

# **Report on the Date of the Wong Tei Tung Archaeological Assemblage**

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30<sup>th</sup> April 2007

## **Abstract**

Previous studies proposed the artifacts found in Wong Tei Tung can be dated to the late Palaeolithic epoch. In order to validate this proposal, a geological survey was carried out in 2006 by two geologists and two Palaeolithic archaeologists from Peking University with two local scholars, and more samples were collected and send to Oxford and Hong Kong University for further OSL dating. The results from the two laboratories indicate that the dates of the Wong Tei Tung artifacts are much younger, probably between 7700-2200 years old. Further, according to two geologists from Peking University, the Wong Tei Tung deposit is an alluvial and disturbed deposit.

## **摘要**

有一種觀點認為黃地峒發現的石器屬於舊石器時代晚期。為了檢驗這一觀點，北京大學的兩位地質學家和兩位舊石器考古學家與兩位本地學者在2006年到該地點進行現場考察，並收集樣品送到英國牛津和香港大學的實驗室重新進行光釋光年代測定。檢測的結果表明黃地峒堆積的年代可能在距今7700到2500年左右；而且根據地質學家的意見，該堆積是經過擾亂的山前堆積。

## **Introduction**

Stone implements have been discovered at Wong Tei Tung in Sham Chung, Sai Kung between 2004 and 2006, and a preliminary Optically Stimulated Luminescence (OSL) dating was conducted by Zhongshan University in 2004 (AMO 2006). It has been argued that Wong Tei Tung represents a late Palaeolithic culture in Hong Kong dated to over 35,000 years ago (Ng *et al.* 2006). If the dates are valid, Wong Tei Tung will be a very important discovery in Hong Kong archaeology.

In order to validate the aforementioned result, further studies were carried out from April 2006 to March 2007, including the following:

- 1) Site visit by geologist Prof. Wei Chunjing of the School of Earth and Space, Peking University and the author of this report in April 2006.
- 2) Site visit by renowned Palaeolithic archaeologists Prof. Lu Zun-e and Prof. Huang Yunping of the School of Archaeology and Museology, and geologist Prof. Xia Zhengkai of the School of Earth and Space, all from Peking University with the author of this report in May 2006. Prof. Lu Zun-e has submitted his comment to AMO.
- 3) Samples for further OSL dating were collected in May 2006. One sample from layer 3 [field code WTT-3(1)], two samples from layer 4 [field code WTT-4(1) and WTT-4(2)] and another two samples from layer 5 [field code WTT-5(1) and WTT-5(2)] were collected by Dr. Li S-H of the Department of Earth Sciences, the University of Hong Kong in May 2006 by standardized OSL sample collection method (Li 2007), and sent to the Luminescence Dating Laboratory of Oxford University, UK for OSL dating. No suitable samples could be collected from layer 2. The Oxford dating was completed in March 2007 and the report is attached as Appendix I (hereafter referred as Schwenninger 2007).

The objective of the aforementioned studies is to examine whether the previous OSL dates are valid. This report is to summarize the outcome of the above studies with some discussions.

## **Results and Discussions**

The Wong Tei Tung deposits have been described by the excavators (Ng *et al.* 2006).

According to Ng and his co-authors, stone implements were discovered in layers 1, 3, 4 and 5,

dated to  $1938\pm64$ ,  $6800\pm600$ ,  $39,000\pm1320$  and  $35,000\pm350$  BP respectively (ibid.; Table 1). It is based on these OSL dates that Wong Tei Tung is defined as a Palaeolithic site (ibid.).

However, OSL dates analyzed by the Oxford University are very different. According to Dr. Schwenninger of the Oxford University, the reliable dates (underlined added) for the Wong Tei Tung deposits are within the middle to the late Holocene (Table 1; Schwenninger 2007: 2). There is one sample from layer 5 dated to  $21,000\pm3500$ , but this is considered NOT reliable (Table 1; Schwenninger 2007: 2). It is further argued that sediments of Wong Tei Tung may be “partially bleached” or “disturbed” (Schwenninger 2007: 2).

Dr. Li of the University of Hong Kong (HKU) raised a similar issue, pointing out that incompletely bleaching of the samples “would result in OSL ages that are systematically too old” (Li 2007:8). The dates given by Dr. Li are also much younger than the dates provided by Zhongshan University (Table 1; Li 2007). Apparently, the two reports by experts from Oxford and the University of Hong Kong have identified one common depositional problem at Wong Tei Tung, which may result in inaccurate and much older OSL dates.

To summarize, we now have three sets of OSL dates from three OSL laboratories for the Wong Tei Tung deposits, as follows:

**Table 1 OSL dates of the Wong Tei Tung site**

Layer	Zhongshan Lab. dates	HKU Lab. dates	Oxford Lab. dates
T4 L1	$1938\pm64$	570	
T4 L2	$2848\pm126$	-	
T4 L3	$6800\pm600$	2230	$2520\pm470$
T4 L4	$39,000\pm1320$	2760	$6470\pm640$ , $5320\pm610$
T4 L5	$35,000\pm1350$	10,300	$7730\pm770$ , [ $21,000\pm3500$ ]

Sources: Ng *et al.* 2006; Li 2007, Schwenninger 2007.

Apparently, dates given by the Zhongshan University laboratory are much more older than the dates given by the other two labs, while the dates tested by Oxford and the University of Hong Kong are relatively closer. In addition, it should be noticed that the Zhongshan

University dates are not in good sequence, with a huge gap between 6800 and 39,000 between layers 3 and 4, and reversed dates between layers 4 and 5; whereas dates given by the other two university laboratories are generally in sequence, except the one sample in layer 5 tested by Oxford, which is considered not reliable and caused by incompletely bleaching (Table 1; Schwenninger 2007).

Still, the majority of these dates are different. While layer 2 of Wong Tei Tung may be about 2500 to 2200 years old based on two reports from HKU and Oxford, there are still approximately three-thousand-years differences between the dates given by HKU and Oxford for layers 4 and 5. This inconsistency may be a result of the sediments of Wong Tei Tung. According to Prof. Xia Zhengkai of the School of Earth and Science, Peking University, the Wong Tei Tung deposits are colluvial or alluvial sediments with sand grains and stones of different ages mixing together, causing difficulties for OSL dating (personal communication, May 2007).

As we all know, the spot at Wong Tei Tung, where stone implements were found and samples were collected, is at the foot of a steep slope. The sediments consist of stone slabs, cobbles and grains of all sizes, representing a typical alluvial or colluvial deposit, which is very commonly found in Hong Kong caused by subtropical to tropical typhoon and landslides every year. As pointed out by Prof. Xia (personally communication 2007), deposits of this type could contain materials of different ages, thus accurate dating will be very difficult.

Taking the geographic factors and the highly diverse OSL dates into consideration, as well as the global climatic and sea level changes from the upper Pleistocene to the Holocene as pointed out by Li (2007), it is more convincing to propose that human activities might have taken place at Wong Tei Tung in the early to middle Holocene. In addition, as some of the stone implements found in Wong Tei Tung are morphologically similar to rough-outs found in the nearby Sha Ha and other Neolithic sites in Hong Kong, it is more prudent to propose that the implements belong to the middle to late Holocene period. In other words, the Oxford dates are more convincing.

### **References:**

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# LUMINESCENCE DATING LABORATORY



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## LUMINESCENCE DATING REPORT

**P256 HONG KONG SEDIMENTS**

**OSL DATING OF FIVE SEDIMENT SAMPLES.**



## Section 1: Project Summary - P256 Hong Kong Sediments

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<b>Address</b>	Department of Anthropology, The Chinese University of Hong Kong, Shatin, N.P., Hong Kong. China.
<b>Nature of samples</b>	Sediment samples
<b>Number of samples submitted</b>	5
<b>Location of site</b>	Hong Kong, China.
<b>Period of interest</b>	Holocene

Field code	Lab. code	Depth (cm)	Palaeodose (Gy)	Dose rate (Gy/ka)	Age (years before 2007)
WTT-3(1)	X2805	67	11.49±1.99	4.56±0.29	2520±470
WTT-4(1)	X2806	100	27.82±2.65	5.23±0.33	5320±610
WTT-4(2)	X2807	100	32.90±2.46	5.09±0.31	6470±640
WTT-5(1)	X2808	130	100.03±15.50	4.77±0.29	[21000±3500]
WTT-5(2)	X2809	130	36.85±2.78	4.77±0.29	7730±770

**Table 1** Summary of the optically stimulated luminescence (OSL) dating results. The results are based on luminescence measurements of sand-sized quartz (180-255µm). All samples were measured using a SAR post-IR blue OSL protocol (Murray and Wintle 2000, Banerjee *et al.* 2001). Dose rate calculations are based on the concentration of radioactive elements (potassium, thorium and uranium) derived from elemental analysis by ICP-MS using a fusion sample preparation technique. The final OSL age estimates include an additional 2 % systematic error to account for uncertainties in source calibration. Dose rate calculations are based on Aitken (1985). These incorporated beta attenuation factors (Mejdahl 1979), dose rate conversion factors (Adamiec and Aitken 1998) and an absorption coefficient for the water content (Zimmerman 1971). The contribution of cosmic radiation to the total dose rate was calculated as a function of latitude, altitude, burial depth and average over-burden density based on data by Prescott and Hutton (1994). Further details regarding individual samples may be found in Appendix 1.



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## Section 2: Comments on the results

For each sample we measured six to twelve multigrain aliquots according to procedures described in further details in sections 4 to 6. The results of the OSL dating are presented in Table 1 and further details regarding individual samples may be found in Appendix 1. The overall luminescence characteristics of the samples were considered to be satisfactory for optical dating, providing good recycling ratios (mean 1.04) and low thermal transfer values (mean 3%). However, a small IRSL signal indicative of feldspar contamination was noticed for most samples and some showed an above average degree of scatter. This could be due to the fact that the sediments were either partially bleached due to insufficient exposure to daylight for complete resetting of the luminescence signal or disturbed. The latter may have resulted through the mixing of younger and older grains through bioturbation or during sampling. We noticed upon arrival of the samples at the laboratory that most containers [X2805-X2808] were not completely filled with sediment and therefore it is possible that a small proportion of the exposed sediment from the end of the containers could have become mixed during handling and transport with the central unexposed material from the centre. This was not the case for X2809 and therefore we consider this result to be more reliable.

From one of the two photographs of the sampling trench which was sent to us we also noticed that the samples appear to have been collected from a deposit containing abundant stony debris (see Figure 1) which can lead to variations in dosimetry [eg. microdosimetric variation]. Because no in-situ radioactivity measurements of the external gamma dose rate are available we can only base our calculations on the dose rate derived from the concentration of radioactive elements present within the OSL sample itself. This does not take account of the surrounding sediment or large stones both of which can have an affect on the dose received by individual quartz mineral grains if the OSL sample is taken within a distance of 30cm or less from sedimentary boundaries or stones characterized by a different mineralogical make-up.

With the exception of sample X2808 which provided a substantially higher palaeodose the remaining samples appear to be in stratigraphic order. We suspect that this sample must have incorporated grains with a residual OSL signal relating to a pre-existing older deposit and which dominates the luminescence signal. For this reason the OSL age estimate obtained for sample X2808 should be considered to be unreliable. The only way to ascertain whether or not this is the case, would be to carry out additional single grain measurements.





**Figure 1** Photograph featuring the location of metal pipe containers used for the collection of five OSL samples. The stony nature of the sediments can cause problems for the correct assessment of the external gamma-dose rate (picture provided by T. L-D Lu).

### **Section 3: The physical basis of luminescence dating**

When ionising radiation (predominantly alpha, beta or gamma radiation) interacts with an insulating crystal lattice (such as quartz or feldspar), a net redistribution of electronic charge takes place. Electrons are stripped from the outer shells of atoms and though most return immediately, a proportion escape and become trapped at meta-stable sites within the lattice. This charge redistribution continues for the duration of the radiation exposure and the amount of trapped charge is therefore related to both the duration and intensity of radiation exposure.

Even though trapped at meta-stable sites, electrons become 'free' once again under certain conditions (e.g. if the crystal is heated and/or illuminated). Once liberated a free electron may become trapped once again or may return to a vacant position caused by the absence of a previously displaced electron (a 'hole'). This latter occurrence is termed 'recombination' and the location of the hole is described as the 'recombination centre'. As recombination occurs, a proportion of the energy of the electron is dissipated. Depending upon the nature of the centre where recombination occurs, this energy is expelled as heat and/or light. When the crystal grain is either heated or illuminated following irradiation (the 'dose') the total amount of light emitted (luminescence) is therefore directly related to the number of liberated electrons and available



recombination sites. This is the fundamental principle upon which luminescence dating is based.

In cases where the duration of dosing is not known (as is the case for dating), estimates can be made from laboratory measurements. The response (the sensitivity) of the sample to radiation dose (i.e. the amount of light observed for a given amount of laboratory radiation, usually  $\beta$ -radiation) must be established. From this relationship the equivalent radiation exposure required to produce the same amount of light as that observed following the environmental dose can be determined, and is termed the 'equivalent dose' ( $D_e$ ). The  $D_e$  (measured in Gy) is therefore an estimate of the total dose absorbed during the irradiation period. When the dose rate (the amount of radiation per unit time, measured in  $\mu\text{Gy/a}$ ) is measured (or calculated from measured concentrations of radionuclides), the duration of the dosing period can be calculated using the equation:

$$\text{Duration of dosing period} = D_e / \text{dose rate.}$$

The technique of optical dating was first applied to quartz by Huntley *et al.* (1985), and methodological details were further developed by Smith *et al.* (1986) and Rhodes (1988). The technique was demonstrated to work well for aeolian samples by Smith *et al.* (1990), and has further proved to provide useful age estimates for a range of sedimentary contexts ranging from aeolian (e.g. Stokes *et al.* 1997) to glacial contexts (Owen *et al.* 1997). Further developmental research has introduced  $D_e$  measurement protocols that use a 'single aliquot regenerative-dose' (SAR) protocol. These protocols have the potential to provide increased precision in the luminescence measurements, and may in some cases provide an indication of incomplete zeroing of the luminescence signal at the time of deposition.

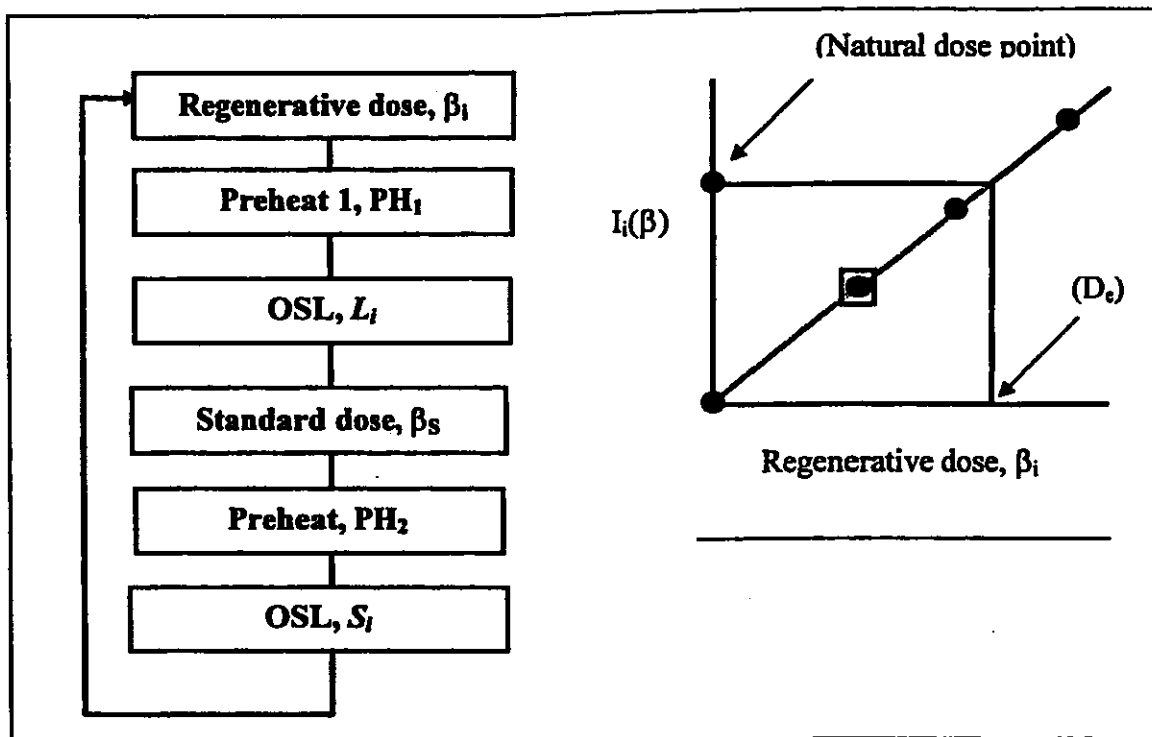
#### **Section 4: The Single Aliquot Regenerative-Dose (SAR) protocol**

The SAR method is a regeneration procedure where the light level of the natural signal is converted into Gy via an interpolation between regenerated (i.e. known dose) points. The natural and regenerated signals are measured using the same aliquot. Sensitivity change commonly observed in quartz TL/OSL has previously precluded meaningful results being obtained this way. A key development reported by Murray and Wintle (2000) is that sample (aliquot) sensitivity is monitored following each OSL measurement ( $L_i$ ) using the OSL response to a common test dose ( $S_i$ ). Plots of  $OSL_1/OSL_2_i$  provide the necessary (sensitivity change corrected) data for interpolation. The procedure is further outlined below, in Figure 4.1.

Murray and Wintle (2000) have introduced two further steps in to the measurement procedure. The first is the re-measurement of the first regenerated data point (indicated by the box in the explanatory Figure 4.1 above). The ratio of the two points (the "recycling ratio") provides an assessment of the efficacy of the sensitivity correction and the accuracy of the technique (large differences being suggestive of an ineffective technique). The second additional step is a measurement of the regenerated OSL due to zero dose. This value gives a



measure of the degree of thermal transfer (to the trap(s) responsible for OSL) during preheating. The ratio of this value to the natural OSL value (both corrected for sensitivity change) gives the "thermal transfer ratio" and this is typically in the range of 0.005-0.020. The "recycling ratio" (ideally unity) is typically in the range 0.95-1.05.



**Figure 4.1** Steps 1-6 are repeated  $n$  times in order to produce the data points required for interpolation (the first dose  $\beta_1$  being zero, to give a measure of the natural signal). Typically  $n=7$  (i.e. the natural plus 6 regeneration points, including one zero dose point and one repeat point).  $PH_1$  and  $PH_2$  are usually different although Murray and Wintle (2000) report no dependence of  $D_e$  on either (over the range of 200-280°C). The OSL signal is integrated over the initial part of the decay (to ~10% of initial intensity) and the background is taken as the light level measured at the end of the OSL measurement.

## Section 5: Measurement procedures / conditions

Luminescence measurements are made using automated Risø luminescence measurement equipment. There are currently three different systems that can be used for routine dating. The major difference between them being the optical stimulation sources. In the first two systems, optical excitation is provided by filtered blue diodes (emitting ~410-510nm), and in the third a filtered Halogen lamp (emitting ~420-560nm) is used. In all three systems, infrared stimulation is also possible using either an array of IR diodes or a single IR laser diode (depending on the measurement system). Luminescence is detected in the UV region on all systems, using EMI 9635Q bialkali photomultiplier tubes, filtered with Hoya U340



glass filters. Sample irradiation is provided in all cases by sealed  $^{90}\text{Sr}$  sources at a rates of 1.5-3 Gy/minute depending on the system used.

In most cases the mean  $D_e$  for each sample is obtained from 6-12 aliquots (see Section 4 for details of calculations). All OSL measurements are made at 125°C (to ensure no retrapping of charge to the 110°C TL trap during measurement) for 100s. The signal in the first 2s (with the stable background count rate from the last 24s subtracted) is normalized using the OSL signal regenerated by a subsequent beta dose ( $\beta_s$ ). To ensure removal of unstable OSL components, removal of dose quenching effects and retrapping necessary to ensure meaningful comparison between naturally and laboratory irradiated signals, 'preheating' is performed prior to each OSL measurement. A preheat ( $\text{PH}_1$ ) at 240°C for 10s was used following the natural and regenerative dose ( $\beta_i$ ), and a preheat ( $\text{PH}_2$ ) of 220°C for 10s was used following each test dose ( $\beta_t$ ). See Section 3 for further details of the SAR method.

## Section 6: Sample preparation

The laboratory procedures were designed to yield pure quartz, of a particular grain size range, from the natural sediment samples. In order to obtain this material, samples were taken through a standard preparation procedure, as outlined below. All laboratory treatments were performed under low intensity laboratory safe-lighting, from purpose-built filtered sodium lamps (emitting at 588 nm).

The sample was wet-sieved to a resolution of  $\sim 50\mu\text{m}$ , and the modal grain size was retained for further processing. Typically the grain sizes used for dating are 90-125 $\mu\text{m}$  or 180-250 $\mu\text{m}$  (see Appendix 1 for details of specific samples). For both samples, the chosen fraction (180-255) was treated with hydrochloric acid (HCl) to remove carbonate and then treated in concentrated HF (48%) for 100 minutes. This treatment serves two purposes: (i) to dissolve feldspar grains, and (ii) to remove (etch) the outer surface of quartz grains (the only part of each quartz grain exposed during burial to natural alpha radiation). Any heavy minerals present were subsequently removed by gravity separation using a sodium polytungstate solution at 2.68 g.cm<sup>-3</sup>. Finally, each sample was re-sieved to remove heavily etched grains. The order of the heavy liquid separation and second sieving are on occasion reversed for practical reasons, and for samples with extremely low yields, either or both of these treatments may be omitted after careful consideration. The prepared quartz samples were mounted on 1cm diameter aluminium discs for luminescence measurement using viscous silicone oil.

Various tests for sample purity are made. Sub-samples of the prepared material are examined using optical microscopy and the sample is exposed (within the Risø measurement system) to infrared (IR) light. Quartz generally does not produce measurable IR luminescence at room temperature whereas feldspar, which can suffer from anomalous fading of the IRSL and OSL signals, or may be less rapidly bleached in some environments, produces an intense luminescence when stimulated with IR. The presence of a strong infra-red stimulated luminescence (IRSL) signal is therefore used as an indication for the presence of feldspar contaminants and is a criterion for rejection. In the rare cases where samples are rejected due



to presence of high levels of IRSL, the prepared sediment sample is treated for ~ 2 weeks in concentrated  $H_2SiF_6$  (silica-saturated HF) which effectively dissolves non-quartz material. If following this treatment, IRSL persists then the sample is subjected to a further two week  $H_2SiF_6$  acid treatment before proceeding to the dating phase (luminescence measurement) and the results are interpreted with caution and the possible contamination of the sample will be discussed. No such extended acid preparation was required for the samples submitted for analysis.

## **Section 7: Dose rate determination**

Radiation dose is described in units of Gray (Gy), the standard SI units of absorbed dose (1 Gy = 1 Joule/kg). The measurement of annual dose rate can be made using a variety of different methods. For most samples, the majority of the environmental dose rate is due to the radioactive decay of unstable isotopes of uranium (U), thorium (Th) and potassium (K). It is therefore necessary to measure the concentrations of each of these elements in each dating sample. An estimation of U, Th and K content can be made using a variety of different methods. These methods are described briefly below.

### **7.1 Field-based gamma-spectrometry**

A portable spectrometer is taken to the sampling site. The probe (housing an NaI scintillator crystal) is inserted in to the cavity left behind following extraction of the sample. Measurements typically take up to one hour and result in direct estimation of the total in-situ gamma radiation field. The spectra are also used to estimate contributions from U, Th and K individually. Through comparison to known concentration standards, quantitative estimates of U, Th and K concentrations are made.

### **7.2 ICP-MS analysis**

A representative sub-sample (typically 10-20g, though as little as a few mg may be used with specialised procedures) of the OSL sample is sent for commercial analysis by fusion ICP-MS to an accredited laboratory. The fusion ensures that the entire sample is dissolved. It is only with this attack that major oxides including  $SiO_2$ , REE and other high field strength elements are put into solution. The sample is first crushed to a nominal minus 10 mesh (1.7 mm), mechanically split (riffle) to obtain a representative sample and then pulverized to at least 95% minus 150 mesh (106 microns). Samples are prepared and analyzed in a batch system. Each batch contains a method reagent blank, certified reference material and 17% replicates. Samples are mixed with a flux of lithium metaborate and lithium tetraborate and fused in an induction furnace. The molten melt is immediately poured into a solution of 5% nitric acid containing an internal standard, and mixed continuously until completely dissolved (~30 minutes). The samples are run for major oxides and selected trace elements on a combination simultaneous/sequential Thermo Jarrell-Ash ENVIRO II ICP or a Spectro Cirros ICP. Calibration is performed using 7 prepared USGS and CANMET certified



reference materials. One of the 7 standards is used during the analysis for every group of ten samples. Totals should be between 98.5% and 101%. If results come out lower, samples are scanned for base metals. Low reported totals may indicate sulphate being present or other elements like Li which won't normally be scanned for. Samples with low totals however are automatically refused and re-analyzed. The measurement of K and Th are usually precise, though samples with low levels of U may be below the detection limit for this element, depending on the interferences from other isotopes.

### **7.3 Alpha-counting and flame photometry**

A sub-sample of sediment (typically 3g) is dried and crushed to a fine powder (<34 $\mu$ m). The powder is then placed in a shallow container that holds a zinc-sulphide  $\alpha$ -scintillator screen directly beneath the sample. The  $\alpha$  emissions of U and Th (during radioactive decay) are then counted, giving a measure of total uranium/thorium concentration. The potassium concentration is estimated using standard flame-photometry methods.

The estimates of U, Th and K concentration are converted to estimates of radiation dose rate (mGy/a) using the standard conversion factors of Adamiec and Aitken (1998) (see Appendix A).

Other factors that influence the annual dose rate, and hence require calculation/measurement, are described below.

### **7.4 Moisture content of the sample**

Moisture within the pore spaces of sediments absorbs  $\alpha$ ,  $\beta$  and  $\gamma$ -radiation. As a result, less is absorbed by the mineral grains. It is therefore important to assess the present day water content of the sediment and to make some assessment of the variability of moisture throughout the burial period of the sample. The moisture correction factors of Aitken (1985) are used in the age calculation (Appendix A).

### **7.5 Cosmic dose rate**

The contribution of cosmic radiation to the total dose rate is calculated as a function of (geomagnetic) latitude, altitude, burial depth and average over-burden density, according to the formulae of Prescott and Hutton (1994).

### **7.6 Radiation attenuation factors**

For coarse grains, the portion of the sample that receives an  $\alpha$ -dose is removed by HF etching. Therefore, no consideration of the  $\alpha$ -dose is made during the age calculation.  $\beta$ -particles (electrons) are significantly attenuated (i.e. a large fraction of the energy is



absorbed) as the  $\beta$ -particle passes through a grain. Account of this effect is needed in order to correctly estimate to dose received by the 'average' grain. The so-called 'attenuation factors' are taken from the empirical work of Mejdahl (1979).

The  $\gamma$ -dose is assumed to be unaffected by attenuation as the penetration of gamma-rays through sediments is several orders of magnitude greater than ( $\sim 10^5$  times) the size of individual grains. Consequently, no attenuation factors are applied to the  $\gamma$ -dose.

Results for the U, Th (ppm) and K (%) concentration of each sample, together with the other parameters used in the age calculation, are given in Appendix A.

## Section 8: Statistics and error calculation

The calculated age depends on the estimate of total absorbed dose ( $D_e$ ) and the annual dose rate ( $D_R$ ). Both of these estimates have uncertainties associated with them. This section gives general details of how the 'error' (the statistical uncertainty) is calculated for each term and combined with the errors on other terms to give an overall estimate of uncertainty on the estimate of age.

### 8.1 $D_e$ estimation

As described in a previous section (Figure 4.1), individual estimates of  $D_e$  are obtained from each of the aliquot (sub-samples) measured, using the SAR technique. The value ( $D_e$ ) is obtained by interpolating between the points of the dose response curve. Statistical uncertainties are calculated for each of the individual points and also on the interpolated value of  $D_e$ . Typically, 12 aliquots are measured for each sample.

Each of the points on the growth curve is defined as

$$I(\beta)_i = \frac{L_i - f \cdot l_i}{S_i - f \cdot s_i} \quad \text{Eq.1}$$

where  $L_i$  is the integrated (initial) OSL from the regeneration dose and  $l_i$  is the measured background signal,  $S_i$  is the integrated (initial) OSL from the test dose (see Section 3) and  $s_i$  is the background;  $f$  is a scaling factor included to take account of the difference in duration of the  $L_i, S_i$  and  $l_i, s_i$  measurements.

The error on each dose-response data point (see Figure 4.1) is calculated by propagating 'counting statistics' errors (assuming Poisson statistics) from the integration of raw OSL data. The error on each term in Equation 1 is given by the square-root of the value. For example, the range for  $L_i$  is given by  $L_i \pm \sqrt{L_i}$ . The errors on each value are propagated in the standard way (see below) to give the uncertainty of  $I(\beta)_i$ .



In cases where the dose response can be (locally) approximated by a straight line, a weighted least squares linear fit is used. The errors in this case are calculated analytically using standard formulae.

In cases where the dose response is significantly non-linear, a single saturating exponential function is used to describe the dose response (a Simplex algorithm is used for fitting in this case). Occasionally an extra linear term is added to the exponential term in order to better describe the form of the dose response, although this is not commonly necessary. The uncertainty for non-linear fitting is calculated using a Monte-Carlo method in which 'random samples' of the dose response data are taken (assuming normally distributed probabilities) and used to obtain a  $D_e$  value. The spread in these values is then used to calculate the error on the mean  $D_e$  for each aliquot, giving a range for each  $D_e$  of  $D_{ei} \pm \sigma D_{ei}$

Once the individual  $D_e$  values have been obtained from each aliquot (and the associated uncertainties calculated) the values are grouped to give a final overall estimate of  $D_e$ . The final estimate ( $\bar{D}_e$ ) is calculated using a weighted average. The weight of each  $D_e$  is referred to as  $w_i$  and defined as

$$w_i = \frac{1}{\sigma D_{ei}^2} / \sum_i \frac{1}{\sigma D_{ei}^2} \quad \text{Eq.2}$$

The weighted mean is defined

$$\bar{D}_e = \sum_i D_{ei} \cdot w_i \quad \text{Eq.3}$$

The weighted standard error,  $\hat{\sigma}_{\bar{x}_w}$ , is calculated from

$$\hat{\sigma}_{\bar{x}_w} = \sqrt{\frac{\sum_i w_i (D_{ei} - \bar{D}_e)^2}{1 - \frac{1}{n}}} / \sqrt{n} \quad \text{Eq.4}$$

where  $n$  is the number of aliquots. The range of the weighted mean  $D_e$  is then defined as

$$\bar{D}_e \pm \hat{\sigma}_{\bar{x}_w} \quad \text{Eq.5}$$

Slight modifications to the approach outlined above are made in special circumstances, though in most cases this description is sufficient.

## 8.2 Dose rate

The errors on the dose rate are due to errors in a range of values, for example, the concentration of U, Th and K, the water content of the sample. The individual components of





the dose rate calculation are shown in Appendix A. The uncertainty on the overall dose rate is calculated by combining the uncertainties according to the standard propagation formula given below.

### 8.3 Age calculation

The calculated age is obtained from dividing the mean  $D_e$  (Eq.3) by the total dose rate (Appendix A). The uncertainty on the final age estimate is calculated using the error propagation formula given below. All calculations were performed using software developed within the laboratory.

### 8.4 Standard error propagation

If a calculated value ( $y$ ) is calculated using a function ( $f$ ) which contains terms  $x_1, x_2, x_3, \dots, x_n$ , then

$$y = f(x_1, x_2, x_3, \dots, x_n) \quad \text{Eq.6}$$

Each term ( $x_i$ ) has an associated uncertainty with a range expressed as  $x_i \pm \sigma_{xi}$ . The overall error of  $y$  can be calculated through the addition of the partial derivatives of  $y$  with respect to each term. Formally, this is written as

$$\sigma_y = \sqrt{\sum_i \left( \frac{\partial y}{\partial x_i} \cdot \sigma_{xi} \right)^2} \quad \text{Eq.7}$$

giving a range for  $y$  as  $y \pm \sigma_y$ .

## Section 9: Selected references

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## Appendix 1

Sample name Laboratory code	WTT-3(1) X2805	WTT-4(1) X2806	WTT-4(2) X2807	WTT-5(1) X2808	WTT-5(2) X2809
<b>Palaeodose (Gy)</b>	11.49	27.82	32.90	100.03	36.85
uncertainty	2.003	2.708	2.546	15.629	2.876
measured	1.99	2.65	2.46	15.50	2.78
Additional laboratory error of 2%	0.230	0.556	0.658	2.001	0.737
<b>Grain size</b>					
Min. grain size (µm)	180	180	180	180	180
Max grain size (µm)	255	255	255	255	255
<b>Measured concentrations</b>					
standard fractional error	0.050	0.050	0.050	0.050	0.050
% K	1.300	1.270	0.990	1.130	1.070
error (%K)	0.078	0.078	0.078	0.078	0.078
Th (ppm)	37.500	47.400	49.300	50.200	43.800
error (ppm)	0.418	0.418	0.418	0.418	0.418
U (ppm)	7.300	8.700	9.700	7.900	9.200
error (ppm)	0.077	0.077	0.077	0.077	0.077
<b>Cosmic dose calculations</b>					
Depth (m)	0.670	1.000	1.000	1.300	1.300
error (m)	0.200	0.200	0.200	0.200	0.200
Average overburden density (g.cm <sup>3</sup> )	1.900	1.900	1.900	1.900	1.900
error (g.cm <sup>3</sup> )	0.100	0.100	0.100	0.100	0.100
Latitude (deg.), north positive	22	22	22	22	22
Longitude (deg.), east positive	114	114	114	114	114
Altitude (m above sea-level))	10	10	10	10	10
Geomagnetic latitude	10.3	10.3	10.3	10.3	10.3
Cosmic dose rate (Gy/ka)	0.178	0.171	0.171	0.164	0.164
error	0.055	0.036	0.036	0.028	0.028
<b>Moisture content</b>					
Measured water content (%)	20.19	20.02	23.42	25.28	22.66
Estimated mean moisture (%)	20.0	20.0	23.0	25.0	23.0
Error (%)	5.0	5.0	5.0	5.0	5.0
<b>Total dose rate, Gy/ka</b>	4.56	5.23	5.09	4.77	4.77
error	0.29	0.33	0.31	0.29	0.29
% error	6.28	6.22	6.19	6.18	6.17
<b>AGE (ka)</b>	2.52	5.32	6.47	20.97	7.73
error	0.47	0.61	0.64	3.52	0.77