

Laboratory Workflows for Integrated Provenance Analyses

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ABSTRACT: Integrated provenance studies are essential for reconstructing the source-to-sink histories, providing key indicators to model regional geodynamics and terrane accretion events. While sedimentary provenance offers critical insights for tectonostratigraphic and paleogeographic reconstructions, the reliability of such interpretations depends fundamentally on an intensive multi-analytical laboratory procedures. This paper details the standardized laboratory workflows implemented at the Department of Geosciences (CURE – UdelaR), emphasizing that optimizing the systematic processing of sedimentary rocks is essential for ensuring high-quality data. The methodology integrates petrography, whole-rock geochemical preparation (comminution), and heavy mineral separation using gravimetric and magnetic techniques. Furthermore, the application of SEM-EDS for chemical characterization of detrital heavy minerals is described. By optimizing decision-making at each stage of sample processing, the laboratory workflows minimize experimental bias and ensure data integrity. Mastering the laboratory work is a fundamental prerequisite for enhancing data and sample quality in subsequent high-precision analyses, ultimately ensuring cost-effective and scientifically rigorous geological models.

KEYWORDS: Laboratory Workflows, Sample Preparation, Sedimentary Provenance, Heavy Mineral Separation, SEM-EDS.

I. INTRODUCTION

The objective of sedimentary provenance is to interpret the history of sedimentary input, from source rock erosion to the final deposition of debris, leading to the reconstruction of the paleogeography by identifying the litho-geochemical characteristics of the source rocks (Nesbitt and Young, 1982; Taylor and McLennan, 1985; Bahlburg, 1998; Blanco et al., 2009, 2011, 2014, 2023; Abre et al., 2011, 2012, 2020, 2026).

Since source rocks and sedimentary basins do not remain stable over geological periods, the analysis of changes in supply areas, drainage networks, climate, tectonic environment and paleorelief can provide critical insights to understand the evolution of the upper continental crust (Cawood et al., 2013). Pioneering works such as those of Nance and Taylor (1976), Condie (1981, 1986) and Taylor and McLennan (1985) have demonstrated that the composition of the upper continental crust can be quantified analyzing the geochemistry of clastic sedimentary rocks, even distinguishing between Archean and post-Archean crust (Taylor and McLennan, 2003; Condie, 2000). It is now widely established that integrated provenance studies, based on the simultaneous application of petrography, whole-rock geochemistry, heavy minerals, and detrital zircon dating (e.g., McLennan et al., 2003; 2006; Abre et al., 2017 a, b, c, 2020, 2026; Blanco et al., 2023) enable detailed inferences regarding the evolution of the continental crust within specific regions and timeframes (Cingolani et al., 2017). In Uruguay, although integrated provenance studies remain scarce, they have provided critical data for understanding the tectonostratigraphic evolution of different crustal sectors (Blanco et al., 2009, 2016, 2021, 2022; Uriz et al., 2016; Abre et al., 2020, 2026).

While sedimentary provenance studies provide a wealth of information to model regional geodynamics, the data are the result of labor-intensive and meticulous laboratory procedures. The reliability of the final geological models depends directly on the quality of work performed during the preliminary stages. Consequently, understanding, optimizing and mastering the laboratory workflows is of paramount importance. This study outlines the methodological protocols employed at the laboratories of the Department of Geosciences (CURE – UdelaR) for integrated sedimentary

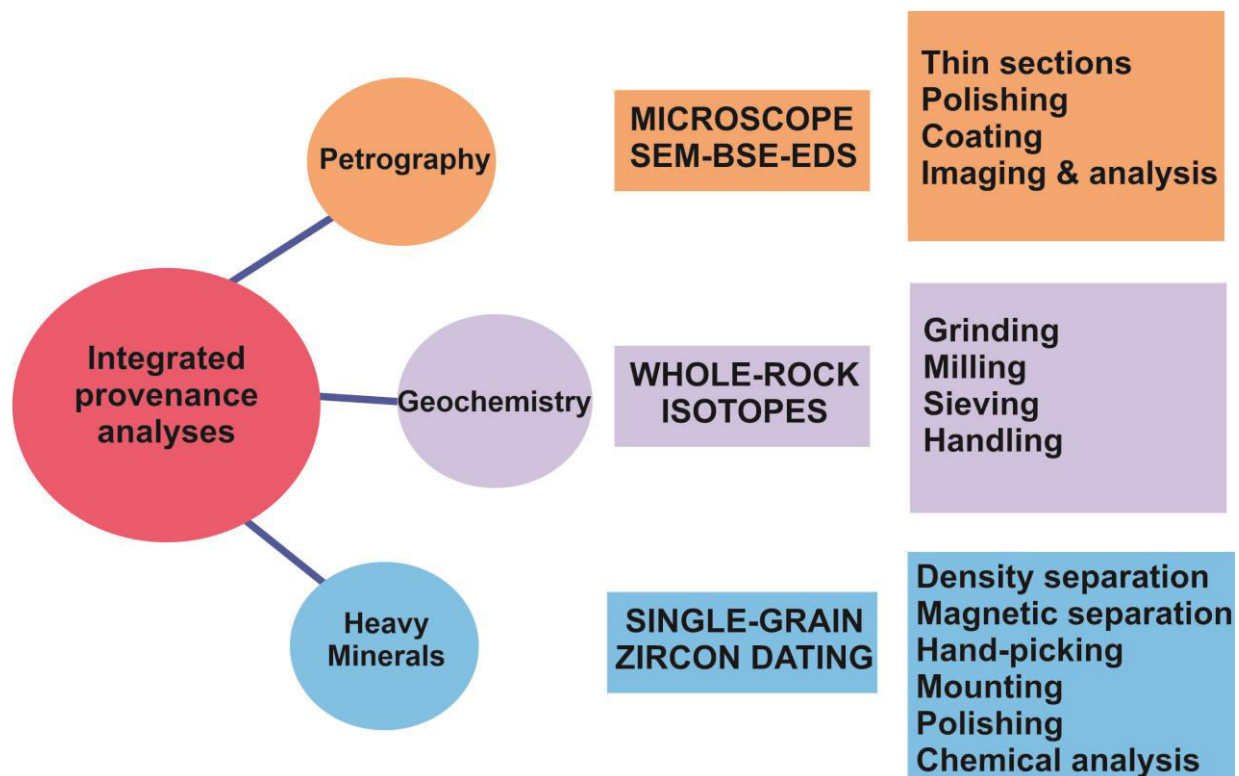
provenance studies. A standardized workflow is essential to ensure the acquisition of accurate data, while simultaneously optimizing time and resources during sample processing and analytical stages. The laboratory workflows implemented at our facilities ensure that the transition from a raw sample to data interpretation is both cost-effective and time-efficient without compromising scientific accuracy and safety.

II. METHODOLOGY

Establishing a research strategy facilitates the scheduling of activities required to achieve stated objectives, helps identify knowledge gaps, and prevents work duplication, thus optimizing time and resources. However, laboratory workflows must remain flexible; the unique characteristics of each sample require an adaptive decision-making process at every laboratory stage, from initial treatment to final analysis. The scientific research methodology to be employed in an integrated provenance study implies the use of the following techniques (Figure 1):

1) Petrography: clastic rocks, particularly sandstones, provide valuable information through the study of their framework grain composition. The laboratory process begins with the preparation of thin sections, followed by identification and quantification of detrital components using a petrographic microscope. These compositional data are then plotted on ternary diagrams to infer depositional environments (e.g. Garzanti, 2019). Further information can be obtained using a scanning electron microscope (SEM) equipped with an Energy Dispersive Spectroscopy (EDS) to determine the precise chemical composition of mineral grains, matrix, and cement.

Figure 1 shows the main techniques used for an integrated provenance analysis. The right column outlines the principal laboratory procedures needed and that can be carried out at the Laboratories of Geology of the Department of Geosciences.



2) Whole-rock geochemistry: refers to the integrated approach that encompasses the analysis of major, trace and rare earth elements (REE) as well as isotopic determinations (e.g. the Sm-Nd system). Although the analytical techniques of

both are different, they share a unified laboratory workflow centred on the production of a high-quality and representative powder. The bulk chemical concentration is a technique that assumes that certain trace elements such as Cr, Th, Zr, Hf, Sc, and REE are transported from the source rock to the sedimentary basin with negligible modifications due to sorting, fractionation, diagenesis, and post-depositional tectono-thermal effects. In particular, the ratios of incompatible (Th, Zr) to compatible elements (Sc) are highly effective in distinguishing the influence of felsic versus mafic sources (McLennan et al., 1993). Additionally, certain major and trace elements (e.g. Si, Al, K, Ca, Th, U) enable the assessment of chemical weathering and paleoclimatic conditions (Nesbitt and Young, 1982; McLennan et al., 1993). The application of the Sm/Nd system to sedimentary rocks is based on the fact that the parent isotope ¹⁴⁷Sm and the daughter ¹⁴³Nd are both REE that do not fractionate during sedimentary processes; thus, they reflect the average composition of the source rock, allowing to estimate model ages and to characterize the source as either mantle or crust derived (DePaolo and Wasserburg, 1976; Sato et al., 1995).

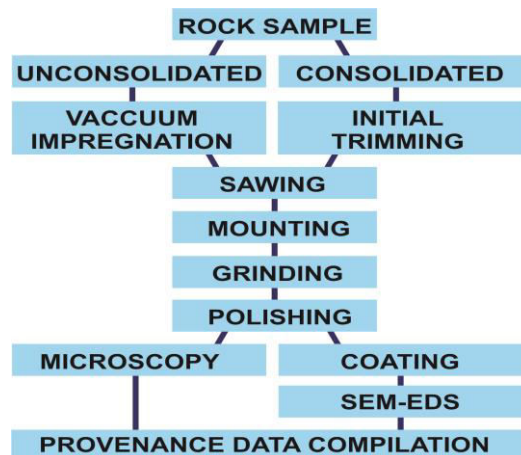
3) Heavy minerals: chemical analysis of specific single-grain detrital heavy minerals provides important clues regarding source-to-sink sedimentary routing systems. In this regard, spinels (e.g. Abre et al., 2009), garnets (e.g. Blanco et al., 2014), tourmaline, amphiboles, are among the most significant. The laboratory workflow to obtain the heavy minerals is the most time-consuming stage, requiring controlled crushing, sieving, gravimetric and magnetic separation, description, hand picking, and mounting. Furthermore, in-situ U/Pb detrital zircons dating using a laser ablation inductively coupled plasma mass spectrometer (LA-ICP-MS) is one of the most effective and widely used techniques in sedimentary provenance analysis. This method allows the statistical evaluation of the ages of source areas (e.g. Blanco et al., 2011, Abre et al., 2011, 2012, 2026). The success of this technique relies on the careful mounting of zircon grains during the final stages of the laboratory workflow to ensure high-quality analytical spots.

IV. LABORATORY WORKFLOWS

Once field samples enter the laboratory, they undergo specific preparation following several workflows depending on the subsequent description and analysis to be performed.

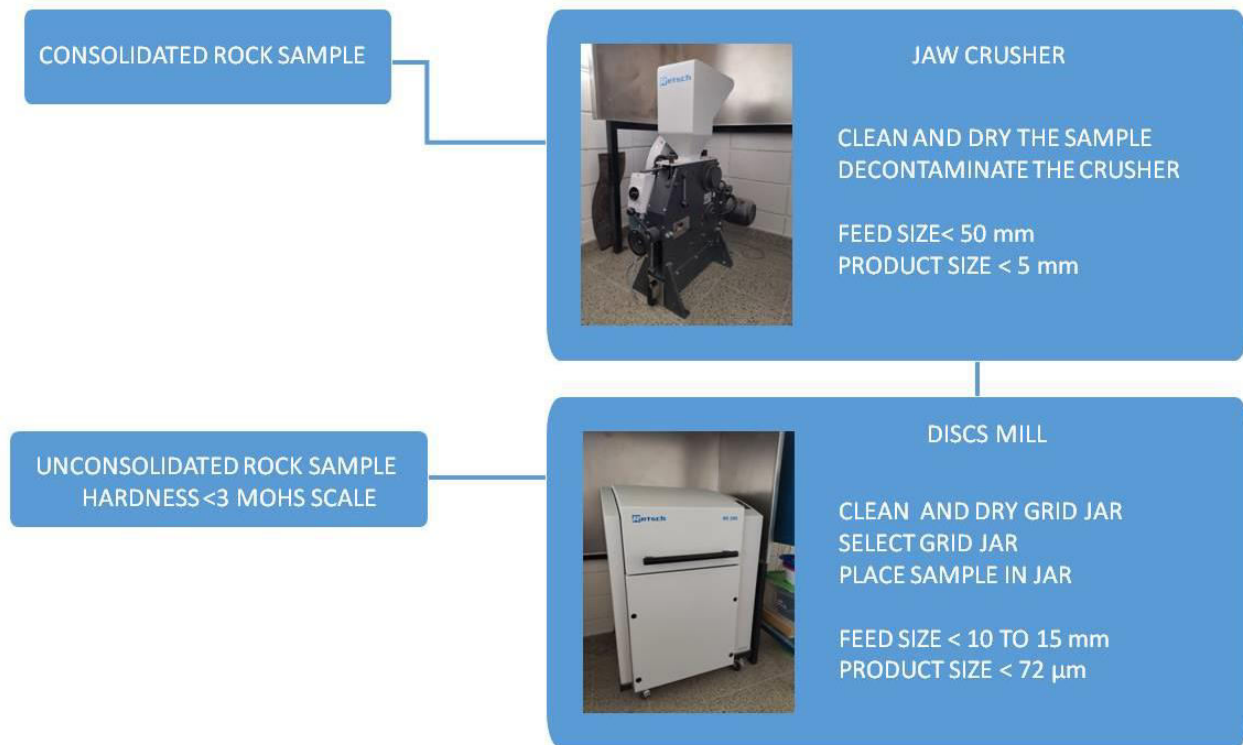
Petrographic analysis: consolidated rock samples are cut with a saw blade and pasted onto glass-slides using a standard epoxy resin. A thin sliver of each sample is cut using a diamond saw and then grounded using progressively finer abrasive grits until a standard thickness of 30 microns is achieved. Unconsolidated or friable samples require vacuum impregnation with epoxy resin before further processing. Instead of using a glass cover slip, thin sections are polished with diamond pastes of progressively finer grading until a smooth and reflective surface is obtained, making them suitable for both, petrographic and electronic microscopy analyses. To ensure electrical conductivity and prevent charging during SEM analysis, a carbon or gold coating is sputtered onto the surface. The JeolNeoscope JCM-6000 Plus allows for secondary electron (SEM) and backscattered (BSE) imaging, coupled to an Energy dispersive X-ray spectrometry (EDS) for chemical identification (patterns) and quantification (chemical analyses).

Figure 2 shows the laboratory workflow for petrographic analysis, illustrating the stages from initial evaluation of sample type to final data compilation via advanced analysis.



Geochemical sample preparation: Although we do not currently perform in-house whole-rock or isotopic analysis, we conduct the comminution required to prepare samples for overseas analysis. As stated in Figure 3, the workflow varies initially depending on the consolidation degree and hardness of the rock sample. Consolidated specimens are processed using a tungsten carbide jaw crusher to reduce grain size to less than 10 mm. Unconsolidated rocks or samples with a Mohs hardness of less than 3, as well as those crushed in the previous step, are then milled. Depending on the grinding jar used (tungsten carbide or mild steel), the maximum feed particle size of the vibratory disc mill ranges from 10 to 15 mm. Operating at 1200 rpm, it takes about 2 to 3 minutes for an average sedimentary rock sample to achieve a grain size of less than 72 microns, meeting the requirements of most commercial and academic laboratories for whole-rock and isotopic geochemical analyses.

Figure 3 shows the comminution workflow for geochemical sample preparation. The procedure outlines the systematic reduction of particle size, starting with primary crushing and ending with pulverization via milling.



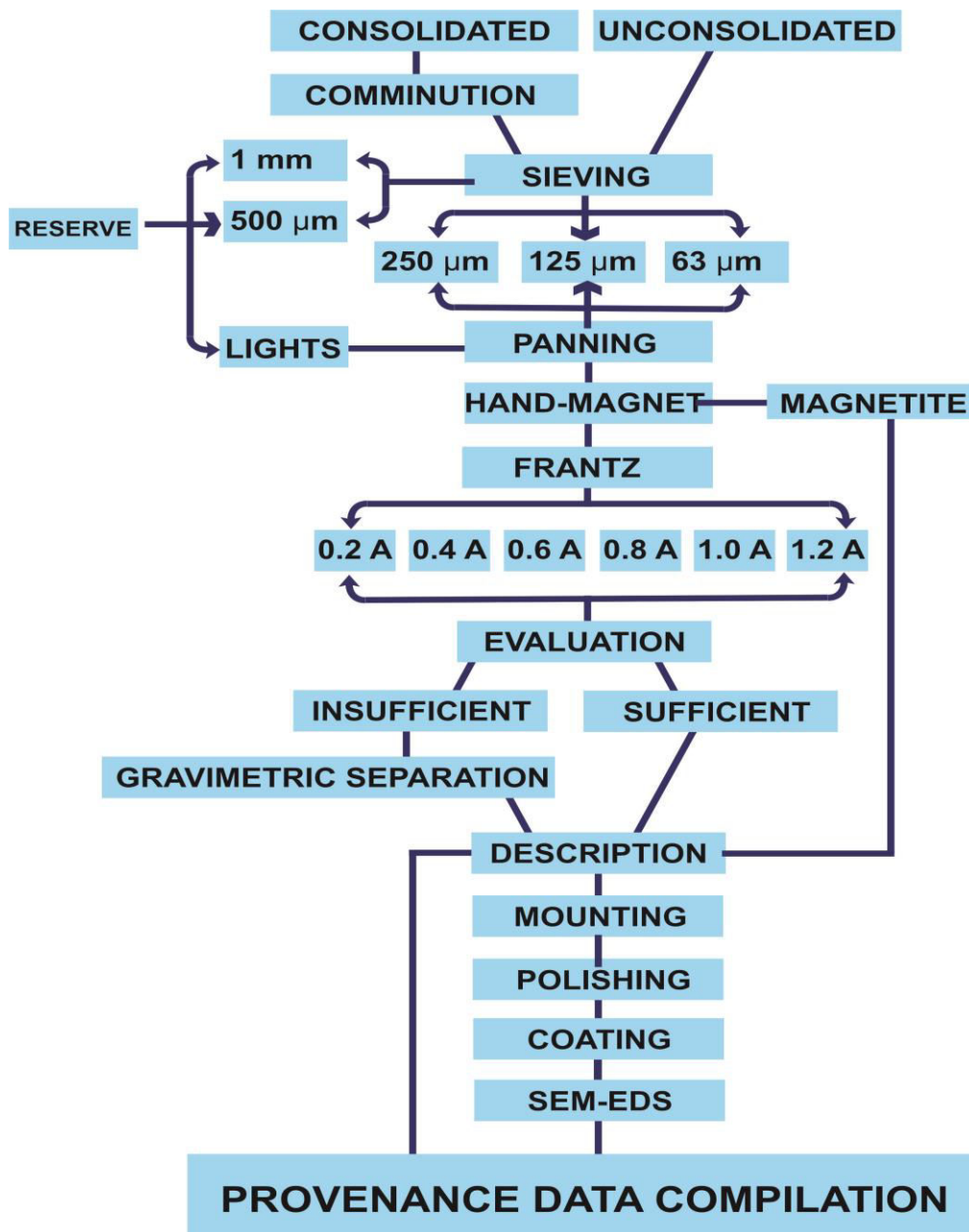
Heavy minerals separation: The separation of heavy minerals for provenance analyses may require initial disaggregation step for consolidated rocks. This is achieved following the comminution workflow, though milling is restricted to a few seconds in order to disaggregate the sample without pulverizing individual grains. Disaggregated samples are dry-sieved using a mesh set of 1 mm, 500 μm, 250 μm, 125 μm and 63 μm; the three finest fractions are typically reserved for subsequent separation. Sieves are cleaned in an ultrasound bath using a proper cleaning solution. The vibratory sieving machine is set at an amplitude of 2 for 7-minute cycles per 100 cm³ of sample.

The next stage involves laboratory panning using a set of small glass or gold-pans; the objective is to pre-concentrate the heavy minerals. Both, the heavy and the light minerals concentrates are dried in an oven at 60°C. The light minerals are archived, whereas the heavy concentrate (250 μm, 125 μm and 63 μm) undergo further processing.

Magnetite of each panned concentrates is extracted using a handheld magnet and stored separately. The rest of each pre-concentrate is then processed through the electromagnetic Frantz Isodynamic Separator (Model LB-1) to obtain fractions based on their magnetic susceptibilities. The separator is typically operated with intensities ranging from 0.2 to 1.2 A, using a standard side slope of 20° and a forward pitch of -15°, unless specific cases that require adjustment.

The procedure involves passing each sample repeatedly until the full range of 0.2, 0.4, 0.6, 0.8, 1.0, 1.2 A intensities are separately recovered.

Figure 4 Flowchart illustrating the laboratory workflow for heavy mineral separation and analysis. The process sequences from initial disaggregation and sieving through density pre-concentration, followed by magnetic and gravimetric separation, mounting and two-stage single-grain characterization (optical microscopy and SEM-EDS). It concludes with the compilation of all chemical and physical properties for a comprehensive provenance study.



To determine the appropriate analytical path, all separated fractions are inspected under a binocular microscope. If a sufficient concentration of heavy minerals is observed, the sample proceeds directly to single-grain analysis. However, for samples with a low concentration of dense phases (typically zircon), a secondary density separation is required. This is achieved using heavy liquids, specifically diiodomethane (CH₂I₂), which has a standardized density of $\delta = 3.325$

g/mL at room temperature. This procedure is conducted within a high-efficiency hood following safety protocols. A key advantage of using heavy liquids at this stage is that only a minimal volume of sample needs to be processed, significantly reducing chemical exposure. The mineral fraction is immersed in the diiodomethane inside a glass separatory funnel; minerals with a specific gravity greater than the liquid settle (e.g., zircon), whereas the lighter fraction floats. Both fractions are thoroughly washed to remove any residual heavy liquid and are subsequently dried inside the hood.

At this stage, the various mineral species are identified, described and photographed using a Leica S8APO binocular magnifier equipped with a DMC2900 digital camera. Textural and physical parameters including color, luster, cleavage, shape, habit, roundness, sphericity, grain size, and degree of dissolution, corrosion or weathering, are recorded for each magnetic fraction following standard criteria (e.g. Mange and Maurer, 1992).

For further analysis, heavy mineral grains are hand-picked, placed onto a double-sided tape and mounted in a mold using epoxy resin. Once cured, the mount is polished in a three-stage process using 320, 600 and 1200 grit abrasive papers, followed by a final polishing sequence using diamond pastes of 3 μ m, 1 μ m, and 1/4 μ m until a reflective finish is achieved.

Detailed characterization of heavy minerals is performed using the SEM-EDS. The polished mounts are carbon- or gold-coated to ensure electrical conductivity and prevent charging. BSE imaging is used to describe internal textures linked to compositional variations, and inclusions, a standard procedure before detrital zircon dating. Furthermore, EDS enables point chemical analysis and elemental mapping, providing precise chemical quantification.

V. CONCLUSION

The integration of multiple analytical techniques constitutes a robust framework for sedimentary provenance studies. The laboratory workflows implemented at the Department of Geosciences (CURE-UdelaR) ensure high-quality samples preparation and thorough characterization through a systematic and standardized approach. Key procedures, including comminution, sieving, multi-step density and magnetic separations, provide an efficient pathway for the recovery of heavy minerals, even in samples with low initial concentrations. Furthermore, the combination of SEM-EDS imaging and chemical quantification provides critical data, enabling the precise identification of source rocks. Ultimately, these standardized methodologies minimize experimental bias and optimize data acquisition, ensuring that the transition from field samples to final analytical data is both, cost-effective and scientifically rigorous.

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