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BLANK LEVELS IN ISOTOPIC ANALYSES AT THE GEOCHRONOLOGICAL RESEARCH CENTER

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Precise and accurate isotope analyses via thermal ionization mass spectrometry (TIMS) and multi-collector inductively coupled plasma mass spectrometry (MC-ICPMS) require both laboratorial low blank (picogram levels) and robust corrections to assure efficient mass discrimination. A combination of these, together with the advances in high-resolution mass spectrometry with outstanding improvement in sensitivity of multi-collector equipments, enables to determine the isotope composition of a number of elements from micro-sized natural matrices such as rocks, minerals, water, aerosols and organic matter.

This work presents a discussion of the importance of low blank values, its sourcesand ways to avoid sample contamination at isotopic analyses.

Some papers [1,2]suggest that blank values should be < 0.1% of sample content, so that the influence of procedural blank on the isotope ratioswould be negligible.To achieve low blank values, the Geochronological Research Center (CPGeo) laboratory follows a rigid procedure protocol.CPGeo assembles a set of chemical laboratories where ion exchange chromatographic techniques are used for isolating specific elements from natural materials in order to determine their isotope compositions either by TIMS or MC-ICPMS.

Blank is a direct parameter to determine how clean the lab is. These labs are equipped with polypropylene-made laminar flow hoods and clean-boxes (class100) working under temperature and pressure-controlled conditions provided by high-efficiency particulate air system (HEPA, class 10,000) which is periodically certified.

The totalprocedure blank is strongly influenced by the degree of purificationand volumeof reagents [3]. At the CPGeo, all the reagents are ordered as P.A. level from Merck® andthen purified by sub-boiling distillationin Savillex® DST-1000 devices, and stored inpreviously decontaminatedteflon bottles. Water is firstly purified by reverse osmosis and thendeionized by aMilli-Q® system, reaching a final resistivity value of 18.2 MΩ·cm at 25ºC. Blanksfor Sr and Pb in all reagents (includingMilli-Q®water) are presently below 1 pg/g.

Besides reagent purity, labware cleaning is also a prerequisite to minimize the total procedural blank in isotope chemistry labs. Tubes and pipette tipsarecleaned up with 50 % (v/v) HNO_3 solution and sonicated for 30 minutesbefore being used. Teflon bottles are half filled with the 50% (v/v) HNO_3 solution and heated at 60 ^{o}C for a week, whileSavillex®teflon beakers are rigorously cleaned. They are firstly washed with reverse osmosis water and neutral liquid soap, followed by boiling in 50% (v/v)HNO_3 for 1 hour, triple rinsing with Milli-Q® water and finally boiling in Milli-Q® water for 1 hour. For some specific elements such as Pb, the cleaning procedure includes successive boiling steps in HNO_3, HCl and HNO_3 again intercalated byMilli-Q®

waterwashings. Acids used in this procedure are neutralized and discarded according to IGc - USP protocol.

To keep blank levels as low as possible, the analyst must be aware of his interference on every step of the chemical procedure, carefully handling the samples in order to avoid cross-contamination from other samples. The use of clean coat, laboratory shoes, cap and gloves is also important to keep the laboratory free from external contamination.Taking full control on these parameters, the CPGeo has achieved maximum blanks levels of 100 pg to Sr and Pb procedures and 40 pg to Nd.The table 1 shows blank data from other laboratories that deal with isotope analysis.

Table 1- Total blank values obtained by ID-TIMS for whole rock isotope analysis.

Reference	Pb (pg)	Nd (pg)	Sr (pg)
Presentwork	**100**	**40**	**100**
Yang, 2011[4]	-	20	-
Saji, 2016[5]	-	100	-
Taylor, 2015[6]	75	-	-
Baker, 2004[7]	80-100	-	-
Avanzinelli, 2005[8]	-	-	120-300
Takahashi, 2009[9]	-	-	21-33

Procedural blank values obtained at CPGeo are in agreement with those measured in other laboratories. Our data are lower than 0.1% of our samples content, so that the influence of procedural blank on the isotope ratios is negligible and no correctionsare necessary.

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