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Research Article

User guide for scanning electron microscopy applications to luminescence dating

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Abstract

Scanning electron microscopy (SEM) methods are widely used in the geosciences to determine grain shape and surface characteristics using SEM–secondary electron and backscatter imagery (SEM-SE/BSE) and elemental composition of minerals using SEM–energy dispersive X-ray spectroscopy (SEM-EDS). We discuss applications and best practices for utilizing widely available SEM methods for luminescence dating, including (1) checking sample purity following mineral separation, (2) imaging grain shape and surface characteristics related to weathering and transport, (3) quantifying feldspar-mineral phases in feldspar separates, and (4) determining internal potassium concentration (wt% K) in feldspars for use in estimating internal beta contribution to the dose rate for a sample.

Quartz and feldspar purification checks of mineral separates require the least sample preparation and instrument time. These methods utilize the "environmental" or "low-vacuum" conditions of SEM. These conditions are less conducive to acquiring high-quality compositional data but can be used to quickly determine sample purity.

Conversely, to acquire higher-quality compositional data, SEM working conditions require high vacuum and accelerating voltages. The resulting semiquantitative SEM-EDS results can be used to determine the phase composition of feldspar separates and more accurately determine the internal potassium content for dose-rate and age calculations.

Keywords: Scanning electron microscopy; Energy dispersive X-ray spectroscopy; Internal potassium content; Test of mineral separation; Dose-rate calculation; OSL; IRSL; Luminescence dating

Introduction

Luminescence dating methods provide an age estimate of the last time that quartz or feldspar minerals were exposed to light or heat, which resets the luminescence signals (Huntley et al., 1985; Rhodes, 2011). After burial or removal from heat, the luminescence signal re-accumulates due to exposure to ionizing radiation from the surrounding environment and internal radioisotopes within grains. Analyzing the purity of quartz mineral separates and characterizing the mineral phases and internal potassium content (wt% K) of feldspar mineral separates are important checks on lab processing procedures. Scanning electron microscopy (SEM) techniques are an important yet underutilized tool for completing mineral pre-checks before luminescence measurements to determine quartz and feldspar separate purity (e.g., Gaar et al., 2014) and internal potassium concentration (wt% K) of feldspars (e.g., Trauerstein et al., 2014), as well as checking the efficacy of laboratory procedures such as hydrofluoric acid etching. This paper outlines methods and best practices for sample preparation and mounting samples for analysis, SEM instrumental parameters, and analysis routines geared toward testing sample purity, mineral content, and internal dose-rate calculation using standard SEM technology, which has become more widely available.

All images and data analysis for this report were collected using a FEI Quanta Field Emission Gun 650 equipped with Oxford Instruments EDS X-MAX detector and Aztec software. We acknowledge the support from the Microscopy Core Facility at Utah State University for their assistance in collecting this data.

SEM basics

SEM utilizes a beam of electrons to scan the surface of a sample. Electrons, created and focused by the SEM, interact with the atoms of a sample and produce a wide variety of secondary products, including electrons, photons, and characteristic X-rays (Ritchie et al., 2017). The type of secondary products used for analysis will be dependent on the data needed for analysis. Herein, we define and summarize the different SEM data types relevant to luminescence dating. It is important to note that each type of secondary product has its own dedicated detector that is subject to availability on each SEM. However, most SEM instruments at research institutions will have the basic detectors we discuss.

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Table 1. Breakdown of scanning electron microscopy (SEM) conditions and sample preparation time for analyzing luminescence samples.

Analysis type	Mineral separate	Vacuum (Torr)	Accelerating voltage (KeV)	Sample prep time	Microscope time/sample
Whole-grain analysis	Quartz	0.08–1.5	10–15	5 minutes	10–15 minutes
Polished-grain analysis	Feldspar	$\sim 5 \times 10^{-5}$	15–25	1–2 hours	1–2 hours

Secondary electrons (SE)

SE are created during the inelastic scattering of incident electrons with the atoms of the sample (Ritchie et al., 2017). As a result of this inelastic scattering, SE have relatively low kinetic energy and can be easily differentiated from electrons sourced from the incident beam of the SEM. Because they source from the top few nanometers of the specimen surface, SE are useful in providing imaging of the surface features of grains. SEM-SE imaging is commonly used to characterize grain texture and shape related to environmental conditions associated with transport processes (fracturing, rounding) and chemical weathering (etching, dissolution) (e.g., DeFarge et al., 1996; Vos et al., 2014).

Backscattered electrons (BSE)

BSE are the result of elastic scattering with the atoms of the sample (Ritchie et al., 2017) and source from the upper 100 nm of the sample surface (Piňos et al., 2017). As a result of this elastic scattering, the electrons retain most of their original energy provided by the SEM beam and can therefore be easily differentiated from the SE. There is a direct correlation between the atomic number of the atom that the electron elastically scatters from and how bright the BSE signal is due to the increased number of elastic scattering events that occur with higher atomic number atoms. This difference in number of scattered electrons allows for a real-time assessment of the relative composition of minerals with and without heavy elements. However, minerals with similar elemental compositions, such as feldspars and quartz, can still be differentiated using SEM-BSE, even when they are intricately intergrown with each other. SEM-BSE analysis is commonly used for analyzing complex mixtures of minerals within rocks (Muscente and Xiao, 2015) and other substances, such as complex steels, polymer blends, and so on (e.g., Piňos et al., 2017).

Energy dispersive X-ray spectroscopy (EDS)

EDS utilizes the interaction of incident electrons with the electrons of atoms within the sample (Ritchie et al., 2017). Some of these interactions eject inner-shell electrons from the sample atom, which then get filled by a higher-shell electron. This movement of electrons from higher to lower shells creates a characteristic X-ray with an identifiable energy that is specific to the element from which it came (Ritchie et al., 2017). Once enough characteristic X-rays are collected, a statistical analysis of the data can be undertaken to determine the semiquantitative to fully quantitative elemental composition of the sample, depending on the use of standards of known composition. These analyses can be done as a spot analysis or as elemental mapping. SEM-EDS is commonly applied to identifying elemental distribution within mineral phases (Muscente and Xiao, 2015; Ritchie et al., 2017; Alencar et al., 2022) and characterizing the microstructure and composition of various other materials (Ritchie et al., 2017). The detection limits for a scanning electron microscope under standard operating conditions is typically around 0.1wt% (~1000 ppm) for heavier elements and >1wt% for light elements (Newbury et al., 2017) and even greater for electron probe microanalysis (Donovan et al., 2011), well within the necessary precision for luminescence-related research.

SEM working conditions

Several key SEM working conditions determine how the electron beam interacts with a sample, and these must be considered when characterizing mineral separates for luminescence dating. The most important conditions relate to the vacuum level, use of accelerating voltage, and working distance (Table 1). The working conditions will be determined by the type of analysis one needs (whole-grain analysis or polished-grain analysis). We will first give a brief overview of these working conditions and then describe how they relate to luminescence sample analysis.

The SEM working conditions will be dependent on the type and resolution of the data to be collected and can be grouped into two categories: high vacuum and low vacuum. Under high-vacuum working conditions ($\sim 5 \times 10^{-5}$ Torr), higher-resolution images and EDS elemental data are possible due to higher accelerating voltage, producing a greater flux of high-energy electrons interacting with the sample and therefore greater data density in return. These higher-vacuum conditions also require a greater level of sample preparation, because they require a higher degree of surficial conductivity to reduce charging—the buildup of electrons in areas of the sample with lower electrical conductivity resulting in a distortion of the image (Ritchie et al., 2017). However, lower-vacuum conditions allow for non-conductive samples to be analyzed with little to no charging due to the interaction of the surficial electrons with the surrounding atmosphere of the chamber (Stokes, 2003; Thiel et al., 2004).

Charging of a sample under electrical bombardment results in the buildup of electrons on the sample surface, resulting in an amplified signal of BSE and SE that can distort images, reduce contrast, and affect the accuracy of analytical data (Ritchie et al., 2017). Depending on the quality of the SEM backscatter detector, certain working conditions can be increased beyond what lesser quality/older SEMs would allow before charging makes collecting images impossible. Therefore, in this report, we suggest starting conditions (accelerating voltage, vacuum, etc.) that can be increased until significant charging is evident. This process will result in different working conditions for each sample and SEM. Working with the SEM manager will be critical to achieving the best imaging and EDS elemental results.

Vacuum

A high vacuum is required to provide the most precise and informative imaging and geochemical data while using the SEM. However, most modern SEMs can run at high- or low-vacuum conditions while providing precise data. For the purposes of analyzing grains for luminescence dating, we discuss the working conditions for different types of analysis. A high vacuum allows for the least amount of interference between the incident electron beam and the sample. This becomes more critical with the higher accelerating voltages needed for more detailed elemental analyses. In contrast,

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under the low-vacuum conditions required for whole-grain (surface) analysis, there is significant buildup of electrons on the sample that can only be dissipated into the surrounding air molecules within the chamber.

The level of vacuum under high-vacuum conditions will be determined by the lab manager based on the microscope specifications and cannot be adjusted. However, low-vacuum conditions are more variable and can be adjusted for each sample to get the best data and images. We recommend starting with as low as 1.5 Torr and slowly reducing vacuum until charging becomes evident (Table 1). If possible, a vacuum of roughly 0.08 Torr is best for low-vacuum conditions for analyzing mineral grains in luminescence samples.

Accelerating voltage (AC)

AC refers to the voltage applied to pull electrons from the electron gun toward the sample; higher accelerating voltages result in more electrons bombarding and interacting with the sample. This effects a variety of functions of data collection, including quality of imaging and EDS elemental data resolution, as well as the amount of charging a sample will experience during electron bombardment. In general, a higher AC allows for higher-quality data collected at a higher rate.

Using the highest AC possible for a sample is important for obtaining high-resolution data. Under low-vacuum conditions, we recommend starting with an AC as low as 10 KeV and slowly raising it until charging becomes evident (overexposure and/or distorted images) (Table 1). Under high-vacuum conditions, AC is typically machine limited; however, high-quality data can be collected with a range of 15–25 KeV.

Working distance (WD)

WD refers to the distance between the sample and the point where the electrons exit the column (called the pole piece) and enter the sample chamber. Changing the WD allows for a shortening or widening of the depth of field, allowing for more focused imaging at shortened WDs or more depth of focus at longer WDs. This difference is critical when analyzing polished samples or whole grains. Imaging whole grains requires a wide depth of field (a longer WD) to allow the entire grain to be imaged in focus. In contrast, polished samples require a small depth of field (a short WD), because the sample has been flattened during polishing.

SEM applications to luminescence dating

The rationale, data requirements, and SEM sample preparation techniques differ between samples for optically stimulated luminescence (OSL) dating of quartz (e.g., Huntley et al., 1985; Murray et al., 2021) and infrared stimulated luminescence (IRSL) dating of potassium feldspar (e.g., Hütt et al., 1988; Buylaert et al., 2012). SEM analysis can provide sample purity checks or the determination of feldspar composition for dose-rate calculations. Sample purity checks are an important part of determining the cause of anomalous data, which can include unusually dim/bright luminescence signals and decay components, unusually high/slow fading rates in feldspars, or the presence of infrared stimulated signals in quartz samples. Similarly, the determination of feldspar sample composition (Porat et al., 2015) and the determination of internal potassium content for dose-rate calculation (e.g., Huntley and Baril, 1997; Smedley et al., 2012; Gorman et al., 2021; Maßon et al., 2025) are important reasons to consider SEM analysis of feldspar separates.

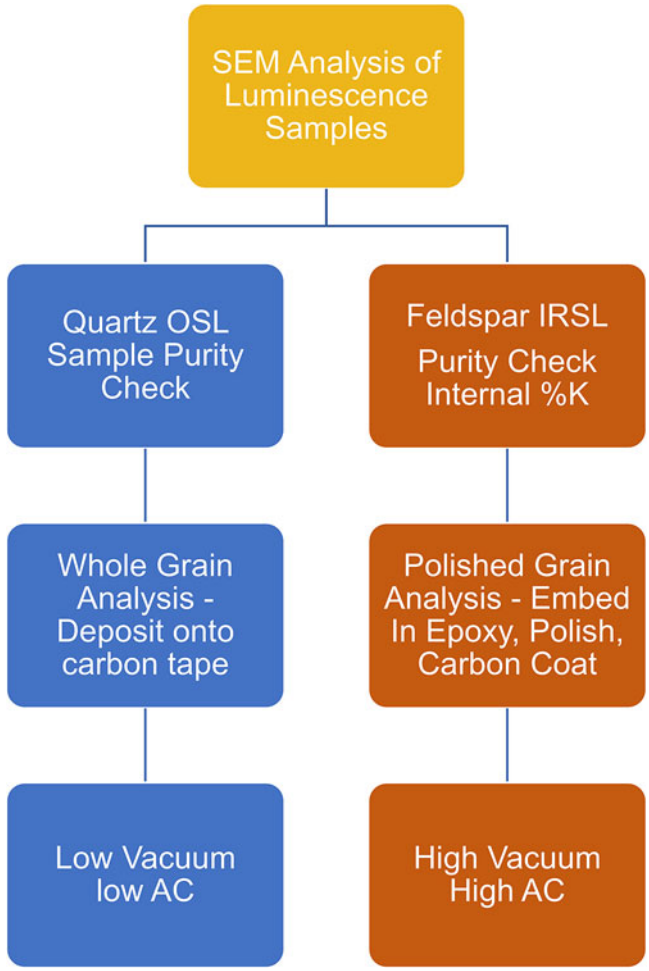


Figure 1. Workflow for analyzing luminescence samples with scanning electron microscopy (SEM). AC, accelerating voltage; IRSL, infrared stimulated luminescence; OSL, optically stimulated luminescence.

Additionally, the addition of fluorite precipitants, a consequence of hydrofluoric acid dissolution of Ca-feldspar grains, can result in anomalous luminescence signals in quartz OSL samples. Therefore, the determination of sample purity following density separation, the extent of hydrofluoric acid etching, and checking for post-hydrofluoric acid fluorite precipitation are major rationales for SEM analysis of quartz OSL samples.

The approach to SEM analysis of luminescence samples is different for quartz OSL and feldspar IRSL samples. The nearly ubiquitous presence of thin coatings on sedimentary grains can complicate determination of the composition of the underlying grains. This is especially true for feldspar grains, which weather more easily than quartz grains and tend to have more complex and thicker coatings as a result. While both quartz and feldspar grains can have weathered grain coatings, the more simplistic composition of quartz (SiO_2) grains is more easily identifiable through the relatively thin weathered coating. However, due to the more complex composition of feldspar grains and their thicker weathered coating, it is more difficult to ascertain the identity of the underlying grain as seen through the grain coatings. This is the main reason we must approach SEM analysis differently when working with samples for quartz OSL and feldspar IRSL.

Whole-grain SEM analysis of quartz OSL samples (Fig. 1, Table 1) is designed to provide a quick assessment of the purity of

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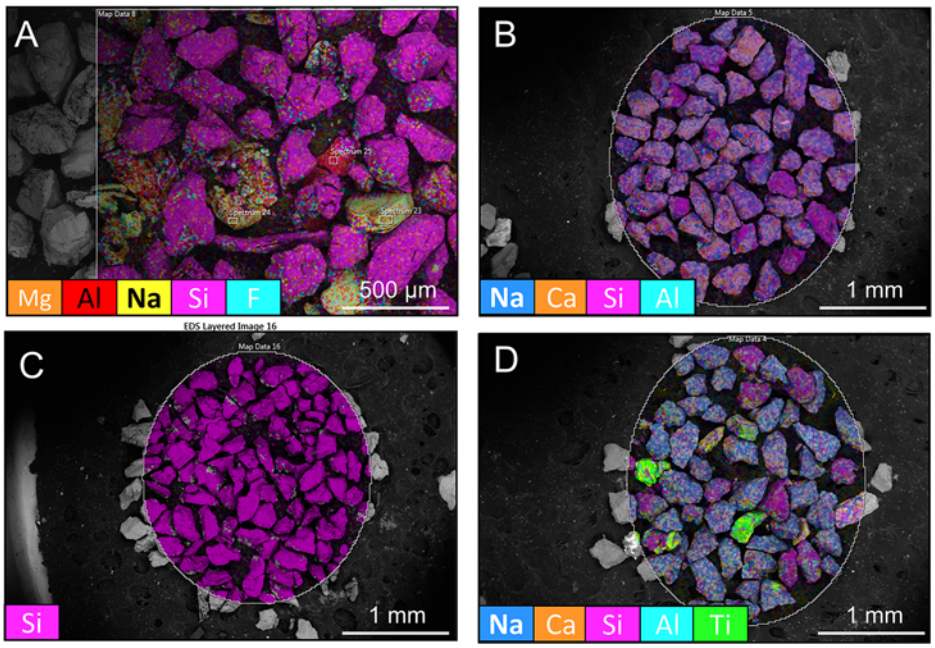


Figure 2. Scanning electron microscopy–energy dispersive X-ray spectroscopy (SEM-EDS) elemental mapping of quartz optically stimulated luminescence (OSL) separates. (**A–D**) SEM-EDS maps of quartz separates with (**A, B, and D**) and without impurities (**C**).

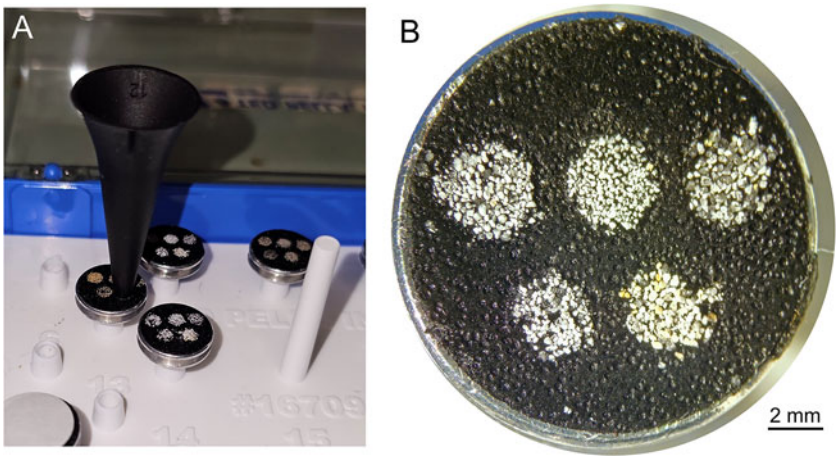


Figure 3. Sediment aliquot (dot) deposition procedure showing the use of the funnel speculum (**A**) to deposit consistently sized dots onto carbon adhesive tab covered scanning electron microscopy (SEM) pins (**B**).

the quartz sample. Feldspar IRSL samples utilize a polished-grain analysis approach (Fig. 1, Table 1), which allows higher-resolution imaging and compositional data for the purposes of determining the types of feldspars in a sample as well as their internal wt% K content for dose-rate calculations.

Whole-grain analysis (quartz OSL applications)

The simple SiO_2 composition of quartz makes determination of sample purity relatively easy, as non-quartz grains will be clearly differentiated in the SEM-EDS elemental map (Fig. 2) in most samples regardless of grain coatings. Whole-grain (non-polished) analysis of the surface of grains is accomplished by adhering dots of sediment onto a carbon adhesive tab (Fig. 3).

Sample preparation for whole-grain analysis

Considering the size constraints and the time required for preparation of samples for SEM analysis, we have developed a technique to achieve the most efficient sample preparation with the smallest sample size. We use a 3 mm funnel speculum (also known as an otoscope speculum, it is typically used by doctors to examine a patient's ear canal and can be purchased from any medical tool supplier) to deposit circular aliquots (dots) of grains onto carbon adhesive tabs (Fig. 3). These dots of sediment are deposited onto the carbon adhesive tab in identifiable rows of differing numbers to allow for easy identification of multiple samples on the same SEM pin (Fig. 3), as the exact orientation of samples will be unknown within the SEM.

To deposit the sediment into discrete dots (aliquots), the SEM pin is first covered with carbon adhesive tab (both supplies can be purchased at TedPella.com). The narrow end of the funnel speculum is placed onto the carbon adhesive tab where you want the dot of sediment to be deposited. A small scoop of sediment is then deposited into the wide end of the speculum, taking care to avoid spilling sand during this transfer. Once the grains have settled

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into the speculum, it is carefully removed from the carbon adhesive tab. Grains may continue falling through the speculum during removal, so care must be taken to avoid these grains contaminating surrounding sample dots. To ensure grains are fully emplaced onto the tape, we have found that pressing down on the sediment dots with a clean, gloved finger works best. Between the deposition of each dot, we use compressed air to blow any loose sediment away from the pin surface and the surrounding sample holder. This will reduce the chance of sample contamination as well as ensure no grains will come loose within the SEM chamber.

SEM conditions for whole-grain analysis

SEM working conditions for whole-grain analysis are designed to reduce charging of the sample surface during electron bombardment (Table 1). The most important settings are low vacuum and low AC, but the procedure also includes a suite of other conditions that are best determined by the SEM manager at the time of analysis. We give generalized suggestions for working conditions, as they may differ between instruments, based on the goal of the analysis.

Imaging is conducted under low-vacuum conditions, with chamber pressure between 0.08 and 1.5 Torr (Table 1), which allows for less charging of the sample surface due to the interaction of the electrons with the atoms in the chamber atmosphere. Lower vacuums allow for better chemical analysis through the EDS detector due to the lower interaction of the outgoing X-rays with the chamber air. Therefore, we suggest starting with roughly 1.5 Torr and slowing increasing vacuum until charging becomes evident (bright, over-exposed, or distorted images).

Due to the complex surficial topography in whole-grain analysis, there is no direct pathway for electrons to leave the area of electrical bombardment (imaging area), leading to charging. The best way to reduce charging under these circumstances is to reduce the number of electrons bombarding the sample by reducing the AC. We suggest starting with 10 KeV and increasing the voltage until charging becomes evident.

Data collection for whole-grain analysis

Typical whole-grain analysis will take less than 15 minutes of machine time per sample. This includes finding the sample dot (aliquot) on the SEM pin, capturing a high-quality BSE and SE image of the dot, and collecting a low-quality SEM-EDS elemental map of the dot. These data will provide the necessary information to determine the purity of the quartz separate for OSL dating.

It is important to note that the elemental composition of weathered coatings will show up in elemental analysis even when very thin. However, the silica of the underlying quartz should be expressed through weathered coatings quite well (Fig. 2), even with short data-collection times. Other than the rare exception, we have found that grain coatings are typically removed by the processing steps undertaken to purify quartz (exposure to HCl, H_2O_2, and hydrofluoric acid treatments), allowing unobscured imaging of the underlying mineral composition. It should be immediately apparent which grains are not quartz when utilizing SEM-EDS elemental maps. We suggest systematizing the color selection for each element to the users' preferences to make identification easier.

After locating the sample dot (aliquot) within the SEM pin, a low-resolution elemental map (~250 × 250 pixels; Fig. 2B and D) will show which grains contain higher elemental content other than the SiO_2 of quartz. Pure quartz samples will show simple elemental maps consisting only of silicon (Fig. 2C). In contrast, samples with grains other than quartz will show up clearly in the elemental map (Fig. 2A, B, and D).

Additionally, even though quartz grains can often be intergrown with feldspar and other minerals, we have found that quartz samples purified following sieving, removal of carbonates (10–30% HCl) and organic material (10% H_2O_2), density separation (2.72 g/cm^3), and etching in concentrated hydrofluoric acid/HCl tend to be pure quartz, with the occasional mixed-phase grains (Fig. 2B and D). Polished-grain analysis (see next section) can provide the information necessary to accurately date mixed phases in a manner similar to Huntley et al. (2003).

Polished-grain analysis (feldspar IRSL applications)

Preparation of samples for IRSL analysis of feldspathic sand involves the use of density separation at 2.58 g/cm^3, following sieving and removal of carbonates (using HCl) and organic material (using H_2O_2) (Fig. 4). Use of SEM-EDS analysis can help confirm the mineral composition of processed samples. While 2.58 g/cm^3 density separation is effective in capturing most of the targeted potassium-rich feldspar grains, we have found that a wide range of feldspar compositions can make it into this fraction (Fig. 4). Use of slightly lower-density heavy liquid (2.565 g/cm^3) has been proposed to produce grain separates with greater proportion of potassium feldspar (Rhodes, 2015) but has rarely produced higher-purity separates for us or other labs (Woor et al., 2022). However, we have found that the success of this approach is highly dependent on sample mineralogy and contributing source rocks. Most commonly, we find that the Na-K feldspar compositional ranges are isolated within the 2.58 g/cm^3 density-separated fraction (Fig. 4D and E). Ca-Na phases can be included at lower concentrations in some samples (Fig. 4B, D, and E) and at higher concentrations in others (Fig. 4C and F). As discussed later, isolation of K-rich feldspar is important for consideration of internal dose rate and luminescence characteristics.

During SEM analysis, the electron beam can only penetrate a few microns into the grain surface before interacting with the sample atoms and producing the variety of SE, BSE, and X-rays that are collected during analysis. The need to determine the internal grain mineralogy and potassium concentration requires a higher degree of SEM sample preparation and analysis compared with that required for quartz OSL samples (Fig. 1). Higher ACs, higher vacuums, and longer analysis times are required to accomplish this level of analysis. Therefore, SEM analysis of feldspars for IRSL dating is significantly more costly and time-consuming than purity checks for quartz OSL dating (Table 1).

While polished-grain analysis has more clearly defined utility in IRSL dating of feldspars (Figure 5; see later for discussion), these techniques can also be utilized in developing a more advanced understanding of luminescence characteristics (Huntley et al., 1983, 1993; Jain et al. 2005; Sittner et al., 2024) and elemental composition (Sittner et al., 2024) of quartz mineral separates used for OSL dating. The limits for elemental detection using SEM-EDS techniques are variable depending on the instrument and its operating conditions. However, trace elemental studies of quartz are well within the analytical capabilities of most scanning electron microscopes.

Sample preparation for polished-grain analysis

Feldspar sample preparation begins with the deposition of aliquots of purified feldspar grain separates (dots) onto carbon sticky tape in rows of differing numbers to allow for easy identification within the SEM (Fig. 6A and D). We use 2.5-cm-diameter (1-inch-diameter)

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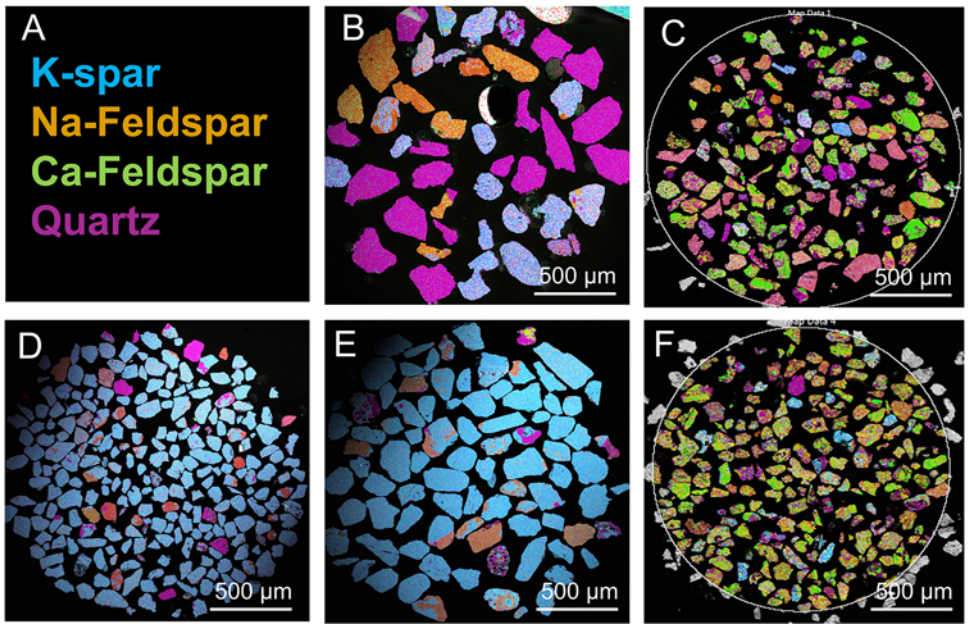


Figure 4. Examples of scanning electron microscopy–energy dispersive X-ray spectroscopy (SEM-EDS) maps of quartz optically stimulated luminescence (OSL) (**B**) and feldspar IRSL (**C–F**) separates. (**A**) Elemental-color representation within SEM-EDS maps (**B–F**). Samples were embedded into epoxy and polished using ¼ micron grit to achieve uniform polish. No difference in purity of samples, from low purity (**B, C, and F**) to high purity (**D and E**). Grain-size fractions are 75–150 µm (**C and F**) and 250–355 µm (**B, D, and E**).

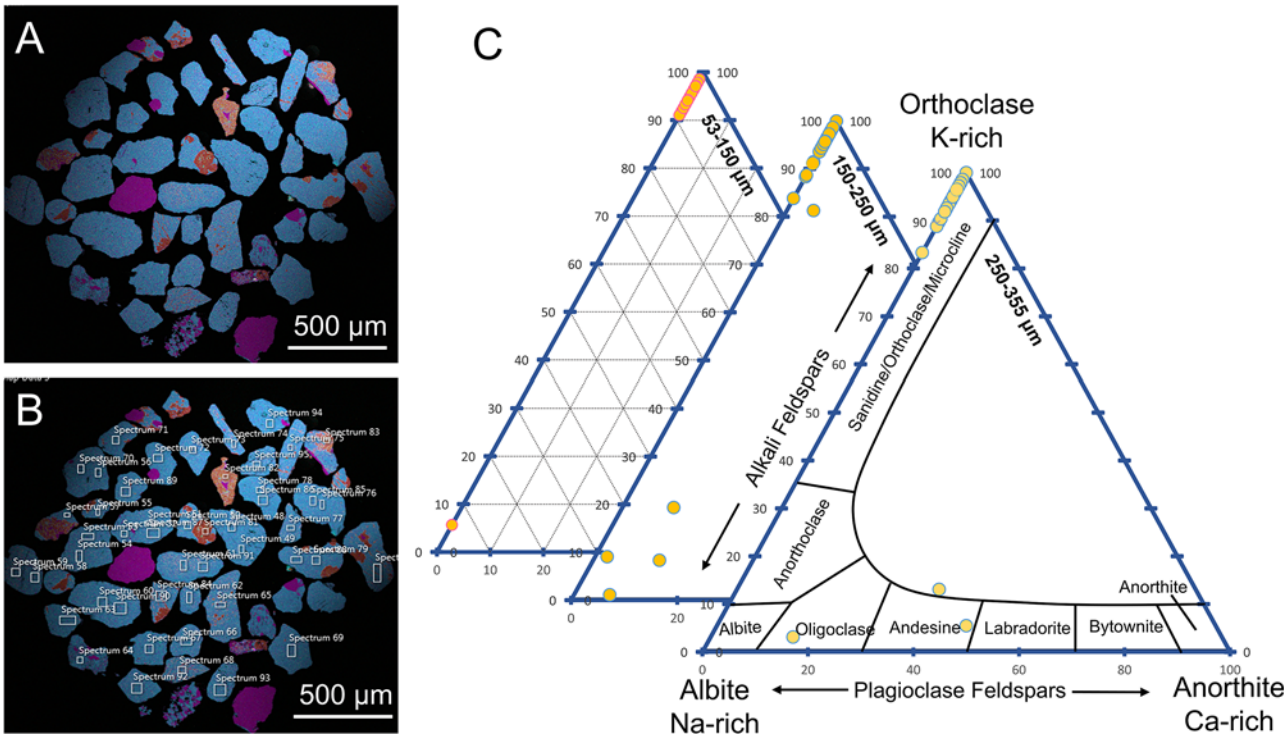


Figure 5. Scanning electron microscopy–energy dispersive X-ray spectroscopy (SEM-EDS) elemental mapping with spot and data analysis of the same sample. (**A**) SEM-EDS elemental map of a post-float 250–355 µm polished feldspar infrared stimulated luminescence (IRSL) sample. (**B**) SEM-EDS elemental map showing spot analysis locations for **C** (see Figure 6A–C for additional pictures of this same sample). (**C**) Ternary plot for data collected from locations shown on **B** as well as for 53–150 µm and 150–250 µm grain-size fractions (elemental maps not included); note bimodal distribution of samples between the potassium-rich and sodium-rich endmembers.

sample billets for this process, which allows for as many as 9 sample dots per billet using a row of 5 and a row of 4 (Fig. 6A and D shows rows of 3 and 2). The same sample deposition technique used for quartz samples using a 3 mm speculum funnel is used for these samples as well (Fig. 3A) to deposit a 3 mm dot onto the tape (Fig. 6B and E).

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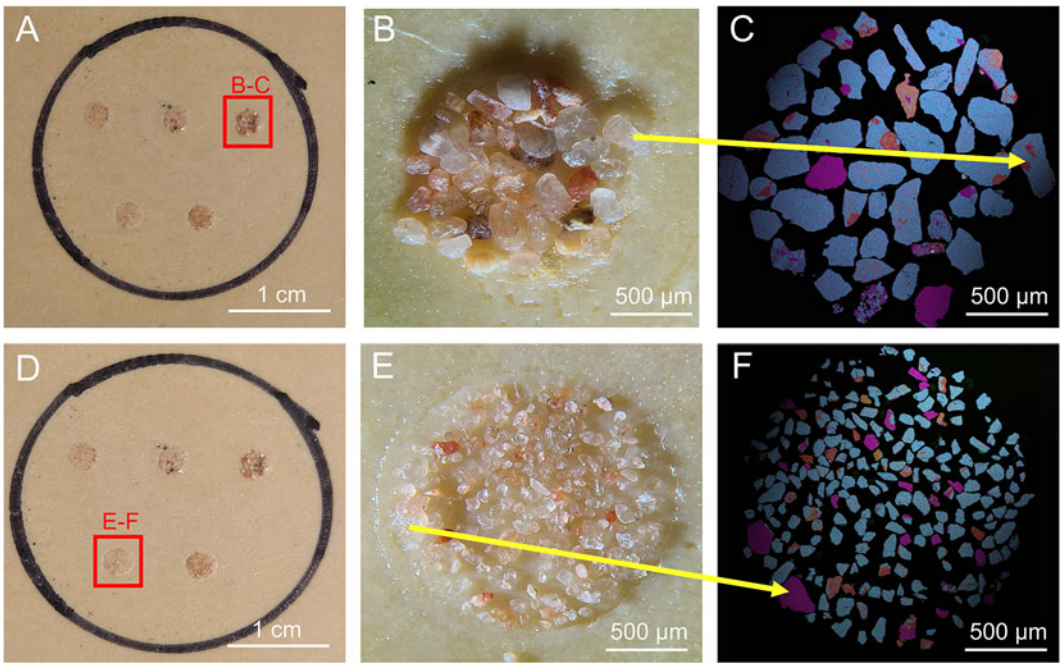


Figure 6. Polished-grain analysis sample processing procedure. (**A and D**) Sample aliquots are deposited onto tape in rows of unequal numbers. (**B and E**) Enlarged image of sample aliquots shown in **C** and **F**. (**C and F**) Scanning electron microscopy–energy dispersive X-ray spectroscopy (SEM-EDS) elemental maps of sample aliquots show in **B** and **E**. Grain-size fractions are 53–150 µm (**E and F**) and 250–355 µm (**B and C**).

Once the samples have been deposited onto the tape surface, a form is placed around the SEM pin and epoxy (we use Buehler Epoxy Resin 20-3440-032 and Epoxy Hardener 20-3442-016) is carefully poured into the billet and allowed to harden. Typical hardening times are roughly 24 hours. Once hardened, the billet can be removed from the tape and the grains can be polished. At this beginning stage, the grains should be visible at the surface of the epoxy. The epoxy billet should be polished starting with a lower grit of your preferred polishing medium (we use aluminum oxide lapping/micropolishing paper) until a cross section through the grains in the billet is produced, taking care not to polish through the entire grain thickness by regularly checking under an optical microscope. Next, move down through sequentially higher grits, giving each 1–2 hours of polish time, until the billet has been polished using the ¼ micron grit. This will provide the best surface for SEM-EDS (Fig. 6C and F). The final step for preparing polished samples for SEM analysis is to deposit an electrically conductive coating on the surface of the polished sample. The SEM lab should have the necessary carbon coating/sputtering equipment for this purpose.

SEM working conditions for polished-grain analysis

SEM working conditions for polished-grain analysis are designed to provide the highest-resolution elemental compositional data (Fig. 1). The most important conditions for this purpose are obtaining a high AC under high vacuum. Other important conditions, such as WD, spot size, bias, and so on, will best be determined by the microscope manager at the time of analysis. Due to the differences between types and brands of scanning electron microscopes, it is difficult to define a set of exact working conditions; instead, we give generalized suggestions on working conditions based on the goal of the analysis.

SEM microscope conditions with high AC under high vacuum allow for the highest interaction of incident electrons with the sample, producing the most data possible at the quickest speeds. Typically, an AC of 15–25 KeV (or higher if the SEM will allow) and a high vacuum of $\sim 5 \times 10^{-5}$ Torr will be conducive to collecting high-resolution compositional data. Due to the highly conductive surface after carbon coating, data collection is not limited by the effects of charging under normal SEM working conditions.

The production and analysis of characteristic X-rays during energy dispersive X-ray spectroscopy (SEM-EDS) under these microscope conditions will provide high-detail, semi- to fully quantitative elemental composition of the sample. However, more advanced techniques, including the use of advanced software and multiple SEM-EDS detectors, as well as microprobes and wavelength-dispersive X-ray spectroscopy, have been shown to provide higher-quality and more easily interpreted data (e.g., Neudorf et al., 2012; Gaar et al., 2014; Jacobs et al., 2019; O'Gorman et al., 2021). While advanced software and hardware products are not available in many SEM labs, the resultant data output allows for easier interpretation and use of SEM data for luminescence applications.

Data collection for polished-grain analysis

Polished-grain analysis is good for acquiring two types of data: elemental maps and point (spot) analysis. Both datasets are useful for different purposes. In general, we first produce an elemental map to provide a quick visual summary of the grain composition within the sample and provide a guide as to where to do point analysis. For quick low-resolution elemental maps, ~500 × 500 (or as low as 250 × 250) pixel maps will provide a good base map for selecting areas for spot analysis. Unless needed for publication, elemental maps should be kept at a lower resolution to reduce microscope time and cost. Low-resolution maps can be collected in 15–30 minutes. However, for publication-quality elemental maps, we suggest at least 1000 × 1000 pixel maps, which are allowed to collect data

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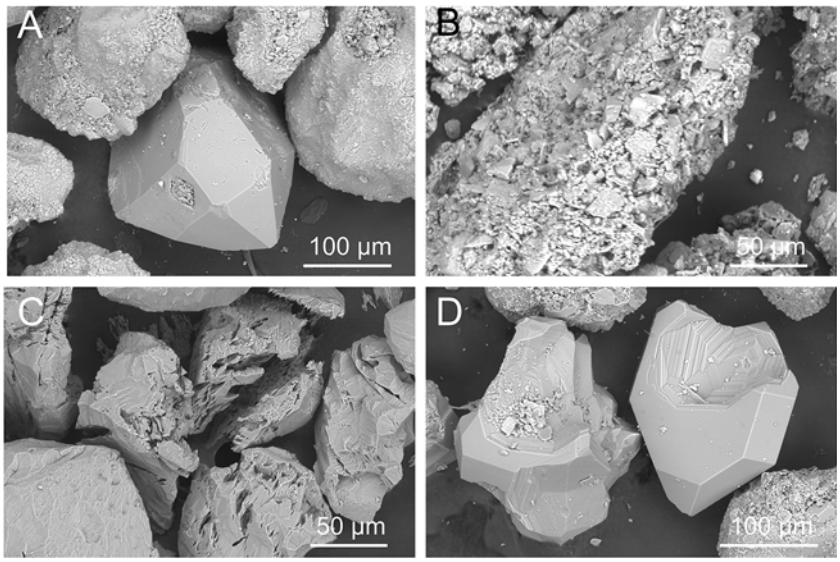


Figure 7. Scanning electron microscopy–secondary electron and backscatter imagery (SEM-SE/BSE)images showing a variety of grain shapes: (**A**) crystalline quartz grain with rounded and weathered feldspar grains; (**B**) highly weathered feldspar grain; (**C**) post-hydrofluoric acid etching of quartz grains; and (**D**) fractured/shattered crystalline quartz grains.

for 1–2 hours (see Fig. 4 for examples). Elemental maps of multiple grains provide a useful indicator of the minerals present within a sample, including feldspar phases. Spot analysis is needed for higher-resolution data on elemental concentrations within mineral grains.

Areas of interest for high-resolution spot analysis are selected using the larger elemental map of the grains. Analysis of multiple potassium feldspar grains is recommended to determine mean internal potassium content for calculation of internal dose rate. When selecting points, we suggest using a range of spot sizes on areas that seem to be pure potassium feldspar and clean of any impurities. Gülgönül et al. (2008) has shown that small impurities within the feldspar surface can result in widely varying elemental composition; therefore, multiple spots and a range of spot sizes will help to avoid the oversized impact of small impurities that do not show in the elemental maps. At least 20 spots should be selected to get a statistically relevant data set to determine mean wt% K within the sample. Each spot needs ~1 minute to collect the necessary data for a robust dataset.

SEM-EDS elemental analysis, either through mapping or spot analysis, will identify the dominant elemental composition of the sample. The SEM program will auto-detect which elements are represented by the collected characteristic X-rays during SEM-EDS analysis. Typically, these suggested elements can be accepted by the user. However, due to the carbon-coating process, carbon must be removed or defined as a coating within the program.

Interpretation and utilization of results

Whole-grain analysis (quartz OSL applications)

SEM data collection and analysis for quartz OSL samples is relatively quick and straightforward due to the simple composition of quartz grains allowing even low-resolution SEM-EDS maps to be used to confirm the purity of a sample and identify contaminants. Pure quartz samples will show simple elemental maps consisting of only silicon (Fig. 2C). In contrast, samples with grains other than quartz will clearly show up in the elemental map (Fig. 2A, B, and D). If grains other than quartz grains are identified in this manner, additional spot analysis can be done to determine the composition of contaminating grains and intergrowths. While the luminescence response of quartz samples to infrared stimulation provides evidence of feldspar contamination or inclusion, other minerals can also contribute to the blue-light stimulation that is assumed to be derived from quartz, leading to problems with isolating the signal from quartz. Knowledge of which minerals are contaminating a sample and whether they are separate grains, secondary precipitants, or found in rock fragments and mineral intergrowths will help design further processing and chemical etch steps needed to remove the contaminants.

Processing samples to isolate quartz sand commonly involves etching the sample with hydrofluoric acid to remove feldspar minerals and to etch the outer ~5 microns from the quartz grains to remove the influence of alpha radiation in the dose-rate calculation. Concentrated HCl is commonly combined with or followed by the hydrofluoric acid etch step to prevent the precipitation of fluorite (CaF_2). Given the highly luminescent nature of fluorite, its removal is required to measure pure quartz luminescence signals for dating and sensitivity analysis (brightness of luminescence signal produced per absorbed dose of radiation). SEM-EDS analysis is key in these cases to test for fluorite contamination. Fluorite contamination is quickly identified with the presence of elemental fluoride on grain surfaces in the elemental map. We have found in these cases that fluorite is easily removed with sample agitation in warm HCl for several hours, alternating between periods of vortexing and sonication. We use a vortex mixer or orbital shaker with sonication to help break up and dissolve the fluorite and then check sample purity afterward with SEM-EDS analyses.

In addition to sample purity checks, analysis of the whole-grain quartz mineral separates can provide more environmental data about a sample beyond its luminescence age. For example, SE and/or BSE data are useful in determining grain shape imparted through erosion and transport sedimentation processes (well-rounded vs. angular vs. crystalline grains; Fig. 7) as well as grain surface textures related to transport processes (e.g., pitting, conchoidal fractures, chatter marks). Moreover, SEM-BSE provides information on the depth and character of hydrofluoric acid etching of the grains (Fig. 7). These analyses do not require the use of elemental maps and therefore take minimal time per sample (~1 minute/image) for data collection.

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Polished-grain analysis (feldspar IRSL applications)

Due to the common weathered coatings and diverse composition of feldspars, a more complex sample processing procedure is needed to prepare IRSL samples for SEM analysis. These steps were described in the polished-grain analysis section. This section will describe the post–data collection analysis and how to incorporate those data into IRSL dose-rate calculations.

The feldspar mineral group is complex and varied. Feldspars can be grouped into three compositional groups: potassium feldspars, sodium feldspars, and calcium feldspars. Represented by the feldspar ternary diagram in Figure 5C, this range of compositional variability is broadly separated between the alkali feldspars along the potassium–sodium compositional range and the plagioclase feldspars along the sodium–calcium compositional range. While mixed-phase feldspar assemblages are common, the style and complexity of mixing is due to the geological source of mineral grains.

Potassium feldspar is preferred for IRSL dating due to the increased internal beta dose rate from ^{40}K within the mineral, as well as its brighter luminescence signals, which are assumed to dominate other mineral phases (e.g., Huntley and Baril, 1997; Smedley et al., 2012). However, other feldspar phases can contribute to the bulk IRSL signal of samples in some settings (cf. Maßon et al., 2025). Given differences in internal dose-rate contributions from ^{40}K, it is important to understand the composition of feldspar phases within a sample, as well as how to effectively remove them during sample processing. Similarly, the use of SEM techniques can help to explain anomalous fading and/or IRSL data acquired on impure feldspar mineral separates; identification of Ca-rich feldspar mineral separates is especially important, as they have been shown to display significantly different fading characteristics than K-rich feldspars (Huntley et al., 2007).

The assumption that heavy mineral separation (at 2.58 g/cm^3 or super-K density 2.565 g/cm^3; Rhodes, 2015) will produce nearly 100% potassium-rich feldspar subsamples has been shown to be incorrect for many samples (e.g., Meyer et al., 2013; Woor et al., 2022). Additionally, alkali feldspar phases can have potassium content ranging from 0 to 14 wt% (Huntley and Baril, 1997). SEM-EDS spot analysis of polished feldspar mounts is needed to determine the actual internal wt% K for a sample for internal beta dose-rate calculations. Commonly assumed internal values are 12.5 ± 0.5 wt% K (Huntley and Baril, 1997) and 10 ± 0.5 wt% K (Smedley et al., 2012) for alkali feldspars that contribute to IRSL signals, although this has been shown to not always be the case, especially for samples dominated by non-potassium feldspar phases (e.g., Maßon et al., 2025).

SEM data can be useful in accounting for potassium content within feldspar grains, as well as providing sample-specific values to correct for the assumed wt% K when calculating internal dose rates and IRSL ages. We collect two separate types of data for this purpose: (1) elemental maps scans of the entire sample (~100 grains) to determine relative abundance of mineral phases (Fig. 4); and (2) point analyses to determine detailed elemental composition of grains, with a focus on potassium content in feldspar minerals. For the spot analysis, care must be taken to not collect a region that crosses between feldspar phases within multiphase feldspar grains. In cases with complex intergrowths and twinning of non-potassium feldspar phases, it is best to collect multiple spot regions. Use of a system that combines microprobe and SEM-EDS abilities provides the best solution (e.g., O'Gorman et al., 2021), but such systems are not available in most SEM labs.

There are several options for how to incorporate internal wt% K content determined by SEM-EDS into dose-rate calculations. First, if the sample is dominated by potassium feldspar, then it is recommended that the mean of the internal wt% K be used for the internal beta dose-rate calculation (e.g., Duran et al., 2015). This is a much better option than assuming 12.5 ± 0.5 wt% K or 10 ± 0.5 wt% K, as previously proposed (e.g., Huntley and Baril, 1997; Smedley et al., 2012). However, given sample-to-sample variability in feldspar content and differences in luminescence contribution between phases (e.g., Maßon et al., 2025), it might be best to collect single-grain IRSL measurements and analyze the same grains with SEM-EDS to select grains with constant wt% K for age calculation.

However, routine analysis of single-grain data for both luminescence dating and SEM-EDS is time-consuming, and not all facilities have access to these specialized resources (IR single-grain reader, SEM-EDS) or funding for extended analysis time. This is why a standard 12.5 ± 0.5 wt% K is used for the purposes of IRSL dating (Huntley and Baril, 1997). However, when this assumed 12.5% is not viable, we recommend starting with mounting and polishing 5–9 aliquot dots of feldspar separates from representative samples to produce a quick SEM-EDS map to get an overview of the dominant mineral phases within a sample. If samples are not dominated by potassium feldspar phases, and density separation efforts to purify K-rich phases are not successful, researchers can instead utilize SEM-EDS spot (small area) analysis of individual grains to characterize wt% K content of multiple (>20) feldspar grains per aliquot dot. This can be used to determine the mean wt% K of grains. This can be applied, with realistic uncertainty added, to calculate the internal dose rate within the feldspar from samples in the study region, assuming similar source-rock contributions in each sample.

Summary

This paper recommends the routine use of SEM to aid in sample processing and data interpretation of luminescence age calculation. We outline sample preparation techniques and data analysis requirements for mineral separates prepared for quartz OSL and feldspar IRSL samples. This paper aims to provide a generalized understanding of how SEM works as well as specialized details on its applications to luminescence dating techniques.

For luminescence practitioners, SEM can aid in the understanding of sample heterogeneity by differentiating between quartz and lithic grains or by determining the relative abundance of target potassium feldspar and other feldspar phases in a sample. Additionally, the collection of high-resolution elemental compositional data of feldspar grains can allow for sample-specific internal potassium concentrations for dose-rate calculation.

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