## **Supplementary material**

## Method for LA-ICPMS spot analysis for U-Pb age determination

- LA-ICPMS spot U-Pb geochronology was conducted at the PAMAL platform in the Institut Pluridisciplinaire de Recherche Analytique en Environnement et Matériaux (IPREM), Pau, France. Similar to the isotopic maps, the method uses a femtosecond laser ablation system (Lambda3, NexeyaBordeaux, France) coupled to an HR-ICPMS Element XR (ThermoFisher Scientific, Bremen, Germany) fitted with the Jet interface. The laser beam produced by the ablation system has a size of ~15 µm. To allow a more obvious comparison with the ablation strategies commonly used for U-Pb dating of carbonates by nanosecond LA-ICPMS (spot size of ~100 µm), we have defined a laser trajectory with the galvanometric scanners, allowing to obtain 100 µm square ablation craters at a repetition rate of 1 kHz, for 35 s of ablation. Samples were
  pre-ablated during 4 s with 150 µm square craters to remove possible contamination. ICPMS set-up was similar to that used for the isotopic maps. For the <sup>207</sup>Pb/<sup>206</sup>Pb ratio, normalization was based on standard sample bracketing to glass SRM NIST614. For the <sup>238</sup>U/<sup>206</sup>Pb ratio, normalization was based on the calculation of a correction factor deduced from all the drift-corrected analyses of the WC1 primary standard made during one session (i.e., half a day), following the method of Roberts et al. (2017). Three analyses of the glass SRM NIST614, and five analyses of WC1 were performed between each 30 ablations of the
- 15 unknown. The variability of the NIST614 standard on Pb-Pb and Pb-U ratios during the session is propagated quadratically over the analytical uncertainty of the unknown sample ratios as an excess variance (see Horstwood et al., 2016). The age of the unknown samples is then determined from a Tera-Wasserburg concordia diagram, without correction for common lead, using IsoplotR software (Vermeesch, 2018). The robustness of the ages is estimated from the alignment of the points and the MSWD value. The uncertainty on the age corresponds to that calculated by the regression, to which systematic uncertainties
- are added (decay constant (0.1%), uncertainty on the age of WC1 (2.7%, Roberts et al., 2017)). It should be noted that the long-term reproducibility of the method was not added, as the method has been applied to 5 sessions only in October 2018 and February 2019. The Duff Brown sample (64.04 Ma, Hill et al. (2016)) was used as a secondary standard, but for one session only (February 2019). The age obtained is  $63.66 \pm 1.97$  Ma without anchoring of common lead, and  $65.02 \pm 0.43$  Ma with common lead anchored at a value of 0.738 calculated from Hill et al (2016) (without propagation of systematic uncertainties)
- 25 (Fig. S1).



Figure S1: TW concordia plot for the Duff Brown samples analyzed by LA-ICPMS spot analysis, with a common Pb value anchored to a value of 0.738 (left), and without anchoring (right).

## 30 References

Horstwood, MSA, Košler, J, Gehrels, G, Jackson, SE, Mclean, NM, Paton, C, Pearson, NJ, Sircombe, K, Sylvester, P, Vermeesch, P, Bowring, JF, Condon, DJ, Schoene, B.: Community-Derived Standards for LA-ICP-MS U-(Th-)Pb Geochronology - Uncertainty Propagation, Age Interpretation and Data Reporting. *Geostandards and Geoanalytical Research*, *40(3)*, 311-332, doi:10.1111/j.1751-908X.2016.00379.x., 2016.

35 Roberts, N. M. W., Rasbury, E. T., Parrish, R. R., Smith, C. J., Horstwood, M. S. A. and Condon, D. J.: A calcite reference material for LA-ICP-MS U-Pb geochronology: *Geochemistry, Geophysics, Geosystems,* doi:10.1002/2016GC006784, 2017 Vermeesch, P.: IsoplotR: A free and open toolbox for geochronology. *Geoscience Frontiers, 9*, 1479-1493, doi: 10.1016/j.gsf.2018.04.001, 2018

40



Figure S2: Left: isotopic maps used for location of areas amenable to dating (preliminary images), along with their related analysis time and dimension; Right: sample images. For samples PXG20-1 and PXG32-2, the ablation pits of the LA-ICPMS spot analyses are visible. Location of isotopic maps is indicated by the frames (preliminary images: black; isotope dating maps: red).

Fig S3: Tera-Wasserburg, 86TW and isochrone plots obtained for samples BH14, PXG20-1, PXG32-2, ETC2 and ARB. The same plots for discretized data (i.e., ellipses) are also given.























