Sample Preparation

All luminescence samples were processed under a 589 nm sodium vapor lamp further filtered with a Lee 101 filter. Following overnight treatments in 10% HCl and bleach, modal size fractions (165-250 μm) of each luminescence sample underwent multiple density separations in solutions of lithium heteropolytungstate to obtain a pure quartz fraction (ρ=2.58-2.66 g/ml). Quartz samples then underwent two 30 minute etches in concentrated HF before a final 30 minute etching in concentrated HCl. All samples were then hand-rubbed and resieved through an 80 μm cloth to break apart low-quality fragments and ensure measurement of intact, high quality grains.

Instrument Calibration

Sample analyses were performed on a Daybreak 2200 manufactured by Bortolot Daybreak. All samples were analyzed using 9.8 mm diameter aluminum discs with a thickness of 0.5 mm. Photons were recorded using a 9235QA photomultilplier tube manufactured by Electron Tubes with a dark count rate of roughly 20 cps. Stimulation was performed with blue LED's (480 nm) and measured light was filtered using a Schott UG-11 and Edmunds UG-340 filter pack with an approximate admittance window from 260 to 390 nm, peaked at ~330 nm.

Irradiations were performed using a 100 miC ^{90}Sr beta source manufactured by Eckert and Ziegler. Source calibration has been performed with 2 different sets of gamma-irradiated calibration quartz: Riso batches 71 and 98. Because the older batch 71 is not thermally annealed, these two calibration standards give very different sensitivity changes during the SAR protocol. Nonetheless, accepting only analyses for which the first and second test dose agree to within 5% yields tight agreement between the two standards and a nominal dose rate of 0.0618 +/- 0.002 gy/sec (2σ uncertainty) as of June 4th, 2015.

Equivalent Dose (De) Determinations

All samples in this study are analyzed using a six-step SAR protocol for quartz following the standard sequence: natural, regen-1, regen-2, regen-3, zero dose, regen-1' (Murray and Wintle, 2000). Data reduction was undertaken using an Excel spreadsheet originally authored by Ronald Goble and subsequently modified by Tammy Rittenour, Sebastien Huot, and Zach Perzan. Equivalent dose (De) for a given disk and associated uncertainties are estimated by fitting a quadratic function to all of the data, including the zero dose. Equivalent doses (D_e) for a sample were then computed using the central age model for all samples (Galbraith et al., 1999). This was considered appropriate given that over-dispersion was always < 20%, skew was <1, and kurtosis between -1 and 1.

A combined preheat plateau and dose recovery test was run by bleaching samples under a 150 W (1800 lumens) lamp with a 5500 k light spectrum for 20 minutes. Samples were then administered a known dose of 8 Gy which was recovered using various cutheat/preheat combinations, using 6 discs per combination. In one case cutheat was held at 160°C and preheat was increased in 40° increments from 160° to 280°C. In a second series cutheat and preheat were both increased in 40° increments, always held equal to each other. Dose recovery was found to be within uncertainty for all combinations except for 280°/280° and 240°/240°. The best precision and accuracy was obtained using a cutheat/preheat combination of 160°/240°, which yielded a recovered dose of 7.99 + 0.38 Gy (1s).

One criteria used to reject disks was the fast ratio. Shine-down curves were consistent with an OSL signal dominated by the fast component as evidenced by fast ratios that were generally > 12, which we adopted as a rejection threshold (Durcan and Duller, 2011). To further minimize slow and medium components all net signals are computed using an early background correction protocol (Cunningham and Wallinga, 2010), which involves subtracting the average counts per second in the 4-5th seconds from that in the first 0.5 seconds.

Four additional quality criteria used to reject De estimates were the recycling ratio, recuperation ratio, test-dose reproducibility ratio (the % difference between the first and second test dose), and the IR check (Murray and Wintle, 2000). The rejection cutoff for the first three tests was set at 10%. The IR check was administered to verify purity after the SAR protocol was complete. This was done by applying an additional test dose to the disc, preheating it, and then measuring the response to infrared stimulation at room temperature. Disks were rejected if the signal to background ratio was greater than 3.5 or if the net signal exceeded 5% of the final test dose that had been measured on the same sample.

To maintain efficiency we utilize a "reconnaissance" method in which the natural and regen-1 steps (including respective test doses) are initially completed on all 60 samples in the carousel. We then undertake an initial data reduction and only proceed to analyze samples whose test dose ratio (TD1/TD2) is between 0.9 and 1.1, and whose fast ratio is > 12. Typically only 1/3 of the discs (~20 discs) pass this step and are fully analyzed. Of the fully analyzed discs, roughly 1/3 of them (~6 discs) then pass the additional quality criteria. This process resulted in a roughly 10% yield of usable discs.

Dose Rate Determinations

Dose rates were calculated through ICP-MS bulk geochemistry of representative samples collected within a 20 cm radius of each sample's location (Guerin et al, 2011). Analyses were performed by ALS Minerals in Reno, NV using a lithium meta-borate fusion followed by ICP-MS analysis for both trace and major elements. Dose rates and ages were then computed using the DRAC online calculator V1.1 (Durcan et al., 2015). The calculator used nuclide to dose rate conversion factors from Guerin et al., 2011, grain size attenuation factors from Brennan et al., 2003, and etch depth attenuation factors from Guerin et al., 2012.

Direct alpha dose was assumed to be zero given that grains were etched in HF prior to analysis.

Water contents were computed on samples collected from a 20 cm sphere using a mass difference calculation after drying in an oven for two days at 60°C (wet weight - dry weight at room temperature). Present day moisture content was used to estimate the radiation attenuation due to water, with an estimated 25% variability over the life of the sample.

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