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COMPARISON OF THREE K-FELDSPAR LUMINESCENCE DATING METHODS FOR HOLOCENE SAMPLES

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Abstract: The luminescence dating of the K-feldspar fraction is an alternative way for samples that cannot yield reasonable equivalent dose (D_e) from quartz fraction with very weak luminescence signal. For testing the reliability of the infrared stimulated luminescence (IRSL) dating of K-feldspar, luminescence dating was applied to quartz and K-feldspar fractions respectively for several Holocene samples in this study. K-feldspar apparent ages using routine single aliquot regenerative-dose (SAR) protocol, K-feldspar ages using g value correction method and ages from isochron dating method were compared with quartz ages. It is found that the g value correction method cannot give reliable ages due to the large errors induced during measurements. The isochron dating method is effective to the sample with problematically external dose rate. However, isochron dating may introduce a relatively greater error during grain sizes – D_e curve fitting, therefore this method could obtain low-resolution ages for Holocene samples. Even K-feldspar apparent age from routine SAR protocol is relatively younger by about 10% than the quartz age, it still could establish reasonable chronological framework for Holocene samples.

Keywords: K-feldspar, Holocene samples, OSL dating, g value correction, isochron dating.

1. INTRODUCTION

Since Huntley *et al*. (1985) presented Optical Stimulated Luminescence (OSL) dating and Hütt *et al*. (1988) presented Infrared Stimulated Luminescence (IRSL) dating for sediments, OSL and IRSL dating have been widely applied to determine the age of Quaternary deposit events (Aitken, 1998). OSL dating was widely applied to aeolian deposit like loess and aeolian sand (Lai, 2010; Murray and Olley, 2002; Sun *et al*., 1998, 2006; Telfer and Thomas, 2007). It was also successfully applied for hydraulic deposit events such as alluvial deposit, colluvium, lakeshore sediment and lacustrine sediment (Berger and Doran, 2001; Eriksson *et al*., 2000; Lang *et al*., 1999, Lang and Zolitschka, 2001; Stone *et al*., 2010). In particular, OSL dating can derive reliable chronology evidence when it is hardly to get available material for ^{14}C dating because OSL dating measures the sediments directly (Argyilan *et al*., 2005; Cupper, 2006). Single aliquot regenerative-dose (SAR) protocol can greatly improve equivalent dose (D_e) measurement accuracy (Wintle and Murray, 2006). The SAR protocols have been established successfully for both quartz (Murray and Wintle, 2000) and potassium rich feldspar (K-feldspar) (Wallinga *et al*., 2000a, b) separations.

K-feldspar grains can emit luminescence signals when they are stimulated by infrared at wavelengths around 800-900 nm (Bøtter-Jensen *et al*., 2003), which is termed

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as IRSL. There are three advantages for IRSL dating from K-feldspar compared with OSL dating from Quartz. Firstly, The IRSL signals from K-feldspar are much brighter than the OSL signal from quartz. It enables high precision of luminescence measurements in laboratory. This leads to high reproducibility for the natural dose measurements (Li *et al*., 2007a). Secondly, D_e values determined from quartz grains can be very scattered for some samples (Zhang *et al*., 2003). For young samples, the random error from D_e determination can be comparable to the age of the sample and thus the interpretation of the ages is difficult. Meanwhile, K-feldspar grains from the same samples give high luminescence intensity and relatively homogenous D_e values (Li *et al*., 2007b). Thirdly, IRSL signal from K-feldspar grains has much higher saturation doses than OSL signal from quartz. It gives potential to date relatively much older sediments than quartz (Kars *et al*., 2008). On the other hand, the main disadvantage of IRSL dating using K-feldspars is anomalous fading of the IRSL signals, which can leads to age underestimation (Auclair *et al*., 2003; Huntley and Lamothe, 2001; Huntley and Lian, 2006; Spooner, 1992, 1994). Anomalous fading rate (g value) correction method and isochron dating method were presented to calibrate the K-feldspar age underestimation.

The g value correction method (Auclair *et al*., 2003; Huntley and Lamothe, 2001) for K-feldspar is a laboratory process which measures the percentage of IRSL signal decay after storage of varying periods following the same laboratory dose irradiation. The method gives a g value in terms of % loss of signal per decade (Aitken, 1985). Usage of this g value allows to calibrate the age underestimation induced by IRSL anomalous fading during SAR protocol.

Isochron dating (IRSL) method for K-feldspar is a protocol that measures D_e of different grain sizes of this mineral (Li *et al*., 2008a). The internal dose rate produced by ^{40}K and ^{87}Rb in the crystal lattice of grains is related to the grain size, which allows the utilisation of an isochron to obtain the relationship between the internal dose rate and the produced D_e, and the corresponding age. As the isochron dating method is totally reliant on the internal dose rate, it could overcome problems related to changes in past external doses rate due to post-depositional environmental changes (Li *et al*., 2008a). This method can also overcome the problem of the anomalous fading of the IRSL signals.

The OSL signal intensity of quartz fraction from some Holocene samples was very low. Some OSL signals were so weak that it was hardly possible to obtain a reliable D_e. A discussion of the use of K-feldspar separation to establishing a dating framework of Holocene samples will be meaningful. This study compares the ages obtained by K-feldspar routine SAR method, g value correction method and isochron dating method with the quartz SAR method for several Holocene samples, and then discusses the reliability and shortcomings of these methods.

2. SAMPLING AND MEASUREMENT

Sampling process

All studied samples were collected from the aeolian-lacustrine deposit sequence in Ulan Buh Desert where is located in the northeastern Alxa Plateau, Inner Mongolia, China (**Fig. 1**). To examine the reliability of the dating results, two almost parallel sections QJD-1 and QJD-1* were investigated (**Fig. 2**). The sediments of these two sections have the same depositional sequence. Therefore, the dating results of section QJD-1 should be consistent with QJD-1* for the same stratum (Zhao *et al*., under preparation). From section QJD-1, samples QJD-1-6, QJD-1-26 and QJD-1-50 were collected from aeolian sand and sample QJD-1-32 from lacustrine silty clay. For comparison, four samples were also collected from the same stratigraphic layers of QJD-1* (**Fig. 2**). All Samples were obtained by hammering iron tubes of 4 cm diameter into the cleaned vertical sections, and then be sealed inside black plastic bags.

Laboratory treatment

In the laboratory, the potentially light-exposed portions at both ends of the tubes were removed. All laboratory processes of sample preparation and luminescence measurement were carried out in subdued red light. All raw samples were treated with 10% HCl and 20% H_2O_2 to remove carbonate and organic matter. The samples were then sieved in water to select the grain size in the size ranges of 63-90 μm, 90-125 μm, 125-150 μm, 150-180 μm, 180-212 μm and 212-250 μm. Heavy liquids of densities 2.62 and 2.75 g/cm^3 separated the grain fraction to obtain quartz and feldspar. After drying, K-feldspar was obtained from the floating part of feldspar fraction separated by heavy liquids of densities 2.58g/cm^3. The quartz grains were treated with 40% HF for 60 min to remove the outer layer irradiated by alpha particles and remaining feldspars. The K-feldspar grains were treated with 10% HF for 40 min to remove the outer layer irradiated by alpha particles. The grains were then treated with 1 mol/l HCl for 10 min to remove fluorides created during the HF etching. K-feldspar fractions for grain size at intervals of 63-90 μm, 90-125 μm, 125-150 μm, 150-180 μm, 180-212 μm and 212-250 μm were obtained. Pure quartz fractions for grain size in the size of 90-125 μm and 125-150 μm were finally acquired.

The OSL measurements were carried out in the Luminescence Laboratory of the Cold and Arid Environment and Engineering Research Institute, CAS, using an automated Risø TL/OSL-DA-15 reader (Markey *et al*., 1997). The OSL signal was detected through two 3 mm thick Hoya U-340 filters. Laboratory irradiation was carried out using $^{90}Sr/^{90}Y$ sources mounted within the reader, with dose rates of 0.104 Gy/s.

The environmental dose rate is created by the radioactive elements existing in grains of the sample and the

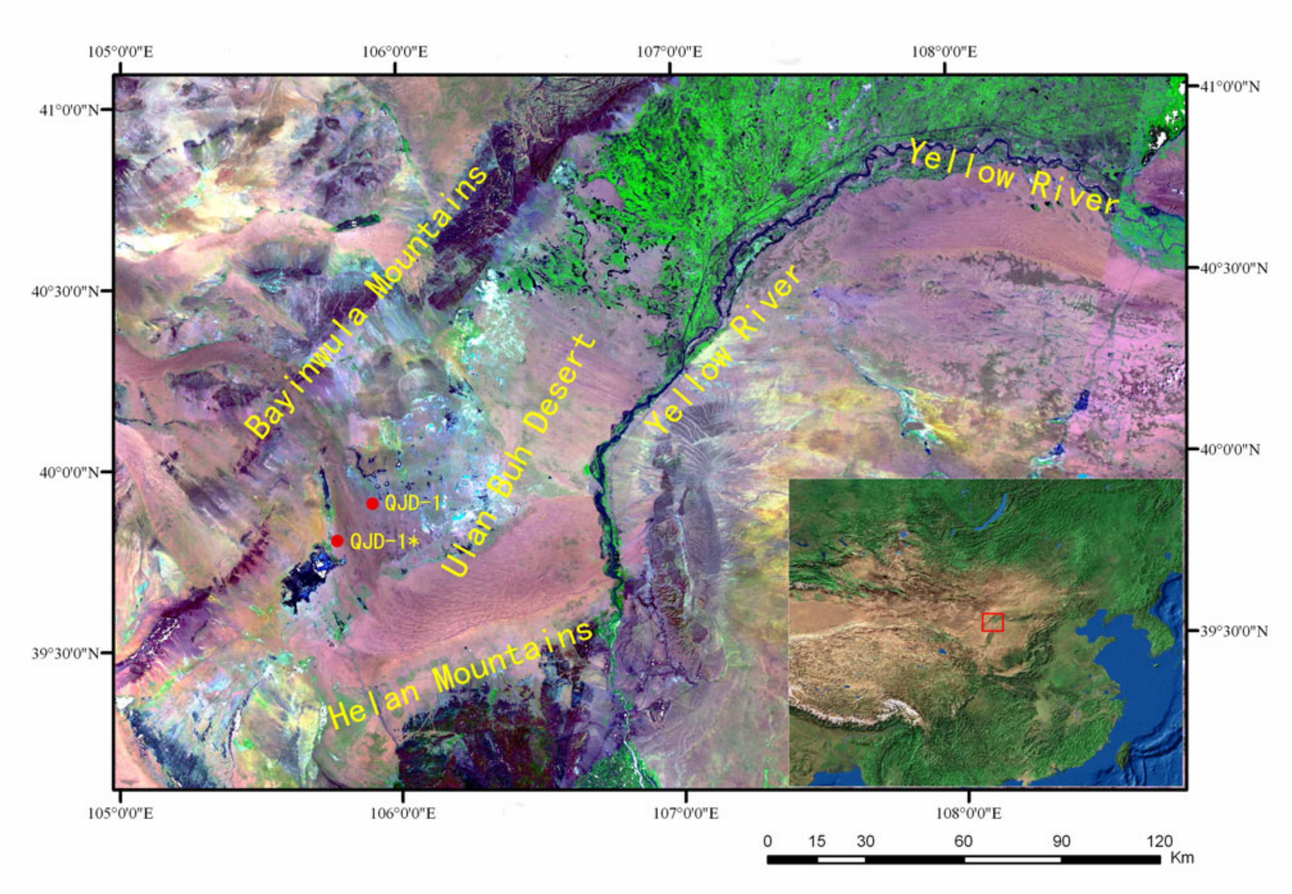

Fig. 1. *The map for location of the section QJD-1 and QJD-1*.*

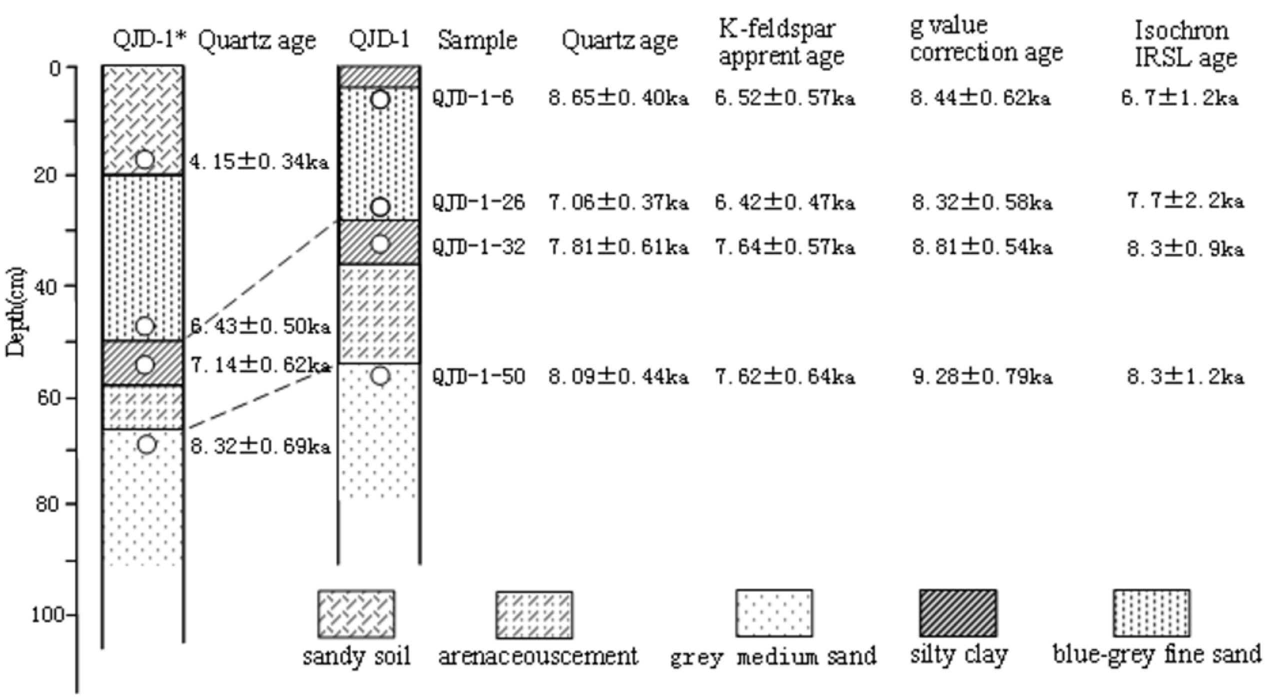

Fig. 2. *Contrast K-feldspar IRSL ages of different methods with quartz ages. Quartz ages from parallel section QJD-1* was shown for comparison.*

surrounding sediments, with a small contribution from cosmic rays. For all the samples measured, U, Th concentrations and K contents were determined by means of Neutron Activation Analysis (NAA). All measurements were converted to alpha, beta and gamma dose rates according to the conversion factors of Aitken (1985). The dose rate from cosmic rays was calculated based on sample burial depth and the altitude of the section (Prescott and Hutton, 1994). The internal dose rate of K-feldspar was calculated with K content of 13±1% (Zhao and Li, 2005; Li *et al.*, 2010). Water content changed drastically after burial for these samples because they were collected from lacustrine sediments in arid area. Since the uncertainty in the mean water content during the sediment burial period, water content of 5±2.5% and 10±5% were introduced in age calculation for eolian sand and lacustrine sediment respectively.

Quartz measurements

Because the optical signal from feldspar is very bright and can suffer from anomalous fading, a small amount of feldspar contamination will greatly affect D_e values from quartz fractions. In order to eliminate the influence from feldspar, the post-IR blue OSL signal was used (Banerjee *et al.*, 2001) and the post-IR single aliquot regenerative dose protocol was used to obtain D_e values from quartz extracts (Zhang and Zhou, 2007). The post-IR SAR protocol was applied to quartz extracts in this study is shown in **Table 1**.

K-feldspar measurements

The D_e for K-feldspar extracts of all samples were measured using a SAR protocol proposed by Wallinga *et al.* (2000a). A preheat plateau test was conducted and it is indicated there is no dependence of D_e on preheat temperature. The results of the dose recovery test also suggest that the protocol in **Table 1** is appropriate for our samples.

For calculating the g value, the single aliquot measurement procedure proposed by Auclair *et al.* (2003) was used. The procedure involves measurements of the sensitivity corrected IRSL signal (L_i/T_i) after different delay times between irradiation and measurement. Four K-feldspar aliquots from each sample were measured. Every sample was bleached by IR and then given a dose of 15.6 Gy prior to fading measurements, where L_i is the response to a laboratory dose of 15.6 Gy and T_i is the response to a test dose of 5.2 Gy, given after the measurement of L_i. Immediately after each irradiation the aliquots were preheated (250°C for 60 s). The measurements of L_i and T_i were made at 50°C with IR stimulation for 80 s. The first measurement took place at a time t_c, fixed as 413.5 s after the mid-point of the irradiation time with a delay of 196s between preheat and measurement. The details of the protocols are demonstrated in **Table 1**.

During isochron dating process, at least 4 grain size fractions of K-feldspar extracts were used for every sample according to isochron dating method (Li *et al.*, 2008a). The D_e for each grain size fraction of K-feldspar was measured using the SAR protocol proposed by Wallinga *et al.* (2000a). It seems that the more grain size fractions used in isochron dating process the higher the precision of the obtained isochron dating results. Six grain size fractions were obtained for samples QJD-1-32 and QJD-1-26. These fractions were used to investigate the precision of the isochron dating method.

3. RESULTS

Ages of Quartz

QJD-1-26 and QJD-1-50 are well-bleached aeolian sand that detected by the correlationship of the D_e values with the sensitive corrected natural OSL signals (Zhang, *et al.* 2003). The ages of their quartz extracts are 7.06±0.37 ka and 8.09±0.44 ka, respectively. QJD-1-32 is also well-bleached lacustrine silty clay with quartz age of 7.81±0.61 ka (**Fig. 2**). At the same time, the quartz ages for sample QJD-1-26, QJD-1-32 and QJD-1-50 are highly consistent with independence quartz ages of correspond-

Table 1. *The single aliquot regenerative-dose (SAR) protocols for quartz, K-feldspar and g value calculation. (a) For the 'natural' sample, i=0 and D_0=0. The whole sequence is repeated for several regenerative doses including a zero dose and a repeat dose. (b) t_i=196 s, 1800 s, 5400 s, 18000 s, 64800 s, 196 s.*

	SAR protocol for quartz		SAR protocol for K-feldspar		g value measurement protocol	
Step	**Treatment**	**Observed**	**Treatment**	**Observed**	**Treatment**	**Observed**
1	Give regenerative dose, D_i[a]		Give regenerative dose, D_i[a]		Give laboratory dose, 15.6 Gy	
2	Preheat at 260°C for 10 s		Preheat at 280°C for 10 s		Preheat at 250°C for 60 s	
3	IRSL measurement at 50°C for 80 s		IRSL measurement at 60°C for 80 s	L_i	Store t_i[b]	
4	OSL measurement at 125°C for 60 s	L_i	Give test dose, D_t		IRSL measurement at 50°C for 80 s	L_i
5	Give test dose, D_t		TL 220°C		Give test dose, 5.2 Gy	
6	TL 220°C		IRSL measurement at 60°C for 80 s	T_i	Preheat at 250°C for 60 s	
7	IRSL measurement at 50°C for 80 s		Return to Step 1		IRSL measurement at 50°C for 80 s	T_i
8	OSL measurement at 125°C for 60 s	T_i			Return to Step 1	
9	Return to Step1					

ing layer of the parallel section QJD-1* (**Fig. 2**). Therefore, the quartz ages for sample QJD-1-26, QJD-1-32 and QJD-1-50 are reliable and will be used to compare with the ages from K-feldspar extracts.

QJD-1-6 is blue-gray aeolian sand overlying lacustrine deposit, collected at the depth of 6-10 cm below the section surface. The true dose rate of it is 95% of the calculated dose rate according to the air-sediment boundary effect of the external γ dose rate (Aitken, 1985). The quartz age of QJD-1-6 is 8.65±0.40 ka, it is reversed to other dating results of section QJD-1 and do not consist with the age (6.43±0.50 ka) of the same stratum on QJD-1*. QJD-1-6 is a well-bleached sample when checking the correlationship of its De values with the sensitive corrected natural OSL signals. This quartz age is not reliable because of the dose rate problem of the waterlain sample (Li, *et al*., 2008b).

Apparent age of K-feldspar

Apparent ages of K-feldspar for sample QJD-1-6, QJD-1-26, QJD-1-32 and QJD-1-50 using the routine SAR method are shown in **Fig. 2**. The K-feldspar apparent age of 6.52±0.57 ka was obtained for the sample QJD-1-6. This result is identical with the same stratum age (6.43±0.50 ka) on section QJD-1*, which is more reliable than the quartz age. The K-feldspar apparent ages of QJD-1-26, QJD-1-32 and QJD-1-50 are 6.42±0.47 ka, 7.64±0.57 ka and 7.62±0.64 ka, respectively, which are smaller than their quartz ages of 7.06±0.37 ka, 7.81±0.61 ka and 8.09±0.44 ka, respectively, due to the anomalous fading of IRSL signals. Anomalous fading led to underestimation of the K-feldspar apparent ages by about 2-9%. Nevertheless, K-feldspar apparent ages using the routine SAR method still enabled establishing the age of the sequence to identify the Holocene deposition event.

g value correction age of K-feldspar

Laboratory measurement process is shown in **Table 1** and the g values were obtained for the studied four samples (**Table 2**). The g value corrected ages of 8.32±0.58 ka, 8.81±0.54 ka and 9.28±0.79 ka for sample QJD-1-26, QJD-1-32 and QJD-1-50 were obtained. They are about 12-20% older than their quartz ages of 7.06±0.37 ka, 7.81±0.61 ka and 8.09±0.44 ka, respectively (**Fig. 2**).

When we measured the g value using the SAR protocol presented by Auclair *et al*. (2003), L_i/T_i values for some aliquots did not decrease monotonously with increasing delay time between irradiation and measurement. **Fig. 3** shows measurement results of four aliquots of K-feldspar extracts for sample QJD-1-32. L_i/T_i values of two aliquots decrease monotonously with increasing delay time (log) between irradiation and measurement; L_i/T_i values of the other two aliquots appeared abnormal, this kind of abnormal decrease induces a large error in the g value calculation. The reason for this is that the IRSL signal decrease with delay time was too small to detect on the laboratory time scale for young samples from the Holocene section. Even the aliquots which L_i/T_i decreased monotonously with delay time (log) were selected to calculate g values, the error induced during the g value calculation is still so large that the calibrated

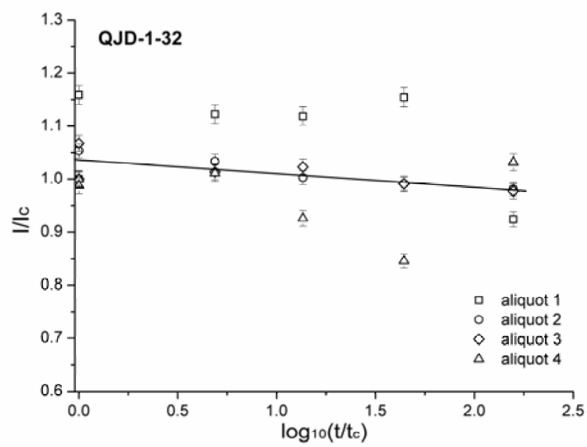

Fig. 3. *g value calculation data of normalized IRSL signal with delay time (log) of K-feldspar for sample QJD-1-32. Each symbol (square, circle, diamond and triangle) represents one of the four aliquots investigated for this sample.*

Table 2. *Relative deviation of K-feldspar ages for different methods compare with quartz age. (a) Sample D4, Dgw5, Sm1 and Sm2 modified according to Li et al. (2008a). (b) Relative deviation for K-feldspar apparent age to quartz age calculated using 13±1% of the K content (Zhao and Li, 2005; Li et al., 2010) and 400±100 ppm of Rb concentration (Readhead, 2002).*

Sample	Quartz			K-feldspar						
	Equivalent dose (Gy)	Dose rate (Gy/ka)	Quartz age (ka)	Apparent age (ka)	Relative deviation (%)	g value (%/10a)	Correction age (ka)	Relative deviation (%)	Isochron age (ka)	Relative deviation (%)
QJD-1-6	14.57±0.38	1.68±0.07	8.65±0.40	6.52±0.57	/	3.99±0.48	8.44±0.62	/	6.7±1.2	/
QJD-1-26	13.79±0.46	1.95±0.08	7.06±0.37	6.42±0.47	9.07/11.33[b]	3.98±0.70	8.32±0.58	17.85	7.7±2.2	9.1
QJD-1-32	14.18±0.43	1.86±0.13	7.81±0.61	7.64±0.57	2.18/5.38[b]	2.30±1.23	8.81±0.54	12.8	8.3±0.9	6.3
QJD-1-50	13.80±0.52	1.71±0.07	8.09±0.44	7.62±0.64	5.81/8.88[b]	3.09±0.82	9.28±0.79	14.71	8.3±2.0	2.6
D4[a]	/	/	3.0±0.2	2.8±0.1	6.67	/	/	/	2.8±0.5	6.67
Dgw5[a]	21.73±0.67	2.61±0.10	8.3±0.3	8.9±0.3	7.23	2.6±0.5	11.4±0.6	37.35	8.7±1.5	4.82
Sm1[a]	/	/	9.1±0.5	8.5±0.4	6.59	3.2±0.4	11.7±0.5	28.57	10.9±1.5	19.78
Sm2[a]	/	/	9.2±0.3	9.0±0.5	2.17	3.4±0.6	12.7±0.8	38.04	8.5±1.2	7.61

K-feldspar ages make a big difference to quartz ages.

Isochron dating of K-feldspar

D_e generated by internal dose rate and external dose rate ($D_f(s)$) (F in **Fig. 4**) is related to internal dose rate for the specified grain size (s), as well as D_e generated by external dose rate ($D_{Fex}(s)$) (F_{ex} in **Fig. 4**). The age t is the slope difference between the trend line F and the trend line of estimated F_{ex} (Li *et al.*, 2008b).

The internal dose rates for different grain size fractions of K-feldspar samples were calculated (**Table 3)**. Internal dose rate $\square_{in}(s) = D_K\Phi_K(s) + D_{Rb}\Phi_{Rb}(s)$, where $\Phi_K(s)$ and $\Phi_{Rb}(s)$ are beta absorption coefficients for K and Rb related to grain size (s) (Fain *et al*., 1999; Readhead, 2002). $\square_{ex}(s)$ for different grain sizes (s) were also calculated using the typical values (K=1.5%, U=3 mg/g, Th=10 mg/g, no radon loss, water content =10% and cosmic ray dose rate = 0.2 Gy/ka) (Li *et al*., 2008b). Then the isochron dating process only needed the measurement D_e for different grain sizes of K-feldspar $D_f(s)$ and extrapolate to obtain the $D_f(0)$, and then using $D_f(0)$ and $\square_{ex}(s)$ value can obtain $\square_{Fex}(s)$ of different grain size fractions. The trend line of $D_f(s)$ and $D_{Fex}(s)$ are then obtained to calculate the age t.

Summary of the data set for constructing the isochron plot are shown in **Table 3.** Ages of all samples by isochron dating method are shown in **Fig. 4**. The isochron age is 6.7±1.2 ka for sample QJD-1-6, which has a problematic quartz age. This result is identical with the independent quartz age of same stratum (6.43±0.50 ka) in section QJD-1*. On the other hand, Sample QJD-1-26, QJD-1-32 and QJD-1-50 have isochron ages as 7.7±2.2, 8.3±0.9 ka and 8.3±1.2 ka, respectively. They are consisting with the corresponding quartz ages but with low precision.

4. DISCUSSION

g value corrected age

For the Holocene samples of QJD-1-6, QJD-1-26, QJD-1-32 and QJD-1-50, using laboratory measured g values, K-feldspar correction ages are 12-20% larger in comparison with the independence reliable quartz ages. It indicates that g value correction method cannot obtain reliable ages for Holocene samples. Wallinga *et al.* (2001) using g value correct samples of 13-300 ka and found that calibrated ages of K-feldspar are younger than quartz ages. Li *et al.* (2008a) using the methods of Auclair *et al*. (2003) measurement of g value modify K-feldspar apparent ages for 10 samples of 11-130 ka. It is found that only two calibration ages have good consistency with quartz ages and the relative deviation values compared to quartz ages are less than 10%, while relative deviation values for the other 8 K-feldspar calibration ages are 15-40% (**Table 2**). Using the g value to correct the K-feldspar apparent age could not lead to obtaining accurate K-feldspar IRSL ages for Holocene samples. The reason for this is that IRSL signal decay of young samples is small in the laboratory time scale compared with old samples.

Isochron dating age

Isochron dating method can be applied to water lain samples like QJD-1-6 with external dose rate problem.

Table 3. *Summary of the data set for constructing the isochron plot*

Sample	Diameter (μm)	$D_f(s)$ (Gy)	$\Phi_K(s)$	$D_{Rb}\Phi_{Rb}(s)/C_{Rb}$ (mGy/ug/g)	$\dot{D}_{in}(s)$ (Gy/ka)	$D_{Fex}(s)$ (Gy)	$\dot{D}_{ex}(s)$ (Gy/ka)
QJD-1-6	63-90	13.34±0.33	0.0301	0.147	0□3644	10.80	2.92
	90-125	13.31±0.42	0.0413	0.182	0.4932	10.69	2.89
	125-150	14.98±0.69	0.0523	0.209	0.6150	10.61	2.87
	150-180	15.67±0.39	0.0623	0.228	0.7244	10.55	2.85
	180-250	15.88±0.82	0.0805	0.253	0.9195	10.40	2.81
QJD-1-26	63-90	15.32±0.19	0.0301	0.147	0.3644	11.17	2.92
	90-125	14.99±0.24	0.0413	0.182	0.4931	11.05	2.89
	125-150	17.43±0.34	0.0523	0.209	0.6150	10.98	2.87
	150-180	18.68±0.43	0.0623	0.228	0.7244	10.90	2.85
	180-250	18.30±0.45	0.0805	0.253	0.9195	10.75	2.81
QJD-1-32	63-90	17.19±0.53	0.0301	0.147	0.3644	15.36	2.92
	90-125	20.43±1.84	0.04133	0.182	0.4931	15.20	2.89
	125-150	18.48±0.78	0.0523	0.209	0.6150	15.09	2.87
	150-180	18.88±1.00	0.0623	0.228	0.7244	14.99	2.85
	180-212	20.06±1.63	0.0736	0.2448	0.8459	14.83	2.82
	212-250	21.18±1.11	0.0863	0.2594	0.9813	14.73	2.80
QJD-1-50	125-150	17.65±0.65	0.0523	0.209	0.6150	12.47	2.87
	150-180	18.17±0.90	0.0623	0.228	0.7244	12.38	2.85
	180-250	19.58±0.33	0.0805	0.253	0.9195	12.21	2.81
	250-300	22.29±1.26	0.1023	0.274	1.1500	11.99	2.76

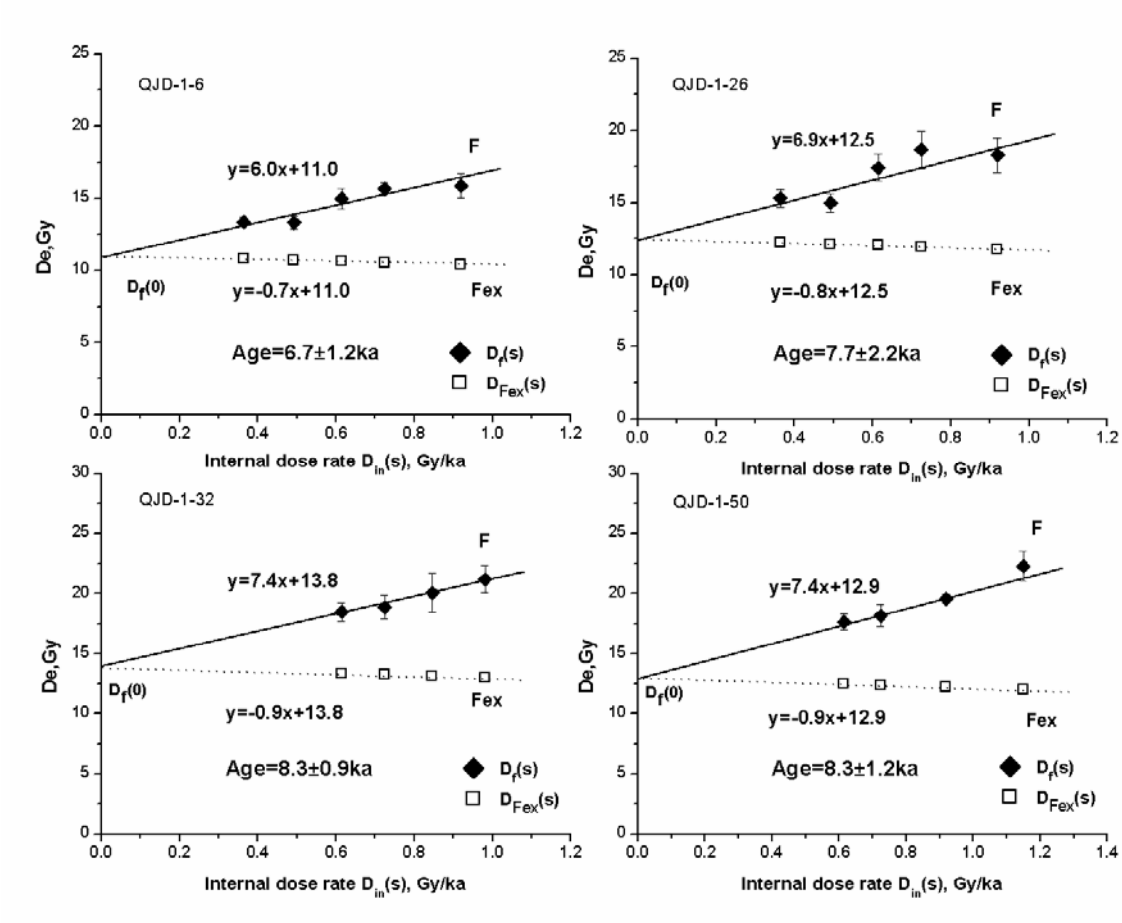

Fig. 4. *Isochron dating results of K-feldspar for sample QJD-1-6, QJD-1-26, QJD-1-32 and QJD-1-50*

However, the precision of isochron dating ages for sample QJD-1-26, QJD-1-32 and QJD-1-50 are low. Because the D_e changes for different grain-sizes are so small that laboratory measurement error may cover the D_e value changes with grain size. As shown in **Table 3**, the D_e values of sample QJD-1-26 do not increase monotonically with grain size. From **Fig. 4,** the goodness of fit of $D_f(s)$ for QJD-1-26 is very low and the isochron age of this sample is not reliable. Using a limited number of points to linear fitting can introduce error. The fitting error may pass to the estimate of $D_f(0)$. It will further increase the error of the estimate linear fitting of external Fex to $\square_{in}(s)$. Isochron dating method can be used to establish a low-resolution framework for the Holocene section.

Six grain size fractions of 63-90 µm, 90-125 µm, 125-150 µm, 150-180 µm, 180-212 µm and 212-250 µm were separated from sample QJD-1-32. In **Fig. 5**, Isochron age of this sample was calculated with 6 and 4 grain size fractions respectively. When all 6 grain size fractions were used, isochron IRSL age of 6.9±1.7 ka were obtained. It is 12% smaller than its quartz age of 7.81±0.61 ka. On the other hand, when 4 grain size fractions of 125-150 µm, 150-180 µm, 180-212 µm and 212-250 µm were used in calculating isochron age, the age of 8.3±0.9 ka was then obtained with a 6% difference with its quartz age. There could not obtain reliable isochron age using all 6 grain size fractions mainly due to the abnormal D_e values of 90-125 µm fraction (**Fig. 5**). If the grain size 90-125 µm fraction was used in routine OSL dating, the apparent age of 8.69 ka will be obtained which overestimates the age by about 11%. It suggests that large error of one grain size D_e would result in significant impact on the isochron IRSL age calculation.

Relative deviation comparison

The relative deviation values for K-feldspar apparent age, g value correction age and isochron IRSL age to their independent quartz age are shown in **Table 2**. The relative deviation values of K-feldspar apparent ages for sample QJD-1-6, QJD-1-26, QJD-1-32, QJD-1-50, D4, Dgw3, Dgw5, Sm1 and Sm2 are between 2-9%. These results do not consider the internal dose rate from Rb concentration as in the routine K-feldspar dating. The relative deviation values will increase when internal dose rate calculated using 13±1% of the K content (Zhao and

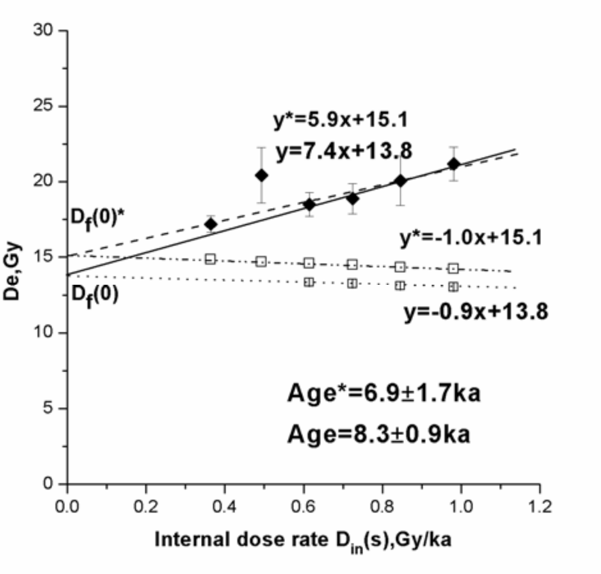

Fig. 5. *The isochron dating results with 7 and 4 grain size fractions linear fitting for sample QJD-1-32. Dashed line is obtained by linear fitting of 7 grain size fractions De and isochron IRSL age of 6.1±1.4 ka was calculated. Solid line is obtained by linear fitting of 4 grain size fractions D_e and isochron IRSL age of 8.3±0.9 ka was calculated.*

Li, 2005; Li et al., 2010) and 400±100 ppm of Rb concentration (Readhead, 2002), but they are still 10% younger than quartz ages. The relative deviation values of K-feldspar g value correction ages for these samples are between 12 and 40%. The relative deviation of isochron ages to quartz ages for sample QJD-1-26, QJD-1-32, QJD-1-50, D4, Dgw5 and Sm2 are less than 10%, while the relative deviation for sample Sm1 is about 20%.

It is indicated that for Holocene samples, the K-feldspar apparent ages are most close to quartz ages. Anomalous fading caused luminescence signal decay for Holocene sample is within systematic errors of laboratory measurement. Li *et al*. (2007b) consider that K-feldspar has advantages over quartz for the dating of very young samples. Although K-feldspar may suffer from anomalous fading, its effect may be negligible for samples younger than 100 years. K-feldspar apparent ages can be used to distinguish deposition events and establish a relatively reliable chronological framework for Holocene sections (see **Fig. 2**). When OSL signals from quartz fractions are too weak to obtain a reliable age for some Holocene samples, K-feldspar apparent age from routine SAR protocol could be used to establish age framework even though it is relative younger no more 10% than independent age.

4. CONCLUSION

IRSL ages obtained by routine SAR protocol, g value correction method and isochron dating method were compared with reliable independent quartz age.

g value correction method cannot obtain reliable ages for Holocene samples in this study due to the large errors induced during g values measurements.

Isochron dating method is effective to the sample with problematic external dose rate and could overcome the anomalous fading of the IRSL signal. However, Isochron dating may introduce relatively larger error during internal dose rate – D_e curve fitting; therefore this method could obtain low precision ages for Holocene samples.

K-feldspar apparent age from SAR protocol could be used to establish age framework even though it is a little bit younger than quartz age.

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REFERENCES

Aitken MJ, 1985. *Thermoluminescence Dating*. Academic Press: 359pp.

Aitken MJ, 1998. *An Introduction to Optical Dating*. Oxford, Oxford University Press: 276pp.

Argyilan EP, Forman SL, Johnston JW and Wilcox DA, 2005. Optically stimulated luminescence dating of Late Holocene raised strand plain sequences adjacent to Lakes Michigan and Superior, Upper Peninsula, Michigan. USA. *Quaternary Research* 63(2): 122-135, DOI 10.1016/j.yqres.2004.12.001.

Auclair M, Lamothe M and Huot S, 2003. Measurement of anomalous fading for feldspar IRSL using SAR. *Radiation Measurements* 37: 487-492, DOI 10.1016/S1350-4487(03)00018-0.

Bøtter-Jensen L, McKeever SWS and Wintle AG, 2003. *Optically Stimulated Luminescence Dosimetry*. Amsterdam, Elsevier: 355pp.

Banerjee D, Murray AS, Bøtter-Jensen L and Lang A, 2001. Equivalent dose estimation using a single aliquot of polymineral fine grains. *Radiation Measurements* 33: 73-94, DOI 10.1016/S1350-4487(00)00101-3.

Berger GW and Doran PT, 2001. Luminescence dating zeroing tests in Lake Hoare, Taylor Valley, Antarctica. *Journal of Paleolimnology* 25(4): 519-529, DOI 10.1023/A:1011144502713.

Cupper ML, 2006. Luminescence and radiocarbon chronologies of playa Sedimentation in the Murray Basin, Southeastern Australia. *Quaternary Science Reviews* 25(19-20): 2594-2607, DOI 10.1016/j.quascirev.2005.09.011.

Eriksson MG, Olley JM and Payton RW, 2000. Soil erosion history in central Tanzania based on OSL dating of colluvial and alluvial hillslope deposits. *Geomorphology* 36: 107-128, DOI 10.1016/S0169-555X (00)00054-4.

Fain J, Soumana S, Montret M, Miallier D, Pilleyre T, and Sanzelle S, 1999. Luminescence and ESR dating Beta-dose attenuation for various grain shapes calculated by a Monte-Carlo method. *Quaternary Science Reviews* 18: 231-234, DOI 10.1016/S0277-3791(98)00056-0.

Huntley DJ and Lamothe M, 2001. Ubiquity of anomalous fading in K-feldspars and the measurement and correction for it in optical dating. *Canadian Journal of Earth Sciences* 38: 1093-1106, DOI 10.1139/cjes-38-7-1093.

Huntley DJ and Lian OB, 2006. Some observations on tunnelling of trapped electrons in feldspars and their implications for optical dating. *Quaternary Science Reviews* 25: 2503-2512, DOI 10.1016/j.quascirev.2005.05.011.

Huntley DJ, Godfrey-Smith DI and Thewalt MLW, 1985. Optical dating of sediments. *Nature* 313:105-107, DOI 10.1038/313105a0.

Hütt G, Jaek J and Tchonka J, 1988. Optical dating: K-feldspar optical response stimulation spectra. *Quaternary Science Reviews* 7: 381-385, DOI 10.1016/0277-3791(88)90033-9.

Kars RH, Wallinga J and Cohen KM, 2008. A new approach towards anomalous fading correction for feldspar IRSL dating—tests on samples in field saturation. *Radiation Measurements* 43: 786-790, DOI 10.1016/j.radmeas.2008.01.021.

Lai ZP, 2010. Chronology and the upper dating limit for loess samples from Luochuan section in the Chinese Loess Plateau using quartz OSL SAR protocol. *Journal of Asian Earth Sciences* 37: 176-185, DOI 10.1016/j.jseaes.2009.08.003.

Lang A and Zolitschka B, 2001. Optical dating of annually laminated lake sediments: A test case from Holzmaar/Germany. *Quaternary Science Reviews* 20(5-9): 737-742, DOI: 10.1016/S0169-555X (99)00043-4.

Lang A, Moya J, Corominas J, Schrott L and Dikau R, 1999. Classic and new dating methods for assessing the temporal occurrence of mass movements. *Geomorphology* 30(1-2): 33-52, DOI: 10.1016/S0169-555X (99)00043-4.

Li B, Li SH, Wintle AG and Zhao H, 2007a. Isochron measurements of naturally irradiated K-feldspar grains. *Radiation Measurements* 42(8): 1315-1327, DOI 10.1016/j.radmeas.2007.09.008.

Li B, Li SH, Wintle AG and Zhao H, 2008a. Isochron dating of sediments using luminescence of K-feldspar grains. *Journal of Geophysical Research* 113: F02026, DOI 10.1029/2007JF000900.

Li B, Li SH, Wintle AG and Zhao H, 2008b. Overcoming environmental dose rate changes in luminescence dating of waterlain deposits. *Geochronometria* 30: 33-40, DOI 10.2478/v10003-008-0003-z.

Li B, Li SH, Duller GAT and Wintle AG, 2010. Infrared stimulated luminescence measurements of single grains of K-rich feldspar for isochron dating. *Quaternary Geochronology*, DOI 10.1016/j.quageo.2010.02.003.

Li SH, Chen YY, Li B, Sun JM and Yang LR, 2007b. OSL dating of sediments from deserts in northern China. *Quaternary Geochronology* 2: 23-28, DOI 10.1016/j.quageo.2006.05.034.

Markey BG, B¢ter-Jensen L and Duller GAT, 1997. A new flexible system for measuring thermally and optically stimulated luminescence. *Radiation Measurements* 27, 83-90. DOI 10.1016/S1350-4487(96)00126-6.

Murray AS and Olley JM, 2002. Precision and accuracy in the optically stimulated luminescence dating of sedimentary quartz: A status review. *Geochronometria* 21: 1-16.

Murray AS and Wintle AG, 2000. Luminescence dating of quartz using an improved single-aliquot regenerative-dose protocol. *Radiation Measurements* 32(1): 57-73, DOI 10.1016/S1350-4487(99)00253-X.

Prescott JR and Hutton JT, 1994. Cosmic ray contributions to dose rates for luminescence and ESR dating: large depths and long-term time variations. *Radiation Measurements* 23: 497-500, DOI 10.1016/1350-4487(94)90086-8.

Readhead ML, 2002. Absorbed dose fraction for ^{87}Rb β particles. *Ancient TL* 20: 25-27.

Spooner NA, 1992. Optical dating: preliminary results on the anomalous fading from feldspar. *Quaternary Science Reviews* 11: 139-145, DOI 10.1016/0277-3791(92)90055-D.

Spooner NA, 1994. The anomalous fading of infra-red stimulated luminescence from feldspars. *Radiation Measurements* 23: 625-632, DOI 10.1016/1350-4487(94)90111-2.

Stone AEC, Thomas DSG and Viles HA, 2010. Late Quaternary palaeohydrological changes in the northern Namib Sand Sea: New chronologies using OSL dating of interdigitated aeolian and waterlain interdune deposits. *Palaeogeography, Palaeoclimatology, Palaeoecology* 288: 35-53, DOI 10.1016/j.palaeo.2010.01.032.

Sun JM, Yin GM, Ding ZL, Liu TS and Chen J, 1998. Thermoluminescence chronology of sand profiles in the Mu Us desert, China. *Palaeogeography, Palaeoclimatology, Palaeoecology* 144: 225-233, DOI 10.1016/S0031-0182(98)00082-0.

Sun JM, Li SH, Han P and Chen YY, 2006. Holocene environmental changes in central Inner Mongolia based on single-aliquot-quartz optical dating and multi-proxy study of dune sands. *Palaeogeography, Palaeoclimatology, Palaeoecology* 233: 51-62, DOI 10.1016/j.palaeo.2005.09.016.

Telfer MW and Thomas DSG, 2007. Late Quaternary linear dune accumulation and chronostratigraphy of the southwestern Kalahari: implications for aeolian palaeoclimatic reconstructions and predictions of future dynamics. *Quaternary Science Reviews* 26 (19–21): 2617-2630, DOI 10.1016/S0031-0182(98)00082-0.

Wallinga J, Murray AS, Duller GAT and Törnqvist TE, 2001. Testing optically stimulated luminescence dating of sand-sized quartz and feldspar from fluvial deposits. *Earth and Planetary Science Letters* 193(3-4): 617-630, DOI 10.1016/S0012-821X(01)00526-X.

Wallinga J, Murray AS and Wintle AG, 2000a. The single-aliquot regenerative-dose (SAR) protocol applied to coarse-grain feldspar. *Radiation Measurements* 32(5~6): 529-533, DOI 10.1016/S1350-4487(00)00091-3.

Wallinga J, Murray AS and Duller GAT, 2000b. Underestimation of equivalent dose in single-aliquot optical dating of feldspars caused by preheating. *Radiation Measurements* 32: 691-695, DOI 10.1016/S1350-4487(00)00127-X.

Wintle AG and Murray AS, 2006. A review of quartz optically stimulated luminescence characteristics and their relevance in single-aliquot regeneration dating. *Radiation Measurements* 41: 369-391, DOI 10.1016/j.radmeas.2005.11.001.

Zhang JF and Zhou LP, 2007. Optimization of the 'double SAR' procedure for polymineral fine grains. *Radiation Measurements* 42: 1475-1482, DOI 10.1016/j.radmeas.2007.06.007.

Zhang JF, Zhou LP and Yue SY, 2003. Dating fluvial sediments by optically stimulated luminescence: selection of equivalent doses for age calculation. *Quaternary Science Reviews* 22, 1123-1129, DOI 10.1016/S0277-3791(03)00054-4.

Zhao H and Li SH, 2005. Internal dose rate to K-feldspar grains from radioactive elements other than potassium. *Radiation Measurements* 40(1): 84-93, DOI 10.1016/j.radmeas.2004.11.004.

Zhao H, Li GQ, Chen FH, Jin M,. Lake-desert changes of early middle Holocene in Ulan Buh Desert in northern China. Under preparation.