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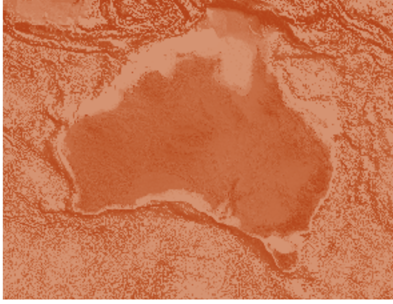

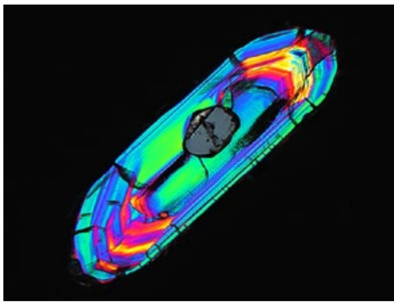

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Handbook of Geochronology Mineral Separation Laboratory Techniques

Emma-Kate Chisholm, Keith Sircombe and David DiBugnara

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GEOSCIENCE AUSTRALIA
RECORD 2014/46

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Contents

Executive Summary

This Record provides a summary of activities undertaken in the Geoscience Australia (GA) Mineral Separation Laboratory, an in-house facility dedicated to the production of high quality mineral separates for isotopic analysis.

Significant resources are expended acquiring isotopic geochronology data; money, time and effort are invested from the earliest stage of identifying a need to date a geological sample, followed by planning and sample collection, mineral separation processes, data acquisition, data processing, report writing and data management. The researcher and end-users need assurance that the best sample possible has been obtained and analysed.

The chain of quality control required for good isotopic analysis begins with field sampling and continues with detailed attention to mineral separation and final sample preparation. Issues faced include finding fresh samples, minimisation of contamination risks, effective Work Health & Safety procedures, efficiencies in sample preparation, and digital documentation of samples. Insufficient sampling and preparation can produce erroneous results, just as surely as any systematic instrument problem, potentially leading to inaccurate reporting, low confidence in data quality and a loss of reputation for the laboratory, researchers and organisation. Therefore, it is essential that measures are taken through all stages of processing to ensure that staff are safe, the risk of sample contamination remains as low as possible, labelling is correct, and that data entered into information and archiving systems are accurate.

Rock samples collected for geochronological analysis from outcrop, drill core or dredges can range in size from 100 g to over 30 kg. Variations in sample type, sample size and target minerals affect the approach to sample preparation. Minerals most frequently separated for isotopic analysis are zircon, monazite and potassium-rich rock forming minerals including K-feldspar and micas. Other minerals separated include titanite, rutile, apatite, xenotime, sulphides, tungstates and gold.

1 Sample Collection

1.1 Sampling philosophy

The quality of isotopic analyses begins with sample collection. High quality samples will boost confidence in the results whereas poor quality samples will always have doubt around their validity. Samples collected for geochronology are typically large, ~20–40 kg, for several reasons:

- The yield of analysable material from a given sample is often unknown. Although minerals like zircon are often ubiquitous in many rock types, it is not uncommon to encounter apparently promising rocks that have very low yields. Until shown otherwise in a newly sampled rock, it is advisable to take a large sample.
- Even if a rock yields suitable minerals, having more material to work with provides for more options in selecting mineral grains for analysis. For example, having 500 grains to choose from is better than 50. It also provides more options for subsequent analysis using other methods.
- Although thorough cleaning of sample processing equipment reduces the chance of contamination processing large samples reduces this chance even further.
- The costs of fieldwork, especially in remote areas, are high, therefore collecting as much sample as possible is a cost effective approach. Consider the cost of a return visit to collect more sample.

1.2 Land access

Sample collection requires significant planning following the identification of geological targets for analysis, and sufficient time should be allocated to investigate all planning aspects thoroughly. Land access is a significant consideration at this stage; areas of cultural sensitivity, traditional ownership, private property, national parks, and catchment regions require specific access permissions or permits. Permission must be sought well in advance of accessing the land and any agreed restrictions respected.

Access is a sensitive issue and must be taken seriously. A local’s negative experience with researchers can quickly become common knowledge in smaller communities and can cause negative responses to later requests in a wide area. Never sample from a location without permission to do so.

1.3 Site Selection and Safety

Choosing an appropriate outcrop for geochronological sampling is essential. Consider the in-situ nature and consistency of rock type throughout the outcrop; collecting rocks that are foreign to the outcrop will contaminate the sample. Where possible, it is best to obtain fresh rock samples (Figure 1.1) as deep weathering of the outcrop and fractures, where weathering can occur (Figure 1.2), may affect final results. A general guide to the quality of a rock is the tone of sound it makes when hit with a hammer. A fresh rock, particularly a granite tor, that is free of cracks and weathering may a have clear metallic tone when struck. Weathered or cracked rock may have a dull or hollow sound when struck.

Although most minerals used for geochronology are generally robust over geological timescales, weathering processes, especially those in a stable continent like Australia, can also occur over long periods to great depth and may induce removal of daughter elements such as lead from the minerals. This is classically seen as ‘zero-age lead loss’ in zircon U–Pb geochronology, where analyses plot on a concordia diagram along a line trending to zero age and in some cases may cause ambiguity in interpretation. If the purpose is to obtain an unambiguous isotopic analysis, evidence of alteration—where thermal events associated with the alteration and veining may have also induced changes in chemical or isotopic composition—should be avoided.

Also consider any cultural or aesthetic sensitivity that may be associated with the outcrop. Even with permission to sample it is best to do so as discretely as possible; for instance, try to sample well away from roads and tracks where a freshly sampled outcrop may contrast strongly with weathered surfaces and be considered unsightly.

Figure 1.1 A ‘suitable rock’: In situ monzonite outcrop, Tharwa, ACT, across the road from the deeply weathered and fractured outcrop in Figure 1.2. Note the fresh rock and lack of a deeply weathered rind and fractures.

Figure 1.2 A 'less suitable rock': In situ monzonite outcrop, Tharwa, ACT. Note the deep weathering from the top of the outcrop (left) and weathering along fractures (left and right).

General safety is a necessary consideration in site selection. Loose rocks, slippery surfaces, deep grass, fauna, creeks, logs, overhanging branches and rocks, rock cliff faces, downed wire fencing, and rubbish are all safety hazards; check beneath feet, head height and above during site selection for hazards. When a suitable site is selected sampling can commence (Figure 1.3).

In areas where contact with the public is possible, it is good practice to assign one member of the sampling party as a contact person to speak with any public bystanders to explain the activity and ensure they keep their distance.

1.4 Drilling and Splitting

Drilling and splitting is one method used to obtain large quantities of fresh sample. The drill operator must wear hearing, eye and respiratory protection as well as gloves, long pants and capped boots to avoid injury and dust inhalation. Bystanders must keep a distance of at least 10 m upwind of the sampling site and wear the same protective equipment to reduce the risk of dust inhalation and fly-rock impact during sledging. The operator and all bystanders should watch for back strain and fatigue of operators throughout drilling and sledging as they are physically intensive processes. Operators should alternate regularly to reduce risks associated with fatigue.

Figure 1.3 Prior to sampling. Note the safety aspects of the site: very few loose rocks, no slippery surfaces, short grass, away from the road, no overhead branches, rocks, cliffs or wires. There is plenty of room for other site staff to stand well away from the drill operator but still be available to assist if required.

Initially the rock is assessed to determine if a natural cleavage plane is apparent and two rows of holes are drilled accordingly (Figure 1.4, top left). Feathers and wedges are greased—to reduce friction and therefore the amount of time and effort required to split the rock—prior to being inserted into the first row of holes to split the facing surface off the rock (Figure 1.4, top right). The feathers and wedges are then inserted into the second row to split a second fresher layer off the rock (Figure 1.4, bottom left). The second layer is broken into smaller pieces using a sledge hammer (Figure 1.4, bottom right).

The rock pieces can be further treated in the field by chipping or napping off any unwanted parts such as weathering rinds or removing sharp edges for safer handling. Removing weathering rinds is an important consideration in reducing potential sample contamination. Even a narrow, seemingly intact rind may contain mineral grains not from the rock. Doing this in the field can save the expense of handling unwanted material and preparation time in the laboratory.

The fresh sample blocks are placed on the outcrop rather than the ground (Figure 1.4, bottom right) to reduce the risk of sample contamination prior to labelling and bagging for transport.

Sample numbers generated prior to field work are written on heavy duty bags and noted in a field notebook with a description of the rock. No more than 20 kg should be placed in one bag due to maximum single person lift limits. Individual bag weights must be assessed by sample collectors; it may be necessary to break the sample into smaller loads taking into consideration operator abilities and distance the samples need to be carried. Ensure sample numbers are on all bags, with bags labelled accordingly, for example: 1 of 2, 2 of 2. Bags are sealed to prevent rocks falling out and mixing samples. Never mix samples from different locations in a single bag.

Figure 1.4 Drilling the first row of holes in the boulder from Figure 3 (top left); Splitting off the face of the rock (top right) with second row of holes visible to the right of the feathers and wedges; Splitting the second layer (bottom left); Breaking the rock into manageable pieces with a sledge hammer (bottom right).

1.5 Site Clean-Up

Prior to leaving the site, soil, plant material and rock dust should be cleaned from equipment to reduce cross-contamination risks of both rock and biological material and all rubbish removed. The site should be tidied and left as natural looking as possible (Figure 1.5). This is particularly important in farming and pastoral lands where small, sharp rock chips may be a hazard to animals, workers or machinery.

Figure 1.5 Site clean-up. After sampling the boulder in Figure 1.3 and Figure 1.4 the site is arranged to look as undisturbed as possible; small chips are placed in crevices or tucked under larger rocks, large pieces are placed close to the outcrop and all rubbish and equipment is removed.

1.6 Further safety considerations

Two rock sampling scenarios warrant further consideration.

Firstly, ultramafic rocks, such as serpentinite, may contain asbestiform minerals that may not be apparent during immediate observation in the field. When sampling such rocks it recommended that extra precautions are taken such as using disposable coveralls (see section 4.2 for example), putting samples in puncture-proof samples bags and clearly labelling those bags with warnings for laboratory staff.

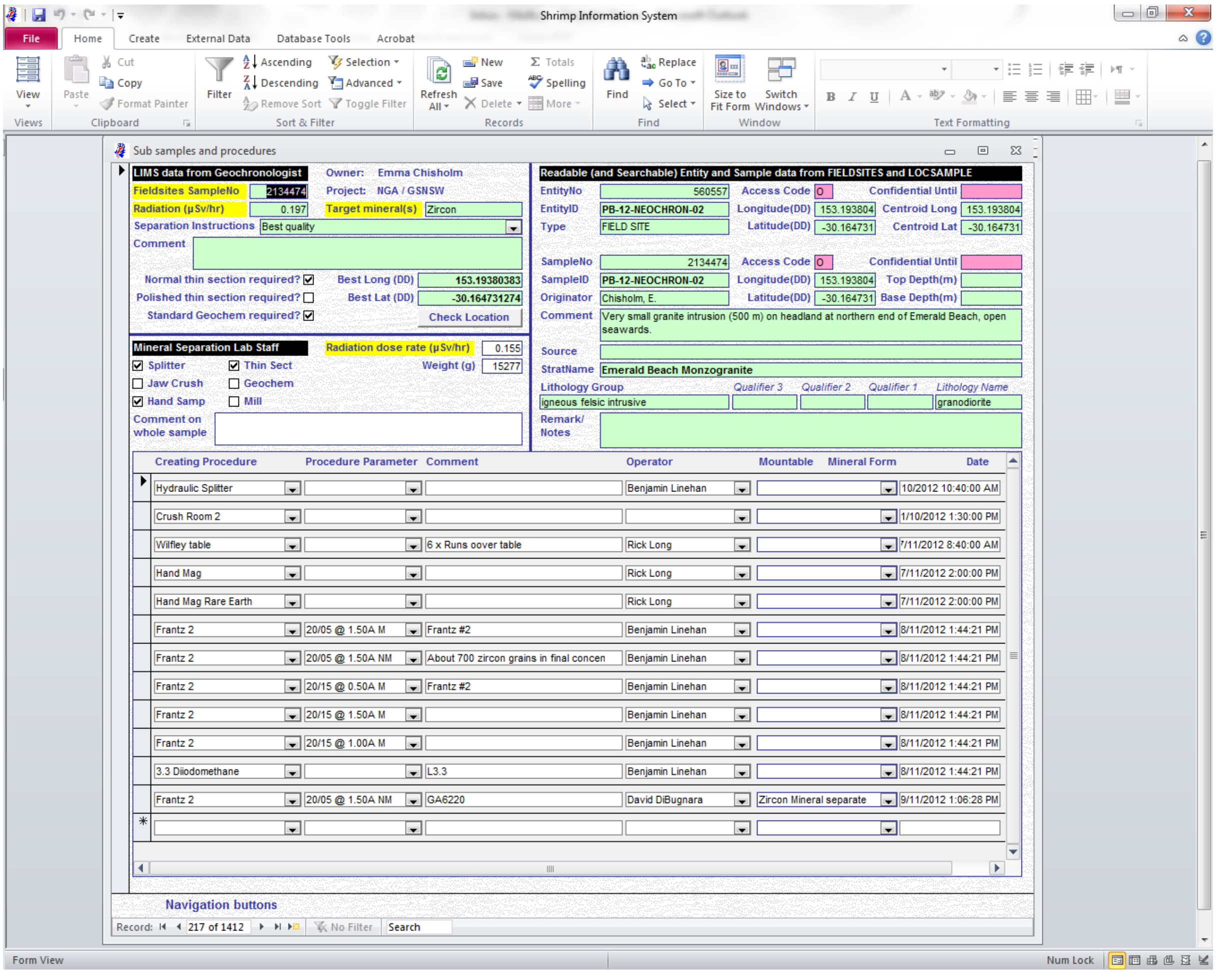
In the second scenario, rocks with naturally high uranium content may not be discernable from low to moderate uranium content during field observations. For this reason, ionising radiation safety protocols should be followed for collection of all samples, including taking radiation measurements in the field, to ensure that any samples are within specified limits to be handled by laboratory staff. Samples should be collected in puncture-proof samples bags with clear warning labels for laboratory staff.

In both scenarios researchers and sampling parties should consult relevant WH&S guidelines and seek advice before undertaking sampling.

2 Laboratory Systems and Sample Priority

On return from the field, the sample is assigned a unique GA Fieldsites Sample Number that correlates to the labels assigned in the field. These sample numbers are entered on all labels associated with sub-samples and are cross-checked throughout sample processing and preparation.

Sample information is then entered into the GA Sensitive High Resolution Ion Micro-Probe (SHRIMP) Laboratory Information Management System (LIMS) (Figure 2.1). Designed in-house, the SHRIMP LIMS is dedicated to acquiring data from all stages of sample processing and preparation to complement analytical procedures and reporting. A measurement of ionising radiation to check that the sample is within safety limits is also a required entry. Processing begins once labelling is complete and the sample is scheduled for isotopic analysis, be it in-house on the SHRIMP, or externally for isotopic analysis such as ID-TIMS.



LIMS data from Geochronologist

Owner: Emma Chisholm
Project: NGA / GSNSW
Fieldsites SampleNo 2134474
Radiation (μSv/hr) 0.197
Target mineral(s) Zircon
Separation Instructions Best quality
Comment
Normal thin section required? ☑
Polished thin section required? ☐
Standard Geochem required? ☑
Best Long (DD) 153.19380383
Best Lat (DD) -30.164731274

Mineral Separation Lab Staff

Radiation dose rate (μSv/hr) 0.155
Weight (g) 15277
☑ Splitter
☑ Thin Sect
☐ Jaw Crush
☐ Geochem
☑ Hand Samp
☐ Mill
Comment on whole sample

Readable (and Searchable) Entity and Sample data from FIELDSITES and LOCSAMPLE

EntityNo 560557
Access Code 0
Confidential Until
EntityID PB-12-NEOCHRON-02
Longitude(DD) 153.193804
Centroid Long 153.193804
Type FIELD SITE
Latitude(DD) -30.164731
Centroid Lat -30.164731

SampleNo 2134474
Access Code 0
Confidential Until
SampleID PB-12-NEOCHRON-02
Longitude(DD) 153.193804
Top Depth(m)
Originator Chisholm, E.
Latitude(DD) -30.164731
Base Depth(m)
Comment Very small granite intrusion (500 m) on headland at northern end of Emerald Beach, open seawards.
Source
StratName Emerald Beach Monzogranite

Lithology Group	Qualifier 3	Qualifier 2	Qualifier 1	Lithology Name
igneous felsic intrusive				granodiorite

Remark/ Notes

Creating Procedure	Procedure Parameter	Comment	Operator	Mountable	Mineral Form	Date
Hydraulic Splitter			Benjamin Linehan			10/2012 10:40:00 AM
Crush Room 2						1/10/2012 1:30:00 PM
Wilfley table		6 x Runs oover table	Rick Long			7/11/2012 8:40:00 AM
Hand Mag			Rick Long			7/11/2012 2:00:00 PM
Hand Mag Rare Earth			Rick Long			7/11/2012 2:00:00 PM
Frantz 2	20/05 @ 1.50A M	Frantz #2	Benjamin Linehan			8/11/2012 1:44:21 PM
Frantz 2	20/05 @ 1.50A NM	About 700 zircon grains in final concen	Benjamin Linehan			8/11/2012 1:44:21 PM
Frantz 2	20/15 @ 0.50A M	Frantz #2	Benjamin Linehan			8/11/2012 1:44:21 PM
Frantz 2	20/15 @ 1.50A M		Benjamin Linehan			8/11/2012 1:44:21 PM
Frantz 2	20/15 @ 1.00A M		Benjamin Linehan			8/11/2012 1:44:21 PM
3.3 Diiodomethane		L3.3	Benjamin Linehan			8/11/2012 1:44:21 PM
Frantz 2	20/05 @ 1.50A NM	GA6220	David DiBugnara		Zircon Mineral separate	9/11/2012 1:06:28 PM

Figure 2.1 Example of sample and sub-sample information entered into LIMS throughout sample processing. Initially the information that is entered provides the laboratory with separation instructions and sample requirements. For example, recording that a thin section and geochemical analysis is required and the type of grains to select in the final stages of processing determines the appropriate separation processing procedures.

3 Receipt of Samples to the Laboratory

Samples not ready for processing are held in a designated warehouse storage area outside of the laboratory (Figure 3.1) to reduce cluttering the workspace and the risk of contamination to samples being processed. Samples are only moved to the layout room (Figure 3.2) prior to commencement of processing.

Figure 3.1 Geochronology sample storage area.

Figure 3.2 Laboratory layout room.

4 Sample Splitting

Samples ready for processing commence separation with pre-treatment procedures. Dirt remaining from the field is washed and rinds are removed on a saw. Appendix A provides an overview of the sample processing workflow undertaken in the GA Mineral Separation Laboratory.

The sample is broken down using one of two hydraulic presses within the laboratory, reducing the large blocks to manageable pieces (Figure 4.1). Any remaining weathering rinds are removed at this stage using a rock saw. The broken pieces are thoroughly washed in an ultrasonic bath to remove dust and contaminants and dried in an oven (<150°C to prevent thermal resetting of any target minerals) prior to progressing to crushing. Sample numbers are written on all bags and cross-checked as the sample progresses to the crushing stage, or are packed away for later processing.

Figure 4.1 Split rocks in the hydraulic splitter.

Two split blocks are chosen; one is retained as a representative hand specimen of the sample which follows the sample through all stages of processing to aid determination of separation methods, the other is sent to an external facility where a thin-section is made. Sample numbers are written on both the blocks and the bags and cross-checked. Once thin-sections are returned to the laboratory the mineral arrangement within the rock is assessed and the target mineral within the matrix observed; this can guide the choice of separation technique, especially for small or precious samples, or unusual separations or minerals.

4.1 Contamination and Cleaning

Although the laboratory has two hydraulic splitters in the splitting room (Figure 4.2), only one sample is processed in the room at any one time to reduce the risk of cross-sample contamination. Equipment is cleaned thoroughly using a vacuum cleaner and compressed air to remove hidden dust, and is then wiped over with a damp cloth.

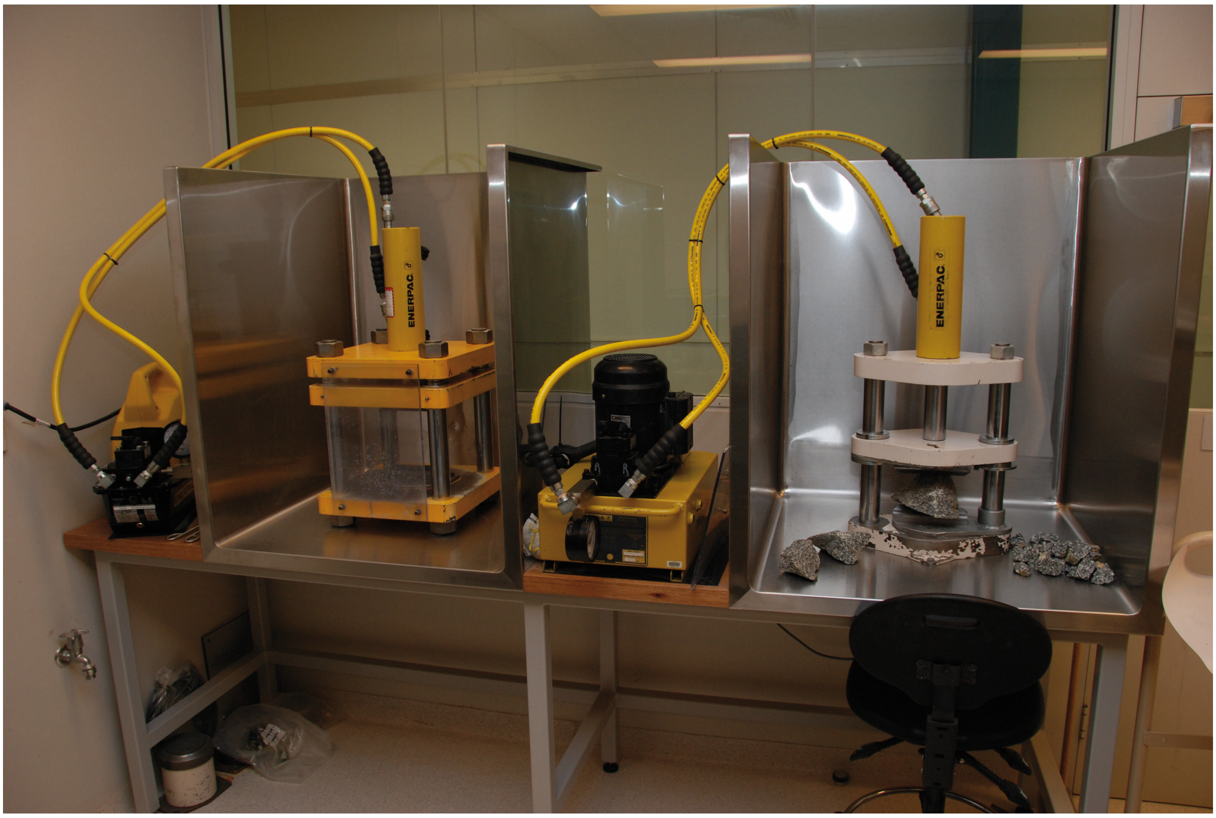


Figure 4.2 Hydraulic splitters and bays in the splitting room.

4.2 Safety

Safety is paramount while using the hydraulic press. Risks include crush injuries to hands and fingers, back strain, fatigue, impact from flying debris—small shards of rock up to fist sized blocks—and inhaled rock dust. For these reasons safety equipment and Personal Protection Equipment (PPE) is essential. Impact grade safety glasses or a full face shield, respiration mask, capped boots and a laboratory coat must be worn. A thick Perspex barrier is used to guard against flying debris. Fatigue and back strain are unique to individuals, however, it is recommended that splitting activities are undertaken for no more than 10 minutes at a time to prevent these risks and no more than 20 kg are lifted by an individual at one time.

Protection from samples that contain potentially dangerous minerals, such as asbestos fibres, is an essential component of sample handling. Figure 4.3 illustrates the PPE used in the GA Mineral Separation Laboratory when handling a serpentinite potentially containing asbestiform minerals.

Figure 4.3 Sample safety. Personal Protective Equipment is an essential component of sample handling, and paramount when handling samples that contain dangerous minerals such as asbestos. In this image, the scientist is wearing a full protective suit, including shoe coverings, earmuffs (the saw in the background is in use), gloves, a respirator and wrap-around goggles.

5 Sample Crushing and Milling

After splitting, the sample is further reduced to rock chips in a jaw crusher and is then milled to a powder to liberate target mineral grains. Target grain size generally falls between 20 and 400 µm, with the majority between 50 and 150 µm.

The GA Mineral Separation Laboratory maintains two crushing rooms, each configured with different crushing and milling equipment. Both rooms have advantages and disadvantages and provide the Mineral Separation facility with significant flexibility in sample processing.

Crushing Room 1 (Figure 5.1) houses a dust extractor, Rocklabs Boyd Crusher, a Rocklabs Continuous Ring Mill (CRM) and a small bench-top Shatter Box. The Boyd Crusher (Figure 5.2, left) has large flat plates and requires samples to be split to a maximum size of ~40 mm diameter to prevent the blocks grinding rather than crushing, however, the final crush produced is consistent in size and readily feeds into the CRM.

Figure 5.1 Crushing room 1: L–R dust extractor, Continuous Ring Mill and Boyd Jaw Crusher.

The CRM is excellent for large samples due to the continuous feed system and its collection drum can hold up to 10 kg of rock flour (Figure 5.2, centre). The CRM is a closed system; the rock flour cannot be checked during processing so this machine is used for routine samples where output consistency is necessary. The shatter box (Figure 5.2, right) is used for small samples up to 500 g and can mill 100 g at a time. Final grain size can be varied in both machines; an automatic dial operates this function in the CRM but grain size determination is a manual process in the shatter box and is checked during processing.

Figure 5.2 Crushing Room 1: Boyd jaw Crusher (left), Continuous Ring Mill (centre), Rocklabs Shatter Box (right).

Crushing Room 2 (Figure 5.3) houses a dust extractor, an older Sturtevant jaw crusher (Figure 5.4, left) and a Fritsch P13 disc mill (Figure 5.4, right) which are used generally for samples up to 10 kg. The jaw crusher can take split blocks up to 100 mm diameter by adjusting the grooved plates. The width between the plates can be easily adjusted to obtain variations in crush size, useful for obtaining a finer or coarser crush on a small sample.

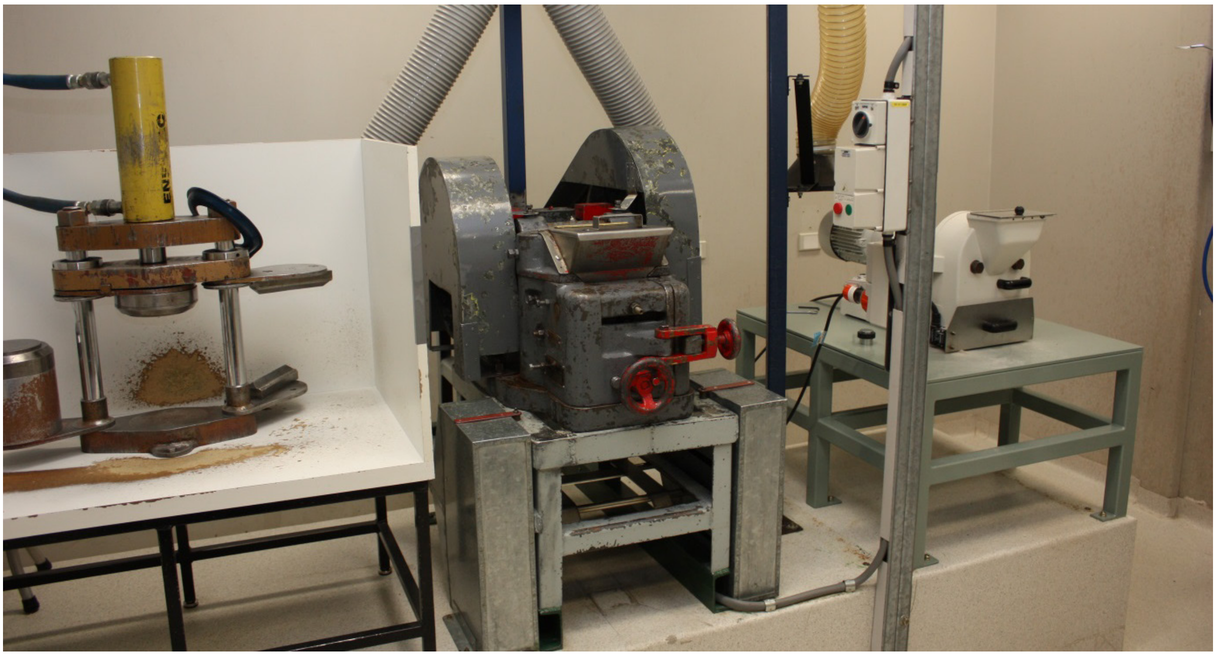


Figure 5.3 Crushing Room 2: L–R Hydraulic Splitter, Jaw Crusher, Disc Mill.

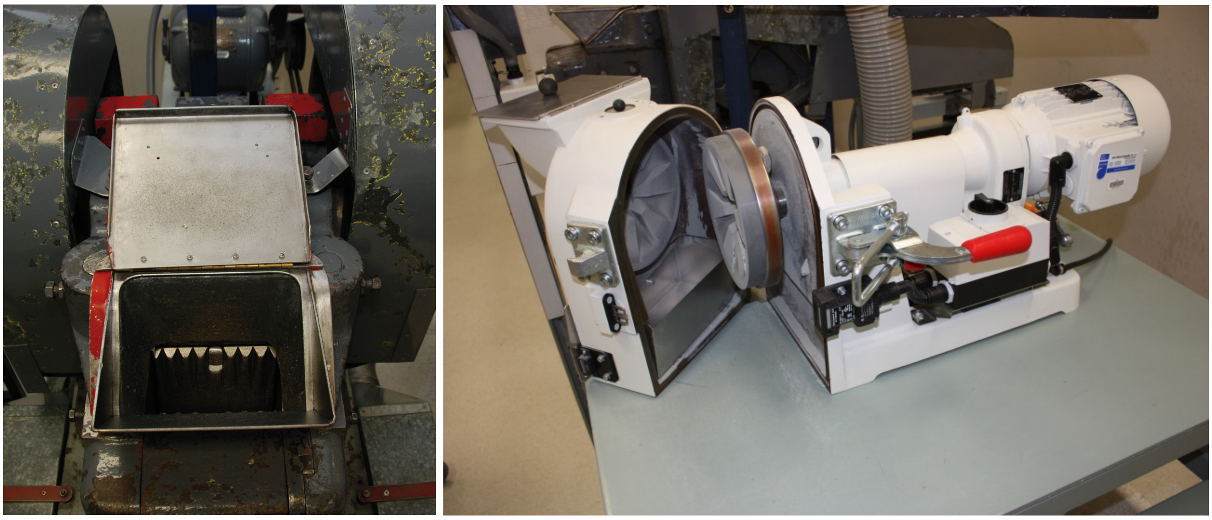


Figure 5.4 Crushing Room 2: Sturtevant Jaw Crusher (left), Fritsch P13 Disc Mill (right).

The disc mill has both advantages and disadvantages compared to the CRM. The spacing between the discs can be significantly altered during processing to vary the final grain size—the feed system in the disc mill is manual and a small collection tray at the base is easily removed between feeds to check grain size. This ability makes the disc mill useful for liberating micas and feldspars for K–Ar and Ar–Ar dating as grain size output can be checked constantly. Large samples take a significant amount of time to process in the disc mill due to the small capacity manual feed system. Friction on hardened steel discs causes the discs to heat rapidly and expand, decreasing the gap and resulting in a finer grind. For this reason, samples are run through a mill with hardened steel discs slowly to ensure grain size consistency; the P13 is fitted with tungsten carbide discs that do not heat up nor expand as much as regular tool steel to avoid grind inconsistency.

After crushing, and prior to milling, two representative sub-samples are taken; one aliquot for geochemical analysis and one as an archived representation. The sub-sample is carefully labelled with all sample numbers and cross-checked. When crushing and milling of the sample is complete the rock flour is bagged in preparation for further separations, sample numbers are written on the bags, cross-checked and stored with the hand specimen in a dry area (Figure 5.5).

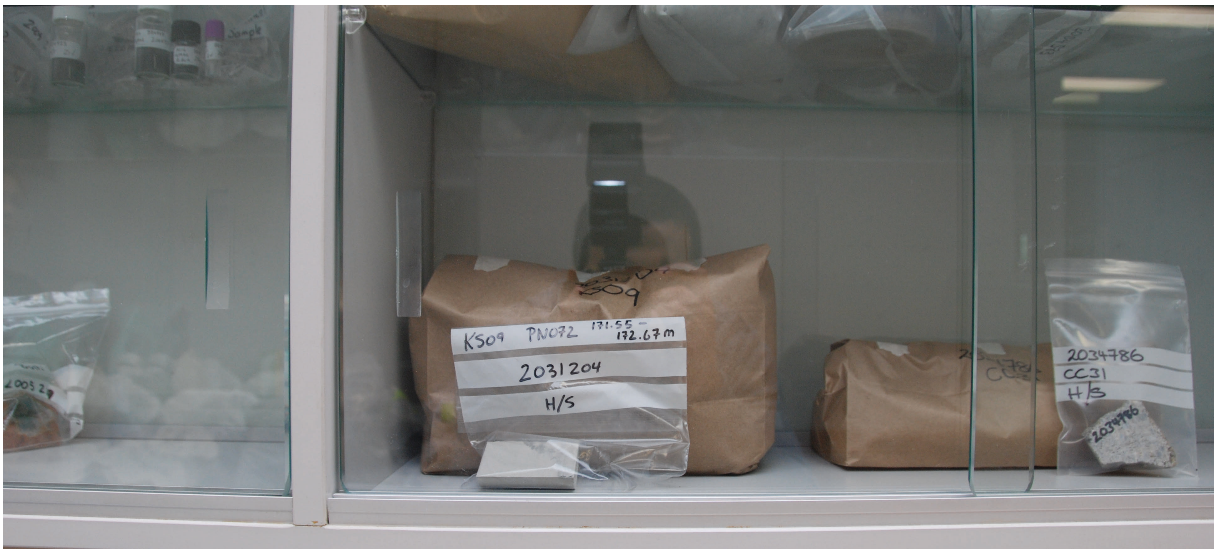


Figure 5.5 After crushing the sample is packed up ready for further separation.

5.1 Contamination and Cleaning

Contamination risks in the crushing rooms are high. There are many places grains can collect in the crushing and milling equipment and the cleaning regime in the crushing rooms, as with all areas of the laboratory, is rigorous. A 20 kg sample will take an hour to be completely crushed in Crushing Room 1, yet cleaning and preparation for the next sample can take up to five hours.

Rooms are thoroughly cleaned between samples; machines are taken apart and vacuumed prior to using compressed air to remove residual dust. Crushing plates, rings and discs are wet-sandblasted using 45–90 µm glass bead media to remove trapped residue and then washed in an ultrasonic bath with bolts, nuts and other equipment components used for processing samples (Figure 5.6). The non-removable components of the machines are thoroughly wiped with a damp cloth and air dried. The floor and all other areas where dust collects (including the extraction fan and motor housing) are thoroughly cleaned and the rubbish bin is emptied between samples. Lab coats are changed between each sample to reduce spread of contaminant grains.

Figure 5.6 Removable components of Crushing Room 1 Boyd Jaw Crusher and Continuous Ring Mill.

5.2 Safety

The variable nature of the equipment in the crushing rooms requires specific understanding of the risks associated with use. Dust is a major hazard while using crushing equipment. While every effort is made to reduce ambient rock dust during machine operation by sealing equipment, dust extractors are used to remove internal dust, and respiratory masks are worn during processing and cleaning to reduce the risk of fine particle inhalation. Dust and particle inhalation is particularly an issue when handling material, such as ultramafic rocks, that may contain asbestiform minerals; full cover such as that in Figure 4.3 is recommended.

The crushing rooms are high-level noise environments and ear protection must be worn in the form of either high rated ear muffs or plugs. Safety glasses are essential to avoid eye injury from rock chips while using crushers and mills, capped boots must be worn to prevent injury to the toes from dropped rocks, tools and crushing equipment during processing and equipment disassembly and reassembly. Laboratory coats are worn to reduce the risk of contamination to clothes and carrying contaminants into other areas of the laboratory.

5.3 Sample effects

Early studies in processing granite samples suggest that there is little or no biasing of zircon morphology caused by simple mechanical crushing (Larsen and Poldervaart, 1957). However, more recent work indicates substantial loss of any low-temperature or altered zircon present (Hay and Dempster, 2009) so mechanical grain liberation processes may need to be avoided with in-situ mounting of material in thin section.

6 Gravity Separation Using Water

Once a sample has been crushed and milled it is ready for gravity separation using water. The GA Mineral Separation Laboratory uses a Wilfley Table (Figure 6.1; Niebur and Fell, 1982) for desliming bulk rock flour as a first step towards reducing sample composition to minerals of interest. Using the vibration and slope of the table, the heavier and lighter minerals are separated by a divider and wash into different collection containers. Lighter minerals such as quartz and feldspars wash into the lower portion of the table and can be captured and retained if these minerals are also to be targeted in a separation. The heavy mineral component of the sample moves along the uppermost portion of the table and is captured as it washes off (Figure 6.2). The majority of target minerals, such as zircon, pyroxenes, micas, amphiboles, magnetite, pyrite, apatite etc. are found in the heavy fraction.

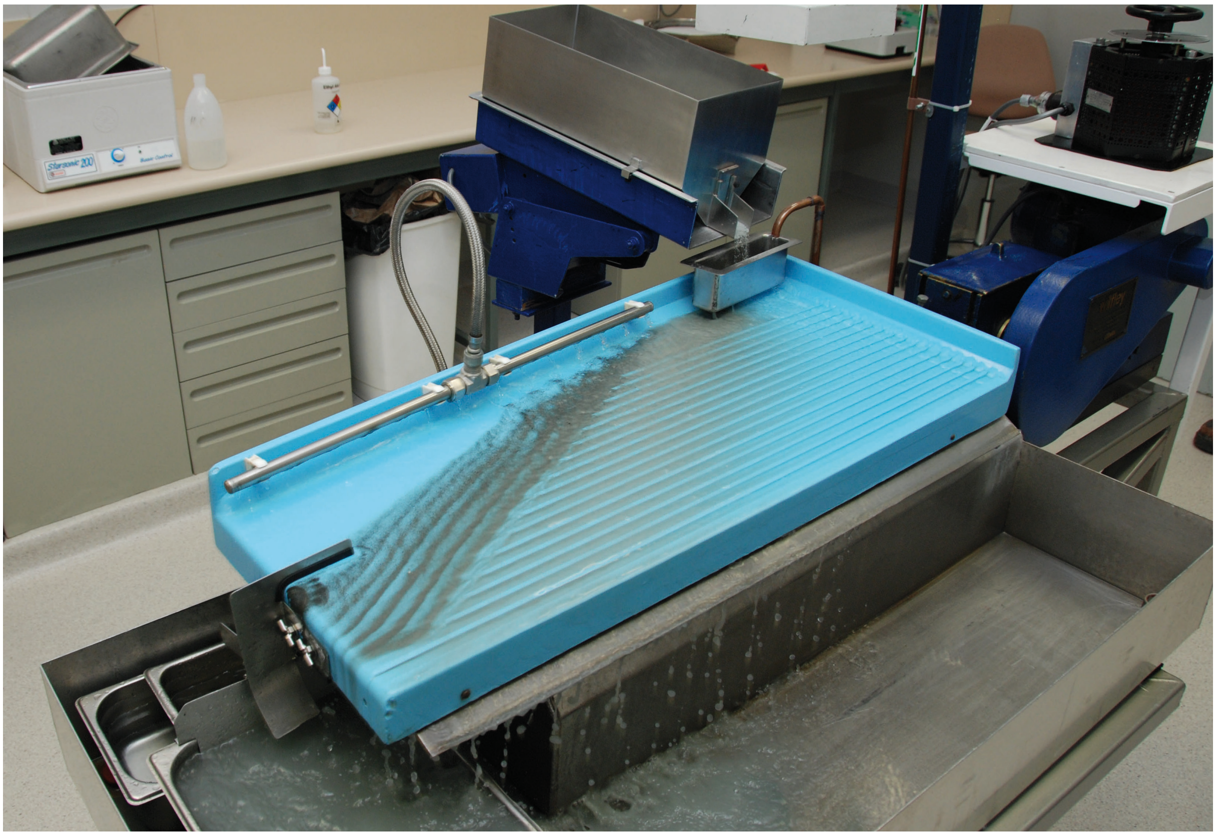


Figure 6.1 Wilfley Table in operation.

Lighter minerals are frequently entrained in the heavy mineral flow on the Wilfley Table. As a result the heavy fraction is run over the table numerous times to reduce the light mineral fraction to a minimum. This additional level of processing has reduced the need in the laboratory for further gravity separation using bulk heavy liquids.

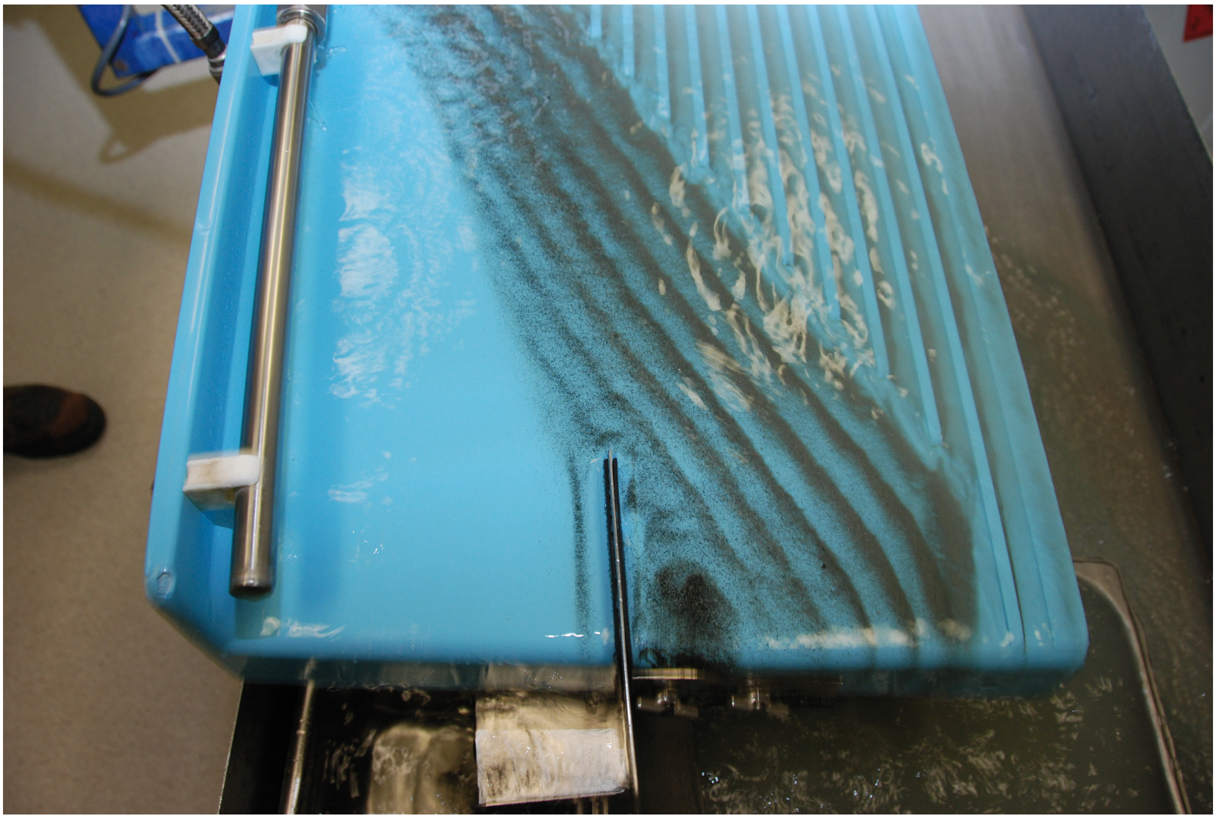


Figure 6.2 Close up image of sample split of heavy minerals and lighter minerals on the Wilfley Table.

Samples smaller than 500 g are deslimed in an elutriation system (Figure 6.3) to prevent potential loss of valuable analytical grains (see examples in Frost, 1959; Van der Westhuizen et al., 1993).

Grains are placed in a large separating funnel and water flows at a controlled rate, allowing the light material to pass out through a top exit port. Stokes Law is applied to this method enabling the determination of settling rates to retain grains of a particular specific gravity and size versus the inflow pressure of water. When the column of water is clear the desliming separation is complete.

After processing, the sample is dried under heat lamps. The more magnetic components are removed by a hand magnet, followed by rare earth magnets to remove the para-magnetic material (see section 7). The sample is then packed up, labelled and stored with the hand sample prior to further magnetic and heavy liquid processing.

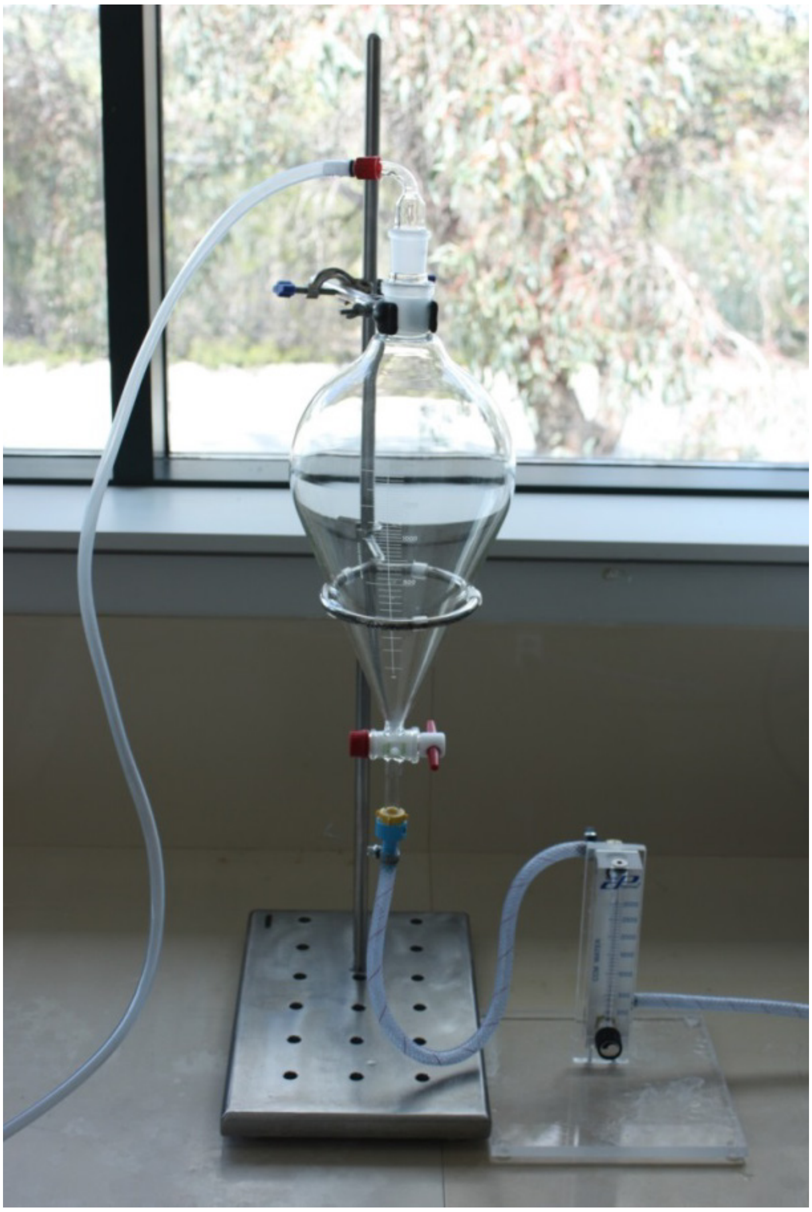


Figure 6.3 Elutriation system.

6.1 Contamination and Cleaning

Only one sample at a time is processed in the Wilfley Room to reduce the risk of sample cross-contamination. Cleaning of the Wilfley Table and associated equipment is thorough as there are grain traps on the table surface and in the removable equipment. Once disassembled, the table and basin are scrubbed and washed to remove residue. Associated equipment is scrubbed, washed, cleaned in an ultrasonic bath and air dried. Finally, the Wilfley Table is wiped over and compressed air is used to remove any residue that may remain.

6.2 Safety

The Wilfley Room is considered a wet area due to running water and associated splashing that may occur on the floor. It is imperative that care is taken in this environment to avoid slips and falls. Rubber soled shoes must be worn at all times. The use of compressed air requires ear and eye protection to be worn and general care is taken to ensure fingers do not become caught in moving parts on the Wilfley Table. Dust in this environment is minimal; however, a small dust extraction fan sits above the dry sample as it moves down the vibrating chute to capture the fine material that is disturbed by the vibration.

6.3 Sample effects

Sláma and Košler (2012) explicitly tested for preferential loss of the small-grain fractions in a synthetic detrital zircon sample when using a Wilfley Table and found no loss occurred.

7 Magnetic Separation

Minerals can be discriminated against using their magnetic properties, allowing for refinement of the mineral concentrate and removal of minerals with varying degrees of magnetic susceptibility.

Initially, minerals with high magnetic susceptibility (such as magnetite) are removed using a low level hand magnet. The hand magnet also removes any metallic residue from the crushing plates. Rare earth magnets can be employed at this stage if there are significant amounts of minerals with a high to moderate magnetic susceptibility (such as amphiboles, pyroxenes, biotite and garnet). The use of the hand magnet and rare earth magnets reduces the time the sample needs to run on the Frantz Magnetic Separator, yet still provide an effective early stage magnetic separation (Figure 7.1).

Figure 7.1 Hand magnet and rare earth magnet separation after Wilfley Table.

Magnetic separation using a Frantz Magnetic Separator is arguably the most essential method for bulk heavy mineral concentration; induced magnetic fields at various electric current settings interact with mineral grains as they pass through the magnets at a set slope and tilt to further separate the sample according to the magnetic susceptibility of the mineral grains (Figure 7.2 and Figure 7.3). Surface slicks, smears, coatings, cracks, inclusions and internal fractures may be discriminated against in order to select the highest quality grains for analysis. The Frantz is the only piece of equipment in a mineral separation laboratory that allows for refined mechanical discrimination of high quality zircons for isotopic analysis.

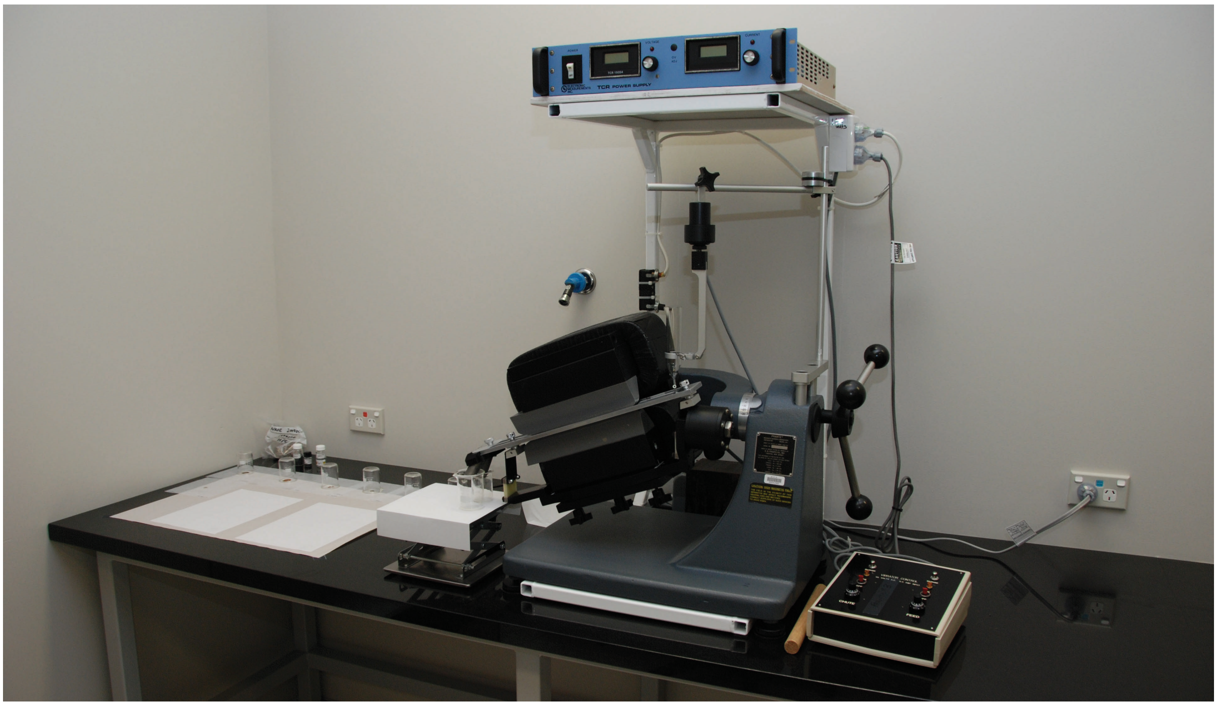


Figure 7.2 Frantz Magnetic Separator.

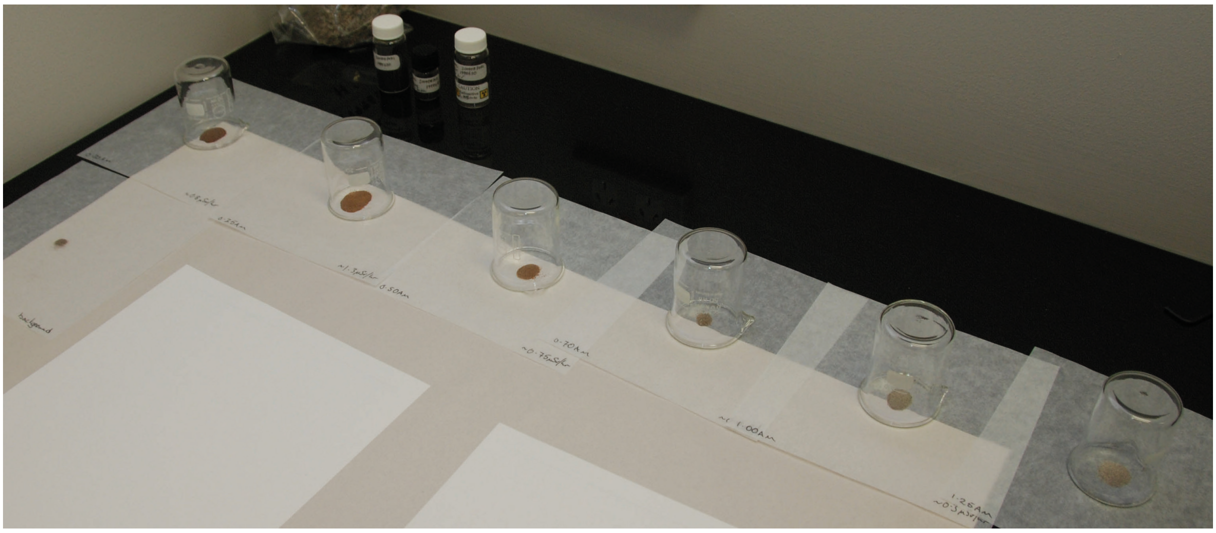


Figure 7.3 Image of Frantz separated mineral fractions.

7.1 Contamination and Cleaning

The risk of contamination is greatly increased at this stage of processing; as the sample is so highly concentrated, one foreign grain introduced at this stage has a much greater chance of reaching final stages of processing and being analysed. Three Frantz Separators run at one time in a purpose built room in order to reduce contamination risk from bystanders. Grains are loose in the Frantz room during separation, often sitting on the bench uncovered; it is important that samples are not handled unnecessarily and benches are not leant on to reduce the risks of cross-contamination.

Cleaning is of extreme importance to avoid contamination between samples run on the same Frantz. The feeder, separator and beakers are all removed and washed in non-abrasive soap, thoroughly rinsed and air dried. The chute is removed and all chute attachment bolts are cleaned in an ultra-sonic bath. The chute is carefully cleaned using a non-abrasive soap and hot running water to avoid scratching the surface and creating holding traps for grains. The chute is then wiped with petroleum ether to remove any traces of grease or oil. All removable Frantz components parts are blown with compressed air. All surfaces of the Frantz are vacuumed to remove trapped grains. The magnet poles are wiped thoroughly with an ethanol soaked cloth. After wiping the equipment and benches with a damp cloth the Frantz is reassembled for the next sample.

7.2 Safety

The Frantz Magnetic Separator has a strong magnetic field (up to 2 Tesla / 20,000 Gauss) that is unsafe for people with pacemakers. Analogue watches are also affected by the magnetic field and can lose time over a day of intense Frantz work. Other significant risks in the area include inhalation if samples are significantly disturbed, or ingestion from transporting grains from fingers to food. Protective equipment, including gloves and eye protection, must be worn while working in the Frantz room; these must be removed and hands washed between samples and prior to eating.

Because mineral grains become highly concentrated when separated, ionising radiation is an issue especially for naturally occurring radioactive minerals such as monazite and uraninite. Finger badges are worn and the sample activity is measured using a scintillometer at all stages of processing and recorded. Risk management is essential—controlled point source, length of time in contact, and distance from the hazard are the best risk management procedures.

7.3 Sample effects

In zircon geochronology magnetic separation is valued because of the correlation between concordance and low (even negative) magnetic susceptibility (Silver, 1963). However in sedimentary provenance studies such focus on the highest quality zircon may bias the proportions of zircon age components in a sample (Sircombe and Stern, 2002). In sedimentary studies the general recommendation is to sample a broad range of magnetic susceptibilities although this needs to be balanced against the need to efficiently collect reasonable quality data (Neumann et al., 2009).

8 Gravity Separation Using Heavy Liquids

Using known variations in specific gravity between different minerals, heavy liquids are used to further concentrate minerals required for analysis (Krumbein and Pettijohn, 1938; Callahan, 1987). Four types of heavy liquid are used in the GA Mineral Separation Laboratory:

- Tetrabromoethane (TBE), density 2.96 (CAS Number: 79-27-6)
- Di-iodomethane (DIM), density 3.3 (CAS Number: 75-11-6)
- Lithium Heterotungstate (LST), density 2.86 (CAS Number: N/A)
- Sodium Polytungstate (NaPT), density 2.9 (CAS Number: 12141-67-2)

The density of these liquids (with the exception of DIM) may be varied with the use of solvents depending on the mineral separates required.

TBE and DIM are toxic substances and their use demands careful handling to reduce risks to personnel. The GA Mineral Separation Laboratory has almost completely eliminated the need to use TBE in day-to-day separation work due to the refinement of bulk heavy mineral concentration techniques using the Wilfley Table, however, some specific targeted minerals still require the use of TBE with modified densities to separate minerals such as feldspar from quartz.

Following initial Frantz work a standard zircon separation will be put in DIM to separate minerals lighter than 3.3 specific gravity, such as apatite, from those that are heavier, such as zircon and pyrite (Figure 8.1). Elutriated samples less than 500 g have their initial density separations done in either LST or NaPT.

Following Heavy Liquid separation, the ‘heavy’ concentrate fraction is returned to the Frantz at varying tilts on the chute and high amplitude to remove the more metamict zircons and achieve the purest zircon concentrate for a particular sample. The sample is then ready for handpicking; the final selection and concentration stage prior to isotopic analysis.

8.1 Contamination and Cleaning

The Mineral Separation Laboratory has six fume cupboards. Only one sample is worked on in each fume cupboard at any one time to reduce the risk of cross-sample contamination. Sample labels are cross-checked numerous times during processing and packing as numerous samples can be worked on in the Heavy Liquids Room at once.

Glassware used for heavy liquids is thoroughly cleaned between samples using solvents and detergent to remove residue. Fume cupboards are wiped out and heavy liquids are filtered regularly to remove possible residual grains and thereby avoid cross-sample contamination. Solvent and heavy liquid residues from sample preparation are disposed of through an authorised chemical disposal facility.

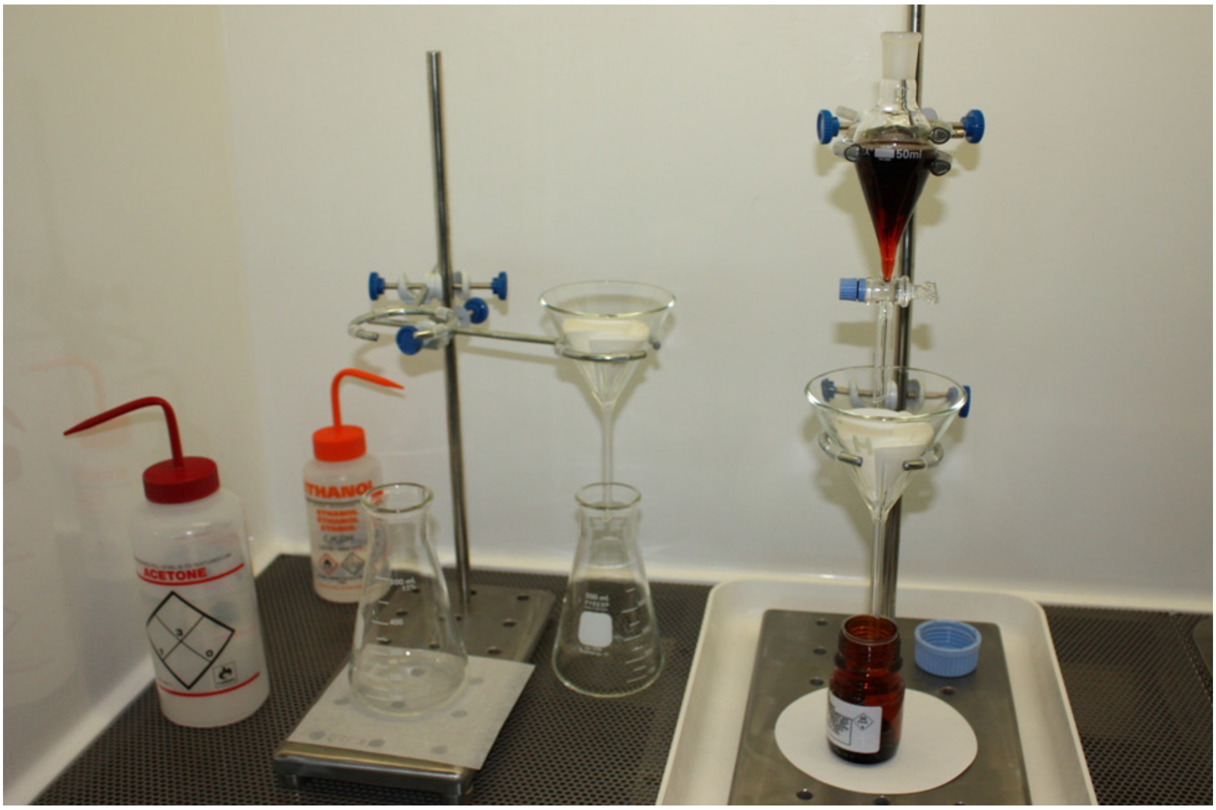


Figure 8.1 Heavy liquid separation set-up using diiodomethane (SG 3.3) in a down-draft fume cupboard.

8.2 Safety

The use of heavy liquids requires specialist equipment, including down-draft fume cupboards as vapours from the heavy liquids sink, rather than rise as lighter vapours do. The six down-draft fume cupboards in the Mineral Separation Laboratory are housed in a in a purpose-built high-draft room.

Heavy liquids are highly toxic; exposure can result in permanent damage to the central nervous system. PPE is essential while using heavy liquids; gloves, splash-proof eyewear, laboratory coats, long pants and boots are mandatory for work in the Heavy Liquids Room. Splashes and spills are avoided by careful handling, and handling only when absolutely necessary.

8.3 Sample effects

Sláma and Košler (2012) found preferential loss of the small-grain fractions in a synthetic detrital zircon sample using heavy liquid separation processes alone, possibly due to electrostatic build-up during handling of the sample more likely to affect the smaller grains.

9 Mineral Concentrates and Grain Selection

Final pure mineral concentrates for isotopic analysis are achieved through hand-picking individual grains using a stereo binocular microscope. Grain selection is based on the information being sought from the sample (such as an emplacement or crystallisation age, a maximum depositional age or a deformation age) and can vary significantly between samples.

A sedimentary sample may contain grains from numerous sources; therefore, grains can be of various ages. A sedimentary rock with no fossils to allow for biostratigraphic assignment can only be assigned an isotopic age relative to the youngest grains analysed in the sample, that is, a sedimentary rock cannot be older than the youngest grain contained within the rock, providing a maximum age for deposition of the unit only. For this reason, detrital grains are not selected for specific characteristics to avoid biasing the sample and generally more grains in a detrital population are analysed in order to obtain a representative spectra of ages than in a single population igneous sample.

Grain selection for igneous populations is based on grain clarity and crystal integrity. Ideally, the ‘best quality’ selection contains grains that are euhedral, transparent, and fracture and inclusion free. Some grains may contain cores and selection of these grains is dependent on analytical requirements.

Grain selection for a metamorphic sample generally focusses on grains with both cores and rims, to aid determination of the range of protolith and metamorphic ages that may be present in the sample, taking into account grain clarity and crystal integrity.

Grains are placed in labelled Petri dishes with ethanol (Figure 9.1). Ethanol provides a liquid base to highlight grain variation and provides a degree of resistance to the grains during handpicking to prevent them flicking out of the Petri dish. Additionally, the Refractive Index (RI) of ethanol is much lower than the RI of minerals targeted in the laboratory which facilitates visual distinction of, for example, analytical quality zircon grains from grains such as quartz and apatite. Where possible, approximately 400 grains per sample are chosen for mounting.

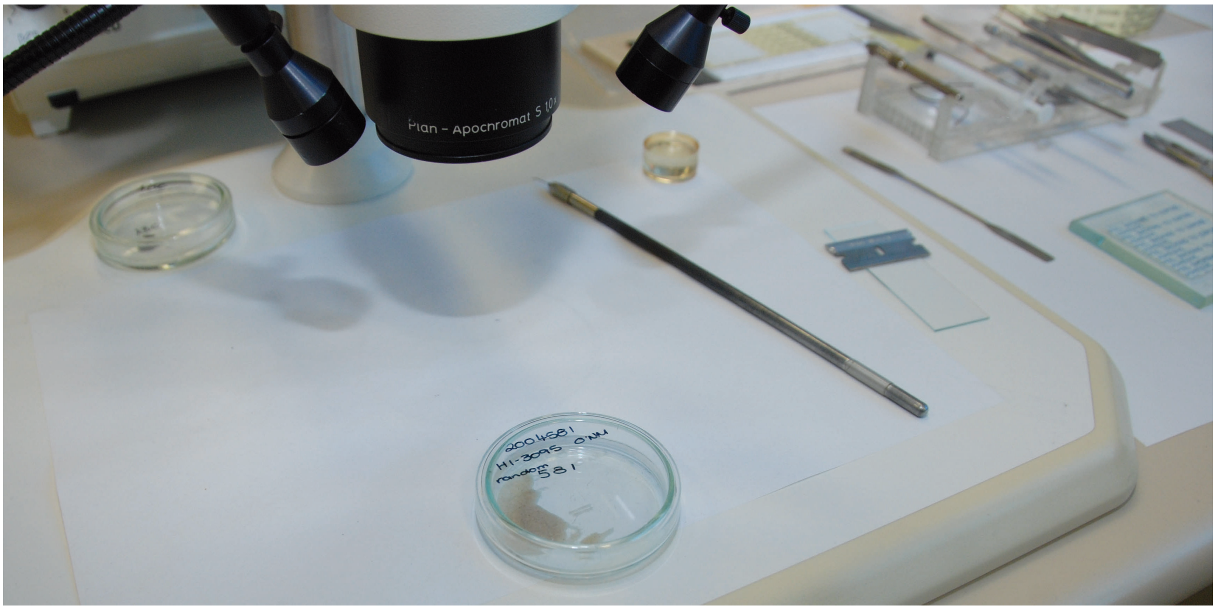


Figure 9.1 Zircon grains being handpicked in a Petri dish with ethanol.

9.1 Contamination and Cleaning

Contamination risk between samples during grain selection can be quite high. Procedures to mitigate the risks include: thoroughly cleaning the station prior to handpicking a new sample, cleaning the hand picking tool regularly and especially when changing samples, maintaining sample numbers on the top and bottom of Petri dishes, and not reusing syringe tips; all standard practice in the laboratory. Petri dishes are cleaned thoroughly between samples using an ethanol rinse, washed in a detergent bath, and are then rinsed with de-ionised water prior to air-drying.

10 Making a SHRIMP Mount

Selected grains are mounted in a 25 mm diameter epoxy resin disc for analysis on the SHRIMP (Figure 10.1).

Making a SHRIMP mount is a precise and time consuming process with exacting requirements (outlined in section 12). Prior to making a mount, double sided tape is smoothed onto a glass plate and a row template is made from plastic film. The template is cut with a row width of 0.4 mm and length of 7 mm (to a maximum of 8 mm); this is the ideal row width for imaging the grains on the Scanning Electron Microscope (SEM), allowing 150× magnification within a single frame, and the ideal row length for analysis on the SHRIMP, as the defined analytical area is 10 mm diameter.

Figure 10.1 SHRIMP mount after gold coating and analysis (note surfaces scratches around the edge from the SHRIMP mount holder). Diameter of mount is 25 mm, grain rows are approximately 7 mm length x 0.4 mm width, placed within the 10 mm x 10 mm SHRIMP analytical area.

Using the row template the grains are carefully transferred between the Petri dish and the mount plate; grains are removed from the Petri dish using a disposable syringe, placed on the edge of a razor blade and transferred from the razor blade to the mount plate using a hand picking tool or tweezers. Grains are then gently pressed onto the tape individually to hold them in place and prevent grain transfer between rows when the resin is poured. Once a row is mounted, the template is moved to create a space between rows of approximately 0.6 mm; the next row is then mounted.

Each row contains between 250 and 400 grains depending on grain size. After all rows have been placed, a plastic mould is placed over the mounted samples and standards. Epoxy is poured into the mould and left to harden overnight; the mount is then placed in an oven set at 60°C for up to 48 hours to cure.

Once the mount is fully cured, the back is cut to a mount thickness of 6 mm to fit the mount holder in the SHRIMP. The back must be cut parallel to the mount face as any gradient will result in analytical issues. The face of the mount is then polished; the mount face and the grains must have minimal relief as the SHRIMP is sensitive to grain relief and this will result in analytical errors. Careful polishing is essential to maintain parallelism and a flat surface on the mount.

Polishing is a semi-automated process undertaken on equipment with predefined polishing programs (Figure 10.2). Polishing begins with a 9 µm diamond abrasive to remove the surface layer of epoxy and cut the grains back to reveal the internal structure. Ideally grains are cut to almost half their depth to reveal cores, rims and other internal structures of interest. Polishing is then refined through 3 µm, 1 µm and 0.25 µm abrasives to produce a high sheen and remove scratches.

Figure 10.2 Struers TegraPol-15, TegraForce-1 and TegraDoser-5 polishing equipment.

Issues occur during polishing when very small grains are placed on the same mount as very large grains. The mount can only be polished to a depth that will reveal the smaller grains; polishing to reveal the larger grains in their entirety will result in smaller grains either plucking-out or being polished out completely. Conversely, polishing to only reveal the smaller grains can result in poor exposure of the larger grains.

The mount is cleaned after polishing using ultra-pure Milli-Q water and is lightly coated with a 2 nm layer of gold. The mount is then imaged using various techniques to aid analysis.

11 Imaging a SHRIMP Mount

The mount is imaged using reflected light, transmitted light and Cathodoluminescence (CL) and/or Back-scattered Electron (BSE) imaging techniques to highlight the physical and bulk elemental mineral properties (Figure 11.1). Initially a map of the entire mount is made at low magnification (2.5× to 5×) using reflected light then each row is imaged at higher resolution (10×) in reflected and transmitted light. CL and BSE imaging is done at much higher magnifications (100–150×).

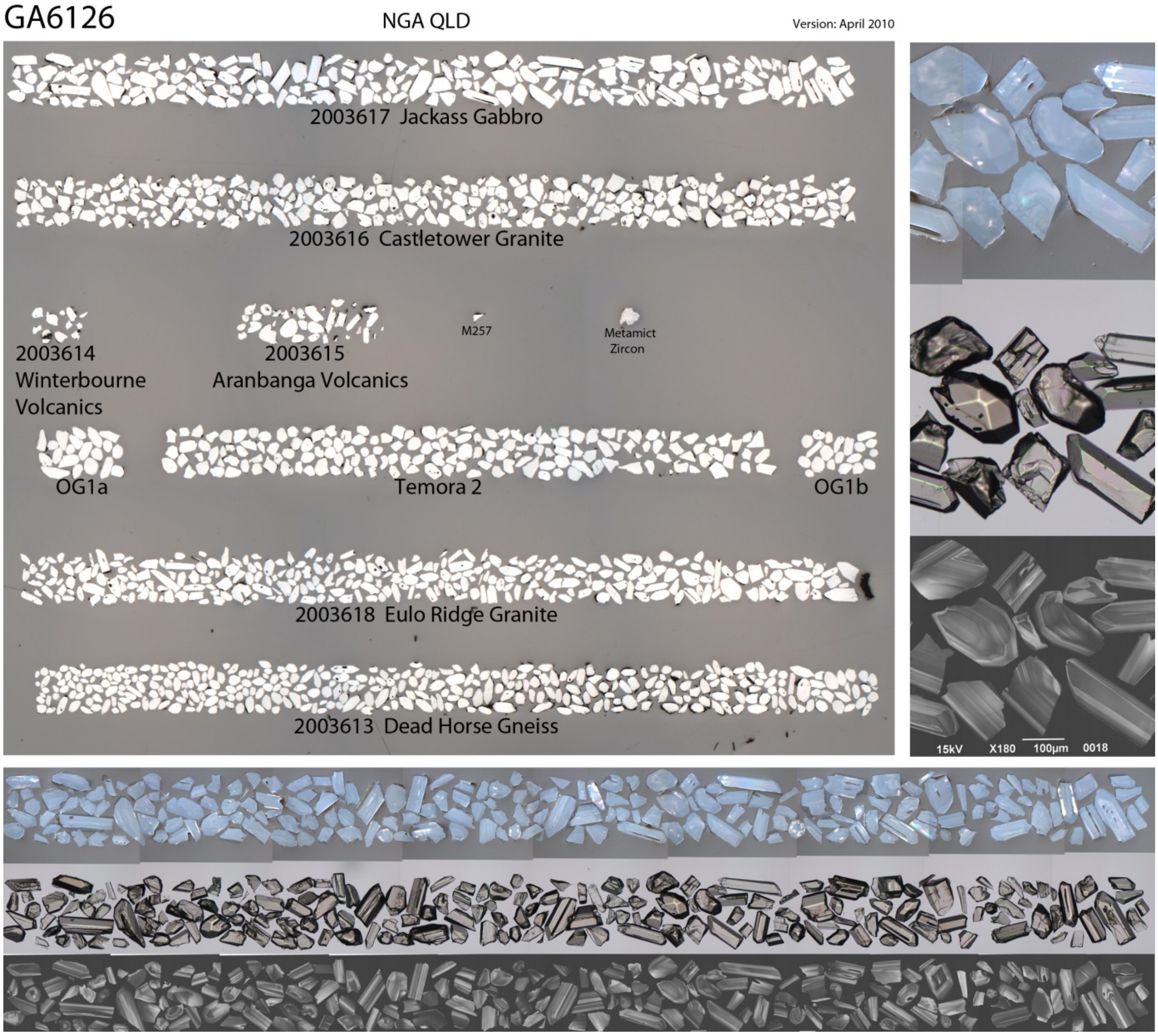


Figure 11.1 Top left: Mount Map imaged at 2.5× in Reflected Light. Bottom: Composite image of the full top row (Jackass Gabbro) in the map with Reflected Light DIC (top), Transmitted Light (middle) and Cathodoluminescence image (bottom). Top Right: cropped portion of full row illustrating the variation in the imaging techniques.

Reflected Light Differential Interference Contrast (DIC) microscopy is used to determine surface topography of the grains. Discontinuities such as slopes and valleys on the surface create differences in the optical pathways; when transformed by reflected light DIC into intensity variations the topographic profile of the grain can be seen. Reflected light DIC images provide information on suitable surface locations for a SHRIMP analytical spot.

In transmitted light microscopy, light is transmitted through the mounted grains at high illumination to reveal internal grain texture and features such as zoning, inclusion and fractures. Transmitted light images provide valuable internal grain information to the analyst to target or avoid specific areas of the grain such as zones, cores, rims, inclusions and/or fractures when selecting a location for analysis.

Cathodoluminescence (CL) and Back-scattered Electron (BSE) imaging provide the best internal compositional and textural determination of target minerals and are undertaken on a (SEM).

CL response results from the electron beam causing orbital transitions of electrons in certain elements—notably some rare-earth elements—that emit photons. The intensity of the CL response is also a function of the quality of the mineral lattice, therefore, as well as indicating compositional variation it also provides information about the quality of the mineral.

BSE is a response to the size of the atomic nuclei encountered by the electron beam. The more abundant large atomic nuclei are in a part of the sample the greater the BSE response. Thus the technique reveals contrasts in the average atomic number of phase regions in a mineral, providing information about bulk elemental distribution.

Different minerals require different methods. BSE is useful for analysis of grains such as monazite (Figure 11.2) where CL does not show the low level of elemental variation. However, the greater range of intensity in CL emission is more useful than BSE for determining growth regions in zircon grains.

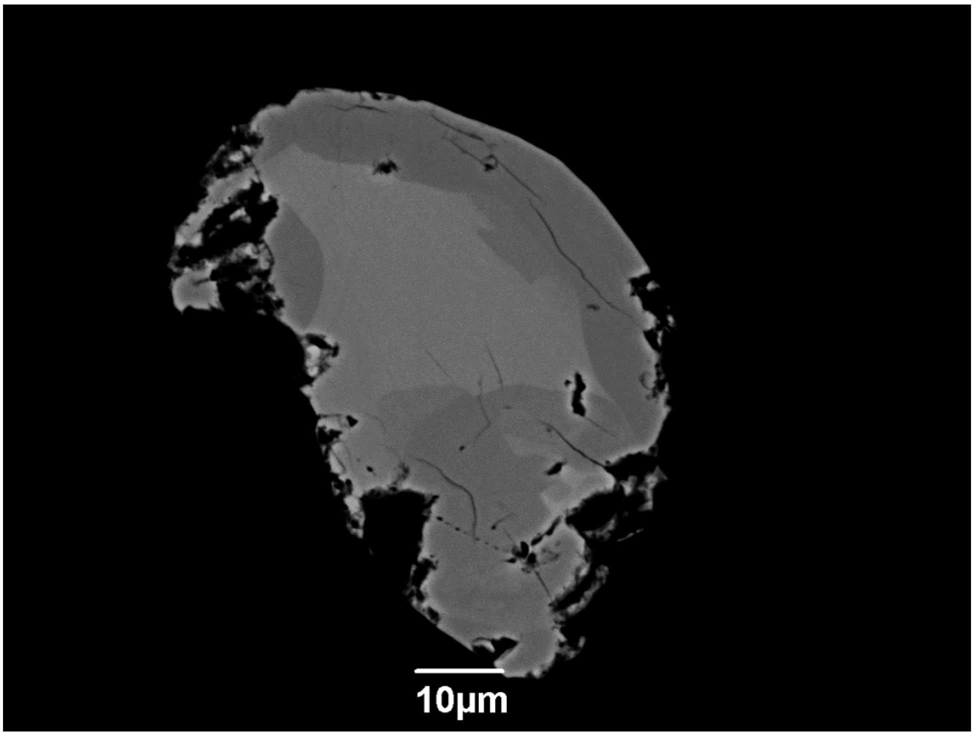


Figure 11.2 Back Scattered Electron (BSE) image of monazite showing elemental variation.

Although the above imaging techniques can stand alone for specific determinations such as surface topography or grain zonation, when used in conjunction all methods provide a complete description of the target grain. This information allows for selection of the most appropriate area for SHRIMP analysis; for example, cores and rims can be targeted and fractures and inclusions avoided with a high degree of positioning accuracy. The information becomes additionally valuable when follow up work is undertaken on the grains using Electron Microprobe, Laser Ablation or Thermal Ionisation Mass Spectrometry.

Once imaging is complete the mount is cleaned with various solvents—such as ethanol, detergent and petroleum ether—and ultra-pure Milli-Q water to remove any contaminants from the mount surface that may disrupt gold coating and affect analysis. After drying in a clean oven at low temperature, the mount is gold coated a second time to 15 nm depth. The mount is now ready for analysis on the SHRIMP. Images are copied to DVD and printed for the analyst and the mount is handed over to the SHRIMP Laboratory for analysis.

12 Sensitive High Resolution Ion Micro-Probe

The Sensitive High Resolution Ion Micro-Probe (SHRIMP; Figure 12.1) is a large mass spectrometer that can measure isotopic ratios in a geological specimen at a micrometre scale. It uses a finely focussed beam of ionised oxygen (O^{2-}) to ablate and ionise a region of the sample typically 20–30 µm in diameter (Figure 12.2).

Figure 12.1 The Geoscience Australia Sensitive High Resolution Ion Micro-Probe IIe (SHRIMP IIe).

The oxygen ions are produced in a duoplasmatron and accelerated by 10 kV to a speed of around 28 million km/h. Around 50 billion oxygen ions are produced per second and over the course of a 15–20 minute analysis will produce an ablation pit 1–4 µm deep. The amount of material removed from the pit is typically ~2 ng, but the amounts of an element of interest such as uranium or lead that is actually ionised and measured can be an order of magnitude smaller.

The ions from the sample, including uranium and lead, are accelerated by 10 kV through an electrostatic and magnetic sector, filtering the ions for energy and mass respectively. The magnetic sector consists of an electromagnet that can be finely tuned to direct atomic and molecular masses of interest into the detector which counts the number of incoming ions in a given period of time.

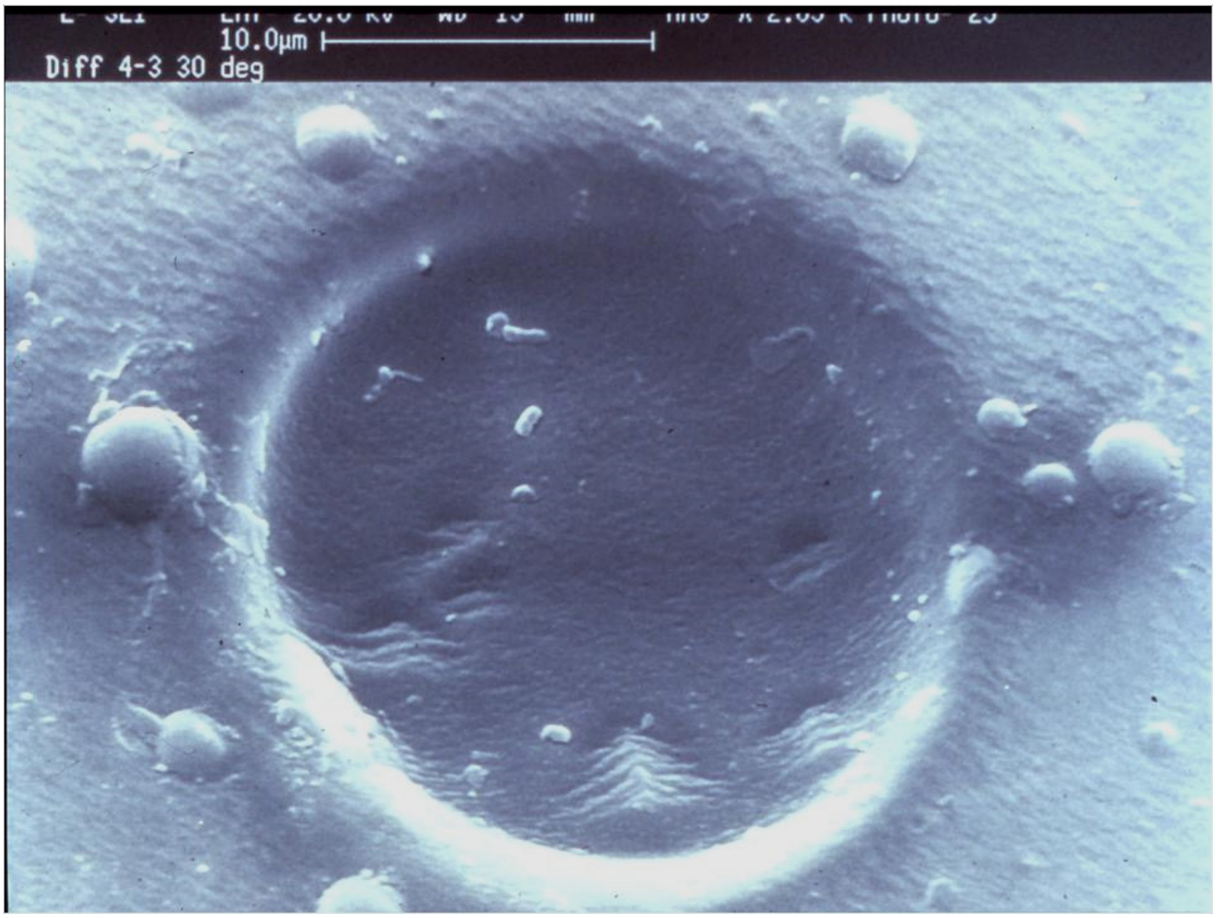


Figure 12.2 Example of a SHRIMP ablation pit.

The need to transmit ions as effectively as possible makes the instrument susceptible to several aspects of the sample mount and preparation.

- Vacuum. The instrument is maintained at vacuum pressures ~10^{-7} and 10^{-8} Torr in order to prevent loss of signal and also to provide a measure of electrical insulation between high voltage components. Higher pressures can degrade the signal, cause contamination and may even allow high voltage discharges that can destroy sensitive instrument components. Samples need to be clean and not outgassing. Ideally the sample mount should be prepared in sufficient time to allow for adequate outgassing in the sample lock, or other vacuum, for a day or more prior to loading into the instrument.
- Relief: Au-coating the sample avoids electrical charges building up around the areas being analysed. Relief or scratches in the mount surface can disrupt the continuity of an Au-coat and encourage high voltage discharges. Charging of the mount surface can disrupt the transmission of ions leading to poor performance and accuracy of results. Discharges can disrupt or even damage sensitive electronic components.
- Gradient: If the surface of the mount is not flat and perpendicular to the imaging on the SHRIMP then focussing problems may occur. This may result in less than optimal ion transmission across the mount during an analytical session which may be particularly apparent during automated running, which relies on focussing parameters being nearly constant.

12.1 Contamination

Being an in situ technique, the SHRIMP measures all elements and molecule ions from the surface of the sample—including any chemical contamination that may have occurred during preparation. Potential contaminants such as water and mercury can cause unresolvable isobaric interferences around the important Pb mass spectra at 204, 206, 207 and 208 amu (atomic mass units). Long-term contamination of the instrument can lead to degradation of vacuum systems particularly a reduced life-cycle on the cryopump.

13 Conclusion

The crucial first step towards obtaining high quality isotopic data is obtaining a high quality sample. Planning the sample collection involves permission for land access, followed by selection of an appropriate outcrop; taking into consideration the physical and cultural aspects—in situ nature, general site safety, aesthetic location—of the outcrop, and the rock properties—weathering, fractures, veining. Sampling is only undertaken when a suitable outcrop is located.

On return to the laboratory the sample details are added to databases, and samples are labelled and stored until required for processing. The unique identifiers obtained at this stage are used and cross-checked throughout preparation, analytical and reporting processes for quality control.

Processing commences with pre-treatment, removing any remaining field contamination, and finishes with handover of the mount and associated images for analysis. Samples are handled with care from the field, into pre-treatment and until the sample analytical process is completed—identifiers are cross checked, processing areas are kept clean and Work Health & Safety (WHS) procedures are followed.

The laboratory is a high risk environment with potential injury resulting from the majority of work. Examples of the cause of an injury and the potential damage that can result include (but are not limited to):

- Sledge hammers, drilling, splitters and crushers:
    - Fly-rock in eyes resulting in eye damage and potential blindness.
    - Incorrect equipment use resulting in crushed limbs.
    - Excessive noise and inadequate hearing protection resulting in permanent hearing loss.
- Crushing and Milling:
    - Inadequate use of masks and respirators resulting in inhalation of fine particulate matter such as asbestos, causing long-term and potentially fatal illness.
- Heavy liquids:
    - Spills or splashes of toxic heavy liquids or solvents resulting in serious illness and central nervous system damage.
- Wet areas:
    - Slipping on wet floors resulting in falls and broken limbs.
- Radioactive samples:
    - Inadequate prevention of exposure to high levels of radiation resulting in illness.
- General tool and equipment use (ranging from large equipment such as saws, to small equipment such as a glass funnel and tools such as screwdrivers and scalpels):
    - Incorrect handling/use of tools for assembling and disassembling equipment resulting in slipping and potential bodily damage.
    - Incorrect use of equipment resulting in loss of limbs, cuts or abrasions.
    - Inadequate protection resulting in injury, such as dropped rocks on feet.

For these reasons it is imperative that WHS procedures are strictly adhered to: only trained personnel operate laboratory equipment; appropriate PPE is provided for, and used by, all personnel; signs indicate hazardous areas or situations; Standard Operating Procedures are used as an essential component for training new staff; hazards are continually monitored; safe chemical handling and radiation safety procedures are followed and chemicals are tracked; among many others.

Cleaning processes throughout the laboratory are rigorous due to the risk of contamination between samples. The higher the level of sample concentration, the greater the risk of contamination; it only takes one grain from a previous sample separation to contaminate the next sample—a grain that, if analysed, must be reported and explained in the interest of scientific transparency.

The acquisition of high quality isotopic data commences in the field, and is continued in the laboratory. The detailed attention that contributes to the sample collection and processing procedures—that precede data acquisition, processing, report writing and data management—is instrumental in the provision of high quality isotopic data.

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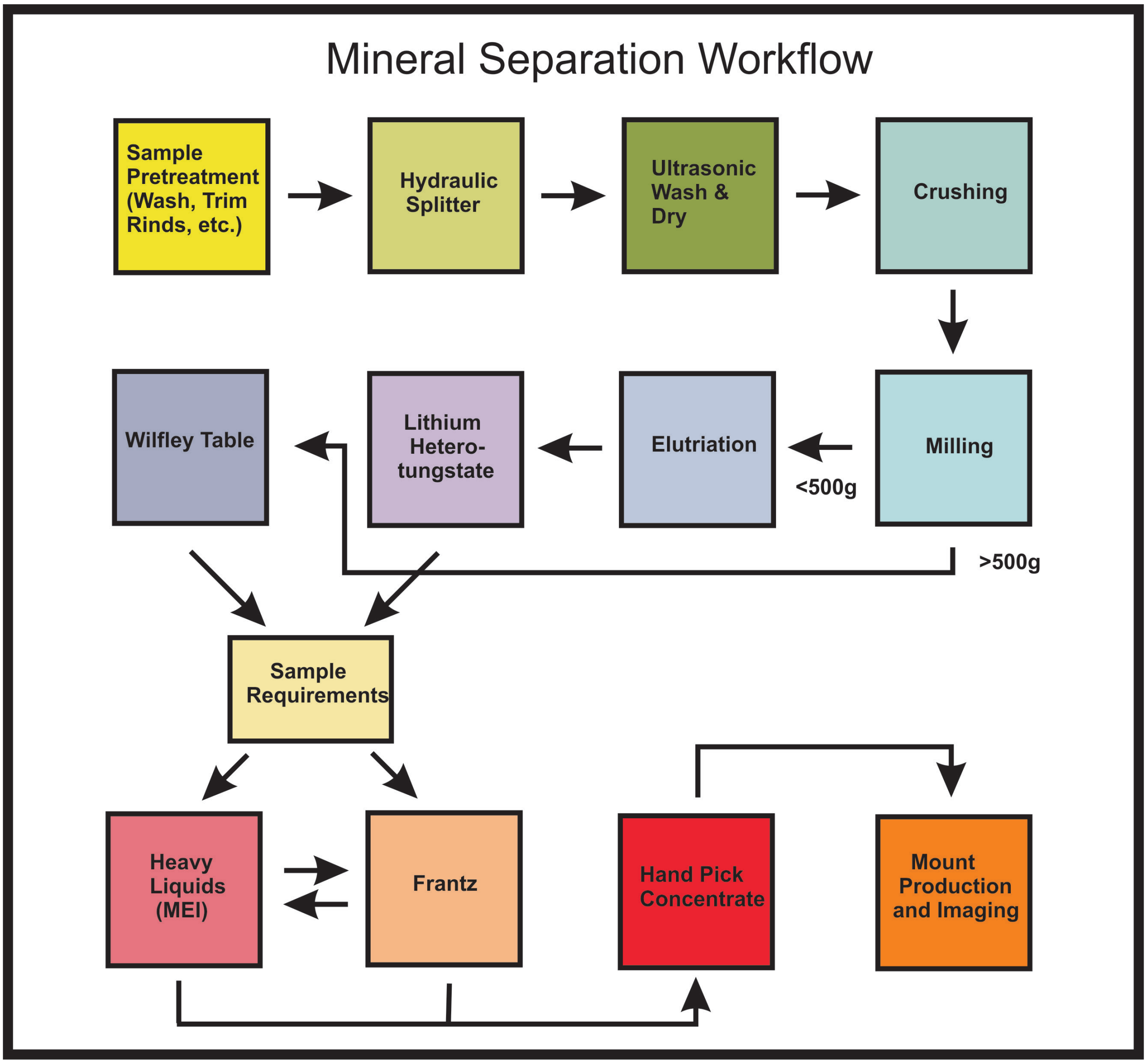
Appendix A Mineral Separation Workflow

The Mineral Separation Workflow in the laboratory commences with sample pre-treatment and finishes with mount production and imaging, outlined in sections 1–11. Appendix Figure A.1 provides a flow chart of mineral separation procedures for the majority of samples processed in the Geoscience Australia Mineral Separation Laboratory. Appendix B provides four examples of processing variations for zircon separates.



Appendix Figure A.1 Workflow of samples through the Geoscience Australia Mineral Separation Laboratory in preparation for analysis on the Sensitive High Resolution Ion Micro-Probe.

Appendix B Examples of Processing Variations

B.1 Granite, 30 kg, to extract zircon.

Considered a fairly ‘standard’ sample, Example B.1 can be processed without the aid of a thin section.

The sample would be processed in the following order (outlined in Appendix Figure A.1):

- Split
- Crushed and milled in Crushing Room 1
- Wilfley Table (run up to 5 times to reduce the concentration to the heaviest mineral components)
- Hand magnet
- Rare earth magnet
- Frantz (up to zircon concentrate stage)
- Heavy Liquid DIM (to remove apatite and remnant lighter mineral components from the Wilfley table)
- Frantz (to purify zircon concentrate to least magnetic fraction)
- Handpicking
- Mounting and imaging

B.2 Drill Core, 100 g, to extract zircon.

Drill core is considered precious as it generally cannot be readily replaced; considerable expense goes into obtaining drill core. Frequently core is cut and the lab receives half or a quarter of a core for processing.

This sample would be processed in the following order:

- Crushed: 100 g of drill core would not require splitting, but would be crushed using the flat crushing plates on the splitter as there is no room to lose sample in the jaw crushers
- Milled: using a small shatter box calibrated for target grain size liberation
- Deslimed in a beaker or the elutriation system
- Heavy liquid LST or NaPT is used to remove most remaining light minerals
- Assessment: the sample is then assessed to determine if heavier minerals make up the bulk of the sample and if is more efficient to remove them via Frantz or DIM at this stage
- Handpicking
- Mounting and imaging

B.3 Drill Core, 2.5 kg, to extract muscovite and zircon.

The sample size required for muscovite separation is generally less than 200 g, but can be as small as a thin section off-cut. In this example, the entire sample is processed to crushing stage as for zircon. After crushing, a representative portion of the sample (up to 100 g) is placed in a separate container. The larger portion is processed as above for Example B.1 to extract zircon.

The target muscovite component is then run through the jaw crusher on the finest possible setting. The fine crush is sieved through numerous fractions to under 250 µm; fractions between 100 and 250 µm are retained for heavy liquid separation.

Targeted specific gravity using modified TBE is used to purify and separate muscovite from other minerals. The density of muscovite is between 2.76 g/cm^3 and 2.88 g/cm^3. Therefore, a primary liquid separation with the TBE density modified (using acetone) from the standard 2.96 g/cm^3 to 2.90 g/cm^3 enables the removal of any minerals with greater density than the target mineral. The liquid is then remodified to 2.70 g/cm^3 to float of any lighter minerals; the ‘heavy’ fraction will contain the target mineral (TBE will return to the standard 2.96 g/cm^3 density when the acetone evaporates).

The muscovite is handpicked to obtain the cleanest mica separate possible.

B.4 Drill cutting from petroleum wells

Samples from petroleum wells may contain cutting fluids and drilling muds that were added as part of the drilling process. Drilling additives must be removed as a first stage of processing otherwise grains will clump together and standard mineral separation processes will be ineffective.

Removal of drilling additives is achieved by immersing the sample in a bath of acetone; the sample is washed several times in the bath until the acetone runs clear. The sample is then washed in a detergent bath and wet sieved to 425 µm. Larger particles are then dried, milled conventionally and recombined with the <425 µm component. Processing then continues as per the workflow outlined in Appendix A, from the Wilfley Table stage to final concentrate, outlined in the example at B.1.

Barite is often used in drilling mud and will be retained in the final concentrate. Barite is both dense and non-magnetic and survives heavy liquid and Frantz procedures; however, it is a soft mineral and susceptible to breakage.

When barite has been used in drilling mud an additional step is employed. Before either handpicking or further runs on the Frantz to purify the zircon, the barite is removed through an abrasion process. The sample is placed in a small beaker of water with a 3 mm layer of steel ball-bearings. The beaker is then placed on an orbital shaker until all traces of Barite have been pulverised and are washed away, leaving only zircon and other remaining heavy minerals. The sample is ready to have the zircon purified (if necessary) or to be handpicked for mounting.