

Sensitive High Resolution Ion Microprobe (SHRIMP IIe/MC) of the Institute of Geosciences of the University of São Paulo, Brazil: analytical method and first results

*Microsonda Iônica de Alta Resolução e de Alta Sensibilidade (SHRIMP IIe/MC)
do Instituto de Geociências da Universidade de São Paulo, Brasil:
método analítico e primeiros resultados*

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Abstract

This paper presents the characteristics of the high resolution secondary ion mass spectrometer coupled with an Ion Microprobe (SHRIMP IIe/MC), installed at the Institute of Geosciences of the University of São Paulo (IGc-USP), as well as the respective analytical procedures and the first results obtained with standard samples, making possible to conduct routine analysis of zircon samples. The standard of the Temora 2 zircon, with age of 416.78 Ma, was analyzed by the SHRIMP IIe/MC at the Institute of Geosciences of the University of São Paulo and yielded the age of 416.8 ± 3.8 Ma. Samples of the Archean zircon OG1 and the Neoproterozoic zircon Z6266, which yielded U-Pb ages of 3465.4 ± 0.6 Ma and of 559 ± 0.2 Ma, respectively, using Thermal Ionization Mass Spectrometry (TIMS), were analyzed by SHRIMP IIe/MC at the IG-USP and yielded U-Pb ages of 3462.6 ± 5.1 Ma and 561.0 ± 0.92 Ma, respectively, in Concordia diagrams. Therefore, these values and the literature values are very close. Both OG1 and Z6266 samples, due to the homogeneity in their composition and isotopic relationship, can be used as SHRIMP standard. These results indicate that data obtained using SHRIMP IIe at the IGc-USP are reliable and compatible with international standards. Dating of samples with ages ranging from Cenozoic to Archean, obtained using SHRIMP IIe/MC U-Pb geochronology on zircon crystals, is presented here to illustrate this point.

Keywords: U-Pb zircon ages; SHRIMP; Geochronology; IGc-USP; Temora standard.

Resumo

Este trabalho apresenta as características de espectrômetro de massa de íons secundários acoplado com uma Microsonda Iônica de Alta Resolução, do tipo SHRIMP IIe/MC, instalada no Instituto de Geociências da Universidade de São Paulo (IGc-USP), bem como os respectivos procedimentos analíticos e os primeiros resultados obtidos em amostra de padrões, tornando-a apta a operar para análises em rotina de amostras de zircão. O padrão de zircão Temora 2, com idade de 416,78 Ma, foi analisado no SHRIMP IIe/MC de IGc-USP e apresentou a idade de $416,8 \pm 3,8$ Ma. As amostras de zircão arqueano OG1 e de zircão neoproterozoico Z6266, respectivamente com idades U-Pb obtidas por Espectrometria de Massa de Ionização Térmica (TIMS) de $3465,4 \pm 0,6$ Ma e de $559 \pm 0,2$ Ma foram analisadas no SHRIMP IIe de IG-USP e apresentaram idades U-Pb em diagramas Concórdia de $3462,6 \pm 5,1$ Ma e $561,0 \pm 0,92$ Ma respectivamente, portanto muito próximos aos valores referenciados na literatura. Tanto a amostra OG1 como a Z6266, em razão da homogeneidade de suas composições e das relações isotópicas, poderão ser utilizadas como padrões para SHRIMP. Esses resultados indicam que o SHRIMP IIe/MC do IGc-USP produz dados confiáveis compatíveis com os padrões internacionais. Como exemplos são aqui apresentadas datações de amostras de idades variáveis, desde cenozoica até arqueana, efetuadas pelo método geocronológico U-Pb por meio de SHRIMP IIe em cristais de zircão.

Palavras-chave: Idade U-Pb em zircão; SHRIMP; Geocronologia; IGc-USP; Temora.

INTRODUCTION

History

Development of Sensitive High Resolution Ion Microprobe

The design of Sensitive High Resolution Ion Microprobe (SHRIMP) I was based on an ion optical description by Matsuda (1974) for a double-focusing high mass-resolution mass spectrometer (Clement et al., 1977). The magnet was made as large as manufacturing capabilities allowed with a turning radius of 1.000 m, thus yielding a secondary-ion beam path of 7 m. The other components in the mass analyzer were an electrostatic analyzer, radius 1.272 m, used to correct the large velocity spread characteristic of (Secondary Ion Mass Spectrometer (SIMS), and an electrostatic quadrupole lens located between the Electrostatic Analyzer (ESA) and magnet, used by Matsuda to reduce second-order aberrations (additional information you can see by Ireland et al., 2008; Compston et al., 1984; Williams, 1998).

The SHRIMP IIe was installed at the Laboratory of High Resolution Geochronology of the Institute of Geosciences of the University of São Paulo (GeoLab-IGc-USP) in October, 2010 by staffs (professor, engineers and technicians) of the Australian Scientific Instruments (Figure 1). The mass spectrometer model is SHRIMP IIe/MC, in which “II” mean second generation and “e” mean extended, and MC is because it has a multicollector detector system with five multi-ion counting (MIC – continuous dynode), one electron multiplier (ETP – discrete dynode) and one Faraday cup. In this machine, it is possible to measure both positive and negative isotopes. Since 2011, the GeoLab-IGc-USP has obtained very accurate zircon U-Pb isotopic data, and there are plans, in the near future, for dating other minerals, as baddeleyite, titanite, xenotime and monazite, and also for analyzing stable isotopes, such as oxygen in zircon. The main purpose of this paper is to present analytical procedures for the U-Pb analysis of zircons using SHRIMP IIe, the first isotopic results obtained on international zircon standard/samples: Temora 2 (Paleozoic – Black et al., 2004), OG1 (Archean – Stern et al., 2009) and Z6266 (Neoproterozoic – Stern and Amelin, 2003), as well as some examples from South American Platform.



Figure 1. Picture of the SHRIMP IIe/MC at Institute of Geosciences of the University of São Paulo.

ANALYTICAL PROCEDURES FOR U-Pb ISOTOPIC ANALYSIS USING SHRIMP IIE/MC AT GeoLab-IGc-USP

Mount preparation and imagery

Zircons from each sample were arranged in rows with Temora 2 zircon reference standard, cast into epoxy resin grain mounts with a diameter of 2.54 cm, and polished with diamond compound (1 – 7 μm) to reveal grain centers. Methodology for sample preparation and hand picking can be seen in the Appendix 1.

Cleaning procedures:

- (1) wash with detergent;
- (2) wash for five minutes with diluted HNO_3 (1%);
- (3) wash three times with Petroleum Spirit;
- (4) wash three times with ultrapure hot water (MILIQ, 50 – 60°C);
- (5) dry at 50 – 60°C for 2 hours in a clean room.

Note: washing procedure described from step 1 to 4 was carried out in ultrasonic bath.

Optical and electronic imagery:

- (1) reflected and transmitted light images were acquired before gold coating of 2 – 3 μm ;
- (2) cathodoluminescence (CL) images were obtained by FEI Quanta 250 Scanning Electron Microscope (SEM) and XMAX CL detector (Oxford Instruments). SEM analytical conditions are: high voltage = 15 kV, work distance = 16.9 mm, detector = PMD, filament emission = 100 μA , and magnification range = 95 to 250x;
- (3) after CL acquisition, the gold was removed and the mount was recleaned;
- (4) the thickness (~3 μm) of the final gold coating was reached after a two-minutes exposition and an electrical conductivity resistance of approximately 15 – 17 Ω (between the border and the center of mount). Then, it was ready to be analyzed by the SHRIMP IIE.

SHRIMP IIE analytical setup

The SHRIMP IIE setup at the GeoLab-IGc-USP is described below:

- (i) Primary beam analytical conditions: Kohler aperture = 120 μm , spot size = 30 μm , and O_2 beam density = around 2.5 – 7 ηA (dependent of brightness aperture).
- (ii) Secondary beam analytical conditions: source slit = 80 μm ; mass resolutions for $^{196}\text{Zr}_2\text{O}$, ^{206}Pb , ^{207}Pb , ^{208}Pb , ^{238}U , $^{248}\text{(ThO)}$ and $^{254}\text{(UO)}$ ranging between 5,000 and 5,500 (1%), and residues < 0.025 (Appendix 2); energy slit = open.

Acquisition table setup

Raster time is 2 – 3 minutes with spot size = 50 μm , plus 0.5 minutes of burning time fixed at the center. Acquisition parameters are shown in Table 1.

Setup conditions of the acquisition table

Primary beam autotune is systematically carried out in the Source Steering Y and Source Steering Z focus lens (Appendix 3), and secondary beam autotune QT1Y and QT1Z are enabled (Appendix 4). Auto run selector is $^{254}\text{(UO)}$, in which auto center method is measured at 50% of the peak height. The analytical cycle number is equal to 5 cycles for Neoproterozoic-Archean samples, and equal to 6 – 7 cycles for Phanerozoic samples. All raw count rates have been corrected for a dead time of 25 ns and analytical rate among standard and sample is 1 standard to 4 zircon samples.

Acquisition and data processing

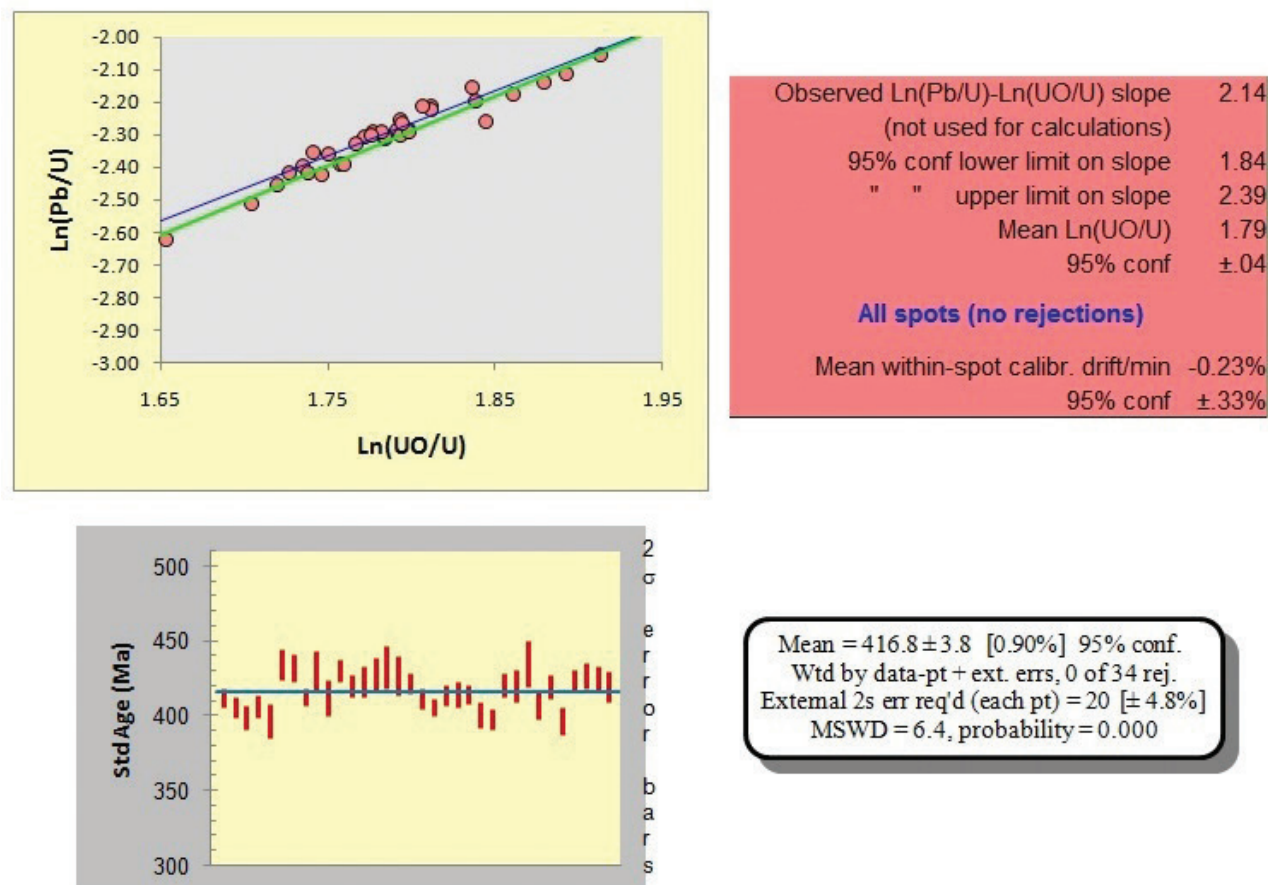
U-Pb calibration: the Pb^+ ionization efficiency is about a factor of two higher than U^+ , and so the $^{206}\text{Pb}^+/^{238}\text{U}^+$ ratio must be calibrated by a standard material (Williams, 1998). Measured $^{206}\text{Pb}^+/^{238}\text{U}^+$ varies with the measured UO^+/U^+ to define a calibration line of known age, in this case 416,78 Ma for Temora 2 (Figure 2) or 572 Ma for SL13. Figure 2 shows $\text{Ln}(\text{Pb}/\text{U}) \times \text{Ln}(\text{UO}/\text{U})$ trend line with slope of 2.14. The age of an unknown sample can then be determined by the ratio of $^{206}\text{Pb}^+/\text{U}^+$ in the unknown to that ratio in the standard at the common UO/U value. For additional information, please see Williams (1998).

SHRIMP softwares are LabVIEW 8.5 and SHRIMP SW, version 2.1. Data was reduced using SQUID 1.06, and the Concordia Diagram was plotted using ISOPLOT 4 (Ludwig, 2009). Common lead corrections usually use ^{204}Pb according to Stacey and Kramer (1975), but the software SQUID has the option to use ^{207}Pb and ^{208}Pb corrections. Temora 2 is used as $^{206}\text{Pb}/^{238}\text{U}$ age reference (416.78 Ma, Black et al., 2004), and SL13 (238 ppm) is used as U composition reference. Analytical uncertainties are shown in Tables 2 and 3 in 1- Σ error for each analysis.

ANALYSIS BY SHRIMP IIE OF TEMORA 2 STANDARD AND Z6266 AND OG1 SAMPLES AT GeoLab-IGc-USP

The Temora 1 zircon, as well as the Temora 2 zircon, is crystallized within the Middledale Gabbroic Diorite (Black et al., 2004). This body forms a small, high-level stock within the Paleozoic Lachlan Orogen of Eastern Australia. The weighted mean U-Pb age (416.78 \pm 0.33 Ma) measured by ID-TIMS (Isotope Dilution - Thermal Ionization Mass Spectrometry) and obtained from the nine Temora 2 analyses is interpreted as the best estimated age for these zircon crystals (Black et al., 2004).

The Z6266 zircon comprises clear, colorless, glassy megacryst fragments from Sri Lanka (Stern, 2001). Evidence for



The weighted mean age for 34 analyses is 416.8 ± 3.8 Ma. For 3 days, the age varied within an error of 0.9% and Ln(Pb/U) × Ln(UO/U) slope = 2.14. (For data, see Table 2.)

Figure 2. (Top) Ln(Pb/U) versus Ln(UO/U) calibration diagram for Temora 2 standard analyzed for 3 consecutive days (72 hours); (bottom) bar graph of $^{206}\text{Pb}/^{238}\text{U}$ ages (Ma) for Temora 2 analyzed for 72 hours.

Table 1. Acquisition setup table.

	Zr ₂ O	²⁰⁴ Pb	204.1	²⁰⁶ Pb	²⁰⁷ Pb	²⁰⁸ Pb	²³⁸ U	²⁴⁸ ThO	²⁵⁴ UO
D (s)	5	3	1	2	1	1	2	2	2
IT (s)	2	10	10	10*	20*	2	10	5	5
AC	ON	OFF	OFF	ON*	OFF	OFF	ON	ON	ON

D: delay; s: seconds; IT: analytical integration time; AC: mass auto center; ON: enabled; OFF: disabled; * integration time to low U concentration or young zircon, < 300 Ma, is changed to 20 s to ²⁰⁶Pb and 30 – 40 s to ²⁰⁷Pb and Auto Center is operated in OFF mode conditions.

U/Pb homogeneity of these materials includes TIMS U-Pb analyses obtained in two different laboratories, and ages are consistent and within analytical error. The Z6266 zircon yielded a weighted $^{206}\text{Pb}/^{238}\text{U}$ age of 559.0 ± 0.2 Ma, and ID-TIMS analysis gave a mean U concentration of 903 ± 23 ppm, total Pb = 79 ± 2 ppm and Th = 199 ± 6 ppm (Stern and Amelin, 2003).

Sample OG1 is a quartz diorite body from Pilbara Craton, Western Australia (Stern et al., 2009). TIMS $^{207}\text{Pb}/^{206}\text{Pb}$ reference value for OG1 zircon was 0.29907 ± 0.00011, which is equivalent to an age of 3465.4 ± 0.6 Ma. The OG1

zircons analyzed by SHRIMP RG at the Australian National University (ANU) yielded weighted mean $^{207}\text{Pb}/^{206}\text{Pb}$ age of 3468.1 ± 1.2 Ma, whereas the robust mean $^{206}\text{Pb}/^{238}\text{U}$ age was 3449 ± 10 Ma.

Results at GeoLab-IGc-USP

Cathodoluminescence images (CL) of Temora 2, Z6266 and OG1 zircon crystals are shown respectively in Appendix 5, 6 and 7, and the analytical results of U-Pb dating of these zircons using SHRIMP IIe at Geo-Lab-IGc-USP are shown in Tables 2 and 3.

Table 2. Ln(UO/U) x (Ln(Pb/U) data and $^{206}\text{Pb}/^{238}\text{U}$ reference age from Temora 2. Ln(UO⁺/U⁺) correlate directly with Ln(Pb⁺/U⁺), so that it was possible to determine a correction factor.

Spot name	ppm U	ppm Th	$^{232}\text{Th}/^{238}\text{U}$	$^{204}\text{Pb}/^{206}\text{Pb}$	% err	% comm ^{206}Pb	Total $^{206}\text{Pb}/^{206}\text{Pb}$	% err	Total $^{238}\text{U}/^{206}\text{Pb}$	% err	Total $^{207}\text{Pb}/^{206}\text{Pb}$	% err	$^{204}\text{Pb}_{\text{corr}}/^{238}\text{U}$ Age	1 σ err	Ln (UO/U)	Ln (Pb/U)	$^{204}\text{Pb}_{\text{corr}}/\text{UO}/\text{U}^2$
TEM-1.1	566	189	0.34	0.00041	35	0.77	0.108	2.3	0.129	2.6	0.056	2.6	413	3	1.913	-2.057	0.00279
TEM-1.2	447	251	0.58	0.00059	32	1.10	0.190	2.0	0.119	2.8	0.063	2.4	406	4	1.879	-2.141	0.00275
TEM-1.3	347	185	0.55	0.00052	41	0.96	0.171	2.5	0.114	2.3	0.058	2.9	399	4	1.861	-2.177	0.00270
TEM-2.1	368	134	0.38	-0.00014	2	-0.27	0.123	3.0	0.121	2.6	0.063	3.8	407	4	1.893	-2.112	0.00275
TEM-2.2	185	65	0.36	0.00086	34	1.61	0.138	3.6	0.106	2.8	0.066	4.3	397	6	1.844	-2.259	0.00269
TEM-3.1	221	111	0.52	0.00047	44	0.88	0.171	4.3	0.117	2.0	0.066	3.4	435	5	1.836	-2.154	0.00295
TEM-3.3	345	166	0.50	0.00013	136	0.24	0.171	2.8	0.101	2.3	0.061	3.9	432	4	1.777	-2.290	0.00293
TEM-3.4	930	281	0.31	0.00027	13	0.50	0.097	2.1	0.102	1.9	0.058	1.9	413	3	1.791	-2.286	0.00280
TEM-4.2	160	44	0.28	0.00084	41	1.57	0.125	4.3	0.107	2.0	0.074	4.5	431	7	1.793	-2.252	0.00292
TEM-4.3	258	125	0.50	0.00094	35	1.75	0.168	4.5	0.091	1.3	0.063	4.8	412	6	1.726	-2.417	0.00279
TEM-5.1	472	148	0.32	0.00035	65	0.66	0.106	2.6	0.110	2.7	0.060	2.4	431	4	1.812	-2.211	0.00292
TEM-5.2	467	163	0.36	0.00065	28	1.22	0.121	2.6	0.096	1.9	0.060	2.6	420	4	1.751	-2.358	0.00285
TEM-5.3	372	206	0.57	0.00088	35	1.65	0.186	2.6	0.101	2.2	0.063	3.8	423	5	1.784	-2.312	0.00287
TEM-6.1	797	287	0.37	0.00018	54	0.33	0.117	3.4	0.109	0.8	0.057	2.0	428	6	1.812	-2.223	0.00290
TEM-7.1	136	66	0.51	0.00047	66	0.87	0.180	8.1	0.111	2.3	0.069	7.7	433	7	1.807	-2.211	0.00294
TEM-7.2	200	91	0.47	0.00093	41	1.75	0.173	3.4	0.101	3.1	0.068	3.8	427	7	1.784	-2.307	0.00289
TEM-8.1	605	379	0.65	0.00030	30	0.57	0.200	1.9	0.102	2.0	0.057	2.4	422	3	1.782	-2.293	0.00286
TEM-8.2	581	165	0.29	0.00019	22	0.36	0.098	2.7	0.101	1.9	0.059	2.6	412	3	1.793	-2.299	0.00279
TEM-8.3	800	310	0.40	0.00021	43	0.38	0.125	2.7	0.092	2.7	0.059	2.1	406	3	1.757	-2.388	0.00275
TEM-8.4	790	276	0.36	0.00043	35	0.80	0.117	2.3	0.087	2.5	0.059	2.3	414	4	1.719	-2.455	0.00280
TEM-9.1	315	210	0.69	0.00082	33	1.54	0.212	3.4	0.113	1.4	0.061	3.0	415	4	1.838	-2.199	0.00281
TEM-9.2	582	138	0.24	0.00047	33	0.87	0.082	2.7	0.103	1.8	0.059	3.1	414	3	1.798	-2.285	0.00280
TEM-9.4	440	203	0.48	0.00019	88	0.35	0.150	2.4	0.089	2.7	0.062	2.7	401	4	1.746	-2.421	0.00271
TEM-9.5	276	151	0.56	0.00042	17	0.78	0.170	2.7	0.082	2.3	0.058	2.3	398	4	1.704	-2.512	0.00269
TEM-10.1	287	141	0.51	0.00099	28	1.85	0.172	2.7	0.103	1.8	0.063	3.7	421	4	1.798	-2.291	0.00285
TEM-10.2	306	155	0.52	0.00029	59	0.54	0.170	2.4	0.092	2.8	0.059	3.8	420	6	1.735	-2.396	0.00285
TEM-11.1	292	141	0.50	0.00046	43	0.86	0.166	2.7	0.096	2.3	0.065	3.0	435	8	1.742	-2.355	0.00295
TEM-11.2	285	138	0.50	0.00087	25	1.62	0.152	3.2	0.093	2.2	0.064	3.4	407	5	1.759	-2.390	0.00276
TEM-11.3	456	226	0.51	0.00039	33	0.73	0.161	2.8	0.090	1.3	0.061	2.8	419	4	1.738	-2.415	0.00284
TEM-11.4	421	210	0.52	0.00071	33	1.34	0.172	2.7	0.074	2.1	0.060	5.3	397	5	1.653	-2.623	0.00268
TEM-12.1	529	280	0.55	0.00042	26	0.79	0.169	2.1	0.105	1.3	0.061	2.4	424	4	1.794	-2.265	0.00287
TEM-12.2	384	211	0.57	0.00055	41	1.03	0.177	2.4	0.101	2.4	0.061	3.0	427	4	1.772	-2.308	0.00290
TEM-13.1	355	180	0.53	0.00052	32	0.98	0.172	3.9	0.101	1.6	0.062	3.7	425	4	1.775	-2.299	0.00288
TEM-14.1	404	196	0.50	0.00050	35	0.93	0.171	2.2	0.098	1.5	0.060	2.6	419	5	1.767	-2.327	0.00284
Wtd mean													416.8				0.00282
2 σ error													0.90%				0.97%

err: error; comm: common; corr: corrected; Wtd: weighted. Jamal et al., unpublished data.

Temora 2 zircon standard

Figure 2 (top left and right) shows the Ln(Pb/U) versus LN(UO/U) diagram of the Temora Standard analyzed for 3 days (34 spots, Table 2). There is a good correlation among the analytical points shown in this diagram. Figure 2 (bottom) presents a $^{206}\text{Pb}/^{238}\text{U}$

ages bar graph diagram indicating a weighted average value of 416.8 ± 3.8 Ma. This number is very close to that obtained by Black et al. (2004), with low error dispersion ranging from 0.9 to 95% confidence. For SHRIMP stability and calibration tests and Temora zircon homogeneity test, we analyzed 26 Temora zircon (8 standard and 18 normal sample). Figure 3A shows a

Table 3. Z6266, Temora and OG1 zircon standards - SHRIMP IIe at Institute of Geosciences of the University of São Paulo, Brazil.

Spot name	ppm U	ppm Th	²³² Th/ ²³⁸ U	ppm Rad ²⁰⁶ Pb/ ²⁰⁴ Pb	²⁰⁶ Pb/ ²⁰⁴ Pb	% err	% comm	Total ²³⁸ U/ ²⁰⁶ Pb	% Total ²⁰⁷ Pb/ ²⁰⁶ Pb	% err	²³⁸ U/ ²⁰⁶ Pb	% err	²⁰⁷ Pb/ ²⁰⁶ Pb	% err	²⁰⁷ Pb/ ²⁰⁴ Pb	% err	²⁰⁶ Pb/ ²³⁸ U	% err	²⁰⁶ Pb/ ²³⁸ U	% err	err corr	err	1σ ²⁰⁴ Pb/ ²⁰⁶ PbAge	err	1σ ²⁰⁴ Pb/ ²⁰⁶ PbAge	err	%Discor-dant
1.1	898	197	0.226	71.3	0.00000	0	0.06	10.83	1.9	0.0596	1.0	10.83	1.9	0.0596	1.0	0.7584	2.2	0.0824	1.9	0.88	569.5	10.5	587	23	3		
1.2	907	199	0.227	72.6	0.00000	0	-0.21	11.03	1.9	0.0575	1.1	10.74	1.9	0.0575	1.1	0.7587	2.2	0.0804	1.9	0.88	574.1	10.5	587	24	3		
2.1	986	219	0.229	76.8	0.00000	0	-0.10	11.03	1.9	0.0580	1.2	10.91	1.9	0.0580	1.2	0.7248	2.2	0.0804	1.9	0.88	569.6	10.3	547	26	3		
2.2	923	203	0.228	72.7	0.00000	0	-0.06	10.91	1.9	0.0584	1.2	10.91	1.9	0.0584	1.2	0.7389	2.2	0.0917	1.9	0.85	565.6	10.3	547	26	3		
2.3	952	211	0.229	76.7	0.00003	37	0.15	10.66	1.9	0.0594	1.1	11.39	1.9	0.0594	1.1	0.7622	2.2	0.0838	1.9	0.85	577.7	10.2	566	25	2		
3.1	906	201	0.230	68.4	0.00005	39	0.15	11.38	1.9	0.0596	1.1	11.39	1.9	0.0599	1.2	0.7123	2.2	0.0878	1.9	0.83	542.3	9.6	562	27	4		
3.2	907	202	0.230	69.8	0.00000	0	-0.13	11.17	1.9	0.0576	1.2	11.17	1.9	0.0576	1.2	0.7406	2.2	0.0895	1.9	0.84	562.9	9.8	543	26	7		
4.1	900	199	0.228	69.5	0.00006	26	0.19	11.13	1.8	0.0602	1.0	11.14	1.8	0.0593	1.1	0.7342	2.2	0.0897	1.8	0.85	554.0	9.8	580	25	5		
5.1	906	201	0.230	70.0	0.00005	19	0.18	11.13	1.8	0.0601	1.1	11.14	1.9	0.0594	1.1	0.7347	2.2	0.0898	1.9	0.85	564.2	9.8	580	24	5		
6.1	893	194	0.224	70.7	0.00003	20	0.02	10.85	1.8	0.0592	1.0	10.85	1.8	0.0587	1.0	0.7461	2.2	0.0921	1.8	0.87	568.2	10.0	557	23	2		
6.2	895	198	0.226	71.1	0.00000	0	-0.04	10.93	1.9	0.0586	1.1	10.93	1.9	0.0586	1.1	0.7393	2.2	0.0915	1.9	0.88	564.5	10.4	552	23	2		
7.1	966	205	0.220	74.7	0.00003	33	0.05	11.11	1.4	0.0590	0.8	11.12	1.4	0.0585	0.8	0.7262	1.6	0.0900	1.4	0.85	555.3	7.4	550	18	1		
8.1	951	200	0.217	74.1	0.00007	30	0.13	11.03	1.4	0.0600	0.7	11.04	1.4	0.0590	0.9	0.7361	1.7	0.0905	1.4	0.82	568.7	7.4	566	21	1		
8.2	1020	217	0.219	78.8	0.00003	50	0.05	11.13	1.4	0.0591	0.7	11.13	1.4	0.0587	0.8	0.7263	1.6	0.0898	1.4	0.87	554.4	7.4	554	18	0		
9.1	1083	237	0.226	87.6	0.00005	45	0.09	10.62	1.4	0.0597	0.7	10.63	1.4	0.0590	0.9	0.7654	1.6	0.0941	1.4	0.83	579.6	7.6	567	20	2		
10.1	1019	216	0.219	77.5	0.00004	25	0.07	11.30	1.4	0.0589	0.7	11.31	1.4	0.0583	0.8	0.7112	1.6	0.0884	1.4	0.88	546.1	7.2	543	17	1		
10.2	1026	219	0.220	80.6	0.00004	11	0.03	10.94	1.5	0.0593	0.7	10.95	1.5	0.0587	0.9	0.7388	2.0	0.0913	1.5	0.76	563.3	8.2	555	16	1		
11.1	1100	239	0.224	87.8	0.00002	74	0.03	10.76	1.4	0.0597	0.7	10.76	1.4	0.0591	0.7	0.7311	1.6	0.0897	1.4	0.89	572.7	7.5	570	16	3		
11.2	943	197	0.216	72.7	0.00000	0	0.00	11.14	1.4	0.0591	0.7	11.14	1.4	0.0591	0.7	0.7311	1.6	0.0897	1.4	0.89	563.9	7.5	570	16	3		
12.1	1008	213	0.219	80.6	0.00000	0	0.00	10.74	1.5	0.0593	0.7	10.74	1.5	0.0593	0.9	0.7615	2.1	0.0931	1.5	0.70	574.1	8.0	578	15	1		
13.1	1043	224	0.222	83.2	0.00000	0	0.00	10.77	1.4	0.0595	0.7	10.77	1.4	0.0595	0.9	0.7617	2.0	0.0928	1.4	0.70	572.3	7.7	586	15	2		
14.1	1009	214	0.219	80.2	0.00000	0	0.00	10.81	1.4	0.0599	0.9	10.81	1.4	0.0599	0.9	0.7637	1.6	0.0925	1.4	0.84	570.2	7.5	600	19	5		
14.2	1013	216	0.220	82.0	0.00001	66	0.01	10.61	1.4	0.0591	0.9	10.62	1.4	0.0590	0.9	0.7667	1.7	0.0942	1.4	0.83	580.3	7.9	568	21	2		
15.1	1006	214	0.219	80.7	0.00000	0	0.00	10.71	1.5	0.0592	0.8	10.71	1.5	0.0592	0.8	0.7619	1.7	0.0933	1.5	0.88	575.3	8.4	574	18	0		
15.2	1062	228	0.222	82.4	0.00003	23	0.06	11.08	1.5	0.0592	0.7	11.08	1.5	0.0588	0.7	0.7309	1.6	0.0902	1.5	0.89	556.8	7.7	558	16	0		
16.1	795	172	0.224	61.9	0.00001	60	0.01	11.03	3.0	0.0588	0.6	11.03	3.0	0.0587	0.6	0.7335	3.0	0.0906	3.0	0.98	559.3	15.8	566	13	1		
17.1	808	174	0.222	61.7	0.00000	0	0.00	11.24	2.9	0.0586	0.8	11.24	2.9	0.0586	0.8	0.7187	3.0	0.0889	2.9	0.97	549.2	15.4	553	17	1		
18.1	804	173	0.222	62.9	-0.00002	49	-0.04	10.97	2.9	0.0586	0.5	10.97	2.9	0.0589	0.6	0.7405	3.0	0.0912	2.9	0.98	562.4	15.7	564	12	0		
19.1	784	168	0.221	60.5	0.00000	240	0.01	11.12	2.9	0.0590	0.5	11.12	2.9	0.0589	0.6	0.7304	3.0	0.0899	2.9	0.98	554.9	15.5	565	12	2		
20.1	879	188	0.221	70.2	0.00000	0	0.00	10.76	1.8	0.0583	0.6	10.76	1.8	0.0583	0.6	0.7472	1.9	0.0929	1.8	0.95	572.9	9.8	541	13	6		
20.2	870	186	0.220	66.2	0.00000	0	0.00	11.29	1.8	0.0582	0.5	11.29	1.8	0.0582	0.5	0.7107	1.9	0.0885	1.8	0.96	546.9	9.5	538	12	2		
21.1	916	196	0.221	72.1	-0.00002	49	-0.03	10.91	1.8	0.0587	0.7	10.91	1.8	0.0589	0.7	0.7445	1.9	0.0917	1.8	0.93	565.6	9.8	563	15	0		
22.1	888	189	0.219	70.7	0.00000	0	0.00	10.80	1.8	0.0586	0.5	10.80	1.8	0.0586	0.5	0.7485	1.9	0.0926	1.8	0.96	570.9	9.8	553	11	3		
22.2	887	190	0.221	71.2	-0.00003	41	-0.04	10.70	1.8	0.0580	0.5	10.70	1.8	0.0591	0.6	0.7620	1.9	0.0935	1.8	0.95	576.3	9.7	571	13	1		
23.1	856	180	0.217	67.3	0.00000	0	0.00	10.92	1.8	0.0590	0.5	10.92	1.8	0.0590	0.5	0.7450	1.9	0.0916	1.8	0.96	565.0	9.7	567	12	0		
24.1	883	187	0.219	69.6	-0.00002	45	-0.04	10.91	1.8	0.0584	0.5	10.90	1.8	0.0587	0.6	0.7420	1.9	0.0917	1.8	0.95	565.7	9.6	555	12	2		
25.1	903	194	0.222	70.7	-0.00002	45	-0.04	10.98	1.8	0.0584	0.5	10.97	1.8	0.0587	0.5	0.7371	1.9	0.0911	1.8	0.96	562.2	9.8	555	12	2		
25.2	874	188	0.222	66.6	-0.00002	49	-0.03	11.27	1.8	0.0589	0.5	11.27	1.8	0.0592	0.6	0.7242	1.9	0.0887	1.8	0.95	548.1	9.6	574	13	5		
26.1	881	190	0.223	67.6	0.00008	38	0.14	11.20	1.5	0.0598	0.7	11.21	1.5	0.0587	1.1	0.7215	1.9	0.0892	1.5	0.81	550.7	8.1	555	24	1		
26.2	889	191	0.223	69.8	0.00013	24	0.23	10.93	1.5	0.0606	0.7	10.96	1.5	0.0587	1.1	0.7390	1.9	0.0913	1.5	0.80	563.0	8.2	557	25	1		
26.3	873	187	0.221	68.1	0.00012	23	0.21	11.01	1.5	0.0606	0.7	11.04	1.5	0.0589	1.0	0.7363	1.8	0.0906	1.5	0.81	559.1	7.8	565	23	1		
27.1	856	182	0.220	67.1	0.00007	38	0.13	10.97	1.6	0.0598	0.7	10.98	1.6	0.0589	1.0	0.7362	1.9	0.0911	1.6	0.85	561.8	8.6	559	22	0		
27.2	845	177	0.217	66.7	0.00003	72	0.05	10.88	1.5	0.0593	0.7	10.88	1.5	0.0589	0.9	0.7482	1.7	0.0919	1.5	0.86	566.6	7.9	564	19	0		
27.3	867	185	0.221	68.7	0.00009	27	0.15	10.84	1.5	0.0602	1.1	10.86	1.5	0.0599	1.3	0.7477	1.9	0.0921	1.5	0.75	567.8	7.9	563	28	1		
27.4	867	186	0.222	68.9	0.00010	34	0.17	10.82	1.5	0.0604	0.7	10.84	1.5	0.0590	1.1	0.7506	1.8	0.0923	1.5	0.80	569.0	7.9	567	24	0		
28.1	1053	223	0.219	82.1	0.00010	25	0.17	11.01	1.1	0.0601	1.2	11.03	1.1	0.0587	1.4	0.7337	1.8	0.0906	1.1	0.62	559.2	5.9	557	31	0		
28.2	1045	222	0.220	82.2	0.00000	206	0.01	10.92	0.9	0.0588	0.5	10.92	0.9	0.0587	0.6	0.7417	1.1	0.0916	0.9	0.85	564.8	5.1	558	13	1		
28.3	1106	239	0.223	87.3	0.00007	34	0.12	10.89	1.0	0.0598	0.8	10.90	1.0	0.0588	1.0	0.7443	1.4	0.0918	1.0	0.72	565.9	5.5	561	21	1		
29.1	1088	233	0.222	84.0	0.00003	68	0.05	11.12	1.0	0.0592	0.5	11.13	1.0	0.0588	0.7	0.7288	1.3	0.0899	1.0	0.82	554.8	5.5	560	16	1		
30.1	1113	239	0.222	86.7	0.00000	0	0.00	11.03	0.9	0.0586	0.5	11.03	0.9	0.0586	0.5	0.7265	1.1	0.									

Table 3. Continuation.

Spot name	ppm U	ppm Th	$^{232}\text{Th}/^{238}\text{U}$	ppm Rad ^{230}Pb	$^{206}\text{Pb}/^{206}\text{Pb}$	% err	% comm ^{206}Pb	Total $^{238}\text{U}/^{206}\text{Pb}$	% err	Total $^{207}\text{Pb}/^{206}\text{Pb}$	% err	$^{238}\text{U}/^{206}\text{Pb}$	% err	$^{207}\text{Pb}/^{206}\text{Pb}$	% err	$^{206}\text{Pb}/^{238}\text{U}$	% err	$^{207}\text{Pb}/^{238}\text{U}$	% err	% $^{206}\text{Pb}/^{238}\text{U}$	err	corr	$204_{206}\text{Pb}/^{238}\text{U}/\text{Age}$	1σ err	$204_{206}\text{Pb}/^{238}\text{U}/\text{Age}$	1σ err	%Discor-dant	
Temora																												
T-10.1	580	264	0.47	33.5	0.0000	0	0.00	14.91	2.4	0.0554	1.9	14.91	2.4	0.0554	1.9	0.51	3.1	0.0671	2.4	0.788	418.5	9.8	0.788	429	42	429	42	2
T-11.1	162	45	0.29	9.5	0.0002	20	0.45	14.62	2.5	0.0595	2.8	14.69	2.5	0.0558	3.4	0.52	4.2	0.0681	2.5	0.599	424.6	10.4	0.599	446	75	446	75	5
T-11.2	156	72	0.48	9.3	0.0000	0	0.00	14.44	2.6	0.0549	3.0	14.44	2.6	0.0549	3.0	0.52	4.0	0.0692	2.6	0.651	431.6	10.9	0.651	409	68	409	68	-5
T-12.1	230	83	0.38	13.3	0.0001	44	0.24	14.83	2.6	0.0571	2.4	14.87	2.6	0.0551	2.9	0.51	4.0	0.0672	2.6	0.666	419.6	10.7	0.666	417	66	417	66	-1
T-13.1	794	378	0.49	46.0	0.0000	66	0.03	14.81	2.4	0.0550	1.4	14.82	2.4	0.0547	1.5	0.51	2.8	0.0675	2.4	0.852	421.0	9.8	0.852	401	33	401	33	-5
T-13.2	352	162	0.47	20.2	0.0001	62	0.20	14.98	2.4	0.0564	2.0	15.01	2.4	0.0548	2.8	0.50	3.7	0.0666	2.4	0.664	415.7	9.8	0.664	403	62	403	62	-3
T-14.1	324	117	0.37	18.3	0.0001	5	0.21	15.18	2.5	0.0561	2.1	15.21	2.5	0.0544	2.2	0.49	3.3	0.0657	2.5	0.744	410.4	9.8	0.744	387	49	387	49	-6
T-14.2	401	140	0.36	22.8	0.0001	45	0.24	15.13	2.4	0.0567	1.6	15.16	2.5	0.0547	2.3	0.50	3.4	0.0659	2.5	0.730	411.7	9.8	0.730	402	51	402	51	-2
T-15.1	2188	1139	0.54	123.9	0.0000	26	0.06	15.17	2.4	0.0556	0.7	15.18	2.4	0.0552	0.8	0.50	2.5	0.0659	2.4	0.953	411.2	9.4	0.953	418	17	418	17	2
T-15.2	765	247	0.33	43.5	0.0000	70	0.08	15.10	2.4	0.0555	1.2	15.11	2.4	0.0548	1.5	0.50	2.8	0.0662	2.4	0.854	413.0	9.6	0.854	406	33	406	33	-2
T-16.1	175	51	0.30	10.6	-0.0001	1	-0.14	14.23	2.9	0.0549	2.4	14.21	2.9	0.0561	2.3	0.54	3.7	0.0704	2.9	0.773	438.5	12.1	0.773	454	52	454	52	4
T-17.1	324	81	0.26	19.0	0.0000	66	0.07	14.68	2.4	0.0550	1.7	14.69	2.4	0.0544	1.9	0.51	3.1	0.0681	2.4	0.793	424.7	10.1	0.793	388	42	388	42	-9
T-17.2	331	171	0.53	19.2	0.0002	45	0.32	14.84	2.5	0.0574	1.7	14.89	2.5	0.0548	2.9	0.51	3.8	0.0672	2.5	0.654	419.1	10.1	0.654	405	64	405	64	-3
T-18.1	449	118	0.27	25.7	0.0001	23	0.07	15.04	2.5	0.0571	1.5	15.08	2.5	0.0549	1.9	0.50	3.1	0.0663	2.5	0.801	413.8	10.0	0.801	409	42	409	42	-1
T-19.1	289	145	0.52	16.4	0.0003	45	0.47	15.16	2.7	0.0590	1.9	15.24	2.7	0.0552	3.8	0.50	4.7	0.0656	2.7	0.579	409.8	10.8	0.579	420	85	420	85	3
T-20.1	393	195	0.51	23.2	0.0000	68	0.07	14.52	2.4	0.0561	1.6	14.53	2.4	0.0556	1.7	0.53	3.0	0.0688	2.4	0.817	429.1	10.1	0.817	435	38	435	38	1
T-21.1	308	124	0.42	17.1	0.0000	66	0.07	15.45	2.5	0.0549	1.7	15.46	2.5	0.0543	1.9	0.48	3.1	0.0647	2.5	0.798	404.0	9.7	0.798	383	42	383	42	-5
T-21.2	334	137	0.42	18.5	0.0001	74	0.09	15.51	2.4	0.0566	1.6	15.52	2.4	0.0558	1.9	0.50	3.1	0.0644	2.4	0.786	402.5	9.5	0.786	446	43	446	43	11
OG1																												
OG1-1.1	141	119	0.88	80.5	0.00006	33	0.11	1.5	5.1	0.297	0.5	1.5	5.1	0.296	0.5	27.12	5.1	0.665	5.1	0.99	3285	130	0.99	3449	8	3449	8	5
OG1-2.1	148	119	0.83	87.5	0.00046	46	0.87	1.45	5.1	0.296	0.5	1.47	5.2	0.292	1.0	27.41	5.3	0.682	5.2	0.98	3350	135	0.98	3427	15	3427	15	2
OG1-3.1	231	178	0.8	135.6	0.00006	35	0.12	1.46	5.1	0.297	0.6	1.46	5.1	0.296	0.6	27.89	5.1	0.683	5.1	0.99	3356	132	0.99	3450	9	3450	9	3
OG1-4.2	61	32	0.53	37.1	0.00016	49	0.3	1.42	5.1	0.304	0.9	1.42	5.2	0.302	1.0	29.24	5.2	0.702	5.2	0.98	3430	137	0.98	3480	15	3480	15	1
OG1-5.1	132	130	1.02	81.2	0.00011	26	0.21	1.39	5.1	0.300	0.5	1.4	5.1	0.299	0.5	29.51	5.1	0.716	5.1	0.99	3482	136	0.99	3464	8	3464	8	-1
OG1-5.2	241	186	0.8	139.5	0.00014	20	0.26	1.49	5.4	0.300	0.4	1.49	5.4	0.299	0.4	27.65	5.4	0.671	5.4	1.00	3310	139	1.00	3464	6	3464	6	5
OG1-5.3	134	108	0.83	75.6	0.00007	34	0.14	1.52	5.1	0.301	0.5	1.52	5.1	0.300	0.5	27.19	5.1	0.657	5.1	1.00	3254	130	1.00	3472	8	3472	8	7
OG1-6.1	120	61	0.53	77.7	0.00017	19	0.31	1.32	5.2	0.300	0.7	1.33	5.2	0.298	0.7	31.01	5.3	0.754	5.2	0.99	3623	145	0.99	3461	10	3461	10	-4
OG1-6.2	128	72	0.58	80.6	0.00014	26	0.26	1.37	6.6	0.299	0.5	1.37	6.6	0.297	0.5	29.92	6.6	0.730	6.6	1.00	3533	179	1.00	3456	8	3456	8	-2
OG1-6.3	135	74	0.56	85.2	0.00016	27	0.3	1.36	5.1	0.302	0.7	1.37	5.1	0.300	0.7	30.24	5.2	0.731	5.1	0.99	3537	140	0.99	3470	11	3470	11	-2
OG1-7.1	153	142	0.95	104.6	0.00008	32	0.16	1.26	5.1	0.299	0.5	1.26	5.1	0.298	0.5	32.57	5.1	0.793	5.1	1.00	3764	145	1.00	3459	8	3459	8	-8
OG1-7.2	187	134	0.74	121.9	0.00006	27	0.12	1.32	5.2	0.298	0.7	1.32	5.2	0.297	0.7	31.06	5.2	0.758	5.2	0.99	3637	143	0.99	3455	11	3455	11	-5
OG1-8.1	235	222	0.98	158.7	0.00011	36	0.2	1.27	5	0.302	0.4	1.27	5	0.301	0.4	32.58	5.1	0.786	5	1.00	3737	143	1.00	3474	6	3474	6	-7
OG1-8.2	205	183	0.92	124.3	0.00006	24	0.12	1.42	5	0.298	0.4	1.42	5	0.298	0.4	28.88	5.1	0.704	5	1.00	3436	134	1.00	3458	6	3458	6	1
OG1-8.3	219	194	0.92	144.1	0.00006	43	0.12	1.3	5.1	0.301	0.4	1.31	5.1	0.300	0.4	31.69	5.1	0.766	5.1	1.00	3666	141	1.00	3471	6	3471	6	-5
OG1-9.1	132	130	1.01	86	0.00016	22	0.31	1.32	5.3	0.299	0.6	1.33	5.3	0.298	0.6	30.92	5.3	0.753	5.3	0.99	3619	146	0.99	3459	10	3459	10	-4

r: radiogenic; err: error; corr: corrected; comm: common; -: not detected, strike-slip cell correspond discordance > 5% (not included for Concordia age).

Concordia diagram with age of 416.7 ± 2.3 Ma and Figures 3B and 3C shows Concordia diagram with U (ppm, color scale) and Concordia diagram with common Pb (color scale). Figure 3D shows a histogram (left) and the respective bar graph (right) of $^{206}\text{Pb}/^{238}\text{U}$ age of Temora 2 analyzed it as an unknown sample. The analytical location points can be seen at CL image (Appendix 5) and the analytical data is in the Table 3. The Concordia age of 416.7 ± 2.3 Ma with low mean square weighted deviation (MSWD) = 0.103 and the $^{206}\text{Pb}/^{238}\text{U}$ weighted average age of 417.0 ± 4.7 Ma with an equivalent error of 1.1% are very close to the true value. The precision and error are dependents of U concentration and common Pb content (Figures 3B and 3C). The histogram (Figure 3D) shows a unimodal Gaussian distribution. The difference of $^{206}\text{Pb}/^{238}\text{U}$ ages among Figure 2 (bar graph) and Figure 3D is in the analytical period. Figure 2 shows $^{206}\text{Pb}/^{238}\text{U}$ ages obtained during 3 consecutive days, demonstrating a good stability for long period (416.8 ± 3.8 Ma – Figure 2) with an

equivalent error of 0.9%, while the $^{206}\text{Pb}/^{238}\text{U}$ average age to half day is 417.0 ± 4.7 Ma (Figure 3D) with an error of 1.1%. For small number of data (10 – 15 analysis), the error expected for $^{206}\text{Pb}/^{238}\text{U}$ Temora age is < 1.5%; therefore, our result can be considered of good quality.

Z6266 zircon sample

The Z6266 zircon (CL image, see Appendix 6) yielded U concentrations for 53 spots, ranging from 804 to 1,113 ppm (mean = 947 ± 100), $^{232}\text{Th}/^{238}\text{U}$ ratios from 0.217 to 0.230 (mean = 0.222 ± 0.002) and total radiogenic ^{206}Pb contents from 60 to 87 ppm (mean = 74 ± 7). The analytical data is seen in Table 3. In the Concordia diagram (Figure 4A), the analytical points defined a U-Pb age of 561.9 ± 1.0 Ma with low MSWD of 0.14. This age is very close to that obtained by Stern and Amelin (2003). $^{206}\text{Pb}/^{238}\text{U}$ (Figure 4B) and $^{207}\text{Pb}/^{206}\text{Pb}$ (Figure 4C) weighted

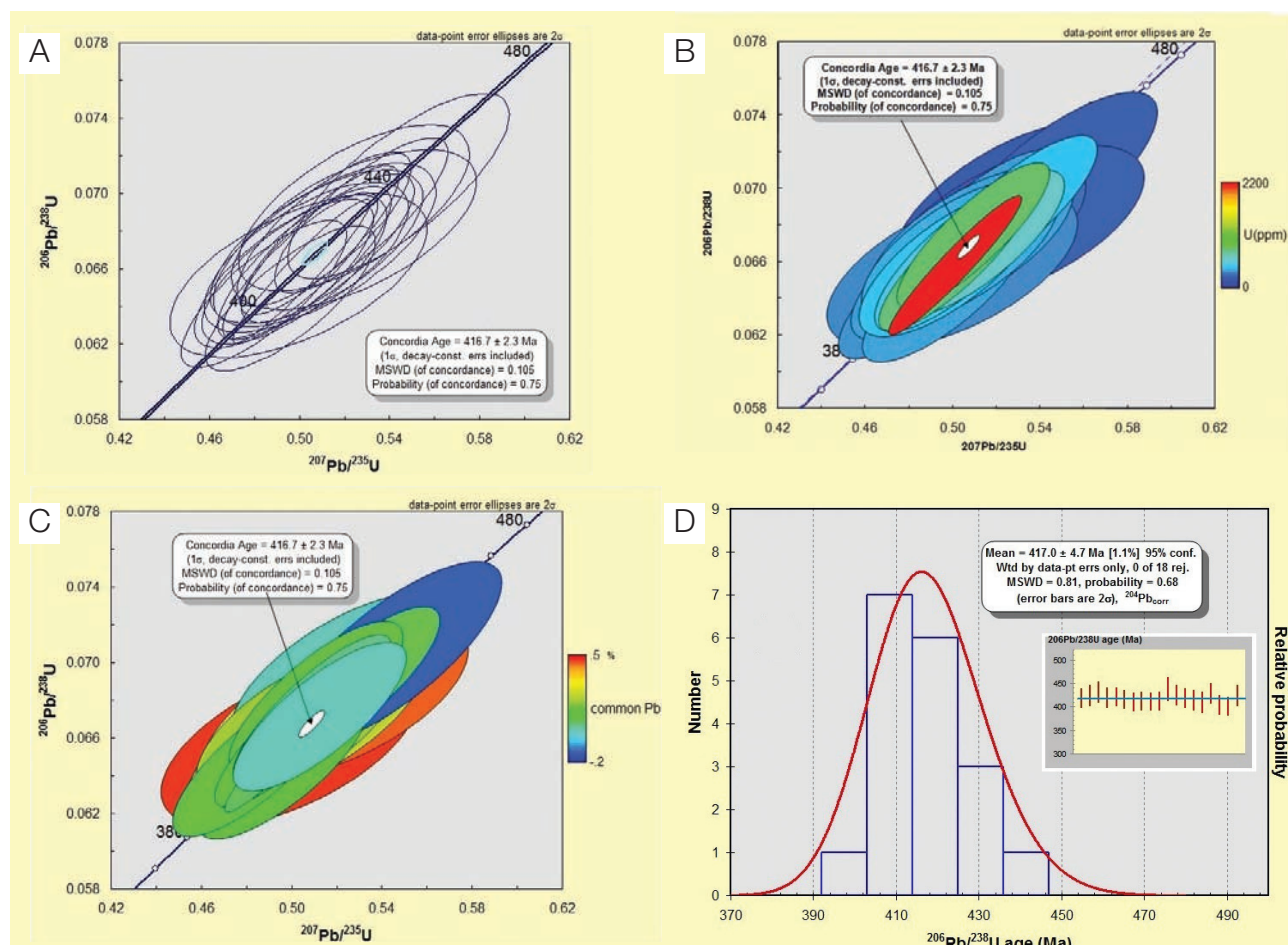


Figure 3. Temora analyzed as unknown sample. (A) Concordia diagram with age of 416.7 ± 2.3 Ma; (B) Concordia diagram with U (ppm – color scale) where the precision is directly proportional to the U concentration, but is dependent of common Pb; (C) Concordia diagram with common Pb (error in %, is directly proportional to the common Pb – color scale); (D) Histogram of $^{206}\text{Pb}/^{238}\text{U}$ age (Ma) and analytical distribution (bar graph) of $^{206}\text{Pb}/^{238}\text{U}$ age (Ma). (For data, see Table 3.)

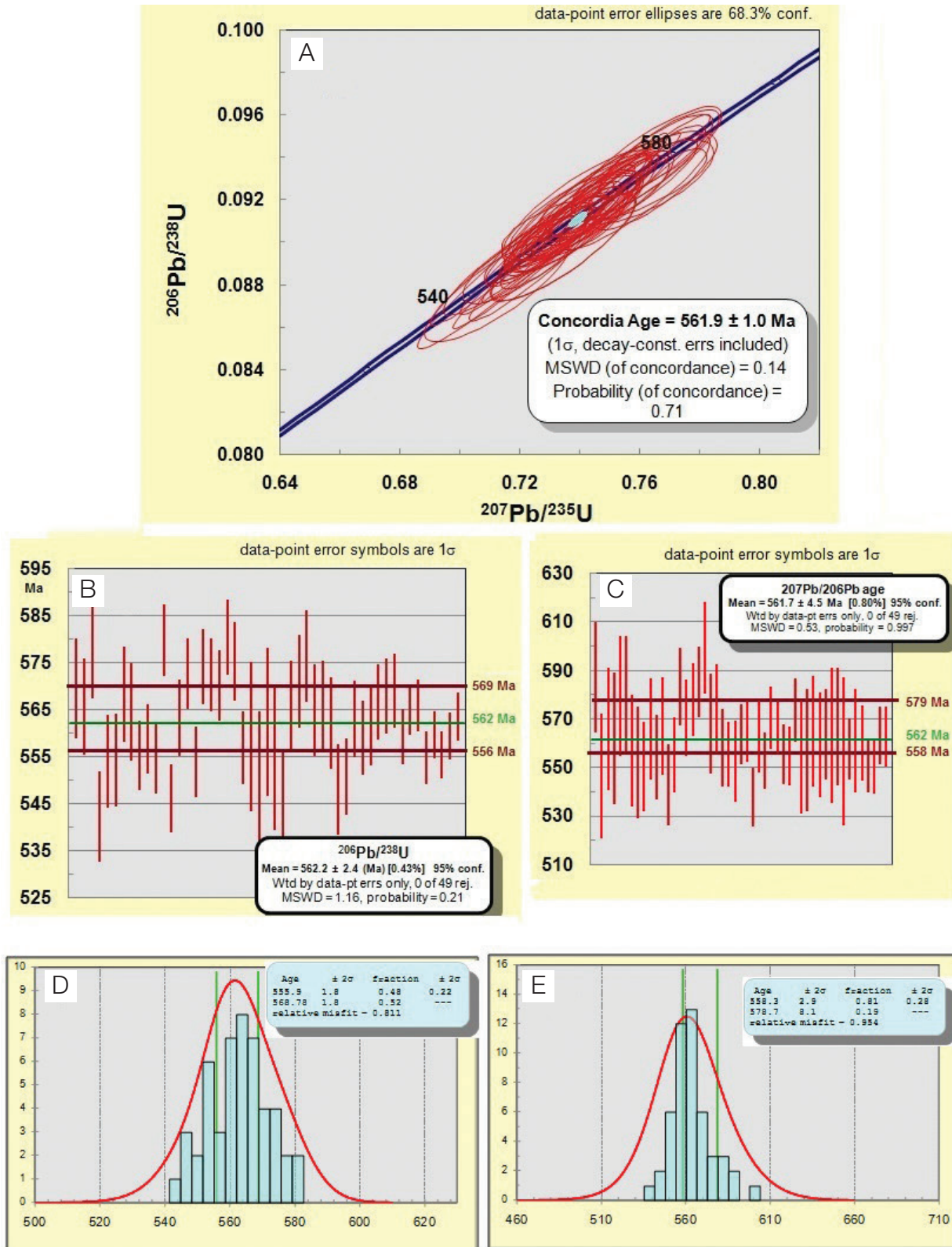


Figure 4. As shown in the Concordia diagram (A), Z6266 sample yielded a Concordia age of 561.9 ± 1.0 Ma, which is in agreement with the ages presented by the Geological Survey of Canada (Amelin and Stern, 2003). (B) and (C) show a $^{206}\text{Pb}/^{238}\text{U}$ and $^{207}\text{Pb}/^{206}\text{Pb}$ weighted average ages of 562.2 ± 2.3 Ma and 567.7 ± 4.5 , respectively. The PDP of $^{206}\text{Pb}/^{238}\text{U}$ and $^{207}\text{Pb}/^{206}$ ages shows a histogram illustrating a unimodal Gaussian distribution (D and E). (For data, see Table 3.)

average ages are 562.2 ± 2.4 Ma and 561.7 ± 4.5 Ma respectively; therefore, these values are very close for Concordia age. The probability density plot (PDP, Figure 4D) of $^{206}\text{Pb}/^{238}\text{U}$ and $^{207}\text{Pb}/^{206}\text{Pb}$ ages shows an unimodal Gaussian distribution. If we considered a possibility of 2 events, then the ages for $^{206}\text{Pb}/^{238}\text{U}$ using unmixing of superimposed Gaussian distributions for 2 components, these ages are 556 and 569 Ma (Figures 4B and 4D) and the $^{207}\text{Pb}/^{206}\text{Pb}$ ages for 2 components are 558 and 579 Ma (Figures 4C and 4E). In the present moment, due to unimodal Gaussian distribution and uniform CL crystal images, we considered the Concordia value of 561 Ma as the correct age. We consider the age variation observed in the Z6266 standard as a small heterogeneity. As we can observe, the same problem occurs in all other standards, which indicates that until today perfect natural zircon standard was not yet found.

OG1 zircon sample

Zircons from OG1 Archean sample (CL image, see Appendix 7) were analyzed by SHRIMP IIe and yielded a U-Pb age (Figure 5A) of 3462.6 ± 5.1 , as shown in the Concordia diagram. The bar graph showed a weighted average $^{207}\text{Pb}/^{206}\text{Pb}$ age of 3462.6 ± 5.1 Ma (Figure 5B and Table 3), which is very close to U-Pb zircon age of 3465.4 ± 0.6 Ma obtained by TIMS (Stern et al., 2009).

EXAMPLES OF SHRIMP IIE U-Pb ZIRCON AGES FROM SOUTH AMERICAN SAMPLES ANALYZED AT GeoLab-IGc-USP

To illustrate the precision of the ages obtained in terms of MSWD values and age errors, samples with different ages along the

geological timeline were selected. Approximately hundreds samples were analyzed at GeoLab-IGc-USP using SHRIMP IIe. Out of these, seven samples are presented, as follows:

- Cenozoic zircon (Henrichs et al., 2012): the zircon sample was collected from Miocene subvolcanic intrusive rocks of the Yarumalito porphyry gold deposit, located at the Marmato Mining district, Colombia. The 16 YARSV zircon grains yielded a $^{238}\text{U}/^{206}\text{Pb}$ weighted mean age of 7.0 ± 0.15 Ma with a MSWD of 1.08. Seventeen U-Pb zircon analyses from the YARNV sample yielded a $^{238}\text{U}/^{206}\text{Pb}$ weighted mean age of 6.95 ± 0.16 Ma, with a MSWD of 1.07;
- Cretaceous zircon (Basei et al., 2012): zircon samples from the Juína kimberlitic Province showed a single population with a $^{206}\text{Pb}/^{238}\text{U}$ mean age of 92.1 ± 2.4 Ma and MSWD = 0.087;
- Cambrian zircon (Janasi et al., 2012): zircon samples from the Santos and Guarujá granite plutons, shown by regional geologic mapping to be post-orogenic, analyzed by SHRIMP IIe, yielded Concordia ages of 497.3 ± 3.5 Ma (MSWD = 0.035) and 496.9 ± 4.3 Ma (MSWD = 0.033), respectively;
- Neoproterozoic zircon (Araujo et al., 2012): zircon samples from the Tamboril-Santa Quitéria Complex, NE Brazil, analyzed by SHIRMP IIe, yielded Concordia ages ranging from 889 ± 9.2 Ma (orthogneiss, MSWD = 0.004) to 618 ± 4.1 (diatexite, MSWD = 0.37);
- Mesoproterozoic zircon (Payolla et al., 2012): the Alto Candeias Intrusive Suite has been interpreted as a product of inboard magmatism related to the Rondonian-San Ignacio orogeny. The analyses by SHRIMP IIe in the oscillatory-zoned cores yielded concordant ages of 1349 ± 9.4 Ma ($n = 7$, MSWD = 1.9) and 1330 ± 12 Ma ($n = 6$, MSWD = 2.4) for samples WB-731 and WB-305, respectively;

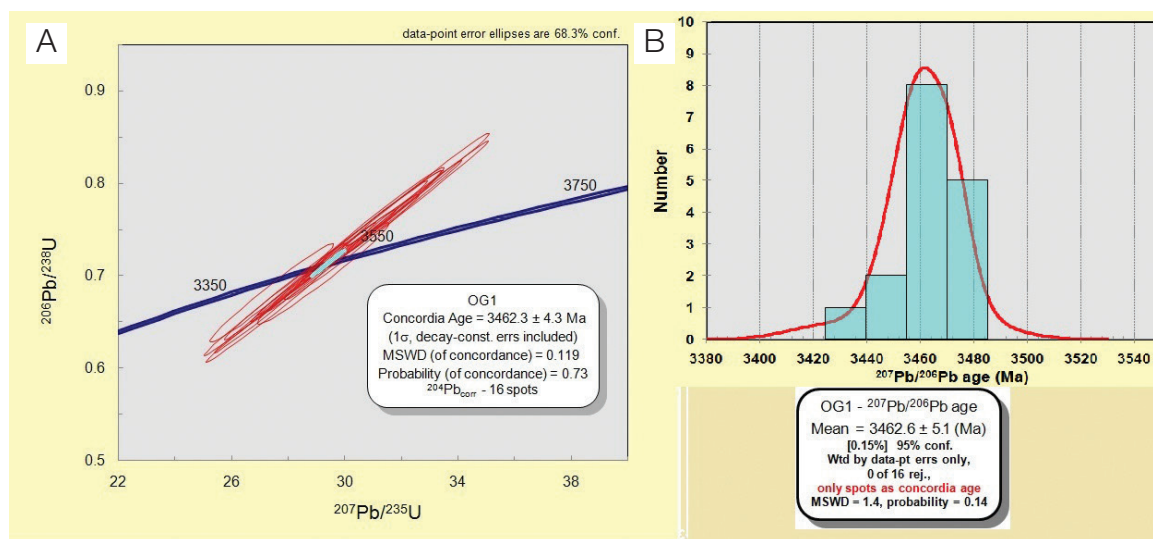


Figure 5. OG1 sample/standard - The Concordia diagram (A) shows a Concordia age of 3462.3 ± 4.3 . The $^{207}\text{Pb}/^{206}\text{Pb}$ histogram (B) shows a weighted average age of 3462.6 ± 5.1 Ma, which is very close to the data by Stern et al. (2009).

- (f) Paleoproterozoic zircon (Sato et al., 2012): sample 2 BBV 40 was collected from the quartzite gneiss located near the Barra Velha region, Santa Catarina State. Among the detrital zircons, two different populations can be identified by their transparent and dark colors (CL images). Eighty five (85) out of one hundred and nine (109) spots are concordant and yielded a Concordia age of 2193 ± 5.7 Ma (MSWD = 0.0018). $^{207}\text{Pb}/^{206}\text{Pb}$ weighted average age is 2191.9 ± 2.5 Ma (mean of 102 spots, error = 0.11%, MSWD = 1.1), which is, therefore, very close to the Concordia age. These ages are very close to the Concordia age of 2182 ± 9 Ma dated by U-Pb TIMS method, and are not very different from Concordia age of 2199 ± 16 Ma dated by the LA-ICP-MS method;
- (g) Archean zircon (Moreto et al., 2012): in the Carajás Domain, the iron, copper and gold oxide deposits from Sossego and Castanha are located in the South Belt, along a regional shear zone trending WNW-ESE, close to the contact between the Carajás basins and basement rocks. The Sossego gabbro-norite and the Castanha porphyry of rhyolitic and dacitic composition were dated by SHRIMP IIe and yielded zircon U-Pb ages of 2739 ± 6 Ma (MSWD = 1.4) and 2745 ± 4 Ma (MSWD = 0.18), respectively.

The examples above show ages from Cenozoic to Archean and do not include Figures and Tables, since the final papers are still being prepared. Dating of young zircons is critical for Cenozoic samples due to the low amount of radiogenic Pb. Until now, the youngest $^{206}\text{Pb}/^{238}\text{U}$ age of zircons is 7 Ma, with an error smaller than 0.2 Ma.

U-Pb DATING FOR OTHERS MINERALS (TITANITE, XENOTIME AND MONAZITE) IN THE NEAR FUTURE

Many tests were already done for dating other minerals, as titanite, xenotime and monazite. U-Pb dating for titanite mineral using Khan and BLR1 titanite standards was tested with success (Sato et al., 2014 in the 9th South American Symposium on Isotope Geology). We also tested with success the dating U-Pb in xenotime using MG standard and monazite using 44069 standard (from Pennsylvania). For near future (2015), we have project to buy cesium gum source to analyze oxygen isotopes in zircon (stable isotopes).

CONCLUSION

The Sensitive High Resolution Ion Microprobe is used at the laboratory of São Paulo University (GeoLab-IGc-USP) to measure U, Pb and Th abundances, and isotopic relationships of these elements in zircon crystals. The spectrometer operates

24 hours per day, obtaining accurate data, which was approved to be published in internationally renowned magazines. The analytical quality of data obtained by SHRIMP IIe at the GeoLab-IGc-USP is of the same standard when compared to the data from leading laboratories, which was confirmed by an Inter-Laboratory Comparison (ILC) conducted in the 6th International SHRIMP Workshop, 2012, Brisbane, Australia. Four samples with unknown ages (Archean, Paleoproterozoic, Paleozoic and Cenozoic) were distributed to 10 international laboratories, and ILC participants anonymously presented the results in this workshop, which were very similar and vary within the experimental error. The Temora 2 zircon standard (416.78 Ma, Black et al., 2004) analyzed for a continuous period of 72 hours by SHRIMP IIe at the IG-USP yielded an age of 416.8 ± 3.8 Ma with a corresponding error of 0.9%. The OG1 Archean zircon samples (TIMS ages of 3465.4 ± 0.6 Ma, Stern et al., 2009) and the Z6266 Neoproterozoic zircon ($^{206}\text{Pb}/^{238}\text{U}$ age of 559 ± 0.2 Ma – TIMS analysis, Stern and Amelin, 2003) analyzed by SHRIMP IIe at the IG-USP yielded concordant ages of 3462.6 ± 5.1 Ma and 561.9 ± 1.0 Ma, respectively, which are, therefore, very close to values found in the literature. Since concentrations of U, Th and Pb are similar for Z6266 zircon, and $^{207}\text{Pb}/^{206}\text{Pb}$ and $^{206}\text{Pb}/^{238}\text{U}$ ratios are relatively constant for OG1 zircon crystals, these crystals can be used as standard for SHRIMP analysis. The precision of the analytical data for U, Pb and Th in zircons from samples collected in Brazil and neighboring countries is compatible with international standards.

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APPENDIX 1

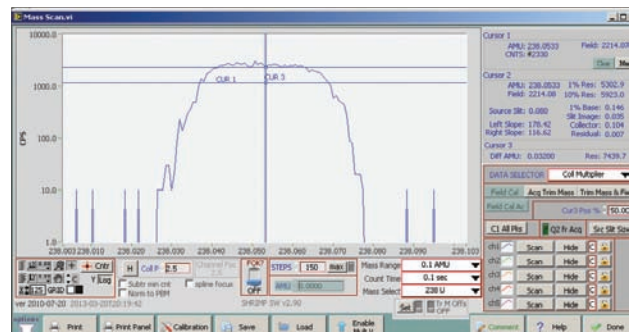
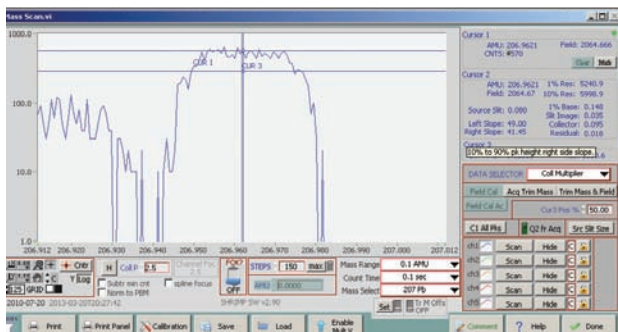
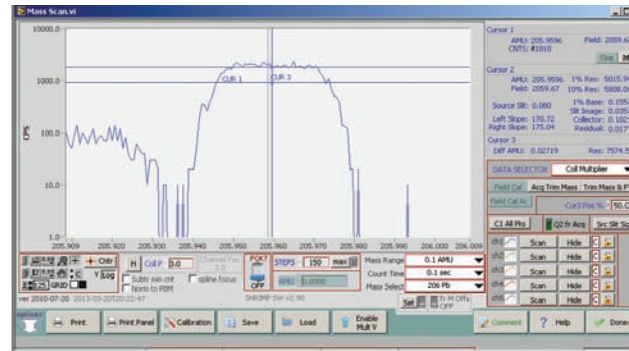
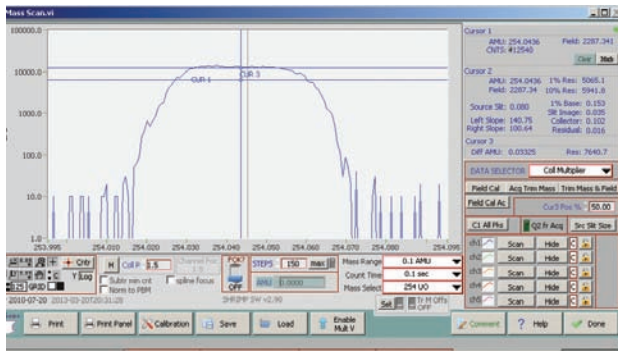
Sample preparation and hand picking

Zircon grains were extracted by standard crushing, milling, sieving (0.150–0.063 mm), Wiffley table, and heavy liquid techniques (bromoform - density = 2.85 and metilene - density = 3.32). Five to seven fractions (magnetic to nonmagnetic) were separated using the Frantz magnetic barrier separator and after removing ferromagnetic minerals by a hand magnet. Normally, the ideal fraction for igneous zircon is non magnetic.

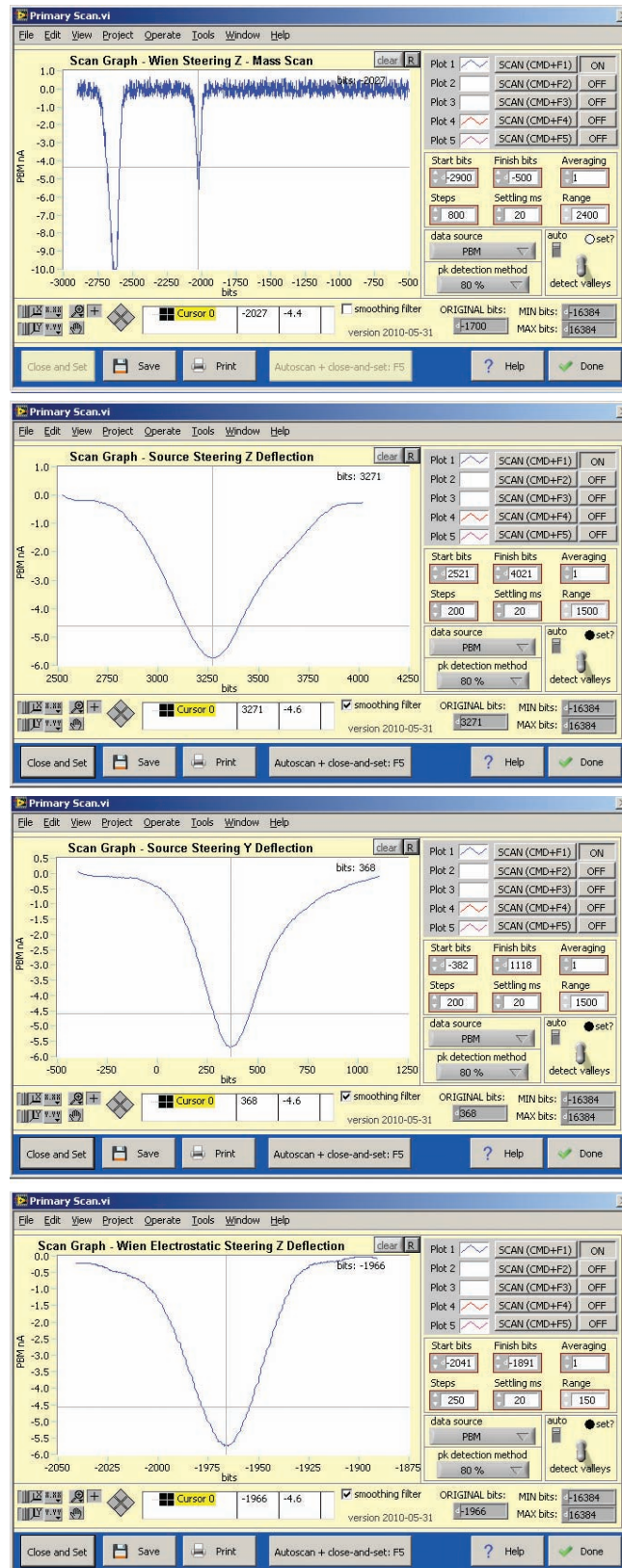
For detrital zircon (Sircombe and Stern, 2002), taking the nonmagnetic fraction at a Frantz setting of 1.8 A 10° side-slope, or a mass magnetic susceptibility of 4.72×10^{-7} e.m.u./g (5.93×10^{-9} m³/kg) is a reasonable compromise between representation and analytical efficiency. The handpicking process in detrital zircon

geochronology attempts to ensure the most-representative sampling possible within the limitations imposed by handling small-sized samples. No preferential selection of zircon was made based on size, color, shape, roundness and metamictization. This aim is best achieved by minimizing individual grain handling and hand picking only to ensure purity of zircon content as a group rather than the quality of individual grains. Extracted zircon grains were poured into a mound under alcohol in a Petri dish. This mound was directly mixed and then a swathe swept from the middle. Non-zircon grains were removed from this swathe, and the remaining zircon grains, typically numbering 100 to 150 grains, were taken from the dish using a pipette. Zircon grains were mounted on double-sided adhesive tape before being set in an epoxy disk and polished to reveal half-sections. Zircon internal structures were analyzed.

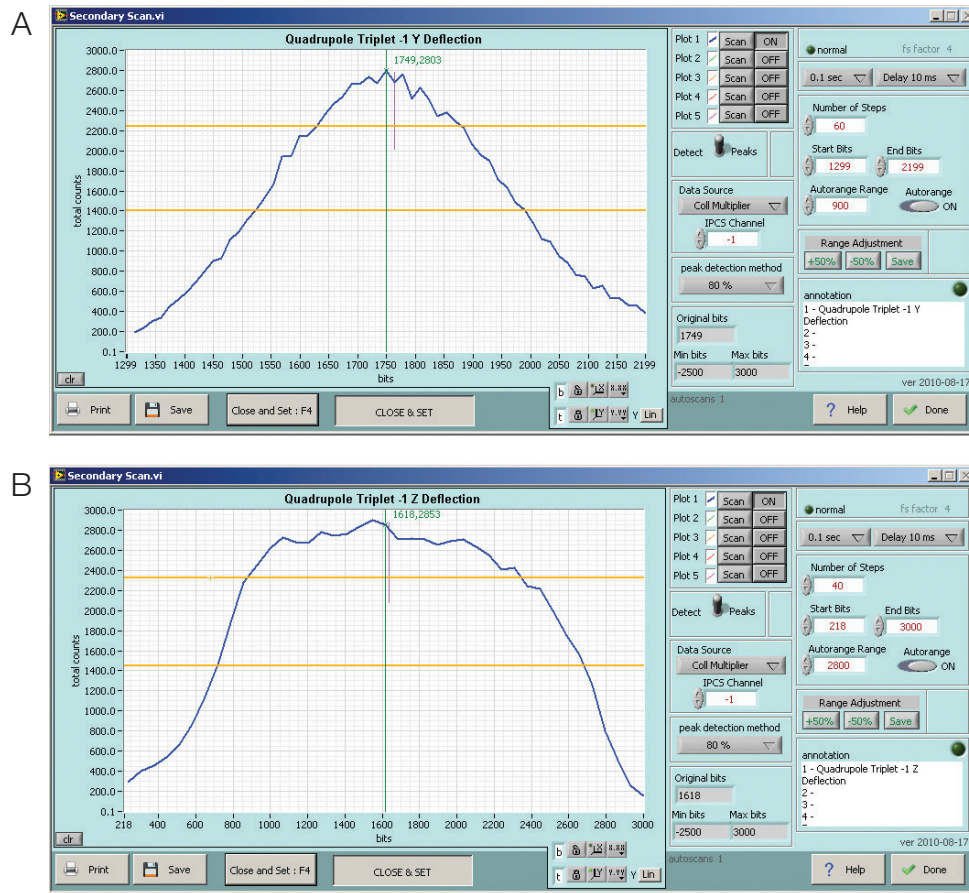
Appendix 2. Secondary beam mass scan: [top (left)] ²⁵⁴U mass scan with resolution of 5065 at 1%; [top (right)] ²⁰⁶Pb mass scan with resolution of 5016 at 1%; [bottom (left)] ²⁰⁷Pb mass scan with resolution of 5200 at 1%; [bottom (right)] ²³⁸U mass scan with resolution of 5300 at 1%.



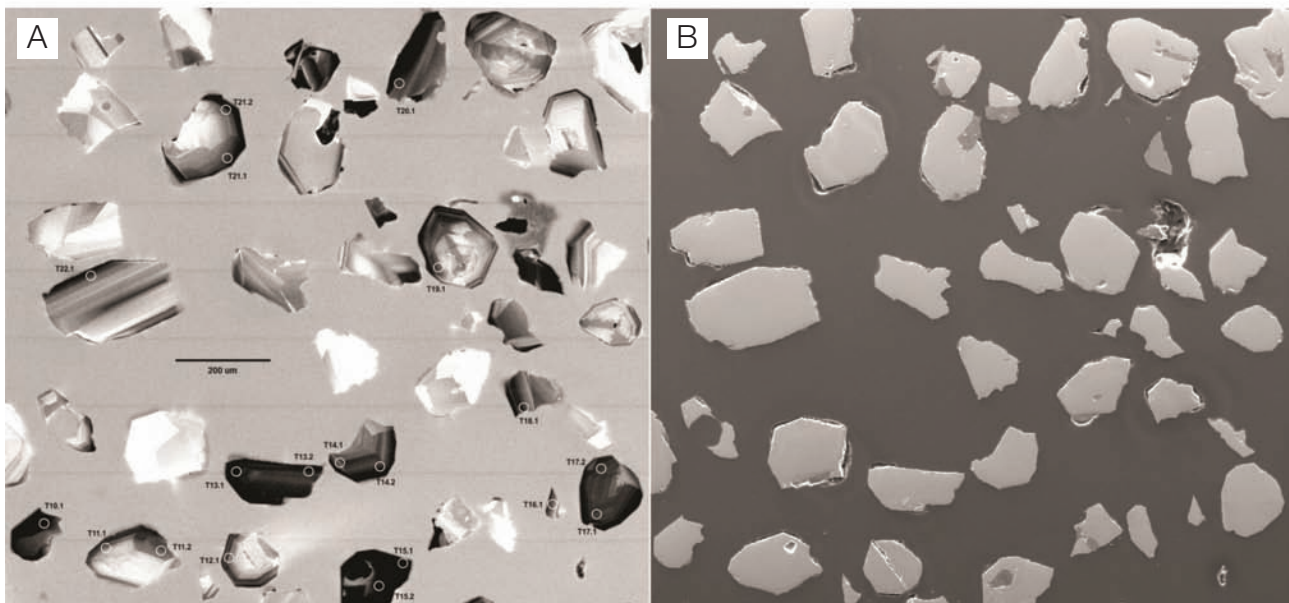
Appendix 3. Primary beam calibration: [top (left)] mass scan of oxygen negative ions O^- (10 hA) and O_2^- (5 hA); [top (right)] Source Steering Z deflection; [bottom (left)] Source Steering Y deflection; [bottom (right)] Wien electrostatic steering Z deflection. Scan done at 2013 April.



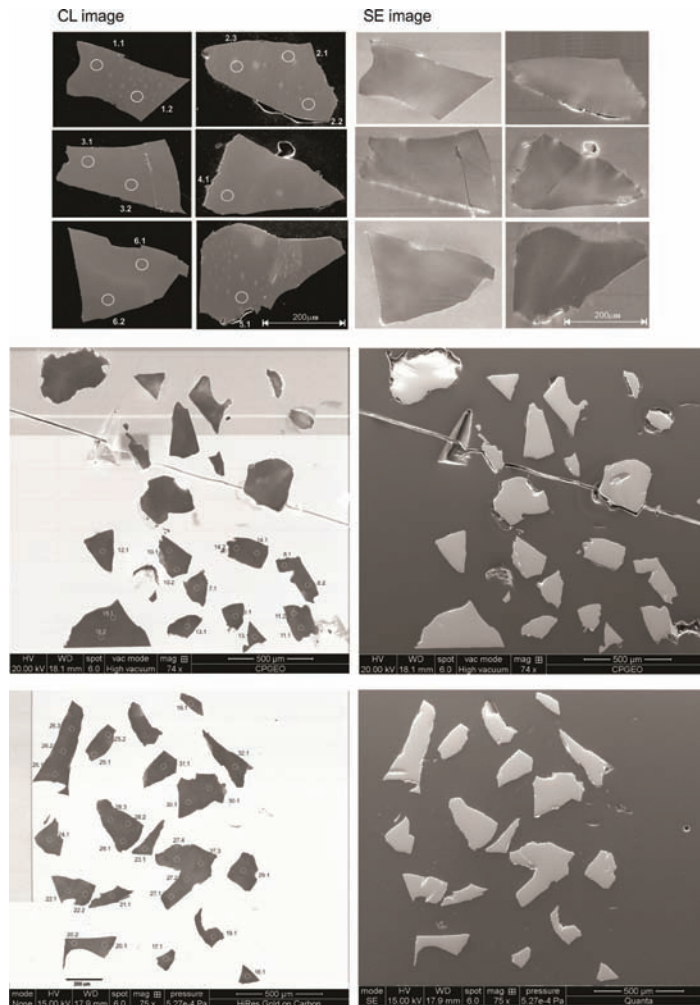
Appendix 4. Secondary beam – focus alibration: (A) quadrupole triplet 1 Y deflection; (B) quadrupole 1 Z deflection.



Appendix 5. Temora 2. (A) Cathodoluminescence image (CL); (B) secondary electron image (SE). The numbers at CL are spot points.



Appendix 6. Cathodeluminescence (CL) image of Z6266 zircon glassy fragments obtained from a single Sri Lankan megacryst, Australia. In fragmented form, it appears clear and colorless with no observable internal heterogeneities, and in the CL no detectable variations were observed.



Appendix 7. Cathodeluminescence (CL) image from OG1 zircon crystal from Owens Gully diorite of Pilbara Craton, Western Australia. These crystals are from SHIMP IIe setup mount. They were generally 100 – 300 μm in largest dimension, stubby prismatic, to sub equant grains dominated by pyramidal faces. CL images typically revealed simple banding parallel to crystallographic c-axis, or sector zoning in equant grains (Stern et al., 2009).

