WORKSHOP DE ESPECTROMETRIA DE MASSA INORGÂNICA
WORKSHOP OF INORGANIC MASS SPECTROMETRY

II – WORKSHOP OF INORGANIC MASS SPECTROMETRY - 2017

GEOCIÊNCIAS USP

LA-MC-ICPMS instrumentation and acquisition of U-Pb ages on zircon, monazite and titanite at CPGeo-USP

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1. Introduction

Acquisition of U- Pb data on silicate minerals is routinely performed in the Geochronology Research Center Laboratory (USP) by laser inductively coupled plasma multicollector mass spectrometry (LA-MC-ICP-MS) technique. The main application of these analytical data is on geochronology and petrogenetic problems, once the results can be applied to determine the age of crystallization and inherited cores of igneous rocks, and the age of metamorphism. Additionally, U-Pb ages can provide information on provenance and age of deposition of sedimentary rocks dating detrital zircons.

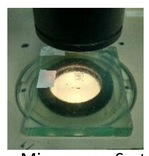
The U-Pb geochronology is based on the disintegration of the 235U (0.704Ga half-life) and 238U (4.47Ga half-life) isotopes into the 207Pb and 206Pb radiogenic isotopes, respectively.

193 nm - Laser Ablation – Photon Machines®

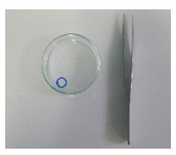
MC-ICP-MS – Neptune - Thermo®

2. Mineral separation

The procedure consists of isolating the minerals from the rocks by conventional hydraulic, magnetic and density techniques, before the final purification by handpicking the minerals under stereomicroscope with a tweezer help.



Olympus SZH10 -Zoom Stereo Microscope System



Petri dish and tweezer

Minerals on adhesive tape

3. Mount preparation

The selected grains are then mounted on specific resin-made disks (“mount”) and polished.

Resin

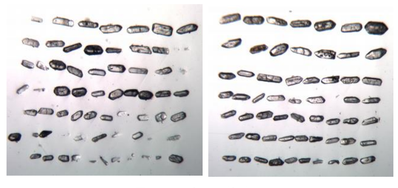
Mount template

Polishing machine for sanding and polishing

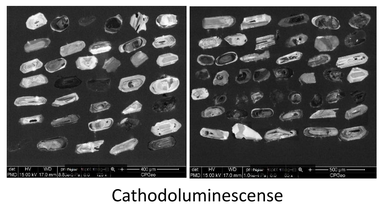
Mount

4. Stereomicroscope and cathodeluminescense images

After the “mount” be prepared, images by transmitted light and cathodoluminescense in a stereomicroscope (Olympus SZH10® - Zoom Stereo Microscope System) and scanning electron microscope (VPESEM – Quanta 250 – FEI Company®) respectively are obtained to identify the internal structure of the grains used to orientated the Laser spot position.



Transmited light

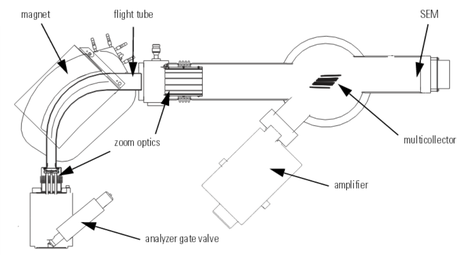


Cathodoluminescense

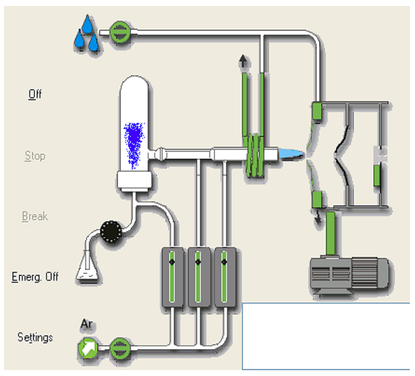
5. LA-ICP-MS – Laser Ablation Inductively Coupled Plasma Multicollector Mass Spectrometry

The mount to be analyzed is placed in the sample chamber of the laser (Photon-machines® - AnalyzerG2 - 193nm - Excimer Laser Ablation System) where the He gas (6.0 purity) transport the material removed from the crystal through a Teflon tube into the MC-ICP-MS (Thermo® - Neptune), where the sample is ionized in the argon plasma (6000 – 10000K). The resulting ions are introduced into the mass spectrometer and separated by a high resolution magnetic sector analyzer as a function of its mass/ charge ratio.

Sample chamber



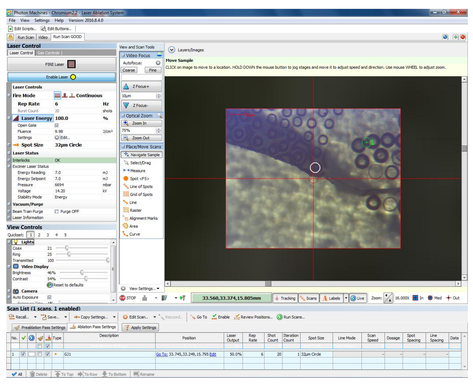
Multicollector module



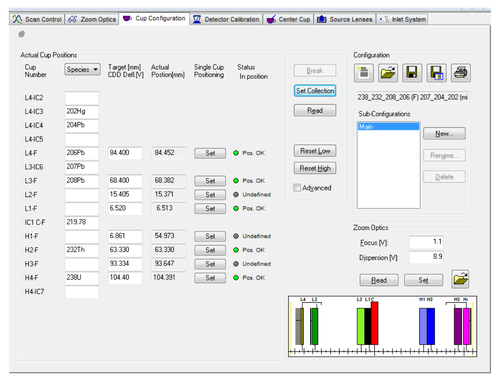
Plasma

6. U-Pb ages in zircon, monazite and titanite

U-Pb radiometric system is applied on U-bearing minerals such as zircons, monazites and titanites. The regular operating conditions for analysis of this methodology in the laser are: 6Hz frequency, 9.98 J/cm² fluence, ablating during aproximate a minute and 32 μm spot size to zircon and titanite, and 19 μm for monazite. The configuration of MC-ICP-MS is mixed between Faraday collectors and ion multipliers (MIC) and they are calibrated to analyze isotopes: 238U, 206Pb, 208Pb in Faradays and 202Hg, 204Pb + 204Hg and 207Pb in MIC. At each 50 minutes of analyses, are measured: four blanks, four synthetic NIST-612 standard and five GJ1 standard that they are used for all corrections, one of the Mud Tank standard is performed as an unknown sample, and twelve measurements of unknown sample that complete one spreadsheet. The data obtained in the MC-ICP-MS are reduced and corrected with in-house software developed by Siqueira et al, 2014, using a R statistical package based on Python programming language. Graphs and ages results are reported using Isoplot 3.0 (Ludwig, 2003) software.

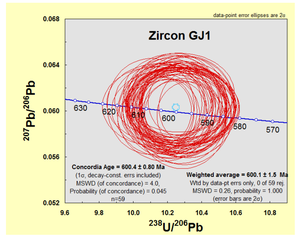


Laser calibration

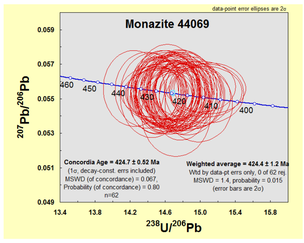


Cup configuration

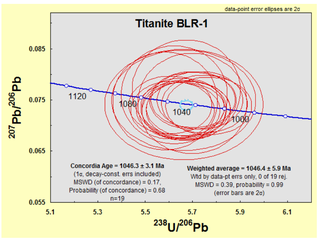
Three results of zircon, monazite and titanite standards are reported below. These results when compared with the TIMS available values in the international literature: 608.5 ± 0.4Ma (Jackson et al., 2004); 424.9 ± 0.4Ma (Aleinikoff et al., 2006) and 1047.1 ± 0.4Ma (Aleinikoff et al., 2007), respectively, illustrated the quality of LA-ICP-MS analysis performed at USP laboratory.



Tera-Wasserburg plot - GJ1 - zircon standard



Tera-Wasserburg plot – 44069 - monazite standard



Tera-Wasserburg plot – BLR1 - titanite standard

7. Reference

(1) R. Siqueira et al, 9th South American Symposium on Isotope Geology, 2014, 306.
(2) K.R. Ludwig, Berkeley Geochronology Center Special Publication, 2003, no.4,74.
(3) S.E. Jackson et al., Chemical Geology 211, 2004, 47-69.
(4) J.N. Aleinikoff et al., Geological Society of America Bulletin 118, 2006, 39-64.
(5) J.N. Aleinikoff et al., American Journal of Science 307, 2007, 63-118.