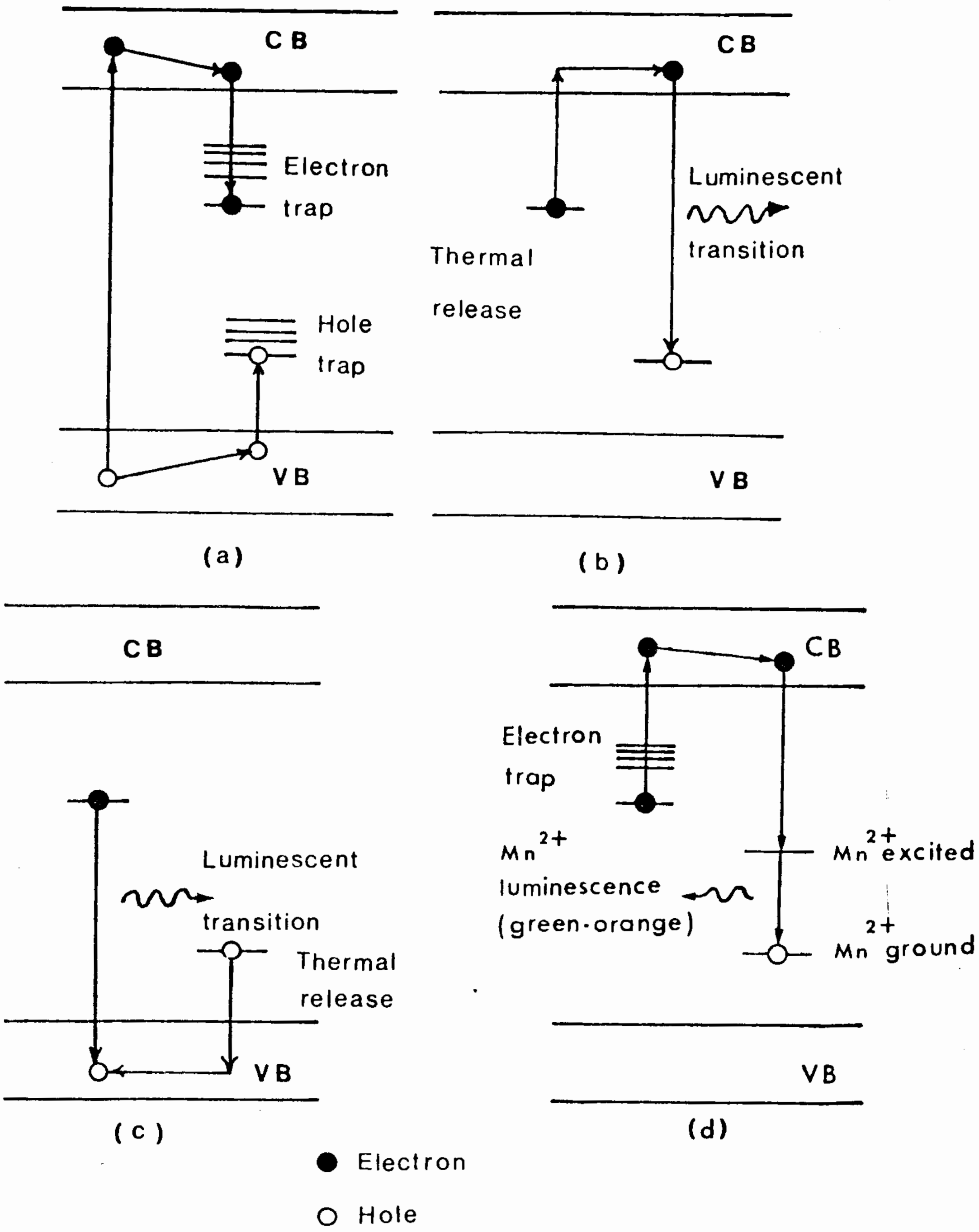

Fig.1.1 TL process.

a) Exposure to irradiation: traps are filled with electrons and holes.
b) Heating: thermally released electrons combines with luminescent centres emitting light (TL).
c) Heating: thermally released holes combine with luminescent cientres emitting light (TL).
d) TL is characteristic of the luminescent centre. eg., Mn^{2+} as the impurity atom; green-orange light characteristic of Mn^{2+} is emitted.

The shape of the glow curve for a single trap can be predicted theoretically, (Randall & Wilkins, 1945). The deeper the trap, the higher is the temperature at which the associated light is emitted.

1.3 TL Dating

The established use of TL for dating pottery is, based on two main factors viz,

1. The last heating of the ceramic should have released all the electrons which had been accumulated in the traps of constituent minerals over their past geological history. This is called 'zeroing' since soon after this event, reheating the material will not give any TL.
2. The TL signal of these minerals increases with time on the subsequent exposure to ionizing radiation, usually both from within and from the surrounding soil in which the pottery had being buried. Decay of the natural radioactive isotopes U, Th and K are primarily responsible for this radiation.

When the minerals from the ceramic objects are heated in the laboratory, a TL signal, usually proportional to their burial time T, and the environmental radiation dose rate D, is observed (Fig.1.2-a). This is the natural TL (NTL). Here, it is assumed that environmental dose rate had been constant and the material's response to radiation had been linear.

This fact is utilized in determining the time of last firing of the material. After measuring the NTL the same sample is exposed to a radiation source such as Sr-90/Y-90 beta source or a Co-60 gamma source for various periods of times and the induced TL is measured at the end of each such irradiations. This way one can obtain what is known as the 'second glow growth curve' (Fig.1.2-b). The dose at which the induced TL matches the NTL is called the equivalent dose (ED). This is the laboratory dose which produces TL equivalent to that from the past natural radiation dose.

Usually, this method is almost impractical since the sensitivity of the sample undergoes changes while heating it for the first time. Hence the common practice is to obtain the first glow growth curve by using a suite of identical samples. Here again the TL of the sample is measured at different doses and the growth curve is extrapolated to give the sample's ED (Fig.1.2-c). A glow curve from a typical pottery sample is shown in Fig.1.3-a; 1) is the NTL, 2) is the TL observed from an identical sample which had been given a laboratory radiation dose.

This procedure is carried out for various temperatures. However a temperature region where the TL originating traps had been stable during the past must be found to determine a reliable ED. This is done by plotting either the ratio 1)/2) or the ED's vs. glow curve temperature (Fig.1.3-b, -c). The region where there has been no loss of NTL is called a plateau.

This whole procedure has to be repeated with a laboratory alpha source as well. The reason for doing this lies in the fact that the TL induced per unit radiation dose is lower for alpha than for beta or gamma radiation. Their sensitivity ratio is called the 'k' value. An alternate ratio calculated on the basis of TL induced per unit track length of alpha radiation is called the 'a' value (Aitken & Bowmann,1975).

The time T, elapsed since the firing of the pottery (i.e, it's age) is then calculated by,[1]

$$T \text{ (years)} = \frac{\text{ED (gray)}}{k(D_{\alpha}) + D_{\beta} + D_{\gamma} + D_{c} \text{ (gray per year)}} \qquad (1.2)$$

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[1] Gray is the SI unit for absorbed dose of ionizing radiation. A gray is defined as the absorption of 1 J of energy per 1 kg of material. 1 gray= 100 rad where rad is the CGS unit defined as 100 erg/g.

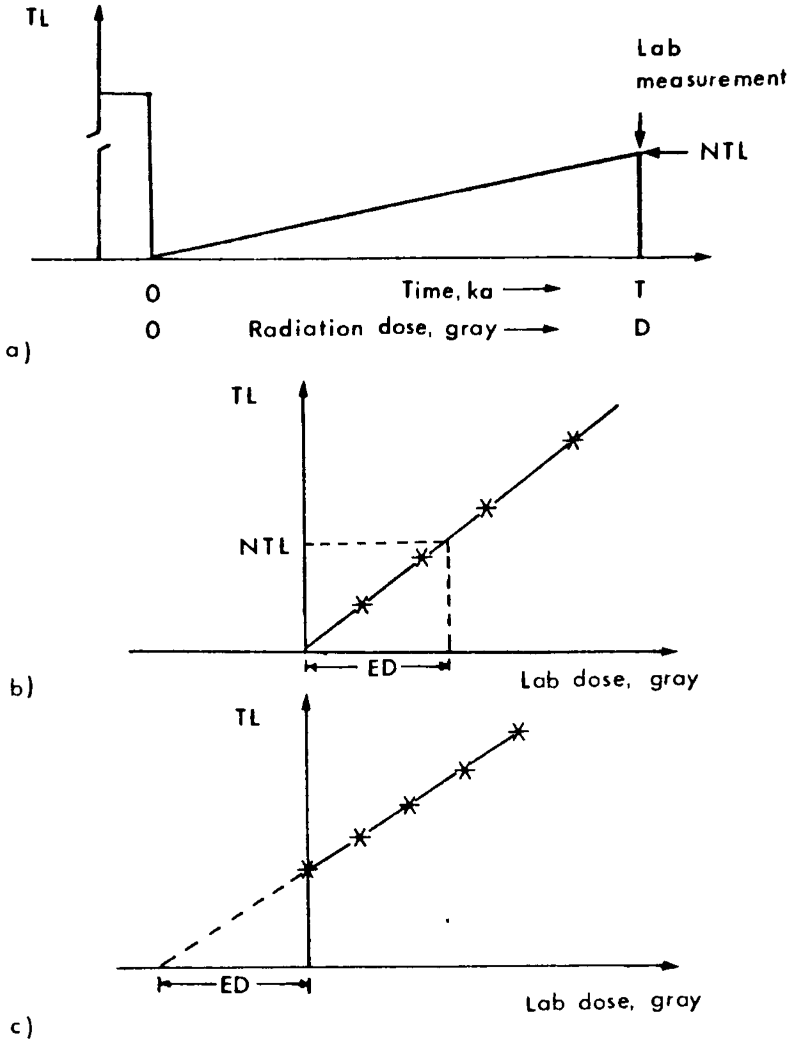
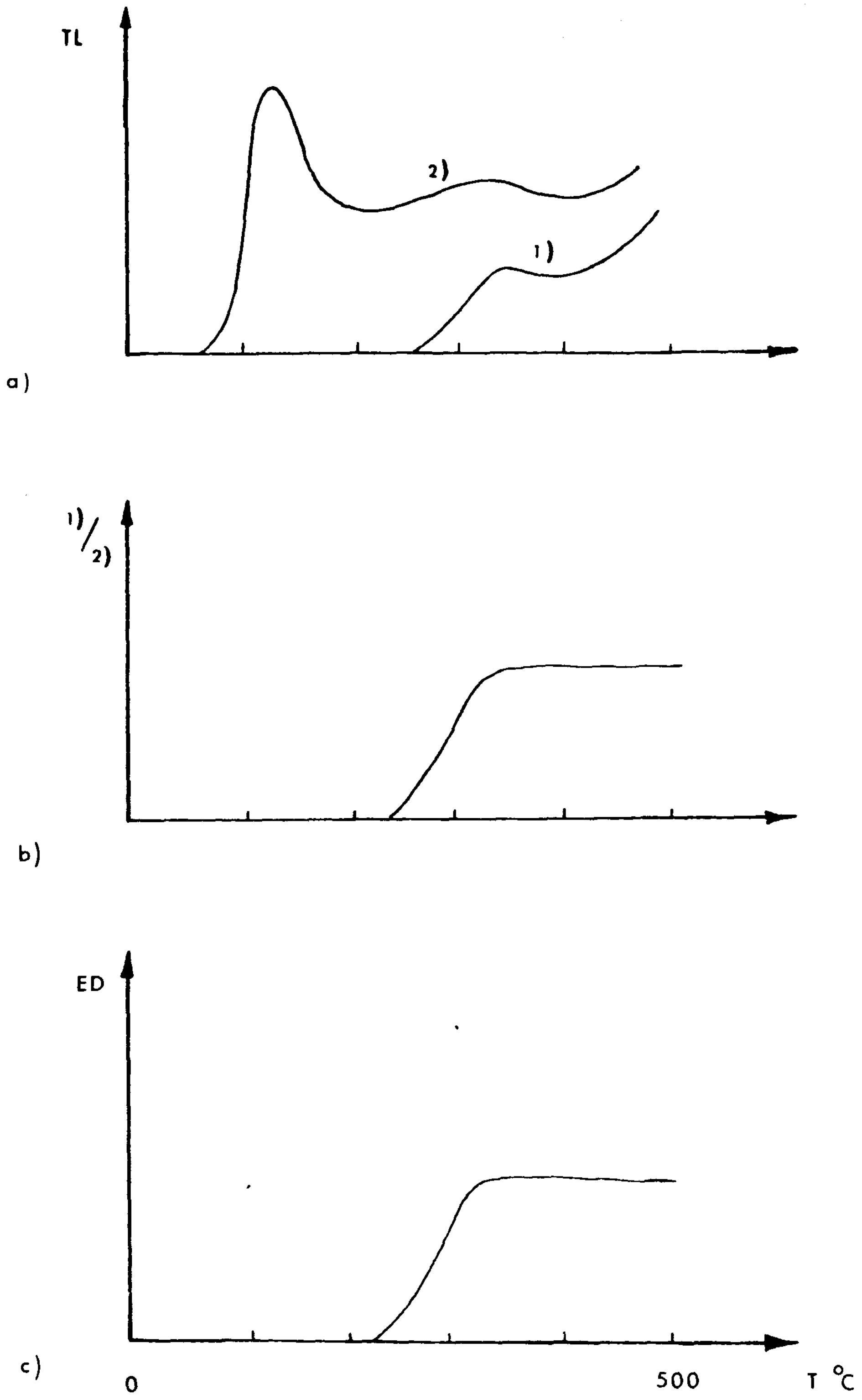


Fig.1.2 The ED determination of pottery.

a) The TL signal vs time for pottery minerals. The TL is zeroed at firing and subsequently grows linearly with time. NTL is the measured value in the lab at age T, dose D.

b) Simple equivalent dose determination: the TL is measured and matched to the TL vs dose curve obtained from subsequent measurements.

c) ED determination circumventing the effect of sensitivity changes with heating which can invalidate b). Several identical samples are given different doses and the ED is obtained by linear extrapolation.



The plateau test for pottery minerals.

Fig.1.3 a) Typical glow curves 1) NTL and 2) Similar sample given added γ dose.

b) 1)/2) showing a plateau.

c) ED obtained as in Fig. 1.2 vs glow curve temperature, also showing a plateau.

where D_α, D_β, D_γ & D_c are the annual dose rates due to alpha, beta, gamma and cosmic ray radiation, respectively. (1.2) is known as the 'age equation'. The relevant dose rates are calculated either by alpha scintllation counting and chemical analysis of the sample and it's surrounding soil or by direct in-situ dosimetry. There are two main ranges of grain sizes of minerals used in dating pottery.

1. The grain size that receives the full alpha dose(2-8 μm) are used in the fine grain method (Zimmerman, 1971).
2. The grains large enough for the average alpha dose to be small but attenuation of beta dose is insignificant(90-125 μm) are used in quartz inclusion method (Fleming, 1970) .

Equation 1.2 is used for the 'fine grains' . In the quartz inclusion method, where quartz grains of about 100 μm obtained from pottery fabric are used after etching away the outer alpha irradiated layer with HF acid treatment, the relevant age equation is,

$$T \text{ (years)} = \frac{\text{ED (gray)}}{D_\beta + D_\gamma + D_c \text{ (gray per year)}} \quad . \qquad (1.3)$$

where the ED has been determined using only quartz grains obtained from the pottery.

1.4 TL Dating of Sediments

The major difference between TL dating of sediments and pottery is the lack of a well defined zeroing of TL of the former. Consequently, there is some uncertainty concerning what event is being dated. But a great deal of evidence for the existence of some kind of zeroing has been accumulated during the past decade of TL research. One of the common features of any sediment section is that the TL increases with depth, which suggests that the alleged zeroing mechanism should be associated with time of weathering, transport and/or deposition of these sediments. If this is not the case, the TL would have been accumulated since the time of crystallization of the minerals. Hence one should be able to see the TL being saturated in them, which is not what one usually finds.

1.5 R-Γ Method

Various workers have studied the effect of light on the TL response of a number of minerals (Tite, 1968). Huntley and Johnson (1976) and Wintle and Huntley (1979,1980) showed that UV light and sunlight drastically reduce the TL of ocean sediments (Fig.1.4). The latter two found that even after a very long sunlamp exposure time there still remained a finite residual TL signal.

They hypothesised that there are two types of traps responsible for TL in sediments viz,

1. light sensitive traps,
2. light insensitive traps.

They suggested that the light sensitive traps have been zeroed at the time of deposition, i.e, the zeroing mechanism was assumed to be the last exposure to sunlight. The natural signal was divided into two components as,

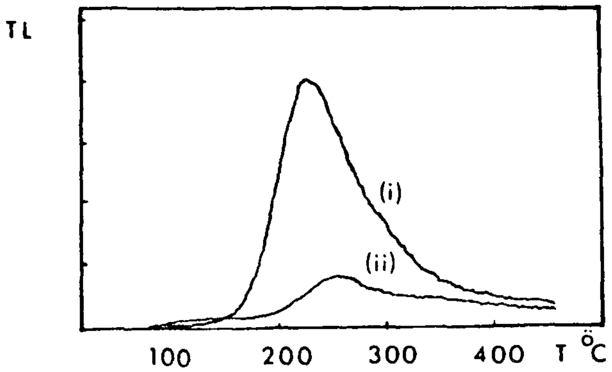


Fig.1.4 Effect of sunlight on an ocean sediment. Glow curve of 4-11 μm grains of a North Pacific ocean sediment. (i) Natural TL, (ii) TL after exposure to October afternoon sunlight for 20 min.

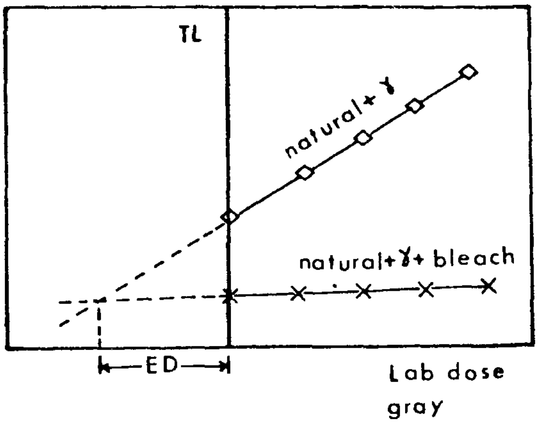


Fig.1.5 The R-Γ method.

$$I(\text{natural}) = I_o + I_d \qquad (1.4)$$

I_o being thought of as the 'residual' TL at sedimentation and I_d being that due to the radiation dose since deposition. They developed a technique called the R-Γ method to determine the radiation dose responsible for I_d component (Wintle & Huntley, 1979). In this method one administers various gamma doses to a number of identical samples, then a half of them including some unirradiated ones are subjected to a fixed sunlamp (SL) exposure. The TL is measured from these samples and the reduction (R) in the TL caused by the SL exposure is plotted versus the gamma dose(Γ). Extrapolation of this plot to R=0 gives an intercept which is taken as the ED. An alternate method of plotting is shown in Fig.1.5, where the ED is obtained by the intersection of N +γ additive growth curve and N +γ+ SL bleach curve.

A summary of sediment TL dates compiled from a recent review paper (Wintle & Huntley, 1982) is given in Table 1.1. In spite of pioneering work on TL dating of sediments by two Soviet scientists, G.V.Morozov and V.N.Shelkoplayas, most of their results have been discredited in the literature, on the grounds of erroneous methodology (Dreimanis et al, 1978; Wintle and Huntley, 1982). According to Wintle and Huntley the results published by most of the other groups, even though they seem to produce plausible dates in comparison with the known dates, are not quite trustworthy because of the lack of published details about the methods used and, in some cases lack of justification of the methods used. The basic hypothesis of the technique about zeroing is not yet proven to be correct. But some workers even claim to produce reliable dates in spite of lack of

Table 1.1

A summary of TL dates reported in the literature.[1]

Authors	AGE RANGE ka[4]	MATERIALS DATED
Morozov,G.V., & Shelkoplayas,V.N. (USSR)	66 - 550 21 - 880	soil, glacial loam loesses
Borsy,Z., Felszefalvi,J., & Szabo,P.P. (Hungary)	105 - 200	loesses, soil
Li,J.-L., Pei,J.-X., Wang,Z.-Z., & Lu,Y.-C. (China)	88 - 660	loesses, soil, sand
Vlasov,V.K., Kulikov,O.A., & Karpov,N.A. (USSR)	1 - 100 60 - 1300	till sediment
Hutt,G.I., Smironov,A.V. (USSR)	26 - 150	till, glaciomarine deposits
Wintle,A.G., & Huntley,D.J. (UK, Canada)	9 - 140	ocean sediments
Wintle,A.G., & Brunnacker,K.[2] (UK, Germany)	15 - 43	loesses
Berger,G.W., & Huntley,D.J. (Canada)	5 - 40	river sediments, soil
Singhvi,A.K., Sharma,Y.P., &[3] Agraval,D.P. (India)	6 - 20	sand dunes

1. References in Wintle and Huntley, 1982.
2. Wintle and Brunnacker, 1982.
3. Singhvi et al., 1982.
4. ka = thousand years.

independent dates to compare with their results. Hence many dates reported in the literature should be regarded with caution. Most of these may be considered as relative dates rather than absolute dates. Wintle and Huntley (1982) proposed a set of criteria for judging TL dates for sediments reported in the literature viz,

1. For each type of material and method used on it, the method should have shown to yield zero age for recently deposited material.
2. For the type of material and method used, the method should have shown to yield correct ages for at least three suites of samples for which reliable ages have been determined independently. These should cover the time span in question.
3. The results of a number of tests which have been found necessary should be reported. These include, but are not limited to, in order of importance: (a)plateau test,(b)anomalous fading test,(c)alpha-effectiveness value and (d)test for non-linearity at low doses.

Once it is assumed that there is an event for dating then the problem is essentially two fold, i.e,

1. Determining the ED, and
2. Determining the effective radiation dose rate.

It has been shown for some time that the R-Γ method yields reasonable ED's for some ocean and land sediments (Wintle & Huntley, 1980; Berger and Huntley, 1982), but before producing acceptable ages by the R-Γ method its applicability should be tested against samples of independently known ages, which is the main concern of this work.

We have selected some peat and silt samples of known ages (C-14) ranging from modern to >70 ka (Table 1.2 & Appendix I) to work with.

In chapter 2 of this thesis the apparatus built for measuring TL is described. The methods used for preparing samples for TL reading is also discussed there. Chapter 3 is devoted to discussing the equivalent dose determinations by the R-Γ method. Evaluation of the effective radiation dose rates is dealt in Chapter 4. Chapter 5 presents the calculation of ages. A discussion and an evaluation of results obtained follows.

Table 1.2

Samples selected for the test program (details are in Appendix I; the sections of the sample sites are shown in Figs.5.1 and 5.2).

SAMPLE	LOCATION	MATERIAL	KNOWN AGE C-14 (ka)
BBP-3	Burns Bog, Delta. B.C.	peat	<0.5[3]
QCIP-1	Cape Ball, Queen Charlotte Islands. B.C.	peat	≥9.16 ± 0.09
MHGP-2	Mary Hill, Port Coquitlam. B.C.	peat	18.60 ± 0.19
MHGP-4	"	peat	18.70 ± 0.17
MHGP-10	"	silt	>27.4, < 28.2
MHGP-9	"	peat	28.2 ± 0.20
MHGP-12	"	silt	>28.2, < 40.2
MHGP-5	"	organic mud	40.2 ± 0.10
MHGP-13	"	silt	>40.2 ± 0.10
LCP-2	Lynn Canyon Park, North Vancouver, B.C.	peat	≥33.00 ± 0.62
LCP-3	"	peat	>33.00 ± 0.62
KRGP-2	Knight Road gravel pit, North Delta. B.C.	silt	$>58.80 {}^{+2.90}_{-2.10}$
KRGP-3	"	silt	$>58.80 \begin{cases} +2.90 \\ -2.10 \end{cases}$
SSP-1	Salmon Springs. Summer. Washington.	peat	$>71.50 \begin{cases} +1.70 \\ -1.40 \end{cases}$

1. ≥ ,> and< signs indicate that C-14 ages are on materials immediately overlying, overlying and underlying the sample respectively.

2. Fission track age of the volcanic ash layer 1m below SSP-1 is 870 ± 210 ka.

3. Age estimated from the sedimentation rate (Hebda, 1977).

II. INSTRUMENTATION

2.1 Introduction

In the early part of this work, some of the samples were measured in the existing TL apparatus (described in Wintle and Huntley, 1980) During the course of the work a new apparatus modelled after the existing one was built with an additional feature.

2.2 TL Apparatus

The TL reader consists of two main components viz,

1. TL oven chamber where the samples are heated at a constant heating rate.
2. Light detection system that measures TL of the sample by photon counting.

A schematic of the entire system is given in Fig.2.1 while a detailed cross section of the TL glow oven is shown in Fig.2.2.

2.2.1 Glow Oven

The sample on an Al disc was heated on a kanthal heating strip (3.5 cm * 2.5 cm * 0.035 cm) in an evacuable chamber. A thermal compound (Wakefield Engineering) was used to make a good thermal contact between the Al disc and the strip. The kanthal strip was mounted on two Cu posts of which one was fixed while the other was sectioned parallel to the strip so that it could slide along a groove to compensate for the thermal expansion of the strip. The heating strip and the two Cu posts were Ni plated by

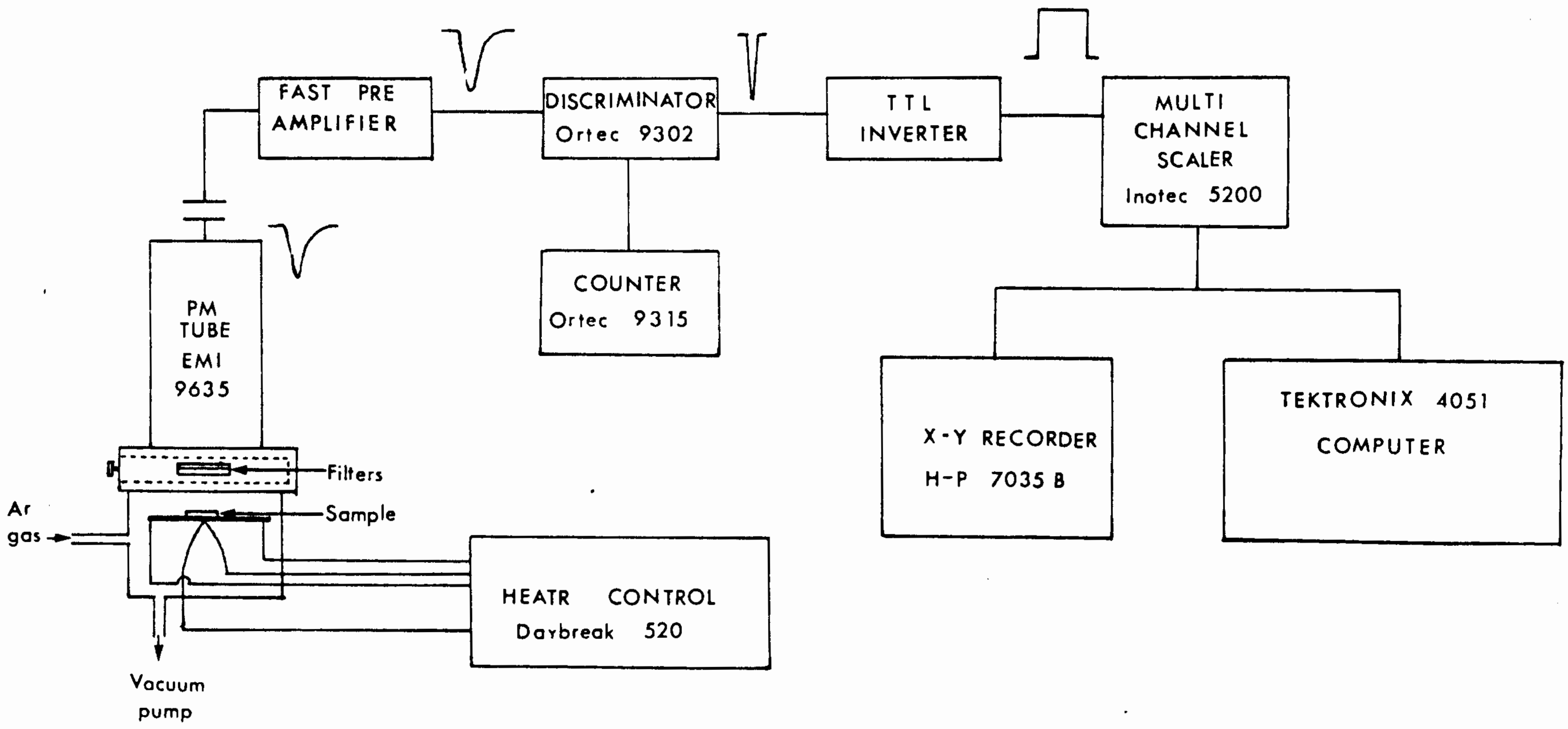


Fig.2.1 Schematic of the TL apparatus.

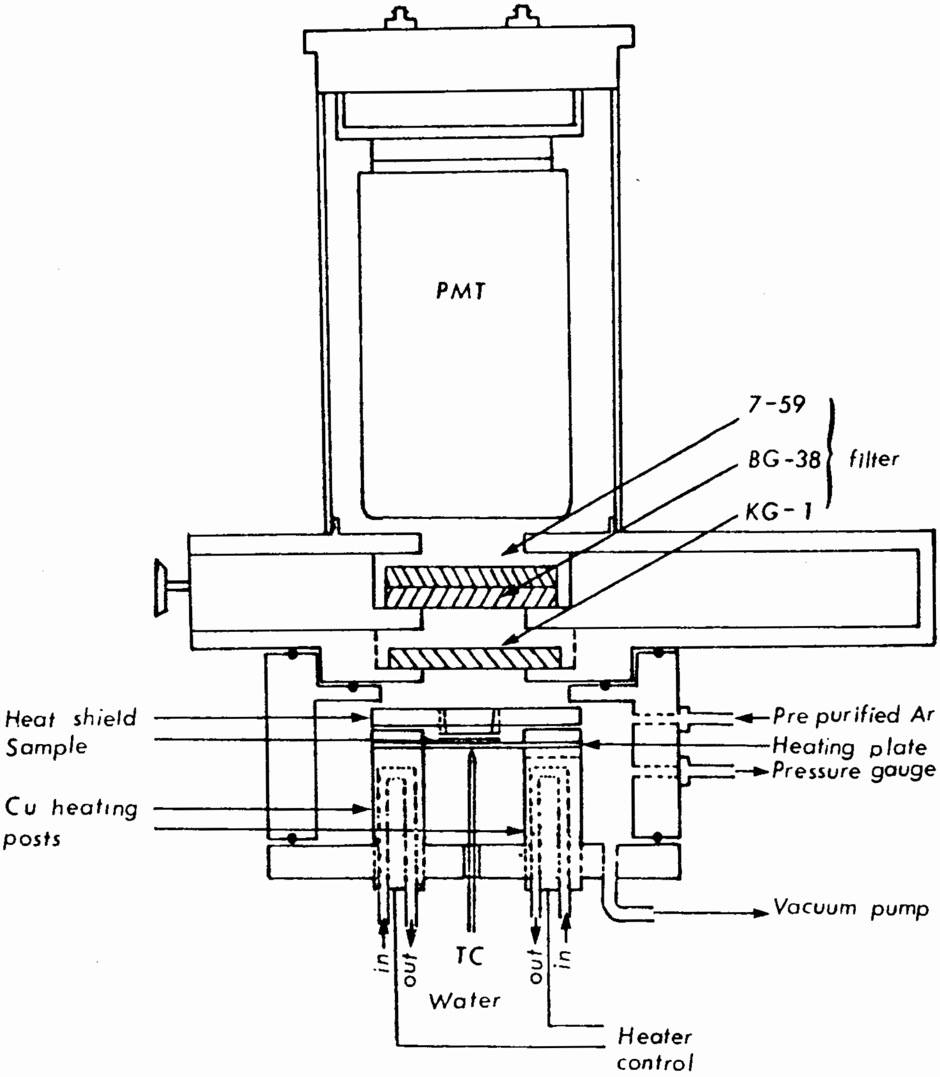


Fig.2.2 Cross section of the TL glow oven. The mineral grains settled on an aluminium disc is placed on the heating plate for TL measurement.

electrodeless plating before mounting them in the apparatus. A ramp generator (Daybreak model 520) connected to the Cu posts provided heating rates from 1-25° C/sec for 0-500° C. A chromel-alumel thermocouple spot welded underneath the kanthal strip monitored the glow temperature. A thermocouple vacuum gauge measured the pressure in the chamber. The two Cu posts were water cooled so that oven recycle time was short and the surrounding was kept at a constant low temperature as well. A vacuum tight gas line carried pre-purified Ar into the oven chamber. Before entering the TL oven the Ar was passed through a purifier cartridge and a getter. The cartridge removed oxygen, hydrogen, carbon monoxide, carbon dioxide and any water vapour left (Matheson[2] Hydrox purifier model 8301). Before heating the sample, the chamber was evacuated (pressure less than 10 micron), flushed with Ar, again evacuated, and finally a continuous flow of Ar was kept at 1 l/min. The presence of oxygen free (<0.1 ppm) Ar was necessary for quenching non-radiation induced ("spurious") TL (Nash et al., 1967). Additionally, a 5 ml beaker filled up to about a quarter with fresh phosphorous pentoxide was kept in the chamber to absorb moisture.

2.2.2 TL Detection System

A photomultiplier tube with bialkali cathode SbKCs (EMI 9635) is used for light detection. The quantum efficiency of the cathode material of the EMI 9635 falls off sharply beyond 600 nm and is high in the blue region. Its insensitivity to thermal radiation and its very low dark counts makes it suitable for TL measurements.

Generally, the reproducibility of the glow curves obtained by the old TL apparatus was very poor at the high temperature end. The cause for this

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[2] Matheson of Canada, Ltd.
P.O.Box 89, 530 Watson Street East
Whitby, Ontario LIN 5R9, Canada.

was thought to be the inadequate filtering of thermal radiation. The old apparatus does not allow more than one filter to be inserted between the sample and the PMT, in addition to the fixed heat absorbing filter (Wild KG-1). A Corning 5-58 filter had been in use along with this, but one can not readily change this filter when one is performing an experiment.

A major innovation in the new TL apparatus is a facility for the easy change of optical filters by incorporating a drawer-like filter holder in between the sample and the PMT. The heat absorbing filter (KG-1) was fixed to the bottom of the filter holder. The Corning 7-59 and BG-38 filter combination was selected for use owing to their better rejection of unwanted thermal radiation (Fig.2.3 shows the characteristics of the filters used). A brass shield placed on the heating strip on top of the sample, thermally isolated from the heating plate, prevented incandescent light from the plate entering the PMT.

The pulses from the PMT were fed into an amplifier-discriminator (Ortec 9302) after passing through a fast preamplifier (gain 10, rise time 5 ns). The (negative) logic pulses from the discriminator were input into a multi channel scaler (Inotech-5200). However since the MCS accepts only positive TTL pulses a TTL pulse inverter had to be used in between the discriminator and the MCS. A photon counter (Ortec 9315) connected to the discriminator output displayed the number of counts for a preset period of time, independently. The discriminator level was set so that it was at the threshold of the constant signal/noise ratio. The dark count of the PMT was then about 15 $\sec^{-1}$. Data collected in the MCS was transfered to a magnetic tape. Data handling (storing and processing) capability was tremendously improved by the use of a Tektronix 4051 minicomputer. A plot of the glow curve was recorded from the MCS on an X-Y recorder as well.

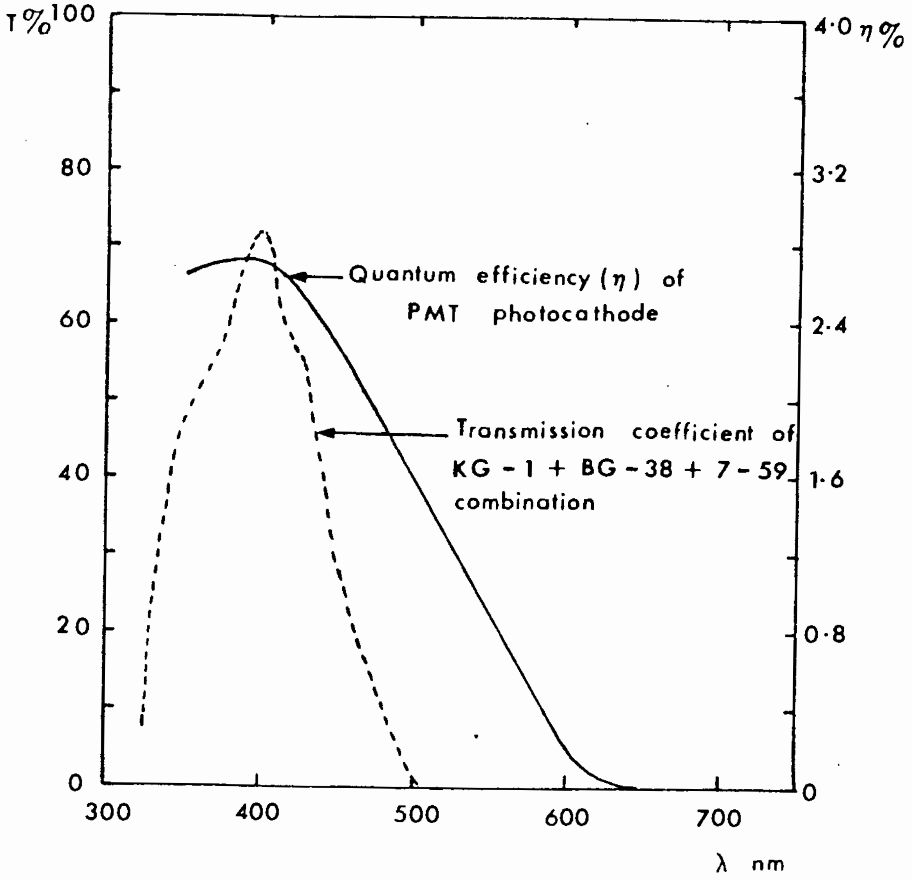

Fig.2.3 The overall transmission characteristics of the filter combination used in the TL apparatus (KG-1 + BG-38 + Corning 7-59), and the quantum efficiency of the PMT photocathode.

Table 2.1

Dose rate to quartz (SiO_2) from Co - 60 source in 'Gammacell -200'.

MONTH	1981 Gy/min.	1982 Gy/min.
January	2.57	2.25
February	2.54	2.23
March	2.52	2.20
April	2.49	2.18
May	2.46	2.16
June	2.44	2.13
July	2.41	2.11
August	2.38	2.09
September	2.36	2.06
October	2.33	2.04
November	2.31	2.02
December	2.28	2.00

1. Dose rates on SiO_2 had been calculated by Dr.D.J.Huntley using the conversion factor 1.05 from the measured value with CaF_2 discs in the Oxford calibration kit by Dr.A.G.Wintle (private communication).
2. The Gamacell 'up-down' time is 5.71 ± 0.28 sec.
3. The uncertainty in the above dose is about 5%.

2.2.3 Laboratory Radioactive Sources

All gamma irradiations of sediment samples were carried out using the Co-60 source in the Department of Biological Sciences at SFU (Gammacell 200). This irradiation unit has been calibrated by Dr.A.G.Wintle in February, 1979 (private communication, Dr.D.J.Huntley) using calcium fluoride discs in the standard Oxford calibration kit. The dose rates to quartz for the period 1980-82 is given in the Table 2.1 after correcting for the half life of Co-60 (5.26 yr) and the absorption of energy in calcium fluoride. The 'gammacell' irradiation time can be controlled by a built in electronic timer from few seconds to 999.9 hrs. The lower limit in the irradiation time was determined by the time taken for moving the sample mechanically to the part where the Co source is located in the irradiation unit. The effective irradiation period for this mechanical movement has to be added to correct the small irradiation times (less than 1 min) as read on the timer. The above correction was determined as follows. The TL induced in a calcium fluoride disc for various irradiation periods set by the timer, ranging from 2-10 seconds was measured. The plot of TL vs. time in seconds extrapolated to TL=0 gave an intercept on time axis which is taken as the effective time. Measurements yielded a value of 5.71 +/- 0.28 seconds.

In the beginning the samples were usually irradiated in small light tight tin boxes by placing them on the circular horizontal irradiation platform of the 'gammacell' and activating the mechanism that moves this platform to the vicinity of Co source. The source is housed in a cylindrical container that encircles the irradiation platform. Subsequent use of the irradiation unit showed some TL variability in irradiated samples, specially at higher irradiation periods. This was attributed partly to sample reproducibility and partly to the spatial variation of the dose rate in the unit. A cylindrical plastic container which fits to the 'gammacell' irradiation platform was found and 12 slots, equally spaced around a circle concentric with the outer rim of the plastic container were made on a thick RTV silicone rubber layer in it. This enables one to irradiate 12 samples simultaneously at a constant dose rate. As expected the use of this sample holder reduced the TL variability in most of the samples. TL measurements with discs prepared from LCP samples showed that the dose rate at the above slots is 4.2% +/- 2.0% higher than that at the centre of the 'gammacell', where the samples were irradiated in a tin box.

All the alpha irradiations were done with the aid of a commercial alpha irradiator (ELSEC Alpha source unit Type 721 S/N 3 with Am-241 2 mCi sources, Aitken, 1978-a). After placing the samples on the irradiation posts the chamber of the unit was evacuated to avoid attenuating the alpha radiation by air. Irradiation time could be controlled up to six hours, by an electronic timer attached, within an accuracy of about one minute.

For the calibration of the dosimeters which were used to measure the in-situ dose rates at the sample sites, a Ra-226 gamma source (10 mCi) was used. This source was calibrated in terms of a laboratory beta source (Sr-90/Y-90) which, in turn was compared with the 'gammacell' by means of the above calcium fluoride disc. This enables one to know the dose rate at the gamma irradiation unit with Ra-226 source in terms of that of Co-60 source in the 'gammacell'. An intermediate source had to be used because of the huge difference in the strengths of the available gamma sources.

An 'order of magnitude' calculation, in the point source approximation, for the Ra-226 linear source gave a tentative value of 2 milligray/hr (Young and Batho, 1964) at 20 cm from the source. The actual distance from the Ra-226 source to the irradiation place is 19.7 cm. Calibration with a calcium fluoride disc yielded the dose rate of Ra-226 source to be 5.33 +/- 0.35 milligray/hr. The higher measured value reflects the fact that neither the effect of scattering nor that of the secondary electrons produced were included in the above calculation.

2.3 Sample Preparation

Usually, the sediment samples were collected with suitable precautionary conditions so as to minimize their exposure to sunlight and were stored in light tight containers. Even so, an outer layer of thickness of about one centimeter is removed from the sample prior to their use to avoid using any material that has been exposed to sunlight. Moreover, all the experiments were carried out in subdued orange light.

TL was to be observed from the minerals in the sediments. Hence one has to isolate them from the collected samples. Primarily, four steps were involved in doing this viz,

1. Removing the materials other than the minerals.
2. Removing the materials on the minerals which obscure TL.
3. Seperating the cleaned minerals into a required grain size.
4. Settling the separated minerals on Al discs for handling them.

Very large grains of minerals (>68 micron) and woody materials were removed from the sample by filtering through a 68 micron sieve. 1/100N HCl acid was then added to dissolve any carbonates in the sample. After about 2 hrs in the solution, the acid was poured away. The sample was rinsed with distilled water and 10% hydrogen peroxide was added to it to oxidise the organic materials. Removal of carbonates speeds the oxidation process. The hydrogen peroxide was replaced daily, and the solution was stirred about twice a day. This procedure was carried out until the material became as light in colour as possible indicating decomposition of organics. This usually took about 3 days. At the end of this period, the sample was rinsed well in distilled water to wash away any hydrogen peroxide left.

The iron oxide layer on minerals is considered to be a TL obscuring agent. Hence the so called CBD (citrate bicarbonate dithionate) treatment was followed for removing iron oxide coating on minerals. For each 10 gm of sample (assuming 5% iron oxide; private communication with Dr. G.W.Berger) 50 ml of a previously prepared solution of sodium citrate and sodium bicarbonate (71 gm of citrate and 8.5 gm of bicarbonate in 1 l of water) was added to which a 1 gm of sodium dithionite was subsequently added stirring the solution vigourously. The sample in this solution was kept at room temperature for 12 hrs (Jackson, 1979). At the end of this period the solution was poured away and the sample was washed thoroughly with water.

When a sample was examined with an optical microscope clumps of mineral grains were usually seen. The presence of such coagulation hampers a good grain size separation. Therefore, the cleaned sample was then placed in 1/100 N sodium oxalate solution for segregating the minerals. Sodium oxalate acts as a disperser here usually, replacing the ions such as calcium and magnesium, which are responsible for such flocculations of minerals, with sodium. The period of dispersion depends on the sample but the optimum was found to be three days (private communication: Dr.G.W.Berger). Usully, dispersion was aided by placing the sodium oxalate solution in an ultrasonic bath forafew minutes.

The well dispersed sample was then separated into the required grain size by Stokes settling. In this study most of the samples were separated into the 2-8 micron range (see Chap.3). To do this the sample was transferred to a tall measuring cylinder and distilled water was then added up to a height of 20 cm. After shaking the cylinder thoroughly the sample was kept undisturbed for a period of 1 hr to settle. At the end of this period all the materials with effective diameters greater than 8 microns would settle down to the bottom of the cylinder as dictated by Stokes law. What remained still in suspension was decanted. This fraction contained particles with effective diameters less than 8 micron, which was again settled as before for a period of 8 hrs at 10 cm column of water. After this settling period, the particles still in suspension are those with effective diameters less than 2 micron. This fraction was decanted away. The rest was mostly grains in the 2-8 micron effective diameter range. To get rid of any smaller particles which could get dragged down with larger ones, the above process was repeated until a very clear supernatant was obtained. It should be noted that this grain size separation was not an exact one. The separated sample may contain a small fraction of grains with diameters less than 2 microns as well as some with diameters greater than 8 microns but the dominant fraction would be those between 2 and 8 microns (Zimmerman, 1971).

The next step was to settle the separated grains on clean Al discs (diameters between 9.99-9.95 mm and thickness 0.5mm) which were previously etched with dilute HF acid. First, the clean Al discs were placed in glass vials of internal diameter of 1 cm. Enough methanol was squirted in to the vials to cover the discs so that by pushing the disc gently against the glass, one could make sure that no air bubbles were trapped between them. This methanol was then discarded.

Meanwhile, the separated grains were made into a thin suspension in methanol in a separate container. One ml of this suspension was transfered to each vial containing an aluminium disc by a micropipette. Before each transfer the suspension was shaken well to make it uniform. A set of vials prepared thus was stored away undisturbed for drying. Usually, about three days were taken for complete drying at room temperature. This could be accelerated by placing the vials in an oven with a temperature of 35°C where one can dry them within 16 hrs. The settled materials adhere to the etched Al discs quite well and the discs can be removed from the vials without losing any material from them once they are dry. It is the usual practice of this laboratory to make the initial suspension of the sample in methanol at such a concentration that one is barely able to read the writings on a test tube containing the suspension by looking through it. This made sure to a certain extent, that the samples prepared at different batches had the same amount of materials on them (roughly 1 mg). For one sample, about 35 discs were prepared at a time, and their TL reproducibility had to be better than 3% for any serious work. Usually, 18 discs were used along with another 11 for R-Γ method and 'a'value measurements respectively. Six discs were generally used for testing for anomalous fading in the sample (see Chap.3). Generally, one could prepare discs with good reproducibilty by following the above procedure.

III. EQUIVALENT DOSE DETERMINATION

3.1. Introduction

A Sylvania 275 watt sunlamp was routinely used to bleach the samples for the R-Γ method. This inexpensive lamp has a somewhat large UV output. Exposure to such light would empty traps that were not emptied during the zeroing mechanism that occured in nature. Ideally, one would like to simulate the exact sunlight exposure that occured in zeroing, which is unknown. However as long as the standard laboratory light exposure is less than or equal to the natural zeroing one at all wavelengths, one should obtain the correct ED. Hence the UV portion of the sunlamp below 300 nm was cut off by an optical filter (Corning 0-52) Fig.3.1-a & 3.1-b. For river sediments the Corning 3-67 filter was used. This filter had yielded good results with such samples (Berger & Huntley, 1982). Samples to be bleached were kept under the sunlamp (SL) about 35 cm below it in an aluminium housing. The bleaching period was usually for 8 hrs or 16 hrs, but for some sensitive samples, exposure periods as short as 2 hrs was not uncommon.

TL was measured from natural (N), gamma irradiated ($N + \gamma$) and sunlamp bleached ($N + \gamma +$ SL) discs (Fig.3.2-a). First, the amount of data collected in the MCS was reduced in size by summing the TL data (in photons per channel) in five channels consecutively, which corrsponds to adding up the TL for about 10° C intervals along the glow curve. This made it easier to handle a large amount of data. The $N + \gamma$ and $N + \gamma +$ SL curves were obtained by fitting least square straight lines to this 'compact' data for

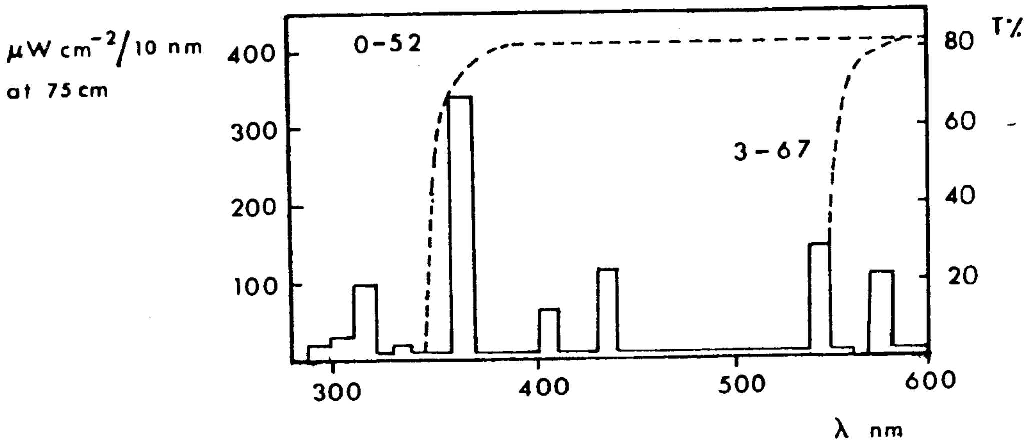

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Fig.3.]-a Spectral energy distribution of the Sylvania sunlamp. The dashed lines are the transmission coefficients of the Corning 0-52 and 3-67 filters which were used with the sunlamp for bleeching.

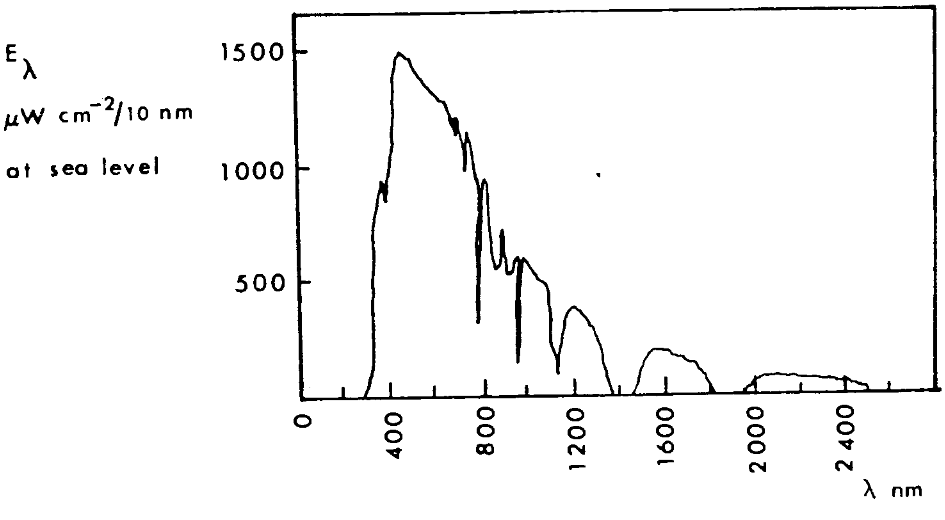

Fig.3.1-b Solar spectral distribution at the sea level

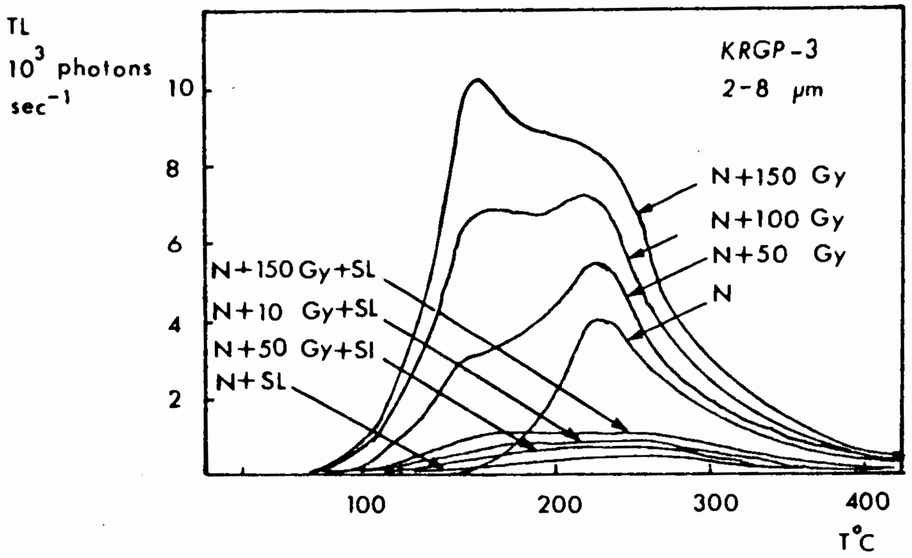

Fig.3.2-a Glow curves of 2-8 μm grains of a typical sediment (KRGP-3). Natural TL, after laboratory γ doses and after exposure to sunlamp for 8 hrs.

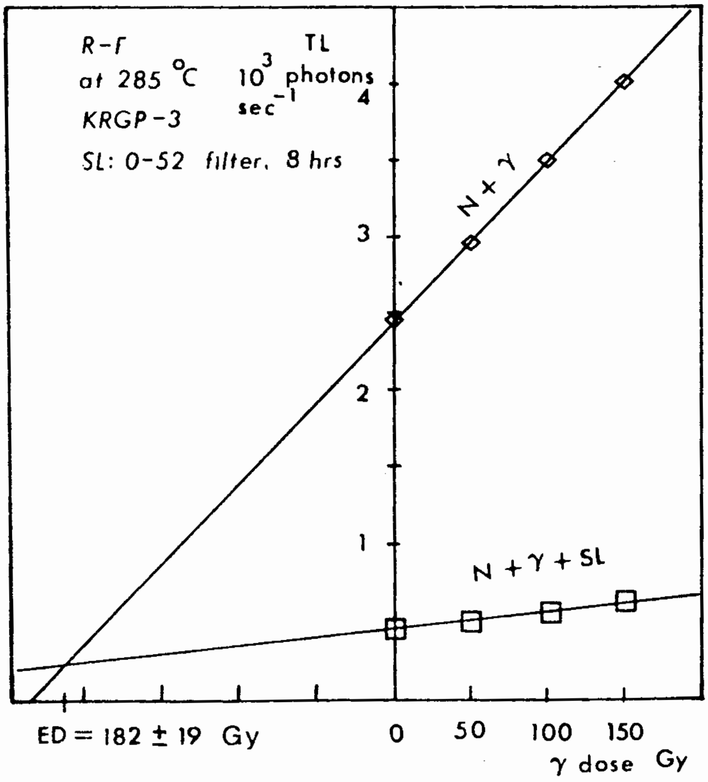

Fig.3.2-b The R-Γ method on the above sample at 285 °C. The point of intersection of Natural (N) +γ and N +γ + bleach curves yields the ED at this glow curve temperature.

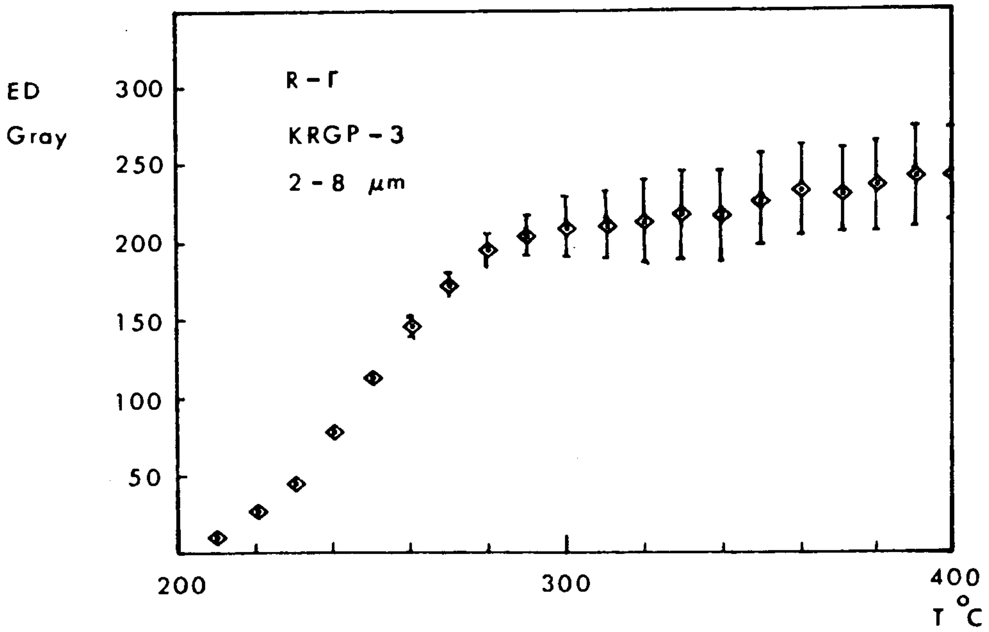

Fig.3.2-c ED vs. glow curve temperature plot. The plateau value is taken as the ED for sample.

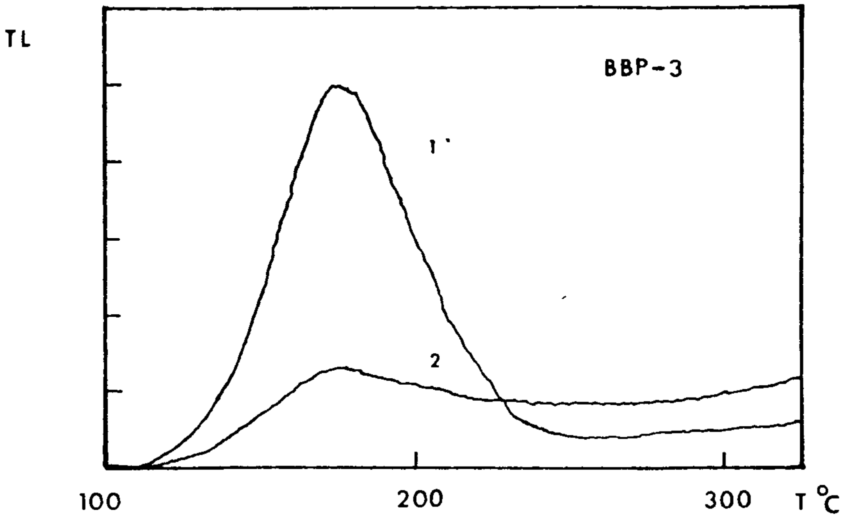

Fig.3.3 The effect of H_2O_2 treatment on BBP-3.
1) The sample had been in 30% H_2O_2 with Na-acetate buffer at pH = 5.00 for 1 day.
2) The sample had been in 10% H_2O_2 for a few days.

each temperature (or 5 channels) at 10° C intervals (Fig.3.2-b). The intersection of the above two lines gave the equivalent dose for that particular temperature. A suitable ED for the sample was determined by obtaining the 'plateau' value (Fig.3.2-c) from a plot of ED vs. temperature.

3.2 Grain Size Dependancy of Equivalent Dose

FRSS-1 and BBP-3 were used to investigate the grain size dependancy of the ED. This investigation was also a check to see if the R-Γ method yielded zero ED for modern samples.

A series of discs with mineral grains in the size ranges of

1. 1-2 micron
2. 2-4 micron
3. 4-8 micron
4. 8-16 micron
5. 16-37 micron

were prepared with both samples. Severe problems were encountered when making reproducible discs with BBP-3 sample. The sample being a 'modern' peat, getting rid of organic materials without altering TL characteristics of the sample was a major problem. The ususal pre-treatment with 10% hydrogen peroxide was found to be inadequate. Two weeks treatment with peroxide still left a lot of organic matter visibly unoxidised and this caused the material to get only weakly attached to the discs. Most of the discs prepared had their materials peeled off from them even before heating. Fig.3.3 shows the glow curves of BBP-3 (37-62 micron) with usual 10% peroxide treated (for few days) and 30% peroxide treated in Na-acetate buffer of pH 5.0 for one day. Even though the latter treatment showed a remarkable improvement in removing organic litter in the sample, its adverse chemical action on the material, as shown by the change in the glow curve shape rendered the method unreliable for TL study.

With most fractions of BBP-3 it was nearly impossible to get a meaningful ED due to poor reproducibility of the discs prepared. However, it was evident that the ED's are <5 Gy, from some fractions which behaved better than the rest but not quite well enough.

The results for sample FRSS-1, obtained by R-Γ method are shown in Fig.3.4-a and Fig.3.4-b. The two bands indicate the one standard deviation error limits calculated for the ED's. Glow curves for all sized fractions were of the same form. The results show that there is no significant dependancy of ED on grain size.

The grains of 2-8 μm range were selected for the routine measurements as they would have received the full alpha dose, the average range of alpha particles from natural radioactivity being about 25 μm (Aitken, 1974) in soil.

3.3 Glow Curve Characteristics

Fig.3.5-a and Fig 3.5-b show the typical glow curves of peat and non-peat materials studied (heating rate 4.5° C/sec). All non-peat samples had glow curves of similar shape while the rest of the samples showed a somewhat diverse behaviour. Specially that of QCIP-1 sample which could be of a different type of minerals from the rest showed the greatest contrast.

The spectral distributions of the TL of the samples were measured with the aid of a series of interference filters, keeping one at a time in the filter holder of the TL apparatus. Measurements were done in the wavelength region of 404-587 nm. Fig.3.6 shows the results of measurements, corrected for both transmission characteristics of the filters and the

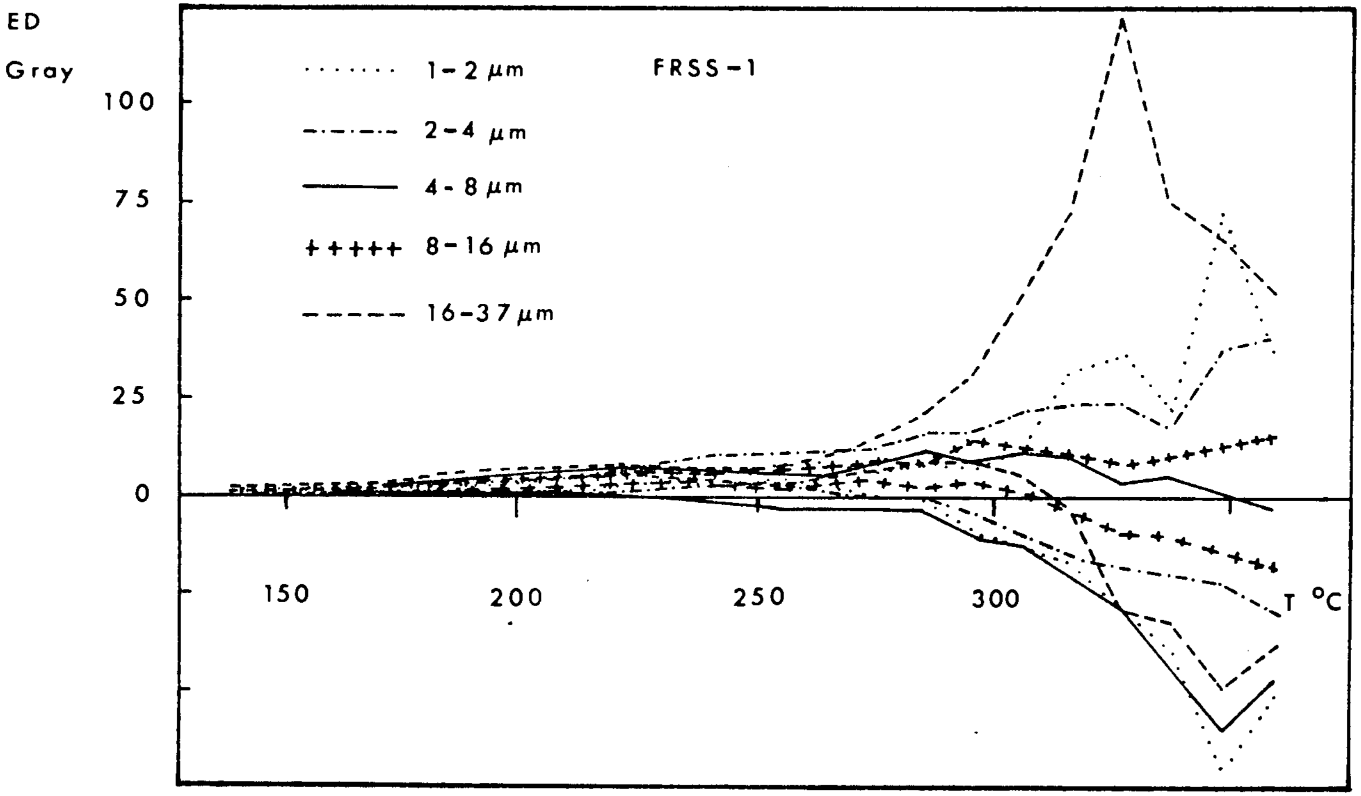

Fig.3.4-a ED vs. temperature for different size ranges of grains of FRSS-1. The two bands indicate 1σ error limits.

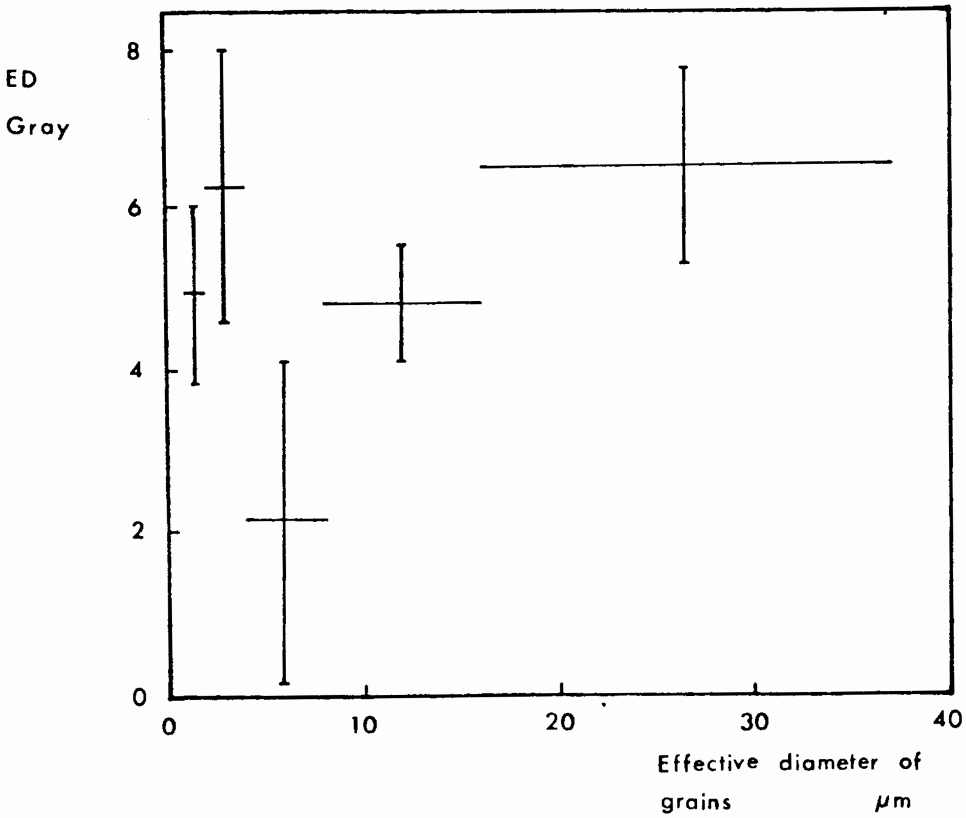

Fig.3.4-b ED vs. grain size plot for FRSS-1 at 285° C (within the plateau region).

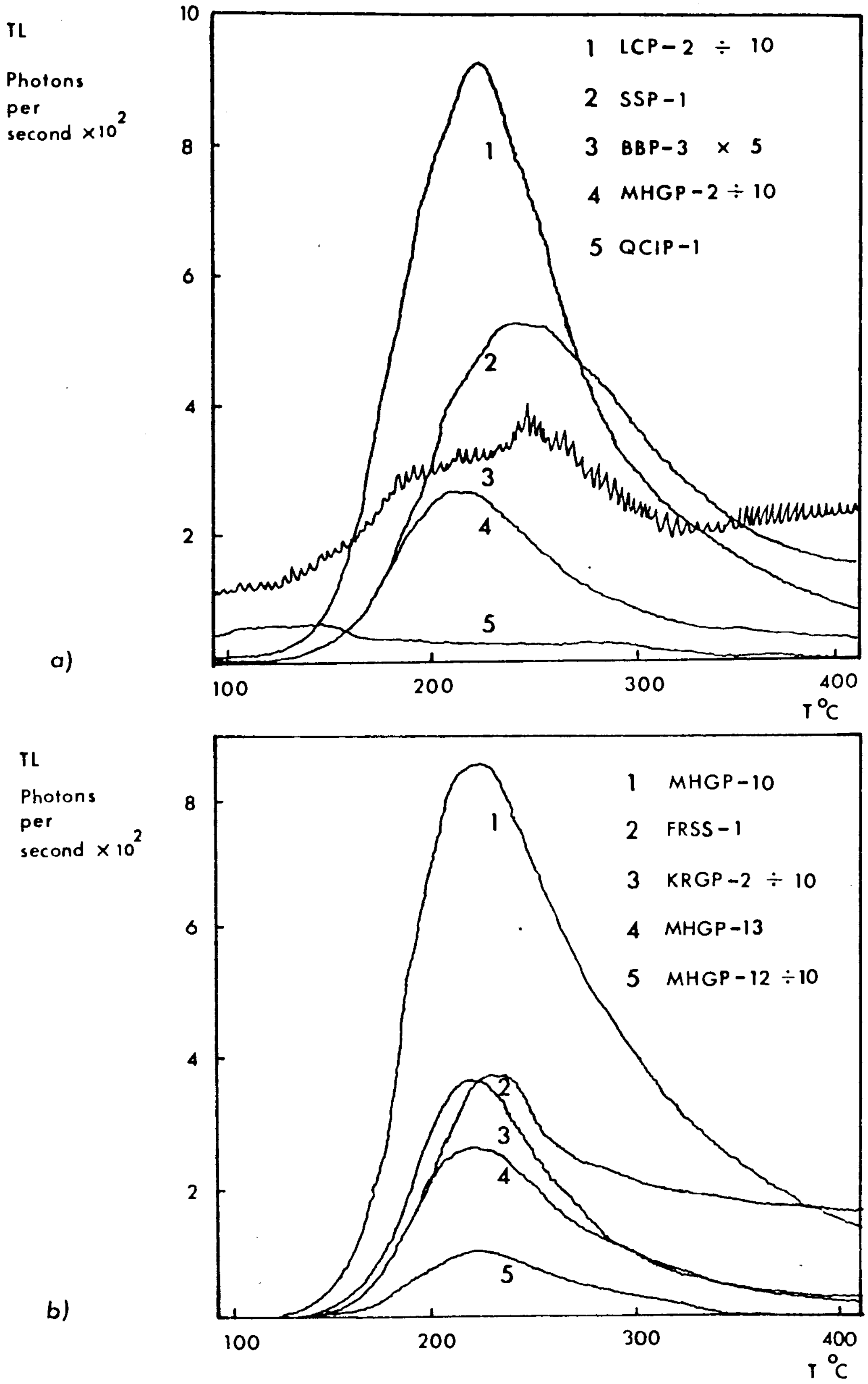

Fig.3.5 Natural glow curves (heating rate 4.5° C s^{-1})
a) Peat samples.
b) Non peat samples.

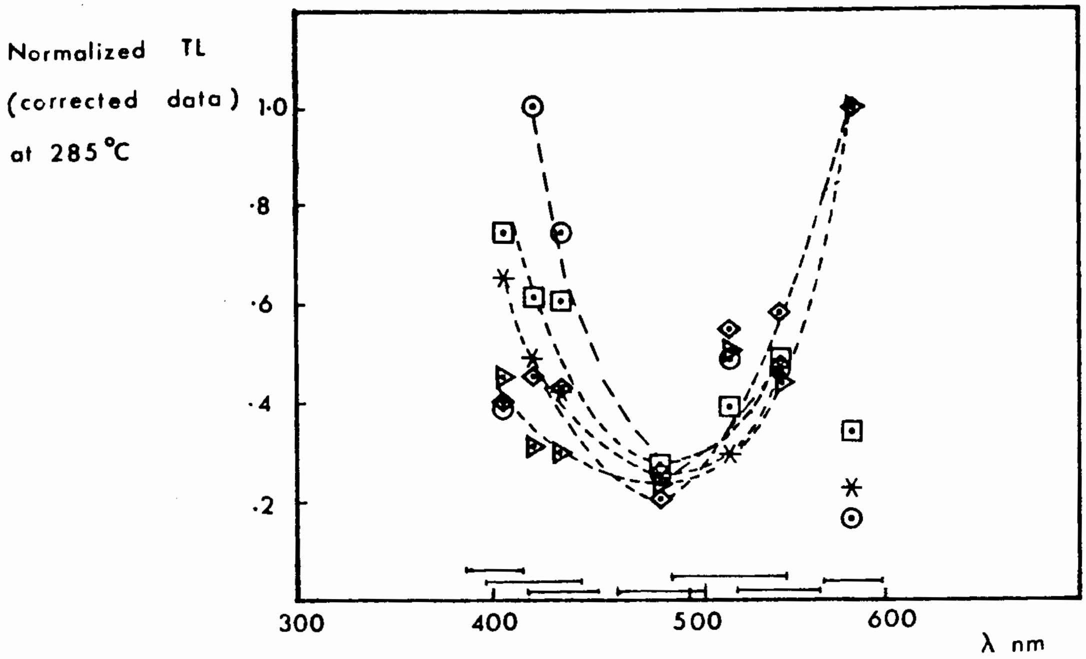

Fig.3.6 Spectral distribution of the TL of samples. Corrections were made for the transmission of the filters used and for the quantum efficiency of the photocathode of the PMT.

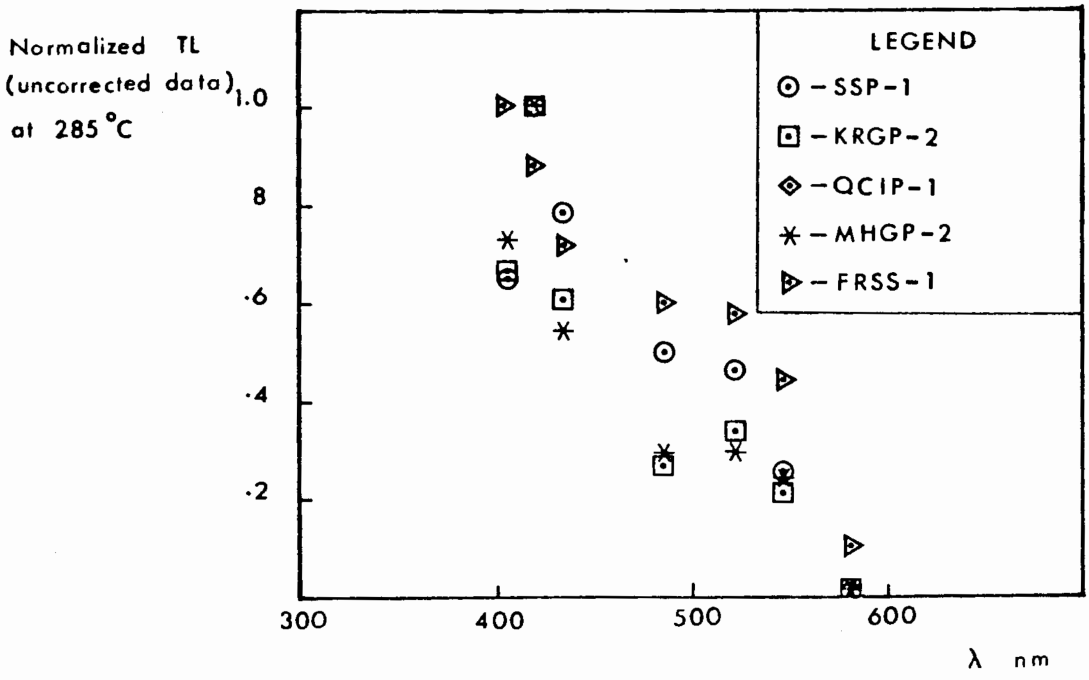

Fig.3.7 Uncorrected data of the Fig.3.6.

quantum efficiency of the photomultiplier tube. The reheat glow curves were subtracted from first glow curves to get rid of the thermal radiation. The red end of the distribution should be regarded as approximate since the figures used for the quantum efficiency were obtained from the general information supplied by the manufacturer and they were very low in this region; the uncorrected results are hence given in a separate plot Fig.3.7. The general characteristics seemed to agree with the distribution curves published by Bailiff (1979) for quartz extracted from pottery. (TL of all samples had a peak in the blue region ($<$420nm)). However it should be noted that the glow curves indicated that the TL of the samples were dominated by feldspars but not by quartz.

3.4 Growth Curve Characteristics

Growth curves obtained by laboratory gamma irradiations ($N+\gamma$) are presented in Fig.3.8. In those cases, where the growth curves showed non-linearity least square quadratic curve fitting was used to obtain ED's and their errors (MHGP-9 in R-Γ ; MHGP-10 and 12 in R-α data, see section 4.3). Even though an exponential fit was desirable where the growth curve started to flatten out noticeably at high doses, a quadratic fit was considered to be adequate in the low dose regime.

3.5.1 Anomalous Fading

When the TL of two discs of a sample which have been given the same laboratory dose at two different times are measured after the last irradiation usually what one observes is shown in Fig.3.9. The loss of TL at low temperature region is due to thermal emptying of traps. However, the loss of TL in high temperature region is in disagreement with the mean lifetimes of electrons in traps which are responsible for that part of the

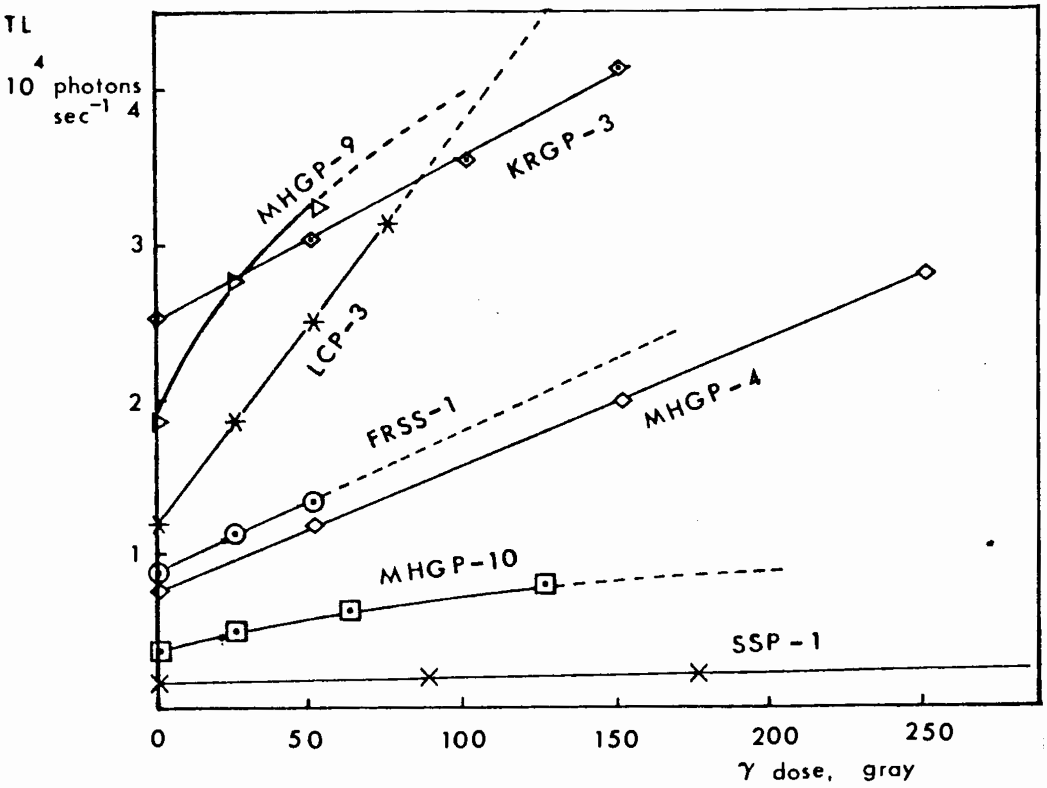

Fig.3.8 The TL growth (1st) curves at 285°C. Least square straight lines and least square quadratic curves are fitted to data.

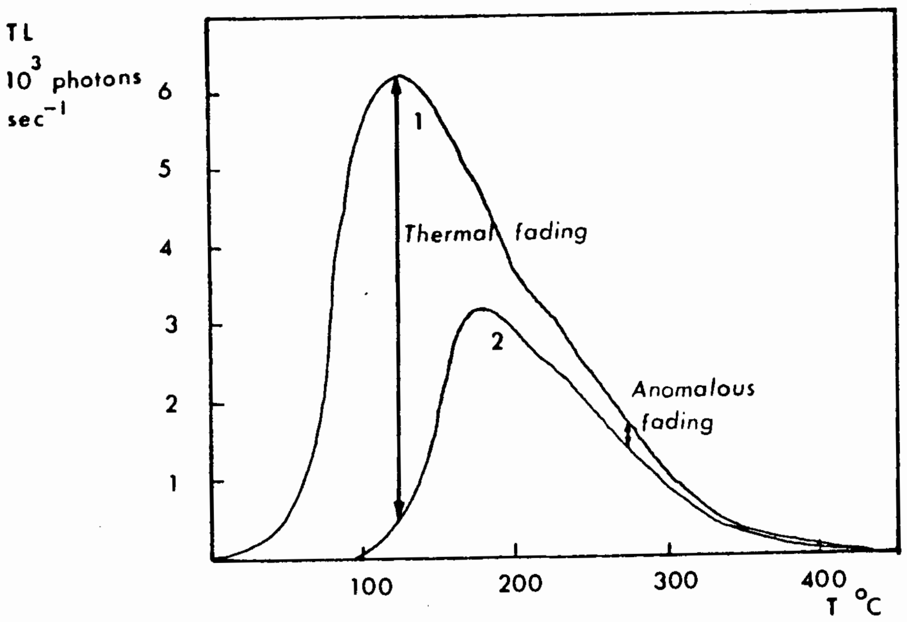

Fig.3.9 Anomalous fading. TL glow curves of 1) a sample that had received 250 Gy γ dose 6 months ago 2) a sample that had received 250 Gy γ dose a few minutes ago.

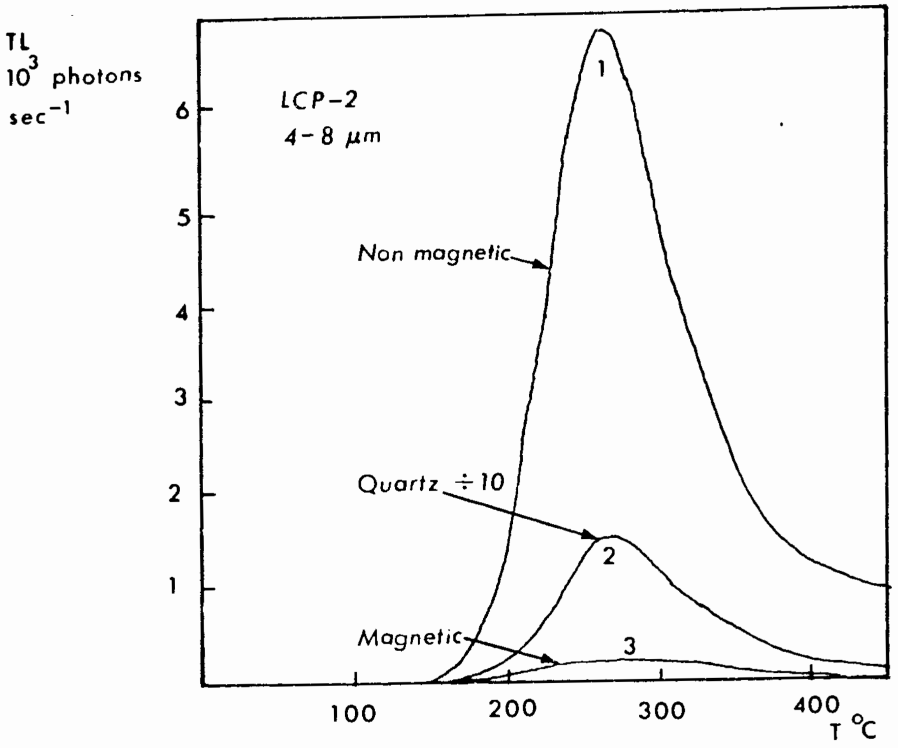

Fig.3.10 Glow curves of LCP-2.
1) 4-8 μm, non magnetic fraction.
2) 4-8 μm, quartz fraction from HF treatment.
3) 4-8 μm, magnetic fraction.

glow curve (Wintle, 1977). This phenomenon is called anomalous fading and is not a process well understood yet.

Tests for detecting the presence of this phenomenon were routinely carried out for all the samples. Six discs per sample were usually used. First, three discs were stored away after being given a laboratory gamma dose . The other three discs were given the same gamma dose after at least one week and TL were measured from all discs. The ratio of the TL of two sets of discs gave a measure of fading during the storage period.

Several workers have studied this worrisome phenomenon which is a major problem in TL dating. Different minerals have different fading characteristics (Wintle, 1977). Quartz does not show fading while some feldspars do .

In an attempt to find a mineral fraction which does not show anomalous fading two methods were tried viz,

1. Magnetic / Non-magnetic fractioning.
2. Quartz separation.

3.5.2 Magnetic / Non-magnetic Separation

Sized (4-8μm) grains of LCP-2 and LCP-3 were separated into two fractions by passing through a Frantz Isodyne magnetic separator as described by Mulhern et al (1981). The effectiveness of the separation was checked by measuring the TL of the two fractions as well as by X-ray diffraction (XRD). Non-magnetic minerals are known to be dominant in emitting TL. Fig.3.10 shows the TL characteristics of the separated fractions where their relative TL level is about 25 times at the peak around 250° C. The emergence of quartz peaks in XRD patterns was also very encouraging (Fig.3.11-a). Another XRD peak of an unidentified mineral was also present in the pattern. The patterns were obtained with two passes from 8.3° to 60° of 2θ at 2° per minute (Cu anode, 40 kv, 30 mA).

Fading tests done on the LCP-2 samples showed that the magnetic fraction did not fade while the non-magnetic fraction did (Table 3.1).

However the ED obtained for the magnetic fraction is not significantly higher (A similar effect has been observed by Dr.G.W.Berger in a sample from a glacial till, Woodbridge Cut, Sunnybrook Till 4-11 μm ; private communication). A surprising result was that another sample (LCP-3) from the same peat layer as LCP-2 did not show fading. The ED's and the results of anomalous fading tests on the samples studied are given in Tables 3.1-a and 3.1-b.

3.5.3 Quartz Separation

The simple procedure of treating the sample with HF acid for short period of time was tried for separating quartz (Fleming, 1979). HF acid is known to dissolve feldspars more readily than quartz. Sized samples were kept in 0.5 N HF acid solution for about one hour at the end of which the sample was washed well with distilled water before preparing discs.

The XRD data obtained from the sample treated with HF acid had the characteristic quartz peaks (Fig. 3.11-a) while the untreated sample yielded only a noisy pattern devoid of any peaks (Fig. 3.11-b).

Quartz (about 70%) separated from LCP-2 yielded a comparable ED with that from the polymineralic sample (Fig. 3.12). The ED obtained from the HF treated fraction of QCIP-1 gave a higher value than that from the untreated fraction but comparable within the experimental error.

Most of the samples from the Mary Hill Gravel Pit and SSP-1 did show fading. KRGP-2 and KRGP-3 did not show detectable fading, but it should be kept in mind that the failure to observe fading in a short term fading

Table 3.1-a.

Equivalent doses and anomalous fading results
of peat samples.

SAMPLE[1]	GRAIN SIZE µm	SL EXPOSURE		ED Gy	ANOMALOUS FADING	STORAGE TIME(d)
		Filter	Time(hr)			
BBP-3	4-8	3-67	16	3 ± 2	6% ± 4%	150
QCIP-1:						
polymineralic	2-8	0-52	16	6 ± 2	4% ± 20%	8
HF treated	2-8	0-52	2	11 ± 4	2% ± 7%	12
MHGP-2	4-11	3-67	16	33 ± 4	2% ± 3%	150
MHGP-4	4-11	3-67	16	45 ± 10	12% ± 3%	12
MHGP-9	4-11	3-67	16	35 ± 6	7% ± 3%	180
LCP-2:						
polymineralic	4-11	0-52	8	18 ± 2	11% ± 3%	15
non magnetic	4-8	-	-	-	10% ± 4%	12
magnetic	4-8	0-52	8	23 ± 2	3% ± 5%	12
HF treated	2-8	0-52	8	19 ± 2	2% ± 7%	8
LCP-3:						
polymineralic	2-4	0-52	8	25 ± 3	2% ± 4%	7
non magnetic	4-8	-	-	-	6% ± 6%	12
magnetic	4-8	0-52	8	22 ± 7	20% ± 11%	12
SSP-1	2-8	0-52	8	327±55	6% ± 10%	14

1.The polymineralic, non magnetic, magnetic and HF treated samples are a) sample after grain size separation,
b) non magnetic fraction of the separated grains,
c) magnetic fraction of the separated grains and
d) grains remaining after 1 hr in 0.5N HF
respectively.

Table 3.1 -b

Equivalent Doses and Anomalous Fading of non-peat samples.

SAMPLE	GRAIN SIZE	SL BLEACHING		ED	ANOMALOUS	STORAGE
	(Micron)	Filter	Time (hr)	Gy	FADING	TIME (d)
FRSS-1	1-2	3-67	16	5 ±1	6 ± 2	44
FRSS-1	2-4	3-67	16	6 ±2	9 ± 4	44
FRSS-1	4-8	3-67	16	2 ±4	1 ± 8	44
FRSS-1	8-16	3-67	16	5 ±1	0.2 ± 2	44
FRSS-1	16-32	3-67	16	6 ±1	12 ± 5	44
MHGP-10	2-8	0-52	16	44 ±4	4 ± 3	11
MHGP-12	2-8	0-52	16	188 ±9	2 ± 3	60
MHGP-5	4-11	3-67	16	25 ±3	8 ± 15	15
MHGP-13	2-8	0-52	16	48 ±11	12 ± 8	11
KRGP-2	2-8	0-52	8	130 ±22	3 ± 7	90
KRGP-3	2-8	0-52	8	184 ±13	5 ± 3	17

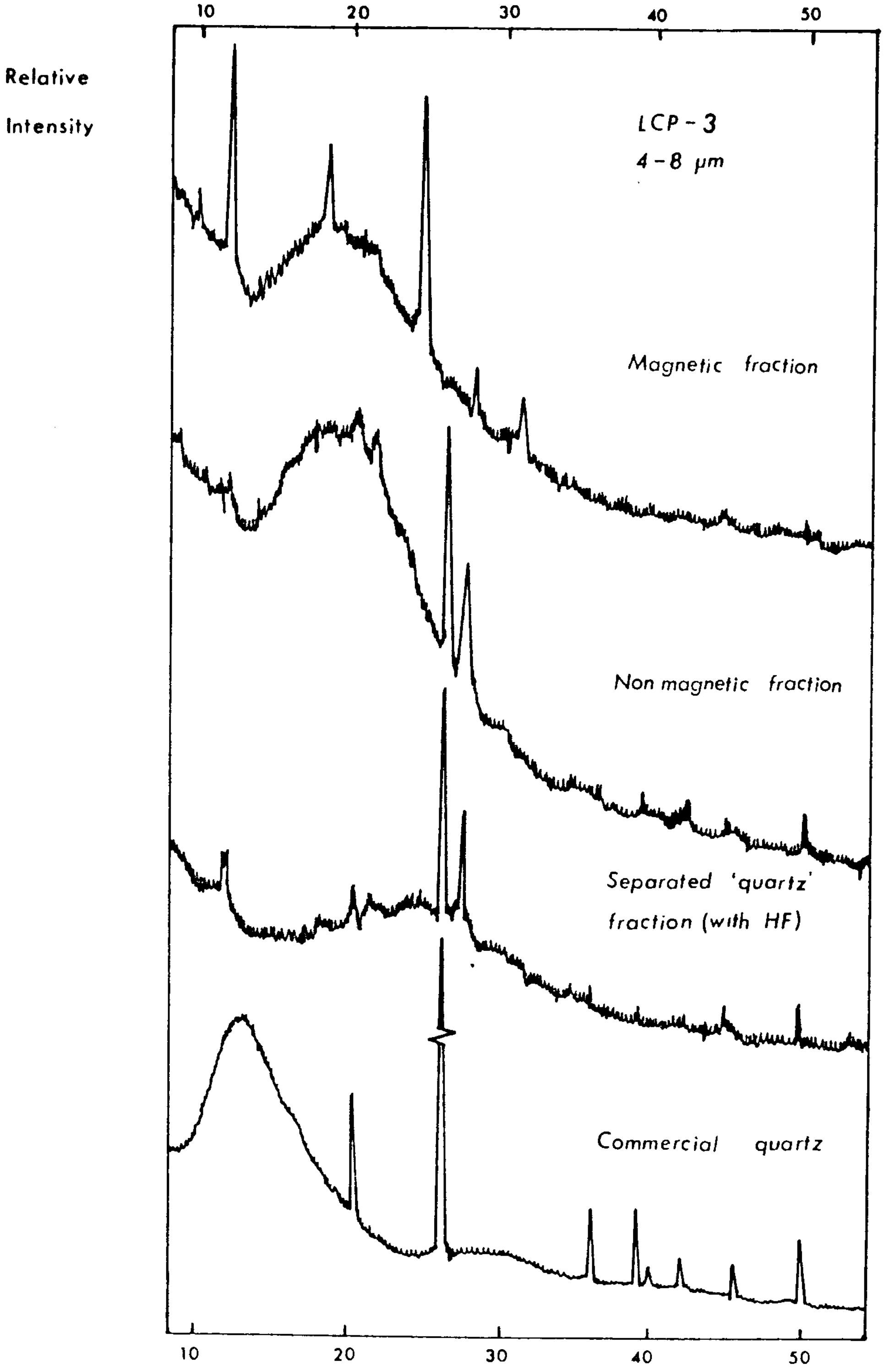

Fig.3.11-a XRD patterns of 4-8 μm grains of LCP-3. Magnetic, non magnetic fractions, and grains treated with 0.5N HF for 1 hr to separate quartz. Commercially obtained quartz' pattern is also shown as a reference.

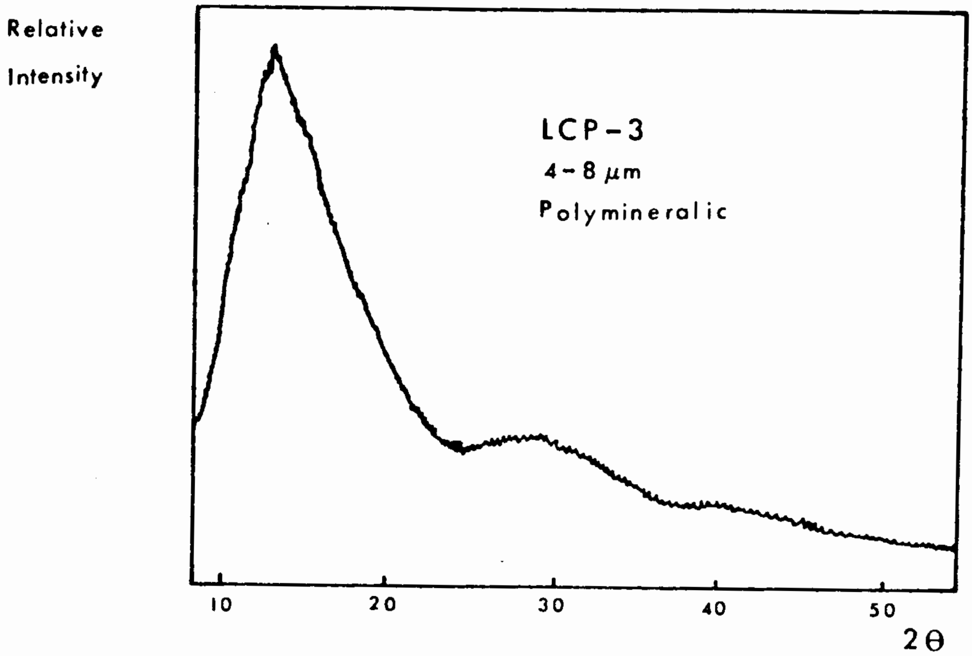

Fig.3.11-b XRD pattern of the untreated sample. (LCP-3, 4-8 μm)

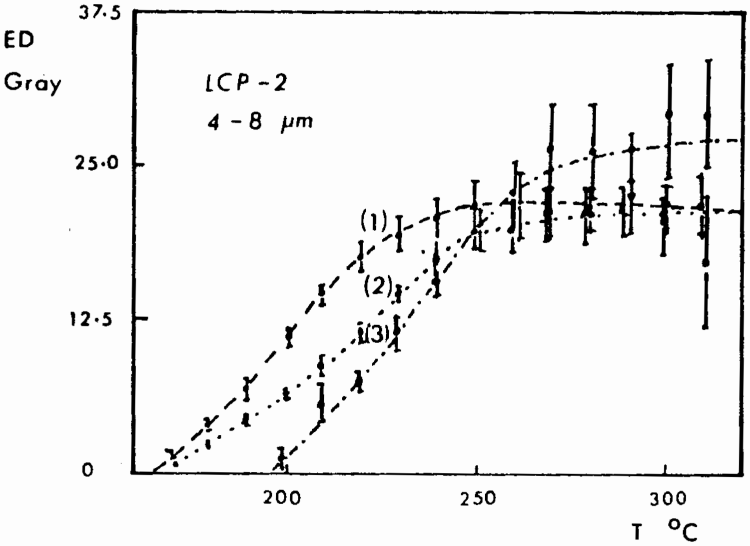

Fig.3.12 ED vs. glow curve temperature for,
(1) Grains treated with 0.5N HF .
(2) Untreated grains.
(3) Magnetic fraction.
8 hrs sunlamp exposure with 0-52 filter was used in all cases.

test does not guarantee that the sample did not fade during its geological history.

IV. EFFECTIVE DOSE RATE EVALUATION

4.1 Introduction

The dose rates suffered by the TL emitting minerals are primarily due to the decay of naturally occuring radioactive isotopes U-238, Th-232 and K-40 (0.012% in natural K) in the sediments. Fortunately, most of the TL emitting minerals themselves contain negligible amounts of these radioactive elements so that their self-dose is not important (Fleming, 1979).

Evaluating the effective dose rate to the mineral grains involves two main steps.viz,

1.Determining the U, Th and K contents of the sample and its surroundings. The first two were measured by alpha scintillation counting while the K content was obtained commercially [3] in the form of $K_2O\%$.

2.Calculating the effective dose rate using the standard dose rate conversion factors (Wintle and Huntley, 1980) for count rates of each decay series allowing for the fact that the alpha particles are less efficient in producing TL than beta or gamma radiation. Additionally, the dose rates were corrected for the presence of water and organic matter in the samples.

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[3] Chemex Labs Ltd,
212, Brooksbank Avenue,
North Vancouver. B.C.
Canada V7J 2C1.

4.2 Alpha Scintillation Counting

The powdered dry sample was placed on a ZnS disc in a lucite container which was then placed on a photomultiplier tube (PMT). Fig.4.1 shows a schematic of the counting equipment described in detail in Huntley and Wintle (1981). The scintillations of ZnS caused by alpha particles from the sample were counted using the PMT. The proper discriminator setting excluded those pulses due to beta particles. Conventionally 82% of the pulses induced by alpha particles from the U-238 decay chain are let in through the discriminator and counted.

The total alpha counts and the counts of alpha particles emitted within 0.4 s of each other (pair counts) were recorded. In addition to the random pairs, pair counts are produced by the decay sequence Rn-220-->Po-216-->Bi-212 which occur in Th-232 decay chain; Po-216 has a half life of 0.145 s. After correcting for the random pair counts, this gave a measure of Th-232 in the sample. A knowledge of decay schemes and the true pair counts enables one to calculate the count rate due to Th-232 series and that due to U series (U-238+U-235) separately, under the assumption that the radioactive decay series are in equilibrium (Aitken, 1978-b).

If alpha count rates from U and Th series are c_U and c_{Th} respectively, and W_K is the weight percent of K_2O in dry sediments, the uncorrected total dose rate D in a dry sample would be given by

$$D = D_\alpha + D_\beta + D_\gamma + D_c$$

where D_α, D_β and D_γ are the 'raw' dose rates from alpha, beta and gamma radiation given by,

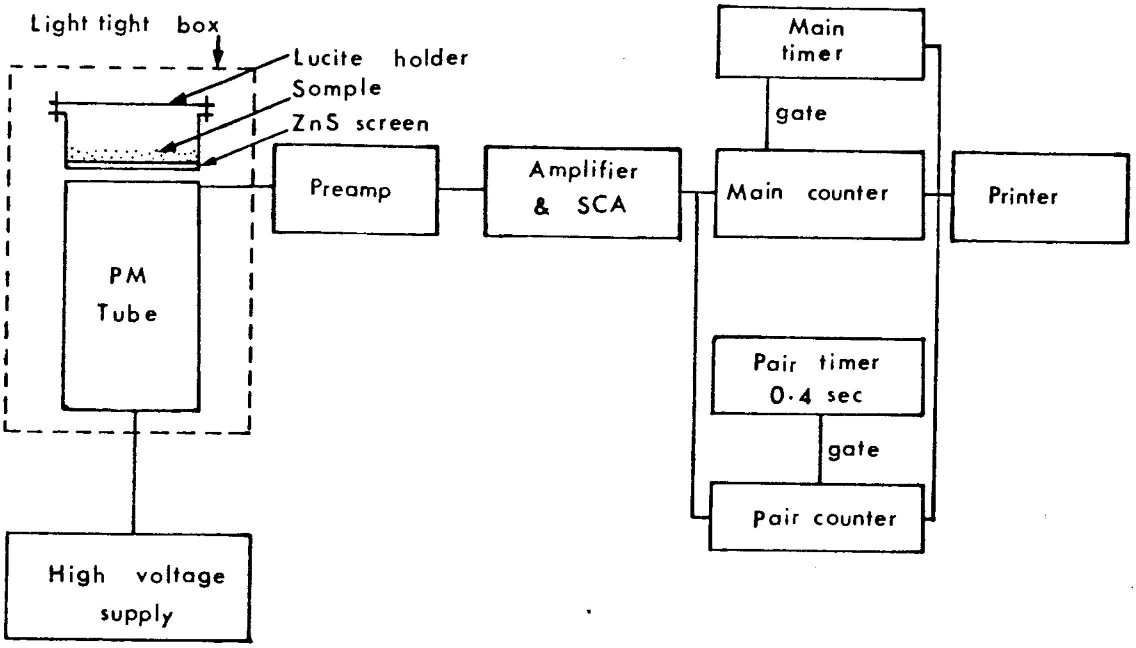


Fig.4.1 Schematic of alpha scintillation counting apparatus.

$$D_{\alpha} = c_{U}\, d_{\alpha U} + c_{Th}\, d_{\alpha Th}$$

$$D_{\beta} = c_{U}\, d_{\beta U} + c_{Th}\, d_{\beta Th} + w_{K}\, d_{\beta K} \qquad (4.1)$$

$$D_{\gamma} = c_{U}\, d_{\gamma U} + c_{Th}\, d_{\gamma Th} + w_{K}\, d_{\gamma K}$$

where d_{α}'s, d_{β}'s, d_{γ}'s are the conversion factors for each type of radiation in corresponding series (Table 4.1) for the standard rock minerals; D_{c} is the cosmic dose rate.

In order to accomodate for the difference in the efficiencies of alpha particles and beta (or gamma) radiation in producing TL a measured quantity called the 'a'-value (Aitken & Bowman, 1975) is conventionally used for expressing the alpha dose rate in terms of an *equivalent beta (or gamma)* dose rate.

4.3 The 'a'-value

Expressing effective alpha dose rate in terms of the equivalent beta (or gamma) dose rate was done first with the aid of the 'k'-value which was defined as the ratio of TL per rad of alpha radiation to that per rad of beta radiation. But this parameter is dependent upon the energy of the alpha particles. Hence an alternate procedure called 'a'-value system has been adopted where the 'a'-value for a sample relates the TL per unit track length of alpha particle (which does not depend on the energy of the alpha particles of interest here (Bowman, 1976)) to the TL per gray of beta particles.

Table 4.1

Dose rates (Gy/ka) for the decay of
K-40, U-238 and Th - 232

Dose rate for K-40 per 1% K_2O.

K-40	$d_{\beta K} = 0.682$	$d_{\gamma K} = 0.205$

Dose rates from an alpha count rate of 1.00 $cm^{-2}ks^{-1}$.

	d_α	d_β	d_γ
U-238 {U-235}	$d_{\alpha U} = 23.30$	$d_{\beta U} = 1.226$	$d_{\gamma U} = 0.983$
Th-232	$d_{\alpha Th} = 20.93$	$d_{\beta Th} = 0.819$	$d_{\gamma Th} = 1.414$

An alpha radiation source (Am-241) of known strength which is expressed in terms of the 'rate at which track length is delivered to a unit volume of the sample ($cm^{-2} sec^{-1}$ or $micron^{-2} min^{-1}$)' is used to measure the 'a'-value of the samples. The 'a'-value is defined by

$$a = \frac{x}{13\ S} \qquad (4.2)$$

where x is the beta (or gamma) radiation dose in grays which produces an equivalent TL response in the sample as one minute of irradiation by the alpha source of strength S $micron^{-2} min^{-1}$ (Aitken and Bowman, 1975). Then the effective alpha dose rates $d'_{\alpha U}$ (from U series) and $d'_{\alpha Th}$ (from Th series) in beta(or gamma) equivalent dose rates are given by

$$d'_{\alpha U} = 18.0\ a\ c_U \qquad (4.3)$$

$$d'_{\alpha Th} = 17.4\ a\ c_{Th} \qquad (4.4)$$

where c_U and c_{Th} are alpha count rates from the U and Th decay series, respectively (Wintle & Huntley, 1980). Hence the effective dose rates D'_α, D'_β, D'_γ due to alpha, beta, gamma radiations are given by

$$D'_\alpha = 18.0\ a\ c_U + 17.4\ a\ c_{Th} \qquad (4.5)$$

$$D'_\beta = 0.682\ w_K + 1.226\ c_U + 0.819\ c_{Th} \qquad (4.6)$$

$$D'_{\gamma} = 0.205\ w_{K} + 0.983\ c_{U} + 1.413\ c_{Th} \quad (4.7)$$

and the total effective dose rate D′ is the sum of D'_{α} , D'_{β} ,D'_{γ} and D_{C}.

The x in equation 4.2 was routinely determined by repeating the R-Γ procedure for the sample by replacing gamma irradiation with alpha irradiation; this is called the R-α method. The ED′s obtined from the two methods (R-Γ and R-α)were used to calculate x. The use of R-Γ and R-α methods are thought to give the correct ′a′-value which should be relevant to the ′bleachable′ (light sensitive) traps of the minerals in the sample.

4.4 Dose Rate Correction Due to Water and Organic Matter

Most sediments are permeated with water and additionally materials like peat contain a significant amount of organic matter. Hence the dose rate calculated for the standard rock minerals should be corrected for the presence of the above materials.

For samples with neglegible organic contents the correction for attenuation of radiation was done by

$$D'_{\alpha\ \text{corrected}} = \frac{D'_{\alpha}}{1 + H_{\alpha}\Delta} \quad (4.8)$$

$$D'_{\beta\ \text{corrected}} = \frac{D'_{\beta}}{1 + H_{\beta}\Delta} \quad (4.9)$$

$$D'_{\gamma\,\text{corrected}} = \frac{D'_\gamma}{1 + H_\gamma \Delta} \qquad (4.10)$$

where,

Δ is the mass of water in sample expressed as a fraction of the mass of dry sediment,

H_α and H_β are the ratios of specific stopping powers of water to dry sediment for alpha and beta particles respectively, and H_γ is the ratio of absorption coefficient of water to dry sediment for gamma radiation (Zimmerman, 1971).

The values $H_\alpha = 1.49$, $H_\beta = 1.25$ and $H_\gamma = 1.00$ were used from Bowman (1976).

The correction for the dose rate in materials with a significant amount of organic content was effected by (see Appendix. I)

$$D'_{\alpha\,\text{corrected}} = \frac{D''_\alpha}{1 + H_\alpha \Delta^w + H^o_\alpha \Delta^o} \qquad (4.11)$$

$$D'_{\beta\ \text{corrected}} = \frac{D''_{\beta}}{1 + H_{\beta}\Delta^{W} + H^{o}_{\beta}\Delta^{o}} \qquad (4.12)$$

$$D'_{\gamma\ \text{corrected}} = \frac{D''_{\gamma}}{1 + H_{\gamma}\Delta^{W} + H^{o}_{\gamma}\Delta^{o}} \qquad (4.13)$$

where D"'s are the dose rates calculated by equations (4.5), (4.6) and (4.7) using the alpha count rates and K contents that had been corrected for the presence of organic matter in the dry sediment (i.e, these are the count rates one would observe if the organic matter was removed).

Δ^{W} and Δ^{o} are the masses of water and organic matter in sample expressed as fractions of the mass of minerals respectively,

H^{o}_{α} and H^{o}_{β} are the ratios of specific stopping powers of organic matter to minerals for alpha and beta radiation respectively, and H^{o}_{γ} is the ratio of absorption coefficients of organic matter to minerals. The H_{α}, H_{β} and H_{γ} are defined as above. The calculated values of H^{o}'s are shown in Table 4.2. Throughout all calculations of the ratios of stopping powers and absorption coefficients (Evans, 1955) the mean composition of organic matter was taken as C: 59%, H: 6%, O: 33% and N: 2% (Rankama & Sahama, 1950).

The Bragg-Kleeman rule states that the range of alpha particles in a material is proportional to its $A^{1/2}_{eff}$ value (Evans, 1955). For an element A_{eff} is its atomic weight while for a compound or a mixture of compounds it is given by

Table 4.2

The calculated H_{α}^{o}, H_{β}^{o} and H_{γ}^{o} values.

a) Ratio of specific stopping powers of α and β particles in organic matter to minerals in peat samples. (H_{α}^{o} and H_{β}^{o}).

b) Ratio of absorption coefficients of γ rays in organic matter to minerals in peat samples (H_{γ}^{o}).

ENERGY (MeV)	H_{α}^{o}	REMARKS
0.5	1.462	Reference;
1.0	1.347	Northcliffe & Schilling
2.0	1.342	(1970).
4.0	1.398	Averaged over typical α
8.0	1.322	spectrum of U,Th in soil.

$H_{\alpha}^{o} = 1.37 \pm 0.06$

ENERGY (MeV)	H_{β}^{o}	REMARKS
0.1	1.241	Reference;
0.5	1.205	Berger and Seltzer (1964).
1.0	1.191	β spectrum unknown; hence
2.0	1.179	the average value for H_o .

$H_{\beta}^{o} = 1.20 \pm 0.03$

ENERGY (MeV)	H_{γ}^{o}	REMARKS
0.05	0.511	Reference;
0.01	0.904	Evans (1968).
0.20	1.028	Assuming 30% of γ are
0.50	1.054	< 2 MeV .
1.00	1.056	Bowman (1976).
2.00	1.051	

$H_{\gamma}^{o} = 0.98 \pm 0.08$

Table 4.3

A_{eff} values and the correction factors for alpha count rates of peat samples.

SAMPLE	A_{eff}	CORRECTION FACTOR c'/c
SSP-1	13.60	2.13
QCIP-1	14.80	1.75
MHGP-5	16.99	1.45
LCP-3	17.87	1.39

$$A_{eff}^{1/2} = \frac{\sum_{i=1}^{r} n_i \, A_i}{\sum_{i=1}^{r} n_i \, A_i^{1/2}} \tag{4.14}$$

where $n_1 \ldots n_r$ are the atomic fractions of the elements whose atomic weights are $A_1 \ldots . A_r$. The A_{eff} value for most of the minerals lies in the range 19 - 22. Hence in calculating H_{α}^{o} , the stopping power for Ne (A = 20) was used in place of the value for minerals (A = 21.4). Moreover, since beta energy absorption depends on the density of material (Evans, 1955), the stopping power of Al (density 2.60 g cm^{-3}) was used (Berger & Seltzer, 1966) for that of the minerals (average density 2.65 g cm^{-3}) while for H_{γ}^{o} the data for concrete was used since the gamma absorption characteristics of soil and concrete are very similar (Murray, 1981).

The discriminator level of the alpha counter was conventionally set to let in 82% (85%) of alpha induced pulses for a dry sample of A_{eff} value 21.4 containing uranium (thorium) in secular equilibrium. However, the presence of organic matter alters both the range of alpha particles and the concentrations of U and Th in the sample. Hence the alpha count rates (both from U and Th decay series) $c_{U,Th}$ were corrected using the Bragg-Kleeman rule and the mass ratio of the dry sample and the minerals in the sample by,

$$c'_{U,Th} = \frac{0.83 \quad c_{U,Th}}{\sqrt{\frac{A_{eff}}{21.4}} - 0.17} \cdot \frac{M_m + M_o}{M_m} \tag{4.15}$$

where A_{eff} is the calculated effective atomic weight for the sample (Table 4.3); M_m and M_o are the masses of minerals and organic matter in the sample respectively (private communication: Dr.D.J.Huntley). The mass ratio corrects the count rate for the change in the concentration of U and Th while the rest in the equation (4.15) takes care of the effect of the change in the alpha particle range and the discriminator level. The weight per cent of K_2O in the dry sediment w_K was also corrected for the presence of organic matter by using the above mass ratio. For LCP-2 the A_{eff} value of LCP-3 was used; MHGP-2, MHGP-4 and MHGP-9 had negligible organic contents (<2%) hence no correction were made for their count rates. These corrected alpha count rates and K_2C weight per cents were used in equations (4.5), (4.6) and (4.7) to obtain $\overset{w}{D}{}'$s in equations (4.11), (4.12) and (4.13).

Water contents of the non-peat samples were determined by measuring the reduction of water in them by heating in an oven at 40 C soon after collecting them. In most samples the saturation water contents were also determined by letting the samples soak water to saturation and subsequently drying them in an oven. Organic content ($\overset{o}{\Delta}$) of a peat sample was determined by heating it in an oven at 500 C for several hours until the sample was completely ashed. The ratio of the reduction in mass to the mass of ash left was taken as $\overset{o}{\Delta}$. The mass of ashed material was taken as the mass of minerals in calculating $\overset{w}{\Delta}$'s and $\overset{o}{\Delta}$'s for peat samples (Table 4.4 and Table 4.5-a,-b).

The average ranges of alpha, beta and gamma radiation from natural radioactivity in soil is about 25 um, 2 mm and 45 cm, respectively. Hence in calculating D'_α and D'_β the alpha count rates and K_2O% of the sample were used while both of the sample and the surrounding were taken in calculating the gamma contribution. The corrections of alpha and beta dose rates due to water and organic contents were carried out using the Δ value of the sample. For the correction of the gamma dose rate a Δ value judged from the geometry of the sample in relation to the surrounding sediment layers was

Table 4.4

Measured water contents (Δ, Δ^w) and organic contents (Δ^o).

SAMPLE	IN-SITU WATER CONTENT Δ (Δ^w)	SATURATION WATER CONTENT Δ (Δ^w)	ORGANIC CONTENT Δ^o
Peat:			
QCIP-1	(2.14)	(2.74)	(0.40)
MHGP-2	0.47	0.64	(<0.02)
MHGP-4	0.35	0.53	(<0.02)
MHGP-9	0.62	1.03	(<0.02)
LCP-2	(0.91)	(1.09)	(0.19)
LCP-3	(0.81)	(0.81)	(0.24)
SSP-1	(2.20)	(2.27)	(0.58)
Silt and other materials:			
MHGP-10	0.23	0.53	
MHGP-12	0.18	0.20	
MHGP-13	0.19	0.58	
KRGP-2	0.19	0.43	
MHGP-5	0.55	1.05	(0.26)

1. The values in brackets are with respect to mass of minerals in the samples while others are with respect to mass of the dry sample.

Δ - mass of water / mass of dry sample.
Δ^w - mass of water / mass of minerals.
Δ^o - mass of organics / mass of minerals.

Table 4.5 - a

Dose rates to peat materials.

SAMPLE	COUNT RATE cm^{-2} ks^{-1}			K_2O %	'a' value	DOSE RATE Gy/ka
	TOTAL	U-238 + U-235	Th-232			
QCIP-1	0.195 ± 0.010	0.158 ± 0.020	0.037 ± 0.017	0.84	0.15 ± 0.03	0.79
MHGP-2	0.434 ± 0.011	0.291 ± 0.021	0.143 ± 0.025	1.34	(0.06)	1.79
	0.228 ± 0.012	0.192 ± 0.024	0.036 ± 0.020	1.53		
MHGP-4	0.383 ± 0.007	0.195 ± 0.020	0.187 ± 0.019	1.59	0.06	1.82
	(0.228)	(0.192)	(0.036)	(1.53)		
MHGP-9	0.295 ± 0.013	0.193 ± 0.033	0.101 ± 0.030	0.77	(0.06)	0.97
	0.278 ± 0.014	0.213 ± 0.032	0.065 ± 0.029	1.00		
LCP-2	0.322 ± 0.009	0.211 ± 0.027	0.112 ± 0.025	1.06	0.064 ± 0.004	1.13
	0.353 ± 0.009	0.192 ± 0.028	0.161 ± 0.027			
LCP-3	0.241 ± 0.006	0.189 ± 0.014	0.052 ± 0.012	0.79	(0.06)	0.97
SSP-1	0.114 ± 0.005	0.061 ± 0.016	0.054 ± 0.015	0.34	0.13 ± 0.02	0.69
	0.477 ± 0.023	0.312 ± 0.003	0.165 ± 0.059	1.38		

See the notes on table 4.5 - b.

Table 4.5 - b

Dose rates (D) to non - peat materials.

SAMPLE	α COUNT RATE cm^{-2} ks^{-1} TOTAL	U-238 + U-235	Th-232	K_2O	'a'- VALUE	D Gy/ka
MHGP-10	0.332 ± 0.014	0.243 ± 0.034	0.090 ± 0.031	1.24	0.073 ± 0.010	1.50
	0.316 ± 0.013	0.179 ± 0.034	0.136 ± 0.032			
MHGP-12	0.724 ± 0.027	0.579 ± 0.066	0.145 ± 0.060	1.24	0.066 ± 0.009	2.60
	0.265 ± 0.011	0.196 ± 0.018	0.069 ± 0.024			
MHGP-5	0.321 ± 0.012	0.261 ± 0.027	0.061 ± 0.025	0.92	0.064 ± 0.002	0.84
	0.283 ± 0.011	0.188 ± 0.024	0.095 ± 0.021	1.08		
MHGP-13	0.304 ± 0.013	0.196 ± 0.031	0.109 ± 0.028	1.24	0.081 ± 0.016	1.51
	0.291 ± 0.012	0.210 ± 0.020	0.081 ± 0.025	1.08		
KRGP-2	0.349 ± 0.014	0.280 ± 0.030	0.069 ± 0.027	1.96	0.028 ± 0.008	2.04
KRGP-3	0.281 ± 0.011	0.122 ± 0.024	0.159 ± 0.026	1.43	0.041 ± 0.007	1.58

1. The alpha count rates of the surroungings are given below the sample data.
2. SSP-1:Only the underlying silt was counted.
3. For SSP-1 & QCIP-1 the saturation water content values were used since the samples are presently saturated and were assumed to have been so at all times.

used.

The water contents of the samples and their surroundings must have fluctuated in the past due to both the drastic changes like glaciation as well as mild variations like seasonal effects. For the lack of a better value for the actual water content of the sample the average value of the in-situ and saturation water contents was used for the preglacial samples while for the postglacial ones the in-situ values were used in the dose rate calculations. Estimation of the cosmic dose rate D_c which is generally a very small quantity compared to D'_α , D'_β and D'_γ was based on the figures given by Aitken (1974) and Prescott and Stephan (1982). The cosmic dose rate at 1 m depth at latitudes greater than 40 N is quoted as 0.15 Gy/ka while that on the ground is about 0.30 Gy/ka. Considering the geological factors such as sedimentation, glaciation and deglaciation the D_c for each sample was estimated. An uncertainty of about 40% was also estimated considering extreme behavior of the above factors. Thus the estimated values for most preglacial samples was as low as 0.05 Gy/ka since they have been under a thick ice sheet during the last glaciation (about 11-22 ka ago for most of the samples, Armstrong & Hicock, 1975). The calculated effective dose rates are given in Table 4.5-a and 4.5-b. A detailed effective dose calculation for one of the samples studied is given in Appendix III.

4.5 In-situ Gamma Dosimetry

The largest uncertainty in dose rates calculated by alpha counting lies in evaluating the gamma contribution. This is because of the possible escape of some decay products in the series which were assumed to be in equilibrium. For instance, at 75% of Rn escape (a gaseous product of U-238 series), the gamma dose rate would be underestimated by 50% (Aitken, 1978-b).

As a check to see how well the gamma dose rate was accounted for by alpha counting, an alternate method of measuring it by phosphor dosimeters at some sample sites were carried out.

CaF_2:Dy thermoluminescence dosimeters (TLD's, 6.35mm 6.35mm 0.889mm) were encapsulated in tubes of 0.0254 mm thick tantalum and 0.0508 mm thick lead, held together in a vinyl holder (Fig.4.2, Technical details are in Denham et al, 1972). Dosimeters were placed in the capsule on a slitted piece of foam plastic. The capsule excludes alpha and beta particles from the phosphor and allows only gamma radiation while providing the phosphor with a flat (within 30%) energy response in 0.05 - 1.25 MeV range. A capsule containing two TLD's was placed in a thin (0.70 mm) stainless steel tube (50 cm long) and the tube was buried at a sample site with a piece of Cu attached to the top so that it could be easily found by a metal detector at the end of the burial period.

CaF_2 :Dy TLDs were buried at sites of LCP-3, MHGP-10 and MHGP-12. The TL of recovered TLD's after several months of burial were measured and the TL level of the most prominent peak (at 140° C) was matched against a subsequently built growth curve in the laboratory using the Ra-226 gamma source. This yielded the total equivalent gamma and the cosmic dose that the phosphor has received during its burial at the sample site. The dosimetric results are given in Table 4.6. Dosimeters yielded values with 13%-24% uncertainty whereas alpha counting gave gamma dose rates with about 10% uncertainty to which the estimated cosmic ray dose rate was added. The cosmic ray dose rates D_c , were estimated for each site using the burial depth dependance of D_c given by Prescott and Stephan (1982). The value at sea level was used since the altitude effect is neglegible for the samples studied.

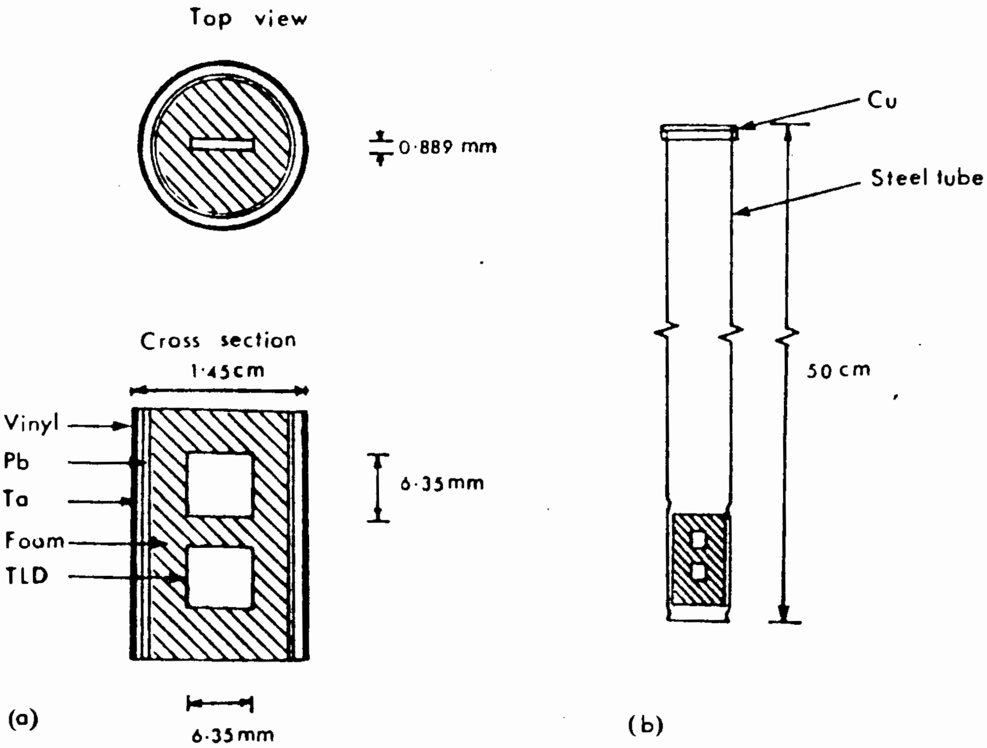


Fig.4.2 CaF$_2$:Dy thermoluminescence phosphor dosimeters (TLD's).

a)TLD capsule with the dosimeter.

b)Steel tube with TLD capsule.

Table 4.6

Comparison between dosimetric results and the dose rates from alpha counting and K analysis.

SAMPLE SITE & BURIAL PERIOD	TLD DOSE RATE Gy/ka	α- COUNT DOSE RATE D_γ Gy/ka	CALCULATED PRESENT COSMIC DOSE RATE D_c Gy/ka	$D_\gamma + D_c$ Gy/ka
LCP-3 (6 months)	0.37 ±0.05	0.30 ±0.03	0.12 ±0.01	0.42 ±0.03
MHGP-10 (82 days)	0.51 ±0.13	0.61 ±0.06	0.18 ±0.01	0.79 ±0.06
MHGP-12 (82 days)	0.52 ±0.07	0.51 ±0.05	0.15 ±0.01	0.66 ±0.05

Table 4.7

Alpha count rates of fresh and dripped samples to detect mobility of radioactive materials.

	TOTAL COUNT RATE cm^{-2} ks^{-1}	
SAMPLE	FRESH SAMPLE	DRIPPED SAMPLE
LCP-3	0.332 ± 0.010	0.309 ± 0.011
MHGP-10	0.332 ± 0.014	0.322 ± 0.007

The estimated attenuation of gamma radiation by the steel tube and the capsule is about 4% in 0.5 - 1 MeV range. The results of the LCP-3 and MHGP-12 are within the uncertainty of the dose rates from alpha counting and K analysis. However, a discrepancy is seen in the case of MHGP-10. A situation like having a somewhat large stone buried nearby that particular sample, blocking away radaiation from the surroundings, could possibly explain this slight discrepancy.

It has been shown for some time that there could be a significant enrichment of uranium in sediments with high organic contents due to mobile uranyl salts that get transported by ground water. Hence an additional experiment was done to see if the radioactive materials in the samples are mobile due to the action of ground water. This experiment was like that of Wintle (1981). Distilled water was continuously dripped through two samples for a period of one week at a rate of about 7 litres per day. Subsequently, alpha counting was carried out on both the fresh and the dripped samples results of which are shown in Table 4.7. LCP-3 sample showed a 7% decrease in total count rate while MHGP-10 was unaltered (within 3%). This results are suggestive of the possibility of the dose rate being variable in the past but are not conclusive.

V. APPARENT TL AGE CALCULATION

5.1 Apparent TL Ages

The age equation discused in section 1.3 was used to assign tentative TL ages to the sediments studied in the test program. These ages may correspond to the times since their deposiion _if_ the initial assumtion about the TL clock being zeroed at the last exposure to sunlight is correct. The calculated 'apparent' ages are given in Table 5.1. In situations where the samples showed detectable anomalous fading the ages given are to be considered only minimum values, hence the > sign.

5.2 Uncertainties

The variability of the water contents of the samples is the major source of uncertainty in the calculated final ages (see Appendix III). The experimental error of ED's are also quite significant in some samples (see Table 5.1). The uncertainty in the measured 'a'-value is about 15%-20%. The alpha count rates had an error of about 10% which corresponds to an uncertainty of about 6% in the calculated final age. In SSP-1, the correction for organic content reduced the dose rate by 8%. The error in the 'a'-value (15%) corresponds to only 2% change in the dose rate. In samples like SSP-1 where one has to estimate a Δ value for gamma radiation from the values of the sample and the surrounding according to the geometry involved, the error in the dose rate is about 10%. The overall uncertainty in the ages are typically about 20%.

Table 5.1

Apparent TL ages.

SAMPLE	ED Gy	DOSE RATE Gy/ka ± 10%	APPARENT TL AGE ka	C-14 AGE ka
Peat:				
BBP-3	3 ± 2	-	-	< 0.5[1]
QCIP-1	6 ± 2	0.79	7 ±3	⩾9.16 ± 0.09[2]
MHGP-2	33± 4	1.79	>18±3	18.60± 0.19
MHGP-4	45± 10	1.82	>25±6	18.7 ± 0.17
MHGP-9	35 ±6	0.97	>36 ±7	28.20 ± 0.20[2]
LCP-2[3]	23 ±2	1.13	>20 ± 2	⩾33.00 ± 0.62[2]
LCP-3	25 ±3	0.97	25 ±4	>33.00 ± 0.62[2]
SSP-1[4]	327± 55	0.69	>480 ± 90	$>71.50\ ^{+1.70}_{-1.40}$[2]
Silt:				
MHGP-10	44 ±4	1.50	29±4	>27.4,< 28.2[2]
MHGP-12	188 ±9	2.60	72±7	>28.2,< 40.2[2]
MHGP-5	25 ±3	0.84	>30±4	40.2 ± 0.10[2]
MHGP-13	48 ±11	1.51	>32±8	>40.2 ± 0.10[2]
KRGP-2	130± 22	2.04	64±12	$\geqslant 58.80\ ^{+2.90}_{-2.10}$[2]
KRGP-3	184± 19	1.58	116±18	$>58.80\ ^{+2.90}_{-2.10}$

---

1. Age estimated from the sedimentation rate (Hebda, 1977).
2. ⩾,> and < signs indicate that C-14 ages are on materials immediately overlying, overlying and underlying the sample respectively.
3. ED for the magnetic fraction is used: unseparated sample showed anomalous fading and a lower ED.
4. Fission track age of the ash layer 1m below peat is 870 ±210 ka (Easterbrook et al, 1981).

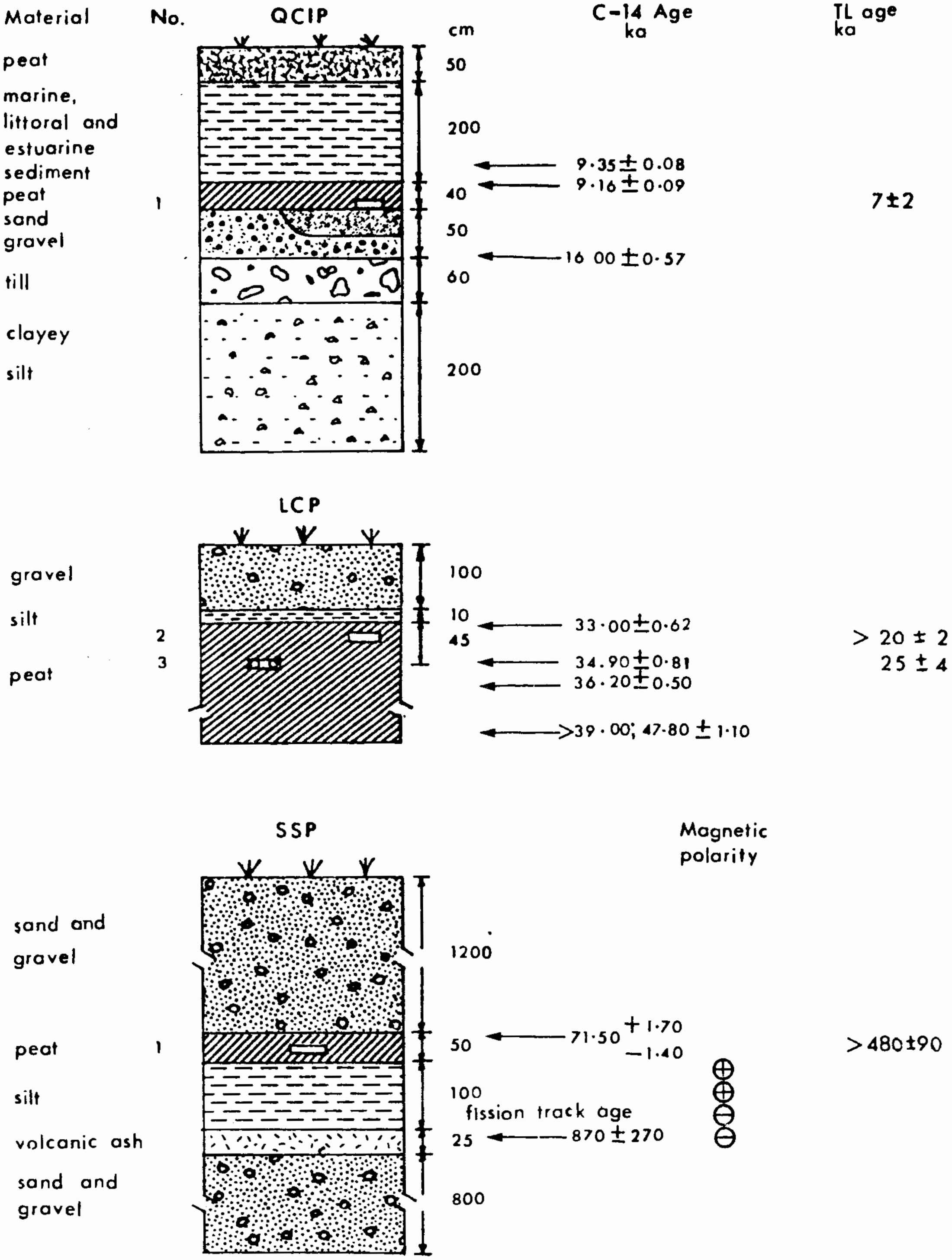
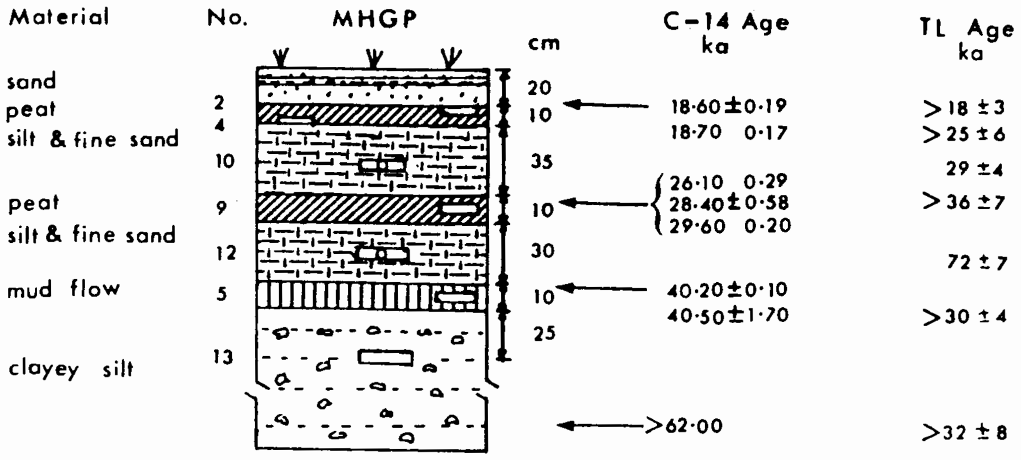
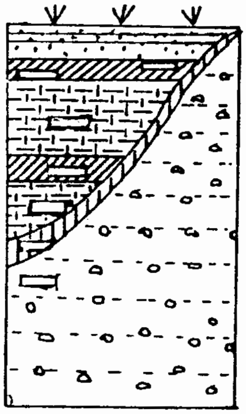


Fig.5.1-a Site sections.
QCIP, LCP and SSP (details in Appendix. I).



cross section



profile

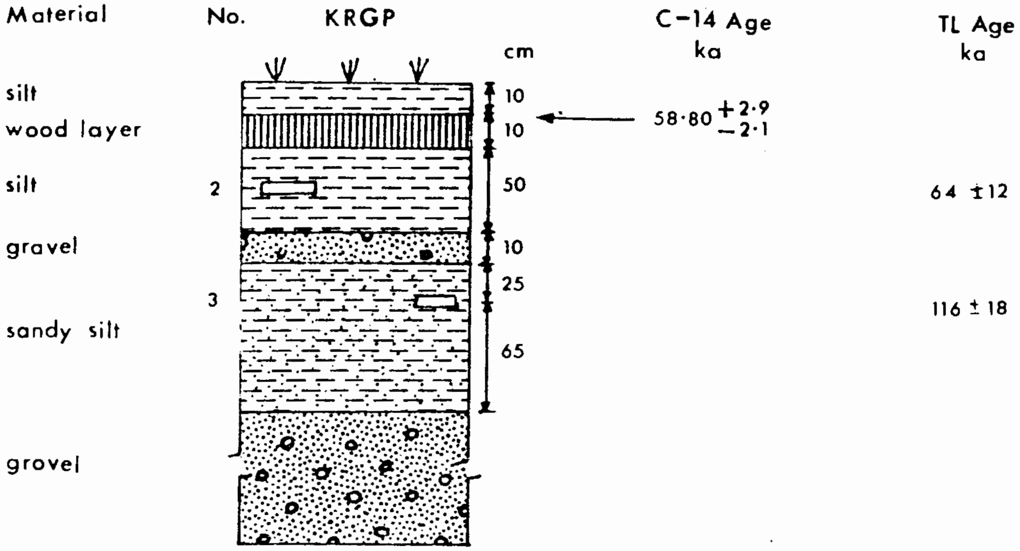


Fig.5.1-b Site sections.
MHGP and KRGP (details in Appendix.I).

5.3 Discussion

The apparent ages calculated for most of the samples from A-horizons (i.e., soils with maximum biological activity; those containing organic matter) are in agreement with known ages; this shows great promise in TL dating. None of them yield too old an age indicating that zeroing has taken place. BBP-3, a modern peat sample yielded a low ED (< 3 +/- 2Gy). However, its alpha activity was so low that the count rate was comparable to that of the background. Even though we have not counted materials underlying BBP-3, this rare, low count rate strongly indicates the possibility of the dose rate being variable throughout the geological history of materials such as peat. It would be instructive to obtain samples at different depths from the same peat layer to check if the dose rate varies. Phenomenon such as uranium uptake by the organic materials have been reported in the literature (Manskaya & Drozdova, 1968; Halbach et al, 1980). Such processes would cause the dose rate to be increased with the progress of time. The cause for LCP-2 giving a low age may be such an effect. Anyhow, the ED value for BBP-3 indicates a certain limitation of using the R-Γ method as it is being used for modern samples. The apparent age of BBP-3 may be roughly estimated as >3 ka.

The anomalous fading observed in almost all the samples were quite small. A mineral fraction whose TL shows anomalous fading, but present in the samples only in small (<10%) quantities may explain the results obtained. However, if this was the case, i.e, if the majority of the TL emitting minerals in samples did not fade, the apparent TL ages may not be much effected by fading.

A reason for having too young ages for some peat samples, in addition to anomalous fading, would be the possibility of their water to mineral ratios being much higher during the first few ka which subsequently decreased dramatically as the material was compressed during glaciation when they were buried under an ice sheet. Moreover, the effects of geochemical actions on TL in sediments are totally unknown yet.

The oldest peat material studied in the test program was SSP-1. This sample has a C-14 age which was recently brought into question by a fission track age about 10 times older (Easterbrook et al, 1981). The apparent TL age supports the older age. For a resolution of this discrepancy it is suggested that the C-14 age which was obtained in the extreme limit of the technique could be in error due to sample contamination. A recent (last century) material which has grown in equilibrium with atmospheric C-14 levels has a disintegration rate of about 13.6 per minute per gram of carbon (Stuiver,1978) while a sample of age 72 ka has only 0.0018 per minute per gram of carbon. Therefore a very old material (with beta activity well beyond the detection limit of the counting apparatus) that had been contaminated 0.13 per mil with modern material will produce the same count rate as the 72 ka old sample.

The young river silt, FRSS-1, gave an ED of <5Gy. Among the ages obtained for the older silt materials, KRGP-2 and KRGP-3 gave satifactory values. The samples from MHGP site showed too young ages. Again, this could be attributed to anomalous fading or variable dose rates. The result of MHGP-12 is quite anomalous; the material may not have been zeroed during deposition. A good knowledge of the mode of deposition, origin of the material and its geological history would give some insight in most of the cases. Adverse effects such as radon escaping could have occured in these sites whereas in the samples collected from peat layers, this effect would have been minimal because of their high water contents.

A preliminary test on LCP-3 showed that the change in ED due to TL sensitvity change caused by bleaching was insignificant. However, for

obtaining accurate ages a correction for this may be required.
In comparing the apparent TL age with the C-14 age, one should not forget that the latter may depart from the true age by a maximum of 2ka in the 9-32 ka BP range (Stuiver, 1978); the errors beyond that period are yet to be determined. Anyhow, it is unlikely that there would be any major deviation.

5.4 Conclusions

Considering the criteria set forth by Wintle and Huntley (1982: see section 1.4), the peat materials appear to be better candidates for TL dating by the R-Γ method than the non-peat deposits studied in the test program. It should be stressed that more detailed studies should be done to clarify the primary problem of zeroing before making R-Γ a standard technique in dating any kind of geological materials. The secondary, but almost equally important issues such as anomalous fading, dose rate variabilty etc, also limit any TL dating technique. However, at present the technique seems to yield much promise in obtaining reliable dates in the future.

5.5. Suggestions for Future Work

1. A better control over the ages of the modern peat materials would be highly desirable. To see the effect of the 'residual' TL signal on the ages of the young samples unambiguously, a more efficient method of removing organic matter from young peat samples is needed. A careful study of the peroxide treatment (section 3.2) should be carried out since this was not investigated to a great extent. One variable, among many that was not studied in this work is the bleaching spectrum. An

improvement in the situation of the young samples could be expected by investigating the effect of this variable carefully (peat samples with ages 0-9ka could be used here).

2. The fact that the $R-\Gamma$ method seem to work for peat and some associated silt materials suggests that either one or a group of minerals behaves well according to the model proposed by Wintle and Huntley (1979). Therefore identifying and possibly separating this mineral component will be of much use in both selecting and dating potential materials. In this context, a further study of peat from different geographic locations will also be of importance.
3. An interesting experiment to examine the initial hypothesis of zeroing would be to carry out the $R-\Gamma$ method on an unseparated old peat sample which had been previously exposed to sunlight for some time and subsequently given a known gamma dose. A young material also could be used without the sunlight exposure if one is able to prepare good reproducible discs with it.
4. Since the two attempts of circumventing the anomalous fading yielded somewhat ambiguous results the experiments may be worth repeating with samples that show considerable fading in a short time.

VI. APPENDIX I.

Sample Site Descriptions

Sedimentology of sites MHGP, LCP and KRGP are due to Dr.S.R.Hicock (personal communication). All the ages quoted are in ka.

BBP-3: From top layer of peat in Burns Bog, South Delta. B.C.
Much of the bog has been mined for peat. This sample is from a depth of 25 cm at a place the mine manager said had not been mined.
The estimated age from the sedimentation rate is <0.5 ka. (Hebda, 1977)

FRSS-1: Suspended river silt collected from the Fraser river near the mouth of Coquitlam river, B.C.

QCIP-1: Peat from a sea cliff at Cape Ball, Graham Island, Queen Charlotte Islands. B.C. (53° 45.6'N , 131° 53.0'W). Fig.5.1-a.
Three dates are available: (Mathewes & Clague, 1982;Clague et al, 1982)
On marine shells at the base of the marine sediments above the buried peat layer: 9.35 +/- 0.08 (GSC 3120)
Top of the buried peat layer: 9.16 +/- 0.09 (GSC 3129)
On wood below the buried peat layer: 16.00 +/- 0.57 (GSC 3340).

MHGP: From the gravel pit at Mary Hill, Coquitlam. B.C. (49° 13′58"N , 122° 46′59"W) Fig.5.1-b and Fig. I.1.

> "Peats: Swamp deposits, probably in shallow glacial melt water or where water table fluctuated above and below ground surface.
> Sand: Glaciofluvial outwash - deposited by meltwater streams from advancing glaciers during the Fraser Glaciation (Armstrong & Clague, 1977).
> Silt and fine sand: Pond deposits - horizontally layered and probably settled out of standing or slowly moving water.
> Organic mud flow: A gravity induced mixture of organic and non-organic material moved down a slope upon water saturation and/or disturbance (eg.earthquake) producing reworked material in a mixture of organics, mud, sand and stones all derived from further upslope. The paleoslope at Mary Hill occurs under the organic mud flow.
> Clayey silt: Glaciolacustrine and glaciomarine deposits fine glacial rock flour settled to the bottom of fresh or salt water bodies with stones dropping into the mud from floating icebergs and rafts, producing a fine mud with stones scattered throughout."

There are 18 C-14 dates on the site (Armstrong & Clague, 1977; Hicock & Armstrong, 1981;Hicock, 1980).

Dates on top peat:

18.60 +/- 0.19 (GSC 2194) 18.70 +/- 0.17 (GSC 2344)

Dates on organic matter in sandy silt:

25.80 +/- 0.31 (GSC 2773); 26.00 +/- 0.31 (GSC 2277);

26.20 +/- 0.26 (GSC 2191); 26.45 +/- 0.52 (GSC 124);

26.90 +/- 0.32 (GSC 2217); 27.18 +/- 0.52 (GSC 530).

Dates on peats:

26.10 +/- 0.29 (GSC 2301); 27.00 +/- 0.49 (GSC 2263);

27.40 +/- 0.42 (GSC 2107); 27.50 +/- 0.36 (GSC 2349);

28.20 +/- 0.20 (GSC 2139); 29.60 +/- 0.20 (GSC 2140).

Dates on organic colluvium and below:

40.50 +/- 1.70(GSC 2167); 40.20 +/- 0.43(GSC 2137).

>62.00 (QL 194); >49.00 (GSC 2120).

LCP: Peat from Lynn Canyon Park, North Vancouver, B.C. (49° 21.0′N, 123° 1.2′W) Fig.5.1-a.

> "Peat: Buried vegetation layer that covered ground surface, probably above the water table - pollen so far indicates mainly shrubs and herbs (very few trees).
> Silt: Pond or back water deposits - probably the sediment settled out of standing or slow moving water alongside a stream.
> Gravel: Coarse glaciofluvial outwash - deposited by glacial meltwater streams as glacial ice retreated up the valley; may in part include colluvium (material moved down valley sides under gravity)."

Dates on peat:

30.00 +/- 0.62 (GSC 2793); 34.90 +/- 0.81 (GSC 2873);

>39.00 (GSC 2726) ; 47.80 +/- 0.11 (GSC 3290). (Hicock, 1980).

KRGP: From a gravel pit south of the western end of 84th Avenue (Knight Road), North Delta, B.C. (49° 9.3′N, 122° 55.6′W) Fig.5.1-b and Fig.I.2.

Samples from a wood layer dated 58.8 +/- 2.9, 2.1 (QL 195).

> "Gravel: Glaciofluvial outwash - coarse sandy gravel deposited by glacial meltwater streams, probably close to ice front (fault in the unit suggests collapse of ice blocks and / or ice face itself):
> Sandy silt: Pond or lake deposit-horizontally layered silt probably settled in a body of standing water or backwater alongside a river.
> Gravel: Stream cleaned deposits - stream shifted course and flowed over site for short time (geologically).
> Silt: Backwater or lake deposit-stream channel shifted again and site again covered by standing or slow moving water.
> Wood layer: Water logged snags that floated and sank to the bottom of the lake or backwater; sample from this layer contained some marine dinoflagellate cysts (Hicock, 1980) so the site may have been estuarine at that time.
> Upper silt: More of the lake or backwater deposit."

SSP-1:Peat from Salmon Springs, Summer. Washington. (47° 30′N, 122° W) with C-14 age 71.50 (QL 110, Stuiver et al, 1978).

Fig. 5.1-b.

The TL sample was collected from the middle of the peat layer. A volcanic ash layer about 1m below the peat is dated at 870 +/- 210 ka by fission track method (Easterbrook et al, 1981). The ash is reversely magnetized whereas the immediately overlying silt and peat are normally magnetized. Hence the peat is < 700 ka as the Brunhes-Matuyama boundary must lie between peat and ash.

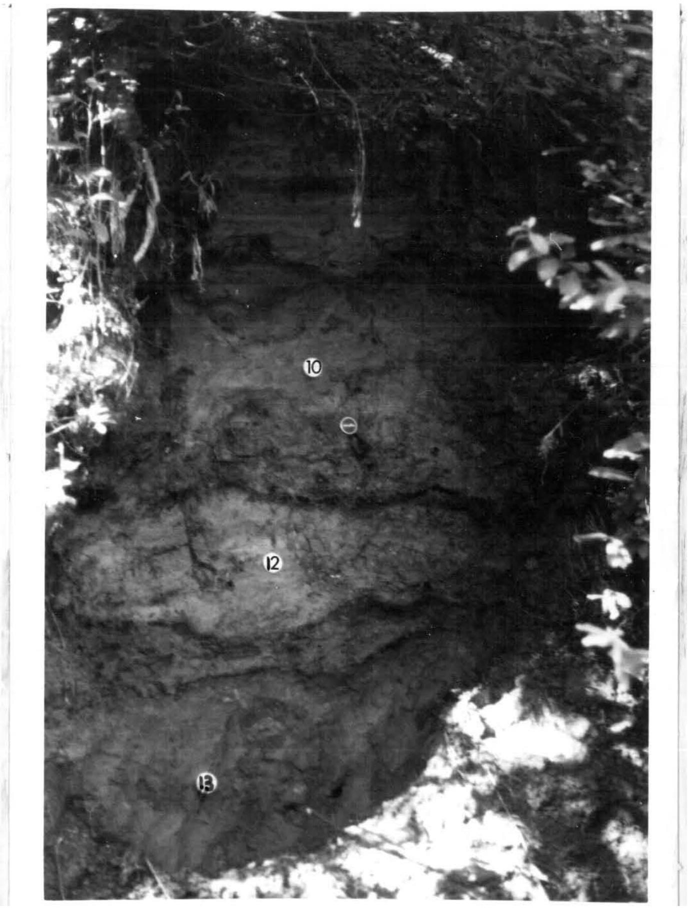


Fig.I.1 MHGP (Mary Hill gravel pit) site.
MHGP-10, 12, 13 samples are shown. TLD' s were buried at MHGP 10 and 12.

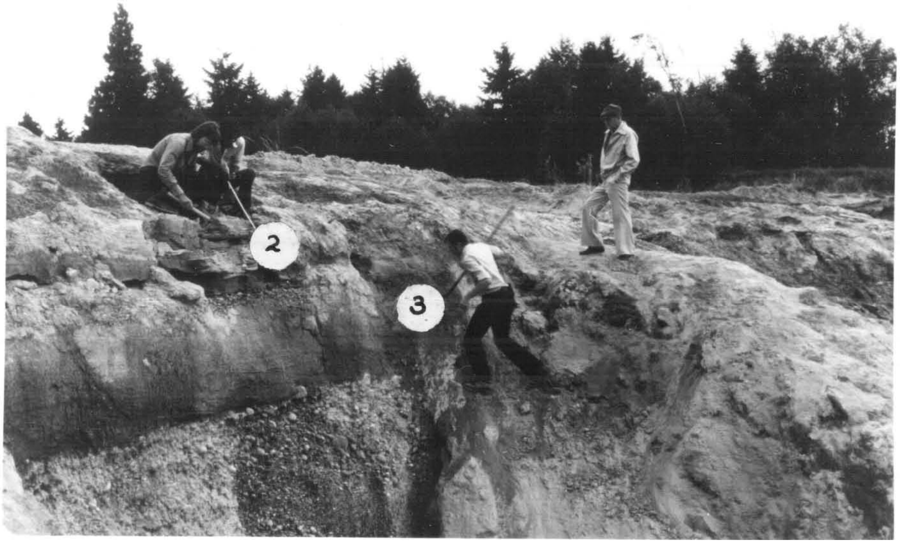


Fig.I.2 KRGP (Knight Road gravel pit) site.
KRGP-2 and 3 samples are shown.

VII. APPENDIX II

Derivation of the Correction Formula for Dose Rates

In a sediment sample containing minerals, water and organic matter the energy E_α of the alpha particles emitted from the radioactive materials in the sample equals the total α energy absorbed by minerals, water and organic matter in the sample at radiation equilibrium.

i.e., $$E_\alpha = E_\alpha^m + E_\alpha^w + E_\alpha^o$$

or

$$E_\alpha^m = \frac{E_\alpha}{1 + (E_\alpha^w / E_\alpha^m) + (E_\alpha^o / E_\alpha^m)} \qquad (1)$$

Let S_α^m, S_α^w and S_α^o be the linear stopping powers of alpha particles (at the average alpha energy) in minerals, water and organic matter respectively. Moreover, let d_m, d_w and d_o be the densities of the respective materials.

The linear stopping power is defined as the amount of energy lost per unit path length of the alpha particle. If we consider a cylindrical volume of cross section area A then,

S/A = Energy lost per unit volume in the material.

Hence

S/Ad = Energy lost per unit mass in the material.

Therefore

$$\frac{E_\alpha^\omega}{E_\alpha^m} = \frac{(S_\alpha^\omega/d_\omega).M_\omega}{(S_\alpha^m/d_m).M_m}$$

and

$$\frac{E_\alpha^o}{E_\alpha^m} = \frac{(S_\alpha^o/d_o).M_o}{(S_\alpha^m/d_m).M_m}$$

where M_m , M_ω and M_o are the masses of minerals, water and organic matter in the sample respectively.

Let

$$H_\alpha^\omega = (S_\alpha^\omega/d_\omega) \ /(S_\alpha^m/d_m),$$

$$H_\alpha^o = (S_\alpha^o/d_o) \ /(S_\alpha^m/d_m),$$

$$\Delta^\omega = M_\omega/ M_m \quad \text{and} \quad \Delta^o = M_o/ M_m .$$

Then the equation (1) can be rewritten as

$$E_\alpha^m = \frac{E_\alpha}{1 + H_\alpha^\omega \Delta^\omega + H_\alpha^o \Delta^o}$$

Since the energy absorbed per unit mass is the dose, we obtain

$$D^{m}_{\alpha} = \frac{D_{\alpha}}{1 + H^{\omega}_{\alpha}\Delta^{\omega} + H^{o}_{\alpha}\Delta^{o}}$$

where D^{m}_{α} is the alpha dose to minerals while the D_{α} is the alpha dose to the minerals in the absence of both water and organic matter.

Similarly one obtains

$$D^{m}_{\beta} = \frac{D_{\beta}}{1 + H^{\omega}_{\beta}\Delta^{\omega} + H^{o}_{\beta}\Delta^{o}}$$

$$D^{m}_{\gamma} = \frac{D_{\gamma}}{1 + H^{\omega}_{\gamma}\Delta^{\omega} + H^{o}_{\gamma}\Delta^{o}}$$

where $H^{\omega,o}_{\beta}$ and $H^{\omega,o}_{\gamma}$ are given by

$$H^{\omega}_{\beta} = (S^{\omega}_{\beta}/d_{\omega})/\,(S^{m}_{\beta}/d_{m}) \quad ; \quad H^{o}_{\beta} = (S^{o}_{\beta}/d_{o})\,/\,(S^{m}_{\beta}/d_{m})$$

$$H^{\omega}_{\gamma} = (\mu^{\omega}_{\gamma}/d_{\omega})/\,(\mu^{m}_{\gamma}/d_{m}) \quad ; \quad H^{o}_{\gamma} = (\mu^{o}_{\gamma}/d_{o})\,/\,(\mu^{m}_{\gamma}/d_{m})$$

where S^{m}_{β} (μ^{m}_{γ}), S^{ω}_{β} (μ^{ω}_{γ}) and S^{o}_{β} (μ^{c}_{β}) are the stopping powers (absorption coefficients) of minerals, water and organic matter for the corresponding radiation. Obviously the same relation holds true for dose rates as well. Thus one obtains the equations (4.11), (4.12) and (4.13). In the absence of organic matter the above equations reduce to equations (4.8), (4.9) and (4.10) respectively.

VIII. APPENDIX III

A Sample Calculation of the Apparent Age

LCP-3 sample:-

Experimental data:

Equivalent dose (Table 3.1-a)	25 ± 3 Gy
'a' - value (Table 4.5-a)	0.064±0.004

(The measured 'a'-value for the LCP-2 sample was taken as that of the LCP-3 sample.)

Alpha count rates (Table 4.5-a):

From U-238 and U-235 decay	$0.189 \pm 0.014\ cm^{-2}ks^{-1}$
From Th-232 decay	$0.052 \pm 0.012\ cm^{-2}ks^{-1}$
The weight per cent of K_2O	0.79 ± 0.04

An uncertainty of 5% was assumed for the K_2O% (private communication: Dr. D.J.Huntley).

Water and organic contents (Table 4.4):

Δ^w (saturation)	0.81
Δ^w (in-situ)	0.81
Δ^o (organic content)	0.24

An uncertainty of about 20% was assumed for Δ^w value considering both in-situ and saturation values for all the other peat samples while that of the Δ^o was assumed to be about 10%. The in-situ water content value is the same as that of saturation for LCP-3 because the in-situ measurement was made in the winter of 1981 when the sample was collected from its wet site.

Calculations:-

The correction factor for the alpha count rates (from equation 4.15) $= c'_{U,Th} / c_{U,Th}$

$$c'_{U,Th} / c_{U,Th} = \frac{0.83 \text{ x } (\text{mass of minerals + organic})}{\left\{\sqrt{\frac{A_{eff}}{21.4}} - 0.17\right\} \text{x}(\text{ mass of minerals })}$$

A_{eff} value (Table 4.3) $= 17.87$

Hence $c'_{U,Th} / c_{U,Th}$
$$= \frac{0.83 \text{ x } 1.24}{\sqrt{\frac{17.87}{21.4}} - 0.17}$$
$$= 1.38$$

The corrected alpha count rates and $K_2O\%$:

U-238 and U-235 $= 0.262 \text{ cm}^{-2}\text{ks}^{-1}$

Th-232 $= 0.072 \text{ cm}^{-2}\text{ks}^{-1}$

$K_2O\%$ $= 0.98$

Now D''_α, D''_β and D''_γ in equations (4.11), (4.12) and (4.13) can be calculated using the equations (4.5), (4.6) and (4.7) respectively. The alpha count rates and $K_2O\%$ of the sample itself were used in calculating the gamma dose to LCP-3 because the sample is surrounded by the same peat layer at least up to about 50 cm in every direction.

$D''_\alpha = 0.38 \pm 0.03$ Gy/ka

$D''_\beta = 1.12 \pm 0.21$ Gy/ka

$D''_\gamma = 0.58 \pm 0.12$ Gy/ka

From equations (4.11), (4.12) and (4.13) the corrected effective dose rates can be calculated as follows.

$$D'_\alpha = \frac{0.38 \pm 0.03}{1 + 1.49x(0.81 \pm 20\%) + 1.37x(0.24 \pm 10\%)}$$

$$= 0.15 \pm 0.02 \text{ Gy/ka}$$

$$D'_\beta = \frac{1.12 \pm 0.21}{1 + 1.25x(0.81 \pm 20\%) + 1.20x(0.24 \pm 10\%)}$$

$$= 0.48 \pm 0.10 \text{ Gy/ka}$$

$$D'_\gamma = \frac{0.58 \pm 0.12}{1 + 1.00x(0.81 \pm 20\%) + 0.98x(0.24 \pm 10\%)}$$

$$= 0.29 \pm 0.07 \text{ Gy/ka}$$

The total effective dose is given by $D = D'_\alpha + D'_\beta + D'_\gamma + D_c$.

The estimated D_c (see section 4.4) $= 0.05 \pm 0.02$ Gy/ka

Hence the total effective dose D $= 0.97 \pm 0.10$ Gy/ka

The 'apparent' TL age $= \dfrac{25 \pm 3}{0.97 \pm 0.10}$ ka

The 'apparent' TL age of LCP-3 $= 26 \pm 4$ ka

The dominant source of error in the final age is due to the uncertainty in the water content. This is clearly seen if one calculates the dose rate assuming the water content alone has an uncertainty. For the 20% uncertainty in the water content alone the corresponding uncertainty in the dose rate is 0.90 Gy/ka compared to the total uncertainty of 0.10 Gy/ka obtained above.

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