User Guide for Luminescence Sampling in Archaeological and Geological Contexts

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INTRODUCTION

Use of geochronologic techniques has become a cornerstone of archaeological research, Quaternary geology, and paleoenvironmental reconstruction. **Luminescence** dating, including **optically stimulated luminescence** (**OSL**) dating (Huntley et al. 1985) and **thermoluminescence** (**TL**) dating (Aitken 1985), can be an important tool for archaeologists and geologists, as the technique is widely applicable to diverse archaeological settings and depositional environments (e.g., see reviews by Duller 2004; Feathers 2003; Jacobs and Roberts 2007; Lian and Roberts 2006; Liritzis et al. 2013; Preusser et al. 2008; Rhodes 2011; Rittenour 2008; Roberts 1997; Wintle 2008). The number of publications reporting luminescence results has increased substantially since the development of **single-aliquot** and **single-grain dating** methods (Figure 1). Due to the increased demand for luminescence dating, we present a sampling guide for archaeologists and geologists who wish to apply luminescence dating to their research. (*Note*: Terms that appear in bold are defined in the glossary in the sidebar.)

ABSTRACT

Luminescence dating provides a direct age estimate of the time of last exposure of quartz or feldspar minerals to light or heat and has been successfully applied to deposits, rock surfaces, and fired materials in a number of archaeological and geological settings. Sampling strategies are diverse and can be customized depending on local circumstances, although all sediment samples need to include a light-safe sample and material for dose-rate determination. The accuracy and precision of luminescence dating results are directly related to the type and quality of the material sampled and sample collection methods in the field. Selection of target material for dating should include considerations of adequacy of resetting of the luminescence signal (optical and thermal bleaching), the ability to characterize the radioactive environment surrounding the sample (dose rate), and the lack of evidence for post-depositional mixing (bioturbation in soils and sediment). Sample strategies for collection of samples from sedimentary settings and fired materials are discussed. This paper should be used as a guide for luminescence sampling and is meant to provide essential background information on how to properly collect samples and on the types of materials suitable for luminescence dating.

La datación por luminiscencia proporciona una estimación directa de la edad del último momento en el que el cuarzo o los minerales de feldespato se expusieron a la luz o al calor y que se ha aplicado exitosamente a depósitos, superficies rocosas y materiales expuestos al fuego en distintos contextos arqueológicos y geológicos. Las estrategias de muestreo son diversas y pueden ser individualizadas dependiendo de las circunstancias locales, aunque todas las muestras de sedimentos deben incluir una muestra segura que no haya sido expuesta a la luz y material para calcular la tasa de la dosis. La exactitud y precisión de los resultados de la datación por luminiscencia están directamente relacionadas con el tipo y la calidad de los materiales muestreados y los métodos de recolección de muestras en el campo. La elección del material de estudio para su datación debe incluir las siguientes consideraciones en torno a la idoneidad de poder reposicionar la señal de luminiscencia (blanqueador óptico y térmico), la capacidad de caracterizar el ambiente radiactivo que rodea la muestra (la tasa de la dosis) y el que no exista evidencia de una alteración posdeposicional (bioperturbación en suelos y sedimentos). Se discuten las estrategias de muestreo para la recolección de muestras de contextos sedimentarios y de materiales expuestos al fuego. Este artículo debe utilizarse como una guía para el muestreo por luminiscencia y tiene la intención de proveer información básica de cómo recolectar muestras y sobre los tipos de materiales apropiados para la datación por luminiscencia.

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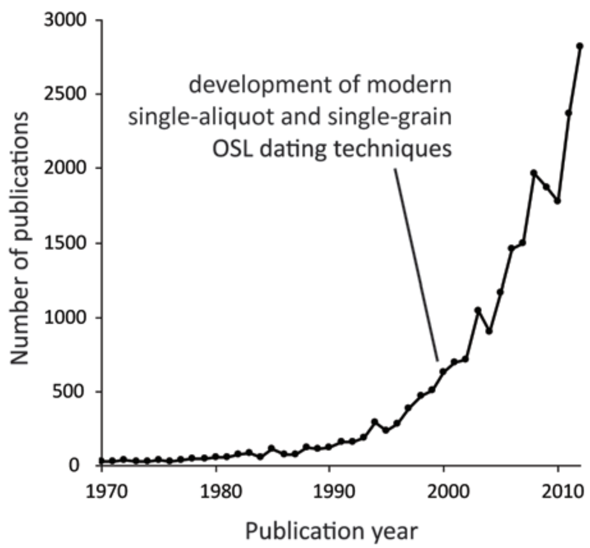

FIGURE 1. Number of publications per year, identified using Google Scholar and the search term "luminescence dating."

BACKGROUND

Luminescence dating provides an age estimate of the last time minerals, such as **quartz** and **feldspar**, were last exposed to sufficient light or heat to reset a prior luminescence signal (Aitken 1998). After removal from heat or from sunlight, electrons accumulate in defects in the crystal lattice of minerals by exposure to **ionizing radiation** (environmental **dose rate, D_R**) from radioisotopes in the sample and the surrounding sediment, and through incoming cosmic radiation. Subsequent exposure to light or heat causes trapped electrons to be evicted and to release a photon of light in the process (luminescence). The intensity of the resultant luminescence signal is directly proportional to the radiation received, the environmental dose rate, and the time since last exposure to heat or light.

Luminescence ages are calculated by dividing the amount of radiation the sample absorbed since exposure (termed the **equivalent dose, D_E**, in Grays (Gy) where 1 Gy = 1 Joule/kg) by the environmental dose rate (D_R, in Gy/ka):

$$\text{Age } (ka) = \frac{D_E\,(\text{Gy})}{D_R\,(\text{Gy } ka^{-1})}. \tag{1}$$

TL dating uses heat to stimulate the luminescence signal, whereas OSL dating uses light. Blue-green light is typically used for quartz OSL dating and **infrared stimulated luminescence** (**IRSL**) is used for feldspar dating. While both TL and OSL can be used to date heated materials, sedimentary deposits—including cultural deposits such as middens, canal spoils, and earthen architecture—are predominantly dated using OSL techniques. Recent technological advances and the development of single-aliquot (Murray and Wintle 2000; Wallinga et al. 2000) and single-grain dating capabilities (Bøtter-Jensen et al. 2000; Duller et al. 1999) have greatly expanded archaeological and geological applications of OSL dating in the last several decades (Figure 1). Precision of OSL ages are often better than 10 percent of the age and TL dating is often near 15–20 percent of the age, but both are dependent on uncertainties related to dose-rate measurements and D_E scatter between individual aliquots/grains (e.g., Murray and Olley 2002).

Luminescence dating provides a number of benefits over other available techniques for dating cultural materials and Quaternary deposits. The maximum age range exceeds the ca. 40,000-year limit of radiocarbon dating and is also applicable to very young (historic) samples. The typical age range for luminescence dating is between ca. 100 and ca. 200,000 years. However, the actual maximum and minimum age range is sample-specific and dependent on the maximum attainable signal of the target minerals (**saturation** level) and the dose-rate environment. High dose-rate environments may limit the upper age range attainable for older samples (> 10,000 years) but can allow for greater signal resolution for younger samples (< 1,000 years).

CONSIDERATIONS FOR SEDIMENT SAMPLE SELECTION

The successful application of luminescence dating is primarily dependent on the choice of sample materials and collection methods. Site selection should always take into account both the numerator (D_E) and denominator (D_R) in the age equation (1). Given the number of considerations involved in sample selection, discussed below, consultation with a geologist, geomorphologist, or geoarchaeologist is recommended when considering luminescence dating of sediments at archaeological sites. While the collection methods described below are relatively simple, contacting the analyzing laboratory prior to collection is also recommended, especially in cases of rock art and architectural features, which are difficult to sample and challenging to date.

Mineral and Grain-Size Composition

Sediments (including cultural deposits) sampled for luminescence dating must contain quartz or feldspar minerals that are very-fine silt (7–11 μm) or very-fine to fine-grained sand (63–250 μm), due to factors related to the calculation of the D_R (Aitken 1998). **Coarse-grained dating** (63–250 μm) using small-aliquot (tens to hundreds of grains) and single-grain dating of purified quartz or feldspar have several advantages over poly-mineral **fine-grained dating** (4–11 μm). Single-grain dating not only is useful for detecting **partial bleaching** or post-depositional mixture, but also allows for selection of grains most suitable for dating (in terms of signal **sensitivity**, intensity, stability, and reproducibility). Fine-grain dating employs a poly-mineral fraction and is typically used where coarse grains are not available (i.e., ceramics or fine-grained sediments). Due to the difficulty of physically separating quartz and feldspar minerals in very fine silt and the greater intensity of the feldspar minerals in response to infrared-stimulation in comparison to quartz, poly-mineral dating is commonly accomplished by IRSL dating of the feldspar signals. Fine-grain IRSL dating has its advantages for dating ceramics and lithics, where the external D_R environment can be complex or unknown. Often, clasts or artifacts are juxtaposed with other cultural features or sediments that are thinly bedded at archaeological sites, creating a heterogeneous external

D_R environment and variability in grain dosing. Moreover, for museum specimens the external D_R is typically unknown. With fine-grained dating of artifacts, most of the ionizing radiation comes from within the artifacts themselves, such that the potentially large external D_R uncertainties are minimized.

Mineral selection will largely depend on the estimated age, abundance, and properties of the quartz and feldspar grains within the cultural material or deposit. In most settings, quartz is preferred to feldspar due to its relatively rapid removal (bleaching) of a previously acquired signal (Godfrey-Smith et al. 1988). While generally dependent on the source geology (for both constituents of pottery and Quaternary sediments), the luminescence signal intensity of quartz (sensitivity) is typically weaker and more variable than feldspar. For example, some quartz samples have 30–50 percent of the grains producing a measurable signal while in others only 1–5 percent of the grains may luminesce (Duller et al. 2000). Additionally, quartz saturates at a lower acquired dose than feldspar, which limits the maximum age attainable. While the luminescence signal in feldspar is stronger, it is also commonly unstable and decays over time, a condition termed anomalous fading (Wintle 1973). Therefore, analysis of feldspars requires calculation of both the D_E and its fading rate (Auclair et al. 2003; Lamothe et al. 2003) or use of modified techniques (e.g., Thomsen et al. 2008) that measure more difficult to reset (bleach) luminescence signals with lower fading rates. The extra measurement steps involved in IRSL dating of feldspars and the higher internal dose D_R require longer instrument and analysis time.

Geologic Source Area

The tectonic history and geologic source of archaeological and geomorphological sediments are an important control on the abundance of target minerals and their luminescence properties. For example, basaltic terrains commonly lack quartz for OSL dating, while regions dominated by clastic sedimentary rocks have abundant quartz and feldspar minerals in surface deposits. Source terrain will also affect the luminescence sensitivity, or brightness, of samples, which affects the ability to measure luminescence signals. Sediments that have undergone several erosion, transportation, and depositional cycles commonly have greater luminescence sensitivity (e.g., Pietsch et. al. 2008; Preusser et al. 2006). Moreover, research has shown that sediments sourced from actively uplifting regions with high erosion rates, volcanic and metamorphic terrains, and sediments sourced from hydrothermal and micro-crystalline quartz commonly have poor luminescence properties that can cause inaccurate age determinations (e.g., Jeong and Choi 2012; Lawson et al. 2012; Pruesser et al. 2006; Sawakuchi et al. 2011; Steffen et al. 2009). If working in geographic regions dominated by these source rocks and processes, involvement of a luminescence specialist is recommended.

Post-Depositional Mixing

Targeted sediments should be examined for evidence of disturbance (e.g., **post-depositional mixing**). Processes such as bioturbation (from roots or animal and insect burrows), soil-formation, desiccation cracks, or frost/ice growth can mix grains of different ages in a sedimentary profile (Bateman et al. 2003; Rink et al. 2013). Pedogenic processes such as translocation of clays, weathering and removal of soluble minerals, and accumulation of carbonates or other salts can alter dose-rate conditions over time, leading to further uncertainty in age calculations. It is important to note that evidence for mixing may not be visible in sandy deposits that lack clear bedding, and single-grain dating may be required in these settings (Bateman et al. 2007; Bueno et al. 2013; Feathers et al. 2006).

Likelihood of Signal Resetting- Partial Bleaching Considerations

An important aspect of sample selection is assessing whether the target material was exposed to sufficient light or heat to fully reset a previously acquired luminescence signal. While experiments have shown that OSL signals are zeroed within less than a minute of direct sunlight exposure (Godfrey-Smith et al. 1988), samples from modern-aged deposits indicate that residual, or partially bleached, signals are common and can lead to age overestimation (Jaiswal et al. 2009; Medialdea et al. 2014; Olley et al. 1998). Researchers need to carefully assess the depositional environment and mode of sediment transport to select the deposits most likely to have had their luminescence signals reset prior to deposition. While eolian and beach facies commonly yield tightly distributed D_E results suggestive of well-bleached sediment (Ballarini et al. 2003; Madsen and Murray 2009), fluvial deposits can be plagued with incomplete solar resetting (e.g., Jain et al. 2004; Summa-Nelson and Rittenour 2012; Wallinga 2002) because of heavy sediment load or high turbidity (e.g., Rittenour 2008; Sohn et al. 2007). For these reasons, well-sorted deposits with sedimentary structures indicative of near-shore or eolian environments with shallow or light-penetrable water are best suited for OSL dating (e.g., Madsen and Murray 2009; Mahan et al. 2014).

Archaeological sediments commonly exhibit partial-bleaching properties. Typically, they are colluvium (e.g., rock shelter sites) or are anthropogenically sourced (e.g., middens) or disturbed (e.g., living surfaces) and often have not been well-exposed to sunlight prior to deposition/formation. For example, fill from artificial earthen mounds is often poorly bleached because the mounds were built from masses of dirt (e.g., basket loading). Sediments from canal excavation or clean-out (Huckleberry et al. 2012) and fossil graves (Kemp et al. 2014) may face the same issue. Similarly, architectural materials (i.e., mortar and bricks) that were minimally processed during building construction are likely to retain unbleached or partially bleached signal components (Feathers et al. 2008). In these contexts, the use of single-grain dating of coarse grains (63–250µm) is strongly encouraged to allow for assessment and correction of partial bleaching. Researchers interested in sampling these materials are advised to consult with a luminescence specialist to aid in sample selection that will minimize the influence of partial bleaching and sediment mixing on dating results.

Heated Materials

Materials such as ceramics, bricks, and siliceous rocks (e.g., chert) that have been heated to > 450°C can be dated with TL or OSL (Feathers 2003; Wintle 2008). Many ceramics and bricks are suitable for dating, although ceramics fired at low temperatures (ca. 500°C) can be problematic. Some ceramics are constructed using materials that do not provide measurable luminescence signals, particularly those of volcanic origin.

TABLE 1. Sample Material and Information Required for Luminescence Dating.

Material/Information	Notes
General information	
Contact and affiliation Project name and location	Provide information on the project, number of samples submitted, types of sample, and whether there are time constraints on when analyses are needed. One should also provide information regarding payment.
Soil importation permit	If transporting samples internationally, include all necessary permits and tags (hand-carry and courier transport). See the USDA website (http://www.aphis.usda.gov/ [USDA 2015]) for rules, regulations, and permit applications.
Transport method	Use a ground-based shipment process whenever possible to avoid additional dose received during air transport (high-altitude cosmic irradiation). For samples that must travel by air or will be stored for long periods, consider including a travel dosimeter.
Dose-rate (D_R) determination	
Representative bulk sediments	Subsample uniformly from within a 15-cm radius of the luminescence sample (see text for volume requirements for different techniques). A different D_R sample is required for each luminescence sample.
Water content sample	Collect within an airtight container or triple bag, indicate if sample is representative of history (required to determine level of water attenuation of dry dose rate).
Burial depth	Provide information on past burial depth history if depth of burial has changed in the past (required for calculation of cosmic contribution to dose rate)
Elevation, latitude, and longitude	Required for calculation of cosmic contribution to dose rate. Resolution to within a tenth of a degree and 10 m is sufficient.
Equivalent dose (D_E) determination	
List of sample numbers and sample types (tubes, blocks, ceramics, etc.)	Use unique sample numbers for your project, labels such as OSL-1, OSL-2 etc. can be confused in the lab. Clearly label the equivalent dose and dose-rate samples for each sample. Use permanent black or dark-colored pens; red ink is not visible in the darkroom.
Sketches and photographs	Provide clear descriptions of what was sampled and how samples relate to each other.
Information on external age control, if available	Needed to bracket initial sample analysis and check for problems with luminescence results.
Samples for D_E determination	Ensure samples are in light-proof containers/wrapping and tightly packed to limit disturbance during shipment.

Note: Refer to http://www.usu.edu/geo/luminlab/submit.html (USULL 2015) for sampling guidelines and an example sample submittal sheet.

The Pacific islands and highlands of Mexico, for example, are two areas where dating ceramics has been difficult. If one is uncertain about the suitability of a ceramic for dating, a pilot test study is recommended. Lithics are datable only if they were fired at high enough temperatures at the time of interest. While heat treatment of chert is common, the heat may not have been sufficiently high to reset a previous signal. Those that show evidence of over-firing, such as pot-lidding, crazing, and thermal fractures, will make better candidates for dating. Fire-cracked or fire-modified rocks are datable, but the archaeologists need to select those rocks that appear to have been fired at the highest temperatures. Our experience is that one-third to one-half of the fire cracked rocks submitted for dating have not been fired sufficiently. Sampling is from the center of the rocks, so even if the surface has been exposed to high temperature, steep thermal gradients may prevent the center from being exposed to high temperatures.

COLLECTION OF MATERIALS FOR LUMINESCENCE DATING

Luminescence laboratories require two separate samples and information related to dose-rate and depositional/stratigraphic setting in order to calculate an age for each sample (Table 1). These include a light-shielded sample for determining the D_E and a bulk sample of the surrounding sediments for determining the D_R. If the D_R sample is not secured in an airtight container, then a third sample may also be requested for measurement of water content. As many non-specialists overlook the importance of the D_R sample for OSL dating, the required information and collection materials for D_R calculation are discussed first.

Dose-Rate (D_R) Sample and Information

The luminescence signal measured in quartz and feldspar minerals is acquired from exposure to radiation from within the sample, from the surrounding sediments, and from incoming cosmic radiation. Collaborating luminescence laboratories will need to

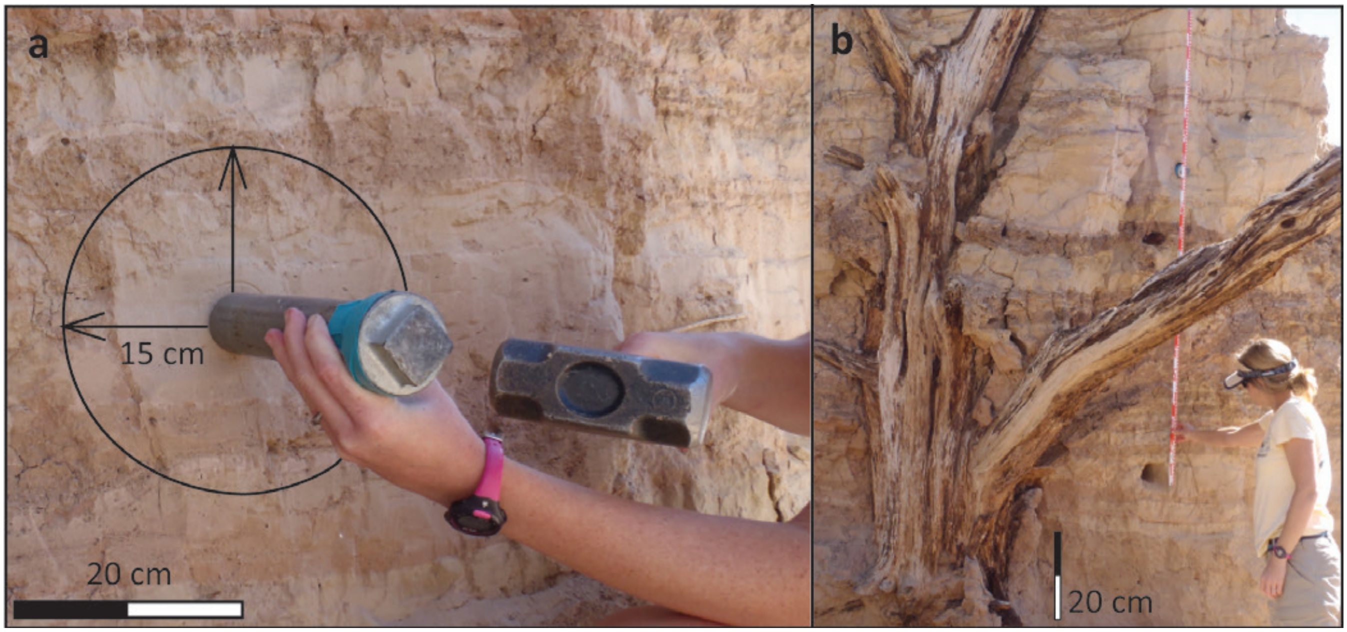

FIGURE 2. Illustration of traditional OSL sample collection by pounding a tube into an outcrop exposure: (a) circle depicts area of surrounding sediment that should be uniformly sampled for dose-rate analysis; (b) measurement of the burial depth, indicating any recent changes to depth through deposition or erosion..

have information on the latitude, longitude, and elevation of the site and the burial depth of the sample to calculate the cosmic contribution to the D_R (Table 1; Figure 2b). It is important to note whether the sample burial depth has changed over time by providing information on buried soils or erosional surfaces (López and Thompson 2012; Munyikwa 2000). Photographs and profile sketches should also be included to help illustrate stratigraphic relationships between samples.

The environmental D_R is generated from exposure to radioisotopes of potassium, uranium, thorium, and rubidium and can be determined through chemical analysis (e.g., inductively coupled plasma mass spectrometry [ICP-MS]) or radiation detection (e.g., alpha or beta counters or gamma spectrometry) of bulk sediments in the laboratory or in situ radiation detection in the field (e.g., field-portable gamma spectrometer or dosimeters). The chosen method depends on the capabilities of the analyzing laboratory.

Representative bulk samples for dose-rate analysis should be collected from a ca. 15-cm radius surrounding the sample (Figure 2a). For ICP-MS, approximately 100–200 g is sufficient for analysis; gamma spectroscopy requires about 500 g, and alpha and beta counting requires close to 70–100 g. Samples should be double-bagged and clearly labeled. Homogenous stratigraphy is favored, as variable lithology, bed thickness, and grain size of the surrounding sediments can result in non-uniform radiation fields and inaccurate D_R calculation. If heterogeneous stratigraphy cannot be avoided, the use of a field-portable gamma spectrometer (Mercier and Flaguères 2008) or of onsite dosimeters is recommended (Aitken 1998). In cases where in situ measurements are not feasible, bulk samples should be collected for each material type within 15 cm of the sample and sample locations clearly indicated on a photograph or profile sketch.

Lastly, a sample for in situ moisture content is needed. It is important to estimate moisture content, as water attenuates radiation affecting the D_R (Aitken 1998; Mejdahl 1979). The water-content sample should be collected as far into the outcrop as possible to avoid affects from surface drying and should be placed in an airtight container, such as a film canister, or triple-bagged in zip-locking baggies. Include notes on whether the collected sample is representative of the moisture history throughout burial. Where possible, avoid mottled sediments or other indicators of past water content change (e.g., Duller 2008). In cases where in situ samples are not representative of the burial moisture conditions, the influence of past water content on D_R can be modeled based on past climate conditions (e.g., Kenworthy et al. 2014) and sediment characteristics (e.g., Nelson and Rittenour 2015).

Equivalent-Dose (D_E) Sample and Information

The sample for D_E analysis must be collected without exposing the sample to sunlight. Common sampling strategies include horizontally pounding opaque pipes into the target horizon after cleaning back sediments to make a fresh exposure surface. Typical collection tubes are composed of a steel pipe that is sharpened on one end and 15–20 cm (6–8 in) in length by 2.5–5 cm (1–2 in) in diameter (Figures 2a, 3). Pipes made from soft metals (i.e., aluminum and copper) frequently buckle during collection. Smaller-diameter tubes may be used for deposits with thin bedding. Smooth, non-threaded pipes are recommended because threads in threaded pipes can hold sediment and present a potential source of contamination. Black or white unthreaded polyvinyl chloride (PVC) pipe can be used if the sample is immediately placed in a light-sealed bag or container (following Mahan et al. 2007), given that PVC is not light-safe. Use of a styrofoam plug inserted into the sharpened end of the

FIGURE 3. Typical sample-collection gear used for luminescence dating. Items identified include: (a) trowel (or field knife or small shovel) for clearing back of sediments from the face of the trench or outcrop and collection of sediment for dose-rate samples; (b) OSL sampling tube (metal or other opaque material) sharpened at one end and pre-loaded with a styrofoam plug on the sharpened end to limit sediment shaking during pounding; (c) end caps for sample tube (tinfoil and duct tape can be substituted if not available); (d) sledge hammer for pounding in sample tube (rubber mallets and light field hammers are not recommended for most sediment types); (e) duct tape to seal ends of tubes; (f) film canister for water-content samples (triple-bagged zip-bags or other airtight containers also acceptable); (g) permanent marker for labeling samples; (h) one-quart (ca. 1 liter) zip-seal bag half-filled for dose-rate sample collection; (i) pounding cap (a 2-in outside threaded plug is shown; it is important not to use pounding caps that fit tightly on tubes or that have internal threads as they can get permanently seized onto pipes); (j) field note book to document stratigraphic context and GPS location and elevation; (k) measuring tape to determine sample depth; and (l) clear packing tape to cover labels so they do not get worn off during shipping. Additional material in a sampling toolkit could include tinfoil for wrapping samples and securing tube ends if end caps are not available, a camera to document sample placement, and light-proof tarps for use if modified sample collection is necessary (e.g., for coarse-grained deposit or sampling under rocks).

tube can help secure sediment from mixing during pounding (Figure 3). Following sample collection, the sample tube should be secured with end caps or aluminum foil and duct tape to prevent light exposure and loss of sediment. Sediment within sample tubes should be tightly packed to prevent mixing during shipment.

In some cases, the target sediment may be too dense to pound a tube into for sampling. Instead, a cohesive block of sediment (ca. 15 cm per side) can be carved out of the sediment and securely wrapped in tin foil and duct tape and then placed in light-proof plastic bags or containers for transport (Roberts et al. 2003). Note that samples for D_R determination and moisture content are still needed for these samples if they come from heterogeneous stratigraphy.

High-energy, coarse-grained alluvial deposits may contain sand for OSL dating only within the matrix between clasts or in sand lenses that are too thin to sample with a tube (Kenworthy et al. 2014; Rizza et al. 2011). In these cases, the sandy matrix within

Glossary of Terms Commonly Used in Luminescence Papers

Aliquot: A subsample. In luminescence dating, aliquots are typically described as large, small, or single-grain depending on the volume of sediment measured in each aliquot.

Central age model (CAM): Statistical model used to calculate a representative D_E value to use in age calculation for a population of individual D_E values that have a normal distribution. The CAM has advantages over the arithmetic mean in that the uncertainty of each D_E value is taken into account.

Coarse-grain dating: Luminescence dating of very fine to fine-grained sand (63-250 µm in diameter).

Equivalent dose (D_E): The dose of laboratory radiation required to produce a luminescence signal that is equivalent to the natural signal of radiation the target mineral acquired since last exposure to heat or light, in Grays (Gy) where 1 Gy = 1 Joule/kg. For single-aliquot and single-grain techniques, a separate D_E value is calculated for each aliquot/grain and a statistical/numerical calculation of the populations of D_E values from a sample is used to calculate the luminescence age.

Dose rate (D_R): Rate at which the target mineral was exposed to radiation in the natural environment. Includes exposure to alpha, beta, and gamma radiation from radioisotopes of potassium, uranium, thorium, and rubidium within the sample and surrounding sediment and external radiation from incoming cosmic rays. Reported in units of Gray (Gy) per thousand years (Gy/ka), where 1 Gy = 1 Joule/kg.

Feldspar ($KAlSi_3O_8$, $NaAlSi_3O_8$, $CaAl_2Si_2O_8$): Stimulated by infrared light for IRSL or heat for TL dating. Typically more sensitive to radioactivity and has higher age limit than quartz; however it needs correction for anomalous fading, or loss of luminescence signal over time.

Fine-grained dating: Dating of polymineral (quartz and feldspar) silt grains that are 4-11 µm in diameter. Typically dated using IRSL techniques.

Finite mixture model (FMM): Statistical model used for calculating representative D_E values from a mixed or multi-modal population of D_E values.

Infrared stimulated luminescence (**IRSL):** Commonly used for dating feldspars; infrared light is used as the stimulation source to release electrons from traps.

Ionizing radiation: Radiation that causes the release of an electron from an atom (ionization) due to exposure to high-energy particles such as alpha, beta, and gamma radiation.

Luminescence age (ka): Related to the time since the last exposure of a sample to light or heat. Calculated by dividing the equivalent dose, D_E (*Gy*) by the dose rate, D_R ($Gy\ ka^{-1}$).

Luminescence: Signal generated by the release of a photon of light after an electron recombines in a lower energy state after being evicted from a mineral lattice defect (trap) by the absorption of light or heat energy. The intensity of the resultant luminescence signal is directly proportional to the number of trapped electrons, which is in turn proportional to the duration and intensity of radiation exposure since the minerals were last exposed to heat or light.

Minimum age model (MAM): Statistical model used to calculate a representative D_E value from a partially bleached population where the youngest population of individual D_E values is expected to have been bleached (reset) prior to deposition.

Optically stimulated luminescence (OSL): Luminescence dating technique in which light is used as the stimulation source to release electrons from defects (traps) in the mineral lattice.

Partial bleaching: Incomplete resetting of a prior luminescence signal due to insufficient sunlight or heat prior to the most recent burial. Note that partial bleaching can refer to conditions in which some grains were fully reset prior to deposition while others were not and therefore accurate ages can be calculated using a minimum age model, or conditions where all grains were not fully reset prior to deposition. Accurate ages cannot be generated in the second scenario.

Post-depositional mixing: Vertical displacement of grains in a sedimentary column through soil processes or disturbance following deposition (i.e., bioturbation, cryoturbation).

Quartz (SiO_2): Stimulated by blue-green light for OSL or heat for TL dating.

Saturation: Upper limit for radiation exposure that can be stored in the crystal lattice. Beyond this point, additional exposure to radiation generates an increasingly non-linear luminescence response; marks the upper limit for dating.

Sensitivity: Amount of luminescence emitted for a given radiation dose. Sensitivity is related to the source geology and history of the sediment and varies regionally and between samples and sand grains. The sensitivity of a sample will affect the precision of resultant luminescence measurements.

Single-aliquot dating: Methods in which an individual D_E value is calculated for each subsample (aliquot) measured. Typically, analysis of 10-100 aliquots or hundreds to thousands of single grains are required to produce an age.

Single-aliquot regenerative dose method (SAR): Developed by Murray and Wintle (2000), involves measurement of the natural luminescence signal followed by subsequent measurement of luminescence signals produced by given laboratory doses on the same aliquot or grain. A test dose of constant magnitude is utilized to correct for sensitivity change during the procedure.

Single-grain dating: One D_E calculated for each grain measured. Note that hundreds to thousands of grains need to be measured to produce an age because not all grains produce a luminescence signal and many individual D_E values do not pass data quality tests.

Thermoluminescence (TL): Luminescence dating method that uses heat as a stimulation source to release electrons from traps. Commonly used for heated samples.

the gravel can be collected in a light-proof container under dark conditions, such as at night or under opaque tarps, with the aid of a red flashlight or headlamp. Be aware that light-exposed sediments will need to be removed under dark conditions prior to sample collection; scraping away the outer ca. 2 cm of exposed sediment prior to sample collection is typically sufficient to remove exposed materials. Coarse gravel units often have heterogeneous D_R environments, so both the larger clasts and the sandy matrix need to be collected for analysis if in situ measurements are not available (Kenworthy et al. 2014).

In many settings, sediment exposures are not available and trenching is not possible, necessitating subsurface collection through coring. Hand augering with a soil recovery auger and an opaque sleeve insert may be sufficient for shallow sediments above the water table. Soil probes or vibracores equipped with opaque sleeves and core-catchers work best for deeper and water-saturated sediments (e.g., Mallinson et al. 2011; Rittenour et al. 2005). Cores can be split and subsampled in a darkroom setting or duplicate cores can be collected, to allow sampling intervals to be selected from an opened core (e.g., Bush and Feathers 2003). Sediments near core-breaks and lining the edges of the core tube should not be used for dating purposes due to the possibility of contamination of sediment from other horizons. For core samples, the D_R and water-content samples will need to come from the core sections above and below the sampled interval.

Information related to the expected age of the sample, stratigraphic relationships between samples, and other age results should also be submitted (Table 1). This information will help speed up analysis by providing constraints on the expected D_E to bracket during initial analyses. Understanding of the depositional environment, relative age, and geologic source area will also help determine the type of analysis most suitable for the sample (e.g., small aliquot vs. single grain, quartz vs. feldspar, coarse- vs. fine-grain dating). Knowledge of the stratigraphic relationships among OSL samples and other age constraints will also help to identify potential problems with partial bleaching or dose-rate determination. Moreover, a priori information on expected age, along with D_E distribution, can guide decisions related to the method used for age calculation. For example, luminescence specialists have a variety of age models within their toolkit that range from the use of a weighted mean (e.g., **central age model** [**CAM**]) to the more complex **minimum age model** (**MAM**) and **finite mixture model** (**FMM**), which are used for partially bleached and multi-modal (mixed) D_E distributions (Galbraith and Roberts 2012).

Sample Collection from Cultural Deposits

In principle, sampling of anthropogenic deposits is identical to sampling other types of Quaternary sediments. In practice, sediment from archaeological contexts often requires special consideration because the target units are commonly thin and the methods described above may be too destructive. Surface sediments such as earthen mounds or rock alignment features may require construction of darkroom conditions in the field using layered tarps and red lights (e.g., Feathers 2012; Feathers et al. 2008). Sampling of sediments encased within artifacts and remains can be conducted in a darkroom setting in the lab (e.g., Lail et al. 2013).

For rock alignments and masonry structures, tarps should be emplaced prior to moving the rocks and collecting the underlying sediment. A core can be driven down vertically after the rock is removed to allow investigation of the change in D_E as a function of depth (e.g., Feathers 2012). D_R samples should be collected from both the rock and the underlying sediment. Previous light exposure of the rock surface itself may also be datable (Pederson et al. 2014; Sohbati et al. 2012). However, it is recommended that researchers contact the collaborating laboratory beforehand to discuss the feasibility of these specialized luminescence dating applications in the study area.

Sampling Heated Materials

Avoidance of light exposure is less important when collecting heated samples, provided the ceramic or lithic material is opaque. More transparent samples should be immediately placed into an opaque container once recovered. In the lab, the outer 2 mm of ceramics and lithics are removed for dosimetric reasons, and this will also eliminate any light-exposed portions for most D_E samples (Feathers 2009). Ceramic sherds should be at least 5 mm thick and 2 cm in diameter to allow enough material for processing. Generally, larger-sized samples lead to greater precision. Chert artifacts need to be at least 10 mm thick and 5 g in weight. The samples for external D_R should be collected in a similar fashion, as mentioned above. Internal D_R is measured from the ceramic or rock itself.

In some cases, samples for D_R are not available, such as in the case of museum specimens or samples from sites that have been destroyed. One possibility is to return to the approximate location to collect D_R samples. In these cases, it is advisable to collect more than one sediment sample in order to evaluate the amount of variation within an area. In a worst-case scenario, where no external sediment sample is available, the laboratory can make estimations of external dose rate based on geographic location, but the dating precision will be lower.

A Summary of Special Considerations for Archaeological Sediments and Materials

While luminescence dating of earthen materials in archaeological contexts is similar to that of Quaternary sediments, archaeological settings commonly pose additional challenges. For example, locations are commonly comprised of finely stratified deposits, architectural materials, and buried surfaces that can make sampling discrete deposits difficult. Moreover, sampling options can be restricted due to permission and access issues, site preservation concerns, and the small spatial distribution of anthropogenic sediments. These restrictions require the following considerations when applying luminescence dating to archaeological contexts.

First, as there may be few alternatives, researchers may attempt to date soils or sediments that have been influenced by post-depositional mixing or partial bleaching. Luminescence dating at the single-grain scale can help diagnose and correct for these issues and is therefore highly recommended for archaeological contexts. It is worth noting that these methods can be labor-intensive and that not all laboratories have single-grain dating capability.

TABLE 2. Example Table for Reporting Luminescence Ages and Dose-Rate Information.

Sample/lab number	Depth (m)	H_2O (wt %)	K (%)[1]	Th (ppm)[1]	U (ppm)[1]	Cosmic dose rate (Gy/ka)[2]	Total Dose Rate (Gy/ka)	Number of aliquots[3]	D_E (Gy)[4]	OSL age ± 1σ (ka)[5]
Unique ID	.5	4.0	1.44 ± .04	3.0 ± .3	.8 ± .1	.15 ± .02	1.90 ± .10	20 (30)	7.41 ± .99	3.89 ± .47

1. Radioelemental determination was conducted using ICP-MS techniques.
2. Cosmic dose rate calculated following Prescott and Hutton (1994).
3. Number of aliquots used in age calculation and number of aliquots analyzed in parentheses.
4. Equivalent dose (D_E) calculated using the mean.
5. Age analysis using the single-aliquot regenerative-dose procedure of Murray and Wintle (2000) on 2-mm small-aliquots of 90-150 μm quartz sand.

Second, many archaeological contexts have complex stratigraphy within small areas, complicating dose-rate estimation. Areas such as irrigation canals, stone-and-mortar architecture, and surfaces buried by rock alignments present contexts where materials of differing radioactivity lie in close proximity. While these contexts have been successfully dated using luminescence (e.g., Feathers et al. 2008; Huckleberry et al. 2012; Huckleberry and Rittenour 2014), it is often necessary to sample additional materials for dosimetric purposes. In situ measurements on radioactivity can be made if a portable gamma spectrometer is available. Note that the field sampling strategy may need to be adapted to meet the peculiar circumstances of archaeological materials and sites and that consultation with a luminescence specialist prior to sampling and project design is highly recommended.

PUBLICATION OF RESULTS

The collaborating luminescence laboratory will send a final report upon completion of luminescence measurements. This report will include most of the information needed to publish the luminescence results. However, many laboratories will provide more details regarding the properties of the luminescence samples than needed for a non-luminescence focused paper. For this reason, we have provided a table that includes the essential information for publication (Table 2). Additional information regarding specifics of luminescence properties and sample processing and analysis can be provided in supplemental documents within the publication if desired. Given the level of involvement and research efforts of the collaborating luminescence specialist, it is recommended that they be included in publication of the results and should be offered co-authorship if justified by contribution to the research and the importance of luminescence ages in the research.

TIME AND COST CONSIDERATIONS

While this guide is meant to provide recommendations for sample selection and collection methods, it should not be considered a substitute for contacting a luminescence laboratory prior to beginning research and sample collection. A website listing North American Luminescence labs is maintained by the United States Geological Survey (USGS 2013). It is important to note that, while some of these laboratories accept external samples for analysis, many are part of research programs and have large internal workloads.

The dating process itself is labor intensive, and obtaining a luminescence age can take 9–12 months or more, depending on the current backlog of the laboratory. Therefore, costs per sample are relatively high and range from about $400 to $1,500 USD per sample, depending on laboratory overhead costs and the type of analysis requested. The demand for luminescence dating is greater than the supply; most laboratories are overbooked, understaffed, and working on many projects at a time. In general, most laboratories can complete analysis on only one sample per week per luminescence instrument, producing a typical maximum capacity of ca. 100–120 samples per year for a laboratory with two luminescence readers. Therefore, users of luminescence dating should plan their budgets and schedules accordingly and make contact with a laboratory prior to sampling to ensure that the laboratory has the capacity to accept samples and conduct analysis within the time constraints of your project.

CONCLUSIONS

Archaeological and geological field investigations require a significant investment of time and resources, and luminescence dating is no exception to this. However, with adequate planning, luminescence sample collection can be performed efficiently while avoiding errors that complicate age determination and unnecessarily consume time, money, and effort. The key to developing a sampling plan is to have a concrete understanding of local site formation processes, a general understanding of the principles of luminescence dating, and a clear focus on the role of the sample in addressing the study questions. Major problems that luminescence laboratory personnel have observed include: (1) poor sample-collection methods (e.g., exposure to light or mixing during shipment); (2) missing essential parts of the sample (e.g., D_R and water content); (3) poorly documented depositional setting, stratigraphic relationships, burial depth, and external age constraints; (4) sampling improper grain sizes, materials, and mineralogy; and (5) selection of deposits/materials that have been affected by post-depositional mixing or incomplete solar bleaching and heat resetting. These problems can be mitigated or completely avoided following the recommendations presented here, as well as by contacting a luminescence specialist prior to sampling.

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