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U-Pb dating by zircon dissolution method using chemical abrasion

LUCY TAKEHARA[1], FARID CHEMALE JÚNIOR[2], LÉO A. HARTMANN[3], IVO A. DUSSIN[4] AND KOJI KAWASHITA[4]

[1]Serviço Geológico do Brasil – CPRM, SGAN 603, Conjunto "J" . Parte A , 1° andar, 70830-030, Brasília, DF, Brasil
[2]Laboratório de Geocronologia, Instituto de Geociências, Universidade de Brasília,
Campus Darcy Ribeiro, s/n, 70904-970, Brasília, DF, Brasil
[3]Instituto de Geociências, Universidade Federal do Rio Grande do Sul,
Av. Bento Gonçalves, 9500, Bairro Agronomia, 91501-970, Porto Alegre, RS, Brasil
[4]Centro de Pesquisa Geocronológicas, Universidade de São Paulo,
Rua do Lago, 562, 05508-080, São Paulo, SP, Brasil

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ABSTRACT

Chemical abrasion was carried out on zircons grains of the Temora II standard for U-Pb dating prior to analyses using *in situ* Laser Ablation-MultiCollector Ion Coupled Plasma Mass Spectrometer (LA-ICPMS) followed by the Isotope Dissolution Thermal Ionization Mass Spectrometer (ID-TIMS) method. The proposed methodology was herein applied in order to reduce primarily the effects of secondary Pb loss, the presence of common lead and/or silicate impurities. Nine Temora II zircon grains were analyzed by the laser ablation method yielding an age of 418.3±4.3 Ma. Zircon grains of a same population were separated for chemical abrasion before dissolution and mass spectrometry analyses. Six fractions of them were separated for isotope dissolution using ^{235}U-^{205}Pb mixed spike after we have checked and assured the laboratory conditions of low blank values for total Pb of less than 2 pg/g. The obtained U-Pb zircon age by the ID-TIMS method was 415.7±1.8 Ma (error 0.43 %) based on four successful determinations. The results are consistent with the published ages for the Temora diorite (Temora I – 416.75±1.3 Ma; Temora II – 416.78±0.33 Ma) and established as 416±0.33 Ma. The technique is thus recommended for high precision U-Pb zircon analyses (error < 1 %), mainly for high resolution stratigraphic studies of Phanerozoic sequences.

Key words: chemical abrasion, ID-TIMS, LA-MC-ICPMS, U-Pb dating, zircon dissolution.

INTRODUCTION

The age determinations of geological events are a significant tool in basic and applied earth science studies. In the last two decades, the *in situ*, high resolution U-Pb dating by SHRIMP (Sensitive High Resolution Ion Microprobe) (Williams 1998, Rasmussen 2005) and LA-ICPMS (Laser Ablation Ion Coupled Plasma Mass Spectrometer) (Kosler et al. 2001, Jackson et al. 2004, Pickhardt et al. 2005) has brought new insights into the understanding of crystallization ages of minerals, especially zircon. The complexity of geological processes registered in the minerals can be dated with these techniques. These solid state, *in situ* determinations are useful

Correspondence to: Lucy Takehara
E-mail: lucytakehara@gmail.com.br

to geoscientists because they can identify the timing of events such as rock generation, metamorphism and deformation in the crust and mantle.

Despite the great advance in U-Pb *in situ* dating with SHRIMP and LA-ICPMS methods, in some cases these techniques do not attain the required accuracy to date geologic events that occurred at the end of the Precambrian and in the beginning of the Phanerozoic. In these cases, more precise techniques are required, such as the isotopic dissolution (ID) methods in minerals using Thermal Ionization Mass Spectrometer (ID-TIMS). However, the total Pb blank (common Pb contamination) in the laboratory has to be less than 2 pg/g. Indeed, in good laboratory conditions the dating of one crystal or part of it by ID-TIMS may yield better precision than 0.1% of error, while the *in situ* dating with 25 μm beam gives 1% or more of error (Bowring and Schmitz 2003).

The chemical abrasion procedure in the zircon dissolution method is widely used because it may avoid the common Pb that is present in the external surface of the crystal. This procedure has improved the high resolution U-Pb dating.

Thus, the aim of this study is to present the U-Pb dating of zircons of the Temora diorite by ID-TIMS by applying the chemical abrasion technique to the analyzed zircon fractions before the isotope dissolution. The sample for this study was provided by the Australian Geological Survey Organization, and the diorite has been systematically used as standard for geochronological studies (Black et al. 2003). In order to separate the zircon grains with a single crystallization phase (zircon with overgrowth), U-Pb *in situ* dating by the LA-MC-ICP-MS method was carried out in the zircon concentrate.

ANALYTICAL PROCEDURES

After the crushing and milling of the diorite Temora II sample, zircon grains were separated by conventional procedures using heavy liquids and magnetic separator after concentration by hand panning. The most clear and inclusion-free zircon grains from the least magnetic fractions were handpicked for *in situ* LA-MC-ICP-MS analyses and exposed to chemical abrasion for subsequent U-Pb ID-TIMS analyses.

U-Pb *in situ* LA-MC-ICPMS Analyses

All zircon grains were mounted in epoxy in 2.5-cm-diameter circular grain mounts and polished to reveal the internal part of the grains. Images of zircons grains were obtained using binocular microscope Leica MZ 12_5 and backscattering images with the scanning electron microscope Jeol JSM 5800. Zircon grains were dated with a New Wave UP213 laser ablation microprobe coupled to a Neptune MC-ICP-MS at the Isotope Geology Laboratory of Universidade Federal do Rio Grande do Sul (UFRGS). Isotope data were acquired using a static mode with the spot size of 25 μm. The Laser-induced elemental fractional and instrumental mass discrimination were corrected by the GJ-1 reference zircon (Simon et al. 2004), following the measurement of two GJ-1 analyses to every ten sample zircon spots. The external error was calculated after the propagation error of the GJ-1 mean and the individual sample zircon (or spot). Laser operating conditions are summarized in Table I, and a detailed description of analytical method can be found in Chemale Jr. et al. (in press).

TABLE I
Operation conditions of the Laser Ablation type New Wave UP213 and MC-ICP-MS Neptune

Laser type New Wave UP213	**MC-ICP-MS Neptune**
• Laser output power 6 J/cm2 • Shot repetition rate 10 Hz • Laser spot 25 and 40 μm	• Cup configuration: Faradays: ^{206}Pb, ^{208}Pb, ^{232}Th, ^{238}U MIC: ^{202}Hg, $^{204}Hg+^{204}Pb$, ^{207}Pb • Gas input: Coolant flow (Ar) 15 l/min Auxiliary flow (Ar) 0.8 l/min Carrier flow 0.75 l/min (Ar) + 0.45 l/min (He) • Acquisition 50 cycles of 1.048 s

U-Pb ID-TIMS Analyses

Water and acid preparation and washing

In the Isotope Geology Laboratory of UFRGS clean laboratory facility (class 100), we obtained the best water and acid blanks for the U-Pb isotope dilution method, taking into account the suggestions of the researchers of Earth Time group (Bowring, personal communication).

The study started by improving the laboratory conditions in order to get low Pb blanks for the analyses. This involved the purification of water to prepare the acids and the cleaning of the polytetrafluoretilene (PTFE) vials, bomb and microcapsules to dissolve the zircon grains. In order to get pure acids, we produced purified water below 2 pg/g of total Pb, from initial Milli-Q® water with blank values lower than 10 pg/g.

To purify the Milli-Q® water, we used two techniques: (i) filtering process with anionic resin (10 ml each time) and (ii) bi-destillation of Milli-Q® water. The second technique was more effective and faster, producing a larger amount of ultrapure water with a blank of total Pb under 2 pg/g. After the preparation of water and acids with total Pb blank values (under 2 pg/g), we proceeded to the cleaning procedure of microcapsules and bombs. Each bomb and microcapsule was washed nine (9) times with the following acids sequence: HF conc + HNO_3 7N at 200° C in the oven for one day (3 times); HCl 6N at 200° C in the oven for two days (3 times); and HF conc + HNO_3 7N at 200° C in the oven for one day (3 times). In each acid washing exchange the bomb and microcapsules were washed three times with purified water with 2.5 Pb pg/g of total Pb.

Zircon pre-treatment (including chemical abrasion)

Zircon crystals from the same igneous population analyzed at Isotope Geology Laboratory of UFRGS by LA-MC-ICP-MS were separated by handpicking for ID-TIMS analyses. These crystals were clear and inclusion free. All zircon crystals were photographed with a binocular microscope. The isotope analyses were done after the following preparation steps.

The zircon grains were pre-treated following the procedures suggested by Mundil et al. (2004), which are: annealing (thermal treatment at 850°C for 36 h); making photograps of all crystals using the binocular microscope; chemical abrasion (HF conc in Teflon vials, as pressurized dissolution, at 200°C for 16 h); washing several times with tridistilled HNO_3; cleaning with aqua regia in an ultrasonic equipment followed by tridistilled HNO_3; making photographs of all crystals using the binocular microscope to estimated the sample weight; zircons were separated into groups of 6 to 8 grains, weighing approximately 10 µg each; and tgrouping the zircon grains that were then transferred to ultraclean microcapsules (PFTE Krogh type).

Zircon dissolution and U-Pb analyses

After the chemical abrasion, the separated zircon grains were dissolved in a microcapsule as follows: 10 drops of ultraclean HNO_3 7N were added and heated in the hot plate for 30 minutes at 100° C; the zircon grains were washed three times with HNO_3 7N, and a drop of acid plus zircon grains was left in the bottom of the microcapsule; 200 µl of 50 % HF was added with 4µl double spike ^{205}Pb-^{233}U-^{235}U (UNB205PC) in the individual microcapsule. Three sets of five microcapsules each were placed on an elevated rack in a 125 ml digestion vessel, allowing the vapor transfer dissolution in a mixture with 50 % HF/HNO_3 conc (30/1). The digestion vessel was placed in a metal jacket and left in an oven for six days at 220 °C. After the zircon dissolution, the sample + spike were transferred to Teflon® vials, which were dried with 20 µl of 3M HCl + 5µl 0.25 M H_3PO_4. The dried dissolved zircon samples were loaded in rhenium filament with silica gel + H_3PO_4.The U and

Pb concentrations were determined in a VG-Sector 54 mass spectrometer with Daly detector and ion counting system of ORTEC, in a single mode. Pb was analyzed as metal and U as oxide. The isotope masses of ^{204}Pb, ^{205}Pb, ^{206}Pb, ^{207}Pb, ^{208}Pb, ^{235}U and ^{238}U were collected. Repeated analyses of NBS-981 and NBS-982 presented a Pb fractionation of ≤ 0.1 % amu, similar to that of NBS 982 analyzed in the same turret and whose zircon samples were also analyzed. All data were reduced using the PBDAT program and Isoplot 3.1 (Ludwig 2003).

We dissolved seven zircon samples and two blanks. Two samples presented either very low ^{206}Pb/^{204}Pb isotope ratio (~200) or unreliable U-Pb data (sample MC 6, not shown in Table III), which we attribute to high Pb blank of the microcapsule or to contamination during the analyses.

RESULTS

U-Pb isotope analyses of nine zircon grains were determined by LA-MC-ICPMS and are shown in Table II.The data from eight zircon spots yielded a concordant age of 418.6±4.3 Ma (±1.02%)(Figure 1). The ^{206}Pb/^{238}U age is 418.1±7.1 Ma (±1.7 %, 95% conf.). This result is in good agreement with the age published for Temora II zircon obtained by ID-TIMS and SHRIMP methods (Black et al. 2004), although the calculated error is too high for geochronological studies of Phanerozoic rocks.

In the present study, the dating by the ID-TIMS method is more appropriate for zircon grains with only one crystallization phase. Thus, the ID-TIMS analyses of the same zircon population of Temora II standard were performed in two important steps before the zircon dissolution, which permitted to get high quality results (Table III). Ultrapure water with very low total Pb blank was used, and this demanded a long time to obtain (approximately 4-6 months). Indeed, it is not possible to obtain a high resolution U-Pb dating without a good laboratory blank (Bowring and Schmitz 2003).

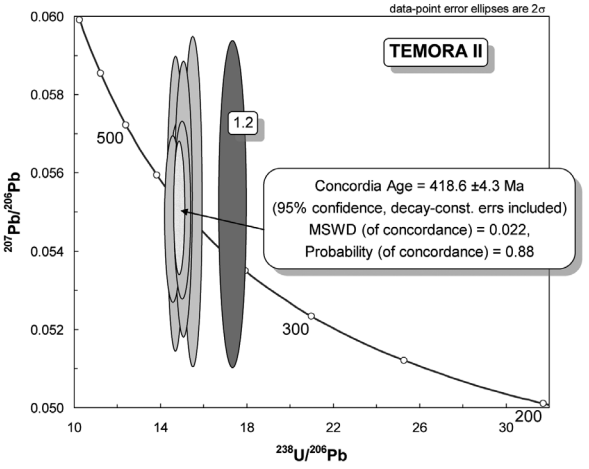

Figure 1. U-Pb concordia diagram for Temora II standard obtained by LA-MC-ICPMS.

Aother important step was the combination of zircon heating and chemical abrasion. This step showed that the results could be improved by leaching the common Pb present in the outer part of zircon grains. The heating procedure can seal small fractures, and, thus, homogenize the external surface, which was leached by chemical abrasion with HF vapor. The chemical abrasion procedure in this work is that described by Mundil et al. (2004).

The results are presented in Table III, which includes the analytical data of analyzed zircon grains and NBS 982 values obtained during the U-Pb isotope analyses.

Two blanks and seven zircon samples were analyzed. The obtained values for blanks (microcapsules without samples) were < 1pg/g of total Pb. Two zircon samples presented either very low ^{206}Pb/^{204}Pb isotope ratios (~200) or unreliable U-Pb data (sample MC 6), which we attribute to high Pb blank of the MC or to contamination during the analyses. From the five reliable analyses, two yielded concordant or almost concordant zircon fractions with concordia age at 416 Ma, and three discordant zircon fractions are used for age calculation (Figure 2). The age obtained is 415.7±1.8 Ma(±0.43%, 95 % conf.) (Figure 3),

TABLE II

U-Pb *in situ* zircon data of Temora II standard obtained by LA-MC-ICPMS. Mean $^{206}Pb/^{238}U$ age = 418.1±7.1 [1.7%] 95% conf.

		Isotope ratios[2,3]								Age (Ma)						
Grain spot	f 206	$^{232}Th^{1}/^{238}U$	$^{207}Pb^{*}/^{06}Pb^{*}$	err %	$^{207}Pb^{*}/^{35}U$	err %	$^{206}Pb^{*}/^{38}U$	err %	Rho[4]	$^{206}Pb/^{38}U$	±	$^{207}Pb/^{35}U$	±	$^{207}Pb/^{06}Pb$	±	%Disc[5]
1.1	0.0006	0.25	0.0552	1.50	0.516	1.84	0.068	1.07	0.58	422	5	422	8	422	6	0
1.2	0.0006	0.46	0.0552	3.11	0.439	3.45	0.058	1.48	0.43	362	5	370	13	420	13	14
1.3	0.0010	0.20	0.0553	1.34	0.507	2.85	0.066	1.10	0.39	415	5	416	12	424	6	2
1.4	0.0011	0.28	0.0552	1.70	0.492	3.33	0.065	1.12	0.34	404	5	406	14	422	7	4
1.5	0.0005	0.37	0.0548	1.60	0.519	1.92	0.069	1.05	0.55	428	5	424	8	404	6	-6
1.5	0.0009	0.37	0.0550	1.69	0.506	2.01	0.067	1.08	0.54	416	5	416	8	413	7	-1
1.7	0.0004	0.28	0.0554	2.21	0.520	2.46	0.068	1.06	0.43	425	5	425	10	427	9	1
1.8	0.0001	0.35	0.0552	2.80	0.519	3.00	0.068	1.06	0.35	425	5	424	13	420	12	-1
1.9	0.0006	0.42	0.0550	2.03	0.498	2.31	0.066	1.10	0.48	410	5	410	9	411	8	0

1. $^{232}Th/^{238}U$ ratios are calculated relative to GJ-1 reference zircon, where Th/U = $^{232}Th/^{238}U$*0.992743
2. Corrected for background and within-run Pb/U fractionation and normalised to reference zircon GJ-1 (ID-TIMS values/measured values); $^{207}Pb/^{235}U$ calculated using ($^{207}Pb/^{206}Pb$)/($^{238}U/^{206}Pb$ * 1/137.88)
3. Sample and standard are corrected after Pb and Hg blanks
4. Rho is the error correlation defined as the quotient of the propagated errors of the $^{206}Pb/^{238}U$ and the $^{207}/^{235}U$ ratio
5. Degree of disconcordance = 100* (1-$^{206}Pb/^{238}U$ age/100/$^{207}Pb/^{206}U$ age)
6. Light gray = discordant (not used for concordia age)

TABLE III

(a) U-Pb zircon data of Temora II standard obtained by ID-TIMS; mean $^{206}Pb/^{238}U$ age = 418.1±7.1 [1.7%] 95% conf. and (b) Analytical data of NBS 982 values obtained during the U-Pb analyses and NIST values.

(a)

					Isotope ratios									Age (Ma)					
Sample	Sample Wt (mg)	Spike Wt (mg)	Pb ppm	U ppm	$^{206}Pb/^{04}Pb$	± (%)	$^{207}Pb*/^{35}U$	± (%)	$^{206}Pb*/^{238}U$	± (%)	Rho	$^{207}Pb*/^{06}Pb*$	± (%)	$^{206}Pb/^{38}U$	± (%)	$^{207}Pb/^{35}U$	± (%)	$^{207}Pb/^{06}U$	± (%)
TU MC 1	0.025	0.004	25	42	954	1.24	0.4437	0.95	0.0581	0.83	0.89	0.0554	0.42	364	3.0	373	3.5	427	1.8
TU MC 2	0.030	0.004	32	46	1975	0.38	0.4978	0.29	0.0655	0.28	0.96	0.0551	0.08	409	1.1	410	1.2	417	0.3
TU MC 3	0.020	0.004	37	60	920	0.62	0.4592	0.78	0.0604	0.74	0.95	0.0551	0.24	378	2.8	384	3.0	417	1.0
TU MC 7	0.015	0.008	75	100	1156	2.20	0.5034	0.79	0.0663	0.51	0.70	0.0551	0.57	414	2.1	414	3.3	416	2.4
TU MC 8	0.010	0.004	75	139	1432	3.58	0.4056	3.44	0.0529	3.35	0.98	0.0556	0.76	332	11	346	12	438	3.3

1. Zircons are annealed/chemically abraded
2. Sample weight calculated from crystal dimensions
3. $^{207}Pb*$ and $^{206}Pb*$ radiogenic Pb
4. Estimated total Pb blank = 2 pg

(b)

	$^{206}Pb/^{204}Pb$	2σ	$^{207}Pb/^{204}Pb$	2σ	$^{208}Pb/^{204}Pb$	2σ	$^{207}Pb/^{206}Pb$	2σ	$^{208}Pb/^{206}Pb$	2σ
NBS 982 11.FEV.2008	36.726	0.031	17.138	0.032	36.665	0.033	0.4667	0.0084	0.9984	0.0118
NIST	36.744	0.050	17.159	0.025	36.738	0.037	0.4671	0.0002	1.0002	0.0004
Correction due fractionation	0.0003		0.0004		0.0005		0.0009		0.0009	

* During the month (February) we analysed repeatedely the NBS-981 and NBS-982, and the Pb fractionation of amu = or < 0.1 %

which is very close to the accepted TIMS age for the Temora I (416.75±1.3 Ma, Black et al. 2003) and Temora II (416.78±0.33 Ma, Black et al. 2004).

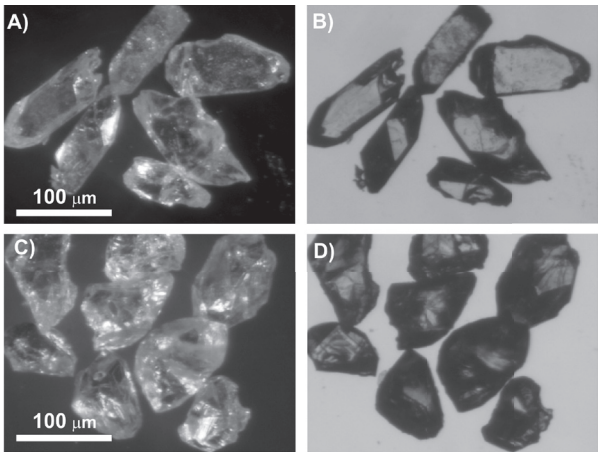

Figure 2. Photomicrography of analyzed zircon grains in transmitted (A, C) and reflected light (B, D), where A and B are long prismatic, and C and D short prismatic crystals.

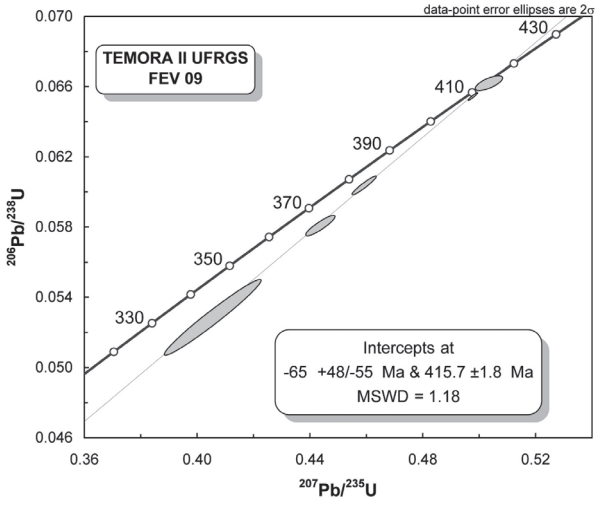

Figure 3. U-Pb concordia diagram for Temora II standard obtained by ID-TIMS.

CONCLUSIONS

Zircon analyses for *in situ* dating by the LA-MC-ICPMS or SHRIMP method can be appropriate for choosing single zircon grains to be used for high resolution dating by the ID-TIMS method.

The analytical results obtained here by the LA-MC-ICPMS method have a great advantage because this is a quick analytical method (usually one day), although the error uncertainty is larger than 1 %. The obtained age and error can be used for regional geological studies or detrital zircon age determination, but it cannot solve some questions such as high resolution stratigraphic studies of Phanerozoic sequences.

On the other hand, the ID-TIMS method is a time consuming method as it takes at least seven days to be performed, and is very sensitive to laboratory conditions. The obtained age and error are more precise and accurate, with an error uncertainty < 0.5 %, so the method can be applied to high resolution, Phanerozoic stratigraphy. The results of the present study, with the ID-TIMS methods combined with a previous *in situ* zircon dating, i.e., LA-MC-ICPMS method, are therefore significant for U-Pb high precision analyses, because the total Pb blank of the laboratory is under 2 pg/g, and the dated zircon phase has a single crystallization phase. The chemical abrasion combined with step heating is also a recommended method since it considerably decreases the amount of common Pb in zircon grains before their dissolution.

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RESUMO

Este trabalho apresenta a técnica de abrasão química em zircões do padrão Temora II aplicada em datação de U-Pb por Dissolução Isotópica e Espectrometria de Massa por Ionização Térmica, método DI-ESIT. O emprego deste método tem como princípio diminuir os efeitos da

perda secundária de Pb radiogênico antes da dissolução. Em primeira instância, os zircões foram analisados pelo método *in situ* com uso de Microssonda Laser acoplada a um Espectrômetro de Massa Multicoletor com Plasma Acoplado Indutivamente (MC-ICP-MS), cujo objetivo foi identificar os grãos de zircão de fase de cristalização simples sem sobrecrescimento. Nove cristais de zircão analisados com microssonda a laser forneceram uma idade 418,3±4,3 Ma. Da mesma população foram separados cristais de zircão para datação utilizando a técnica de abrasão química e posterior análise pelo método DI-ESIT. Seis amostras de zircão foram separadas para diluição isotópica com adição de um traçador isotópico combinado U^{235}-Pb^{205}, em condições laboratoriais de brancos analíticos de Pb total menor que 2 pg/g. A idade U-Pb em zircão obtida foi 415,7±1,8 Ma (erro 0,43 %) com base em cinco amostras de zircão. Os resultados são concordantes com aquelas idades publicadas para o diorito Temora (Temora I – 416,75±1,3 Ma; Temora II – 416,78±0,33 Ma). A técnica descrita aqui é, portanto recomendada para ser aplicada em análises isotópicas de U-Pb com alta precisão (erros menor que 1%) para estudos de estratigrafia de alta resolução de sequências fanerozóicas.

Palavras-chave: abrasão química, ID-TIMS, LA-MC-ICPMS, datação U-Pb, dissolução de zircão.

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