REPORT DOCUMENTATION PAGE

Form Approved OMB NO. 0704-0188

1. REPORT DATE (DD-MM-YYYY)	2. REPORT TYPE	3. DATES COVERED (From - To)
29-10-2015	Final Report	1-Jan-2015 - 30-Sep-2015

4. TITLE AND SUBTITLE
Final Report: Luminescence Dating on a Timescale of Days: A Submission to Earth Materials and Processes

5a. CONTRACT NUMBER
W911NF-14-1-0676

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER
611102

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

6. AUTHORS
Edward Rhodes

7. PERFORMING ORGANIZATION NAMES AND ADDRESSES
University of California - Los Angeles
Office of Contract and Grant Administration
11000 Kinross Avenue, Suite 211
Los Angeles, CA 90095 -1406

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS (ES)
U.S. Army Research Office
P.O. Box 12211
Research Triangle Park, NC 27709-2211

10. SPONSOR/MONITOR'S ACRONYM(S)
ARO

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
66081-EV-II.1

12. DISTRIBUTION AVAILIBILITY STATEMENT
Approved for Public Release; Distribution Unlimited

13. SUPPLEMENTARY NOTES
The views, opinions and/or findings contained in this report are those of the author(s) and should not contrued as an official Department of the Army position, policy or decision, unless so designated by other documentation.

14. ABSTRACT
The objective of this project was to assess the potential of using natural environmental mineral samples to determine duration of burial since a target event on a timescale of days and weeks. The approach adopted assessed TL (thermoluminescence), OSL (Optically stimulated luminescence) and IRSL (infrared stimulated luminescence) signals, using variants of standard luminescence dating protocols. Minerals measured included quartz, potassium feldspar, plagioclase feldspar and fluorite. Comparisons were made with low range dosimeters made of aluminium oxide doped with carbon. A simple dose rate conversion kit was constructed and tested successfully, allowing the

15. SUBJECT TERMS
OSL dating, Dose recovery, Short timescales

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	15. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			Edward Rhodes
UU	UU	UU	UU		19b. TELEPHONE NUMBER
					310-825-3463

Standard Form 298 (Rev 8/98)
Prescribed by ANSI Std. Z39.18

Report Title

Final Report: Luminescence Dating on a Timescale of Days: A Submission to Earth Materials and Processes

ABSTRACT

The objective of this project was to assess the potential of using natural environmental mineral samples to determine duration of burial since a target event on a timescale of days and weeks. The approach adopted assessed TL (thermoluminescence), OSL (Optically stimulated luminescence) and IRSL (infrared stimulated luminescence) signals, using variants of standard luminescence dating protocols. Minerals measured included quartz, potassium feldspar, plagioclase feldspar and fluorite. Comparisons were made with low range dosimeters made of aluminium oxide doped with carbon. A simple dose rate conversion kit was constructed and tested successfully, allowing the use of conventional luminescence equipment at low dose ranges for these measurements. Some of the target minerals do display characteristics that allow low doses to be measured, in particular K-feldspar. However, inefficient bleaching by natural sunlight of the luminescence signal measured rendered our field simulations for dating sediment ineffective. In contrast good potential for dating contexts involving heating of natural sediment or construction materials is demonstrated on timescales of weeks.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

UK Luminescence workshop, Glasgow, UK, July 2015

Number of Presentations: 1.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received   Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received   Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received   Paper

TOTAL:

Number of Manuscripts:

Books

Received   Book

TOTAL:

Received Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

Graduate Students

NAME	PERCENT_SUPPORTED	Discipline
Nathan Brown	0.67	
FTE Equivalent:	**0.67**	
Total Number:	**1**	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

NAME	PERCENT_SUPPORTED	National Academy Member
Edward Rhodes	0.06	
FTE Equivalent:	**0.06**	
Total Number:	**1**	

Names of Under Graduate students supported

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:...... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):...... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:...... 0.00

Names of Personnel receiving masters degrees

NAME

Total Number:

Names of personnel receiving PHDs

NAME

Total Number:

Names of other research staff

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

Please see attachment

Technology Transfer

ARO technical report

1 Foreword

The objective of this project was to assess the potential of using natural environmental mineral samples to determine duration of burial since a target event on a timescale of days and weeks. The approach adopted assessed TL (thermoluminescence), OSL (Optically stimulated luminescence) and IRSL (infrared stimulated luminescence) signals, using variants of standard luminescence dating protocols. Minerals measured included quartz, potassium feldspar, plagioclase feldspar and fluorite. Comparisons were made with low range dosimeters made of aluminium oxide doped with carbon. A simple dose rate conversion kit was constructed and tested successfully, allowing the use of conventional luminescence equipment at low dose ranges for these measurements. Some of the target minerals do display characteristics that allow low doses to be measured, in particular K-feldspar. However, inefficient bleaching by natural sunlight of the IRSL signal measured rendered our field simulations using this approach for dating sediment ineffective. In contrast, we achieved excellent sediment dating results using TL signals for one of the simulation experiments. This is an exciting result, demonstrating good potential for using these signals to date contexts involving either light exposure or heating of natural sediment (or construction materials such as brick, concrete or stone) on timescales of weeks, and possibly with further refinement and optimization, days. Quartz OSL and TL displayed some interesting behaviour which may be of significance in understanding the physical and chemical mechanisms of trapping charge in this mineral.

3 List of Tables and Figures

4 Statement of the problem studied

Luminescence dating has been used for the past 50 years to date heated material and disturbed sediments (Wintle, 2008). What remains less well known is how well this technique can work to date heating or sunlight-exposure events younger than a year on natural materials. This study aims to explore behaviour around the lower age limit for luminescence dating of natural quartz and potassium feldspar sediments.

5 Summary of the most important results

The intensity of a measured luminescence signal in depends directly on the total radiation dose experienced during burial (Aitken, 1985). Because luminescence is traditionally used to measure burial events lasting 10^2 to 10^5 years, the beta source strength is designed to provide irradiation in the range of 0.1 to 1000 Gy; the existing $^{90}Sr/^{90}Y$ beta radiation source within UCLA's luminescence reader machine delivers a dose-rate of ~0.1 Gy/s to mineral grains. The dose delivered to a sample after 1 second (the smallest achievable exposure time) using our unmodified source is more than 10^3 times as much as the dose a natural sediment sample would receive in 1 week (Table 1). Therefore, we constructed a spacer and attenuator system to raise the source vertically, and separate it from the sediment by an additional 3/16". Aluminium attenuators were machined to thicknesses of one-, two-, and three-sixteenths of an inch. These modifications allowed us to irradiate natural and synthetic materials over a range of radiation doses, with a lower limit of 9 µGy, or the geologic dose received in about one to two days (Table 2).

A dose recovery test was performed to assess whether doses of several µGy could be administered in the laboratory and then accurately recovered as a 'pseudo-natural' dose (Wintle and Murray, 2006; Fig. 1). The test consisted of removing the natural TL, IRSL and OSL signals of several samples, including natural K-feldspar by heatring to 350ºC. Next, laboratory doses of either 5 or 50 s using the 3/16" attenuator were administered, as a proxy for natural burial irradiation (of around 5 and 50 days, respectively). Finally, the samples were measured using the same protocol as would be used for naturally-dosed samples, using a sensitivity-corrected regenerative-dose protocol. The test thereby assesses whether a known dose can be accurately recovered with a given protocol. For our lower dose values (5s irradiation), using the TL signal measured at relatively low temperatures, the administered dose values of 45µGy agreed well with the measured equivalent dose in the range of 40-60µGy, supporting the use of this protocol for determination of natural burial doses, at least for heated material, where signal zeroing problems are minimized.

Having modified the luminescence reader machine to be capable of reproducing such low doses, we next determined whether measurable luminescence could be naturally produced after 33 and 99 days, and whether the ages of those burial events could be accurately recovered. To emulate a natural anthropogenic disturbance, we dug a pit in the ground near Pearblossom, CA (N 34° 30.355', W 117° 45.108'). While digging the pit, quartz and feldspar from several previously separated natural sediment samples were set out in the bright sunshine to bleach any natural luminescence; the total sunlight exposure time for this was 3 hours and 10 minutes, after which time the samples were enclosed in paper packets and placed within three identical aluminium tubes. Within each tube, we deposited two natural quartz samples, two natural potassium feldspar samples, and two synthetic Al_2O_3:C high sensitivity dosimeters. The samples were chosen as they were among the brightest samples in our sample collection. These tubes were filled to the top with unearthed sediment. Within the pit, we buried these three tubes at an approximate depth of 50 cm below the surface, before refilling the pit with the original sediment. After 33 days the first tube was exhumed, the second after 99 days, and the third tube is still buried. The natural gamma radioactivity within the burial pit was measured directly with a calibrated NaI gamma spectrometer (Fig. 2). Concentrations of U, Th and K in the desert sand at this site were measured using ICP-MS and ICP-OES, in order to estimate the dose-rate from beta radiation for the buried samples (Adamiec and Aitken, 1998), and the internal beta dose rate was estimated according to observations of internal potassium content within sedimentary K-feldspars (Huntley and Baril, 1997). Total dose rates for two locations were 4.1 ± 0.1 and 4.2 ± 0.1 mGy/year for quartz, and 4.8 ± 0.3 mGy/year for feldspar. For feldspar, this corresponds to around 13 µGy per day.

Once each tube was exhumed, the individual quartz or feldspar samples were taken back and removed from the tube in the UCLA luminescence laboratory under controlled laboratory lighting conditions. The natural luminescence signals produced during burial were then measured and compared with the luminescence responses to comparable radiation doses administered in the lab (Fig. 3).

After measuring various luminescence signals resulting from 33- and 99-day burial periods, we can make several observations. First, the low-temperature infrared-stimulated luminescence (IRSL) signals from the two K-feldspar samples (J0560 and J0589), and the optically-stimulated luminescence (OSL) signals from the two quartz samples (J0049 and J0239) grow significantly during burial. In other words, the sensitivity-corrected signals produced during the 99-day burial are uniformly higher than those produced from the 33-day burial.

Second, the thermoluminescence signal (TL) of K-feldspar detected in the blue-green detection window shows promise. Whereas the IRSL signal from J0589 seems to be incompletely reset by the 3-hour exposure to sunlight (Fig. 3a), the TL signal from the same sample seems to be more sensitive to light exposure and to recover accurately the known burial dose (Fig. 3b). The geologic dose calculated for the 33-day burial period is 431 ± 25 µGy and the laboratory equivalent dose measured with the TL signal is 435 ± 8 µGy (n=3 discs). For the 99-day burial period, sample J0589 yielded a significant underestimate of the burial dose; the potential causes for this discrepancy are discussed below.

Third, for all observed samples, the sensitivities to sunlight and to received dose seem to depend significantly on the measurement protocol conditions, such as test dose magnitude, preheat temperature, and preheat duration (Murray and Wintle, 2003). For example, we observe a marked decrease in test dose response from the first SAR cycle to the second in both quartz and feldspar, and for all signals measured (Fig. 4). In quartz, this may reflect dose quenching (Zimmerman, 1971), though this effect normally occurs after higher doses (e.g., 30 Gy; Bailey, 2001; 2004). In feldspar, it is possible that a significant amount of athermal fading occurs at such low doses during and after laboratory irradiation (Kars et al., 2008). This could account for the differences in burial dose estimates between the 33- and 99-day burial ages estimated from sample J0589, given that samples experienced a greater time delay between irradiation and stimulation during the measurement of the 99-day-burial sample, though other possible explanations are plausible.

In summary, the results are exciting, and the project appears successful in achieving a clear outcome. For natural K-feldspar samples, the TL protocols applied are capable of achieving dose values representative of those administered either in the laboratory or simulating natural burial.

6 Bibliography

Adamiec, G., Aitken, M., 1998. Dose-rate conversion factors: update. Ancient TL 16, 37-50.

Aitken, M.J., 1985. Thermoluminescence dating. Academic Press, London, 359 pp.

Bailey, R.M., 2001. Towards a general kinetic model for optically and thermally stimulated luminescence of quartz. Radiation Measurements 33, 17-45.

Bailey, R.M., 2004. Paper I-simulation of dose absorption in quartz over geological timescales and its implications for the precision and accuracy of optical dating. Radiation Measurements 38, 299-310.

Huntley, D., Baril, M., 1997. The K content of the K-feldspar being measured in optical dating or in thermoluminescence dating. Ancient TL 15, 11-13.

Kars, R.H., Wallinga, J., Cohen, K.M., A new approach towards anomalous fading correction for feldspar IRSL dating-tests on samples in field saturation. Radiation Measurements 43, 786-790.

Murray, A.S., Wintle, A.G., 2003. The single aliquot regenerative dose protocol: potential for improvements in reliability. Radiation Measurements 37, 377-381.

Wintle, A.G., 2008. Fifty years of luminescence dating. Archaeometry 50, 276-312.

Wintle, A.G., Murray, A.S., 2006. A review of quartz optically stimulated luminescence characteristics and their relevance in single-aliquot regeneration dating protocols. Radiation Measurements 41, 369-391.

Zimmerman, J., 1971. The radiation-induced increase of the 100 °C thermoluminescence sensitivity of fired quartz. J. Phys. C: Solid St. Phys.

Table 1: Accumulated radiation dose for sedimentary quartz or feldspar, buried for various durations.

Burial time (days)	Realistic geologic dose range (μGy)*
7	38 – 96
30	165 – 411
90	493 – 1232
365	2000 – 5000

*Assuming a range of geologic dose-rates between 2 and 5 Gy/ka. For the pit excavated here, dose rates of 4.1 to 4.8 mGy/year were measured.

Table 2: Laboratory dose-rates corresponding to various machine modifications to the laboratory $^{90}Sr/^{90}Y$ beta source.

Experimental setup	Laboratory dose-rate (μGy/s)
Unmodified radioactive source	104974
3/16" spacer, no attenuator	42593
1/16" aluminum attenuator	6657
2/16" aluminum attenuator	76
3/16" aluminum attenuator	9

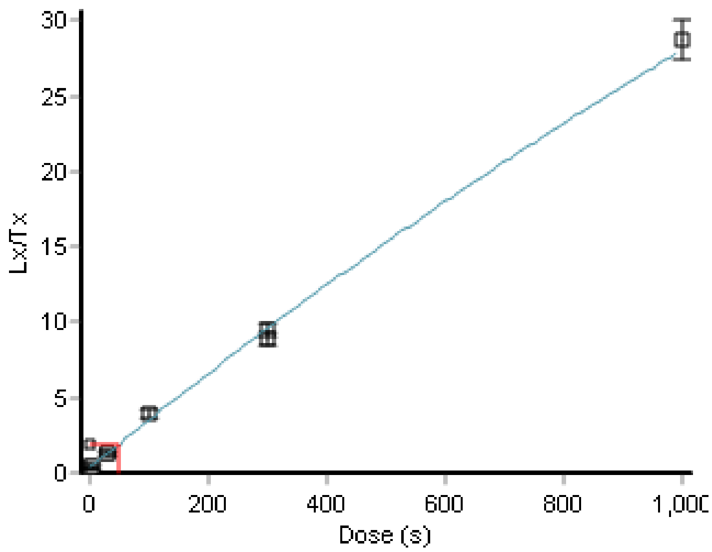

Figure 1: Simulation of dose determination using a laboratory irradiation of a natural K-feldspar sample, mounted in a stainless steel cup. This sample was exposed to the attenuated beta source for 50s, and this dose was treated as a natural dose, which was then recovered as an apparent dose of 51.2 ± 5.6 s exposure. The signal used was the TL between 140 and 280ºC. For reference, 1 s irradiation equates to about 1 days of burial.

Figure 2: The ambient gamma radiation field within the first burial hole is measured with a sodium iodide gamma spectrometer.

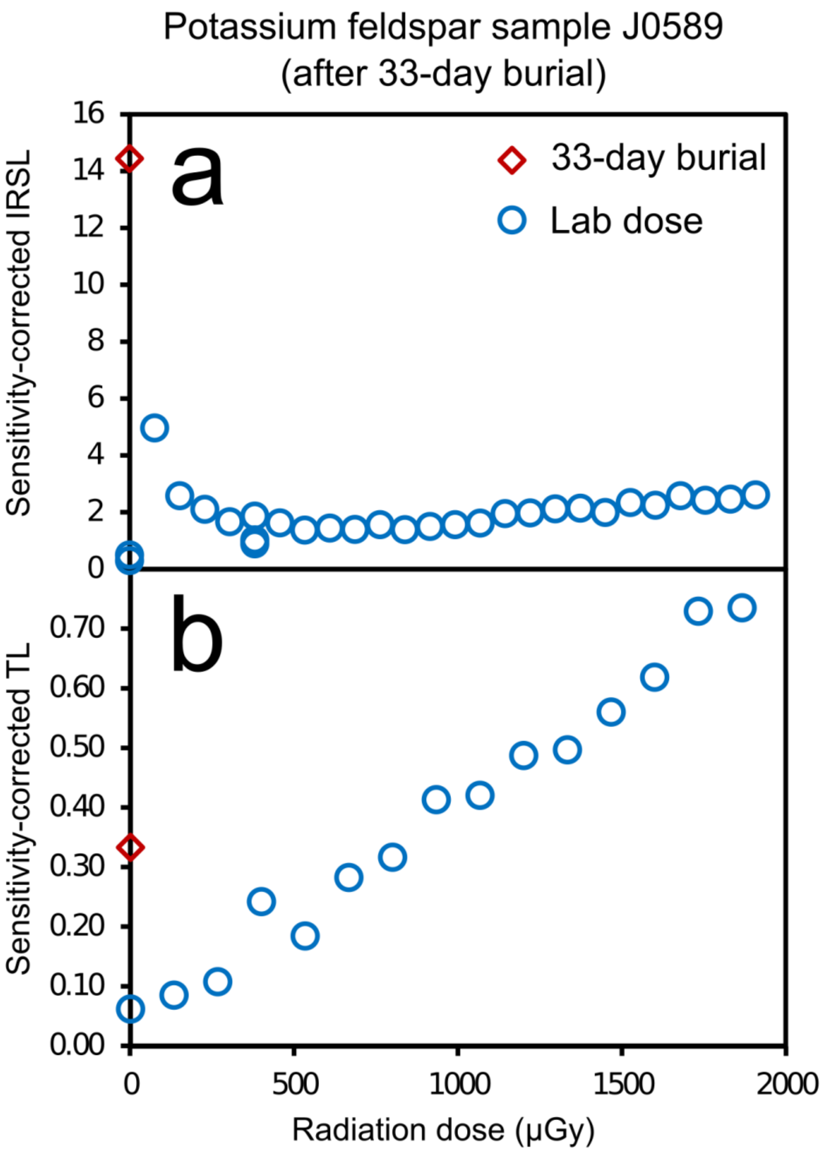

Figure 3: The luminescence responses produced after 33 days of burial are shown as red diamonds for the IRSL (a) and TL (b) signals of sample J0589, enriched K-feldspar separated from a natural sand collected in Southern California. Natural sunlight at the burial site was used to bleach (reduce) the stored electron population, before burial in the ground and subsequent recovery. The equivalent dose estimated with the TL signal (435 ± 8 μGy; n=3 discs) agrees within 1σ with the known geologic dose of 431 ± 25 μGy. The IRSL signal appears to suffer from poor bleaching and dose artefacts, and does not appear suitable for dating in this low range using the protocol applied.

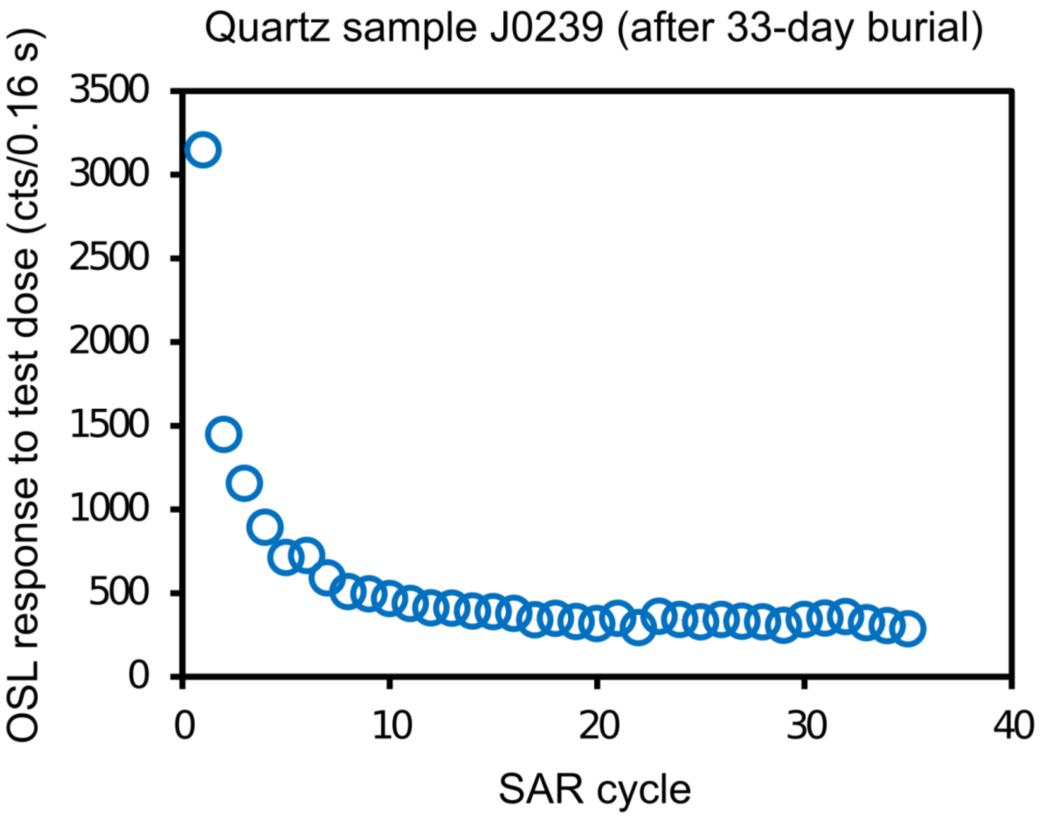

Figure 4: The systematic decrease in OSL sensitivity throughout subsequent SAR cycles implies that the measurement conditions may need to be optimized in order to measure very low doses in quartz. This behaviour, which produces growth curves that decrease with dose initially, represents the phenomenon of dose quenching (Bailey, 2001), and its form may have significant implications for the mechanisms responsible for quartz OSL and TL production.