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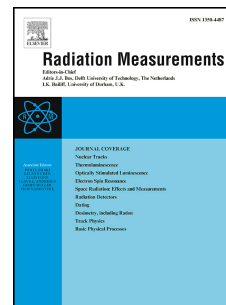
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Age determination using feldspar: evaluating fading-correction model performance

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Abstract

The recent introduction of post-IR IRSL measurement protocols has prompted a resurgence in luminescence applications using feldspar, some of which are affected by anomalous fading related signal loss. Many fading-corrected feldspar ages are reported in the literature, however few of those ages have been corrected using the model of Huntley (2006) [Huntley, D.J., 2006. *An explanation of the power-law decay of luminescence. Journal of Physics: Condensed Matter* 18(4), 1359-1365]. Here we present a new **R** function that calculates fading-corrected ages using the model of Huntley (2006), implemented with either a single-saturating exponential (1EXP) or general-order kinetic (GOK) fit. We evaluate the performance of the model through (i) contrasting measured and modelled field saturation values for a suite of 41 published saturated samples, and (ii) through using the model to fading-correct feldspar ages of samples with independent age control. Our results indicate that when implemented with 1EXP this model has an accuracy of 10 % for predicting sample saturation, but that independent ages may be overestimated when the model is used to fading-correct samples across a range of timescales. In contrast, providing that the dose response curve has been characterised beyond 600 Gy, implementing the Huntley (2006) model with a GOK fit yields accurate age estimations. Modelled age overestimation for 1EXP is associated with dose response curve deviation from a single saturating exponential. Finally we contrast the laboratory measured light levels of a suite of 50 saturated samples with their corresponding fading rates. We show that these saturated samples may yield D_e values below $2D_0$, and thus that $2D_0$ is not an effective screening criterion for sample saturation where no anomalous fading correction is made.

1. Introduction

33 Since the introduction of post-infrared infrared stimulated luminescence (post-IR IRSL)
34 measurement protocols (Thomsen et al., 2008; Buylaert et al., 2009; Li and Li, 2011; Thiel et al.,
35 2011), which measure signals that are less affected by anomalous fading (i.e. athermal signal
36 loss over time due to quantum mechanical tunnelling), there has been a rapid uptake of these
37 new methods in the luminescence dating community. However, high temperature post-IR IRSL
38 signals are hard to bleach in nature (e.g. Kars et al., 2014; Zhang et al., 2015), resulting in the
39 proposal of lower temperature post-IR IRSL measurements for young (e.g. Reimann and
40 Tsukamoto, 2012) or poorly bleached sediments (e.g. Kars et al., 2014). Whilst not suffering
41 from anomalous fading to the same extent as the IRSL₅₀ feldspar signal, low-temperature post-
42 IR IRSL signals often require fading-correction, arguably limiting the benefits of post-IR IRSL
43 measurement protocols. On the basis that fading measurements of quartz minerals (a mineral
44 thought not to fade) yield g_{2days} values of up to 0.98 ± 0.09 %/decade, Buylaert et al. (2012)
45 suggested that it may not be appropriate to fading correct post-IR IRSL signals which yield g_{2days}
46 values of 1-1.5 %/decade. However, non-negligible fading rates (i.e. >1.5%/decade) have been
47 reported for post-IR IRSL₂₂₅ and even for post-IR IRSL₂₉₀ measurements (e.g. Lowick et al.,
48 2012; Lauer et al., 2017), indicating a requirement for fading-correction. Finally, the
49 development of low-temperature OSL-thermochronometry, which utilises low temperature
50 IRSL signals, requires accurate fading-correction in order to model luminescence signal
51 accumulation under changing thermal conditions (e.g. Guralnik et al., 2015a; King et al., 2016a;
52 Valla et al., 2016).

53 Various models for anomalous fading have been proposed, which can be used to correct
54 luminescence ages (e.g. Aitken, 1985; Lamothe and Auclair, 1999; 2000; Huntley and Lamothe,
55 2001; Lamothe et al., 2003; Huntley, 2006). The model of Huntley and Lamothe (2001) is the
56 most widely used approach but is only applicable to the linear part of the dose response curve,
57 although experiments by Buylaert et al. (2008) indicate that it may also be effective beyond this
58 range. A function for age correction using the Huntley and Lamothe (2001; H+L) approach has
59 been implemented in the **R** package 'Luminescence' (Kreutzer et al., 2012; 2018) and is
60 commonly used to correct feldspar luminescence ages (e.g. Bickel et al., 2015; Diaz et al., 2016).
61 The dose rate correction method (DRC) of Lamothe et al. (2003) has the advantage of
62 potentially being applicable over the full range of the dose response curve, and has also been
63 successfully applied in some studies (Auclair et al., 2007; Buylaert et al., 2009; Kars and
64 Wallinga, 2009). Huntley (2006) proposed a model on the premise that rates of fading can be
65 explained by the density and distance of recombination centres from the IRSL trap. Kars et al.
66 (2008) and Li and Li (2008) used this model together with a single-saturating exponential fit
67 (1EXP) to evaluate measured luminescence data, and Kars and Wallinga (2009) also

68 investigated the impact of charge trapping competition on rates of athermal loss. Guralnik et al.
 69 (2015a) also used the Huntley (2006) model to fading correct bedrock thermochronometry
 70 samples, but implemented it with a general-order kinetic (GOK) fit. Here we evaluate the
 71 Huntley (2006) model for age fading correction, using a new **R** function that is freely available
 72 in the ‘Luminescence’ package (v.0.8.1, Kreutzer et al., 2012; 2018), `calc_Huntley2006()`,
 73 (King and Burow, 2018) which calculates fading-corrected ages and sample specific field
 74 saturation values, from which the ability to derive a finite age can be evaluated. We highlight the
 75 effects of fading over laboratory timescales and show why $2D_0$ is not an effective screening
 76 criterion for sample saturation for samples which experience anomalous fading.

77 **2. The model**

78 Huntley (2006) proposed a model that explains the power-law decay of luminescence
 79 signals through tunnelling. Various detailed descriptions of this model have been given in the
 80 literature (cf. Kars et al., 2008; Kars and Wallinga, 2009; Li and Li, 2008; Morthekai et al., 2011;
 81 King et al., 2016a) and it is only briefly described here. The model is based on the premise that a
 82 trapped electron will tunnel to its nearest neighbouring recombination centre, with the lifetime
 83 of trap occupancy governed both by recombination centre density (ρ) and recombination centre
 84 distance from the electron trap (r), (Figure 1). The density of recombination centres is assumed
 85 to be much greater than trapping centres (Huntley, 2006) and can be quantified from fading
 86 measurements (for example using the `analyse_FadingMeasurement()` function in the **R**
 87 package ‘Luminescence’, Kreutzer and Burow (2017)), whereby the luminescence signal
 88 remaining after increasing measurement delays is fitted with equation 5 of Kars et al. (2008):

$$89 \quad IRSL_{faded} = IRSL_{initial} e^{-\rho' \ln(1.8 s t^*)^3} \quad [1]$$

90 where $IRSL_{faded}$ is the luminescence signal measured at time t^* , which is the time between
 91 irradiation and measurement, including half the time of irradiation (after Auclair et al., 2003).
 92 $IRSL_{initial}$ is the luminescence signal without any anomalous fading, ρ' is the dimensionless
 93 density of recombination centres and s is $3 \times 10^{15} \text{ s}^{-1}$ following Huntley (2006), which is the
 94 attempt-to-escape frequency of an electron from an atom-sized box.

95 *[Figure 1]*

96 Li and Li (2008), Kars et al. (2008), Kars and Wallinga (2009) and Valla et al. (2016)
 97 have investigated the model of Huntley (2006) in detail, and we review their investigations
 98 here. The Huntley (2006) model is based on the assumption that the density of recombination
 99 centres, ρ , remains effectively constant. However, following Huntley and Lian (2006), Li and Li
 100 (2008) proposed that a changing density of recombination centres may be required to account

101 for laboratory measured fading-rate dose-dependency which has been reported in a number of
102 studies (Huntley and Lian, 2006; Kars et al., 2008; Li and Li 2008). Li and Li (2008) observed
103 that for sample Sm1, the g-values (normalised to $t_c = 1200$ s, rather than 2 days) increased from
104 3.1 ± 0.3 %/decade to 5.4 ± 0.2 %/decade with given doses of 32 and 2050 Gy respectively
105 (Figure 2A). However where they plotted the normalised natural luminescence signals (L_{nat}) of
106 the same sample suite against their estimated palaeodoses, the data can be well described by a
107 single saturating exponential dose response curve predicted from a single density of
108 recombination centres using the Huntley (2006) model (Figure 2B). It is unclear how this
109 discrepancy can be reconciled. Kars and Wallinga (2009) tested the performance of the Huntley
110 (2006) model, as implemented for age fading correction by Kars et al. (2008) for a suite of
111 samples with independent age control. Using a single saturating exponential curve for dose
112 response, Kars and Wallinga (2009) obtained fading-corrected ages commensurate with, albeit
113 older than independent ages. Following Huntley and Lian (2006) and Wallinga et al. (2007),
114 Kars and Wallinga (2009) also investigated whether charge-trapping competition effects with
115 increasing dose could affect model performance. However, they found that incorporation of
116 charge trapping competition led to greater deviation between fading corrected ages and
117 independent age controls. Valla et al. (2016) evaluated the performance of the Huntley (2006)
118 model as implemented by Guralnik et al. (2015a), i.e. using a general order kinetic model
119 (Guralnik et al., 2015b) rather than a single saturating exponential fit for dose response. Valla et
120 al. (2016) contrasted laboratory measured saturation ratios with those predicted for athermal
121 steady state for 32 bedrock samples, recording a correlation between measured and modelled
122 levels of saturation for some samples, inferring that the majority of their dataset reflected
123 athermal signal loss rather than rock cooling histories. Our study complements the earlier work
124 of Kars et al. (2008), Li and Li (2008), Kars and Wallinga (2009) and Valla et al. (2016) through
125 evaluating how well the Huntley (2006) model can predict field saturation for 41 saturated
126 samples taken from existing literature when implemented with 1EXP or GOK, and also its age
127 fading correction performance for a further suite of samples with independent age controls.

128 *[Figure 2]*

129 **3. The function**

130 The `calc_Huntley2006()` function, which has been recently added to the **R** package
131 'Luminescence', requires the user to input:

- 132 • the regenerative dose times used to measure the dose response curve (s),
- 133 • the sensitivity corrected luminescence data (i.e. L_x/T_x , a.u.) and their individual
134 uncertainties,

- 135 • the reader dose rate \dot{D}_{reader} (Gy s⁻¹) on which the Lx/Tx data were measured,
 136 • the environmental dose rate \dot{D} (Gy ka⁻¹), and
 137 • the sample specific ρ' (dimensionless).

138 Calculation of the model comprises three stages:

139 (1) Simulation of an unfaded dose response curve for the given \dot{D}_{reader} and ρ' , from which the
 140 sample's level of saturation (n/N) can be calculated (e.g. Figure 3), where n is the number of
 141 trapped electrons and N is the maximum number of electron traps.

142 (2) Simulation of a natural dose response curve for the given \dot{D} , ρ' and unfaded D_0 (e.g. Figure 3)
 143 from which the fading-corrected age can be determined through interpolation of the sensitivity
 144 corrected natural signal, L_{nat} .

145 (3) Determination of field steady-state (i.e. the equilibrium between charge trapping due to
 146 exposure to ionizing radiation and charge detrapping due to athermal loss), (n/N)_{ss}, for the
 147 given \dot{D} , ρ' and unfaded D_0 .

148 The simulated unfaded and natural dose response curves, as well as the laboratory measured
 149 dose response curve are shown in the `calc_Huntley2006()` model output (Figure 3),
 150 enabling comparison of the different levels of saturation and the influence of anomalous fading
 151 on laboratory, and natural dose response.

152 *[Figure 3]*

153 **4. Model performance**

154 **4.1 Determining natural signal saturation**

155 In order to test the performance of the model of Huntley (2006), we have contrasted the
 156 measured (n/N) values of a range of different saturated samples reported in the literature, with
 157 their calculated (n/N)_{ss} values calculated either using 1EXP or GOK (Figure 4). Kars et al. (2008)
 158 tested the performance of this model implemented with 1EXP for five saturated samples from
 159 the Lower Rhine (reported average value included in Figure 4), and observed that the model
 160 underestimated the average measured field saturation level by 9 % (note that no information on
 161 scatter between the five samples is given in the original publication). We have also calculated
 162 (n/N)_{ss} for the IRSL₅₀ and post-IR IRSL₂₉₀ signals of sample 62213 (for both early and late
 163 background subtraction) reported in Thomsen et al. (2011), and also report data for a number
 164 of saturated bedrock samples, which were measured as the limitations of OSL-
 165 thermochronometry were explored (Guralnik et al., 2015a; Valla et al., 2016; King et al., 2016b).

166 Only samples with adequate independent age control, confirming their saturation, have been
167 included; the data used to produce Figure 4 are summarised in Table 1. Where a 1EXP fit is
168 used, the majority of the samples investigated here, which are derived from a variety of
169 different geological and geographical settings, exhibit (n/N) within 10 % of $(n/N)_{ss}$ (Figure 4A).
170 In contrast where a GOK fit is used, the data are more scattered and exhibit a trend towards
171 overestimation of $(n/N)_{ss}$ relative to (n/N) (Figure 4B). The cause of the discrepancy between
172 these two model implementations is unclear, but may indicate that feldspar do not follow GOK
173 dose response in the natural environment (cf. Li and Li, 2012), which is discussed further below.
174 The data in Figure 4A indicate that the Huntley (2006) model has approximately 10 % accuracy
175 when applied with a 1EXP fit, but that there is no apparent systematic bias towards over or
176 underestimation of sample field saturation. The scatter in the 1EXP results likely reflects
177 uncertainties in the laboratory-constrained rates of anomalous fading and dose response.
178 Huntley and Lian (2006) report differences in rates of anomalous fading between individual
179 aliquots of the same sample, by up to a factor of two, and some of this scatter may also be
180 related to averaging of anomalous fading rates.

181 *[Figure 4]*

182 **4.2 Fading-correction: Comparison with independent age control**

183 The adequacy of any fading model is determined by its ability to accurately correct ages.
184 Kars and Wallinga (2009) investigated the performance of the Kars et al. (2008)
185 implementation of the Huntley (2006) model with 1EXP on a suite of samples from Bortel, for
186 which quartz OSL ages from 15 ka to 325 ka provided age control. Although contrasting
187 luminescence chronologies can be limited by commonalities in \dot{D} and D_e values, such
188 comparisons are often practical. Kars and Wallinga (2009) report that whereas one feldspar age
189 overestimated its partner quartz age by 150 ka (175 %), the remaining 10 samples exhibited
190 better agreement, whilst all systematically overestimating the quartz ages. Incorporating charge
191 trapping competition effects into the model resulted in greater discrepancies between quartz
192 and feldspar fading-corrected ages (Kars and Wallinga, 2009, their Figure 1) and is not
193 considered here. Morthekai et al. (2011) also investigated applying the approach of Kars et al.
194 (2008) and Huntley and Lamothe (2001) to fading-correct basaltic feldspars with known ages
195 ≥ 400 ka. They found that both methods underestimated sample age, and furthermore that the
196 approach of Huntley and Lamothe (2001) exhibited more severe (~ 80 %) age
197 underestimations. More recently Li et al. (2018) used the Kars et al. (2008) and Lamothe et al.
198 (2003) approaches to fading correct ages of samples from the Heidelberg Basin, Germany. From
199 contrasting their resulting ages with a palaeomagnetic stratigraphy, they determined that the

200 Kars et al. (2008) model gave more reliable results. In this study, we evaluate performance of
201 the Huntley (2006) model for the IRSL₅₀ signals of two sets of sedimentary samples with
202 independent age control. The first sample set (GOS/ZEL) are from Lowick et al. (2012) and the
203 second set (MFRB/BRR) are from this study.

204 4.2.1 Sample details

205 *GOS3 and GOS4*

206 Polymineral fine-grained samples GOS3 and GOS4 were taken from the Gossau gravel pit
207 in Kanton Zürich, Switzerland (Preusser, 1999; 2003), which hosts some of the most
208 comprehensively investigated Marine Isotope Stage 3 sediments in this region (the Gossau-
209 Interstadial-Complex). The samples were collected from overbank deposits adjacent to
210 previously dated peat horizons (Figure 5). For sample GOS4 the adjacent peat has been dated to
211 ~32.3 cal. ka BP using radiocarbon dating and 34.7 ± 4.0 ka using ²³⁰Th/U TIMS (Geyh and
212 Schlüchter, 1998; Preusser, 2003). Sample GOS3 is stratigraphically older, and overlies a peat
213 horizon with radiocarbon ages of ~48.0-50.5 cal. ka BP (Schlüchter et al., 1987; Preusser, 2003)
214 as well as a ²³⁰Th/U TIMS age of 49.4 ± 3.3 ka (Geyh and Schlüchter, 1998). These samples have
215 previously been dated using a multiple-aliquot IRSL approach, resulting in polymineral fine-
216 grain ages of 51.8 ± 5.4 ka for GOS3 (Preusser, 1999) and 29.0 ± 3.9 ka for GOS4 (Preusser,
217 2003). More recently Lowick et al. (2012) used a 225 °C and 290 °C post-IR IRSL protocol to re-
218 investigate these samples, resulting in fading-corrected ages of 60 ± 4 ka to 78 ± 10 ka for GOS3
219 and 41 ± 2 ka to 45 ± 4 ka for GOS4 dependent on signal and including IRSL₅₀ signals measured
220 within the post-IR IRSL₂₂₅ protocol. Lowick et al. (2012) measured fading rates for two aliquots
221 of a range of samples from Switzerland, including GOS3 and ZEL7 (see below). They found that
222 measured g_{2days} values were similar across the entire sample suite, resulting in average values of
223 2.33 ± 0.84 %/decade for the IRSL₅₀ signal measured within the post-IR IRSL₂₂₅ protocol, $1.62 \pm$
224 0.40 %/decade for the post-IR IRSL₂₂₅ signal and 2.13 ± 1.39 %/decade for the post-IR IRSL₂₉₀
225 signal. Lowick et al. (2012) used the approach of Huntley and Lamothe (2001) and these
226 average g_{2days} values for fading correction. Here we use the data of Lowick et al. (2012) for the
227 IRSL₅₀ signal measured in the post-IR IRSL₂₂₅ protocol, in order to test fading-correction using
228 the Huntley (2006) model. Rather than using the average g_{2days} value, we use a g_{2days} value of
229 2.65 ± 0.42 %/decade, determined for the IRSL₅₀ signal of sample GOS3 (Lowick et al., 2012
230 their Figure 2). As no value for sample GOS4 is reported in Lowick et al. (2012), we also use the
231 value for GOS3 for this sample.

232 *ZEL4 and ZEL7*

233 Polymineral fine-grained samples ZEL4 and ZEL7 were collected from the 'Marti' gravel
234 pit in the Luthern Valley of central Switzerland (Preusser et al., 2001). The samples are taken
235 from a succession of fine-grained fluvial deposits (Figure 5), which are bracketed between two
236 coarse-gravel units. A peat layer within these strata has been dated to 95 ± 3 ka using $^{230}\text{Th}/\text{U}$
237 TIMS (Geyh et al., 1997) and previous IRSL dating of these samples yielded a mean age of 96 ± 4
238 ka (Preusser et al., 2001; Lowick et al., 2012). Lowick et al. (2012) reported ages for samples
239 ZEL4 and ZEL7 of between 107 ± 9 ka and 292 ± 36 ka following fading-correction of IRSL_{50}
240 (measured in a post-IR IRSL_{225} protocol), post-IR IRSL_{225} and post-IR IRSL_{290} ages (using the
241 average $g_{2\text{days}}$ values described above). Here we also use the data of Lowick et al. (2012) for the
242 IRSL_{50} signal in order to test fading-correction using the Huntley (2006) model. A $g_{2\text{days}}$ value of
243 2.03 ± 0.93 %/decade was determined for this signal for sample ZEL7 (Lowick et al., 2012 their
244 Figure 2). As no value for sample ZEL4 is reported in Lowick et al. (2012) we also use the value
245 for ZEL7 for this sample.

246 *BRR-2*

247 Sample 205/BRR-2 was collected from Blackman Ridge Road ($46^{\circ}27'29''$ N, $118^{\circ}50'46''$
248 W), which comprises a 3.5 m exposure of the Palouse loess, Washington State, USA. The Mazama
249 tephra (modern Crater Lake, Oregon, USA) has been identified at this site previously (Sweeney,
250 pers. comm.), which is the most widespread Quaternary tephra on the Columbia Plateau
251 (Busacca et al., 1992), and has been dated to 7.6 ± 0.2 ka BP (ice-core derived calendrical age
252 from Zdanowicz et al., 1999). At this exposure, the Mazama tephra is diffuse and has been highly
253 diluted by large volumes of loessic material which make it barely visible in the roadside
254 exposure. Whilst diffuse the deposit is continuous and is interpreted as being in-situ. Sample
255 205/BRR-2 (0.85 m) underlies this diffuse tephra horizon by approximately 0.3 m (Figure 5). In
256 addition, a sample of the tephra horizon (BRRMAZ) was also collected for major, minor and
257 trace element chemical characterisation to confirm its assignment to the Mazama tephra.

258 *MFRB-1 and MFRB-2*

259 Samples 204/MFRB-1 and 204/MFRB-2 were taken from the McFeeley Road site
260 ($46^{\circ}18'54''$ N, $118^{\circ}27'10''$ W) of the Palouse loess deposits of Washington State, USA. The
261 section comprises a 3 m exposure containing a 30 cm thick tephra horizon which has been
262 assigned to Glacier Peak using single-shard major-element analysis (Spencer and Knapp, 2010)
263 and trace-element analyses (Kuehn et al., 2009). This tephra horizon has an age of 13.5 ± 0.1 cal.
264 ka BP (Kuehn et al., 2009); sample 204/MFRB-2 (0.55 m) was taken 0.10 m above the tephra
265 horizon, and sample 204/MFRB-1 (0.92 m) was taken 0.10 m below the tephra horizon (Figure

266 5). A sample of the tephra horizon (MFRGP) was also taken for minor, major and trace element
267 chemical characterisation.

268 *[Figure 5]*

269 **4.2.2 Tephra sample preparation, measurement & results**

270 The tephra samples from BRR and MFRB were prepared using standard methods (cf.
271 King et al., 2016c) in the Department of Geography and Earth Sciences at Aberystwyth
272 University. Samples were wet sieved to obtain the 180-212 μm grain size, before density
273 separation at 2.5 g cm^{-3} using sodium polytungstate to isolate the glass fraction from
274 contaminating minerals. The extracted shards were mounted in resin and polished using
275 increasingly fine polishing papers and suspensions ($\sim 25 \mu\text{m}$ to $0.3 \mu\text{m}$) and were finished using
276 $0.02 \mu\text{m}$ colloidal silica.

277 The major element chemistry of individual glass shards was measured using
278 wavelength-dispersive electron probe microanalysis (EPMA) at the Research Laboratory of
279 Archaeology and Art History at Oxford University. A JEOL 8600 electron microprobe with four
280 wavelength-dispersive spectrometers was used to analyse ten major elements (Na, Mg, Al, Si, P,
281 K, Ca, Ti, Mn and Fe). An accelerating voltage of 15 kV, a beam current of 6 nA and a defocussed
282 $10 \mu\text{m}$ beam were used for glass shard analyses to minimise Na migration. The instrument was
283 calibrated using a suite of mineral standards prior to analysis and secondary standards of
284 ATHO-G, StHs6/80-G and GOR132-G were measured throughout analysis to monitor the system
285 reproducibility and the precision and accuracy of glass analyses (secondary standard data are
286 listed in Supplementary Table S.11). All major element concentrations have been normalised to
287 an anhydrous basis.

288 Trace-elements were measured for the same glass shards using laser ablation
289 inductively-coupled plasma mass spectrometry (LA-ICP-MS) at the Department of Geography
290 and Earth Sciences, Aberystwyth University. The system comprises a Coherent Geolas 193 nm
291 Excimer laser with a Thermo Finnigan Element 2 high-resolution sector field mass
292 spectrometer (Pearce et al., 2007; 2011). Depending upon shard size, either a $10 \mu\text{m}$ or $20 \mu\text{m}$
293 diameter beam was used to ablate the shards at a laser energy of 10 J cm^{-2} , at a pulse rate of 5
294 Hz and with an acquisition time of 24 s. Trace element concentrations were calculated using the
295 method of Perkins and Pearce (1995) and Pearce et al. (2007). NIST SRM 612 silicate glass was
296 used as the calibration standard relative to concentrations from Pearce et al. (1997), and ATHO-
297 G reference glass (Jochum et al., 2006) was used as a secondary standard during each run (listed
298 in Supplementary Table S.12). ^{29}Si was used as an internal standard, determined from the same
299 shards using EPMA and normalised to an anhydrous basis.

300 Comparison of the tephra chemistry with previously published data (Westgate and
301 Evans, 1978; Busacca et al., 1992; Gaylord et al., 2001; Kuehn et al., 2009) using a similarity
302 coefficient (Borchardt et al., 1972) indicates that the horizon sampled at Blackman Ridge Road
303 (BRRMAZ) is the Mazama tephra, and also that the horizon sampled at McFeeley Road (MFRGP)
304 is the Glacier Peak tephra (Figure 6; Supplementary Tables S.9-12). Although material sampled
305 from MFRGP has previously been assigned to layer B of this tephra (Spencer and Knapp, 2010;
306 Kuehn et al., 2009), our sample has greater chemical similarity with layer G (Table S.12). This
307 may reflect sampling of a different part of this thick (>30 cm) horizon and does not affect this
308 horizon's use as a geochronological marker because the ages of layers B and G are
309 indistinguishable. Both the Mazama and Glacier Peak tephra horizons have robust age
310 estimations from independent dating techniques and provide ideal independent age control in
311 this investigation of fading-correction.

312 *[Figure 6].*

313 **4.2.3 Luminescence sample preparation & measurement**

314 Details of the preparation, luminescence and environmental dose rate measurement of
315 polymineral fine-grained samples GOS3, GOS4, ZEL4 and ZEL7 are given in Preusser (1999,
316 2001) and Lowick et al. (2012). Samples 205/BRR-2, 204/MFRB-1 and 204/MFRB-2 were
317 prepared using standard methods for polymineral fine grains at the Aberystwyth Luminescence
318 Research Laboratory. All samples were treated with a 10 % v.v. dilution of HCl to remove
319 carbonates, and 20 % H₂O₂ to remove organic material. The 4-11 µm grain size was isolated
320 using Stokes' Law, and settling in 0.01 N sodium oxalate. Samples were measured using 0.98 cm
321 diameter aluminium discs, and 1 mg of sample material was deposited from suspension in
322 acetone onto each disc. Luminescence measurements were done using a Risø TL/OSL reader
323 (Bøtter-Jensen et al., 2003) equipped with a 1.48 GBq ⁹⁰Sr/⁹⁰Y beta source and an EMI 9235QA
324 photomultiplier. The radiation dose rate of the reader was 0.08 Gy s⁻¹ and IR stimulation used
325 diodes emitting at 870 nm, with irradiance of ~135 mW cm⁻² when set at 90 %. The
326 thermocouple of the instrument was checked prior to measurement, and no deviation between
327 programmed and measured temperatures was recorded. Detection was restricted to the blue
328 region using a 4 mm Corning 7-59, a 3 mm Schott GG-400 and a 2 mm BG-39 filter, giving a
329 detection range of 400-500 nm. All measurements were made using a post-IR IRSL₂₂₅
330 measurement protocol which was selected using preheat plateau and dose-recovery preheat
331 plateau experiments (cf. Roberts, 2012). Signals were integrated over the first 4 s, and
332 backgrounds taken from the final 20 s of stimulation. All measured aliquots fulfilled the
333 acceptance criteria of 1) aliquot recycling within 10 % of unity; 2) maximum equivalent dose

334 uncertainty <10 %; 3) maximum test dose signal (T_x) uncertainty <10 %, 4) signal $>3\sigma$ about
335 background, 5) recuperation <5 %.

336 Fading was measured using a short-stimulation protocol (method 'b' of Huntley and
337 Lamothe, 2001), and aliquots were preconditioned through multiple dose stimulation cycles
338 prior to measurement. Aliquots were preheated immediately following dosing (Auclair et al.,
339 2003). The IRSL₅₀ measurements were not made within a post-IR IRSL protocol, but rather in a
340 protocol comprising only IRSL₅₀ stimulations, to facilitate measurement using a short optical
341 stimulation of 0.38 s. An additional 'témoins' aliquot was measured to provide a control for
342 signal loss due to successive signal bleaching (Huntley and Lamothe, 2001). The maximum
343 delay was 39 days, fading rates for all samples are listed in Table 2. Raw measurement data are
344 available in the Supplementary Material.

345 The environmental dose rate of the samples was calculated using DRAC v.1.2 (Durcan et
346 al., 2015) using a combination of in-situ gamma spectrometry, thick-source alpha and beta
347 counting (full details of the calculations are given in the Supplementary Material Tables S.13
348 and S.14). The conversion factors of Guérin et al. (2011) were used together with the beta grain
349 size attenuation factors of Mejdahl (1979) and alpha grain size attenuation factors of Bell
350 (1980). Water content was estimated at 10 ± 5 % for all samples and an alpha efficiency value of
351 0.0860 ± 0.0038 was assumed (Rees-Jones, 1995).

352 **4.2.4 Comparison with independent age control**

353 The IRSL₅₀ ages of all samples were fading-corrected using the Huntley (2006) model
354 implemented with both a single-saturating exponential (1EXP, Kars et al., 2008) and general
355 order kinetic fit (GOK, Guralnik et al., 2015a) as well as the approach of Huntley and Lamothe
356 (2001); note that the Huntley and Lamothe (2001) fading correction was only applied to D_e
357 values determined with a 1EXP fit. The details of these samples are summarised in Tables 2 and
358 3, and the results of the fading-correction relative to independent age control are shown in
359 Figure 5. It was not possible to run the Huntley et al. (2006) model with a GOK fit for samples
360 GOS4 or ZEL4 because the dose response curves are not sufficiently constrained; the
361 uncertainty of the kinetic order, α , of sample ZEL7 is extremely high for the same reason (Table
362 3). It is immediately apparent that for all samples, the Huntley and Lamothe (2001) approach
363 yields the youngest ages, whilst with the exception of GOS3, the Huntley (2006) model
364 implemented with 1EXP yields the oldest ages, although almost all of the ages are within 2σ
365 uncertainty. All three fading-correction approaches yield ages consistent with independent age
366 control for samples GOS4, 204/MFRB-1 and 205/BRR-2, whilst the 1EXP implementation of the
367 Huntley (2006) model overestimates age for sample 204/MFRB-2. However, whereas the

368 Huntley and Lamothe (2001) fading corrected ages are within 2σ uncertainties of independent
369 ages for samples GOS3 and ZEL4, the Huntley (2006) fading corrected ages overestimate the
370 independent age of these samples. The greatest age discrepancy is recorded for sample ZEL7,
371 however the exact location of this sample relative to the independent age control is unclear
372 (Figure 5).

373 It is promising that the performance of the Huntley (2006) model using either the 1EXP
374 or GOK approach is similar to that of the Huntley and Lamothe (2001) model, although the
375 lower ages obtained using the latter approach may relate to its application beyond the linear
376 part of the dose response curve. However, the age discrepancies between the 1EXP and GOK
377 implementations of the Huntley (2006) model are concerning. A potential explanation for this
378 may relate to constraint of dose response during laboratory measurements, which especially for
379 younger samples may not characterise the complete dose response curve. Applying the Huntley
380 (2006) model involves interpolating the measured natural luminescence signal (L_{nat}) onto a
381 simulated natural dose response curve (Kars et al., 2008) calculated using the unfaded D_0 value,
382 fading rate and kinetic order (α), if a GOK fit is used. To explore the effect of changing dose
383 response curve constraint on calculated age, we used the measurement data of samples
384 204/MFRB-1, 204/MFRB-2 and 205/BRR-2, which were dosed until saturation (i.e. using a
385 maximum regenerative dose of 4.7 kGy). Starting with a dataset that allowed interpolation of
386 L_{nat} between at least two regenerative dose points, we progressively re-fitted the data including
387 additional regenerative doses of 147 Gy, 295 Gy, 590 Gy, 2358 Gy and 4717 Gy with the
388 `calc_Huntley2006()` function using either a 1EXP or GOK fit to calculate age (Figure 7).

389 All samples exhibited similar behaviour; in contrast to the 1EXP fit, 590 Gy was the
390 lowest, maximum regenerative dose value that could be fitted using a GOK fit for these samples.
391 With increasing regenerative doses, D_e values determined using a 1EXP fit increased by 13-16%
392 (i.e. 7 Gy for sample MFRB2), whereas those determined using a GOK fit increased by only 2-4%
393 (Figure 7A). The characteristic dose of saturation (D_0) increased by 50-60% with the inclusion
394 of greater regenerative doses for 1EXP, but reduced by 35-64% for the GOK fit (Figure 7B).
395 Estimation of $(n/N)_{ss}$ reduced by only 2-4% for either fit, whereas (n/N) values reduced by 43-
396 52% for 1EXP and increased by 35-60% for the GOK fitting (Figure 7C). Huntley (2006) model
397 fading corrected ages calculated with 1EXP increased by 20-24% (i.e. 4 ka for sample
398 204/MFRB-2), whereas those calculated with GOK exhibited a reduction of 2%. The Huntley and
399 Lamothe (2001) model fading corrected ages, calculated using D_e values determined from a
400 1EXP fit, exhibited an increase of 13-16% following the increase in D_e values (Figure 7A, Figure
401 7D). In contrast where D_e values determined from a GOK fit were used, Huntley and Lamothe
402 (2001) model fading corrected ages increased by only 2-4% (Figure 7A, Figure 7D).

403 Sample 204/MFRB-2 has the most robust independent age control, as it directly overlies
404 the Glacier Peak tephra, which has known depositional age of 13.5 ± 0.1 cal. ka BP (Kuehn et al.,
405 2009) and we focus on this sample to determine the implications of our results. Whereas an age
406 overestimation is obtained when all of the regenerative data are fitted with the Huntley (2006)
407 model and 1EXP (17.1 ± 1.4 ka), fewer regenerative doses yield ages within uncertainty of the
408 independent age (Figure 7D), however a clear trend of increasing age with increasing
409 regenerative dose is apparent. In contrast the Huntley (2006) and GOK fading corrected ages
410 are commensurate with the independent age control for all regenerative dose combinations,
411 reducing by 2% with increasing regenerative dose (Figure 7D). The key difference between the
412 two implementations of the Huntley (2006) model is the form of the dose response curve that
413 L_{nat} is interpolated on.

414 Kars et al. (2008) assumed that the luminescence dose response of feldspar followed a
415 single saturating exponential, and this implementation has been used in a number of studies
416 (e.g. King et al., 2016a,b; Li et al., 2017). However for some samples, feldspar luminescence dose
417 response can be better fitted using a GOK (e.g. Guralnik et al., 2015a,b; Li et al., 2018) or double
418 saturating exponential function (e.g. Buylaert et al., 2012; Li et al., 2015), which is also true for
419 the three samples investigated here (e.g. 204/MFRB-2, Figure 8A). Whereas either a GOK fit or a
420 double exponential fit passes through all of the data-points, the 1EXP fit exhibits clear bias,
421 overestimating dose at low doses and underestimating dose at high doses (Figure 8B). The
422 improved fit of a double exponential or GOK curve is clearly demonstrated when the impact of
423 fitting increasingly high dose, dose response data is explored (Figure 7A). In contrast to the
424 16% variation in D_e values recorded for the single saturating exponential fit, the D_e values
425 obtained from the double saturating exponential fit or GOK fit vary by only 0-4% (Figure 7A).
426 Thus the Huntley (2006) and 1EXP approach seems to be affected by interpolation of the
427 natural signal (L_{nat}) onto a simulated natural dose response curve of inappropriate form.

428 The investigations presented here indicate that the Huntley (2006) model implemented
429 with a GOK fit is most appropriate for sample fading correction, and this approach has been
430 used previously (Guralnik et al., 2015b; Valla et al., 2016; Lambert et al., In Revision) however a
431 number of caveats remain for its use. First it is only possible to fit well characterised dose
432 response curves, as demonstrated by the model failure to determine finite ages for samples
433 GOS4 and ZEL4. Whilst it was possible to fit sample GOS3, which has a maximum regenerative
434 dose of only 210 Gy, it was not possible to fit the dose response data of a number of samples
435 from Valla et al. (2016) where the maximum regenerative dose was 2.5 kGy, which is partly
436 related to the very high rates of fading for these samples. Samples 204/MFRB-1, 204/MFRB-2
437 and 205/BRR-2 could only be fitted where a maximum regenerative dose of ~ 600 Gy or greater
438 was used, and thus we tentatively propose this as a minimum threshold, but note that this will

439 be sample specific. Secondly the GOK implementation of the Huntley et al. (2006) model yielded
440 less accurate (n/N) values for samples known to be in field saturation (Figure 4) than the 1EXP
441 model implementation. Little difference in $(n/N)_{ss}$ was recorded between the two fitting
442 methods (Figure 7C), and this may indicate that the GOK implementation performs less well for
443 samples that interpolate further along the dose response curve, however we are unable to test
444 this within the present study. Finally, and related to the former point, it is unknown whether
445 laboratory feldspar dose response always mimics the form of natural dose response. Previous
446 studies on the luminescence and electron spin resonance of quartz have revealed markedly
447 different laboratory behaviour in comparison to naturally accumulated signals (Chapot et al.,
448 2012; Timar-Gabor et al., 2015; Tsukamoto et al., In Revision), and it seems reasonable that
449 some feldspar may behave similarly. However, Li and Li (2012) constructed a natural
450 luminescence dose response curve for K-feldspar extracted from loess at Luochuan, China.
451 Using the independent age control at this site, they found that natural dose response for
452 multiple-elevated temperature luminescence measurements followed a single saturating
453 exponential. Furthermore for these samples laboratory generated dose response curves also
454 exhibited similar form. Further studies on feldspar of different origins and chemical
455 compositions are required to confirm whether laboratory dose response always mimics natural
456 dose response, and also whether this is true for all measurement protocols. Despite these
457 limitations, the performance of the Huntley (2006) model implemented with GOK seems to be
458 appropriate for feldspar fading age correction, whilst using a 1EXP fit may result in a slight bias
459 towards age overestimation (Figure 7); we also note that other curve forms may yield accurate
460 fading corrected ages, providing that they fit the measured data adequately.

461

462 *[Figure 8]*463 **5. Threshold values: What does $2D_0$ mean for feldspar?**

464 Wintle and Murray (2006) proposed an upper D_e interpolation value of $2D_0$ for the
465 derivation of finite age estimates from quartz. Beyond this threshold, which is equivalent to 86
466 % of saturation, interpolation of L_{nat} results in a large uncertainty because of the relatively small
467 rate of change (per unit dose) in the dose response curve. In contrast, below this threshold,
468 luminescence signals are often regarded as finite. However the validity of applying this
469 threshold to feldspar data remains questionable unless anomalous fading has been corrected
470 for, because fading can cause a saturated sample to have $D_e < 2D_0$ resulting in misidentification
471 of a saturated sample, as a sample of finite age.

472 For a non-fading sample, the laboratory and natural dose response curves are
 473 considered to be equivalent (although see for example the discussion in Chapot et al., 2012). In
 474 contrast, for a sample which is affected by time-dependent athermal signal loss (i.e. anomalous
 475 fading) the laboratory dose response and natural dose response curves are not equivalent,
 476 because of the different time periods over which dose accumulates; this is shown in Figure 3.
 477 Because the laboratory dose response curve accumulates over a very short time period a much
 478 greater amount of charge can be stored and the dose response curve is able to grow to a greater
 479 level of saturation than would be possible in nature. In contrast, the natural dose response
 480 curve for the IRSL₅₀ signal of sample NB124 (as simulated using the `calc_Huntley2006()`
 481 function) reaches a much lower level of saturation, with $(n/N)_{ss}$ of $38 \pm 5 \%$ (Figure 3). The
 482 difference between the laboratory and natural dose response curves is greatest for the samples
 483 that fade most, and thus post-IR IRSL₂₂₅ and post-IRSL₂₉₀ signals, which fade least, may not be
 484 affected. The consequence of this mismatch between the laboratory and natural dose response
 485 curves means that the notion of $D_e < 2D_0$ as a threshold for feldspar signal saturation is not
 486 applicable to feldspars which experience anomalous fading, unless this fading is corrected.

487 To illustrate this effect graphically, the equivalent of the laboratory-measured
 488 saturation ratio $(L_{nat})/(L_{labmax})$ for a continuum of synthetic saturated samples was computed
 489 using the Huntley (2006) model with 1EXP for g_{2days} values ranging from 0 % to 30 %/decade
 490 (Figure 9). We chose to use 1EXP rather than GOK because of the improved performance of
 491 1EXP in Figure 4, but both fits would yield similar results. This calculation comprised three
 492 stages: (i) $(n/N)_{ss}$ was computed based on an unfaded D_0 of 400 Gy, \dot{D} of 5 Gy ka⁻¹, and ρ' values
 493 of 0 to 1.65×10^{-5} (approximately equal to g_{2days} of 0 % to 30 %/decade). (ii) Fading over
 494 laboratory timescales means that the maximum measured light intensity (L_{labmax}) is lower than
 495 would be measured for a non-fading sample. This can be seen through the difference between
 496 the measured and simulated unfaded dose response curves in Figure 3. The level of laboratory
 497 saturation (N_{lab}/N) achievable for a given sample can be calculated using the same approach as
 498 in stage (i), but substituting the environmental dose rate for \dot{D}_{lab} , which in this calculation was
 499 set to 0.134 Gy s⁻¹. (iii) Once (N_{lab}/N) has been calculated, it is possible to derive the equivalent
 500 laboratory measured saturation ratio, $(L_{nat})/(L_{labmax})$ where L_{nat} is the sensitivity corrected
 501 natural luminescence signal and L_{labmax} is the maximum light level measured in the laboratory,
 502 equivalent to the maximum possible L_x/T_x measurement for that specific sample under the
 503 same experimental conditions. $(L_{nat})/(L_{labmax})$ can thus be derived from $1/(N_{lab}/N) * (n/N)_{ss}$.

504 The modelled relationship between $(L_{nat})/(L_{labmax})$ and g_{2days} is validated through
 505 comparison with the previously published $(L_{nat})/(L_{labmax})$ values (Table 1) for 50 published
 506 samples reported to be in field saturation, across a range of different signals (IRSL₅₀, post-IR

507 IRSL_{100/150/225/290}) (Figure 9). It is apparent that fading rate must be considered when evaluating
508 whether a sample is in saturation (cf. Valla et al., 2016). The importance of this can be
509 particularly illustrated through considering that a sample with g_{2days} of 3.77 %/decade in field
510 saturation has $(L_{nat})/(L_{labmax})$ that interpolates onto the laboratory dose response curve at $1D_0$
511 (Figure 9). Applying the $2D_0$ criterion to this sample would result in it being mistakenly
512 interpreted as having a finite age.

513 [Figure 9]

514 5. Conclusions

515 Anomalous fading remains challenging for luminescence dating of feldspars. Using a new **R**
516 function that is freely available in the 'Luminescence' package (v.0.8.1) and through comparison
517 of a suite of samples with independent age control from radiocarbon dating, ²³⁰Th/U TIMS and
518 tephrochronology, we show that the model of Huntley (2006) implemented with a general
519 order kinetic fit (Guralnik et al., 2015a) is appropriate for sample fading correction.
520 Implementing the Huntley (2006) model with a single saturating exponential fit following Kars
521 et al. (2008) results in age overestimation for the samples investigated in this study, because
522 their luminescence dose response is not well described by this form. If a general order kinetic fit
523 is used, sample dose response curves must include a maximum regenerative dose that is greater
524 than ~600 Gy, otherwise a robust fit is not obtained. We also tested the performance of the
525 Huntley (2006) model for determining sample saturation through applying it to a set of
526 saturated samples from literature, the model implemented with a single saturating exponential
527 fit performed best, yielding an accuracy of ~10 %. In contrast using a general order kinetic fit
528 performed less well, possibly indicating that feldspar do not follow this form of natural dose
529 response in nature. Further research is required to investigate the form of feldspar natural dose
530 response in comparison to that obtained using laboratory measurements.

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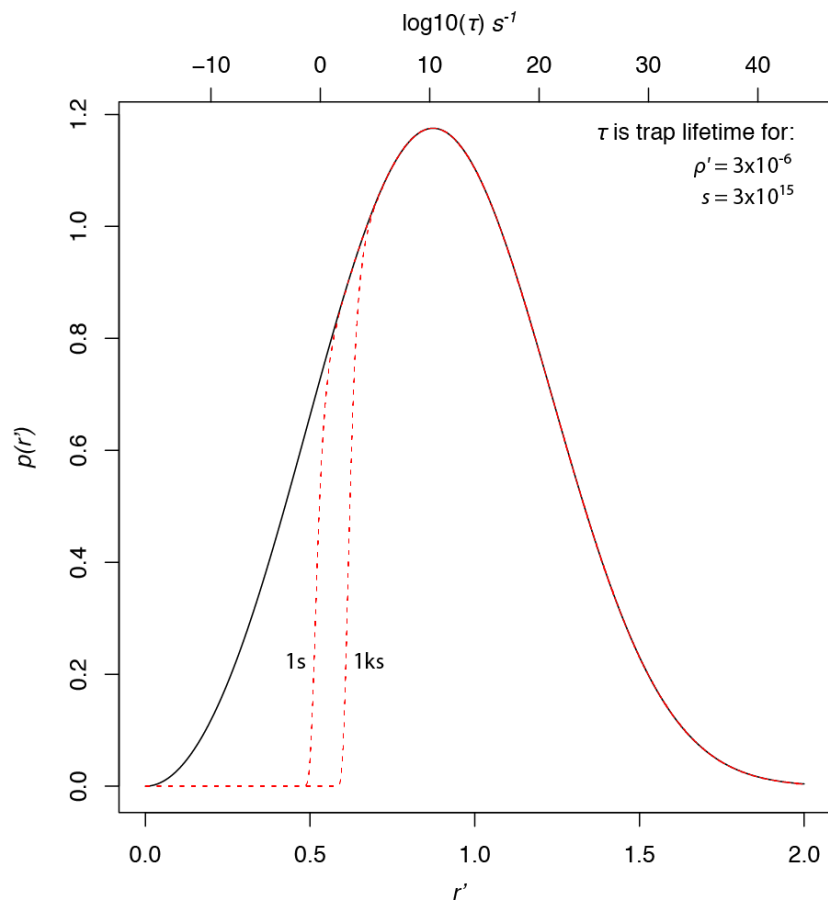
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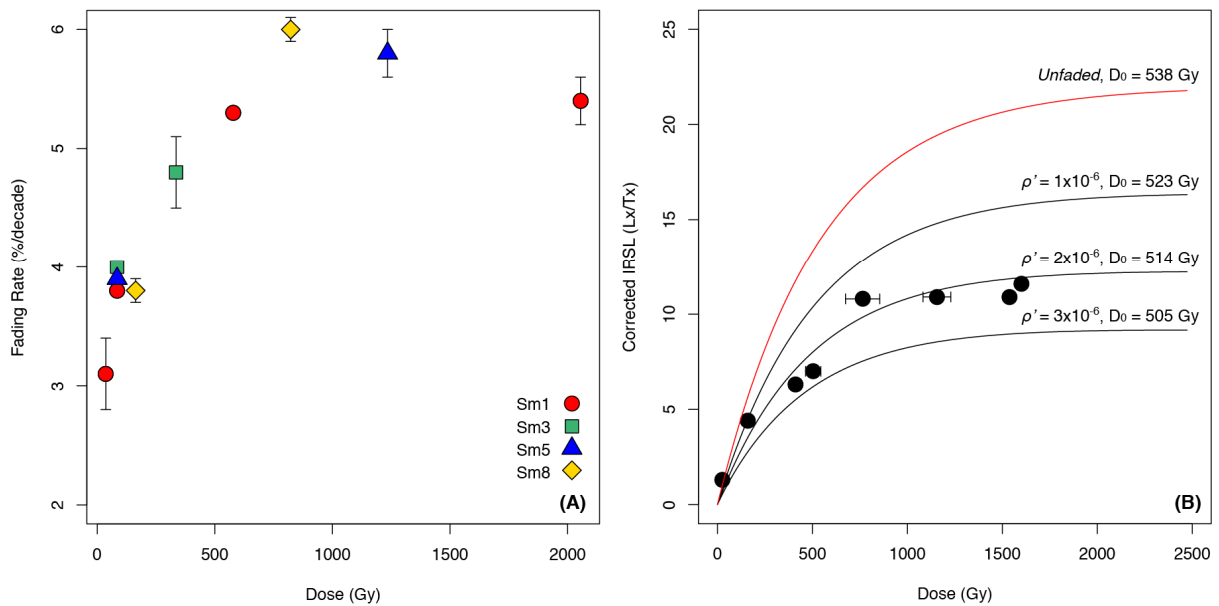
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713 **Figure 1:** Probability distribution ($p(r')$) of dimensionless distances (r') between trapped
 714 electrons and recombination centres following Huntley (2006) for a given dimensionless
 715 density of recombination centres ($\rho' = 3 \times 10^{-6}$) after an instantaneous pulse of irradiation. The
 716 lifetime (τ) of an electron in a given trap increases with increasing distance between the trap
 717 and the recombination centre, thus trapped charge with nearby recombination centres (left-
 718 side of the figure) are unstable over laboratory timescales. The dashed red lines indicate the
 719 remaining trapped electron population after 1 s and 1 ks, i.e. following recombination of
 720 electrons in the most unstable traps.

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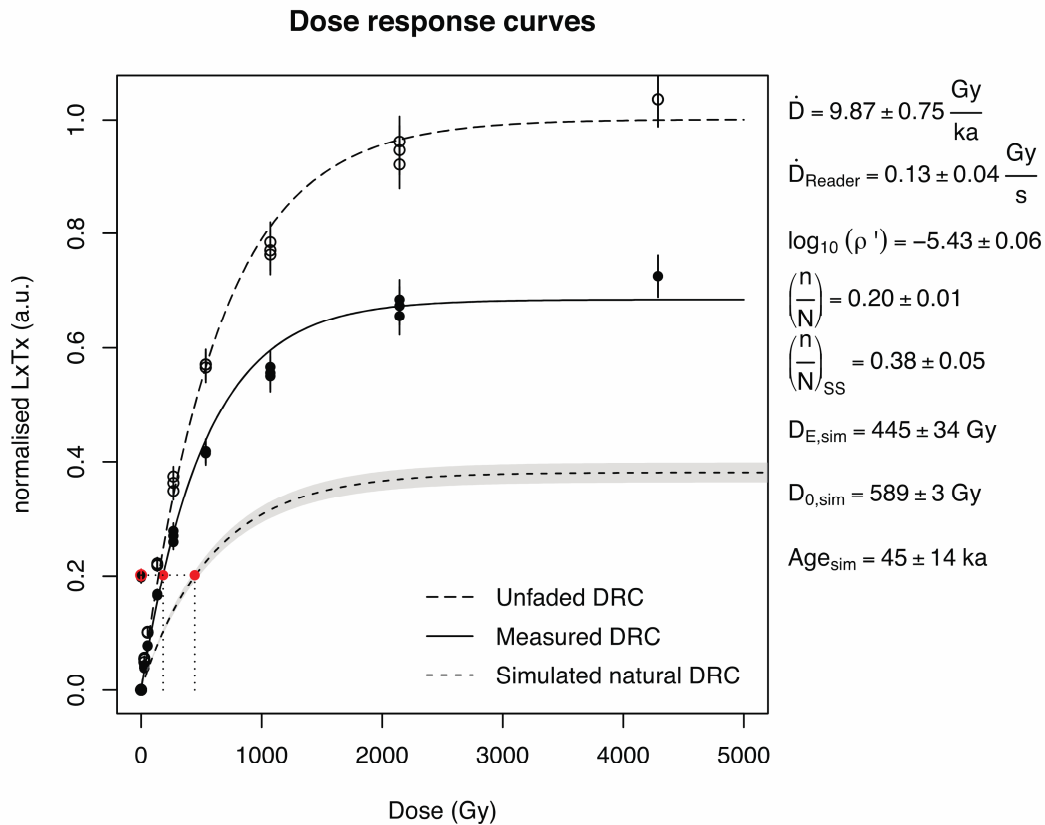


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723 **Figure 2:** (A) Fading rate as a function of given dose, redrawn from Li and Li (2008, their Figure
 724 11). (B) Natural luminescence signal intensities plotted against estimated palaeodose,
 725 contrasted with modelled dose response curves for different rates of athermal signal loss,
 726 redrawn from Li and Li (2008, their Figure 12a). Modelled dose response curves were
 727 calculated using an average environmental dose rate of 3.9 Gy/ka and equation: $\frac{Lx}{Tx}(t) =$

728 $e^{-\rho' \ln(1.85t)^3} A (1 - e^{-\frac{Dt}{D_0}})$ (King et al., 2016a) where A is a constant scaling factor of 22, to
 729 match the original figure (Li and Li, 2008). Whereas the data in (A) suggest that the rate of
 730 sample fading will vary significantly with dose, in (B) it is apparent that the data are well
 731 described by a single saturating dose response curve computed based on a single density of
 732 recombination centres using the model of Huntley (2006).

733



734

735 **Figure 3:** Laboratory measured, simulated unfaded, and simulated natural dose response
 736 curves for the IRSL_{50} signal of sample UNIL/NB124 (King et al., 2016a) output from the
 737 `calc_Huntley2006()` function of the **R** package ‘Luminescence’ (Kreutzer et al., 2012; 2018)
 738 fitted with a single-saturating exponential fit (following Kars et al., 2008). The simulated
 739 unfaded dose response curve is calculated using equation (1) after Kars et al. (2008), whereas
 740 the simulated natural dose response curve is calculated using the sample specific \dot{D} : 9.87 ± 0.75
 741 Gy ka^{-1} , $\log_{10}(\rho')$: -5.43 ± 0.06 and unfaded D_0 : $603 \pm 12 \text{ Gy}$. Note that the latter two parameters
 742 have been recalculated relative to the original publication using the
 743 `analyse_FadingMeasurement()` (Kreutzer and Burow, 2017) and
 744 `calc_Huntley2006()` functions respectively.

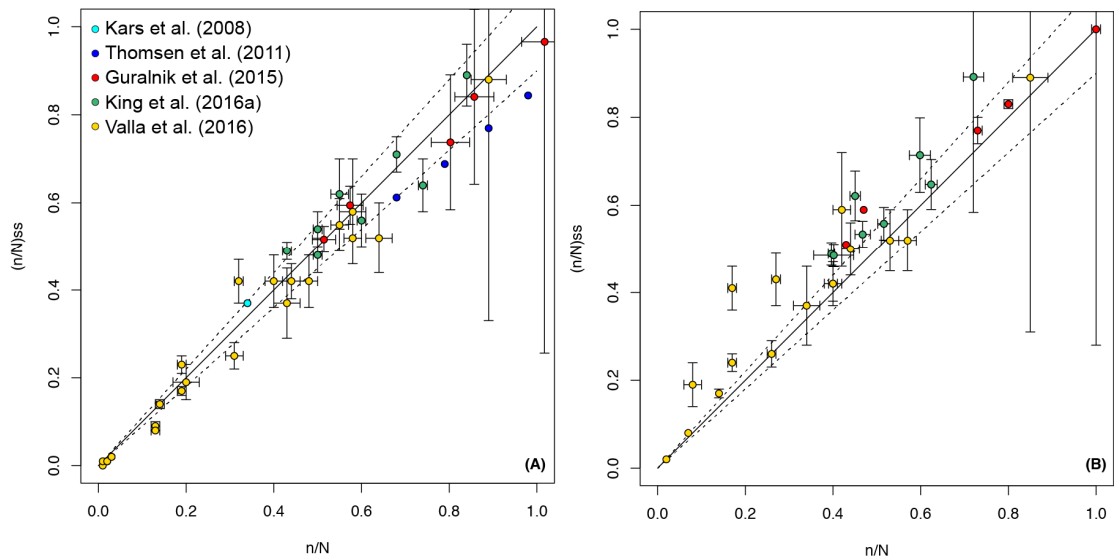


Figure 4: Comparison of (n/N) and $(n/N)_{ss}$ for published saturated samples (data summarised in Table 1) fitted with (A) a single-saturating exponential fit (1EXP, after Kars et al., 2008) and (B) a general order kinetic fit (GOK, after Guralnik et al., 2015a). A sample in saturation should have $(n/N) = (n/N)_{ss}$ and if the model performs perfectly, will sit on the 1:1 line (solid line). The data from Kars et al. (2008) and Thomsen et al. (2011) are not included in plot (B) as the raw data were not available for fitting with a GOK fit. Some of the data from Valla et al. (2016) could not be fitted robustly with a GOK fit and are not included in plot (B) (see Table 1). It is apparent that the data are less scattered when fitted with 1EXP rather than GOK which for the data of Valla et al. (2016) may relate to incomplete characterisation of the full feldspar dose response (maximum dose for some samples ~ 2.5 kGy). Although the data are scattered, for fitting with 1EXP no systematic under or overestimation of $(n/N)_{ss}$ is recorded. The majority of the data are within 10 % of the 1:1 line (dashed lines) indicating that the model has c. 10 % uncertainty when applied with 1EXP.

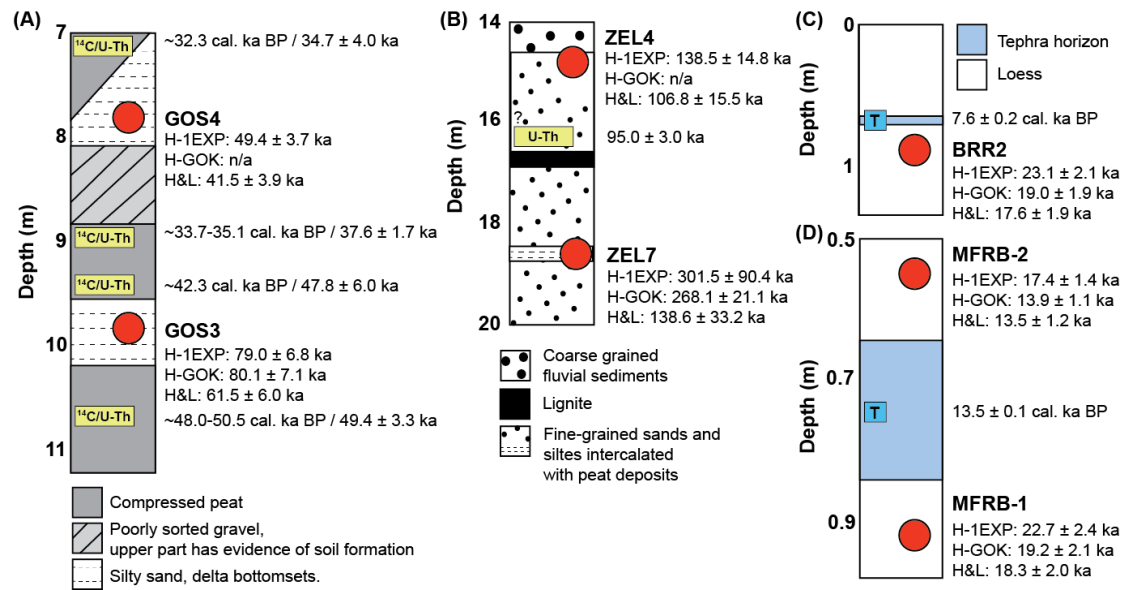


Figure 5: Approximate sample stratigraphic location and independent age control. (A) Sample locations of GOS3 and GOS4 were taken from Preusser (1999) with independent age control from Schlüchter et al. (1987) and Geyh and Schlüchter (1998). (B) Sample locations of ZEL4 and ZEL7 were taken from Preusser et al. (2001) with independent age control from Geyh et al. (1997) as described in Lowick et al. (2012). It was not possible to determine the exact location of the U/Th age at this site relative to the samples, as it is simply described as from the same unit (Lowick et al., 2012). (C) At Blackman Ridge Road (location of sample 205/BRR2) the tephra horizon is correlated to the Mazama tephra (see Figure 6); the reported age is from Adams (1990). (D) At McFeeley Road (location of samples 204/MFRB-1 and 204/MFRB-2) the tephra horizon is correlated to the Glacier Peak tephra (see Figure 6); the reported age is from Kuehn et al. (2009). H&L = Huntley and Lamothe (2001), H-1EXP = Huntley (2006) implemented with a single saturating exponential following Kars et al. (2008), H-GOK = Huntley (2006) implemented with a general order kinetic model following Guralnik et al. (2015a).

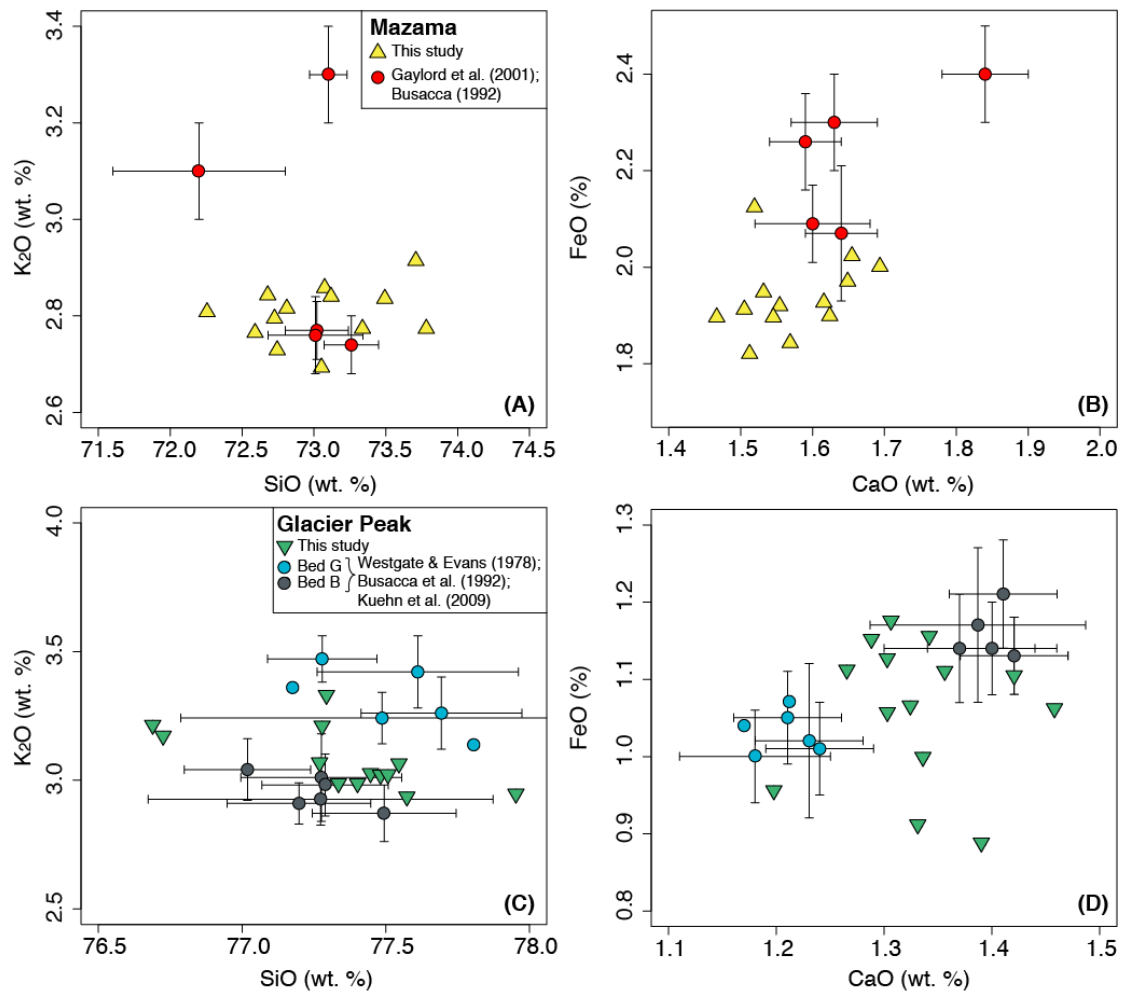


Figure 6: Comparison of tephra major element chemical data from this study, relative to previous studies (A,B) for sample BRRMAZ from Blackman Ridge Road and (C,D) for population 3 of sample MFRGP from McFeeley Road. Uncertainties are shown at 1σ for previously published data. Full chemical data are given in Supplementary Tables S.9-12.

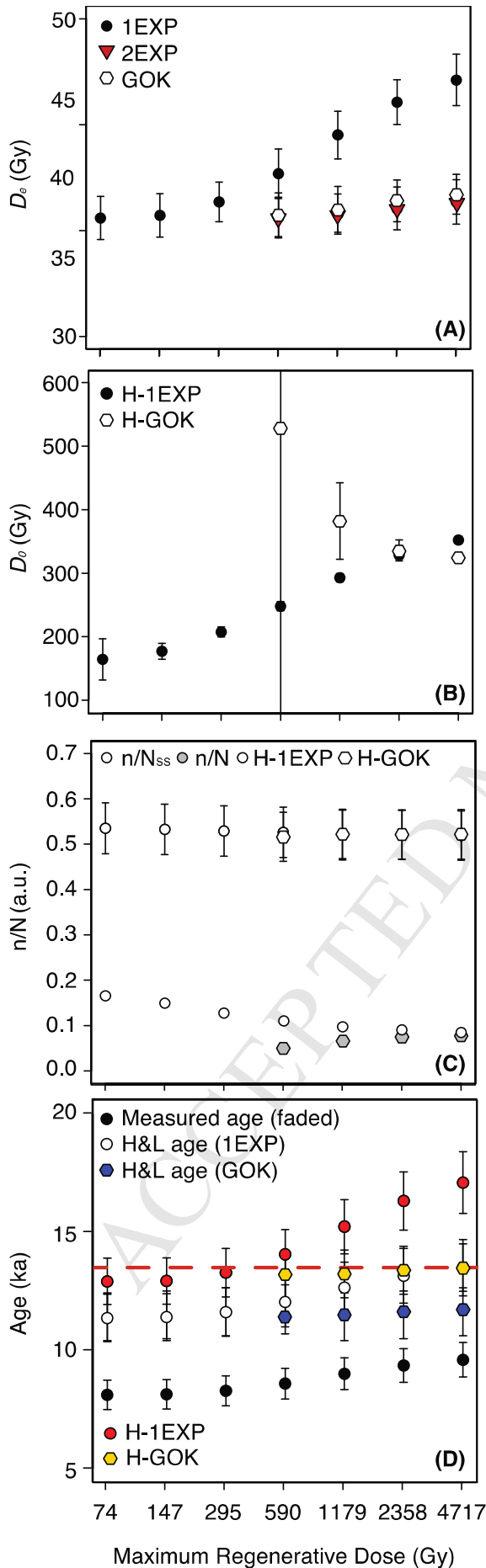


Figure 7: Effect on fitting of including additional regenerative dose points for representative sample 204/MFRB-2. (A) Changing D_e values fitted using a single or double (2EXP) saturating exponential fit, or a general order kinetic (GOK) fit using the `plot_GrowthCurve()` function (Kreutzer et al., 2012; 2018). No data are shown for the 2EXP or GOK fits for maximum regenerative doses below 590 Gy as the dose response curve is not sufficiently characterised for fitting. (B) Changing D_0 values for the 1EXP and GOK fits. (C) Changing $(n/N)_{ss}$ and (n/N) calculated using either the Huntley (2006) model after Kars et al. (2008) i.e. assuming a 1EXP fit or after Guralnik et al. (2015a,b) assuming a GOK fit. (D) Changing measured age (fitted with 1EXP), and fading corrected ages determined using the `calc_Huntley2006()` function and the Huntley and Lamothe (2001) model using the `calc_FadingCorr()` function (Kreutzer et al., 2012; 2018). The independent age control in the form of the underlying Glacier Peak tephra (Kuehn et al., 2009) is given as a red-dashed line which the luminescence ages should be younger than.

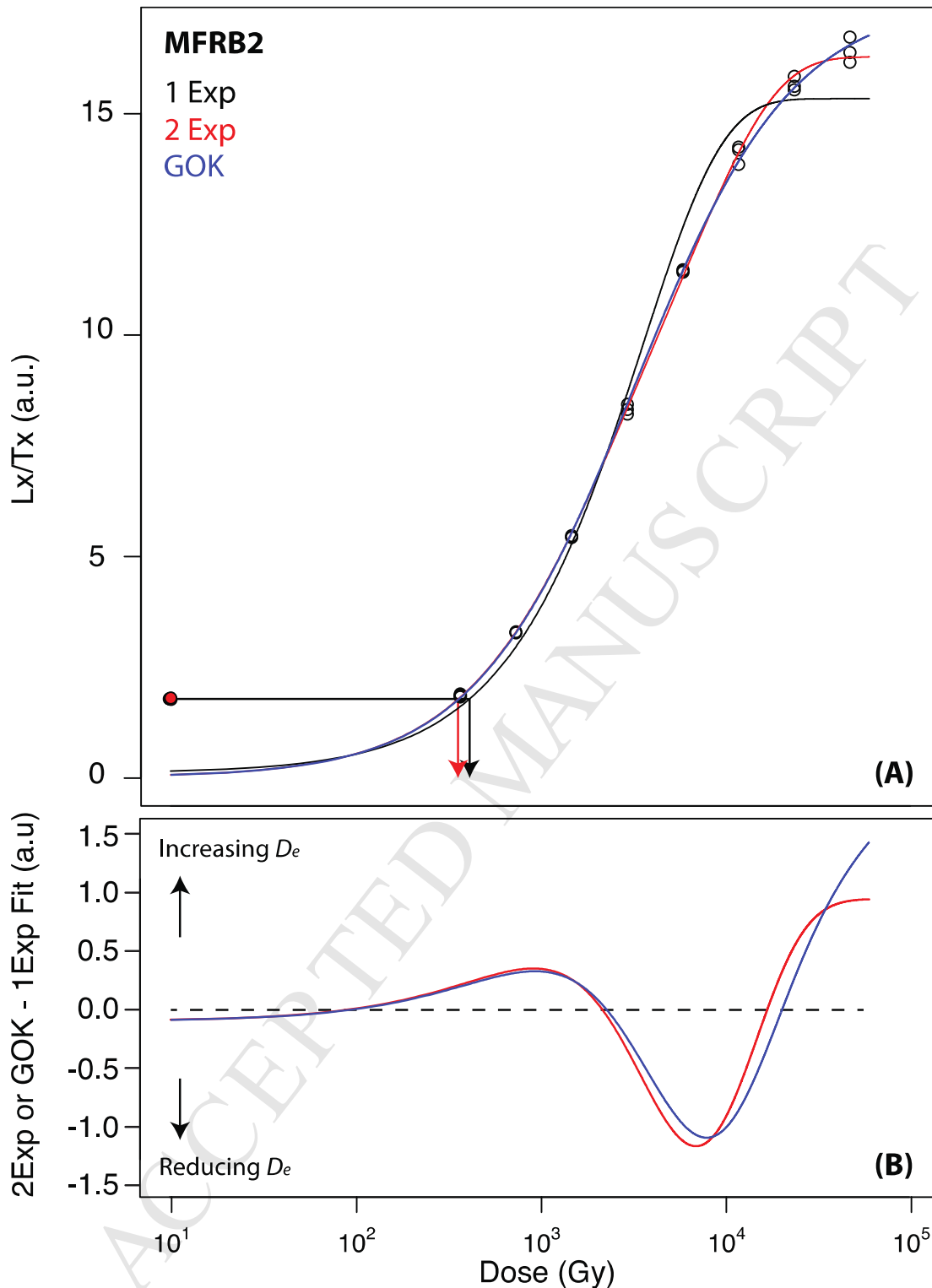


Figure 8: (A) Luminescence dose response curve of representative sample 204/MFRB-2 fitted with a single (1EXP) and double (2EXP) saturating exponential fit, and a general order kinetic (GOK) fit. Arrows show how interpolation of the same L_{nat} measurement yields different equivalent dose values depending on fit (note that interpolation of L_{nat} onto the 2EXP or GOK fit is indistinguishable). (B) Deviation of the 1EXP fit relative to the 2EXP and GOK fits.

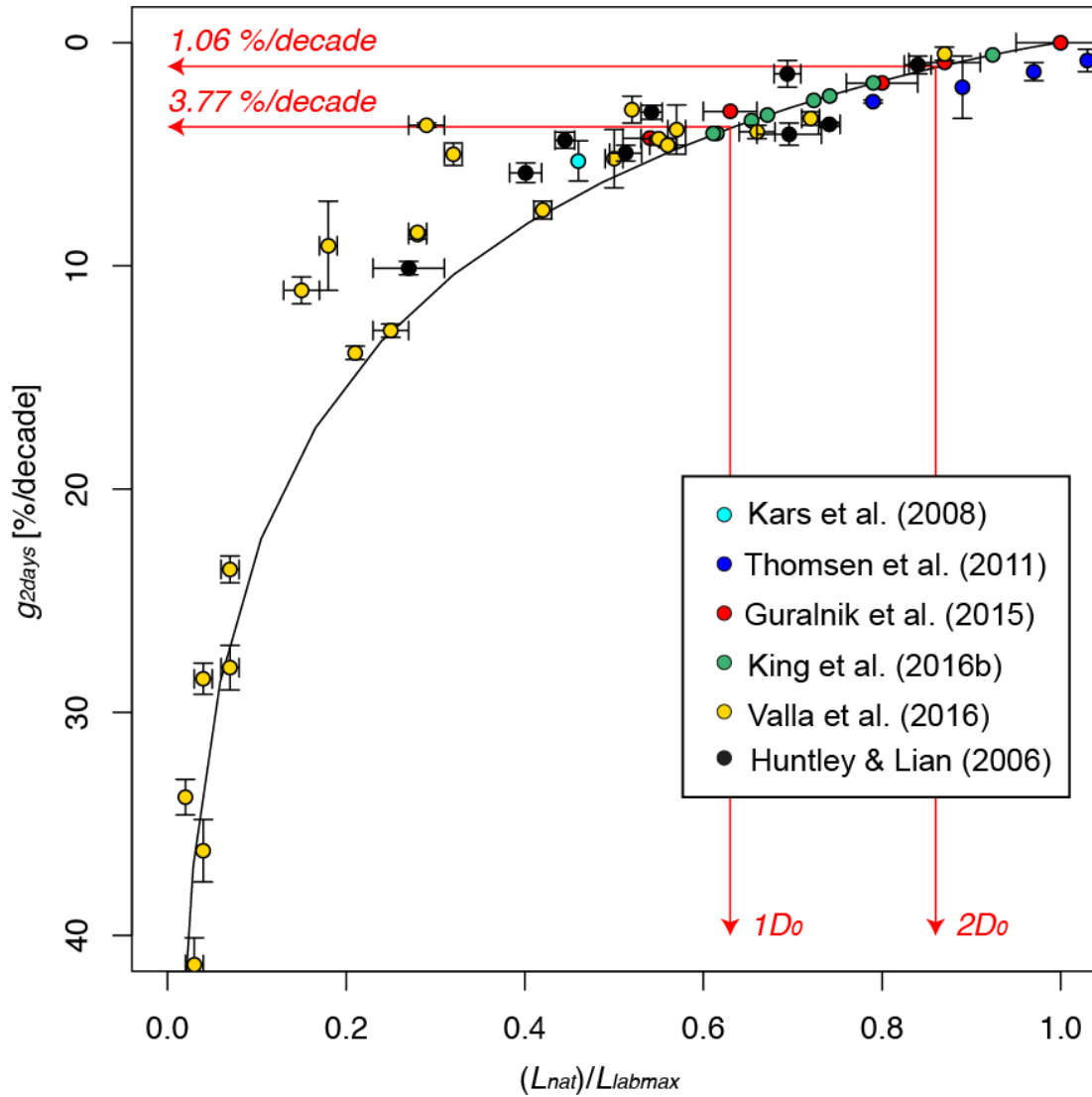


Figure 9: Modelled (line) and measured (datapoints, Table 1) $(L_{nat})/(L_{labmax})$ values for 50 samples in field saturation reported in Huntley and Lian (2006), Thomsen et al. (2011), Guralnik et al. (2015b), Valla et al. (2016) and King et al. (2016b). Modelled values are calculated assuming \dot{D} of 5 Gy ka⁻¹, D_0 of 400 Gy and a laboratory dose rate \dot{D}_{lab} of 0.134 Gy s⁻¹ using the model of Huntley (2006) implemented with a single-saturating exponential fit. The calculation is described in the main text. The red lines relate to $(L_{nat})/(L_{labmax})$ values equivalent to 1 and 2 D_0 which would be measured for saturated samples with g_{2days} of 3.77 and 1.06 %/decade respectively. Some of the scatter in the data of Valla et al. (2016) may be related to samples not being dosed completely to saturation, which could result in L_{labmax} being underestimated.

Table 1: Sample properties.

Sample Name	Description	Independent Age Control	Signal	\dot{D} (Gy ka ⁻¹)	\dot{D}_{lab} (Gy s ⁻¹)	g_{2days} (%/decade)	$\log_{10}(\rho^*)$	L_n/L_{labmax}	D_0 (Gy)	(n/N)	(n/N) _{ss}	N_{lab}/N	Pred. L_n/L_{labmax}	D_0	α	(n/N)	(n/N) _{ss}
<i>Thomsen et al. (2008)</i>										1EXP			GOK				
62213	Coarse grain, K-rich feldspar	Pliocene Marine Level, ~3.6 Ma	IRSL50 (Early background, post-IR protocol)	3.6	0.14	2.64 ± 0.07	1.86E-06*	0.79	425	0.68#	0.61#	0.86#	0.71#	-	-	-	-
			IRSL50 (Late background, post-IR protocol)	3.6	0.14	2.00 ± 1.40	1.38E-06*	0.89	280	0.80#	0.69#	0.89#	0.78#	-	-	-	-
			post-IR IRSL290 (Early background)	3.6	0.14	1.30 ± 0.40	8.71E-07*	0.97	610	0.90#	0.79#	0.93#	0.85#	-	-	-	-
			post-IR IRSL290 (Late background)	3.6	0.14	0.80 ± 0.50	5.13E-07*	1.03	660	0.99#	0.87#	0.96#	0.91#	-	-	-	-
<i>Kars et al. (2008)</i>																	
NCL-4406042 to 4406047	Coarse grain, K-rich feldspar	Late Pliocene - Early Pleistocene, ~1-3 Ma	IRSL50	2.5	0.15	5.30 ± 0.90	3.60E-06 ± 0.50E-06	0.46	529	0.34	0.37	0.74	0.50	-	-	-	-
<i>Guralnik et al. (2015)</i>																	
19B	Coarse grain, Na-rich feldspar	Temperature <= 33 ± 2 °C	IRSL50	1.50	0.153 to 0.180	1.68 ± 0.35	1.06E-06 ± 2.54E-07	0.80 ± 0.04	185 ± 12	0.80 ± 0.01	0.74 ± 0.01	0.91	0.82	170 ± 8	1.81 ± 0.56	0.73 ± 0.01	0.77 ± 0.03
28B				1.03	0.153 to 0.180	0.01 ± 0.12	-4.67E-09 ± 1.43E-07	1.00 ± 0.05	89 ± 11	1.02 ± 0.01	1.00 ± 0.74	1.00	1.00	80 ± 10	1.33 ± 1.36	1.00 ± 0.01	1.00 ± 0.72
105B				2.92	0.180	1.06 ± 0.21	6.93E-07 ± 1.54E-07	0.87 ± 0.04	193 ± 15	0.86 ± 0.01	0.83 ± 0.01	0.95	0.98	175 ± 11	1.71 ± 0.68	0.80 ± 0.01	0.83 ± 0.01
146A				2.84	0.153 to 0.180	3.66 ± 0.18	2.49E-06 ± 1.41E-07	0.54 ± 0.03	267 ± 21	0.51 ± 0.01	0.51 ± 0.00	0.80	0.58	261 ± 22	2.14 ± 0.99	0.43 ± 0.00	0.51 ± 0.00
218A				2.96	0.153 to 0.250	2.84 ± 0.18	1.95E-06 ± 1.66E-07	0.63 ± 0.03	266 ± 23	0.57 ± 0.01	0.58 ± 0.00	0.85	0.65	260 ± 59	2.25 ± 2.24	0.47 ± 0.00	0.59 ± 0.00
<i>King et al. (2016)</i>																	
UNIL/NB19	Coarse grain, K-rich feldspar	Paired sample NBk-36-23 has an Apatite Helium age of 1.79 ± 0.06 Ma, a Zircon Helium age of 2.48 ± 0.09 Ma and an Argon-argon in Biotite age of 4.12 ± 0.1 Ma (Zeitler et al., 2014).	IRSL50 (MET)	6.62 ± 0.56	0.136	3.97 ± 0.19	2.68E-06 ± 1.43E-07	0.61%	539 ± 17	0.43 ± 0.01	0.49 ± 0.03	0.79	0.62	517 ± 18	1.75 ± 0.42	0.40 ± 0.01	0.49 ± 0.03
UNIL/NB19			post-IR IRSL100 (MET)	6.62 ± 0.56	0.136	2.56 ± 0.21	1.76E-06 ± 1.37E-07	0.72%	661 ± 23	0.55 ± 0.01	0.62 ± 0.05	0.86	0.72	667 ± 64	2.29 ± 0.92	0.45 ± 0.01	0.62 ± 0.06
UNIL/NB19			post-IR IRSL150 (MET)	6.62 ± 0.56	0.136	3.17 ± 0.17	2.16E-06 ± 1.24E-07	0.67%	667 ± 20	0.60 ± 0.02	0.56 ± 0.03	0.83	0.67	648 ± 56	2.11 ± 0.87	0.52 ± 0.01	0.56 ± 0.04
UNIL/NB19			post-IR IRSL225 (MET)	6.62 ± 0.56	0.136	2.39 ± 0.18	1.66E-06 ± 1.46E-07	0.74%	518 ± 19	0.74 ± 0.02	0.65 ± 0.05	0.87	0.74	475 ± 44	2.30 ± 0.96	0.62 ± 0.01	0.65 ± 0.06
UNIL/NB19			IRSL50 (MET)	5.90 ± 0.30	0.136	3.46 ± 0.19	2.37E-06 ± 1.40E-07	0.65%	475 ± 18	0.50 ± 0.02	0.54 ± 0.03	0.82	0.65	448 ± 18	1.74 ± 0.47	0.47 ± 0.02	0.53 ± 0.03
UNIL/NB19	Coarse grain, Na-rich K-feldspar	Indicative of exhumation rates too low to be resolved by OSL-thermochronometry (cf. King et al., 2016d)	post-IR IRSL100 (MET)	5.90 ± 0.30	0.136	4.05 ± 0.21	2.68E-06 ± 1.40E-07	0.61%	618 ± 37	0.50 ± 0.06	0.48 ± 0.02	0.79	0.61	660 ± 120	2.75 ± 1.64	0.40 ± 0.05	0.49 ± 0.03
UNIL/NB19			post-IR IRSL150 (MET)	5.90 ± 0.30	0.136	1.92 ± 0.19	1.27E-06 ± 1.33E-07	0.79%	601 ± 19	0.69 ± 0.03	0.71 ± 0.09	0.89	0.79	576 ± 50	2.09 ± 0.90	0.60 ± 0.02	0.71 ± 0.08
UNIL/NB19			post-IR IRSL225 (MET)	5.90 ± 0.30	0.136	0.68 ± 0.22	4.01E-07 ± 1.59E-07	0.20%	473 ± 17	0.83 ± 0.03	0.88 ± 0.31	0.96	0.92	427 ± 36	2.15 ± 0.90	0.72 ± 0.02	0.89 ± 0.31
<i>Valla et al. (2016)</i>																	
GRA-03	Coarse grain, Na/Ca feldspar	Exhumation rates are ~0.1-0.5 km Ma ⁻¹ (e.g. Spotila et al., 2004; Berger et al., 2008)	IRSL50	1.12 ± 0.24	0.18 to 0.26	47.73 ± 0.41	2.18E-05 ± 6.75E-07	0.03 ± 0.01	622 ± 72	0.01 ± 0.00	0.00 ± 0.00	0.17	0.01	-	-	-	-
GRA-04				1.22 ± 0.23	0.18 to 0.26	28.55 ± 0.29	1.55E-05 ± 4.04E-07	0.04 ± 0.01	658 ± 90	0.02 ± 0.00	0.01 ± 0.00	0.27	0.03	-	-	-	-
GRA-05				1.31 ± 0.14	0.18 to 0.26	26.60 ± 0.80	1.44E-05 ± 1.05E-06	0.07 ± 0.01	477 ± 51	0.03 ± 0.00	0.02 ± 0.00	0.32	0.05	-	-	-	-

GRA-06			1.06 ± 0.14	0.18 to 0.26	31.23 ± 0.53	1.61E-05 ± 7.44E-07	0.04 ± 0.00	569 ± 43	0.01 ± 0.00	0.01 ± 0.00	0.29	0.04	-	-	-	-
GRA-08			1.34 ± 0.13	0.18 to 0.26	34.67 ± 1.19	1.75E-05 ± 1.31E-06	0.02 ± 0.00	562 ± 47	0.02 ± 0.00	0.01 ± 0.00	0.26	0.03	-	-	-	-
GRA-09			0.70 ± 0.15	0.18 to 0.26	24.24 ± 0.50	1.33E-05 ± 5.60E-07	0.07 ± 0.01	598 ± 54	0.03 ± 0.00	0.02 ± 0.00	0.35	0.06	708 ± 443	2.19 ± 4.13	0.02±0.00	0.02± 0.00
GRA-10			2.72 ± 0.53	0.18 to 0.26	14.92 ± 0.59	8.84E-06 ± 4.95E-07	0.25 ± 0.02	478 ± 36	0.13 ± 0.01	0.09 ± 0.00	0.51	0.19	-	-	-	-
GRA-11			1.33 ± 0.16	0.18 to 0.26	11.12 ± 0.51	6.91E-06 ± 4.32E-07	0.15 ± 0.02	410 ± 76	0.14 ± 0.01	0.14 ± 0.01	0.57	0.20	-	-	-	-
GRA-12			1.46 ± 0.22	0.18 to 0.26	15.03 ± 0.41	8.98E-06 ± 3.60E-07	0.21 ± 0.00	440 ± 22	0.13 ± 0.01	0.08 ± 0.00	0.51	0.15	547 ± 84	2.93 ± 1.30	0.09±0.01	0.07± 0.01
GRA-13			2.37 ± 0.61	0.26 to 0.27	0.47 ± 0.52	3.37E-07 ± 4.08E-07	0.87 ± 0.00	147 ± 9	0.90 ± 0.04	0.88 ± 0.59	0.98	0.92	434 ± 23	1.65 ± 1.88	0.85±0.02	0.90± 0.05
GRA-14			1.79 ± 0.20	0.28 to 0.29	3.49 ± 0.52	2.42E-06 ± 3.90E-07	0.72 ± 0.01	252 ± 10	0.63 ± 0.03	0.51 ± 0.08	0.83	0.61	241 ± 18	1.80 ± 0.91	0.59±0.02	0.51± 0.08
GRA-17			1.11 ± 0.35	0.12	2.95 ± 0.64	2.00E-06 ± 4.42E-07	0.52 ± 0.00	188 ± 11	0.58 ± 0.03	0.58 ± 0.13	0.85	0.66	171 ± 4	3.03 ± 0.28	0.43±0.02	0.57± 0.04
GRA-18			1.02 ± 0.39	0.12	7.59 ± 0.51	4.91E-06 ± 4.42E-07	0.42 ± 0.01	253 ± 11	0.31 ± 0.01	0.25 ± 0.03	0.68	0.38	245 ± 25	2.02 ± 1.24	0.28±0.01	0.26± 0.01
GRA-19			1.95 ± 0.39	0.12	9.02 ± 1.63	5.35E-06 ± 1.57E-06	0.18 ± 0.01	624 ± 62	0.19 ± 0.03	0.19 ± 0.05	0.60	0.25	934 ± 498	4.55 ± 4.83	0.10±0.01	0.15± 0.07
GRA-BR			3.62 ± 0.49	0.12	4.99 ± 0.62	3.22E-06 ± 4.68E-07	0.32 ± 0.01	326 ± 14	0.40 ± 0.02	0.41 ± 0.05	0.76	0.49	513 ± 192	5.81 ± 3.95	0.23±0.04	0.38± 0.03
Valla et al. (2016)																
SOG-02			8.45 ± 3.44	0.12	8.64 ± 0.46	5.38E-06 ± 4.09E-07	0.28 ± 0.01	647 ± 71	0.19 ± 0.01	0.23 ± 0.02	0.62	0.37	707 ± 419	1.85 ± 5.48	0.17±0.01	0.23± 0.02
SOG-06			9.01 ± 3.01	0.12	4.97 ± 0.41	3.23E-06 ± 3.02E-07	0.55 ± 0.02	602 ± 76	0.44 ± 0.01	0.42 ± 0.04	0.76	0.58	615 ± 282	1.58 ± 5.39	0.41±0.04	0.44± 0.09
SOG-10	Coarse grain, K-feldspar	Exhumation rates are ~0.01-0.05 km Ma ⁻¹ (e.g. Hendriks et al., 2007; Nielsen et al., 2009)	8.68 ± 3.24	0.12	4.90 ± 0.45	3.28E-06 ± 3.66E-07	0.56 ± 0.02	564 ± 64	0.48 ± 0.02	0.42 ± 0.05	0.77	0.58	590 ± 343	1.78 ± 5.96	0.40±0.05	0.44± 0.01
SOG-17			7.51 ± 3.94	0.12	4.90 ± 0.26	3.27E-06 ± 2.19E-07	0.29 ± 0.02	389 ± 82	0.33 ± 0.01	0.43 ± 0.06	0.79	0.57	383 ± 270	1.88 ± 5.01	0.29±0.02	0.45± 0.01
SOG-21			8.87 ± 3.10	0.12	10.88 ± 0.29	6.80E-06 ± 2.77E-07	0.28 ± 0.01	618 ± 95	0.19 ± 0.01	0.17 ± 0.01	0.59	0.31	728 ± 831	2.33 ± 9.10	0.16±0.01	0.18± 0.01
SOG-22	Coarse grain, Na/Ca feldspar		1.00 ± 0.40	0.12	3.48 ± 0.31	2.35E-06 ± 2.35E-07	0.66 ± 0.02	253 ± 89	0.55 ± 0.02	0.52 ± 0.05	0.82	0.58	226 ± 161	1.40 ± 9.85	0.52±0.02	0.48± 0.05
SOG-25	Coarse grain, K-feldspar		8.65 ± 3.19	0.12	5.63 ± 1.16	3.78E-06 ± 8.10E-07	0.50 ± 0.01	518 ± 126	0.43 ± 0.04	0.37 ± 0.09	0.75	0.54	583 ± 812	2.38 ± 11.99	0.36±0.02	0.41± 0.06
SOG-38		8.40 ± 3.34	0.12	3.72 ± 0.31	2.50E-06 ± 2.45E-07	0.57 ± 0.01	512 ± 87	0.58 ± 0.02	0.52 ± 0.06	0.79	0.57	564 ± 806	2.40 ± 12.39	0.44±0.02	0.45± 0.05	
Huntley and Lian (2006)																
BSG1		Baltic Sea granite, Åland, Finland	-	-	1.40 ± 0.60	-	0.69 ± 0.02	-	-	-	-	-	-	-	-	-
CGS		Coryell granite, Westbridge, BC	-	-	5.83 ± 0.44	-	0.40 ± 0.02	-	-	-	-	-	-	-	-	-
MCG		Granodiorite, McKay Cr., North Vancouver, BC	-	-	1.00 ± 0.40	-	0.84 ± 0.02	-	-	-	-	-	-	-	-	-
RMG		"red granite", Rannoch Moor, UK	-	-	4.95 ± 0.35	-	0.51 ± 0.02	-	-	-	-	-	-	-	-	-
WCRS		Sanidine crystal, Kettle River Valley, BC	-	-	10.1 ± 0.30	-	0.27 ± 0.04	-	-	-	-	-	-	-	-	-
CBSS	Coarse grain, K-feldspar	Eocene or Oligocene sandstones, China Beach, Vancouver Island, Bc (Yoreth and Nasmith, 1995, p.129)	-	-	3.12 ± 0.31	-	0.54 ± 0.01	-	-	-	-	-	-	-	-	-
GP10		Pliocene Yorktown Formation, Gomez Pit, Virginia, USA (Lamothe and Auclair, 1999)	-	-	4.37 ± 0.36	-	0.45 ± 0.01	-	-	-	-	-	-	-	-	-
DY23		Red sand, Diring Yuriakh, Siberia, Russia (Mochanov, 1988)	-	-	4.10 ± 0.50	-	0.70 ± 0.04	-	-	-	-	-	-	-	-	-
TML1		Pliocene Unit, Bluefish basin, Yukon (Lamothe and Auclair, 1999)	-	-	3.66 ± 0.09	-	0.74 ± 0.01	-	-	-	-	-	-	-	-	-

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* ρ' not provided in the original publication; calculated from the relationship of $g_{2\text{days}}$ and ρ' shown in the Supplementary Material. *Values estimated based on approximated ρ' parameter. Data from Huntley and Lian (2006) are only included in Figure 9.

2 Table 2 : Environmental dose rate, laboratory dose rate and fading rates for samples used in this
 3 study. Full details of the dose rate calculation for the Palouse samples (i.e. 204/MFRB-1,
 4 204/MFRB-2 and 205/BRR-2) are given in Supplementary Material Tables S.13 and S.14. Data
 5 fitting to determine g_{2days} and ρ' for samples 204/MFRB-1, 204/MFRB-2 and 205/BRR-2 is
 6 shown in Supplementary Material S.2-4.

Sample	Environmental Dose Rate (Gy/ka)	Laboratory Dose Rate (Gy/s)	g_{2days} (%/decade)	$\log_{10}(\rho')$	Reference
204/MFRB1	4.94 ± 0.17	0.081/0.074	3.17 ± 0.28	-5.69 ± 0.05	This study.
204/MFRB2	4.39 ± 0.20	0.081/0.074	3.84 ± 0.37	-5.62 ± 0.04	This study.
205/BRR2	4.47 ± 0.22	0.081/0.074	5.62 ± 0.41	-5.45 ± 0.04	This study.
GOS3	2.70 ± 0.20	0.105	$2.65 \pm 0.42^{\S}$	-5.74^*	Preusser (1999; 2003); Lowick et al. (2012).
GOS4	2.51 ± 0.16	0.105	$2.65 \pm 0.42^{\S}$	-5.74^*	Preusser (1999; 2003); Lowick et al. (2012).
ZEL4	2.60 ± 0.20	0.105	$2.03 \pm 0.93^{\S}$	-5.84^*	Preusser et al. (2001), Lowick et al. (2012).
ZEL7	2.70 ± 0.20	0.105	$2.03 \pm 0.93^{\S}$	-5.84^*	Preusser et al. (2001), Lowick et al. (2012).

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8 § Taken from Figure 2 of Lowick et al. (2012). * ρ' not provided in the original publication;
 9 calculated from the relationship of g_{2days} and ρ' shown in the Supplementary Material.

10 Table 3: Measured equivalent dose values, and corresponding ages, as well as fading-corrected ages calculated using the Huntley (2006) model and
 11 either a single-saturating exponential (1EXP) or general order kinetic (GOK) fit, and the Huntley and Lamothe (2001) approach.

Sample	n	1EXP					GOK					Independent Age Control
		Measured De (Gy)	Measured Age (ka)	Measured D ₀ (Gy)	Age (ka)	H & L Age (ka)	Measured De (Gy)	Measured Age (ka)	Measured D ₀ (Gy)	α	Age (ka)	
MFRB-1	3	68.42 ± 5.79	13.85 ± 1.47	359 ± 6	22.75 ± 2.45	18.34 ± 2.03	60.72 ± 5.03	12.29 ± 1.29	332 ± 5	1.84 ± 0.29	19.20 ± 2.12	Below Glacier Peak tephra, 13.5 ± 0.1 ka cal BP (Kuehn et al., 2009)
MFRB-2	3	42.12 ± 1.54	9.59 ± 0.77	353 ± 6	17.43 ± 1.36	13.46 ± 1.24	36.72 ± 1.45	8.36 ± 0.68	356 ± 5	1.95 ± 0.25	13.86 ± 1.08	Above Glacier Peak tephra.
BRR-2	3	44.70 ± 2.06	10.32 ± 0.94	328 ± 6	23.11 ± 2.12	17.57 ± 1.88	39.36 ± 1.89	9.09 ± 0.83	297.41 ± 5.81	1.84 ± 0.21	18.99 ± 1.88	Below Mazama tephra, 7.6 ± 0.2 ka cal BP (Adams, 1990)
GOS3	4	128.83 ± 4.23	47.71 ± 3.97	200 ± 9	79.01 ± 6.83	61.48 ± 5.98	129.54 ± 4.77	47.98 ± 4.07	421.37 ± 224.54	3.08 ± 2.93	80.09 ± 7.06	Above ¹⁴ C ages of ~48 ka cal BP and a U/Th age of 49.4 ± 3.3 ka (Schlüchter et al., 1987; Geyh and Schlüchter, 1998; Preusser et al., 2003)
GOS4	5	81.20 ± 3.28	32.35 ± 2.52	202 ± 8	49.35 ± 3.72	41.46 ± 3.85	-	-	-	-	-	Adjacent to peat deposit with ¹⁴ C age of ~32.3 ka cal BP and a ²³⁰ Th/U TIMS age of 34.7 ± 4 (Geyh and Schlüchter, 1998). GOS4 is stratigraphically younger than GOS3.
ZEL4	4	228.02 ± 12.35	87.70 ± 8.42	274 ± 13	138.54 ± 14.79	106.76 ± 15.52	-	-	-	-	-	Samples taken from within a unit where a peat layer has been dated to 95.0 ± 3.0 ka using ²³⁰ U SIMS (Geyh et al., 1997; Preusser et al., 2001; Lowick et al., 2012). ZEL4 is stratigraphically younger than ZEL7.
ZEL7	5	306.57 ± 44.33	113.54 ± 18.57	223 ± 7	293.90 ± 82.67	301.45 ± 90.38	314.47 ± 16.07	116.47 ± 10.71	361.95 ± 6.51	2.90 ± 39.75	268.11 ± 21.07	

- Fading correction of feldspar luminescence is explored using known age samples.
- Dose response deviation from a single saturating exponential causes age overestimation.
- A general order kinetic fit results in accurate age determination.
- $2D_0$ cannot be used to evaluate feldspar saturation, unless fading is corrected for.

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