# 40Ar/39Ar Mica Dating – Analytical Protocol

The samples were crushed and sieved to obtain 180 – 250 μm fractions. The finer particles were decanted in tap water and the coarser residue further ultrasonically washed in acetone and deionized water several times. The optically best grains void of any coatings or inclusions were handpicked under a stereomicroscope. The samples were packed in aluminum capsules together with the Hb3gr (PP20) flux monitor standard along with zero age reagent grade K2SO4 and optical grade CaF2 salts for interference corrections. The samples were irradiated at the BNC reactor (Hungary) for c. 6.5 hours, with a nominal fast neutron flux density of c. 5.513 n\*(cm−2 \*s−1). The interference correction factors for the production of isotopes from Ca and K are located in the supplementary raw datatable (ES-6). Samples were placed in a 3.5mm pit size aluminum sample disk and step heated using a defocused 3.5 mm CO2 laser beam from Photon Machine Fusions 10.6 with a flat energy spectrum. The extracted gases from the sample cell were expanded into a Piston Free Stirling Cryocooler for trapping potential water vapor and further into a two-stage low volume extraction line (c. 350 cm3), both stages equipped with SAES GP-50 (st101 alloy) getters, the first running hot (c. 350 °C) and the second running cold. They were analyzed with a MAP 215–50 mass spectrometer in static mode, installed at the Geological Survey of Norway. The peaks and baseline (AMU = 36.2) were determined during peak hopping for 10 cycles (15 integrations per cycle, 30 integrations on mass 36Ar) on the different masses (41–35AMU) on a Mascom electron multiplier in analogue mode and linearly regressed back to zero inlet time. Blanks were analyzed every third measurement. After blank correction, a correction for mass fractionation, 37Ar and 39Ar decay and neutron-induced interference reactions produced in the reactor was undertaken using in-house software AgeMonster, written by M. Ganerød. It implements the equations of McDougall and Harrison (1999) and the newly proposed decay constant for 40K after Renne et al. (2010). A 40Ar/36Ar ratio of 298.56 ± 0.31 from Lee et al. (2006), was used for the atmospheric argon correction and mass discrimination calculation using a power law distribution of the masses. We calculated J-values relative to an age **of** 1080 ± 1.1 Ma for the PP20 hornblende flux monitor (Renne et al., 2010). We define a plateau according to the following requirements: at least three consecutive steps overlapping at the 95% confidence level (1.96σ) using the strict test:

(if errors quoted at 1σ),

>= 50% cumulative 39Ar released, and mean square of weighted deviates (MSWD) less than the two tailed student T critical test statistics for n - 1. Weighted mean ages are calculated by weighting on the inverse of the analytical variance. The uncertainties are expanded in cases where MSWD > 1 using to account for this excess error contribution.

# References

Lee, J.-Y., Marti, K., Severinghaus, J. P., Kawamura, K., Yoo, H.-S., Lee, J. B., and Kim, J. S., 2006, A redetermination of the isotopic abundances of atmospheric Ar: Geochimica et Cosmochimica Acta, v. 70, no. 17, p. 4507-4512.

McDougall, I., and Harrison, T. M., 1999, Geochronology and Thermochronology by the 40Ar/39Ar Method, New York, Oxford University Press.

Renne, P. R., Mundil, R., Balco, G., Min, K., and Ludwig, K. R., 2010, Joint determination of 40K decay constants and 40Ar∗/40K for the Fish Canyon sanidine standard, and improved accuracy for 40Ar/39Ar geochronology: Geochimica et Cosmochimica Acta, v. 74, no. 18, p. 5349-5367.