Appendix. U–Pb SHRIMP-RG zircon geochronology techniques

Zircon mineral separates were produced at the U.S. Geological Survey in Denver using conventional methods. Samples weighing between 1 and 5 kg were crushed, pulverized, sieved (less than 150 µm), washed, and magnetically split to obtain a non-magnetic fraction. The non-magnetic fraction was then put through a heavy liquid (MeI; $\rho = 3.33$), and the zircon concentrate collected from the heavy fraction. After briefly cleaning with acetone, the analyzed zircon fractions were handpicked from this heavy-concentrated, non-magnetic fraction.

SHRIMP-RG (Sensitive High-Resolution Ion MicroProbe-Reverse Geometry) procedures used in this study are similar to those reported by Old et al. [19] and Kim and Travers [11]. Briefly, zircons and chips of standard zircon R33 were mounted in epoxy, ground to nearly half their thickness using 1500-grit, wet-dry sandpaper, and polished with 6- and 1-µm-grit diamond suspension abrasive. Transmitted- and reflected-light photos were taken of all mounted grains (not shown) to aid in the spot selection to perform the SHRIMP analyses. In addition, scanning electron microscope–cathodoluminescence images (SEM–CL on a JEOL 5600 instrument) of all zircons (Figs. 8–12) were obtained at Stanford University prior to analysis, and used to reveal internal zoning related to chemical composition, in order to avoid possible problematic areas within grains. The mounts were cleaned in 1 N HCl and distilled water to minimize surface related common lead, and gold-coated for maximum surface conductivity.

U–Th–Pb analyses were made on 10 to 15 individual zircon grains per sample using the SHRIMP-RG housed in Green Hall at Stanford University, California, and co-owned by the U.S. Geological Survey. The primary oxygen ion beam, operated at about 2–4 nA, excavated an area of about 20–40 µm (adjustable depending on grain size) in diameter to a depth of about 1–2 µm; sensitivity ranged from 5 to 30 cps per ppm Pb. Data for each spot were collected in sets of five scans through the mass range. Nine peaks are measured sequentially for zircons with a single collector: $^{90}Zr^{216}O$, ^{204}Pb, background (0.050 mass units above ^{204}Pb), ^{206}Pb, ^{207}Pb, ^{208}Pb, ^{238}U, $^{248}Th^{16}O$, and $^{254}U^{16}O$. The reduced $^{206}Pb/^{238}U$ ratios were normalized to the zircon standard R33 (418.9 ± 0.4 Ma, ID-TIMS age at 2-sigma; from monzodiorite, Braintree Complex, Vermont; [1]). For the closest control of Pb/U ratios, one standard was analyzed after every four to five unknown samples. Uranium concentrations were monitored by analyzing a standard (CZ3) with ~550-ppm U composition. U and Pb concentrations are accurate to about 10–20%. SHRIMP isotopic data were reduced and plotted using the Squid and IsoplotEx programs of [7,8] and are presented in Tables 1 and 2. Uncorrected U–Pb spot data are projected to the Tera–Wasserburg Concordia diagrams (Figs. 3–7) along a model common-Pb line, after [16]. For every sample, a preferred weighted mean $^{206}Pb/^{238}U$ age is calculated at 2-sigma level of precision and shown in Tera–Wasserburg diagrams.

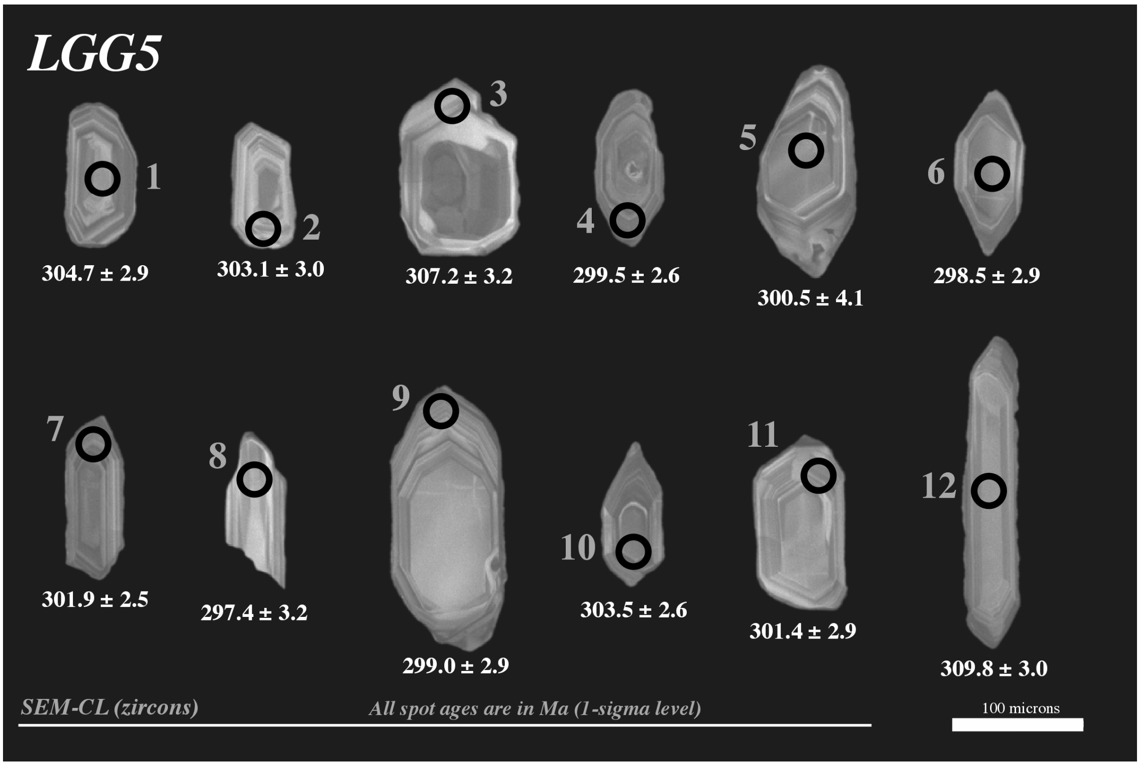

Fig. 8. Scanning electron microscope–cathodoluminescence (SEM–CL) images of 12 dated zircons from the Osor leucogranite sample LGG5. Circles and the adjacent numbers here and in the following figures represent the spot size (~25 μm) and the spot number, respectively. The $^{206}Pb/^{238}U$ ages here and in the following figures are reported in Ma at the 1σ level of precision.

Fig. 8. Images de cathodoluminescence, obtenues au microscope à balayage (SEM–CL), des 12 zircons datés du leucogranite d'Osor LGG5. Cercles et numéros représentent, ici et dans d'autres figures, la taille et le numéro des analyses (~40 μm). Les âges $^{206}Pb/^{238}U$ sont présentés ici et dans les autres graphiques avec une précision de 1σ.

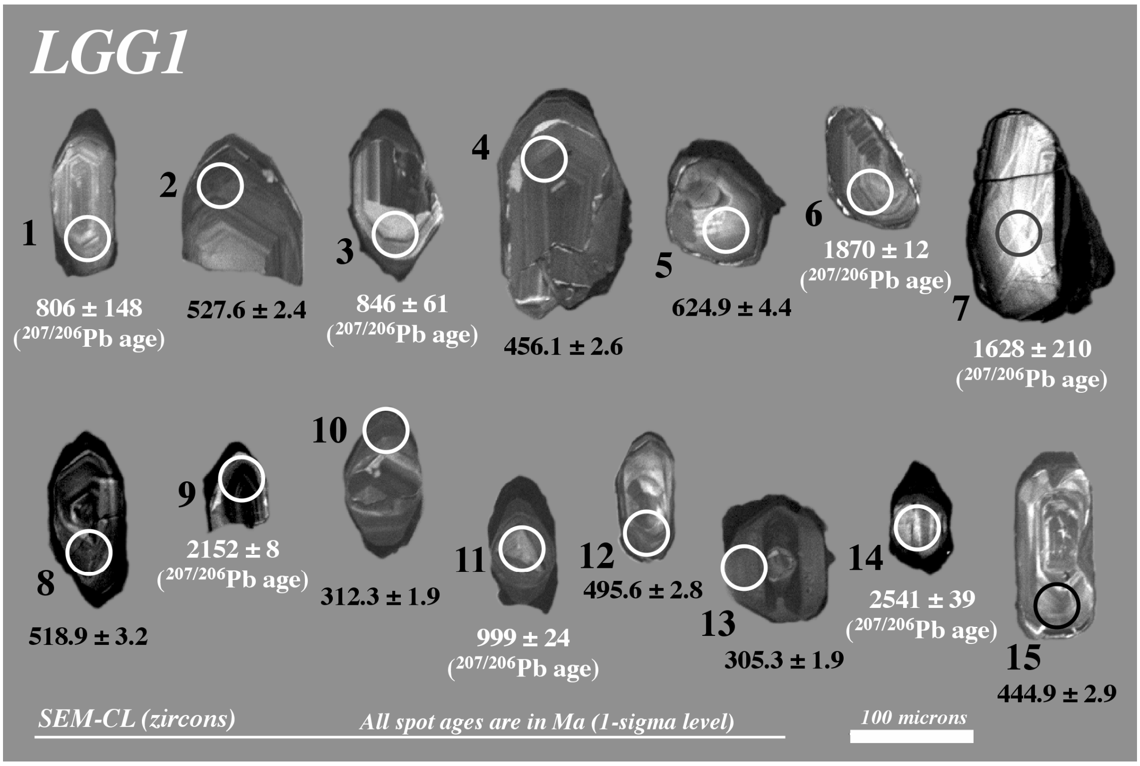

Fig. 9. Scanning electron microscope–cathodoluminescence (SEM–CL) images of 15 dated zircons from the Osor leucogranite sample LGG1. Spot size ~40 µm. Ages older than 700 Ma are reported as $^{207}Pb/^{206}Pb$ ages (see Table 2).

Fig. 9. Images de 15 zircons datés du leucogranite d'Osor LGG1. Tailles des analyses ~40 µm. Les âges les plus anciens, de 700 Ma, correspondent à des âges $^{207}Pb/^{206}Pb$ (voir Table 2).

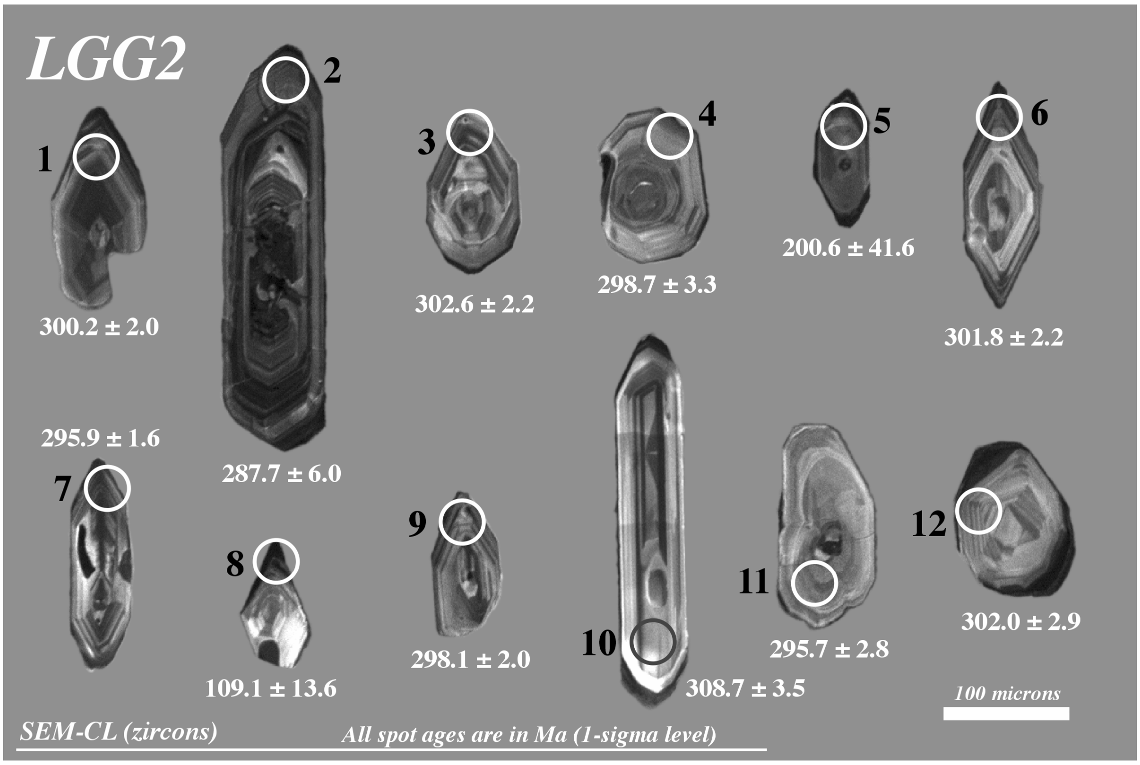

Fig. 10. Scanning electron microscope–cathodoluminescence (SEM–CL) images of 12 dated zircons from the Osor leucogranite sample LGG2. Spot size ~35 µm.

Fig. 10. Images de 12 zircons datés du leucogranite d’Osor LGG2. Tailles des zones analysées ~35 µm.

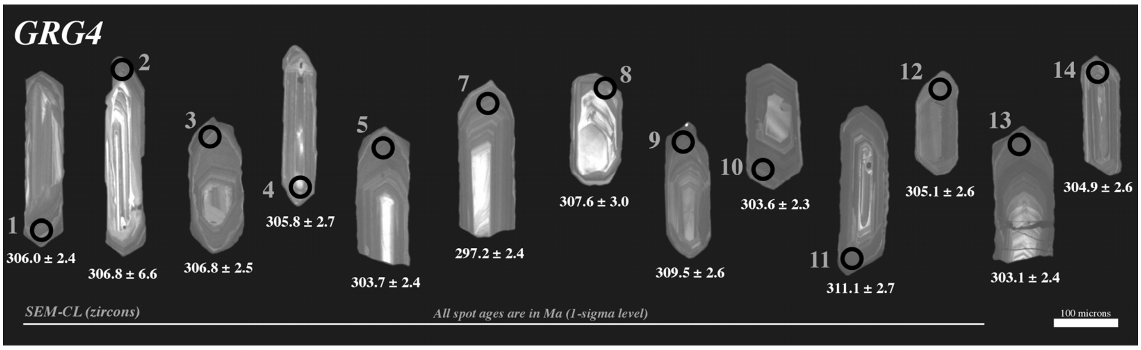

Fig. 11. Scanning electron microscope–cathodoluminescence (SEM–CL) images of 14 dated zircons from the biotite granite sample GRG4. Spot size ~30 µm.

Fig. 11. Images de 14 zircons datés du granite à biotite GRG4. Tailles des zones analysées ~30 μm.

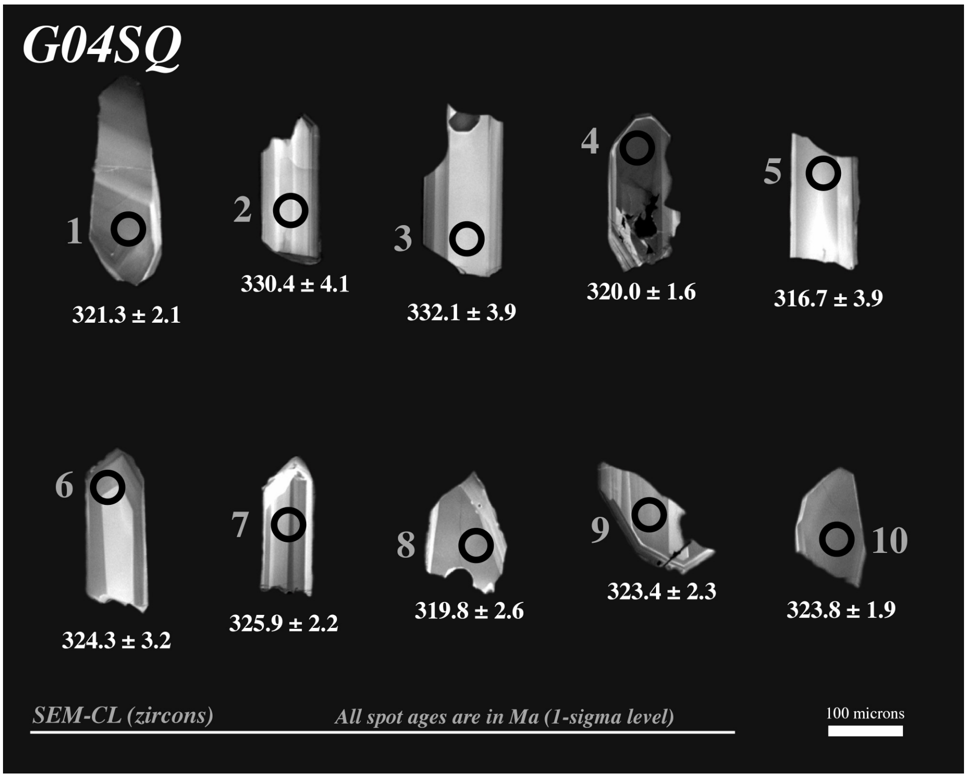

Fig. 12. Scanning electron microscope–cathodoluminescence (SEM–CL) images of 10 dated zircons from Susqueda diorite sample G04SQ. Spot size ~35 μm.

Fig. 12. Images de 10 zircons datés de la diorite de Susqueda G04SQ. Tailles des analyses ~35 μm