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NOTE

An improved U-Pb age dating method for detrital zircon by LA-MC-ICP-MS

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Detrital zircon U-Pb dating is a powerful tool to trace sources for sedimentary basin and to understand of geological history in structurally complex areas. This study established an analytical protocol for measurement of detrital zircon U-Pb age by LA-MC-ICP-MS. Because detrital zircons normally have a large Pb content variation (several ppm to hundred ppm), which leads to large variations for Pb signal intensity, two sets of detector arrays were used. For Pb-rich zircons, ^{206}Pb was collected with Faraday cup detector array, while for Pb-poor zircons, ^{206}Pb was collected with ion counter detector array. The results of ^{206}Pb/^{238}U age and ^{207}Pb/^{235}U age for six well-known and widely used zircon standards agree within 0.5% and 1% respectively of the preferred values measured by ID-TIMS. The precision of ^{206}Pb/^{238}U age and ^{207}Pb/^{235}U age of the six zircons vary from 0.6% to 2.3% and 0.9% to 2.3% (2SD), respectively.

Keywords: detrital zircon, laser ablation, MC-ICP-MS, U-Pb age, ^{206}Pb/^{238}U

INTRODUCTION

Zircon, a widely distributed accessory mineral in various types of rocks, is the most popular mineral for U-Pb age dating due to: 1) U is compatible to zircon crystal lattice, whereas Pb is much incompatible which leads to ignorable common Pb in zircon; 2) The stable crystal structure makes zircon resist weathering and low degree metamorphism so that the U-Pb isotope system remains close after formation. Detrital zircon, selected from sedimentary rocks, is a powerful tool to trace source of sedimentary rocks and study tectonic geodynamics. In order to have a meaningful result on statistics, abundant zircons (normally about 100 zircons per sample) are needed to analyze. Therefore, although detrital zircon U-Pb age can be measured on several kinds of mass spectrometry, such as thermal ionization mass spectrometry (TIMS), Secondary ion mass spectrometry (SIMS) and laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS), LA-ICP-MS, with the highest efficiency among these techniques, is the most widely used method to perform detrital zircon U-Pb age measurement. With laser ablation, a single-collector mass spectrometry (LA-ICP-MS) or a multi-collector mass spectrometry (LA-MC-ICP-MS) can be used. Due to its high sensitivity, flat top of mass peak and the character of simultaneous collection, LA-MC-ICP-MS has a higher precision than LA-ICP-MS (e.g., Gehrels *et al*., 2008; Jackson *et al*., 2004; Xie *et al*., 2017). Willigers *et al*. (2002) first established in-situ Pb-Pb dating method for several accessory minerals by LA-MC-ICP-MS with all mass signals but ^{204}Pb were detected by Faraday cups. Because most zircons have Pb content one order less than U content, the following zircon U-Pb dating studies by LA-MC-ICP-MS used a Faraday cup-ion counter detector array that the signal of ^{238}U was collected by Faraday cup, whereas all lead isotopes were detected by ion counters (e.g., Gehrels *et al*., 2008; Bühn *et al*., 2009). To enhance the instrumental sensitivity, some study collected signals of all U and Pb isotopes by Faraday Cups equipped with 10^{12} Ω resistor instead of 10^{11} ohm resistor (Kimura *et al*., 2015). Recently, a detector array making up totally of ion counters for zircon U-Pb dating by LA-MC-ICP-MS has been developed (Hattori *et al*., 2017; Xie *et al*., 2017).

Here, rather than attempting to improve analytical precisions, this study focuses on developing an accurate and efficient procedure for detrital zircons U-Pb age measurement by LA-MC-ICP-MS. Detrital zircons selected from a sedimentary rock have much large Pb content variation which leads to a large challenge for detectors to

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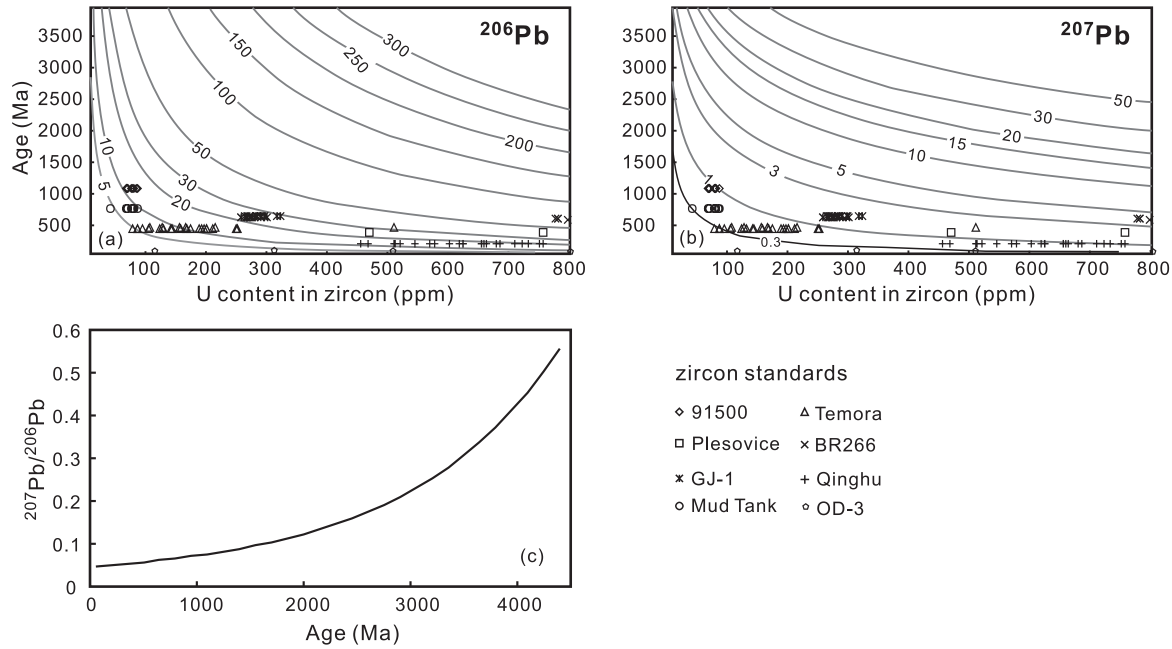

Fig. 1. The theoretical relationship between ^{206}Pb (a), ^{207}Pb (b) content and U content, formation age in zircon, which are calculated from the following equations: ^{206}Pb content = $[exp(\lambda_1 * t) - 1] * U_c/U_{aw} * 137.88/138.88 * {}^{206}Pb_{aw}$; ^{207}Pb content = $[exp(\lambda_2 * t) - 1] * U_c/U_{aw}/138.88 * {}^{207}Pb_{aw}$; λ_1 and λ_2 are the decay constants of ^{238}U and ^{235}U, respectively. U_{aw}, $^{206}Pb_{aw}$ and $^{207}Pb_{aw}$ are the atomic weights of U, ^{206}Pb and ^{207}Pb, respectively. Numbers on the curves represent ^{206}Pb or ^{207}Pb content in ppm. Several reference zircons are also plotted. The U contents and formation ages of these zircons are from Yuan et al. (2008), Li et al. (2013) and Iwano et al. (2013). c is the theoretical relationship between $^{207}Pb/^{206}Pb$ and formation age in zircon.

collect Pb signals. It is difficult for previous studies, either used Faraday cups or ion counters to collect Pb isotope signals, to measured zircons with Pb content varying from several ppm to more than one hundred ppm. This is because ion counter has a much higher sensitivity than Faraday cup, but its linearity range is limited to small signal intensities (e.g., <~20 mV) and large signals may cause ion counter self-protection and even damage. On the contrary, Faraday cup can measure much larger signal, but it has large uncertainty when measuring small signals. Thus, due to the large variation of Pb content in detrital zircons, only Faraday cup or ion counter may not be available to carry out the whole analysis. In this study, with Faraday cup and ion counter, two sets of collector arrays are established. The main difference of the two collector arrays is whether ^{206}Pb detected with Faraday cup or ion counter. To establish our analytical procedure, the two sets of collector arrays were applied to six well-known reference zircons first, whose ages were well confirmed by ID-TIMS.

Experimental

^{206}Pb and ^{207}Pb contents in zircon

For zircon U-Pb age measurement by LA-MC-ICP-MS, at least the signals of ^{206}Pb, ^{207}Pb and ^{238}U should be detected and these signal intensities determine which kind of detectors (Faraday cup or ion counter) to be used. Because zircon is a uranium rich mineral (most more than 50 ppm, e.g., Belousova *et al.*, 2002), ^{238}U is normally detected with Faraday cup (e.g., Bühn *et al.*, 2009). At the formation of a zircon, little Pb can enter its lattice and almost all Pb in a zircon comes from U radioactive decay. Therefore, with different U content and formation age, zircons have a large variation of their Pb contents. Figure 1 shows the relationships between Pb content and U content, zircon formation time, which indicates that ^{206}Pb content is much higher than ^{207}Pb content in a zircon and the younger is a zircon, the smaller is the ratio of ^{207}Pb/^{206}Pb. For a zircon with age less than 1500 Ma, the content of ^{207}Pb is less than 10% of the content of ^{206}Pb. Thus, ^{207}Pb is set to be collected with ion counter whereas ^{206}Pb is collected with ion counter or Faraday cup according to its signal intensity.

Instrument

All zircon U-Pb age measurement in this study were performed on a Neptune Plus MC-ICP-MS (Thermo Scientific), coupled with a RESOlution M-50 193 nm laser ablation system (Resonetics), which are hosted at the State Key Laboratory of Isotope Geochemistry, Guangzhou Institute of Geochemistry, Chinese Academy of Sciences

(GIG-CAS). The detailed description of the both machines can be found in our previous study (Zhang *et al*., 2014). In our experiment, two sets of detector arrays were used (Supplementary Table S1). For Pb-rich zircon (the intensity of ^{206}Pb > 15 mV), ^{206}Pb was collected with Faraday cup (FC array), for Pb-poor zircon (the intensity of ^{206}Pb < 15 mV), ^{206}Pb was collected with ion counter (IC array). The IC array detects the signals of ^{202}Hg, ^{204}Pb, ^{206}Pb, ^{207}Pb, ^{232}Th and ^{238}U simultaneously, whereas due to the limitation of the geometry design of the multi-collector, the FC array used two detector sequences to collect the signals of ^{206}Pb, ^{207}Pb, ^{208}Pb, ^{232}Th and ^{238}U. ^{208}Pb, which was collected by both detector sequences, was used to cancel out the instrumental drift between each sequence. The integration time was set to 0.131 sec and each point collected about 200 valid cycle data. The laser ablation system, equipped a two-volume ablation cell supplied by Laurin-Technic, can change the laser beam size in the range of 7–380 μm in diameter, and the repetition rate in the range of 1–20 Hz. For Pb-rich zircon, to ensure that sample signals are high enough for Faraday cups, a laser beam with 33 μm spot size and 6 Hz repetition rate was used. For Pb-poor zircon, because IC has much higher sensitivity than Faraday cup, the laser shots condition was set to spot size of 17 μm and repetition rate of 4 Hz. Before each spot measurement, five laser ablations were performed to remove any contamination on zircon surface. Each spot measurement was started with 25 sec gas blank collection with laser off and followed with 30 or 45 sec laser ablation to collect sample signals with laser on. When IC detector array was used, the ablation time was 30 sec, whereas when FC detector array was used, the ablation time was 45 sec. A "squid" smoothing device was equipped on the gas line to the ICP to give a smooth signal. During our experiment, the instrumental blanks of ^{206}Pb and ^{238}U were less than 50 cps and 0.2 mV, respectively and the oxide production, indicated by ^{254}UO/^{238}U, was less than 0.5%. The instrumental drift and mass bias for all isotope ratios were corrected with a standard-sample bracketing method (SSB). Both instrumental settings during our experiment are supplied in Supplementary Table S2.

Samples

Six reference zircons (91500, MudTank, BR266, Plešovice, Qinghu and OD-3), with ages ranging from 1064 Ma to 33 Ma, were measured in this study. According to their Pb contents, the six reference zircons were measured with the FC detector array or the IC detector array. Zircon Plešovice was selected as the external calibrator. In the case of Plešovice U-Pb age dating, 91500 or BR266 was used as calibrator. Finally, the U-Pb ages of 77 zircons selected from a sedimentary rock in Yinggehai Basin, South China Sea, were measured with the analytical protocol established in this study to show the application. All zircon crystals were mounted in one-inch diameter epoxy resin discs, which were polished with diamond abrasive. Before analysis, the resin discs were cleaned in an ultrasonic bath with 0.1% HNO_3 three times and rinsed with double-distilled Milli-Q water (18.2 MΩ cm, 25°C), and then dried with a nitrogen jet.

Data reduction

All time-resolved raw data were exported from the MC-ICP-MS in ASCII format with Neptune Plus software and Off-line data reduction was performed on an in-house created spreadsheet program. First, instrumental blank was subtracted from all sample signals. Then the ratios of ^{207}Pb/^{206}Pb and ^{206}Pb/^{238}U were calculated cycle by cycle. After that, ^{207}Pb/^{235}U was derived from the following equation:

$$^{207}\text{Pb}/^{235}\text{U} = {}^{207}\text{Pb}/^{206}\text{Pb} * {}^{206}\text{Pb}/^{238}\text{U} * 137.88 \quad (1)$$

where the 137.88 is the constant ratio of ^{238}U/^{235}U. The instrumental mass bias was corrected with the SSB method. Every five analyses of sample were preceded and followed with two analyses of reference standard. Then the ratios of the analyzed reference standard measured before and after each five analyses of samples were averaged to calculate correction factors. For each isotope ratio, the standard errors (SE) of ^{207}Pb/^{206}Pb and ^{206}Pb/^{238}U of each measurement were derived from the external reproducibility of calibrator reference and their within-run precision following the equation in Bühn *et al*. (2009) as:

$$\text{SE } (^{207}\text{Pb}/^{206}\text{Pb or } ^{206}\text{Pb}/^{238}\text{U}) = (\text{SD}^2 + \text{SE}^2)^{0.5}$$

where SD being the standard deviation of the calibrator and SE (standard deviation/$(n–1)^{0.5}$) being the within-run precision for individual samples.

RESULTS AND DISCUSSION

Reference zircons

The six reference zircons were measured with FC detector array or IC detector array based on their Pb contents. When IC detector array was used, a laser spot with diameter of 17 μm and repetition rate of 4 Hz was used. When FC array was used, to increase the signal intensity, the laser spot size and repetition rate were changed to 33 μm and 6 Hz, respectively. The results are displayed in Figs. 2 and 3 and Supplementary Table S3. 91500, Mud Tank, Qinghu and OD-3 have relatively low Pb contents (about 6–17 ppm, Li *et al*., 2013; Yuan *et al*., 2008; Iwano *et al*., 2013) among the measured reference zircons. Therefore, these four zircons were measured with IC detector array. With a 17 μm laser spot, the signal intensity of ^{206}Pb

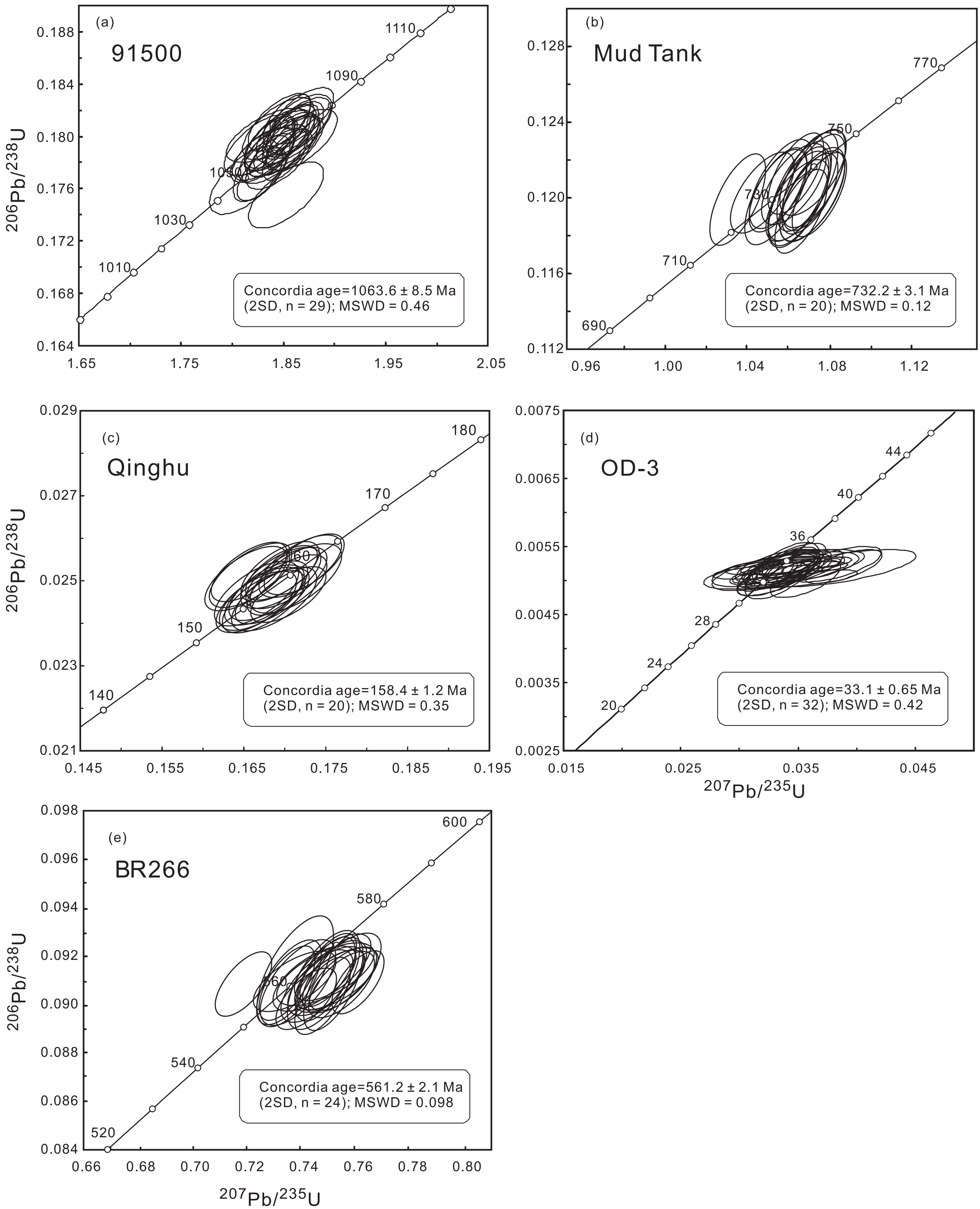

Fig. 2. U-Pb concordia diagrams showing the results for reference zircon 91500 (a), Mud Tank (b), Qinghu (c), OD-3 (d) and BR266 (e). Data in (a)–(d) were measured with IC detector array and data in (e) were measured with FC detector array. Data-point error ellipses are 1 SE.

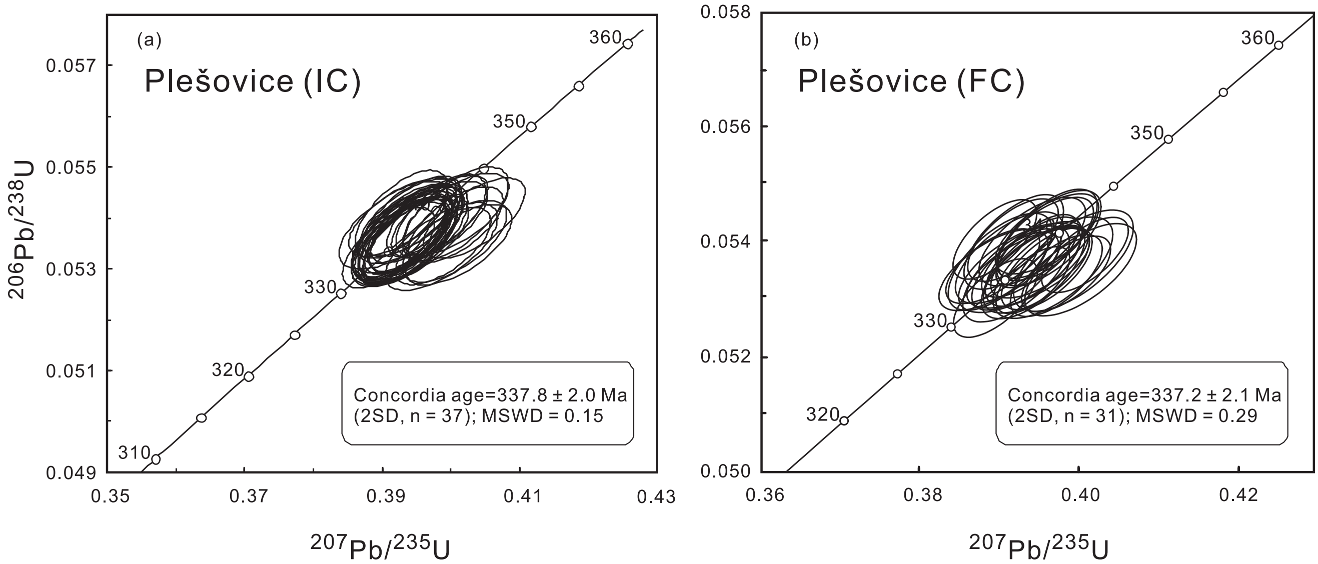

Fig. 3. U-Pb concordia diagrams showing the results for reference zircon Plešovice measured with IC detector array (a) and with FC detector array (b). Data-point error ellipses are 1 SE.

Table 1. U-Pb ages of five reference zircons measured in this study and by ID-TIMS in previous studies

Sample	This study		ID-TIMS		Reference
	$^{206}Pb/^{238}U$ age	$^{207}Pb/^{235}U$ age	$^{206}Pb/^{238}U$ age	$^{207}Pb/^{235}U$ age	
91500	1063.6 ± 15.2	1062.6 ± 9.8	1062.4 ± 0.8	1065.4 ± 0.6	Wiedenbeck *et al.* (1995)
Mud Tank	732.4 ± 5.9	735.5 ± 9.8	732 ± 5[c]		Black and Gulson (1978)
Qinghu	158.6 ± 3.7	158.2 ± 3.7	159.38 ± 0.12	159.68 ± 0.22	Li *et al.* (2009)
BR266	561.3 ± 3.5	566.2 ± 10.5	559.0 ± 0.2	562.6 ± 0.4	Stern and Amelin (2003)
Plešovice	337.4 ± 2.6[a]	338.3 ±4.7	337.13 ±0.37	337.19 ± 0.48	Sláma *et al.* (2008)
	337.1 ± 3.3[b]	337.5 ±4.7			
OD-3	33.1 ± 0.8		32.853 ± 0.016		Lukács *et al.* (2015)

Lukács et al. (2015).
[a] Measured with IC detector array.
[b] Measured with FC detector array.
[c] This is a concordia intercept age.

was about 150000 cps, 70000 cps, 120000 cps and 50000 cps for 91500, Mud Tank, Qinghu and OD-3, respectively. 29 analyses of 91500, 20 analyses of Mud Tank, 20 analyses of Qinghu and 32 analyses of OD-3 gave concordia age of 1063.6 ± 8.5 Ma (2σ), 732.2 ± 3.1 Ma (2σ), 158.4 ± 1.2 Ma (2σ) and 33.1 ± 0.65 Ma (2σ), respectively. BR266 has the highest Pb content (~90 ppm, Yuan *et al.*, 2008) among the measured zircons and even with a 17 μm laser spot, its ^{206}Pb intensity is more than 30 mV. Thus, to avoid IC damage, BR266 was measured with the FC array with a 33 μm laser spot. 24 analyses of BR266 gave a concordia age of 561.2 ± 2.1 Ma (2σ). Plešovice measured in this study has a moderate Pb content (~28 ppm) among these zircons. With a 17 μm laser spot, ^{206}Pb signal intensity of Plešovice was about 430000 cps and 37 analyses of Plešovice under this laser condition gave a concordia age of 337.8 ± 2.0 Ma (Fig. 3a). When laser spot size changed to 33 μm, the signal intensity of ^{206}Pb was about 42 mV, and 31 analyses of Plešovice gave a $^{206}Pb/^{238}U$ age of 337.2 ± 2.1 Ma (Fig. 3b).

Table 1 summarizes our results of these reference zircons and ID-TIMS results from previous studies. The precision of $^{206}Pb/^{238}U$ age and $^{207}Pb/^{235}U$ age of the six zircons vary from 0.6% to 2.3% and 0.9% to 2.3% respectively and their accuracy, compared to ID-TIMS results, are from –0.5% to 0.4% and –0.9% to 0.6%, respectively. Thus, with the proper detector array and laser ablation condition, precise and accurate ages can be obtained.

Detrital zircons

To show the application of the established analytical procedure, 77 detrital zircons selected from a sedimen-

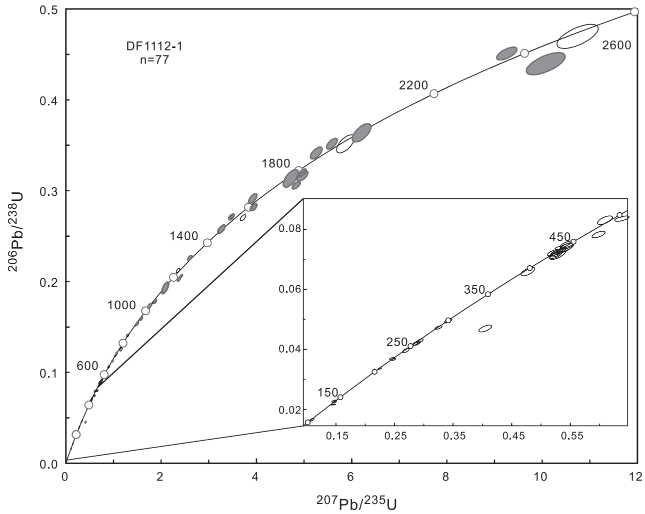

Fig. 4. U-Pb concordia plot for detrital zircon from Yinggehai basin, South China Sea. Open circles (n = 50) were measured with IC detector array and filled circles (n=27) were measured with FC detector array. Note that all zircons measured with FC detector array have Precambrian ages (>600 Ma). Data-point error ellipses are 1 SE.

tary rock, in Yinggehai Basin, South China Sea, were measured. Zircons selected from the same sedimentary rock (DF1112-1) were previously measured by laser ablation quadruple inductively coupled plasma mass spectrometry (Wang *et al.*, 2014). To decide which detector array to apply, the magnetic field was changed to ^{206}Pb for the center Faraday cup and all zircons intended to be measured were ablated with a 17 μm laser spot for about three seconds to record the ^{206}Pb signal intensity. After that, the detector array was changed to FC detector array and zircons with ^{206}Pb intensity more than 15 mV were measured with a 33 μm laser spot first. Then the detector array was changed to IC array to measure zircons with ^{206}Pb intensity less than 15 mV with a 17 μm laser spot. The results are displayed in Fig. 4 and Supplementary Table S4, in which filled circles represent data measured with FC detector array, whereas open circles measured with IC detector array. The results indicate the measured zircons have a large range of ages from about 100 Ma to 2500 Ma, which are consistent with the previous study (figure 7b in Wang *et al*. (2014)). Compared to the results in Wang *et al*. (2014) which shows about 20% data of ^{207}Pb/^{235}U age and ^{206}Pb/^{238}U age having concordance less than 95%, with the established method in this study, except for one point, all other analyses of ^{207}Pb/^{235}U age and ^{206}Pb/^{238}U age have concordance more than 96%. In addition, our results show a much smaller internal analytical error for ^{207}Pb/^{235}U (normally around 1–2%, Table S4) than previous study (3–6%, Wang *et al*., 2014).

CONCLUSIONS

In this study, we refined the analytical protocol for detrital zircon U-Pb dating by LA-MC-ICP-MS. Two sets of detector arrays were used. For Pb-rich zircons, a 33 μm laser spot was used and ^{206}Pb was collected with Faraday cups, whereas for Pb-poor zircons, the laser spot size changed to 17 μm and ^{206}Pb was collected with ion counter. The measurements of reference zircons suggest that with a proper detector array and laser ablation condition, precise and accurate ages can be obtained. The result of the reference zircon OD-3 shows it was possible to measure zircon ages as low as about 33 Ma with the present method. The application of our method to detrital zircons from Yinggehai Basin, South China Sea, shows an improved precision and age accordance over previous study.

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SUPPLEMENTARY MATERIALS

URL (http://www.terrapub.co.jp/journals/GJ/archives/data/52/MS529.pdf)
Tables S1 to S4