Journal of Visualized Experiments

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--Manuscript Draft--

Article Type:	Invited Methods Collection - JoVE Produced Video
Manuscript Number:	JoVE62706R3
Full Title:	Isolation of quartz grains for optically stimulated luminescence (OSL) dating of Quaternary sediments for paleoenvironmental research
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TITLE:

Isolation of Quartz Grains for Optically Stimulated Luminescence (OSL) Dating of Quaternary Sediments for Paleoenvironmental Research

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KEYWORDS:

quartz, mineral separation, quaternary dating, optically stimulated luminescence, OSL, depositional environments.

SUMMARY:

This protocol is for the isolation of quartz grains by size for luminescence dating of sediments. Outlined are physical cleansing and chemical digestions by soaking sequentially in H_2O_2, HCl, HF, and HCl again to isolate quartz grains. The quartz purity is quantified with microscopic assessment, Raman spectroscopy, and IR depletion ratio.

ABSTRACT:

Optically stimulated luminescence (OSL) dating quantifies the time since mineral grains were deposited and shielded from additional light or heat exposure, which effectively resets the luminescence clock. The systematics of OSL dating is based on the dosimetric properties of common minerals, like quartz and feldspar. The acquired luminescence with exposure to natural ionizing radiation after burial provides a depositional age for many quaternary sedimentary systems, spanning the past 0.5 Ma. This contribution details the procedures for separating pure quartz grains of a known range of particle sizes to facilitate luminescence analysis with small or single grain aliquots. Specifically, protocols are given for the needed data and interpretations for effective OSL dating of terrestrial sediment cores or sample tubes from exposures. These cores, 5–20 m long in 1.2 m sections, are split lengthwise and crown-cut leaving 80% of core volume

undisturbed, which facilitates sampling of light-protected sediment for OSL dating deep within the core. Sediment samples are then subjected to a series of physical separations to obtain a certain grain-size interval (e.g., 150–250 µm). Magnetic minerals are removed in wet and dry states using magnets. A series of chemical digestions starts with soaking in H_2O_2 to remove organic matter, followed by HCl exposure to remove carbonate minerals, followed by density separation. Subsequently, grains are soaked in HF for 80 min and after in HCl to render quartz grains solely. The mineralogic purity (>99%) of the quartz extract is quantified with grain petrographic assessment and Raman spectroscopy. Repeating this quartz isolation procedure may be necessary with sediment that contains <15% quartz grains. Excitation of the purified quartz grains by LED-derived blue and IR light allows calculations of the fast and IR depletion ratios, which are metrics to assess the dominance of luminescence emissions from quartz.

INTRODUCTION:

Optically stimulated luminescence (OSL) geochronology yields the time from the last light or heat exposure after sediment erosion, deposition and burial; and further exposure to light or heat. Thus, natural sedimentary processes or heating events (>300 °C) reduces the previously inherited luminescence signal to a consistently low level. In the past two decades, there have been substantial advances in luminescence dating, such as single aliquot and grain analysis of specific mineral grains, like quartz. These experiment-based dating protocols with blue or green diodes can compensate effectively for sensitivity changes induced in the laboratory, rendering OSL ages for the past ca. 500 ka[1–3].

Silicate minerals such as quartz and potassium feldspar have varying crystal lattice-charge defects; some formed mineral crystallization and others due to subsequent exposure to ionizing radiation, resulting in geochronometric potential. These defects are probable locations of electron storage with trap-depth energies of ~1.3–3 eV. A subpopulation of contained electrons in lattice-charge defects of quartz grains is a source for time-diagnostic luminescence emissions with excitation by blue light. Thus, this luminescence emission increases with time, above the solar or heat reset level with exposure to ionizing radiation during the burial period. This signal is reduced to a low, definable level ("zeroed") with subsequent sunlight exposure with sediment erosion, transport, and deposition. This luminescence "cycle" occurs in most depositional environments on Earth and other planets. Thus, OSL dating of sedimentary quartz grains provides a depositional age, reflecting the time elapsed since the last light exposure with deposition and burial (**Figure 1**).

Luminescence dating is a dosimetric-based technique that yields age estimates for selected mineral grains, like quartz, from eolian, fluvial, lacustrine, marine, and colluvial sediments associated with enumerable contexts for geomorphic, tectonic, paleontologic, paleoclimatic, and archaeologic research[2,4–7]. OSL dating is also being evaluated to constrain surface processes on other planets, particularly on Mars[8,9]. Often, the most used mineral in OSL dating on Earth is quartz, reflecting its natural abundance, an inherent sensitivity as a geochronometer, signal stability, and rapid resetting with sunlight exposure (seconds to minutes)[4,10–12]. However, the

accuracy of OSL dating is compromised if the quartz extract is impure, particularly if contaminated by potassium and other feldspars, which can have luminescence emissions ten to hundred-fold brighter than quartz and can yield age underestimates[13]. Therefore, the absolute (>99%) purity for extracts of quartz grains from sediment is pivotal for accurate OSL dating. Thus, the focus of this contribution is to provide detailed procedures for isolating highly purified quartz grain separates from a variety of polymineral sediments, integrating knowledge of mineralogy, crystal chemistry; and optical and Raman imaging to effectively apply laboratory protocols to render OSL ages on quartz grains from carefully sampled strata from retrieved sediment cores. The sediment cores were collected by a push and percussion coring method, which retrieved intact sediment down to a depth of 20–25 m.

The OSL time-sensitive signal is reset relatively rapidly with minutes to hours of sunlight exposure. The geological OSL signal accumulates from this solar reset level. Although the OSL emissions of quartz are considerably variable, reflected original crystalline structure, lattice impurities, sensitization with luminescence resetting cycles (**Figure 1**), and prior radiations[14]. Thus, there is inherent variability in the dose sensitivity of quartz, and dating protocols need to be devised for specific mineralogic and sedimentary provenance. Fortunately, the emergence of single aliquot regenerative (SAR) dose protocols for quartz[1,2] yielded systematics to redress variability in the OSL emissions and metrics to evaluate laboratory changes in apparent OSL sensitivity. Sediment grains function as long-term radiation dosimeters when concealed from further light exposure, with the luminescence signal serving as a measure of radiation exposure during the burial period. The radiation dose that is equivalent to the natural luminescence emission of isolated quartz grains is referred to as the equivalent dose (D_e: in grays, Gy), which is the numerator of the OSL age equation (**Equation 1**). The denominator is the Dose rate (D_r: Grays/yr.), defined by contributing α, β, and γ radiation, originating from the radioactive decay of daughter isotopes in the ^{235}U, ^{238}U, ^{232}Th decay series, ^{40}K, and with lesser contributions from the decay of ^{85}Rb and cosmic and galactic sources.

$$\text{OSL age (yr)} = \frac{\text{Equivalent dose } (D_e; \text{ grays})}{\text{Dose rate } (D_r; \text{ grays/yr}) = D_\alpha w + D_\beta w + D_\gamma w + D_c} \qquad \text{(Equation 1)}$$

Where, D_α = alpha dose D_β = beta dose D_γ = gamma dose D_c = cosmic dose and w=water attenuation factor

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Another method for U and Th determinations in the laboratory or the field is gamma spectrometry, with the Germanium variant able to quantify U and Th isotopic disequilibrium with suitable adjustments to the dose rate. The beta and gamma components of the environmental dose rate need to be modified for mass attenuation[15]. However, there is an effectively insignificant alpha dose for grains >50 µm with the outer 10–20 µm of grains removed by treatment with undiluted HF during preparation. A critical component in dose rate assessment is the quantification of the cosmic and galactic dose during the burial period, which is calculated

for specific points on Earth with adjustments for longitude, latitude, elevation, burial depth, and density of overlying sediment[16,17].

Sediments that contain >15% quartz are usually relatively straightforward for separating out a high purity quartz fraction. However, sediments with <15% quartz often require added time to ensure needed mineralogic purity for OSL dating. Approximately 500–1000 quartz grains are needed for this analysis, but often thousands of grains are separated for duplicate analyses, archiving to expand a calibration library, and future advancements. The mineralogic composition of sediment samples is initially assessed, grain by grain, by petrographic analysis through a binocular microscopic (10–20x) and associated imagine analysis. The mineralogy of individual grains is tested further by Raman spectroscopy to measure grain spectra using an excitation laser (455 nm, 532 nm, 633 nm, or 785 nm) and statistically compare grain emissions to known mineral spectra from the RRUFF System Database[18].

Once the visual and spectral inspection is satisfactory, the purity of the OSL signal is further checked, utilizing an automated luminescence reader system., Three to five aliquots of the sample are exposed to infrared excitation (IR = 1.08 watts at 845 nm ± 4 nm), which preferentially stimulates feldspar minerals, and this emission is compared to emissions by blue light excitation (Bl = 470 nm ± 20 nm), which preferentially stimulates quartz. If the ratio IR/Bl ≥ 5%, the test indicates feldspar contamination and acid digestions are repeated. If the ratio IR/Bl <5%, then the samples are deemed quartz fraction satisfactorily for dating.

Single aliquot regeneration (SAR) protocols on quartz grains is an often-used approach in OSL dating sediments with procedures tailored for a specific sample, a study site, or an area. The reproducibility of these protocols is determined by giving quartz grains a known beta dose (e.g., 30 Gy) and evaluating what heat pretreatment recovers this known dose (**Figure 2**). In practice, determining a D_e with the SAR protocols involves the calculation of a ratio between the natural luminescence and the luminescence from a known test dose (L_n/T_n ratio), which is compared to the luminescence emissions for regenerative doses divided by the luminescence from the same test dose (L_x/T_x) (**Figure 2**). A correction, a consistently applied test-dose (e.g., 5 Gy), has been devised to compensate for quartz grain(s) sensitivity changes with measurement through SAR cycles. Often the OSL emissions increase by >5% with each successive SAR cycle, though given the same dose (e.g., 5 Gy)[7].

At least forty aliquots of quartz or 500 grains are analyzed with TL/OSL reader system, with blue light excitation. The luminescence data generated is analyzed by software associated with the Risø TL/OSL-DA-20 reader system. The D_e and D_r values and age estimates are calculated using the Luminescence Dose and Age Calculator (LDAC)[17]. This platform applies statistical models to determine equivalent dose (De) values and render corresponding OSL age with constrained errors.

The extracted light-shielded sample from a core is prepared for two reasons: 1) To obtain a mineralogic fraction of quartz grains with a purity of >99%, and 2) To isolate grains of specific size fraction, e.g., 150–250 µm, for assessment of the environmental D_r for OSL dating[17]. In many sedimentary settings, quartz grains are common; but mixed with other silicate and non-silicate minerals, rock fragments, and organic matter. Previously, procedures were briefly outlined, indicating some specific steps and reagents needed to isolate pure quartz grains in the context of OSL dating [13,19–23]. This contribution has benefited greatly from these previous approaches. This paper outlines revised, and more detailed protocols using petrographic imaging and Raman technology to monitor grain mineralogy and render highly pure (>99%) quartz extracts for luminescence dating. These quartz isolation protocols have been developed after preparing hundreds of samples from diverse geological environments in the Americas, Eurasia, China, and Africain, the Baylor Geoluminescence Dating Research Laboratory, reflecting analytical experience over thirty years, and are not definitive methods, with suitable variations used by other labs. These are not static protocols, and modifications and additions for improvement are welcomed.

PROTOCOL

NOTE: This section presents the procedures to separate a nearly pure (>99%) quartz fraction from polymineral sediments taken from long (15–20 m) sediment core and are equally applicable to individual tube-like samples collected from outcrops[23]. This methodology has been divided into three components: (1) Sediment core opening, description, and interpretation of sedimentary environments to place the resultant OSL age into a paleoenvironmental context, (2) Retrieval of a small OSL sediment sample from a core without exposure to ambient light, and (3) Separation of a mono-mineralogic quartz extract at a specific size fraction (e.g., 150–250 µm). The first step is conducted under ambient light conditions. The second and third components are undertaken with illumination by a sodium vapor bulb, equivalent LEDs, or bulbs with a red to orange filter. Test have shown that these safe light conditions with emissions centered on 589 nm with about 1–0.5 W/m^2 on the bench surface do not cause inadvertent reset during grain preparations.

1. Open, describe and interpret sediment cores (Figure 3)

NOTE: Use an electric saw at about the quarter diameter (0.5-radian position) of the circumference of the core to open them lengthwise. Perform this “crown” core cut instead of a half-cut to preserve more unlighted-exposed sediment for OSL dating and other analysis without compromising careful visual inspection, sampling, and description of the core.

1.1. Log and evaluate the sedimentologic and pedologic features of a core.

1.1.1. Evaluate the variation in sedimentologic features such as particle size changes, sedimentary and diagenetic structures, bedding if visible, Munsell colors[24], the basis for unit boundaries[25] and identifying sequences of strata.

1.1.2. Ascertain macro-pedologic features including carbonate, argillic and cummlic morphologies; rubification and associated horizon designation; and trace fossils.

1.1.3. Take 1–2 g of the sediment with a spatula, put it in a 50 mL acid-resistant beaker to assess the carbonate content gasometrically.

1.1.3.1. Place the beaker in a well-ventilated box oven (40 °C) for at least 8 h to dry the sample, then weigh on a precision scale and annotate the weight for each sample in the lab book.

1.1.3.2. Add 30 mL of 15% HCl to the sample, place it uncovered inside a fume hood and let it react for at least 30 min. Add acid until the reaction is complete.

CAUTION: HCl acid should always be used inside a fume hood, with the sash no more than a quarter open. A lab coat, chemical-resistant gloves, safety goggles, and a shield are required when handling HCl. Place this mixture in a fume hood for 8 h covered by a wax paper sealant. The reaction of HCl with $Ca/MgCO_3$ is exothermic. Thus, place the beaker in a 300 mL ceramic bowl filled with 100 mL of cold tap water to cool the reaction and capture reaction spillage.

1.1.3.3. Wash the sample with 100 mL of deionized water (DIW), carefully decant the supernatant in to sink without losing the sediment.

1.1.3.4. Return the sample to the oven (40 °C) for at least 24 h until dry; weigh and record the value.

1.1.3.5. Quantify the mass difference between oven-dried samples before and after soaking in 15% HCl to assess carbonate content (%).

1.1.4. Remove 0.5–1.0 g of sediments for particle size analysis every 5–10 cm down the core. Place each sediment sample in a 100 mL acid-resistant beaker. Label the samples in beakers accordingly.

1.1.5. Sieve the sediments through a 2000 µm mesh. Discard the sediment >2000 µm (larger than sand). Continue the process with the remainder sediment <2000 µm.

1.1.6. Add 30 mL of 15% HCl to remove carbonate from the sample. Repeat steps 1.1.3.1–1.1.3.5

1.1.7. Remove the organic matter using 30 mL of 12% H_2O_2 and let it stand for >12 h; do not heat.

CAUTION: H_2O_2 promotes rapid oxidation, is corrosive, and can be very harmful to the eyes, skin, and respiratory system. A lab coat, chemical-resistant gloves, safety goggles, and a shield are required when handling reagent-grade H_2O_2. The addition of H_2O_2 to sediment containing organic matter is an exothermic reaction. The rapid increase in temperature is proportional to the abundance of organic matter disseminated in the sample. The addition of DIW may be

necessary to keep reaction temperature <40 °C. Continue to add H_2O_2 and monitor reaction temperature simultaneously. Let the mixture remain inside a fume hood for 8 h covered by a wax paper sealant. Place the beaker in a 300 mL ceramic bowl filled with 100 mL of cold tap water to cool the reaction and capture reaction spillage.

1.1.8. Determine grain sizes for each sample with a laser diffraction particle size analyzer and classify the range of grain sizes according to the Wentworth scale[26,27].

1.1.9. Assess the data and iteratively resample using finer spacing (2–5 cm) to characterize the unit contacts better or the imprint of pedogenesis (see **Figure 4**).

1.2. Interpret the sedimentary and stratigraphic sections.

1.2.1. Use the resultant logs of sedimentology, stratigraphy, pedology, granulometry, and carbonate percentage to define the depositional units and pedosedimentary facies observed in cores.

1.2.2. Draft the respective sedimentary sections for each core (**Figure 4**).

1.2.3. Interpret the sedimentary and environmental information based on an integrated assessment of the physical core description and granulometry, carbonate content, micromorphology, and facies analysis. Discuss the interpretation of sedimentary environments with others in the research group.

1.2.4. Determine specific depth levels of the cores to be sampled for OSL dating to decipher depositional events[7].

2. Collect OSL sample (Figure 5)

NOTE: The core sections are transferred to the luminescence lab to sample for OSL dating in safe light conditions.

2.1. Moisten the core face with DIW using a squeeze bottle to ensure sediment cohesion.

2.2. Define the sampling area by scoring with a spatula a 2-cm diameter circle from the center point of the core face.

2.3. Scrape off the upper 1 cm of light exposed sediment with a utility knife. Put this sediment in a labeled ceramic evaporation dish to dry for at least 8 h in a box oven at 40 °C. Pulverize and homogenize this dried sediment sample for U, Th, K, and Rb content (assuming secular equilibrium) for dose rate calculations.

NOTE: As an example, assign the sample a consecutive laboratory number (e.g., BG4966) to label on each container that harbors any derivative of the original sample (e.g., BG4966 <200 µm). Link this BG number to the electronic laboratory log, co-registered with the sample field or submittal number. Include other information such as the core number, year collected, drive designation (e.g., B drive), and depth. Labeling subsamples in the lab is a critical task and should be done with exactitude to maintain the chain of sample custody.

2.4. Extract (10–30 g) the light-shielded sediment carefully with a spatula from the circular, scored central area of the core. Place the extract in a labeled 250 mL polyethylene beaker. Clean this sample physically and chemically to isolate a quartz fraction for luminescence dating.

NOTE: Perform core sampling in one direction (usually top to bottom) and one at a time to avoid sampling errors and contamination. Process the samples individually, in numerical order, to maintain the chain of custody.

2.5. Fill the remaining sample cavity in the core with a ball of aluminum foil to designate sample position and prevent sidewall collapse of the split core. Moisten the core face with DIW using a spray bottle, wrap in plastic, and seal the core for archiving.

3. Extract mono-mineralogic quartz (Figure 6)

NOTE: All personnel prior to initiating procedures in the lab are required to wear personal protective equipment (PPE), which includes a heavy and impermeable lab coat, accompanied by nitrile disposable gloves and goggles, and dust masks. This PPE is complemented with heavy PVC gloves and body-long apron, acrylic face shield, and reusable silicone waterproof shoe covers when using solvents at full strength for digestions.

3.1. Remove organic matter: Add slowly 30 mL of 25% H_2O_2 to 30–60 g of the sediment in a 250 mL polyethylene beaker to remove organic matter. Stir well with a glass rod to facilitate the reaction. Add H_2O_2 until there is no visible effervescence with the release of CO_2; let it sit inside the fume hood for at least 12 h.

CAUTION: Perform this procedure under a fume hood. H_2O_2 promotes rapid oxidation, is corrosive, and can be very harmful to the eyes, skin, and respiratory system. A lab coat, chemical-resistant gloves, safety goggles, and a shield are required when handling reagent-grade H_2O_2. The addition of H_2O_2 to sediment containing organic matter is an exothermic reaction. The rapid increase in temperature is proportional to the abundance of organic matter disseminated in the sample. The addition of DIW may be necessary to keep reaction temperature <40° C. Continue to add H_2O_2 and monitor reaction temperature simultaneously. Let the mixture remain under a fume hood for 12 h covered by a wax sealant. Place the beaker in a 300 mL ceramic bowl filled with 100 mL of cold tap water to cool the reaction and capture reaction spillage.

NOTE: If the organic matter content is >3%, the sample may require 1–3 days of soaking in H_2O_2 to react with organic carbon fully. Monitor the exothermic heat evolved and add DIW to keep it below 40 °C. Do not heat the sample above 40 °C. Higher temperatures may cause partial resetting of the luminescence signal and sensitivity changes detrimental to dosimetric measurements.

3.2. Wash the sample five times with 100 mL of DIW to remove any remaining H_2O_2 and halides present in the sediment. After settling for 30–60 min, decant the supernatant into the sink with the water running. Take care to preserve the sediment at the beaker bottom during decanting.

3.3. To remove calcium/magnesium carbonate from the sediment, slowly add 30 mL of 15% HCl for each 5 g of sediment in a 250 mL beaker to react with the $Ca/MgCO_3$ disseminated in the sample. Initially add ≤ 1 mL to assess effervescences and modulate further HCl additions to control better reaction. Stir well with a glass rod to facilitate the completion of the reaction. Add more HCl if necessary until there is no visible effervescence with the release of CO_2.

CAUTION: Use HCl inside a fume hood, with the sash no more than a quarter open. A lab coat, chemical-resistant gloves, safety goggles, and a shield are required when handling this and other acids. The reaction of HCl with $Ca/MgCO_3$ is exothermic. The addition of DIW may be necessary to keep reaction temperature <40 °C. Continue to add HCl and simultaneously monitor reaction temperature. Let the mixture remain inside a fume hood for 8 h covered by wax paper. Place the beaker in a 300 mL ceramic bowl filled with 100 mL of cold tap water to cool the reaction and capture reaction spillage.

3.3.1. Wash the sample with 100 mL of DIW five times and decant carefully to remove excess (diluted) HCl into a sink with the water running.

3.3.2. Dry the sediment overnight in a box oven at 40 °C.

3.4. Remove the magnetic, paramagnetic, and diamagnetic minerals.

NOTE: Most sediments contain <10% magnetic minerals. Perform magnetic mineral removal of the sediment in a dry state using neodymium magnets or wet state using the dispersant Na-pyrophosphate ($Na_4P_2O_7 \cdot 10H_2O$) solution (0.3%). Removal of magnetic and associated minerals is necessary as these components compete with HF etching of quartz and dissolution of other silicate minerals.

3.4.1. Wrap a ~2.5 cm long neodymium magnet with a 38 µm nylon mesh sleeve for dry sediment removal of magnetic minerals.

3.4.2. Place the wrapped magnet on the outside wall of the beaker and move in a circular motion to attract magnetic minerals.

3.4.3. Move the magnet slowly to the top of the beaker to extract the minerals into a 20 mL ceramic dish. Remove the magnet and detach the magnetic minerals attached to the nylon sleeve.

3.4.4. Repeat steps 3.4.1–3.4.3 until to remove the magnetic grains completely; usually after 5 to 6 repeats.

3.4.5. To remove the magnetic grains in a water-based solution, place the sediment in a 250 mL glass beaker with ~100 mL of 0.3% Na-pyrophosphate solution and stir thoroughly until the sediment is well disaggregated.

3.4.6. Place the beaker on a hot plate with a built-in magnetic stirrer; set stir rate at 800 RPM at ambient laboratory temperature. Submerge the magnetic rods and stir the sediment for 5 min.

3.4.7. Remove the rods to clean off attracted magnetic grains by rubbing with a cloth or another magnet before returning the magnets to the solution. Repeat until no magnetic minerals are recovered; up to five repeats may be necessary.

NOTE: A binocular microscopic inspection of the sample is advised to assess the status of magnetic mineral removal. Together, the dry and wet magnetic mineral removal is usually >95% effective.

3.5. Separate a specific grain-size fraction.

NOTE: The particle size range of quartz grains to be separated is based on the previously determined particle size distribution for each sample (see step 1.1.5). Common particle size ranges to separate quartz grains are 500–450 µm, 450–355 µm, and 355–250 µm for medium sand, 250–150 µm and 150–100 µm for fine sand and 100–63 µm for very fine sand.

3.5.1. Cut 15 cm x 15 cm squares from rolls of nylon mesh of two sizes (e.g., 150 µm and 250 µm) for particle size isolation using wet sieving with disposable meshes.

3.5.2. Frame the cut mesh in a 10 cm-inner diameter circular plastic guide. For example, to target the fine sand fraction 150–250 µm, use two framing meshes sequentially: 250 µm first and 150 µm second.

3.5.3. Label three beakers with the laboratory sample number (BGXXXX) and sieving limits; >150 >250 µm, and 250–150 µm (Inset **Figure 6A**).

3.5.4. Place the circular sieving guide tightly with framed mesh, e.g., first, use 250 µm (coarser grain size) over a 1-L beaker rim (10.5 cm diameter).

3.5.5. Sieve sample to the targeted particle size range, e.g., 250–150 µm. Set up 1-L beaker with 250 µm mesh guide on top; ready to sieve.

3.5.6. Add ~100 mL of 0.3% solution of Na-pyrophosphate to a 250 mL beaker that contains the nonmagnetic sediment obtained in step 3.4.7 and stir thoroughly with a glass road to facilitate particle dispersion.

3.5.7. Continue to manually swirl the dispersed sediment mixture, and slowly pour through the 250 µm mesh. The sediment of particles <250 µm size passes through the mesh into the below beaker and is the target for further size separation. Archive the sediment remaining on the mesh (>250 µm) for possible future analysis.

3.5.8. Set up the 150 µm mesh over a new dry 1-L beaker. Take the dispersed sediment mixture of step 3.5.7, continue to swirl in hand, and slowly pour through the 150 µm mesh. The sediment of particles <150 µm size passes through the mesh into the below beaker. Archive the sediment for possible future analysis. The sediment remaining on the 150 mesh is the target size fraction, 150–250 µm, for OSL dating.

3.5.9. Dry the sediments in a box oven overnight at 40 °C.

3.6. Isolate quartz grains from the 250–150 µm size separately (Inset **Figure 6B**).

NOTE: This procedure includes two density separations using the non-toxic heavy liquid Sodium Polytungstate (SPT-Na_6 ($H_2W_{12}O_{40}$) _H_2O) at densities 2.6 g/cc and 2.7 g/cc. Mix the powder with DIW to constitute this heavy liquid. To prepare 100 mL of the heavy liquid with a density of 2.6 g/cc, add 205.5 g of SPT to 54.5 mL of DIW. Whereas, to prepare 100 mL of the heavier liquid with a density of 2.7 g/cc, add 217.5 g of SPT to 52.7 mL of DIW. Assess the density of the heavy liquid with pre-calibrated density beads and a hydrometer.

CAUTION: Use only DIW to prepare heavy liquids because tap water contains dissolved ions that react and change the composition of the SPT powder. To generate a homogeneous solution of the desired density, add the SPT powder to the water and not the counter.

3.6.1. Label two 100 mL beakers with the sample number adding "<2.6" to one beaker and ">2.6" to the other beaker. Keep a 1 L beaker ready to collect the heavy liquid washed from the sample with DIW.

3.6.2. Mix thoroughly 80–70 mL of 2.6 g/cm^3 heavy liquid with the dry fraction of the sediment obtained in step 3.5.8. Pour the mixture into a well labeled 100 mL graduate cylinder. Cover the top with a wax sealant to avoid evaporation. Place the cylinder inside a fume hood to remain undisturbed and shielded from light. Wait for at least 1 h to allow the sample to separate in two markedly different zones. The higher floating, lighter minerals are often enriched in K-feldspar and Na-rich plagioclases, and the lower heavier grains are rich in quartz and other heavier minerals.

NOTE: The separation times using the 2.6 g/cc heavy liquid for smaller particle sizes, <100 µm, may take >4 h.

3.6.3. Place a plastic funnel and place a disposable paper filter over a 250 mL beaker. Filter the solution with a tight fit.

3.6.4. Decant the floating sediment of the 2.6 g/cm^3 heavy liquid through the filter slowly and carefully, with suspended grains captured on the filter. Preserve the lower zone of settled grains carefully. Let the liquid pass through the filter; wash with DIW as needed.

3.6.5. Transfer the washed light sediment to the beaker labeled as "sample number <2.6", placing the paper filter in the beaker and washing carefully with DIW. Discard the filter after washing off all the grains.

3.6.6. Wash the sample five times with DIW to remove vestiges of heavy liquid.

3.6.7. Dry the sediments in the oven overnight at <40 °C. Store this feldspar-rich fraction for future analyses.

3.6.8. Put a new filter paper on the plastic funnel and place it tightly on a 1 L glass beaker. Decant the lower settled mineral grains in the graduated cylinder with 2.6 g/cm^3 solution. Then, wash out the cylinder with DIW using a squirt bottle.

3.6.9. Transfer the washed "heavy" sediment to the beaker labeled with the "sample number >2.6". Place the paper filter in the beaker and wash carefully with DIW. Discard the filter after washing off all the grains.

3.6.10. Wash the sample three times in the sink with DIW.

3.6.11. Dry the sediments in the oven overnight at <40 °C for further density separation using 2.7 g/cc heavy liquid.

3.6.12. Continue with quartz separation with a 2.7 g/cc heavy liquid. Combine the dry "heavy" separate from the beaker labeled "sample number >2.6" with 70–80 mL of 2.7 g/cc heavy liquid.

3.6.13. Decant the floating sediment (quartz-rich) onto a funnel-filter pair over a 1 L beaker slowly and carefully. Wash the floating sample on the filter thoroughly with DIW and collect the wash in the beaker below.

3.6.14. Transfer the washed sediment on the filter to a 250 mL polypropylene beaker labeled with the "sample number + for HF". Place the paper filter in the beaker and wash carefully with DIW; discard the filter after washing off all the grains. Immerse the sample in HF.

3.6.15. Put a new paper filter on the plastic funnel, and place both on a new 1 L glass beaker. Add DIW to the cylinder where the 2.7 g/cc density separation occurred, decant and wash with DIW until the lower separated grains are transferred completely to the filter. Repeat steps 3.6.10–3.6.12 and archive this heaviest fraction.

3.7. Etch the quartz grains by immersing in hydrofluoric acid

NOTE: This procedure has two main goals: 1) to dissolve any remaining minerals other than quartz; 2) To etch the external 10–20 µm of quartz grains, affected by the alpha radiation[28].

CAUTION: Concentrated hydrofluoric acid (HF) is a highly toxic and hazardous liquid. Special training and care are needed to use HF because of the high dermal and pulmonary toxicity. Lab personnel must be familiar with the HF Material Safety Data Sheets. Always handle HF inside an operational laboratory fume hood, near an eyewash and safety shower station. Never work with HF alone. Ensure that non-expired 2.5% calcium gluconate gel antidote is at hand before handling HF. The following PPE must be worn prior to handling HF: Long pants and sleeves, closed-toe shoes, heavy lab coat, acid-resistant apron, thick nitrile gloves (10–20 mil), PVC or neoprene gloves that cover the hands, wrists, and forearms, dust mask, goggles, acrylic face shield, and silicone waterproof shoe covers.

3.7.1. Prepare a timer for 80 min and cut wax paper sealant to cover a 250 mL beaker.

3.7.2. Turn on both the DIW and regular water taps at the sink and have a bottle of DIW at hand as a safety precaution.

3.7.3. Put on the appropriate PPE to use HF acid.

3.7.4. Place a 250 mL heavy-duty polypropylene beaker with the sample obtained in step 3.6.14 inside the fume hood; lower the sash to near closure to be safe and comfortable to work. Add HF to the beaker by pump increments (20 mL) for every 2 g of quartz and cover the beaker with wax paper sealant.

NOTE: For enhanced safety, use an HF bottle dispenser that delivers set volumes of acid, e.g., 20 mL/pump, to control the amount and direction of acid delivery. High-density plastic containers are used with HF because this acid reacts with and etches glass.

3.7.5. Start the 80 min timer and remove the HF-PPE. Keep in mind to wear the PPE again to clean the sample 5 min before the time is complete.

3.7.6. Wash the sample five times under the hood. Fill the beaker with DIW to dilute the acid and decant it into a satellite container used for HF waste.

3.7.7. Remove the sample from the fume hood and wash the sample three more times with DIW at the sink, keeping both the DIW and regular water taps open to dilute any remaining HF further.

3.7.8. Decant and move the sample into a 250 mL glass beaker, add ~150 mL of 0.3% Na-Pyrophosphate ($Na_4P_2O_7 \cdot 10H_2O$) solution to the sediment and place the beaker in a sonicator bath for 20 min to fully disaggregate the grains and particles.

3.7.9. Wash the sample five more times with DIW at a sink to remove the Na-pyrophosphate. Decant and label the beaker "Sample Name" for HCl".

3.8. Immerse the mineral grains remaining after HF digestion (step 3.7.9) in concentrated HCl.

CAUTION: Concentrated HCl (~36%) is considered a toxic and corrosive fluid that can cause chemical burns upon contact and eye damage if splashed, and injury to the mouth, throat, esophagus, and stomach if ingested. Workers are required to be familiar with the HCl Material Safety Data Sheets. Always handle concentrated HCl inside an operational fume hood, near an eyewash and safety shower station. Never work with HCl alone. Before starting the digestion of the sediment with HCl, be sure to wear the PPE listed in step 3.7.

NOTE: As with concentrated HF, it is safer to use a bottle dispenser to control the amount and direction of the discharge. Use glass containers when working with HCl. Before removing the PPE, wash the gloves with soap and water, and after removing the PPE, wash hands and forearms.

3.8.1. Prepare the wax sealant to cover the beaker with the sample immersed in the acid.

3.8.2. Turn on both the DIW and regular water taps at the sink and have a bottle of DIW at hand as a safety precaution.

3.8.3. Put on the acid PPE.

3.8.4. Place the 250 mL glass beaker with the sample obtained in step 3.7.9 inside the fume hood. Lower the sash to near closure to be safe and comfortable to work. Add HCl to sample by pump increments (20 mL) for every 5 g of quartz and then cover the beaker with wax sealant paper.

3.8.5. Remove the acid PPE.

3.8.6. Leave the sample for HCl digestion for 8 h in the fume hood.

3.8.7. Put on the acid-PPE before cleaning the HCl.

3.8.8. Wash the sample five times under the hood; decant supernatant into the satellite container to collect HCl waste.

3.8.9. Wash the sample three more times with DIW at the sink, keeping both the DIW and regular water taps open for further dilution. Make sure to continue wearing the necessary PPE.

3.9. Re-sieve the sediments through the smallest prior mesh (e.g., 150 µm) to remove fractured and broken grains.

3.10. Decant and label the beaker "Sample Name for OSL" and dry the sediments in the oven for at least 8 h at <40 °C to evaluate the purity of quartz separation of this finished product.

3.11. Quantify quartz separate purity

3.11.1. Use a dissecting needle to place 200–400 mineral grains on a glass slide and inspect under a 10x or 20x binocular and/or petroscopic microscope to identify grain minerals. Quantify the percentage of quartz grains by point counting and record the mineralogy of 100 individual grains. If a subsample exhibits >1% non-quartz minerals and is an unwanted mineral with high photon output (e.g., K-feldspar) or remains unidentified, cue the sample for Raman spectroscopy.

3.11.2. Use Raman spectroscopy and associated image to confirm the grain mineralogy and identify minerals unrecognized under microscopic inspection. Use a blue beam with a width of 5 µm and 100-grain point counts to assess the percent purity of quartz and identify the unknown grain minerals.

3.12. Assess the quartz purity spectra by infrared stimulation

3.12.1. Prepare five ultra-small aliquots of quartz separates for IR stimulation by shaking grains onto a circular aluminum disc (1 cm diameter). Each aliquot usually contains approximately 20–100 quartz grains corresponding to a 1 mm or less circular diameter adhered (with silicon) to a disc.

3.12.2. Load the discs on a sample carousel for stimulation by IR LEDs (845 nm ± 4 nm) delivered by an automated TL/OSL reader system and compare it with the blue light excitation (470 nm ± 20 nm), which is preferential for quartz.

3.12.3. Ensure that the ratio between IRSL and blue light emissions of quartz grain aliquots is <5%. If such is the case, the sample is ready for further analysis. Otherwise, the sample requires additional cleaning with HF (step 3.7).

REPRESENTATIVE RESULTS

The laboratory procedures outlined are focused on enhancing the separation of pure quartz grains (700 to 50 µm size) needed for OSL dating without inadvertent light resetting in the laboratory (**Figure 1**). A pure quartz separate, mineralogically and optically, is a prerequisite for applying SAR and TT-OSL dating procedures (**Figure 2**). These procedures explain the necessary steps for effectively understanding and sampling continuous sediment cores, avoiding zones of pedogenesis and diagenesis, retrieving unlight-exposed sediments from cores (**Figure 3** and **Figure 4**); to isolate quartz grains for OSL dating protocols to constrain the timing of sediment

deposition in the past ca. 500 ka (**Figure 5**). The mineralogy of grains of the unprepared sample and prepared separates are assessed continuously through the preparation process to identify the contaminating mineralogy and actively assess the process of removal of unwanted minerals (**Figure 6** and **Figure 7**). The quartz mineralogic purity is determined for subset grains (100–400) through binocular microscopic inspection (10–20x) and by Raman spectroscopy. The use of this technology and prerequisite knowledge is vital to assess and confirm the needed purity (>99%) of quartz separations for OSL dating (**Figure 8**).

The process for quartz separation is started with the removal of organic matter with H_2O_2 and then the subsequent purging of Ca/$MgCO_3$ with soaking in HCl (**Figure 6A** inset). Subsequently, a size fraction is designated by sieving with disposable nylon mesh (e.g., 150 and 250 µm), which is necessary for calculating dose rate values (in mGy/y). The purity of the quartz separate is enhanced by two density separations at 2.6 and 2.7 g/cc, the bounding density of quartz (**Figure 6B** inset). The subsequent soaking of sized grains in HF for 80 min removes non-quartz minerals. This treatment also etches the outer 10–20 µm of grains to remove the alpha-dose affected area, simplifying dose rate calculations (**Figure 6**). The purity of the quartz separate is never assumed but assessed through binocular microscopic inspection and Raman-based measurements at the end of grain separation. Density separations and/or HF treatment can be repeated to rid the separate contaminating grains if a representative aliquot contains >1% non-quartz grains, particularly feldspar minerals (**Figure 7**). The quartz purification procedure was repeated up to four times with quartz contents of <15% to render shine down curves with a fast ratio of >20, characteristic of pure quartz (**Figure 8**).

FIGURE AND TABLE LEGENDS

Figure 1: Processes with OSL dating. (**A**) Mineral grains acquire OSL with ionizing radiation exposure. (**B**) Grain OSL is reset by sunlight with erosion/ transport. (**C**) Exposure to ionizing with burial; luminescence acquired. (**D**) Light exposure resets OSL with erosion/ transport. (**E**) Grains are re-buried, and OSL is acquired with exposure to ionizing radiation. (**F**) Shows sampling without light exposure. The resultant measured natural OSL is followed by a normalizing test dose (L_n/T_n) which is equated to the regenerative dose curve to yield an equivalent dose (D_e). This figure has been modified from Forman, S. L. et al.[7].

Figure 2: Optical Stimulating Luminescence- Single Aliquot Regeneration (OSL-SAR) protocols for quartz grains. (**A**) Equivalent dose using SAR protocols; the natural OSL is L_n/T_n, and the regenerative dose is L_x/T_x; sensitivity changes are corrected by giving a test dose (e.g., 5 Gy). (**B**) Generalized SAR protocol. This figure has been modified from Forman, S. L. et al.[7].

Figure 3: Flow diagram outlining the steps necessary to open, describe, and interpret a recovered sediment core. This figure shows retrieval of sediment core using percussion corer, followed by the opening, cleaning, description, and study of the core to obtain the optimal sample for OSL dating.

Figure 4: Example of a typical log of a core sedimentary and stratigraphic section. Units and pedosedimentary facies are defined using sedimentology, stratigraphy, pedology, granulometry, and carbonate percentage. The soils horizons found in the stratigraphic column from top to bottom are A: Surface organic-rich horizon, B: subsoil with weak structure and color (Bw), and buried B horizon Btb with clay accumulation, Btkb with secondary calcium carbonate and clay accumulation, and Bkb with an accumulation of secondary calcium carbonate. The dominant particle size of sedimentary units is shown on the lower horizontal with medium sand (MS), fine sand (FS), very fine sand (VFS), and Silt (Si).

Figure 5: Flow diagram for the steps necessary to collect an OSL sample from a sediment core. This figure presents a flow diagram with the main steps followed to prepare a quartz separate for OSL dating. The protocols start with the extraction of a polymineral sediment from light-shielded areas of the core in the light safe OLS lab and continue with the extraction of the mono-mineralogic fraction of quartz, comprising the removal of organic matter with peroxide, carbonates with HCl, magnetic minerals using hand magnets, proceeding by the separation of the specific fraction of sand-size sediment by sieving, separation of minerals less dense and heavier than quartz using density liquids (ρ = 2.6 g/cc and 2.7 g/cc). The final steps of cleaning require immersion of the sediment into HF and HCl full strength to isolate quartz from any other mineral in the fraction. The purity of the separate is evaluated by binocular inspection, RAMAN spectroscopy, and further verification of IRSL (Infrared) emissions. The goal is to obtain a sample with a purity ≥99%. Failure to do so requires that some of the steps must be repeated.

Figure 6: Flow chart depicting all the steps necessary to obtain a pristine quartz separate from a sediment sample from a core. This clean quartz fraction will be used for OSL-SAR analyses for age assessment.

Figure 7: Comparison of two collected in two different areas: White Sands and Mongolia. (**A**)The first panel shows raw samples under the binocular microscope, as collected in the field. The sample from White Sands contains sulfates (mainly gypsum), halides, and very little quartz. Its correspondent process sample in panel (**B)** shows a separate fraction (63–100 µm) that contains mostly quartz, but still, there are some vestiges of gypsum, as shown by the Raman Spectroscopy results in panel (**C**). The ratio between the OSL IR and blue responses is 9%, corroborating that the sample needs to go back to be better separated; density at 2.6 g/cc could work in separating the lighter gypsum (2.36 g/cc) from heavier quartz. The Mongolian sample is very rich in felsic feldspars, predominantly K-feldspar. After the cleaning procedures, abundant quartz was isolated in a 100–150 µm separate (panels **B** and **C**), rendering a satisfactory IR/Bl ratio of 3.7%.

Figure 8: Comparison of fast ratio for the natural in three samples that represent different degrees of quartz fraction purity. (**A**) The ideal fast ratio distribution in a pristine eolian sample from Red River, with fast ratio= 72. Contrasting figures (**Figure 7B,C**) have a less fast component

with blue LED stimulation, which is below 20. (**B**) A sample with incomplete quartz and plagioclases. The L2 and L3 components are a significant % of the L1 component (see **Equation 2**). (**C**) A shine-down curve for feldspathic quartz, with a dominant medium component (L2).

DISCUSSION

Quartz mineralogical purity is critical for OSL dating. However, quartz spectral purity is equally important and is usually enhanced with the careful concentration of quartz grains. Ideally, quartz grains under blue LED light (470 nm ± 20 nm) stimulation for 40 s should emit ≥ 90% of the luminescence within the first ~0–2.5 s of stimulation, termed the fast component, with < 10% of light emission between ~2.5 and ~15 s (medium component), and a final low emission post ~15 s, (slow component) (**Figure 8**). A luminescence emission dominated by a fast component is preferred because it is rapidly solar reset (in seconds) and shows high sensitivity to applied β radiation in the laboratory, enhancing equivalent dose determinations. An important metric to assess the dominance of fast components for OSL dating of quartz is the calculation of a "fast ratio"[29,30] with an example shown by **Equation 2** and in **Figure 7**. A fast ratio of >20 for quartz shine down curve is considered a robust luminescence emission suitable for OSL dating[29] (see **Figure 8A**). Separates that have contamination with K-feldspars and plagioclase or feldspathic inclusions often yield fast ratios of <10 (see **Figure 8B,C**) and are unsuitable for SAR quartz dating protocols.

$$\text{Fast Ratio} = \frac{L1-L3}{L2-L3} \qquad \text{(Equation 2)}$$

Where L1: Fast component emission for ~0–2.5 s L2: Medium component emission ~2.5–15 s L3: Slow component emission ~ 15–40 s

An important test on the spectral purity of isolated quartz grains is the response of aliquots to infrared excitation from LEDs (845 nm ± 4 nm). Most quartz grains yield a low or negligible luminescence emission with IR stimulation at or within a few hundred counts of background emissions. A metric has been developed to assess IR-based emissions, called the IR depletion ratio, which is calculated as a SAR ratio (L_x/T_x) for irradiated (5–10 Gy) quartz grains stimulated with IR LEDs and then blue LEDs. Specifically, the ratio of IR luminescence divided by blue emissions should be <5%, which indicates a spectrally pure quartz fraction amenable for OSL dating (**Figure 7**). However, there are instances that mineralogically pure quartz grains can yield errant luminescence emissions with IR stimulation. This IR signal may reflect adhering lithic fragments or feldspathic inclusions in quartz. In such instances, quartz grains should be dated by feldspar protocols[31]. These protocols with modifications can be used to separate and confirm the purity of other minerals for OSL dating, such as k-feldspar, plagioclase, and olivine and pyroxene for other planetary applications.

The ability to isolate a >99% quartz separate and confirm the purity at the grain level is a prerequisite for accurate luminescence dating. Single-grain and ultra-small aliquot (10–50 grains) dating requires additional verification that the luminescence emissions of all grains were from quartz. In turn, the application of thermal transfer approaches that can yield credible OSL ages

up to one million years is predicated on pure quartz signals from mineral grains[6]. A mono-mineralogic quartz separate is foundational for applying OSL-SAR protocols, which provides a sequence of ages for deciphering the depositional history of eolian and fluvial systems for the late Quaternary[1,2,32,33] (**Figure 1** and **Figure 2**). Contamination of quartz aliquots by the errant K-feldspar grains or feldspathic inclusions in quartz or adhering lithic fragment yields a mixed dosimetric signal and prone to anomalous fading often yields underestimates[4]. However, a pure quartz separate does not absolutely ensure spectral purity and appropriate emissions for quartz dating. Effective OSL dating requires careful and complete isolation of quartz grains and OSL associated metrics to verify a pure quartz separate mineralogically and spectrally[2,33,34].

ACKNOWLEDGEMENTS

Support of the Geoluminescence Dating Research Laboratory has been provided by Baylor University and grants from the National Science Foundation (GSS-166023), National Geographic (#9990-1), and Atlas Sand. Discovery and learning in this lab were enhanced by our many collaborators, students, and visitors that have brought new perspectives, ideas, and approaches.

DISCLOSURES

The Baylor University Geoluminescence Dating Research Laboratory, within the Dept. of Geosciences and the associated personal do not have any conflicts of interest or financial interest that can affect the design of experiments or analysis, protocols, outcomes of the research or educational activities conducted in the lab. This Lab, including all the technology within and software, is used solely to conduct research, discovery, education, and mentoring.

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Figure 1

Fig. 1

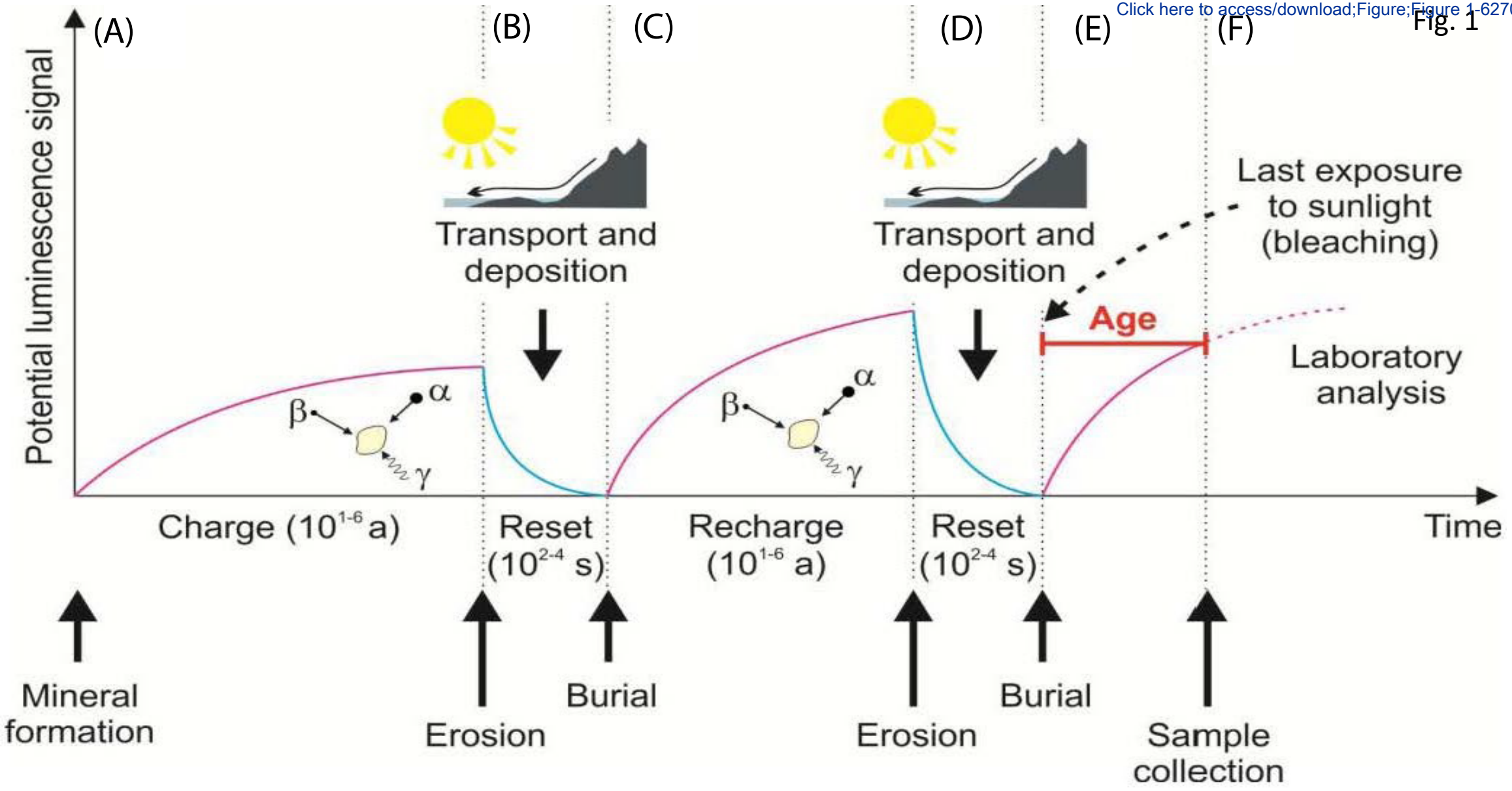
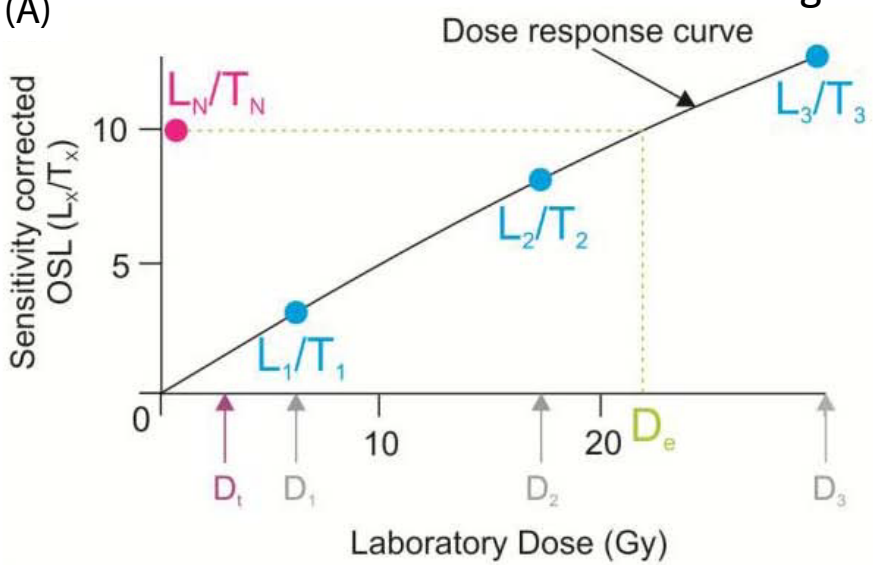


Figure 2

Fig. 2

(A)



(B)

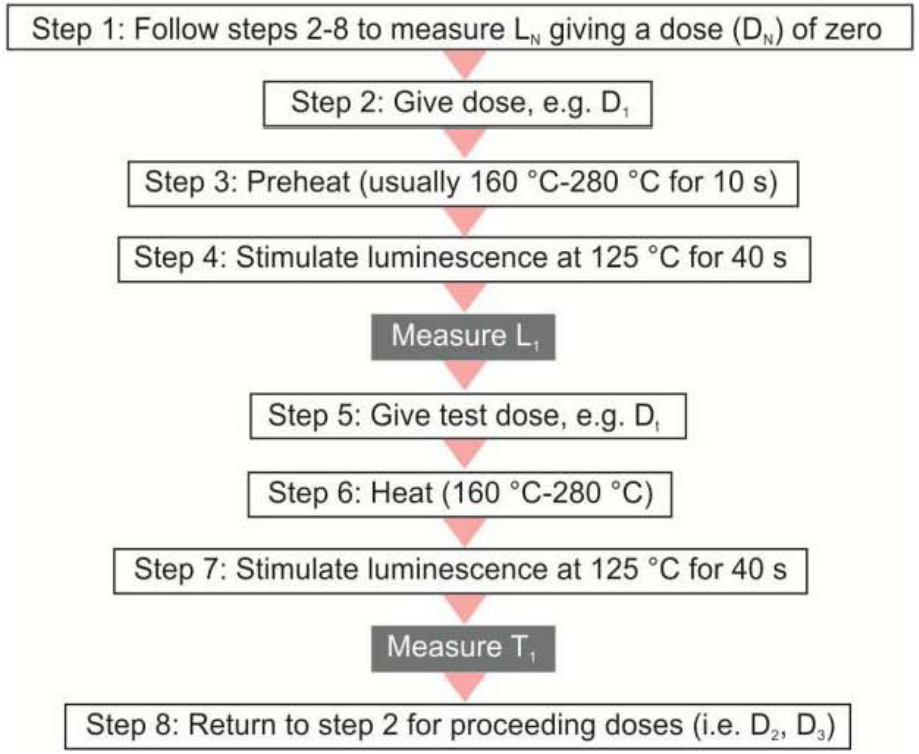


Fig. 3

Sediment Core: 5-20 m long; 1.2 m drives, 7 cm in diameter

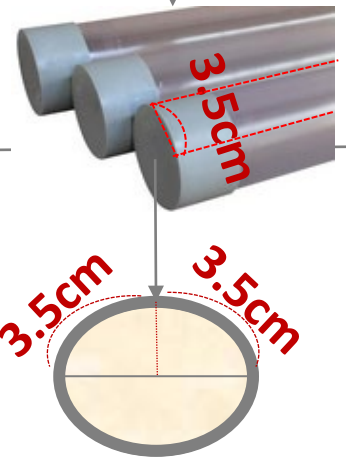
Split core length, crown cut 7 cm across the tube

Scrape off the upper 2 cm of surface to access undisturbed sediments

Apply 15% HCl to core-length to assess presence of carbonates

Describe sedimentology pedology and stratigraphy

- Compensated Depth
- Texture
- Munsell color
- Bedding
- Other pedo-sedimentary & biogenic features



Sample every 10 cm for carbonate by gasometry content and particle size Laser Diffraction

Locate OSL sample levels: C-horizon top and bottom; burial of soil

Figure 4

Fig. 4

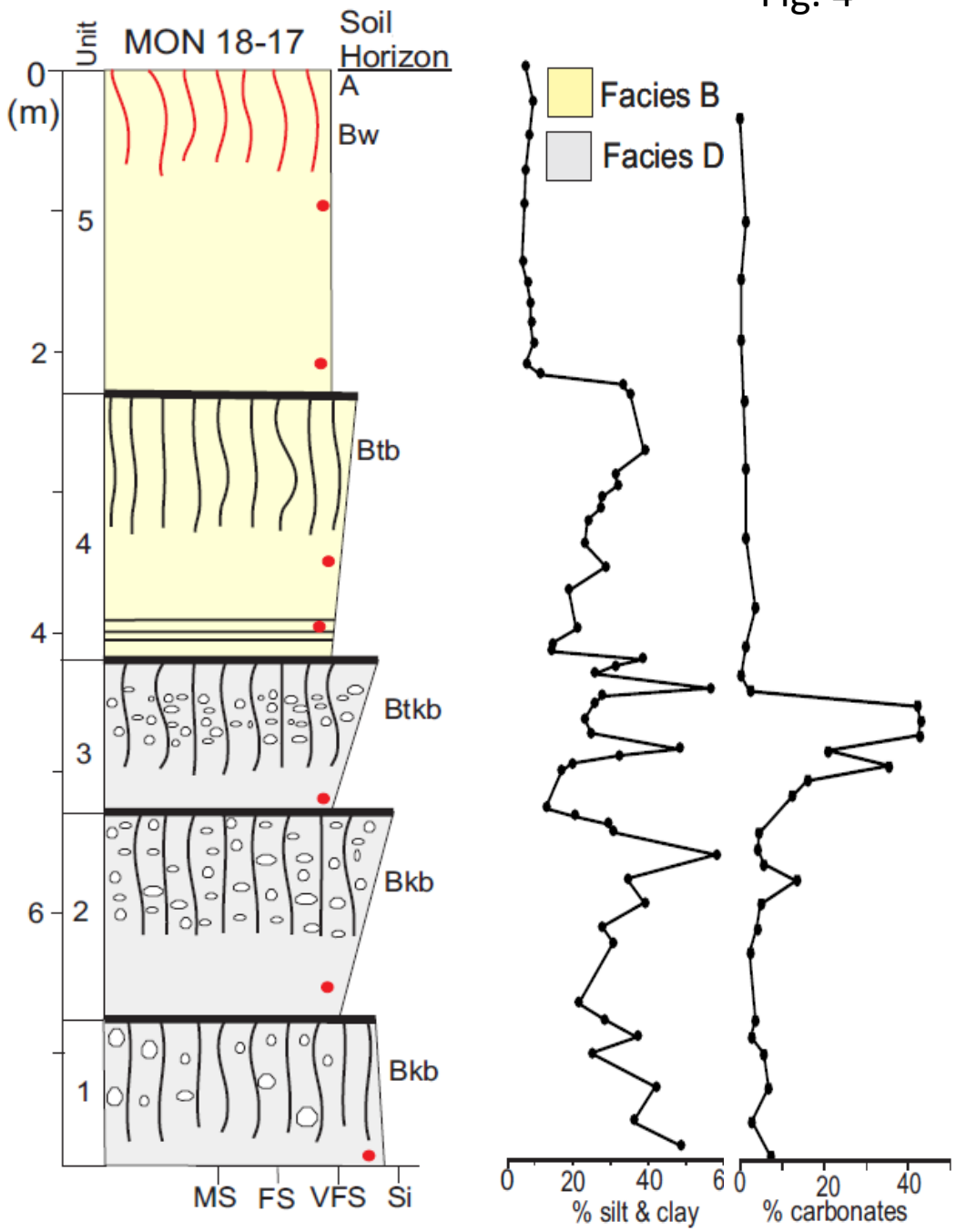


Fig. 5

Move core from the lighted Sedimentology Lab to the low-intensity (<1mW/cm^2) mono-spectrum (590 nm) illuminated OSL Lab

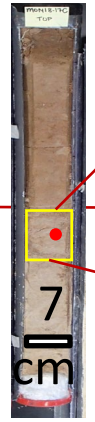
Moisten core face for cohesion. Remove the upper 1 cm at sample site to unlight exposed strata

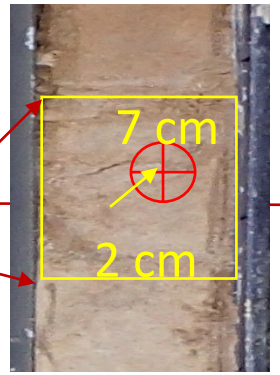
Score a 2 cm diameter circle on the core face to designate sample location

Extract light shielded sediment from the core. This sediment will be cleaned physically and chemically to isolate quartz grains for equivalent dose estimate.

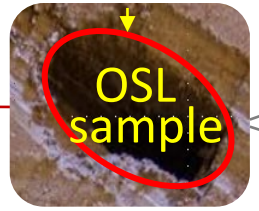
Use this sediment for elemental analysis for dose rate estimate

Fill sample cavity with aluminum foil to prevent side wall collapse. Moisten, wrap in plastic, and seal the core for archiving





Core: MON18-17. Drive B



Lab sample No.BG4653 Core MON18-17B-55cm

Figure 6

Click here to access/download;Figure;Figure 6.pdf

Sample Preparation for OSL Dating

2.3 Extract dose rate sediment 15 cm radius from OSL sample

2.4 Acquire sample from light shielded areas of the core. This sample will be physically and chemical cleaning for OSL

3. Extract mono-mineralogic fraction of Quartz

3.1 Removal of organic matter with 25% H_2O_2

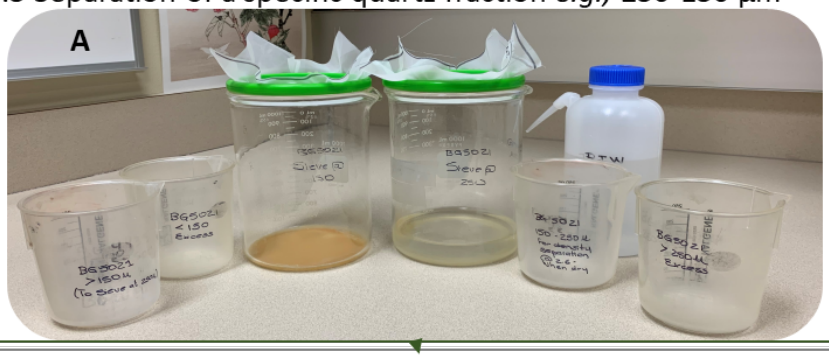
3.3 Removal of Calcium/magnesium Carbonate using 15% HCl

3.4 Removal of magnetic, paramagnetic, and diamagnetic minerals

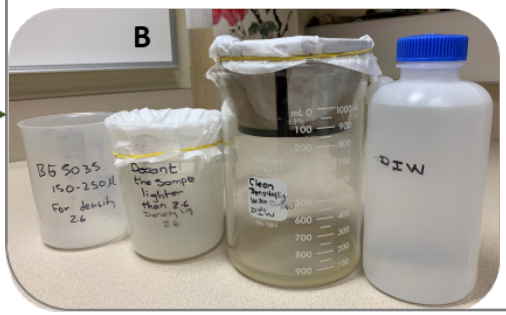
3.4.1 Dry magnetic separation with Neodymium magnets

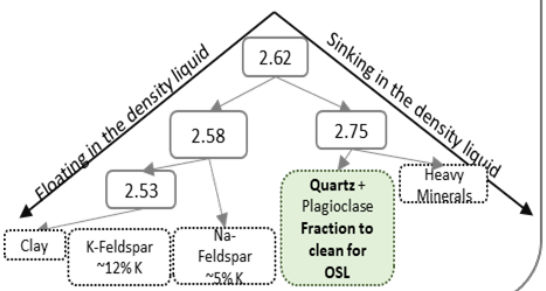
3.4.5 Wet magnetic separation using magnetic rods in sediment dispersed in 0.3% Na-Pyrophosphate solution

3.5 Separation of a specific quartz fraction *e.g.,* 250-150 µm



3.6 Heavy liquid isolation of quartz grains, using non-toxic heavy liquid Sodium Polytungstate (SPT-Na_6 ($H_2W_{12}O_{40}$)_H_2O) .





3.7 Etching of quartz grains with immersion in HF for 80 min.

3.8 Immersion of sediment in HCl to remove fluorite

3.9 Re-sieve through the smallest prior mess size (*e.g.,* 150 µm)

3.10 Label the sample for OSL analysis

3.11 Quantify the purity of the quartz aliquot of the original sediments

3.11.1 Use a 10 to 20X binocular microscope to check the purity of quartz

3.11.2 Use a RAMAN spectroscopy for mineral purity

3.12.2 Check for the IRSL emissions of quartz grains in OSL/TL reader

IRSL>10%

IRSL<10%

Repeat heavy liquids or HF digestions for 20-40 min.

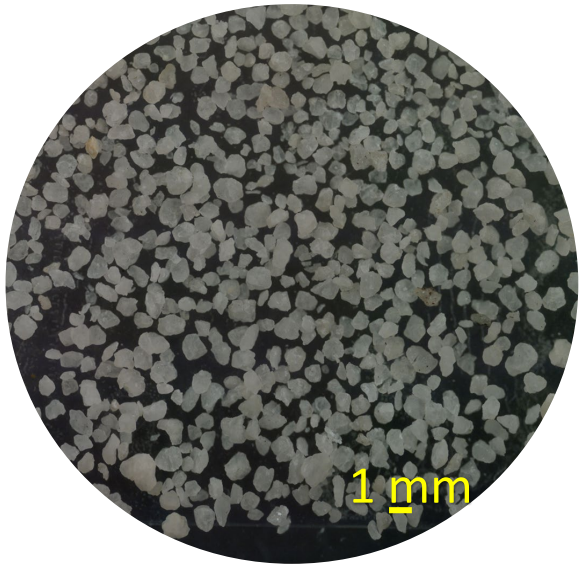
Prepare ~40 aliquots to perform OSL Analysis

Figure 7

Fig. 7

White Sands

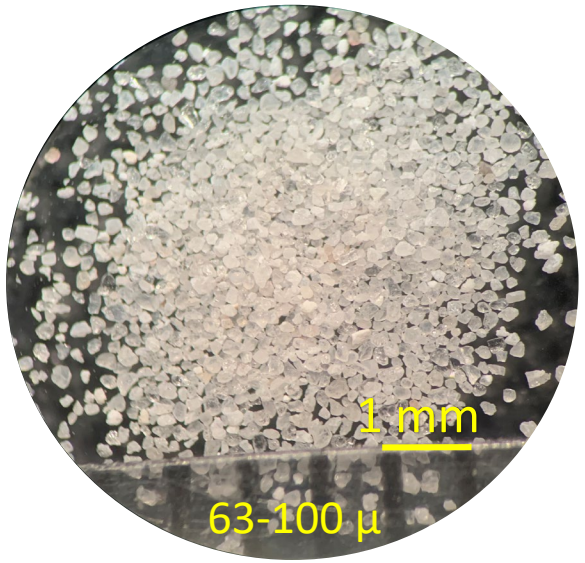
A. Raw Samples



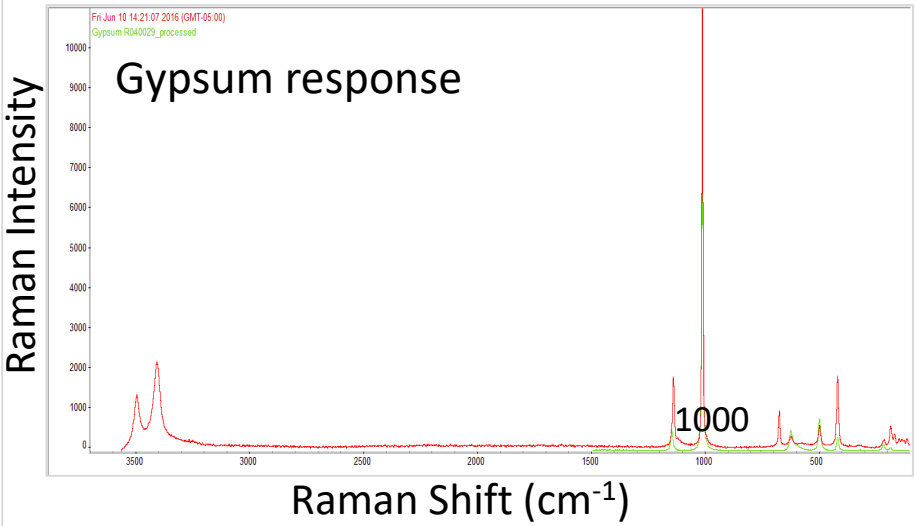
Needs recleaning to remove some gypsum

IR/Bl=9%

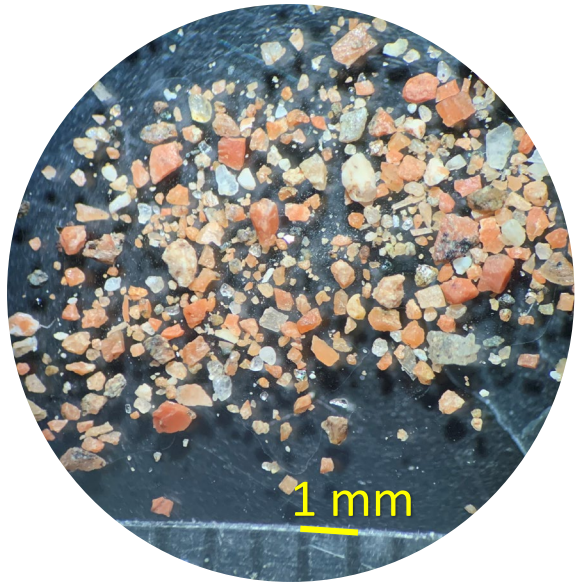
B. Processed Quartz fraction



C. Raman Spectroscopy Results

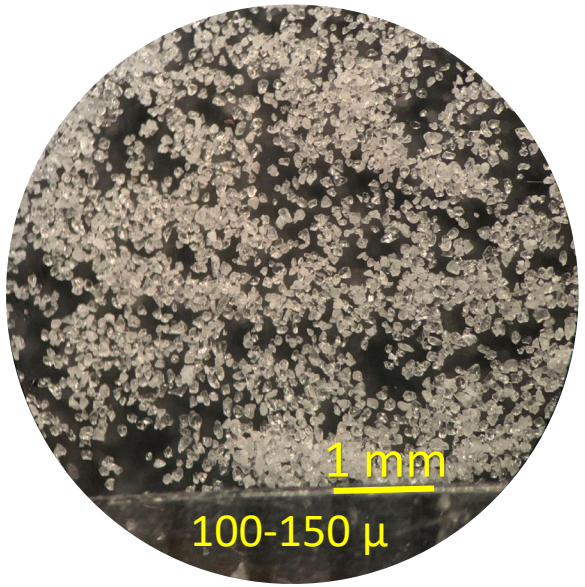


Mongolia



No further cleaning is necessary

IR/Bl=3.7%



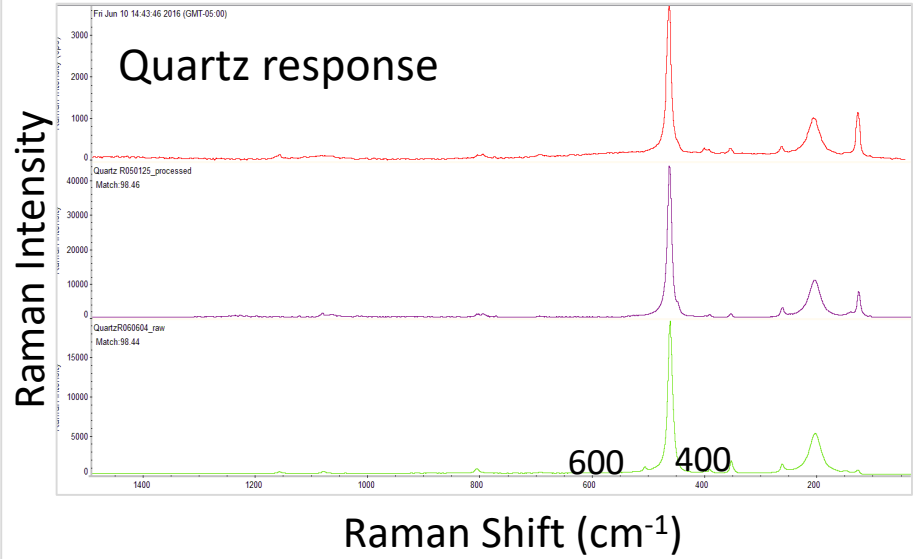
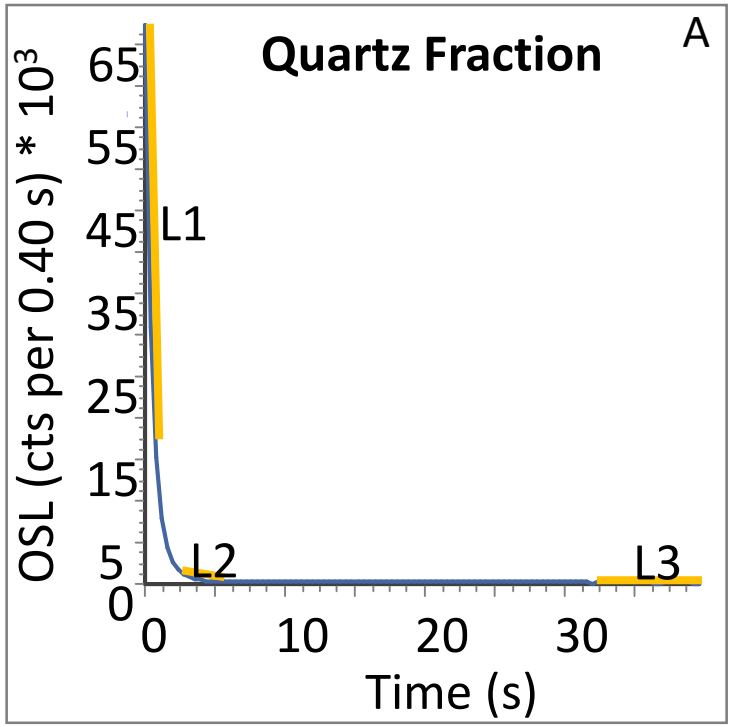
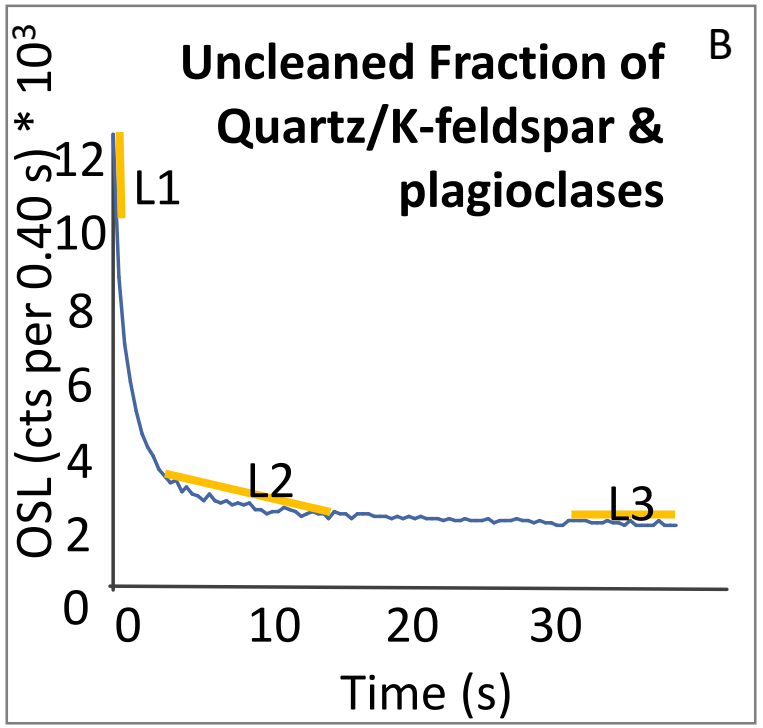


Fig. 8



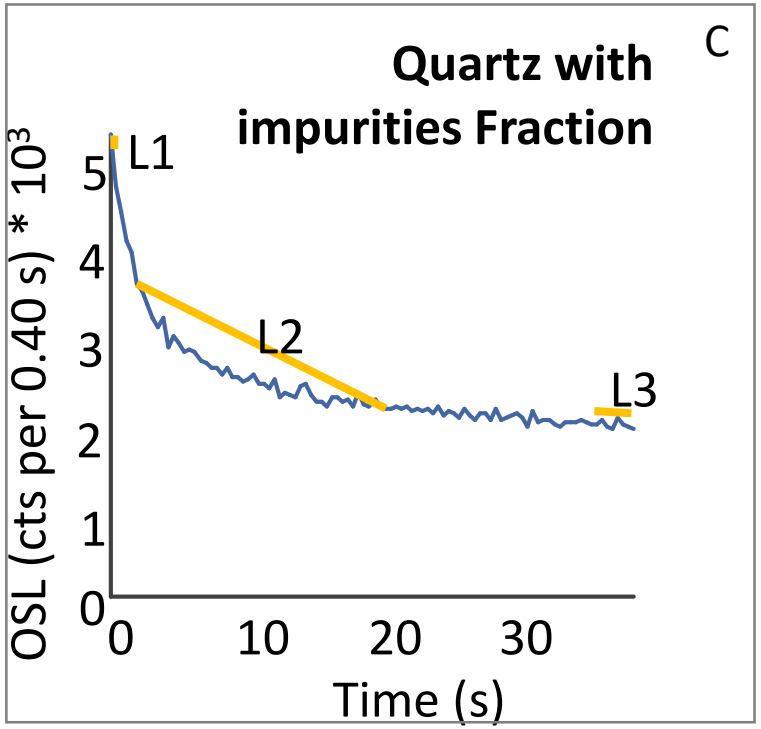
RR-18-OK D. 17: BG501X 250-150

Fast Component: 72



Asia-20 D.18: BG492X 150-250

Fast Component: 6.13



WA-20 D. 18: BG494X 250-150

Fast Component: 2.67

Response to Reviews

***Editorial* comments:**

Changes to be made by the Author(s):

1. Please take this opportunity to thoroughly proofread the manuscript to ensure that there are no spelling or grammar issues.

Response: We have carefully read and re-read this manuscript and hopeful rectified all typos, spelling and grammar issues.

2. Please ensure that all text in the protocol section is written in the imperative tense as if telling someone how to do the technique (e.g., "Do this," "Ensure that," etc.). The actions should be described in the imperative tense in complete sentences wherever possible. Avoid usage of phrases such as "could be," "should be," and "would be" throughout the Protocol. Any text that cannot be written in the imperative tense may be added as a "Note.

Response: We have labored to carefully edit this manuscript to the imperative voice. In places we have removed text, added phrases to notes and moved text.

3. The Protocol should contain only numbered action items that direct the reader to do something. Please move other details to the intro/discussion section as applicable. e.g., lines 184-195, 198-221, etc.

Response: We have condensed text from lines 184-195, but this text contained details that are necessary for full understanding of subsequent text and procedures. Text from lines 206-215 were moved into the introduction as suggested.

4. The Protocol should be made up almost entirely of discrete steps without large paragraphs of text between sections. Please simplify the Protocol so that individual steps contain only 2-3 actions per step and a maximum of 4 sentences per step.

Response: We have gone through the protocol and have simplified and clarified steps such that there are only 2-3 actions/step and four or less lines of text, as suggested.

5. Lines 184-195: Some of the details can be moved to the introduction while some can be converted to a note and moved under the step where directly applicable.

Response: Please see response 3.

6. Please add more details to your protocol steps. Please ensure you answer the "how" question, i.e., how is the step performed? This can be done by including mechanical action, button clicks in the software, knob turns, command lines, etc.

Response: We went through all the steps asking the "how" question which resulted in clearer directions. This was a useful exercise.

7. Please use the degree symbol from insert symbol feature of the word and do not use superscripted o.

Response: We have fixed this usage error.

8. Please expand all abbreviations during the first-time use.

Response: This usage has been applied throughout the manuscript.

9. There is a 10-page limit for the Protocol, but there is a 3-page limit for filmable content. Please highlight 3 pages or less of the Protocol (including headings and single line spacing between the steps)

that identifies the essential steps of the protocol for the video, i.e., the steps that should be visualized to tell the most cohesive story of the Protocol.
Response: We have highlighted in blue the three pages of filmable content, as requested.

10. Please include at least one paragraph of text to explain the Representative Results in the context of the technique you have described, e.g., how do these results show the technique, suggestions about how to analyze the outcome, etc. The paragraph text should refer to all of the figures. Data from both successful and sub-optimal experiments can be included.

Response: We have rewritten and structured the first two paragraphs of the discussion, which are shown below. In these two paragraphs we refer to all figures as requested, and show the outcome of the separation procedure in terms of grain purity, Raman spectra and OSL shine-down curve.

"The laboratory procedures outlined are focused to enhance the separation of pure quartz grains (700 to 50 µm size) needed for OSL dating without inadvertent light resetting in the laboratory (Figs. 1). A pure quartz separates, mineralogically and optically, is a prerequisite for applying SAR and TT-OSL dating procedures (Fig. 2). These procedures explain the necessary steps for effectively understanding and sampling of continuous sediment cores, avoiding zones of pedogenesis and diagenesis, retrieving unlight exposed sediments from cores (Fig.3 and 4); to isolate quartz grains for OSL dating protocols to constrain the timing of sediment deposition in the past ca. 500 ka (Fig. 5). The mineralogy of grains of the unprepared sample and prepared separates are assessed continuously through the preparation process to identify the contaminating mineralogy and actively assess the process of removal of unwanted minerals (Figs. 6 and 7). The quartz mineralogic purity is determined for a subset grains (100-400) through binocular microscopic inspection (10-20X) and by Raman spectroscopy. The use of this technology and prerequisite knowledge is vital to assess and confirm the needed purity (>99%) of quartz separations for OSL dating (Fig. 8).

The process for quartz separation is started with the removal of organic matter with H_2O_2 and then the subsequent purging of $Ca/MgCO_3$ with soaking in HCl. (Fig. 6). Subsequently, a size fraction is designated by sieving with disposable nylon mesh (e.g., 150 & 250 µm) which is necessary for calculating a dose rate values (in mGy/y). The purity of the quartz separate is enhanced by two density separations at 2.6 and 2.7 g/cc, the bounding density of quartz (Fig. 6). The subsequent soaking of sized grains in HF for 80 min removes non-quartz minerals. This acid treatment also etches the outer 10-20 µm of grains to remove the alpha-dose affected area, simplifying dose rate calculations (Fig. 6). The purity of the quartz separate is never assumed, but assessed through binocular microscopic inspection, and Raman-based measurements at the end of grain separation. Density separations and/or HF treatment can be repeated to rid the separate of contaminating grains if a representative aliquot contains > 1% non-quartz grains, particularly feldspar minerals (Fig. 7). We have repeated this quartz purification procedure up to four times with quartz contents of < 15% to render shine down curves with a fast ratio of > 20, characteristic of pure quartz (Fig. 8)."

11. Please include figures or tables in the Representative Results showing the effectiveness of your technique backed up with data. Please discuss all figures in the Representative Results. However, for figures showing the experimental set-up, please reference them in the Protocol.

Response: There is now a section on Representative Results on P. 18. In this section we discuss a metric (fast ratio) to assess quality of luminescence emissions that is representative of pure quartz, which is

shown now in Figure 8. Also, in Figure 8 are positive and negative results that may occur after following this protocol because of contamination by non-quartz minerals or feldspathic inclusions in quartz.

12. Please include all the Figure Legends together at the end of the Representative Results in the manuscript text.
Response: Moved figure captions to this location, as requested.

13. As we are a methods journal, please ensure that the Discussion explicitly cover the following in detail in 3-6 paragraphs with citations:
a) Critical steps within the protocol: *This is now outlined in paragraph 2 in the discussion on p. 18.*
b) Any modifications and troubleshooting of the technique: This covered in the representative results section (p. 18) with the discussion of the fast ratio as a tool for assessing quartz purity.
c) Any limitations of the technique: Yes, we discussion on p. 18 pernicious contamination by feldspathic inclusions of quartz which is an obstacle for OSL dating.
d) The significance with respect to existing methods: This is discussed in the final paragraph on p. 18 in terms of significance for single grain and TT-OSL dating.
e) Any future applications of the technique: Yes, we now discuss future applications at the end of paragraph 1 on p. 18 with text: "These protocols with modifications can be used to separate and confirm purity other minerals for OSL dating such as k-feldspar, plagioclase; and olivine and pyroxene for other planetary applications."

14. Please include an Acknowledgements section, containing any acknowledgments and all funding sources for this work.
Response: *We have added an acknowledgement statement after the Representative Results section.*

15. Please include a Disclosures section, providing information regarding the authors' competing financial interests or other conflicts of interest. If authors have no competing financial interests, then a statement indicating no competing financial interests must be included.
Response: A disclosure statement has been added after the acknowledgements.

16. Please sort the materials table in alphabetical order.
Response: This change to the table has been completed.

17. Please obtain explicit copyright permission to reuse any figures from a previous publication. Explicit permission can be expressed in the form of a letter from the editor or a link to the editorial policy that allows re-prints. Please upload this information as a .doc or .docx file to your Editorial Manager account. The Figure must be cited appropriately in the Figure Legend, i.e. "This figure has been modified from [citation]."

Figures 1 and 2 are from Forman et al (2015) and can be reproduced as part of Rightlinks Copyright Clearance Center.

Reviewers' comments:

Reviewer #1:

I am a little confused about this submission. Is it supposed to be the text that accompanies a video? It makes a little more sense if that is what it is, because it reads like an instruction manual. The video then would show how to do the tasks the manuscript describes. I am also unclear about the intended

audience. The only people who would need this instruction are luminescence practitioners. I suspect these people already know how to do this. The general protocol is fairly standard. Perhaps the paper is for students who want to work in a lab, but at least the students who work in my lab get hands on experience. Or perhaps these instructions are for people who want to set up a lab, but again my advice to those people would be to spend some time in an established lab to get the hands-on experience. Most people I know in this situation do this. Or perhaps it is intended as a quality control, so that all labs can affirm that they follow best practices, although there might be disagreement about what the best practices are. There are things in these instructions that most labs do not do, like Ramon Spectroscopy - and I am not sure many practitioners think this is necessary.
Response: I understand and appreciate this reviewer's uncertainty on the purpose of this manuscript and accompanying video because no such publication currently exists. We are plowing new ground with this contribution. We hope that this manuscript will be helpful for beginning students, post-doctoral scholars and faculty to better refine OSL methods in written procedures and as hands-on demonstrations, with this and future JOVE contributions. We believe that scholarship with documented laboratory procedures, as published by JOVE, will improve analytical geochronology.

I also question to some extent the need to produce high purity quartz during sample preparation. Such separation can also take place during the luminescence measurements. If one does single-grain analysis, it is quite easy during the luminescence measurements to detect and remove feldspars. With a little more effort, one can also select only those grains that display mainly the quartz fast component. Multi-grain aliquots are more of an issue. In fact, I find it very difficult, even following these procedures, to separate out feldspars. Some of them are found as inclusions within the quartz grains, and unless you measure single grains, you are often facing feldspar contamination. When I measure quartz signals with multi-grain aliquots, I find pulsed OSL to be the most useful. Because the charge movements are much faster in feldspar than quartz, one can with selection of proper pulse width effectively eliminate the feldspar signal.
Response: We agree with the reviewer's statements regarding the difficulty of isolating quartz and feldspar grains. However, we believe that these protocols offer an enhanced methodology using petrographic and Raman techniques to make such separations nearly routine. The pulse method sounds intriguing and would like to attempt on mixtures with varying feldspar contamination of quartz, which we have quantified. Unfortunately, the reviewer does not provide a reference for this approach nor a set of protocols; another reason for JOVE articles for geochronology.

Many of the procedures described reflect what the Baylor lab does. I do not object to their procedures for the most part. In fact they are very thorough. But the paper should say that this is Baylor's procedure, not the only possible procedure. Other labs may use different procedures that are just as effective. I mention some of these in the specific comments.
Response: We agree with this reviewer's statement, but nowhere do we state, nor imply that this is only method or approach for separating or testing quartz. This is the implicit assumption of this reviewer. To belie this "definitive procedure" concern we have now added text at lines 191 to 194 "These procedures are used in the Baylor Geoluminescence Dating Research Laboratory reflecting analytical experience over thirty years, and are not definitive methods, with suitable variations used by other labs. This is not a static protocol and welcome modifications and additions for improvement."

Manuscript Summary:
Specific comments:
Line 64 - It is not clear how much defect creation is caused by ionizing radiation, at least over the time

scales used in dating. If producing free electrons and creating defects are both caused by ionizing radiation, this would surely complicate dating.
Response: The sentence has been modified as shown in red to read: Common silicate minerals on Earth, like quartz and potassium feldspar, contain lattice-charge defects formed during crystallization and with some defects from subsequent exposure to ionizing radiation, which results in geochronmetric potential.

Lines 109-112 - This sentence, which is kind of garbled, should be reworded.
Response: Yes, we agree with the text clarified as shown in red: The denominator is the Dose rate, defined by contributing α, β, and γ radiation, originating from the radioactive decay of daughter isotopes in the ^{235}U, ^{238}U, ^{232}Th decay series, ^{40}K, and with lesser contributions from the decay of ^{85}Rb and cosmic sources on Earth.

Lines 119-121 - There are many other alternatives to measuring dose rate than the two mentioned. This should be acknowledged.
Response: This sentence has been modified as shown in red: One method for determining the radioisotope concentration in sediments is by inductively coupled plasma-mass spectrometry, assuming secular equilibrium in the U and Th decay series." It should be noted that in the succeeding sentence we also discuss an alternative method, gamma spectrometry which belies this comment.

Line 127 - "Compensates" is not the right word. Maybe "is accounted for by".
Response: We have replaced "compensate" with "account" as the reviewer suggested.

Lines 859-860, Figure 1 caption. The last sentence is confusing as written. Please reword.
Response: Yes, that sentence was unclear and have now split into two and reworded: "(f) Shows sampling without light exposure. The resultant measured natural OSL is followed by a normalizing test dose (L_n/T_n) which is equated to the regenerative dose curve to yield an equivalent dose (D_e) (Forman, 2015).

Lines 147-151 - It is not absolutely necessary to know the mineralogy of your sample before processing. Most labs probably do not do this if they do not have easy access to the equipment.
Response: Respectfully, there is no basis, nor scholarship to support this statement. It is an absolute imperative to know the mineralogy because of the different dose response and anomalous fading characteristics. This statement demonstrates the need to educate the luminescence dating community on the protocols for separating and confirming pure quartz, with this contribution.

Lines 153-159 - The IR depletion test described here comes after the sample preparation, not after the mineral-spectral analysis (if that is done). This is mentioned at the end of the paper. The idea of the IR depletion test is to make sure your sample preparation has succeeded in removing feldspars. It also is not necessary as a separate step if you can separate out feldspars during the luminescence measurements. For example, in single-grain dating, and often in multi-grain dating, the IR depletion test is built into the SAR protocol.
Response: We agree with this statement. However, we incorporate IR tests prior to dating to assess quartz purity for aliquot or single grains, not after. This prior test identifies potential contamination, allows, further chemical treatments to remove errant minerals, and improves quartz-based luminescence emissions and the reproducibility in dating.

Lines 161-167 - This paragraph is not germane to the subject of the paper and can be eliminated.

Response: We agree and have removed these lines.

Lines 176-7 - You don't actually talk about petrographic imaging and Ramon spectroscopy all that much. If these were to be the main improvement of standard preparation protocols, the focus should have been on them. Especially if you want to convince the reader that these are necessary.
Response: The use of petrographic and Raman spectrometry approaches is discussed in section 3.11 at the appropriate place to assess quartz purity. Training in petrographic analysis and use of a Raman spectrometer is beyond the focus of this protocol. We advocate broad training in geosciences, physics and chemistry for those interested in geochronology. We have now added in section 1.3 in the third step the third: " Discuss interpretation of sedimentary environments with others in the research group." We believe that through discussion of core record with others in the research group deficiencies in interpretation can be rectified.

Lines 184-185 - While coring is important, it is not the only way to collect samples for luminescence. Most samples are collected by just pushing a tube into an exposed profile.
Response: Yes, we agree with this statement and there are other ways to collect samples. To address this comment we have added the phrase (in red) to the indicated sentence "This section presents the procedures to separate a nearly pure (>99%) quartz fraction from polymineral sediments taken from long (5-20 m) sediment core and are equally applicable to individual tube-like samples collected from outcrops.

Line 191 - Not everyone uses a sodium bulb. In fact, finding suitable housing for them that will work in the lab can be difficult. Most labs use some kind of red bulb with appropriate filters.
Response: We understand the basis of this comment. Though, Na-vapor bulbs and housings are readily available on the secondary market. Yes, there are suitable replacements from LEDs to bulbs with red or orange filters. This is now indicated in the mentioned sentence (in red): "The second and third components are undertaken with illumination by a sodium vapor bulb, equivalent LEDs or bulbs with red to orange filter."

Lines 198-206 - Somewhere in this paragraph you should cite Nelson et al. (2019, Methods and Protocols), which deals with coring in depth.
Response: Thank you for pointing out this important paper which is now cited and in the references.

Lines 215-220 - These steps require a trained geologist. Not every luminescence person knows how to make such descriptions. These steps and others (particle size analysis, for example) described later on might be useful but not necessarily essential for successful dating.
Response: One purpose of this contribution is to underscore the need of new knowledge content and technology to improve sample selection and mineral specificity, and thus quality of generated OSL ages. Yes, a horse with a buggy is a suitable conveyance but an electric car is cleaner and faster.

Lines 231-233 - Some of these precautionary steps are lab specific. For example, safety folks have ascertained that our sash can be ¾ open and still be effective.
Response: The precautionary steps are those approved by Baylor University's Environmental Health and Safety staff and are a prerequisite to prevent accidents with caustic acids and other reagents. We have worked closely with our chemical safety staff to develop the safest possible procedures because we are

working in limited light settings. Thus, the precautionary notes are necessary for those with less training and experience.

Lines 254-260 - This is repeat of something just said. Repeated again in lines 376-382.
Response: This modest apparent repetition is needed to provide utmost clarity in the sequence of steps.

Lines 314-319 - Labelling is certainly critical, but how it is done depends on laboratory preferences. Your method is just an example of what can be done.
Response: Yes, we agree with statement. Thus, we have added in red: "As an example, a sample is assigned a consecutive laboratory number (e.g., BG4966)…"

Lines 336-340 - PPE requirements will vary, according to specifications established by a university's environmental health and safety department. Our department has fairly stringent restrictions but it does not require shoe coverings, for example. (And it does not allow flushing hydrogen peroxide down the sink - line 366.).
Response: The PPE prescribed is from Baylor University laboratory safety staff to minimize risks for student exposure and accidents. H_2O_2 is flushed through running water and this reagent readily disassociates to H_2O and O, which are non-toxic. Again, this is the prescribed manner for the disposal of this reagent, as specified by Baylor University.

Lines 348-357 - This is again a repeat of information already given.
Response: Redundancy in safety precautions are needed to stress safe comportment in the laboratory.

Line 361 - Where does the 40°C threshold come from? What about 50°C or even somewhat higher? Some people use preheats of 160°C for 16 hours.
*Response: We set a low threshold for heating of the sample during preparations to obviate fully and temperature related sensitivity changes or inadvertent partial resetting of the signal. We have modified the wording to now read: "*The sample should not be heated above 40° C. Higher temperatures may cause partial resetting of the luminescence signal and sensitivity changes detrimental to dosimetric measurements."

Line 372 - One should add initially only a small amount (<1 ml) of HCl and see what happens before adding more. 30 ml, even if added slowly, can cause a large reaction if there are a lot of carbonates in the sample and risks having your sample boil over.
Response: This is an useful consideration and have change the procedure to read: "Add slowly 30 ml of 15% HCl for each 5 g of sediment in a 250 ml beaker to react $Ca/MgCO_3$ disseminated in the sample. Initially add ≤ 1 ml to assess effervescences and modulate further HCl additions to control better reaction."

Line 389 and beyond - The procedure for removing magnetic materials is interesting and of value, but I would point out that most labs do not do it and assume that a lot of such materials are removed in density separation.
Response: Unfortunately, we wish that density separations rid the sample of magnetics but the non-spheroidal, elongate shapes, confounds settling velocities.

Line 429 - Wet sieving is not necessary for very sandy sediments. Dry sieving is easier and just as effective.

Response: We respectfully disagree, particularly for small volume samples from cores. Wet sieving is advised to obtain maximum fraction weight with a known size. This may be true for larger volume samples, but wet sieving is still advised, for certain size separation.

Lines 500-510 - We use stirring and centrifuging to make the separation. It seems to work pretty well.
Response: Yes, centrifugation is an alternative approach but with transfers from beaker to centrifuge tubes and repeated decanting sample is lost and is ill advised for small volume samples from sediment cores.

Lines 515-517 - How do you keep the settled sediment preserved while pouring off the suspended part? We use liquid nitrogen to freeze the settled part. Much more effective than just carefully pouring off, in my experience.
Response: This is an interesting analytical approach to freeze the heavier fraction in situ with liquid nitrogen. I would like to see a detailed protocol for this approach. However, there are attendant safety issues in using this very cold substance (-196° C) particularly with student involvement. We have no problems in decanting carefully the supernatant and once to achieve maximum yield. Decanting a supernatant is an easy skill to teach to students.

Line 596 - An 80 minute etch with HF is longer than what many labs use (40 min). It depends on the density of HF, I suppose, but the authors do not specify that.
Response: *We recommend a 80 minute emersion in HF instead of 40 min because too often the 40 min emersion need to be repeated to rid the sample of non-quartz minerals. We have indicated the concentration of HF (36%) used, which is the undiluted reagent grade.*

Reviewer #2:
Manuscript Summary:
The procedure follows a common and well-known set of sample preparation procedures for extraction of quartz grains. The protocol section is unusually detailed and informative as far as chemical reactions. Section 3.5 gave me the most questions. All of the figures were quite well drawn and helpful to what I assume will be the video. The flowcharts were also quite informative and will provide a ready made QA/QC method should someone want it.

Major Concerns:
There were frequent issues with grammar, etc which I shall leave for the Journal Editors. For example: line 423 3.5 "separation a specific grain-size fraction" which I assume should read "separation for a specific grain-size fraction". These were pretty common.
Response: Yes, we agree there were errors in grammar and presentation, which we have remedied during the revision process.

Minor Concerns:
1).The way it is written it appears that the first thing that is done with a core sample if to sieve it (Sediment samples are then subjected to a series of physical separations to obtain a certain grain-size interval (e.g., 150-250 µm) and then to use magnets to remove the iron rich grains. I'm not sure why this would be before the grains are cleaned in acid and peroxide but every luminescence lab has their own procedure and I won't quibble.
Response: We removed the magnetics first because these iron-based heavy particles can chemical interact with HCl and H_2O_2 competing with steps to remove carbonate and organic matter. Lastly, high density magnetic particles also interfere with density separations for quartz.

2). I admire the use of Raman spectroscopy to check for quartz purity and feel this is a useful addition to the procedure. I am not sure what the RRUFF System Database is but like the idea of some sort of standard spectra to compare with.
Response: Thank you for this positive comment!

3). Personally, I would move the paragraph 130-140 down with the paragraph 161-167 since the are speaking of the SAR procedures and that seems the most focused way to keep track of the measurement system without wandering back to the quartz purification techniques.
Response: We agree and have moved these paragraphs together in response to the editor's suggestion.

4). Section 3.5 gave me the most questions. I do not know why you would add the density solution at this point (line 445) if you are simply sieving to a certain grain size. Because it appears the technician then washes the density solution to reclaim it (sediments are dried). It wasn't clear to me why this step needed the density liquid treatment unless it was an overabundance of caution against the one possible feldspar grain or was it to keep clays from flocculating?
Respond: I may not understand this comment. We wash the grains with a standard dispersant solution of Na-pyrophosphate to keep clay and silt particles from adhering to grains and to isolate better each grain for density separations.

Response to Editorial comments:

1. The manuscript has been formatted to remove personal pronouns (e.g., "we", "you", "our," etc.) to fit the journal standard. Please note that some comments to be addressed are included in the attached manuscript file.
Response: This change is fine, and we have gone through the manuscript and clarified at your prompts.

2. Please revise the following lines to avoid previously published work: 35-37, 60-61, 62-64, 66-79, 99-109,123-131, 152-162.
Response: We have in most places reworded phrases, but not numeric values or equation, which should not be changed. It should be noted that the sentences with the requested changes are from S. L. Forman's previous publications of the basics of OSL dating, which has been clearly cited. These changes are unnecessary and often degrade the quality of text.

3. In the current version, the figures are structured into the discussion (line 734-762). Please move them/ rename the title as "Representative Results" and ensure that the section "Discussion" explicitly the critical steps within the protocol, any modifications, and troubleshooting of the technique, any limitations of the technique, the significance concerning existing methods
and any future applications of the technique. Restructuring the "Representative results" and the "Discussion" section will be sufficient.
Response: We find this comment cryptic and difficult to understand what changes are needed. I have reordered paragraphs and now have one concluding section "Representative Results and Discussion."
4. Figure legends: Please include a brief description of figures 3 and 5.
Response: Done and included
5. Figure 4: Please define the terms A, Bw, Btb, Btkb, MS, FS, VFS, Si, etc., shown in the figure in the figure description.
Response: Done and included
6. Figure 7: Please ensure that the graphs include the X and Y titles. The first graph of panel C has only half of the parenthesis in the Y-axis. Please revise them to be consistent with the other graphs of the panel.
Response: Done and included
7. Figure 8: Please use uppercase letters to label the figure.
Response: Done

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Luminescence Dating in Paleoseismology

Author: Steven L. Forman
Publication: Springer eBook
Publisher: Springer Nature
Date: Jan 1, 2015

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Expected publication date	Sep 2021
Order reference number	SLF.Jove1
Portions	Figures 1 and 2
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