SUPPLEMENTAL INFORMATION ON LUMINESCENCE METHODS

accompanies Thackray, G.D., Rittenour, T.M., and Shulmeister, J., 2020, Ice-thickness variation during marine oxygen isotope stage 4–2 glaciation determined from kame terraces in the Rangitata Valley, New Zealand, *in* Waitt, R.B., Thackray, G.D., and Gillespie, A.R., eds., Untangling the Quaternary Period—A Legacy of Stephen C. Porter: Geological Society of America Special Paper 548, https://doi.org/10.1130/2020.2548(11).

OSL Measurements Of Quartz

The single-aliquot regenerative-dose (SAR) technique of Murray and Wintle (2000) was used for small-aliquot (2-5mm diameter, ~10-60 grains per disk) optically stimulated luminescence (OSL) dating of quartz sand. Optical measurements were performed on Risø TL/OSL Model DA-20 readers with blue-green light emitting diodes (LED) (470±30 nm) as the stimulation source for small-aliquot measurements. The luminescence signal was measured through 7.5-mm UV filters (U-340) over 40-45 seconds (250 channels) at 125°C with LED diodes at 70-90% power (~45 mW/cm²). Preheat conditions prior to natural, regenerative and test dose signals were at 240°C held for 10 seconds. The resultant signal was calculated by subtracting the average of the last 5 seconds (channels 230-250; background signal) from the first 0.6 seconds (channels 1-4; peak signal) of the signal-decay curve. An early background subtraction was applied to aliquots with non-fast OSL signal components, where seconds 1.6-2.4 (channels 10-15; background signal) were subtracted from the first 0.6 seconds (channels 1-4; peak signal) of the signal decay curve. Dose-response curves were fit with saturating-exponential and saturating-exponential plus linear fits to calculate equivalent dose (D_E) values. Aliquots were rejected if they had a response to infrared stimulation (feldspar contamination), if the recycled dose was >20% of unity, if the signal measured at the zero dose step was >10% of the natural signal, or if the natural D_E was higher than the largest regenerative dose given.

Equivalent dose (D_E) values are reported at 2-sigma standard error and were calculated using the central age model (CAM) or minimum age model (MAM) of Galbraith and Roberts (2012). Samples with evidence of partial bleaching as indicated by significant positive skew (USU-920) were calculated using the minimum age model. OSL age estimates were calculated by dividing the D_E value by the environmental dose rate the sample experience during burial. OSL ages are reported at 1-sigma standard error and include errors related to instrument calibration, dose rate and equivalent dose calculations. Errors were calculated in quadrature using the methods of Aitken and Alldred (1972) and Guérin et al. (2011).

IRSL Measurements Of Feldspar

Three samples were dated by infrared stimulated luminescence (IRSL) on potassium-rich feldspathic sand (USU, 919, USU-1085, USU-1370). Luminescence measurements were made following the IRSL SAR protocol of Wallinga et al. (2000) on 2-mm aliquots (~10-20 grains per disk). Infrared measurements were performed on Risø TL/OSL Model DA-20 readers with infrared light-emitting diodes (LEDs) (870±40 nm) and reader dose rates of 0.10-0.11 Gy/sec from a decaying ⁹⁰Sr beta source (Bøtter-Jensen et al., 2003). The luminescence signal was measured through a blue filter pack of 2-mm and 4-mm thick filters (BG-39 and Corning 7-39, respectively) over 100 seconds (250 channels) at 50°C with LED diodes at 85% power (~120 mW/cm²). The resultant signal was calculated by subtracting the average of the last 10 seconds (chnls 225-250; background signal) from the first 0.8 seconds (chnls 1-2; peak signal) of the signal decay curve. Preheat conditions prior to natural, regenerative and test dose signals were at 250°C held for 60 seconds (Blair et al., 2005). Dose response curves were fit within

saturating-exponential and saturating-exponential plus linear fits to calculate equivalent dose (D_E) values.

The IRSL age was calculated by correcting individual D_E measurements for fading (loss of signal with time, g_{2days} %/decade) using the method of Auclair et al. (2003) and the age correction model of Huntley and Lamothe (2001). Cumulative D_E and IRSL age values were calculated using the Central Age Model (CAM) of Galbraith and Roberts (2012). Aliquots were rejected if recycled (repeated) doses were >10% of unity, produced >10% of signal in the zero-dose step (recuperation) or if they had a natural D_E value greater than the highest regenerative dose given. Errors on D_E are reported at 2-sigma standard error and age estimates are reported at 1-sigma standard error and include errors related to instrument calibration, dose rate and equivalent dose calculations, and were calculated in quadrature using the methods of Aitken and Alldred (1972) and Guérin et al. (2011).

Dose-Rate Determination

Samples for dose-rate determination were collected from a 30 cm diameter area surrounding the sample tube. Sediments were homogenized and representative samples were analyzed for radioisotope concentration using ICP-MS and ICP-AES techniques (Table 1S). These concentration values were converted to dose rate following the conversion factors of Guérin et al. (2011) and beta attenuation values of Brennan et al. (2003) (see Table 2S). For the IRSL ages, the beta dose included contribution from 12.5% internal potassium (Huntley and Baril, 1997) and 400 ppm Rb (Huntly and Hancock, 2001) and an a-value of 0.086 (Rees-Jones, 1995). Contribution of cosmic radiation to the dose rate was calculated using sample depth, elevation and latitude/longitude following Prescott and Hutton (1994). Total dose rates (Table 3) were calculated based on water content, radioisotope concentration, and cosmic contribution (Adamiec and Aitken, 1998; Aitken, 1998).

Use Of Quartz OSL Versus Feldspar IRSL

Due to potential added uncertainties with regard to the correction for anomalous fading (loss of signal overtime, Wintle, 1973) and slower bleaching (resetting) rates (Godfrey-Smith et al., 1988) associated with feldspars, we preferentially dated samples using OSL on the quartz fraction. In a few cases the IRSL age is presented instead of the OSL age because the quartz OSL signals were not dominated by the fast-decaying components needed to accurate age determination (Wintle and Murray, 2006; Steffen et al. 2009) and resultant ages were out of stratigraphic and geomorphic sequence suggesting age underestimation. Table S3 provides completed age estimate results for samples used in the interpretations of this paper and preliminary results from the alternate mineral fraction. Note that samples with less than 15 accepted aliquots should be interpreted as preliminary data.

Equivalent Dose Distributions

Equivalent dose (D_E) distributions are shown in Figure S. Overall, despite the samples having been collected from ice-proximal settings, most of the samples are normally distributed and have minimal skew. Sample USU-920 was the only one calculated using the minimum age model (MAM, Galbraith and Roberts, 2012).

Site	Sample num.	USU num.	Depth (m)	In-situ H ₂ O (%) ¹	Grain size (μm)	K (%) ²	Rb (ppm) ²	Th (ppm) ²	U (ppm) ²
Е	NZ-130111-10	USU-917	>20	9.6	150-212	1.99±0.05	97.7±3.9	10.3±0.9	2.2±0.2
Н	NZ-130111-11	USU-918	2	4.9	75-150	1.74±0.04	81.4±3.3	8.8±0.8	2.0±0.1
Н	NZ-130111-12	USU-919	0.9	2.9	63-90	1.81±0.05	85.2±3.4	9.9±0.9	2.3±0.2
Н	NZ-130111-13	USU-920	2.0	4.6	150-250	1.81±0.05	85.4±3.4	9.1±0.8	2.4±0.2
В	NZ-111211-16	USU-1085	1.5	13.5	125-212	1.69±0.04	87.5±3.5	10.3±0.9	2.1±0.2
G	NZ-131211-18	USU-1087	12.8	23.2	150-250	1.71±0.04	84.0±3.4	9.3±0.8	1.9±0.1
А	NZ-150213-31	USU-1366	3.5	21.7	90-150	2.03±0.05	86.8±3.5	9.6±0.9	2.1±0.2
А	NZ-150213-32	USU-1367	3.5	20.0	150-250	1.99±0.05	90.1±3.6	9.1±0.8	2.0±0.1
D	NZ-160213-34	USU-1369	1.4	14.4	180-250	2.28±0.06	104.5±4.2	9.8±0.9	2.3±0.2
С	NZ-160213-35	USU-1370	1.8	10.1	90-180	2.26±0.06	97.3±3.9	12.1±1.1	2.6±0.2
F	NZ-170213-36	USU-1371	6	27.2	90-125	2.07±0.05	87.7±3.5	10.5±1.0	2.3±0.2
F	NZ-170213-37	USU-1372	1.4	4.5	125-180	1.95±0.05	81.7±3.3	9.8±0.9	2.3±0.2
F	NZ-170213-38	USU-1373	4.5	25.7	75-125	1.93±0.05	83.6±3.3	11.0±0.1	2.5±0.2
Ι	NZ-180213-39	USU-1374	8.0	23.0	150-250	1.72±0.04	72.3±2.9	8.3±0.8	1.9±0.1
I	NZ-180213-40	USU-1375	20.0	20.1	90-150	1.92±0.05	82.4±3.3	10.9±1.0	2.2±0.2

Table S1. Radioelement Chemistry, Water Content, and Grain Size Information

¹Assumed 7.0±3.0% for samples with in-situ values <7% as moisture content over burial history.

 2 Radioelemental concentrations determined by ALS Chemex using ICP-MS and ICP-AES techniques.

Site	Sample num.	USU num.	Alpha (Gy/ka) ¹	Beta ² (internal) (Gy/ka)	Beta (external) (Gy/ka)	Gamma (excl. cosmic) (Gy/ka)	Cosmic ³ (Gy/ka)	Total DR ⁴ (Gy/ka)
Е	NZ-130111-10	USU-917	-	-	1.78	1.12	0.02	2.92 ± 0.13
Н	NZ-130111-11	USU-918	-	-	1.67	1.00	0.15	2.82 ± 0.13
Н	NZ-130111-12	USU-919	1.07	0.32	1.81	1.10	0.19	4.49 ± 0.20
н	NZ-130111-13	USU-920	-	-	1.69	1.07	0.15	2.91 ± 0.13
В	NZ-111211-16	USU-1085	1.12	0.53	1.51	1.00	0.16	4.19 ± 0.18
G	NZ-131211-18	USU-1087	-	-	1.32	0.85	0.05	2.22 ± 0.09
А	NZ-150213-31	USU-1366	-	-	1.62	0.96	0.15	2.73 ± 0.11
А	NZ-150213-32	USU-1367	-	-	1.54	0.94	0.15	2.63 ± 0.10
D	NZ-160213-34	USU-1369	-	-	1.84	1.11	0.19	3.14 ± 0.12
С	NZ-160213-35	USU-1370	2.21	0.45	2.08	1.29	0.18	5.21 ± 0.23
F	NZ-170213-36	USU-1371	-	-	1.61	0.97	0.10	2.68 ± 0.10
F	NZ-170213-37	USU-1372	-	-	1.84	1.12	0.18	3.14 ± 0.12
F	NZ-170213-38	USU-1373	-	-	1.58	1.00	0.12	2.70 ± 0.11
T	NZ-180213-39	USU-1374	-	-	1.32	0.82	0.08	2.22 ± 0.09
I	NZ-180213-40	USU-1375	-	-	1.62	1.01	0.03	2.66 ± 0.11

Table S2. Dose Rate Information

¹Alpha contribution to dose rate attenuated using an efficiency factor, or 'a-value', of 0.089±0.05 after Rees-Jones (1995).

²Internal grain beta dose rate was determined assuming 12.5% K (Huntley and Baril, 1997) and 400ppm Rb (Huntley and Hancock, 2001) attenuated to grain size using Mejdahl (1979).

³Contribution of cosmic radiation to the dose rate was calculated by using sample depth, elevation, and longitude/latitude following Prescott and Hutton (1994).

⁴ Dose rate is derived from concentrations by alpha, beta, and gamma conversion factors from Guérin et al. (2011).

Site	Sample number	Lab-#	OSL Age ± 1σ (ka)	IRSL Age ± 1σ (ka)	Number of aliquots: OSL / IRSL ¹	Reported age OSL/ IRSL
Е	NZ-130111-10	USU-917	25.52 ± 2.76	22.4	36(48) / 2(4)	OSL
Н	NZ-130111-11	USU-918	21.12 ± 2.27	24.1	24(39) / 9(10)	OSL
Н	NZ-130111-12	USU-919	11.4 ± 1.6^2	17.21 ± 1.58	19(39) / 15(15)	IRSL
Н	NZ-130111-13	USU-920	17.67 ± 2.70	36.3 ³	24(54) / 5(10)	OSL
В	NZ-111211-16	USU-1085	40.1 ²	68.12 ± 3.23	7(43) / 19(22)	IRSL
G	NZ-131211-18	USU-1087	31.02 ± 3.06	NA	25(47)	OSL
А	NZ-150213-31	USU-1366	18.62 ± 1.68	9.9 ⁴	20(51) / 8(8)	OSL
А	NZ-150213-32	USU-1367	19.08 ± 1.64	12.5 ⁴	17(28) / 8(11)	OSL
D	NZ-160213-34	USU-1369	>26.5 ± 2.4 ²	53.0 ⁵	17(37) / 3(4)	OSL
С	NZ-160213-35	USU-1370	12.3 ²	37.02 ± 3.42	3(15) / 18(18)	IRSL
F	NZ-170213-36	USU-1371	19.35 ± 2.05	45.0 ³	17(33) / 4(4)	OSL
F	NZ-170213-37	USU-1372	22.29 ± 2.79	NA	18(58)	OSL
F	NZ-170213-38	USU-1373	20.60 ± 2.63	NA	19(50)	OSL
I	NZ-180213-39	USU-1374	44.35 ± 8.09	NA	21(50)	OSL
L	NZ-180213-40	USU-1375	52.56 ± 9.23	NA	18(33)	OSL

Table S3. Comparison of Quartz OSL and feldspar IRSL ages. Note that samples with less than 15 accepted aliquots are considered incomplete results and therefore the error uncertainty is not reported. Most samples were dated with OSL on the quartz sand component

¹ Number of aliquots used in age calculation and number of aliquots analyzed in parentheses. Aliquot tallies for the OSL results are reported first and separated from the IRSL aliquot tally by a slash '/'.

² Quartz OSL results produced an age underestimation as indicated by stratigraphic and geomorphic reversal, due to signal contribution from unstable medium-decay components

³ Feldspar IRSL results produced age overestimate as indicated by stratigraphic and geomorphic reversal, likely due to incomplete resetting of the luminescence signal (partial bleaching)

⁴ Feldspar IRSL results produced age underestimate as indicated by stratigraphic and geomorphic reversal, likely due to inadequate correction of anomalous fading.

⁵ Future analyses will target collecting more IRSL data on this sample.

OSL Equivalent Dose (DF) Radial Plots



Figure S1. Radial plots of equivalent Dose distributions (part 1)





Figure S1. Radial plots of equivalent Dose distributions (part 2)



Figure S2. Geomorphic map of study area, similar to Fig. 3 with laboratory sample numbers included for reference. Geomorphic map modified from Borsellino et al. (2017). Letters refer to site locations used in text, tables, and figures. OSL and IRSL ages are shown with small circles and triangles; ages marked "surface" are inferred to be associated in time with kame terrace construction, while those marked "buried" are inferred to record depositional processes prior to kame terrace construction. Black squares mark CRN boulder exposure ages, with relevant age ranges given (Shulmeister et al., 2018a, and erratum). Inset box shows locations and radiometric ages in middle and lower Scour Stream, including three CRN boulder ages near the important lowermost Scour Stream exposure.

REFERENCES CITED

- Aitken, M.J., Alldred, J.C., 1972. The assessment of error limits in thermoluminescence dating. Archaeometry 14, 257-267.
- Aitken, M.J., 1985. Thermoluminescence Dating. Academic Press, London.
- Aitken, M.J. 1998: An Introduction to Optical Dating: The dating of Quaternary sediments by the use of photon-stimulated luminescence. New York, Oxford University Press, 267 p.
- Aitken, M.J., Xie, J., 1990. Moisture correction for annual gamma dose. Ancient TL 8 (2), 6-9.
- Auclair, M., Lamothe, M., Huot, S., 2003. Measurement of anomalous fading for feldspar IRSL using SAR. Radiation Measurements 37, 487-492.
- Balescu, S., Lamothe, M., 1994. Comparison of TL and IRSL age estimates of feldspar coarse grains from waterlain sediments. Quaternary Science Reviews 13, 437-444.
- Blair, M., Yukihara, E.G., McKeever, S.W.S., 2005. Experiences with single-aliquot OSL procedures using coarse-grain feldspars. Radiation Measurements 39, 361-374.
- Bøtter-Jensen, L., Andersen, C.E., Duller, G.A.T., Murray, A.S., 2003. Developments in radiation, stimulation and observation facilities in luminescence measurements. Radiation Measurements 37, 535-541.
- Galbraith, R.F., Roberts, R.G., 2012. Statistical aspects of equivalent dose and error calculation and display in OSL dating: An Overview and some recommendations. Quaternary Geochronology 11, 1-27.
- Godfrey-Smith, D.I., Huntley, D.J. and Chen, W.H., 1988. Optical dating studies of quartz and feldspar sediment extracts. Quaternary Science Reviews *7*, 373-380.
- Guérin, G., Mercier, N., Adamiec, G., 2011. Dose-rate conversion factors: update: Ancient TL 29, 5-8.
- Huntley, D.J., Baril, M.R., 1997. The K content of the K-feldspars being measured in optical dating or in the thermoluminescence dating. Ancient TL, 15(1): 11–13.
- Huntley, D.J., Hancock, R.G.V., 2001. The Rb contents of the K-feldspar grains being measured in optical dating. Ancient TL v. 19 (2), 43-46.
- Huntley, D.J., Lamothe, M., 2001. Ubiquity of anomalous fading in K-feldspars and the measurement and correction for it in optical dating. Can. J. Earth Sci. 38, 1093–1106.

- Mejdahl, V., 1979. Thermoluminescence dating: Beta-dose attenuation in quartz grains. Archaeometry 21, 61-72.
- Prescott, J. R., Hutton, J.T., 1994. Cosmic ray contributions to dose rates for luminescence and ESR dating: Radiation Measurements 23, 497-500.
- Rees-Jones, J., 1995. Optical dating of young sediments using fine-grain quartz: Ancient TL v. 13, 9-14.
- Steffen, D., Preusser, F. and Schlunegger, F., 2009. OSL quartz age underestimation due to unstable signal components. Quaternary Geochronology 4, 353-362.
- Wallinga, J., Murray, A., Wintle, A., 2000. The single-aliquot regenerative-dose (SAR) protocol applied to coarse-grain feldspar. Radiation Measurements 32, 529-533.
- Wintle, A.G., 1973. Anomalous fading of thermo-luminescence in mineral samples. *Nature 245*, 143.
- Wintle, A.G., 1997. Luminescence Dating: Laboratory Procedures and Protocols. Radiation Measurements 27, 769-817.
- Wintle, A.G. and Murray, A.S., 2006. A review of quartz optically stimulated luminescence characteristics and their relevance in single-aliquot regeneration dating protocols. Radiation measurements 41, 369-391.