

Use of magnetic separation for purifying quartz for luminescence dating

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Introduction

The increased use of the single aliquot regenerative (SAR) dose protocol and its current acceptance as the preferred protocol for dating quartz calls for efficient methods for purifying quartz. Common components of the sediment that need to be removed are salts, clays, carbonates, organic matter, heavy minerals and feldspars. Salts are usually water soluble; clays can be removed by sieving or decanting; carbonates are dissolved by 5-10% HCl; and organic matter is oxidized by concentrated peroxide or bleach.

Removing heavy minerals and feldspars is normally the most time consuming part of the procedure. Commonly, heavy minerals, feldspars and other contaminant minerals are removed by a 2-step density separation, using heavy liquids with densities that are slightly heavier and slightly lighter than quartz (Wintle, 1997). As it is imperative that the purified quartz contains no measurable feldspar, concentrated (40-48%) hydrofluoric acid (HF) is typically used to dissolve the remaining feldspars and at the same time etch the quartz.

An alternative method for removing heavy minerals and most feldspars is magnetic separation. Whilst this is not a new procedure (Rosenblum, 1958), the use of it within luminescence is currently not common. Aitken (1985; p.18) mentions it as one of the many techniques used for mineral separation, and in the past it was used in the Research Laboratory for Archaeology and the History of Art, Oxford (Fleming, 1966). This paper aims to provide a description of the procedure.

The advantages of magnetic separation are effectiveness and simplicity. The modern magnetic separators are stable and the results reproducible. The non-magnetic fraction remaining after separation consists of almost pure quartz and can be etched with relatively small volumes of HF, thus saving time, chemicals and heavy liquids.

The need for a different method for mineral separation in the luminescence dating laboratory at the Geological Survey of Israel (GSI) arose when attempting to extract quartz from sediments of alluvial fans in hyperarid regions. Besides quartz and feldspars, these poorly sorted sediments contained large amounts of very angular sand-sized chert. These chert grains could not be dated by OSL as, first, they were mostly not allochthonous but formed within the sediment by shattering and disintegration of chert pebbles due to action of salts, and had thus not seen sunlight; and second, due to their opacity they do not bleach during transport. Therefore it was crucial to remove the chert for successful dating.

The chert could not be separated from the quartz by the physical or chemical methods commonly used at the time in the laboratory, such as density separation or selective dissolution, because the density of chert is close to that of quartz, and it is only marginally more soluble than quartz in HF. Fortunately, magnetic separation using a Frantz LB1 with a high current on the magnet proved suitable for removing the chert as well as most feldspars, insoluble dolomite and the majority of heavy minerals. Subsequent heavy liquid separation at a density of 2.7 g/cm³ showed that only several grains of heavy mineral remained, mostly apatite and fluorite. Zircon, a luminescent mineral, is very rare in sediments and is highly unlikely to remain after magnetic separation.

Magnetic separation is a common physical method used for separating minerals with different magnetic properties. In industry it is used to concentrate ferromagnetic and paramagnetic ore minerals (e.g. Augusto and Martins, 1999). In Earth Sciences it is used for extracting diamagnetic mineral fractions from igneous and metamorphic rocks for dating or for geochemical analyses (e.g. Kolodner et al., 2006).

Equipment description

Magnetic separators used in Earth Science laboratories comprise an electromagnet, a feeder, and a sloping and tilting chute that pass the grains by the magnet (Figure 1). The grains fall from the feeder and move along the chute due to its vibration. As each grain runs by the magnet it is either attracted to the magnet or it falls to the sloping side of the chute. A split in the chute separates the more magnetic from the less magnetic grains, and each are collected in separate, designated boxes. By adjusting the current on the magnet using guides from published tables (Rosenblum, 1958) and trial and error, almost any two minerals can be separated. Adjusting the slope and tilt is useful for different grain sizes and shapes.

Magnetic separation is now used at the GSI laboratory as part of the routine mineral separation protocol. It follows sieving and dissolution of carbonates. The sample needs to be washed of fines (<40 μm) and dry. The Franz magnetic separator is situated in the dark laboratory and separation is carried out under the required subdued orange-red light. A small red pin-light is used to check the grain flow from the feeder onto the chute.

The feeder may contain up to 100 g, but most samples for OSL dating are much smaller. Sample flow can be as high as 10 g/min, still with effective separation. However a more practical flow rate is about 2 g/min, which would take about 10 minutes to separate a characteristic sample weighing 20 g. There is essentially no sample loss and even very small samples (a few hundred mg) can easily be separated and retrieved using low flow rates.

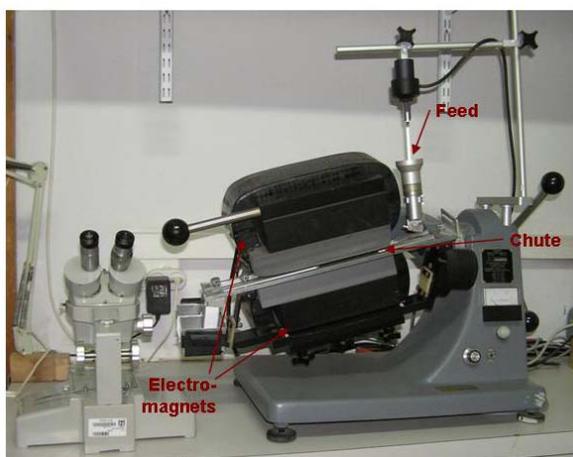


Figure 1: The Frantz Magnetic Barrier Laboratory Separator Model LB-1 at the GSI.

So far, separation has proved to be efficient for a large range of grain sizes, from 64 μm to 350 μm , and the resulting quartz is very clean (Figure 2). Over the years, quartz has been extracted in this way from a variety of soils (Terra Rossa, basaltic soils, rendsina), desert loess, and fluvial and aeolian sediments.

Effective separation is judged visually by placing a small sample from the non magnetic fraction under a binocular (using white light) and checking that no dark or opaque mineral grains are visible and that the extracted fraction is essentially quartz. Further tests for the presence of feldspar are routinely carried out during OSL measurements using the presence and magnitude of the IRSL signal. In 95% of the samples prepared at the GSI laboratory the IRSL signal is less than 5% of the total OSL signal (measured as the ratio between an ordinary recycling point and an additional post IR recycling point).

For fine sand sediment (74 to 180 μm) the optimal setting was found to be:

Slope of 25°, tilt of 17°
Current -1.4-1.5 Amp on the magnet
Sample flow rate: 2-3 g/min

One run through the magnetic separator is usually sufficient to extract clean quartz. Re-running the magnetic fraction may result in extracting a few additional quartz grains and this is recommended when sample size is very small. Re-running the non magnetic quartz fraction is not necessary as this fraction will undergo etching with HF.

The magnetic fraction in a sample may vary between 10 and 70% of the sample. In some cases the magnetic fraction contains quartz grains which will not be separated by a second pass through the magnet. Usually those are quartz grains coated by iron oxides or containing heavy mineral inclusions. Reducing the current on the magnet to 1.2-1.3 Amp may increase the yield of this type of quartz into the non-magnetic fraction.

For most samples the great majority of feldspar grains are removed by magnetic separation. As it is then not necessary to dissolve large quantities of feldspars, only small amounts of HF are needed for obtaining pure quartz. Stoichiometrically, only 2 cm^3 of concentrated (40%) HF per 1 g of quartz are necessary for dissolving the quartz, however routinely much more is used when dissolving feldspar. We found experimentally (by checking for the presence of IR signals as mentioned above) that for the majority of samples, 5 cm^3 HF per 1 g quartz

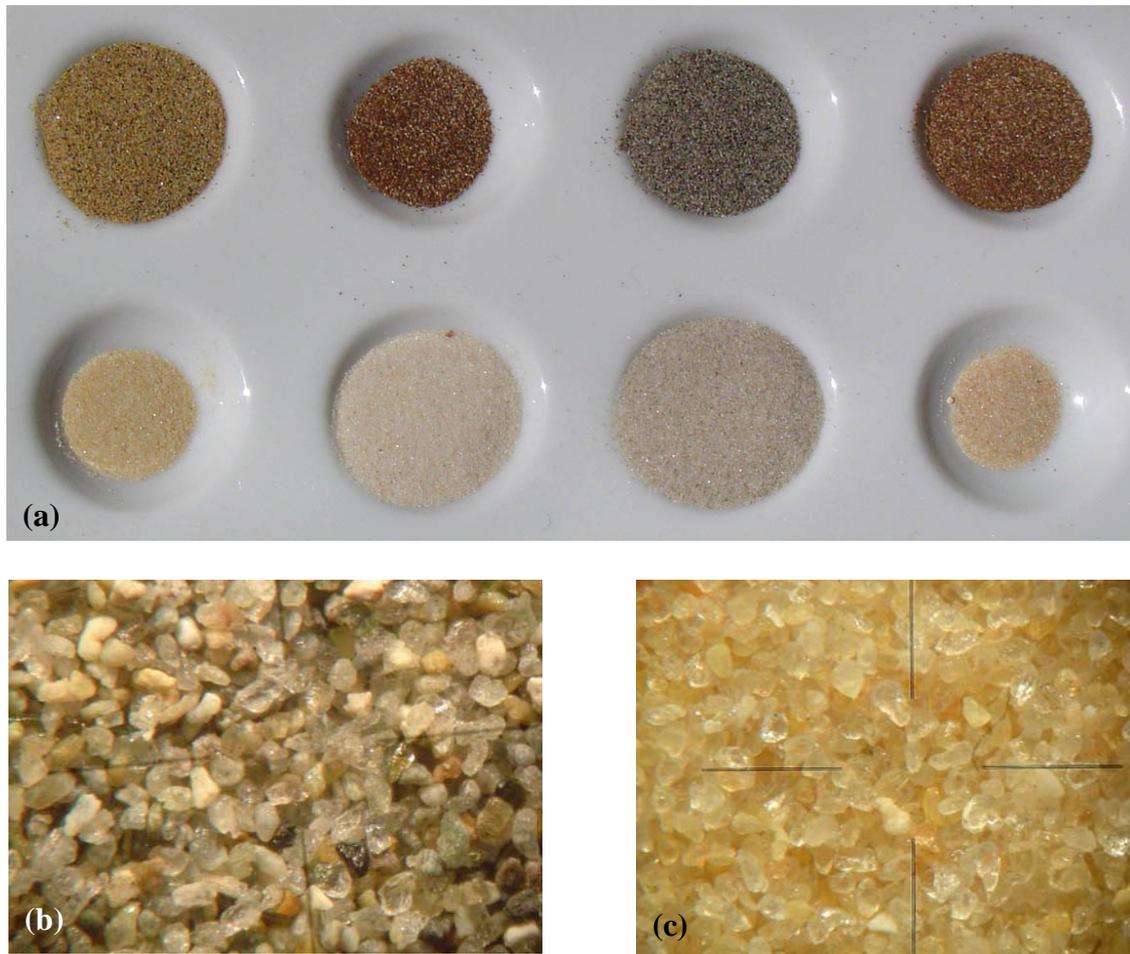


Figure 2: Images of samples after magnetic separation. The non magnetic fraction is essentially pure quartz.
 (a) Magnetic (top) and non magnetic (bottom) fraction of four sand samples.
 (b) Close up of sample before magnetic separation. Note abundant quartz, feldspar (pale, opaque) and some heavy mineral grains (125-150 μm).
 (c) Close up of the non-magnetic fraction. A few remaining feldspar grains are visible (125-150 μm).

will remove any remaining feldspars while etching the quartz. A smaller volume of HF occasionally resulted in IR signals. It should be noted that even in samples with some IR signal, the amount of feldspar remaining after HF etching is so low that it cannot be detected with X-ray diffraction.

Practicalities

The model used in this study is the Frantz Magnetic Barrier Laboratory Separator Model LB-1, which is about 15 years old (for more information see <http://www.sgfrantz.com/labsep.htm>). The separator is located in a light-tight room and can be used either under normal neon lights or with subdued orange lights. It is shared with other geologists requiring mineral separation. Using it is very straight forward

and students can use it independently after a single demonstration.

A binocular microscope in the laboratory is necessary, used to examine tiny amounts of the magnetic and non magnetic fractions under white light, to verify that separation was successful and that essentially only quartz is present in the non-magnetic fraction. Cleaning between samples is carried out using pressurized air and a brush.

Magnetic separators are usually found in geology and mineralogy departments. The most common instruments are made by S.G. Frantz Company, Inc. Before making any major purchases, it is worth going to the nearest laboratory with a magnetic separator to try out a sample that can be exposed to light. The

features one should look for are ease of operation in semi darkness and the stability of the magnetic field. In very old models the magnet tends to overheat and the current on the magnet drifts to lower values.

In most dating labs, magnetic separators will be used only for part of the time. Since the cost of a modern magnetic separator is in the range of US\$18,000, it is worth considering buying it jointly (or using an available one) with other departments. As long as the laboratory can be made light-tight and fitted with appropriate lighting for OSL sample preparation, sharing an instrument is feasible.

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Reviewer

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