SAMPLING FOR FISSION-TRACK ANALYSIS

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Field collection

Fission-track analysis can be preformed on apatite and zircon crystals from certain bedrock lithologies and clastic sedimentary environments that contain sand-sized sediments, including modern environments such as fluvial and beach facies. Sampling techniques are different for different kinds of lithologies and both rocks and loose sediments, as for fission-track analysys apatite and zircons in the 200 – 80 µm range are needed. Sampling strategies depend on the intended study and in the following we will distinguish between bedrock studies (granite, gneiss, micaschists, basalts, rhyolites, andesites etc.) and detrital studies (ancient sandstone and modern sand-sized clastic sediments). For exhumation and provenance studies one should carefully consider where in the field samples are collected. For example, bedrock exhumation studies may require sampling of “vertical” profiles from bedrock outcrops in the target area, while detrital provenance and exhumation studies require samples collected from river deltas of large-scale drainages, from marine turbidite sequences in the outcrop, or from drill-cores, if available. In any case, the most common mistake is that too little sample material is collected (see suggestions below) and too few apatite or zircons are separated for proper analysis.

Apatite and zircon in different source rocks. A crucial first step is to understand the nature of the source rock and whether that rock will yield apatite and zircon in an appreciable quantity. Geologic maps usually provide reasonable information about the potential apatite and zircon yield that can be expected in any given study area, but obviously the source for ancient sandstones can be more difficult to infer. Zircon and apatite are common accessory minerals in many acidic and sodium rich igneous rocks such as granite, granodiorite, tonalite or rhyolite and their metamorphic equivalents (see Table 1; Poldervaat 1955, 1956; Deer et al. 1992). As such, apatite and zircon occur in siliciclastic deposits derived from such source rocks. In many river drainages the variety of gravels in the riverbed will provide a quick overview of lithology in the source area, but they are likely to be biased towards more resistant lithologies.

Table 1. Relative apatite and zircon concentration by source lithology

Source lithology	High concentration	Intermediate concentration	Low to very concentration	No concentration
Igneous rocks	granite, granodiorite, tonalite	rhyolite, ignimbrite	gabbro, basalt	ultramafic rocks
Metamorphic rocks	orthogneiss	paragneiss, meta-rhyolite, meta-sandstone, phyllites	eclogite	marble
Sedimentary rocks	arkose	conglomerates, quartz arenite, litharenite, siltstone	mudstone claystone	dolomite carbonate rocks

Note: Relative zircon concentrations are based on Poldervaart (1955, 1956) and Deer et al. (1992). Basalt may contain some apatite but no zircon. This is given for apatite and zircon in the 200 – 80 μm range.

Owing to its stability (hardness of 7.5; lacks distinct cleavage) zircon survives significant weathering and transport while other detrital components are selectively removed. This trend is reflected in the zircon-tourmaline-rutile (ZTR) index for heavy minerals in clastic sedimentary rocks. This index is used to semi-quantitatively evaluate sediment maturity and source rock weathering, and increases when these three very stable minerals are relatively enriched in the heavy mineral fraction of clastic sediment by either transport or dissolution (e.g. Morton 1984; Mange and Maurer 1992). For example quartz arenite and quartzite have a particularly high ZTR index and commonly at least an intermediate zircon yield. Lithologies with unusually low zircon yield include carbonate rocks, mafic rocks, and ultramafic rocks (Table 1).

Apatite (hardness of 5) is much less stable in the sedimentary environment than zircon, but can still be found in many clastic sediments and sedimentary rocks. While apatite is stable during burial diagenesis, it is affected by weathering during sediment transport and early diagenesis as well as outcrop weathering. In general, it is better to sample fresh bedrock for apatite fission-track analysis and to avoid strongly weathered bedrock. Similar to zircon, apatite is more abundant in acidic and sodium rich igneous rocks such as granite, granodiorite, tonalite or rhyolite, tuffs in ignimbrites and their metamorphic equivalents, but also occurs in low quantities in gabbros and basalts.

Sampling for bedrock apatite and zircon fission-track analysis

Bedrock samples are either collected from crystalline rocks (granite, granodiorite, gneiss, mica schist etc.) along age-elevation profiles or individually isolated locations, depending on the study objective.

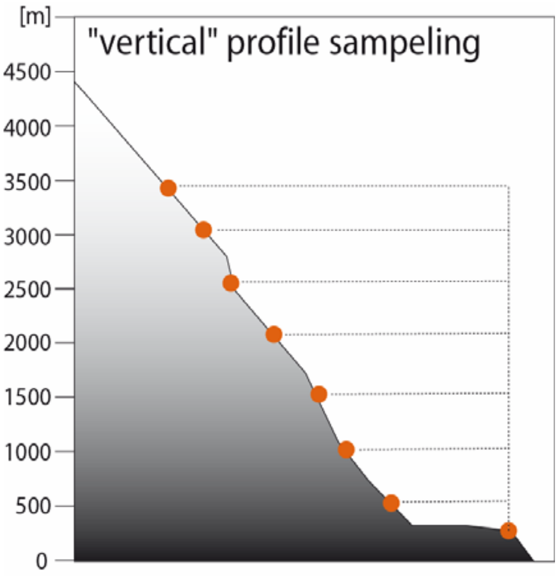

Fig. 1 Schematic sampling plan for a vertical profile. Sample spacing will depend on outcrop access and lithology.

The number of samples necessary for sampling a vertical profile depends on the local relief (Fig. 1). Usually ~300-500 m of altitude difference between samples provides sufficient information for obtaining a good age elevation profile. However, in many cases outcrop accessibility and lithology control the sample spacing.

Good lithologies for age-elevation profiles include granite, gneiss, rhyolites and metasandstones. Poor lithologies include gabbros, serpentintes, shale, slate, metapelites, phyllites and fine grained schists. Intermediate volcanic rocks, such as dacites and andesites are possible to sample, but can be difficult (Table 2).

Table 2. Bedrock sample size for vertical profiles and individual locations

Lithology	sample weight in the field
Granite	4-5 kg
Rhyolite	4-5 kg
Gneiss	4-5 kg
Metasandstone	5-7 kg
Andesite	8-10 kg
Dacite	8-10 kg
Amphibolites	10-15 kg
Gabbro	10-15 kg
Basalt	10-15 kg

avoid these lithologies

Carbonate rocks
Mudstone, shale, slate
Marble
Metapelites, phyllites, schists
Eclogites, sepentinites

Sampling for age dating of hydrothermal systems or the thermal influence of volcanic intrusions depends on the size of the veins/intrusions as well as visible contact metamorphism.

In general it is good to sample the host rock in direct contact to the hydrothermal vein or intrusion and in increasing vertical or lateral distance to the vein/intrusion such as for example: 0 cm, 10 cm, 50 cm, and 100 cm distance (Fig. 2). For km-scale intrusions the sampling distance is on the km-scale as well.

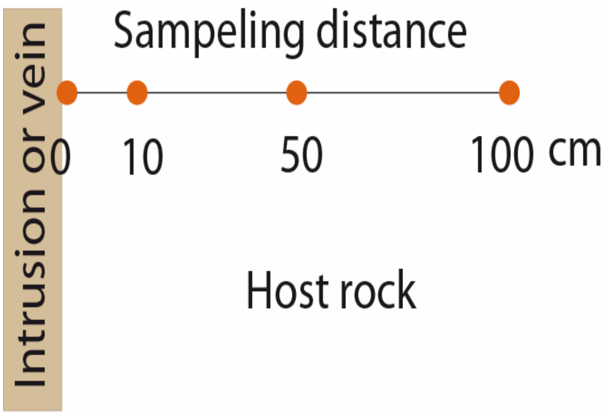

Fig. 2 Schematic sampling plan for dating hydrothermal intrusions of the thermal effect of intrusions on host rock.

Sampling for detrital apatite and zircon fission-track analysis

Recent sediment. Collecting detrital apatite and zircon from Recent sediment and loosely consolidated sedimentary rock is relatively simple. Apatite and zircon, commonly of fine sand size in detrital samples, have densities of 3.1-3.3 g/cm^3 for apatite and ~4.55-4.65 g/cm^3 for zircon (Deer et al. 1992), so their settling velocities are similar to quartz grains of medium sand size (Fig. 3). For this reason apatite and zircon are typically deposited with somewhat coarser grained material, and samples should be preferably collected from sand bars and beaches with coarse- to medium-sand grain sizes. Simple gravity separation in the field (i.e. gold panning), can easily concentrate apatite and zircon, so that a final density separation in the lab involves only a small quantity of material (200-300 g instead of 2-4 kg or more). Therefore, loose sediment can be directly processed in gold pans, and panning removes the lighter material (quartz, feldspar, micas etc.) and enriches the heavy minerals such as apatite, zircon, garnet, magnetite and even gold. In general, it is sufficient to pan between 12-14 pans of material, but the final outcome depends on zircon and apatite yield, panning efficiency, etc.

It is also possible to collect samples from gravel bars. In this case, gravel and all finer grained material can be run through a coarse sieve. The finer fraction (coarse to fine sand and smaller sizes) should be retained and processed further in the gold pan, while all coarse material (> 2 mm) can be discarded. It is worthwhile to look for heavy mineral placer deposits, which can be easily recognized by black and reddish colors from magnetite and garnet. If placer deposits are available it is not even necessary to use gold pans, because the top layers of the placer deposits can be scraped from the surface. If only loose sediment is collected for processing in the lab, without panning in the field, one should collect at least 4-7 kg of sample material. However, even this size of sample may not have sufficient apatite or zircon if the source lithologies are not apatite or zircon bearing.

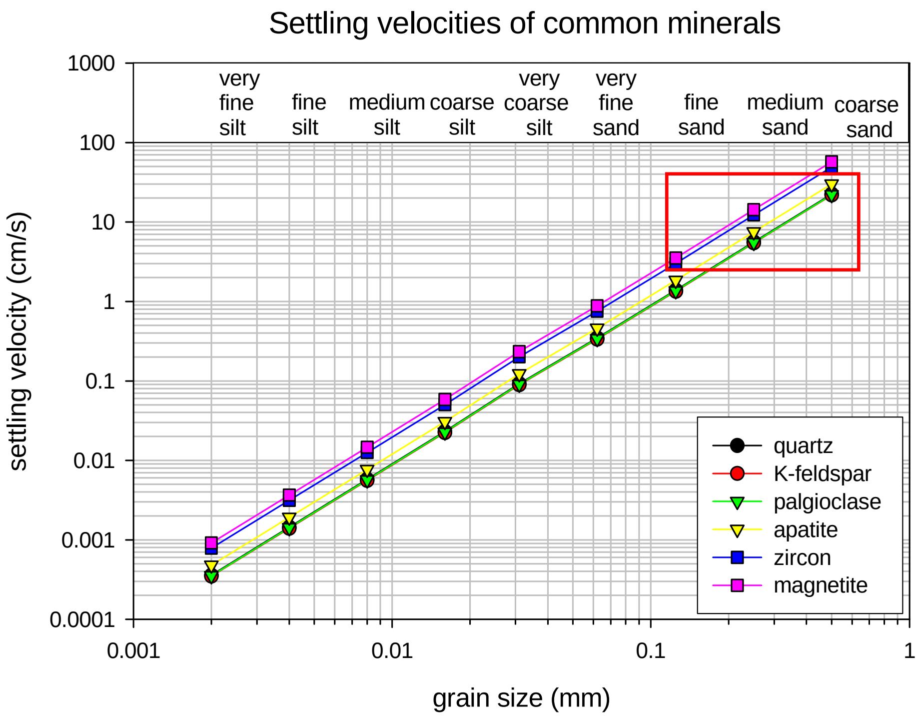

Fig. 3 Settling velocities of apatite, zircon, quartz and Feld-spar minerals depending on grain size.

Fig. 4 Panned samples and placer deposits

Warning: If it is also the objective to do a quantitative analysis of the complete heavy mineral suite, it is necessary to collect a bulk sediment sample that was not panned in the field!

For modern river samples it is a good idea to note the different lithologies present in the pebble fraction at the sampling site of the sampled river.

Ancient sandstone. Collecting samples from sandstone outcrops for detrital apatite and zircon fission-track analysis is routine, but there are some important considerations to bear in mind. All sample sizes suggested here are based on our experience in a number of different geodynamic settings of different composition and age. We find that the best samples are medium-grained arkosic sandstones and 2 kg of sample is generally sufficient, but many sandstone compositions are appropriate for collection and apatite and/or zircon extraction. Samples of lithic sandstone samples should be 4-7 kg. The presence of visible quartz is generally a good indicator, because quartz-rich lithologies require smaller samples for zircon (Table 3), but they are less good for apatite. The target grain size should be medium- to coarse-grained sandstone: fine-grained sandstone should be avoided, but collected only as a last resort as >80 µm zircons or apatite are very difficult to impossible to analyze). For graded beds (i.e. sandy turbidites), this observation requires that in some cases only the base of a bed is sampled.

Table 3. Sandstone sample size for detrital fission-track analyses

Lithology	sample weight in the field
Arkose	2-4 kg
Quartzo-feldspathic sandstone	~4 kg
Quartz-bearing volcaniclastic sandstone	5-7 kg
Lithic sandstone	5-7 kg
Silicic volcaniclastic sandstone	~4 kg

The yield of zircon from most sandstone is usually satisfactory because many common lithologies produce appreciable yield and post-depositional modification is not significant. However, detrital apatite is much less predictable because it is more variable in source rock, and it can be severely affected by weathering and dissolution. If the aim is to analyze detrital apatite as well, then it is important to avoid weathered sandstones, especially those with excessive iron oxide and evidence of interstratal dissolution. These strata may have very poor yields of apatite, and there may be significant secondary minerals such as pyrite, siderite, or barite.

Sampling for fission-track analysis of volcanic rocks and glasses

Volcanic rocks are in principal suitable for fission-track analysis. Rhyolithe normally contains apatite and zircon (and titanite), and 4-5 kg of sample material should be sufficient. In intermediate volcanic rocks such as dacites and andesites the apatite and zircon content is somewhat lower and 8-10 kg should be sampled. Basalt has in general low apatite content and contains no zircon. 10-15 kg should be sampled.

In addition to dating apatite and zircon (or titanite), volcanic glass like obsidian can be used for fission-track analysis.

Tephra layers: Tephra is unconsolidated pyroclastic debris, usually derived form explosive, acidic volcanism. Depending on the thickness of the tephra layers, it is to avoid the base and the top during sampling (Fig. 5).

Four to five kg of material should be collected from the middle the layer to avoid contamination by detrital grains. Attention needs to be paid to possibility that the tephra has bee re-deposited. Re-deposited tephra are to be avoided and not sampled. **Volcanic glass** can be recovered from tephra layers usually just by sieving the samples, recovering the >100 µm fraction.

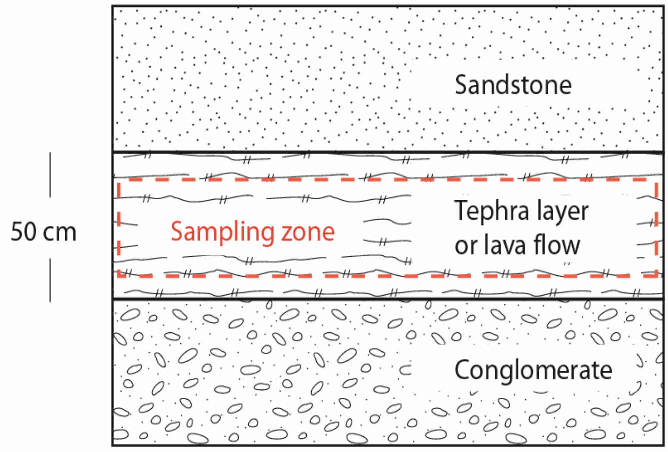

Fig. 5 Schematic diagram for sampling of volcanic rocks. Example is given for a 50 cm thick tephra layer or lava flow. The top and base of the unit should be avoided

Lava flows: Similar to tephra layers, **rhyolitic lava flows** should be sampled in the middle to avoid detrital contamination if the flow overlies sedimentary rock. For **basaltic lava flows** the rapidly cooled crust should be avoided, and samples should be collected from the lower third of the flow, if possible. If the lava flow is in contact with underlying sedimentary rocks, then the base of the flow has to be avoided.

Ignimbrites: In the case of ignimbrites, the sampling should be done in parts without large rock fragments to minimize the contamination from country rock that was included in the ignimbrite flow.

Sample preparation starts in the field.

It is necessary to break down the sample in the field to a hand specimen size that can easily be processed with the jaw crusher in the lab (e.g. 5 cm x 5 cm x 10 cm). Clean the sample in the field, which means removing all soil, vegetation and weathering crusts.

Label the samples (or at least the sample bags) correctly and clearly in the field. Keep extra sample material if other analyses are planned (for thin sections or geochemical analyses for example).

WARNING: The guidelines provided here do not guarantee a successful sampling campaign. Only after all mineral separation steps it is known how many apatites or zircons were in the sample. It is always to be expected that 10-15% of the samples collected in the field will not work.