

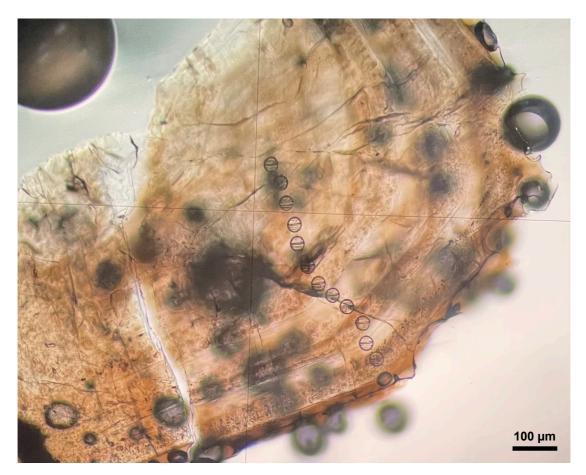


LA-ICP-MS Laboratory Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry

Annual Report 2023

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Plane polarized light microscope image of a plagioclase crystal with laser ablation pits (30 µm spot size).





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Introduction

This document reports the activities carried out by the LA-ICP-MS laboratory, including: 1) personnel and user accesses; 2) technical implementations, 3) costs and incomes, 4) education and outreach activities, and 5) scientific production. For the year 2023, the operation costs were fully covered by the laboratory incomes from the analytical work carried out for internal (CISUP) and external users. Extraordinary maintenance costs derived from the unforeseen substitution of some laser optics, required after their accidental damage during an infrastructural work done in the laboratory. The growing number of users registered in 2023 and their general level of satisfaction for the analytical work set a good premise for the economic self-sustainability of the laboratory for the year 2024.

Brief history of the laboratory

The installation of the Laser Ablation (LA) system ESI NWR-193 (ArF Excimer Laser) and the Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) PerkinElmer NexION 2000 was completed in December 2020. During the first year of activity of the LA-ICP-MS laboratory, most of the analytical time has been dedicated to the setup and tuning of the two instruments for the different types of analyses that are currently performed. Note that the two instruments operate independently, and analyses are performed upon setting a physical connection between the two, for which no specific training was given by the instrument selling companies. Following the installation of the LA-ICP-MS system (late 2020), the laboratory infrastructure required several implementations, including the air extraction system and the He+Ar gas supply lines (upgraded in 2022). As more people got to know the LA-ICP-MS laboratory, the number of users has increased over time, reaching in 2023 a total number of 16 users involved in more than 30 research projects.

The LA-ICP-MS laboratory is currently operative for the following type of analyses:

- ♦ U-Pb-Th geochronology (on crystals of zircon, calcite, monazite and allanite)
- ♦ Trace element analysis in silicate glasses, minerals and melt inclusions
- ♦ Trace element analysis in non-silicate matrixes (metal, oxides, sulfides and carbonates)
- ♦ Measurement of isotopic ratios (major elements) for paleo-thermometry





1. Personnel and user access

The LA-ICP-MS laboratory is currently managed by Prof. Matteo Masotta (RAR), with the collaboration of Dr. Pier Paolo Giacomoni (Researcher) and Dr. Stefano Iannini Lelarge (PhD student). Owing to the complexity in the use of the instrumentation and the requirement of postprocessing of the raw data (*data reduction*) with a dedicated software, the access to the laboratory is limited to trained users only.

During 2023, the laboratory received the access of 16 users (8 internal users and 8 users from other universities and research institutions. In addition to these users, the laboratory has provided service for BSc, MSc and PhD theses (8 students), as well as for the traineeship activities (4 students). Most of the analytical work carried out in the LA-ICP-MS laboratory is inherent to scientific projects related to Earth Sciences disciplines.

2. Technical implementations

New optics and mass flow controller for N_2 . In October 2023 a general maintenance of the laser ablation system has been performed by the ESI company and part of the optics (those showing signs of degrading) were replaced with new ones (Figure 1). The replacement was followed by a realignment of all the optics that lead to a significant improvement of the beam energy transmission toward the ablation chamber. In this phase, a second mass flow controller for N_2 make-up gas was also installed. The addition of the N_2 make-up gas decreases the ionization temperature in the plasma and improves the instrument sensitivity.

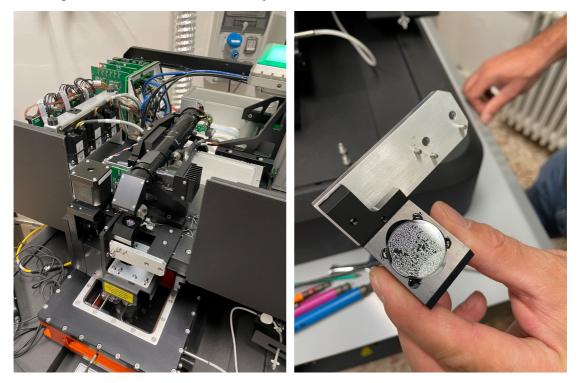


Figure 1. Insight into the laser ablation system exposed during the technical maintenance of the instrument (left) and image of an optic showing an advanced state of degrading due to atmospheric humidity (right).





3. Costs and incomes for 2023

The LA-ICP-MS laboratory income for 2023 amounts to a total of 12,400 \in . This includes the income from internal (CISUP) user projects (7,500 \in) and external user projects (4,900 \in). No income from third parties (industry) has been obtained this year. The presence of a technician dedicated to routine and customised analytical work for third parties could possibly increase the income of the laboratory. The total income fully covered the laboratory costs of 2023 (6,000 \in) and part of the regular maintenance costs of the ICP-MS (15,000 \in per year). An unexpected additional maintenance cost came from the substitution of some optics of the laser ablation system (9,000 \in), due to their accidental damage during unplanned infrastructural work in the laboratory.

4. Education and outreach

The LA-ICP-MS laboratory is yearly involved in laboratory activities and practicals carried out for BSc, MSc and PhD students (24 hours), as well as in demonstration activities carried out for high school students visiting the Department of Earth Sciences in the frame of the orientation program of the University of Pisa. A new specific course on LA-ICP-MS technique applied to Earth Sciences will be offered starting from 2024 for PhD students from the University of Pisa and from other Italian universities. During 2023, a large amount of time (approximately 80 hours) has been dedicated to analytical work performed for BSc, MSc and PhD theses (8 students) and for traineeship (4 students).

5. Scientific production

In 2023, the first article containing data obtained in the LA-ICP-MS laboratory was published. At present, there are 6 papers that have been submitted or are under review:

- 1. Fulignati P., Mulas M., Villalta Echeverria M.D.P., Fornasaro S., Larreta E., Mendoza Arteaga P.L., Menoscal Menoscal M.A., Romero-Crespo P., Gioncada A. (2023). The propylitic alteration in the Ponce Enriquez Gold Mining district, Azuay province, Ecuador: genetic constraints from a mineral chemistry and fluid inclusions study. *Front. Earth Sci.* **11**, 1255712.
- 2. Jacobs J., Rocchi S., Bach W., Masotta M., Pedersen L.E. R. (----). Serpentinite-sediment associations: provenance controlled by competing extensional-contractional tectonic processes during the evolution of the Northern Apennines (eastern Elba Island, Tuscany). *Tectonics (under review)*
- 3. Coletti G., Borromeo L., Fallati M., ... Taccola G. (----) Combining geological and historical archives to reconstruct floods variability in northwestern Italy during the last thousand years. *Submitted to Scientific Reports*
- 4. Colle F., Masotta M., Costa S., Giacomoni P., Trua T., Marani M. (----). A micro-scale insight into a back-arc trans-crustal plumbing system: the case of Marsili Volcano, Southern Tyrrhenian Sea. *Submitted to Lithos*
- 5. Di Rosa, M., Farina, F., Marroni, M., Jeon, H., and Pandolfi, L. (----) U–Pb ages from felsic rocks of External Ligurian sedimentary mélange (Northern Apennine, Italy): Tracing the pre-Jurassic history of the hyper-extended Adria continental margin. *Submitted to Journal of the Geological Society*
- 6. Iannini Lelarge S., Masotta M., Folco L., Ubide T., Suttle M.D., Pittarello L. (----) Melting experiments of a L6 ordinary chondrite: implications for the formation of alkali-rich achondrites. *Submitted to Journal of Petrology*
- 7. Tortelli G., Gioncada A., Pagli C., Braschi E., Gebru E.F., Keir D. (----) From melt- to crystal-rich magmatic system during rift localization: Insights from the Stratoid and Afar Gulf mineral chemistry in Central Afar (Ethiopia). *Submitted to Lithos*





Cite the laboratory

We are grateful that the papers which benefited of the work carried out in the LA-ICP-MS laboratory do acknowledge the laboratory correctly in their methodology chapters. Please check our laboratory web page, how to correctly cite our laboratory/instrument. For instance, a sentence like the following one: "Laser ablation-ICP-MS analyses were performed using a PerkinElmer NexION 2000 ICP-MS coupled with a NWR-193 Ar-F 193 nm excimer laser at the Centro per la Integrazione della Strumentazione della Università di Pisa (CISUP)"