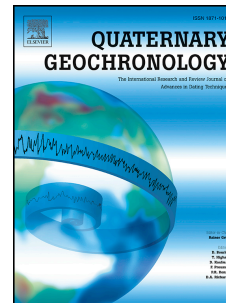


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1 **An (U-Th)/He age for the small Monturaqui impact structure, Chile.**

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14

15 **Abstract**

16 Single-crystal (U-Th)/He dating of 32 apatite and zircon crystals from an impact breccia  
17 yielded a weighted mean age of  $663 \pm 28$  ka ( $n = 3$ ; 4.2%  $2\sigma$  uncertainties) for the Monturaqui  
18 impact structure, Chile. This ~350 m diameter simple crater preserves a small volume of  
19 impactite consisting of polymict breccias that are dominated by reworked target rock clasts. The  
20 small size, young age and limited availability of melt material for traditional geochronological  
21 techniques made Monturaqui a good test to define the lower limits of the (U-Th)/He system to  
22 successfully date impact events. Numerical modelling of  $^4\text{He}$  loss in apatite and zircon crystals  
23 shows that, for even small craters such as Monturaqui, the short-lived compressional stage and  
24 shock metamorphic stage can account for the observed partial to full resetting of (U-Th)/He ages  
25 in accessory minerals. Despite the distinctly different  $^4\text{He}$  diffusion parameters of apatite and  
26 zircon, the  $2\sigma$ -overlapping youngest ages are recorded in both populations of minerals, which  
27 supports the inference that the weighted mean of the youngest (U-Th)/He population is the age of  
28 formation of this impact structure.

29

## 30 Introduction

31 Bolide impacts represent instantaneous events on geological timescales, and  
32 geochronological dating of crater formation should produce precise and accurate ages. However,  
33 most isotopic dating systems are not completely reset by impact processes. As little as ~10% of  
34 the target rocks affected by an impact event record impact-generated ages (Bogard et al., 1988;  
35 Schärer and Deutsch, 1990), which results in ambiguity when dating whole rock samples or  
36 mineral separates (Deutsch and Schärer, 1994). Of the 200 currently known terrestrial impact  
37 structures (excluding 4 historical events), 45% have been geochronologically analyzed, but only  
38 21 % yield ages with <5%  $2\sigma$  uncertainties (Earth Impact Database, 2020; Schmieder and Kring,  
39 2020). Typical geochronological dating of impact samples utilizes U-Pb,  $^{40}\text{Ar}$ - $^{39}\text{Ar}$ , K-Ar,  $^{14}\text{C}$ ,  
40 fission track,  $^{10}\text{Be}$ ,  $^{26}\text{Al}$ , U-Th series, paleomagnetic, thermoluminescence, and optically-  
41 stimulated luminescence techniques (Schmieder and Kring, 2020 and references therein).

42 The amount of melt produced at impact structures scales up with crater dimensions, and  
43 the proportion of target rock clasts in impact melt material decreases with the volume of melt  
44 produced (Grieve and Cintala, 1977). Large impact sites are more straightforward to date than  
45 small ones because melt products are both more abundant and less contaminated with unmelted  
46 xenocrysts and xenoliths, thus dating of these materials often yields valid reset ages using  
47 traditional geochronological methods such as U-Pb and  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  (e.g., Hodych and Dunning,  
48 1992; Kelley and Gurov, 2002). Nearly all (98%) of the crater ages with >5%  $2\sigma$  precision  
49 comes from impact structures with diameters of ~5-250 km (Earth Impact Database, 2020;  
50 Schmieder and Kring, 2020). However, 86% of know impact craters on Earth are medium- to  
51 small-sized (simple to complex central-uplift-peak craters up to 30 km in diameter), and 26% of  
52 all craters have diameters of <3 km (Earth Impact Database, 2020; Schmieder and Kring, 2020).

53 Because of the small volume of melt produced from <3 km craters, traditional geochronological  
54 techniques that rely on impact melt or neoblastic minerals from crystallized impact melts are not  
55 tractable. Therefore, dating of these small impact structures instead relies on methods such as  
56  $^{14}\text{C}$ , cosmogenic nuclides, luminescence and paleomagnetic techniques, which often have age  
57 range limitations or low precision (e.g., Veski et al., 2004; Salminen et al., 2006; Nakamura et  
58 al., 2014; Sighinolfi et al., 2015). Of the forty-nine <3 km diameter terrestrial craters (excluding  
59 4 historical craters), only one impact structure has an age with a  $2\sigma$  precision of <5%; the  $7.315$   
60  $\pm 0.080$  ka Macha Field craters ( $1.1\%$   $1\sigma$ ;  $^{14}\text{C}$  charcoal; Gurov and Gurova, 1998).

61 More recently, the (U-Th)/He thermochronological method has been successfully applied  
62 to both melt materials and unmelted impact breccias from impact structures. The technique relies  
63 on the resetting of  $^4\text{He}$  in U- and Th-bearing minerals, and has been successfully applied to  
64 apatite, titanite and zircon from multiple impact structures (van Soest et al., 2011; Wartho et al.,  
65 2012; Young et al., 2013; Wielicki et al. 2014; Biren et al., 2014, 2016, 2019). While a single-  
66 crystal (U-Th)/He analysis generally has lower precision (~6-10% and sometimes up to 30%  $2\sigma$   
67 uncertainties) compared to typical  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  and U-Pb analyses, multiple replicate (U-Th)/He  
68 analyses can be very accurate (Hourigan et al., 2005) and have resulted in robust impact  
69 formation ages. (U-Th)/He studies of minerals from impact structures include: (i) impact melt  
70 rocks from Manicouagan, Haughton and Clearwater East and West (van Soest et al., 2011;  
71 Young et al., 2013; Biren et al., 2016); (ii) lightly shocked (5-15 GPa) impact breccias from  
72 Wetumpka (Wartho et al., 2012); (iii) rapidly exhumed and cooled rocks from the Manicouagan  
73 central uplift peak (Biren et al., 2014); (iv) shocked and brecciated impactites from Morokweng  
74 (Wielicki et al., 2014); and (5) impact melt ejecta from an Ocean Drilling Program drill core  
75 sample, located ~390 km from the Chesapeake Bay impact structure (Biren et al., 2019).

76 This Monturaqui study utilizes the (U-Th)/He technique to date single apatite and zircon  
77 grains in two impactite breccia samples, containing clasts of local target rocks, from the small  
78 (350 m diameter) Chilean impact crater. In addition,  $^4\text{He}$  diffusion modeling of apatite and zircon  
79 grains during (i) impact compression and (ii) adiabatic decompression with associated shock  
80 metamorphism, illustrate the effects of these thermal processes in fully or partially resetting (U-  
81 Th)/He ages, even in very small (<1 km) diameter craters.

82

### 83 **Background**

84 The Monturaqui impact crater is located at the southern end of Salar de Atacama in the  
85 Precordillera of northern Chile (3015 m elevation;  $23^{\circ} 55' 39.28''$  S,  $68^{\circ} 15' 41.63''$  W). It is the  
86 second smallest crater in South America, and existing  $^{26}\text{Al}$ ,  $^{36}\text{Cl}$  and thermoluminescence ages  
87 suggest that it is the oldest of the 25 smallest craters on Earth (Schmieder and Kring, 2020).  
88 Monturaqui is a well-preserved, simple crater that formed in a Paleozoic basement granite ( $441 \pm$   
89  $8$  Ma;  $2\sigma$ ; Rb-Sr whole rock; Mpodozis et al., 1983) containing zircons and apatites (Bunch and  
90 Cassidy, 1972), which is cut by 1-2 m wide mafic dikes and is overlain by a thin (~5 m) sheet of  
91 Pliocene Tucucaro ignimbrite ( $3.2 \pm 0.3$  Ma; K-Ar whole rock; Ramirez and Gardeweg, 1982;  
92 Fig. 1). The crater has a sub-circular morphology with a preferential NW-SE elongation (370 m  
93 E-W, 350 m N-S, and 34 m deep; Ugalde et al., 2007). Both granite and ignimbrite are exposed  
94 in the walls of the crater, but the rim crest is dominantly draped by ignimbrite outcrop. It was  
95 first proposed as an impact structure in 1966 based on the presence of iron shale, inferred to be  
96 altered fragments of the Fe-Ni impactor. The impactites are described as “porous cindery  
97 aggregates containing fragments of granite and bonded with glass”, which are preferentially  
98 deposited on the southern and southeastern crater flanks (Sanchez and Cassidy, 1966;

99 Klobberdanz, 2010; Ukstins Peate et al. 2010; Rathbun et al. 2017). The impactor is postulated to  
100 be a Group I coarse octahedrite iron meteorite based on the composition of Fe-Ni spherules  
101 found in the impactite breccias and the structure of iron shale fragments (Sanchez and Cassidy,  
102 1966; Buchwald, 1975).

103 Initial studies on the Monturaqui impact structure assigned an age of Pleistocene to  
104 Recent, based on the apparent disruption of the local Pleistocene drainage patterns (Sanchez and  
105 Cassidy, 1966). Thermoluminescence analysis of quartz grains extracted from an impactite  
106 produced an age estimate of  $590 \pm 60$  ka ( $1\sigma$ ; Verdugo and Cartes, 2000). Valenzuela et al.  
107 (2009) used cosmogenic radionuclide dating techniques on quartz mineral separates from granite  
108 outcrops within the crater and residual activities of iron shale samples, to both evaluate the age  
109 of the crater and to obtain ages of the fragmented impactor. Age data from the granite produced  
110 concordant results for  $^{10}\text{Be}$  with an age range of 200 to 250 ka, but this is likely to be a minimum  
111 age due to subsequent erosion of the crater walls. The iron shale produced  $^{36}\text{Cl}$  and  $^{26}\text{Al}$  ages of  
112 500-600 ka, and paleomagnetic analyses suggested a granite remagnetization age of 780 ka,  
113 therefore the Monturaqui impact age was estimated to be between 500-780 ka (Valenzuela et al.,  
114 2009).

115

### 116 **Description of samples and electron microprobe imaging and analyses**

117 The impactite breccia samples were described by Bundy and Cassidy (1972) as cindery,  
118 highly vesicular agglomerations of shocked and unshocked granite fragments (several microns to  
119 4 cm in size) and nickel-iron and sulphide spherules tightly bound in a highly heterogeneous  
120 glass matrix. The impactites have a twisted, ropy appearance, similar to volcanic bombs, which  
121 was probably caused by distortion during flight when most of the components were still hot. In

122 addition, the outer surfaces of the impactites have embedded ignimbrite pebbles, which are  
123 common on the terrain surfaces, suggesting that the impactites were still very hot and viscous  
124 when they struck the ground. The  $441 \pm 8$  Ma Rb-Sr (Mpodozis et al., 1983) granite country rock  
125 is hypidiomorphic-granular in texture, consisting of zoned antiperthitic oligoclase-andesine,  
126 quartz, microcline, chlorite, biotite, magnetite (some grains with exsolved ilmenite), apatite, and  
127 zircon, as well as sericite and other alteration minerals (Bundy and Cassidy, 1972). The apatite  
128 and zircon grains (U-Th)/He dated in this study were most likely sourced from the granitic target  
129 rocks, rather than the welded dacitic ignimbrite, due to abundance.

130 Four zircon grains and fragments from each of two samples (CIUP 08099 and 08100)  
131 were photographed using a Leica MZ16 binocular microscope (Figs. 2-3). The grains were  
132 mounted on double-sided copper tape, carbon coated, and imaged and analysed using secondary  
133 electron (SE) and energy-dispersive spectrometry (EDS) on a JEOL JXA-8530F Hyperprobe  
134 field-emission electron microprobe, located at the John M. Cowley Center for High Resolution  
135 Electron Microscopy, ASU (Figs. 2-3). Operating conditions were 10 kV, a working distance of  
136 11 mm, and for the EDS analyses a current of 305 pA was used, with a counting time of 20  
137 seconds. Please note that it is not possible to undertake SE imaging and EDS analysis of the (U-  
138 Th)/He dated zircon and apatite grains, and hence only binocular microscope photomicrographs  
139 were obtained for the (U-Th)/He dated apatites and zircons (examples are shown in Fig. 4). The  
140 carbon coating plus sticky tape residue that is required for EMP analysis and SE imaging can  
141 cause extensive contamination of the noble gas extraction line and quadrupole mass  
142 spectrometer, resulting in interferences with the He isotopic measurements. For example, singly  
143 ionized deuterium ( $^2\text{H}$ ) interferes with  $^4\text{He}$  measurements, while ionized HD interferes with the  
144  $^3\text{He}$  spike isotopic measurements. Therefore, we only undertook SE imaging and EDS analysis

145 of non-dated zircon grains. Unfortunately, there were no remaining apatite grains or fragments  
146 found in the two samples available for EDS analysis and SE imaging.

147

#### 148 **Methods – (U-Th)/He dating**

149 We selected two impactite breccia samples from the Monturaqui crater, representing  
150 different quadrants of the ejected material. Sample CIUP 08099 was collected from the south-  
151 eastern flank of the crater, along the inferred impact trajectory, and CIUP 08100 originated from  
152 the crater rim directly to the south (Fig. 1). Impactite fragments ranged in size from 2 mm to 3  
153 cm, and a suite totalling 785 g and 840 g were selected for mineral separation from samples  
154 CIUP 08099 and 08100, respectively. The samples were crushed, dry and wet sieved, and  
155 magnetic separation and heavy liquid separation were used to generate apatite- and zircon-  
156 bearing separates. Zircon and apatite grains were hand-picked for (U-Th)/He analysis on the  
157 basis of their euhedral habit and apparent lack of inclusions. As many suitable apatites as  
158 possible were selected from each sample as it was expected that apatite had the greatest chance  
159 of recording the impact age due to its lower  $^4\text{He}$  closure temperature (Farley, 2000) compared to  
160 zircon (Reiners et al., 2004). A total of 10 zircon and 22 apatite grains were analyzed, 5 zircons  
161 from each sample, plus 8 and 14 apatites from samples CIUP 08099 and CIUP 08100,  
162 respectively.

163 The dimensions relevant for the application of the alpha ejection correction were  
164 measured for each grain using photomicrographs taken at high magnification (184 x) with a  
165 digital video camera attached to a Leica MZ16 binocular microscope (Fig. 4). The camera  
166 images were calibrated to specific magnification stops on the microscope so that computer  
167 software could be used to determine the relevant dimensions of the grains (Table 1).



168 Single grains were loaded into individual niobium (for zircon) and platinum (for apatite)  
169 microcrucibles, which were crimped to close and loaded into the sample holder of the Australian  
170 Scientific Instruments Alphachron at the Group 18 Laboratories at Arizona State University. The  
171 laser chamber was pumped down overnight and helium was extracted from each sample using a  
172 45 W, 980 nm infra-red diode laser, using an energy output of approximately 10 W for apatite  
173 and 15 W for zircon. The  $^4\text{He}$  gas was spiked with  $^3\text{He}$  and cleaned of any reactive gases by  
174 exposure to a hot SAES NP-10 getter, before analysis on a Balzers Prisma QMS 200 quadrupole,  
175 equipped with Faraday and Channeltron electron multiplier detectors, which has a room  
176 temperature SAES NP-10 in the analysis chamber. All analyses were performed using the  
177 Channeltron electron multiplier. Between sample analyses, a known  $^4\text{He}$  aliquot was spiked with  
178  $^3\text{He}$  and analyzed to allow the amount of unknown  $^4\text{He}$  in the sample to be calculated. In addition  
179 to the samples, empty Nb and Pt tubes were analyzed as blanks, and several shards of Durango  
180 fluorapatite ( $32.0 \pm 1.8$  Ma;  $2\sigma$ ,  $n = 11$ ) and grains of Fish Canyon zircon ( $27.7 \pm 2.5$  Ma ( $2\sigma$ ;  $n$   
181  $= 5$ ) were analyzed as age standards.

182 After  $^4\text{He}$  analysis, the samples were unloaded from the laser chamber and dissolved for  
183 U and Th analysis. The apatites in their Pt capsules were loaded into 2 ml polypropylene vials  
184 and dissolved using 25  $\mu\text{l}$  of 50 % nitric ( $\text{HNO}_3$ ) acid that contained  $\sim 5$  ng of  $^{230}\text{Th}$  and  $\sim 15$  ng  
185  $^{235}\text{U}$ , which is used as a spike (Evans et al., 2005). The zircons require a more intense dissolution  
186 procedure, utilizing high temperature and pressure Parr digestion vessels, and concentrated  
187 hydrofluoric (HF), nitric ( $\text{HNO}_3$ ), and hydrochloric (HCl) acids, which was modified slightly  
188 from the procedure described by Reiners (2005). After dissolution, samples, together with  
189 specially prepared batches of spiked standard solutions, were analyzed for  $^{238}\text{U}$  and  $^{232}\text{Th}$   
190 concentrations on a ThermoElectron X-series inductively coupled plasma source mass

191 spectrometer in the W.M. Keck Foundation Laboratory for Environmental Biochemistry at ASU.  
192 Analytical errors were regularly between 1.0-2.6% ( $1\sigma$ ) for both  $^{238}\text{U}$  and  $^{232}\text{Th}$ , but in some  
193 apatites the errors were significantly larger (up to 12%;  $1\sigma$ ; Table 1) due to the lower  $^{238}\text{U}$  and  
194  $^{232}\text{Th}$  concentrations.

195 The  $^4\text{He}$ ,  $^{238}\text{U}$ , and  $^{232}\text{Th}$  data were then used to calculate raw dates using an iterative  
196 approach to solving the (U-Th)/He age equation as documented in Farley et al., (1996). Within  
197 this calculation, the concentration of  $^{235}\text{U}$  is derived from the measured  $^{238}\text{U}$  concentration using  
198 the known ratio of these isotopes in nature. The raw ages were then corrected for alpha ejection  
199 losses following the models of Farley et al. (1996) for apatite, and Hourigan et al. (2005) for  
200 zircon, assuming a homogeneous distribution of U and Th throughout the crystals. Analytical  
201 errors were propagated, but no errors were assigned to the values input into the alpha ejection  
202 correction calculation following standard protocols used in the (U-Th)/He dating community. (U-  
203 Th)/He dates for a single non-detrital sample are frequently overdispersed with regards to what  
204 would be expected from their analytical errors. This dispersion is assumed to be associated in  
205 part with the assumptions of the FT correction (ie. homogeneity of U-Th throughout the crystal,  
206 and perfect geometric shape) and can be quite dramatic especially for zircon, e.g., Hourigan et al.  
207 (2005), but for well-behaved samples, this is observed to be  $\sim 2-4\%$   $2\sigma$ . The analyses yielded a  
208 total of 10 (from 10) successful zircon ages, and 12 (from 22) successful apatite ages. The lower  
209 success rate in the apatites may have been due to undetected mineral inclusions ( $n = 1$ ; observed  
210 from subsequent high  $^4\text{He}$  re-extraction values, indicating the presence of inclusions), low  
211 concentrations of U and Th ( $n = 0$ ), or extremely low  $^4\text{He}$  contents in many of the grains ( $n = 9$ ;  
212 Table 1).

**213 Methods – Modeling of <sup>4</sup>He diffusive loss via impact-related heating of apatite and zircon**

214 Modelling of <sup>4</sup>He diffusive loss from minerals requires knowledge of the <sup>4</sup>He diffusion  
215 parameters, the sizes of the grains, and the temperatures and durations of the thermal events  
216 associated with the formation of an impact structure. We have determined durations and  
217 temperatures for each of the dominant impact-formation stages associated with the Monturaqui  
218 impact structure, and used the grain diameters from the (U-Th)/He analyses of the apatites and  
219 zircons.

220 During a bolide impact event, the short-lived initial contact and compressional stage  
221 (~0.0003-1 second for 0.35-250 km final crater diameters<sup>1</sup>) between the impactor and target  
222 rocks can generate temperatures in excess of 10,000 K near the impact point, with surrounding  
223 target rocks typically reaching temperatures of 500-3000°C (French 1998; Collins et al 2005). To  
224 calculate the duration of the initial compression stage for Monturaqui, we used the following  
225 equation:

$$226 \tau = d/V_i \quad [1]$$

227 where  $\tau$  is the duration of contact,  $d$  is the projectile diameter, and  $V_i$  is the impact velocity. In  
228 order to determine a Fe-Ni projectile diameter we used two online programs utilizing Pi-scaling,  
229 with the following parameters for the Monturaqui impact structure: final rim-to-rim crater  
230 diameter (350 m), transient crater diameter (224 m), impact velocity (18 km/s; Gillet and El  
231 Goresy, 2013), impact angle (45°), acceleration of gravity (9.8 m/s<sup>2</sup> for Earth), and target and  
232 projectile densities of 3.0 and 8.0 g/cm<sup>3</sup>, respectively. The first online calculator used was *Crater*  
233 (Melosh and Beyer, 2002), which yielded a projectile diameter of 4.9 m and a total crater

---

<sup>1</sup> Calculated using the *Crater* software (Melosh and Beyer, 2002) and equation [1], with bolide diameters of 4.9 m and 9.3 km, an impact velocity of 18 km/s, an impact angle of 45°, and projectile and target densities ranging from 8.0-1.5 and 3.0-1.5 g/cm<sup>3</sup>, respectively.

234 formation time of 2.4 seconds. The second online impact crater calculator used was *Impact and*  
235 *Explosion Effects*, version 4.02 (Holsapple, 2020). Using the same parameters listed above, a  
236 projectile diameter of 7.8 m and a total crater formation time of 2.3 seconds was determined.  
237 The estimated energy from the impact crater formation was calculated to range from  $8 \times 10^{13}$  to 2  
238  $\times 10^{14}$  J (19-38 kt TNT).

239         Compared to the short-duration initial contact and compression stage, the subsequent  
240 adiabatic decompression associated with shock metamorphism affects target rocks for longer  
241 durations (milliseconds to minutes) with temperatures ranging from ~900-2500°C and up to  
242 5,000°C (Walton et al., 2006; Schwenger et al. 2008; Stöffler et al., 2018). Dynamic  
243 crystallization experiments in meteorites produced shock-melt pockets and shock veins and  
244 yielded shock metamorphism durations in the range of 8-17 minutes (Walton et al., 2006;  
245 Schwenger et al., 2008), to shorter durations of 1.75 minutes to 1.2 seconds (Shaw and Walton,  
246 2013), with an estimated minimum duration of ~0.2 seconds (Beck et al., 2007). As the total  
247 formation time of the Monturaqui impact structure is determined to be 2.3-2.4 seconds, we have  
248 modelled the shock metamorphism stage in apatite and zircon grains using maximum and  
249 minimum durations of 1 and 0.1 seconds, respectively (Table 2).

250         Shock metamorphism at Monturaqui is estimated to range from <10 GPa up to very high  
251 shock pressures of 65 GPa (Bunch and Cassidy, 1972), which can result in post-shock  
252 temperatures of ~1500-1700°C (Stöffler et al., 2018). As no decomposition or melting was  
253 observed in the apatite and zircon grains from Monturaqui, both compression and shock  
254 metamorphism temperatures of 1500 and 1600°C were used in the modeling. A maximum  
255 temperature of 1600°C was estimated as this is just below the apatite melting temperature (1608-

256 1670°C; Bhatnagar, 1969) and it is also lower than the decomposition temperature of zircon  
 257 (1690°C; Finch and Hanchar, 2003).

258 Post-impact hydrothermal processes can affect target rocks with temperatures of ~100-  
 259 400°C, and can last for thousands to millions of years in medium to large impact structures  
 260 (Newsom et al., 1986; Parnell et al., 2005; Rasmussen et al., 2019; Kring et al., 2020). However,  
 261 due to the small size of the Monturaqui impact crater and the lack of information on post-impact  
 262 hydrothermal temperatures and durations in such small craters, no <sup>4</sup>He loss modelling was  
 263 performed on this impact crater stage.

264 Therefore, modeling of <sup>4</sup>He losses were undertaken for apatites and zircons for the initial  
 265 contact and compression stage using durations of 0.00027 and 0.00043 seconds, temperatures of  
 266 1500 and 1600°C, apatite grain radii of 27.4, 52.9 and 32.9 μm (minimum, maximum and the  
 267 average of the 2 reset grains; Table 1), and zircon grain radii of 24.8, 40.6, and 40.6 μm  
 268 (minimum, maximum and the radii of the single reset zircon grain; Table 1). Longer durations of  
 269 0.1 and 1.0 seconds were used for the shock metamorphism decompression stage, with  
 270 temperatures of 1500 and 1600°C. An author-written MathCAD version 14 program was used  
 271 (modified from Wartho et al., 2003), with the appropriate <sup>4</sup>He diffusion parameters and diffusion  
 272 geometries (cylinder for apatite and sphere for zircon; Farley, 2000; Reiners et al., 2004; Table  
 273 2) and the diffusion equations of Crank (1975) listed below.

274 The fractional loss of <sup>4</sup>He from a sphere (zircon) was calculated using the following  
 275 equation:

$$276 \quad 1 - \frac{6}{\pi^2} \sum_n \frac{1}{n^2} \exp\left(\frac{-Dn^2\pi^2 t}{a^2}\right) \quad [2]$$

277 where  $a$  is the grain radius (cm),  $t$  is time (seconds), and  $D$  is the activation energy calculated  
 278 from the following equation:  $D = D_o \exp\left(-\frac{E}{RT}\right)$ , where  $D_o$  is the frequency factor ( $\text{cm}^2/\text{s}$ ),  $E$  is  
 279 the activation energy (cal/mol), and  $R$  is the gas constant (1.987 cal/mol).

280 The fractional loss of He from a cylinder (apatite) was calculated using the following  
 281 equation:

$$282 \quad 1 - \sum_n \left( \frac{4}{a^2 (\alpha_n)^2} \right) \exp(-D (\alpha_n)^2 t) \quad [3]$$

283 where the integer  $\alpha_n$  is the root of  $J_0(a\alpha_n) = 0$ , and  $J_0(x)$  is the Bessel function of first kind of  
 284 order zero. Because diffusion is strongly affected by grain size, the modelling used the smallest,  
 285 largest and average radii of the reset grains for the apatites and zircons (i.e., the R2 values in  
 286 Table 1; Table 2).

## 287 **Results: Optical and SE imaging, and EDS analyses**

288 EDS analyses of 8 grains indicated that they had zircon compositions, and the majority of  
 289 the grains were optically clear and had smooth, occasionally rounded surfaces (Figs. 2a-d).  
 290 Optical microscopy on two conjoined zircon grains showed that they were milky white and  
 291 completely opaque (Fig. 3a). SE imaging of these conjoined grains showed pervasive micro-  
 292 fractures and possible lamellae on the crystal surfaces of both grains (Figs. 3b-d), which were not  
 293 present in the other zircon grains (Figs. 2a-d). Total or partial opaqueness was also observed in  
 294 some optical images of the (U-Th)/He dated apatite and zircon grains (Figs. 4a-b and d-e).  
 295 Alternatively, we have evidence of clear apatite and zircon grains (Figs. 2a-d and 4c, f-j)  
 296 showing little evidence of shock metamorphism.

## 297 **Results: (U-Th)/He ages**

300 Ten zircon grains gave (U-Th)/He ages ranging from  $0.662 \pm 0.029$  to  $197.3 \pm 7.0$  Ma  
 301 ( $2\sigma$ ), and 12 apatite (U-Th)/He analyses yielded ages ranging from  $0.62 \pm 0.11$  to  $61.5 \pm 1.9$  Ma  
 302 ( $2\sigma$ , Figs. 4-5; Table 1). The three youngest ages yielded an inverse variance weighted mean age  
 303 of  $663 \pm 28$  ka ( $2\sigma$ ;  $n = 3$ ) with acceptable Mean Square of Weighted Deviates (MSWD = 2.4)  
 304 and Probability ( $P = 9.4\%$ ) values (Fig. 5).

305 The ‘calculated  $^4\text{He}$  losses’ from the 12 apatite and 10 zircon single grain analyses (Table  
 306 1) were calculated using the following equation:

$$307 \quad 100 - \frac{(\text{Single crystal apatite or zircon Age}_{\text{corr}} - 0.663 \text{ Ma}) * 100}{\text{Oldest apatite or zircon age} - 0.663 \text{ Ma}} \quad [4]$$

308 assuming maximum (U-Th)/He ages of 61.51 and 197.25 Ma, respectively, and 0.663 Ma as the  
 309 age of the impact event (this study). The target rock lithology is volumetrically dominated by  
 310 granite, and geochemical modeling of the compositions of the impactites suggested that the  
 311 impact melt was derived almost exclusively from the granite target (Ukstins Peate et al. 2010), so  
 312 we feel confident that the apatite and zircon grains in the impactite samples were obtained from  
 313 the local granite target rock, rather than the 3.2 Ma ignimbrites. This is confirmed by the overall  
 314 older (U-Th)/He ages of the apatites and zircons from the two impactites. Excluding the 4  
 315 youngest ages ( $0.62 \pm 0.11$  to  $2.309 \pm 0.071$  Ma;  $2\sigma$ ), the remaining 18 (U-Th)/He analyses all  
 316 yield ages older than the 3.2 Ma ignimbrite ( $4.69 \pm 0.31$  to  $197.3 \pm 7.0$  Ma;  $2\sigma$ ; Table 1).

317

### 318 **Results: Modelled impact resetting of the (U-Th)/He geochronological system**

319 For the initial contact and compression stage (0.00027 and 0.00043 seconds at 1500 and  
 320  $1600^\circ\text{C}$ ) the apatite minimum, maximum and average reset grain radii yielded modeled  $^4\text{He}$   
 321 diffusion losses of 61-85, 34-52 and 52–76%, respectively (Table 2). For the zircon grains in the

322 same contact-compression stage, modeling produced much smaller  $^4\text{He}$  loss values of 4.9-8.3,  
323 3.0-5.1 and 4.0-6.7% for the respective minimum, maximum and reset grain radii (Table 2).

324 Modeling of  $^4\text{He}$  losses in the apatites and zircons during the following adiabatic  
325 decompression shock metamorphism stage (0.1 to 1.0 seconds at 1500 and 1600°C), resulted in  
326 large  $^4\text{He}$  losses of 100% in all the apatite grains (minimum, maximum and average reset radii).  
327 Similarly, the modeled zircon grains yielded high  $^4\text{He}$  loss values ranging from 71-100, 49-99.7  
328 and 62-100% for the minimum, maximum and reset grain radii, respectively (Table 2).

329

### 330 Discussion

331 The (U-Th)/He zircon and apatite mean age of  $663 \pm 28$  ka ( $2\sigma$ ) obtained from this study  
332 agrees within  $2\sigma$  errors with the previously obtained age of  $590 \pm 60$  ka ( $1\sigma$ ) from quartz  
333 thermoluminescence dating, and an age range of ~500-780 ka inferred from paleomagnetic and  
334 cosmogenic nuclides studies for the Monturaqui impact crater (Verdugo and Cartes, 2000;  
335 Valenzuela et al., 2009. As both apatite and zircon (U-Th)/He analyses contribute to the  
336 youngest ages, the weighted mean age of this cluster most likely reflects the formation age of the  
337 impact structure. If the young ages had been the result of a later reheating or a slow cooling  
338 event, the distinctly different  $^4\text{He}$  diffusion parameters for apatite and zircon (Farley, 2000;  
339 Reiners et al., 2004) would not have yielded overlapping young ages, thus this gives us  
340 confidence that the  $663 \pm 28$  ka (U-Th)/He age is the Monturaqui impact structure formation age.

341 Milky white opaque zircon grains are a common indicator of shock metamorphism  
342 (Corfu et al., 2003). Owing to the milky opaque nature of two conjoined zircon grains in imaged  
343 but not dated grains (Fig. 3a), the presence of pervasive micro-fracturing and possible planar  
344 features or lamellae (Figs. 3b-d), plus partially or total opaque apatite grains from the (U-Th)/He



345 analyses (Figs. 4a-b, d-e), we suggest that some of the accessory minerals in the two impactite  
346 samples underwent shock metamorphism. Bunch and Cassidy (1972) and Ugalde et al. (2007)  
347 noted a spectrum of shock metamorphism textures in the Monturaqui impactite samples, ranging  
348 from completely unshocked regions, to weak shocking (10-25 GPa) observed as micro-  
349 deformation and planar features in quartz and feldspar, kinking of biotite, and multiple cleavage  
350 development in apatite grains. Moderate shock levels (25-50 GPa) were identified by partly to  
351 completely vitrified quartz, transformation of quartz to coesite, and conversion of feldspars to  
352 maskelynite, while high shock levels (50-65 GPa) were identified by the presence of vesiculated  
353 quartz and feldspar glasses, melting of biotite grains, and formation of brown and green impact  
354 glasses. Therefore, our observations of both shocked and unshocked apatite and zircon grains  
355 verify previous petrological observations regarding the heterogeneous nature of the shock regime  
356 experienced by the Monturaqui impactite samples (Bunch and Cassidy, 1972; Ugalde et al.,  
357 2007).

358         There were no observable correlations between the (U-Th)/He single crystal ages and (i)  
359 optical microscope imaging of the apatite and zircon grains in terms of opaque versus clear  
360 grains (Fig. 4, Table 1), (ii) the Th/U ratios (Fig. 6a), or (iii) the apatite and zircon grain radii  
361 (Fig. 6b). If the compression and shock metamorphism temperatures had been uniform across the  
362 impactite samples, then we would expect a more similar range of ages for the zircon and apatite  
363 populations, and the smaller grains would have undergone more  $^4\text{He}$  loss than the larger grains.  
364 However, no correlation of the (U-Th)/He ages with grain size was observed (Fig. 6b), which  
365 again indicates that the heating of the impactites was heterogeneous. The three youngest apatite  
366 and zircon (U-Th)/He ages ( $0.616 \pm 0.107$  to  $0.845 \pm 0.183$  Ma;  $2\sigma$ ) were sourced from the same  
367 sample (CIUP 08100 with ages up to  $61.5 \pm 1.9$  Ma;  $2\sigma$ ), located on the south flank of the crater

368 (Fig. 1). The second sample (CIUP 08099), located on the SE flank of the crater (Fig. 1) yielded  
369 older apatite and zircon ages ranging from  $4.7 \pm 1.7$  to  $197.3 \pm 7.0$  Ma ( $2\sigma$ ). The 0.616-197 Ma  
370 age range in all the grains from both samples is interpreted to reflect partial to complete resetting  
371 of the (U-Th)/He ages in apatite and zircon grains, which may be due to heterogeneous heating  
372 effects (Fig. 5). These new results further substantiate the heterogeneous nature of the different  
373 pressure-temperature environments experienced by these impactite breccia samples. A regional  
374 apatite (U-Th)/He and fission track study from the nearby Salar de Atacama Basin (Henrique et  
375 al., 2018) yielded similar ages to our oldest Monturaqui apatite and zircon (U-Th)/He ages. (U-  
376 Th)/He ages from the Quimal Intrusive (~100 km NNW of Monturaqui) ranged from  $38.0 \pm 1.2$   
377 to  $70.1 \pm 3.2$  Ma ( $2\sigma$ ;  $n = 10$ ), which overlap within  $2\sigma$  errors with our 4 oldest (U-Th)/He  
378 apatite ages ( $35.6 \pm 2.8$  to  $61.5 \pm 1.9$  Ma; Table 1). Apatite fission track central ages of  $57 \pm 13$   
379 to  $170 \pm 37$  Ma ( $2\sigma$ ;  $n = 2$ ), collected from two samples ~ 45 km NW of Monturaqui are similar  
380 to our 3 oldest Monturaqui zircon (U-Th)/He ages of  $53.5 \pm 2.0$  to  $197.3 \pm 7.0$  Ma, despite the  
381 differences in the zircon He closure temperature (~200°C; Reiners et al., 2004) and the fission  
382 track annealing temperature (~120°C; zircon (U-Th)/He = ~200°C; Ketcham et al., 1999).  
383 Therefore, the Henrique et al. (2018) apatite (U-Th)/He and fission-track regional cooling ages  
384 verify our oldest apatite and zircon (U-Th)/He ages, and gives us confidence in our calculated %  
385  $^4\text{He}$  loss values (Table 1), which are directly compared to our modeled %  $^4\text{He}$  loss values (Table  
386 2; Fig. 7).

387 Comparing modeled amounts of  $^4\text{He}$  loss at temperatures of 1500 and 1600°C from the  
388 (i) initial contact and compression and (ii) the adiabatic decompression and shock metamorphism  
389 stages indicates that the latter is probably the main contributor to the partial to complete resetting  
390 of the (U-Th)/He apatite and zircon ages in the Monturaqui impactites (Fig. 7). This is especially

391 true for the zircons, as  $^4\text{He}$  diffusion is observably slower in zircon compared to apatite (Fig. 8),  
392 and only the longer duration shock metamorphism stage is capable of producing 100%  $^4\text{He}$   
393 resetting in zircons (Fig. 7).

394 Modeling of  $^4\text{He}$  losses in 25 and 50  $\mu\text{m}$  apatite and zircon grains (commonly utilized for  
395 (U-Th)/He dating), at temperatures of 1500 and 1600°C, indicates that the very short duration  
396 (0.3-0.4 milliseconds) initial contact and compression stage is insufficient to completely reset  
397 (U-Th)/He systematics (Figs. 7-8; Table 2). A contact and compression stage duration of >0.51  
398 seconds with a temperature of 1600°C would be sufficient to reset the (U-Th)/He systematics in  
399 25 and 50  $\mu\text{m}$  radii apatites and smaller zircon grains (25  $\mu\text{m}$  radius; Fig. 8). However, this  
400 would require a very large impactor (~9.3 km diameter) resulting in final rim-to-rim impact  
401 crater diameters of ~130-290 km (using projectile and target rock densities of 1.5-8.0  $\text{g}/\text{cm}^3$  in  
402 the *Crater* software (Melosh and Beyer, 2002)).

403 In contrast, modelling of the longer duration (1.4-0.76 seconds at 1500 and 1600°C,  
404 respectively) decompression and shock metamorphism stage at Monturaqui indicates that it is  
405 capable of causing  $^4\text{He}$  losses of 100% in apatites and 49-100% in zircons (Fig 7; Table 2). This  
406 agrees relatively well with the calculated  $^4\text{He}$  losses of 14-100 and 28-100% in the Monturaqui  
407 apatites and zircons, respectively (Fig. 7). The large variability of the shock levels in  
408 Monturaqui impactite samples could also explain the observed range of partial to total resetting  
409 in the (U-Th)/He apatite and zircon ages (Figs. 5 and 7).

410 Assuming temperatures of 1500 and 1600°C during both the compression and shock  
411 metamorphism stages,  $^4\text{He}$  loss modeling of the reset zircon grain (grain # z003; 30.5  $\mu\text{m}$  radius;  
412 Table 1) yields combined compression and shock metamorphism durations of 1.4 to 0.76  
413 seconds to achieve 100%  $^4\text{He}$  loss, respectively (Fig. 8). If the 55.4-99.6% calculated  $^4\text{He}$  losses

414 in the remaining 8 zircon grains (ignoring the 0% loss zircon grain, which requires a temperature  
415 of  $<270^{\circ}\text{C}$ ) are assumed to be due to temperature inhomogeneities in the two samples, then  
416 reverse modeling of the calculated %  $^4\text{He}$  losses and grain radii from each of the (U-Th)/He  
417 analyses (Table 1) yields a temperature range of  $1015\text{-}1529^{\circ}\text{C}$ , which would be equivalent to  
418 shock pressures of  $50\text{-}60\text{ GPa}$  (Stöffler et al., 2018) in the two impactite samples. Similar reverse  
419 modelling of the  $14.0\text{-}100\%$  calculated  $^4\text{He}$  losses in the 11 analysed apatite grains (ignoring the  
420  $0\%$  loss apatite grain, which requires a temperature of  $<210^{\circ}\text{C}$ ) yields a lower temperature range  
421 of  $512\text{-}902^{\circ}\text{C}$ , which is equivalent to lower shock pressures of  $35\text{-}45\text{ GPa}$  (Stöffler et al., 2018).

422 We have shown that the (U-Th)/He method can be successfully applied to the dating of  
423 variably shocked impactite samples from impact structures, including very small  $<1\text{ km}$  diameter  
424 craters, which have thus far yielded few geochronological ages due to lack of suitable material  
425 for the more routinely used U-Pb and  $^{40}\text{Ar}\text{-}^{39}\text{Ar}$  techniques (i.e., impact melts or tektites). Based  
426 on this study and previous (U-Th)/He studies of impact structures (van Soest et al., 2011; Wartho  
427 et al., 2012; Young et al., 2013; Wielicki et al. 2014; Biren et al., 2014, 2016, 2019), we  
428 recommend analyzing numerous single grains (i.e.,  $>20\text{-}30$ ), especially for non-impact melt  
429 material, distal ejecta, and smaller impact structures, where only  $\sim 10\%$  of grains are found to be  
430 reset. We also recommend analyzing multiple mineral phases to confirm the validity of the  
431 youngest ages as impact event ages.

432

### 433 **Conclusions**

434 The  $350\text{ m}$  diameter Monturaqui impact structure has been dated at  $663 \pm 28\text{ ka}$  ( $2\sigma$ )  
435 using the (U-Th)/He geochronological technique. The dating method is capable of expanding the  
436 field of geochronologically datable impact materials and successfully determining accurate ages

437 from very small (<1 km diameter) impact structures, by utilizing the relatively rapid diffusion  
438 and low-temperature resetting properties of the  $^4\text{He}$  daughter product in U- and Th-bearing  
439 minerals.

440         Modeling of  $^4\text{He}$  losses in apatite and zircon grains from the Monturaqui impact crater  
441 suggests that the short duration initial contact and compression stage is not capable of causing  
442 sufficient resetting of the (U-Th)/He ages, especially in the more retentive zircons. However, the  
443 longer duration adiabatic decompression and shock metamorphism stage can cause complete  
444 resetting of the (U-Th)/He systematics in both apatite and zircon grains from this 350 m diameter  
445 impact structure.

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638 **Figure and table captions**

639 **Fig. 1.** (a) The location of Monturaqui impact structure in Northern Chile is shown with a star.

640 (b) Geologic map of Monturaqui with two marked impactite sample locations (circles) that were  
641 selected for (U-Th)/He dating. The asymmetric base of crater is filled with lake sediment  
642 deposits. After Ugalde et al. (2007), Ukstins Peate et al. (2010) and Rathbun et al. (2017).

643 **Fig. 2.** Optical (small white boxed images) and secondary electron photomicrographs of four  
644 unshocked zircon grains from impactite samples CIUP 08099 (a-b) and CIUP 08100 (c-d).

645 **Fig. 3.** (a) Optical and (b-d) secondary electron photomicrographs of shocked conjoined zircon  
646 crystals (CIUP 08099). The white boxes in Fig. 3b indicate the positions of the  
647 photomicrographs shown in Figs. 3c-d. The dashed black and white lines in Figs. 3c-d indicate a  
648 conjugate network of subplanar and occasionally curved micro-fractures (2-13  $\mu\text{m}$  in length),  
649 and the black arrows indicate lamellae-like features (2  $\mu\text{m}$  in length) in these zircon grains.

650 **Fig. 4.** Optical photomicrographs (a-j) of some (U-Th)/He dated apatite and zircon grains from  
651 the two impactite samples, with associated (U-Th)/He ages and  $2\sigma$  uncertainties, and grain  
652 lengths and diameters.

653 **Fig. 5.** Relative probability plots for (U-Th)/He single crystal apatite and zircon ages from two  
654 impactite samples. The dashed box in the main plot indicates the area demarked for the more  
655 detailed insert plot of the younger 0-10 Ma (U-Th)/He ages.

656 **Fig. 6.** (a) Th/U ratios versus (U-Th)/He single crystal ages for apatites and zircons. (b) Apatite  
657 and zircon grain radii ( $R_2$  in Table 1) versus (U-Th)/He single crystal ages. The squares indicate  
658 the 3 youngest apatite and zircon (U-Th)/He ages used to calculate the Monturaqui weighted  
659 mean impact age.

660



661 **Fig. 7.** Comparison of (a) modeled %  $^4\text{He}$  losses (Table 2) from zircon and apatite grains during  
662 the initial contact and compression stage (0.27-0.43 milliseconds duration at 1500 and 1600°C),  
663 and the adiabatic decompression and shock metamorphism stage (0.1-1 seconds duration at 1500  
664 and 1600°C), versus (b) calculated %  $^4\text{He}$  losses (Table 1) for the Monturaqui impact structure.

665 **Fig. 8.** Modeled %  $^4\text{He}$  loss from the 25 and 50  $\mu\text{m}$  radii apatite (black areas) and zircon (grey  
666 areas) grains typically used for (U-Th)/He analysis, heated from 1500-1600°C for varying  
667 durations. 100%  $^4\text{He}$  losses would be achieved with impact heating durations of 0.93 to 0.51  
668 seconds (25 $\mu\text{m}$  radius zircon) and 3.7 to 2.0 seconds (50 $\mu\text{m}$  radius zircon), at 1500 and 1600°C,  
669 respectively. Much shorter durations are required to achieve 100%  $^4\text{He}$  losses in apatites -  
670 0.0028 to 0.0017 (25  $\mu\text{m}$  radius) and 0.011 to 0.0068 seconds (50  $\mu\text{m}$  radius) at 1600 and  
671 1500°C, respectively. \* = Modeling of the 30.5  $\mu\text{m}$  radius 100% reset zircon grain (Table 1)  
672 from Monturaqui, yielding 100%  $^4\text{He}$  losses with durations of 1.4 seconds (1500°C) and 0.76  
673 seconds (1600°C).

674 **Table 1.** (U-Th)/He geochronological analyses of apatites and zircons from the Monturaqui  
675 impact crater.

676 **Table 2.** Modeling of  $^4\text{He}$  loss from apatite and zircon grains during Monturaqui impact crater-  
677 forming stages.

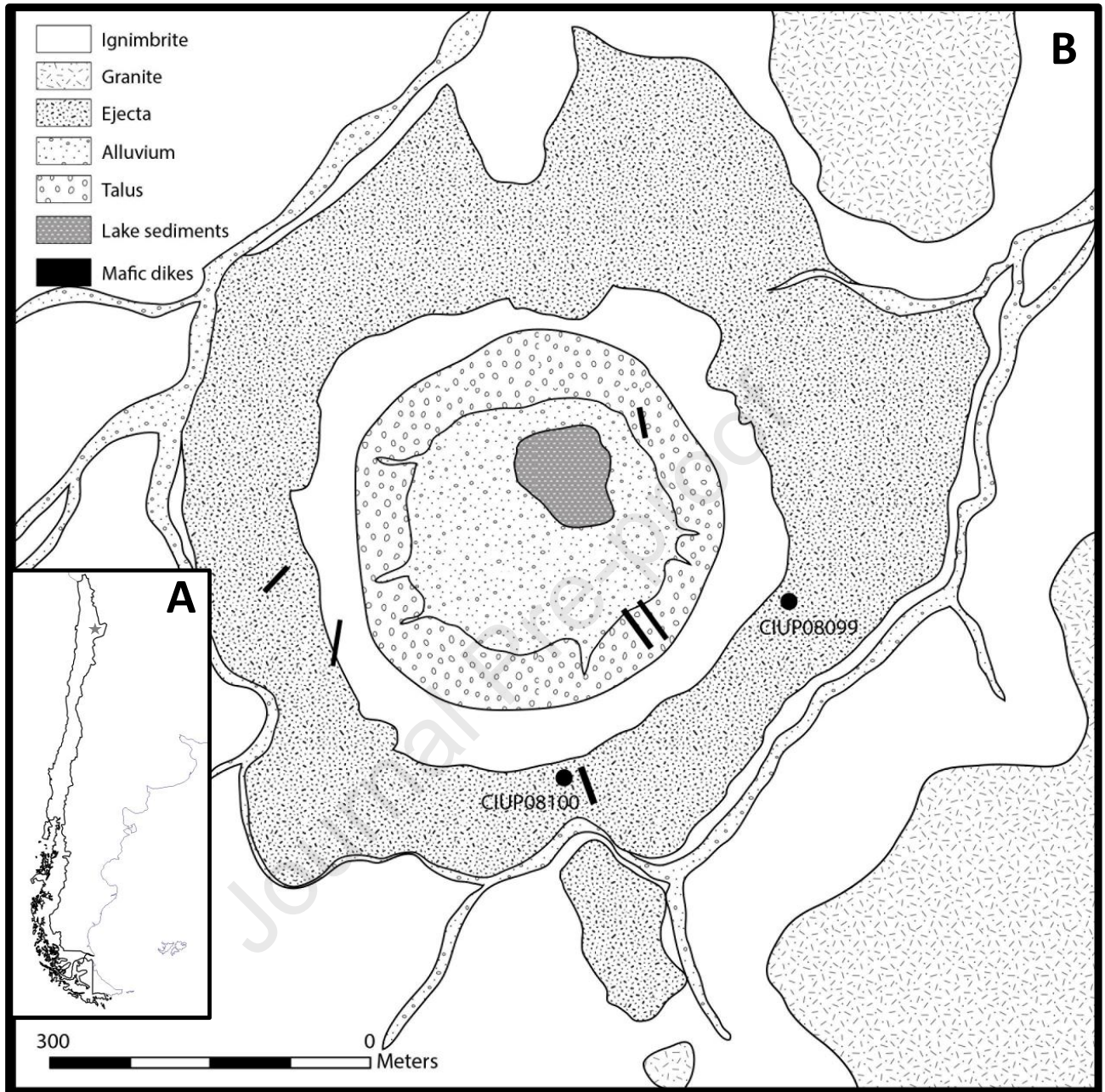
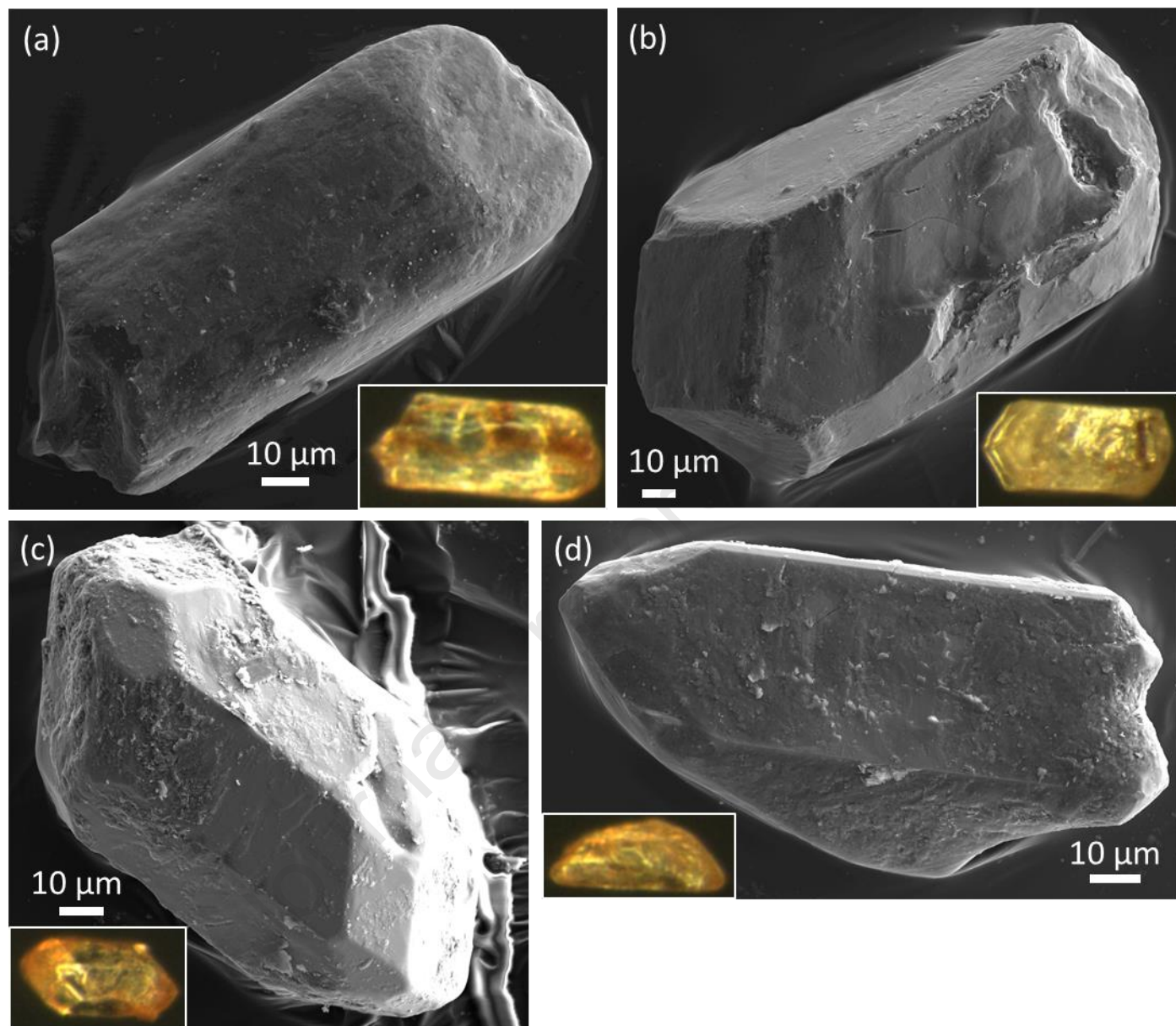
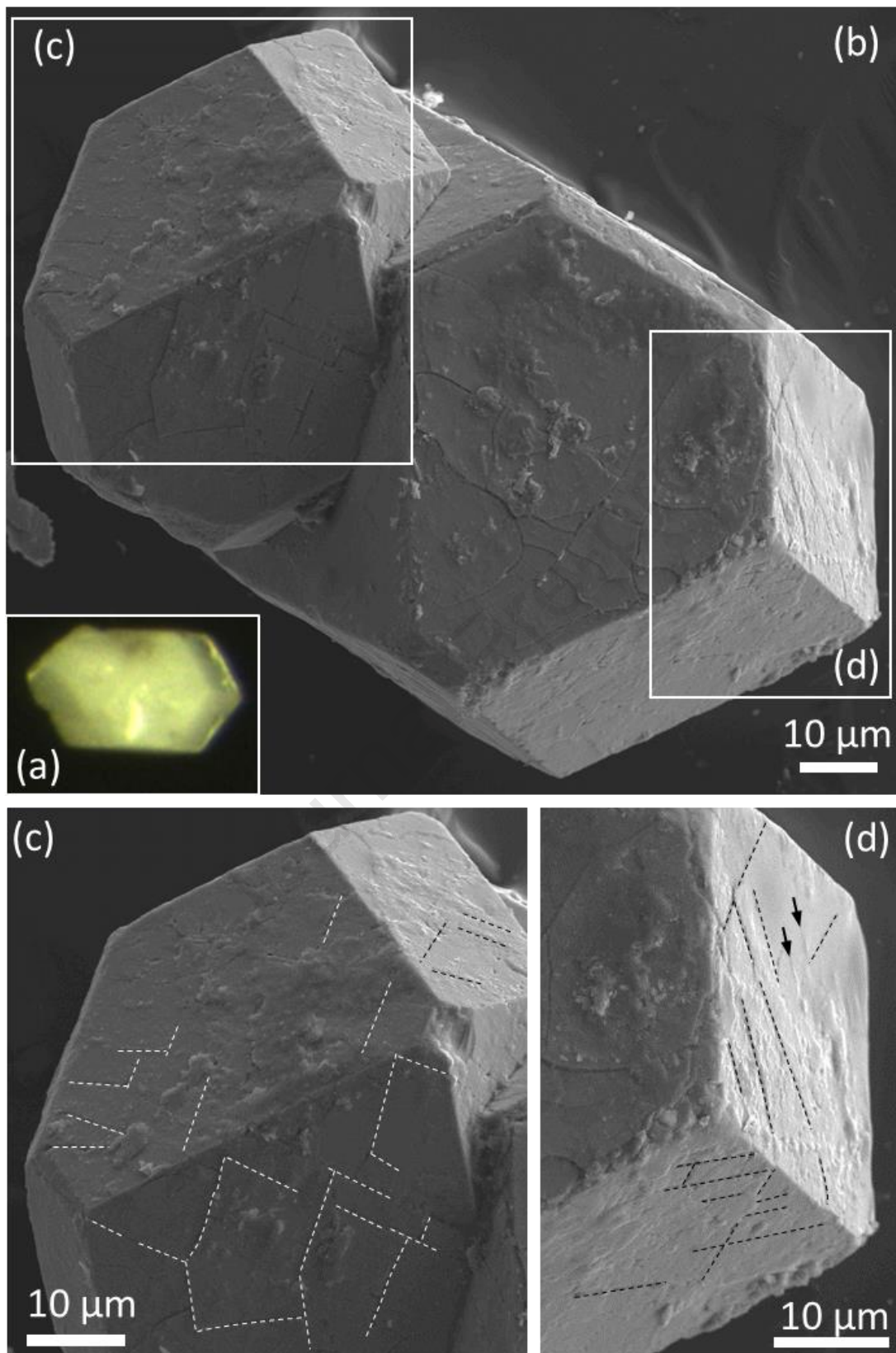
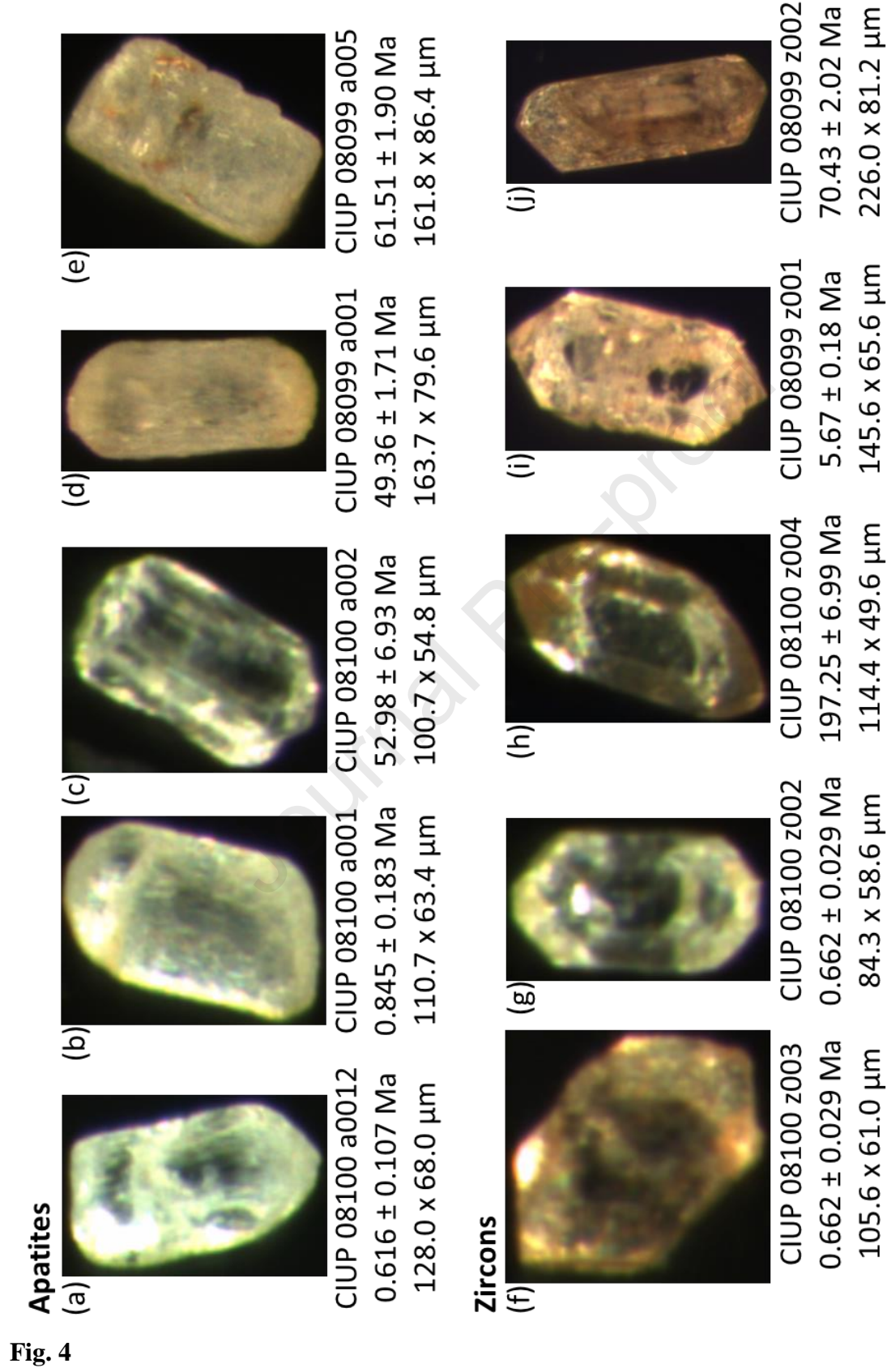


Fig. 1

**Fig. 2**

**Fig. 3**



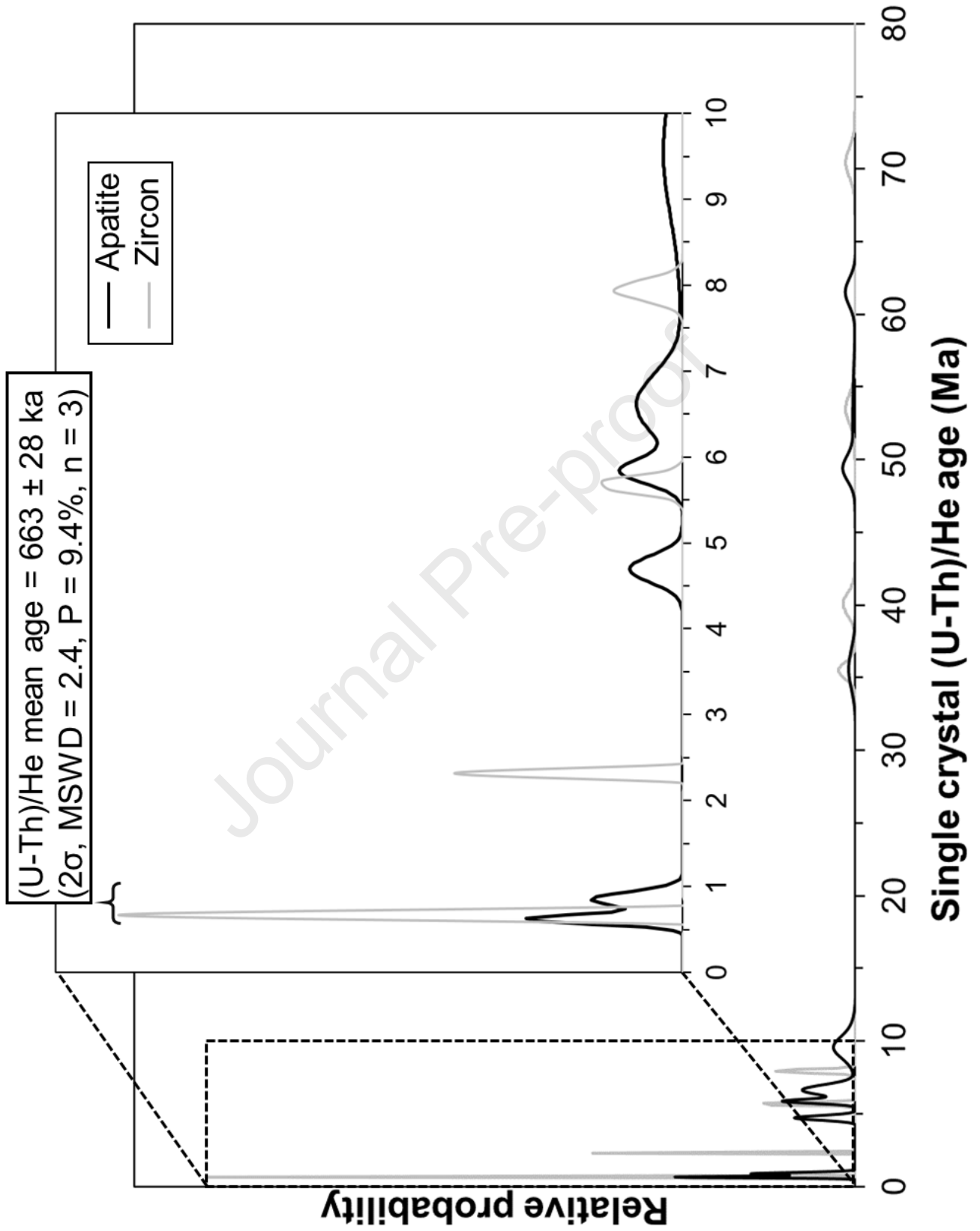


Fig. 5

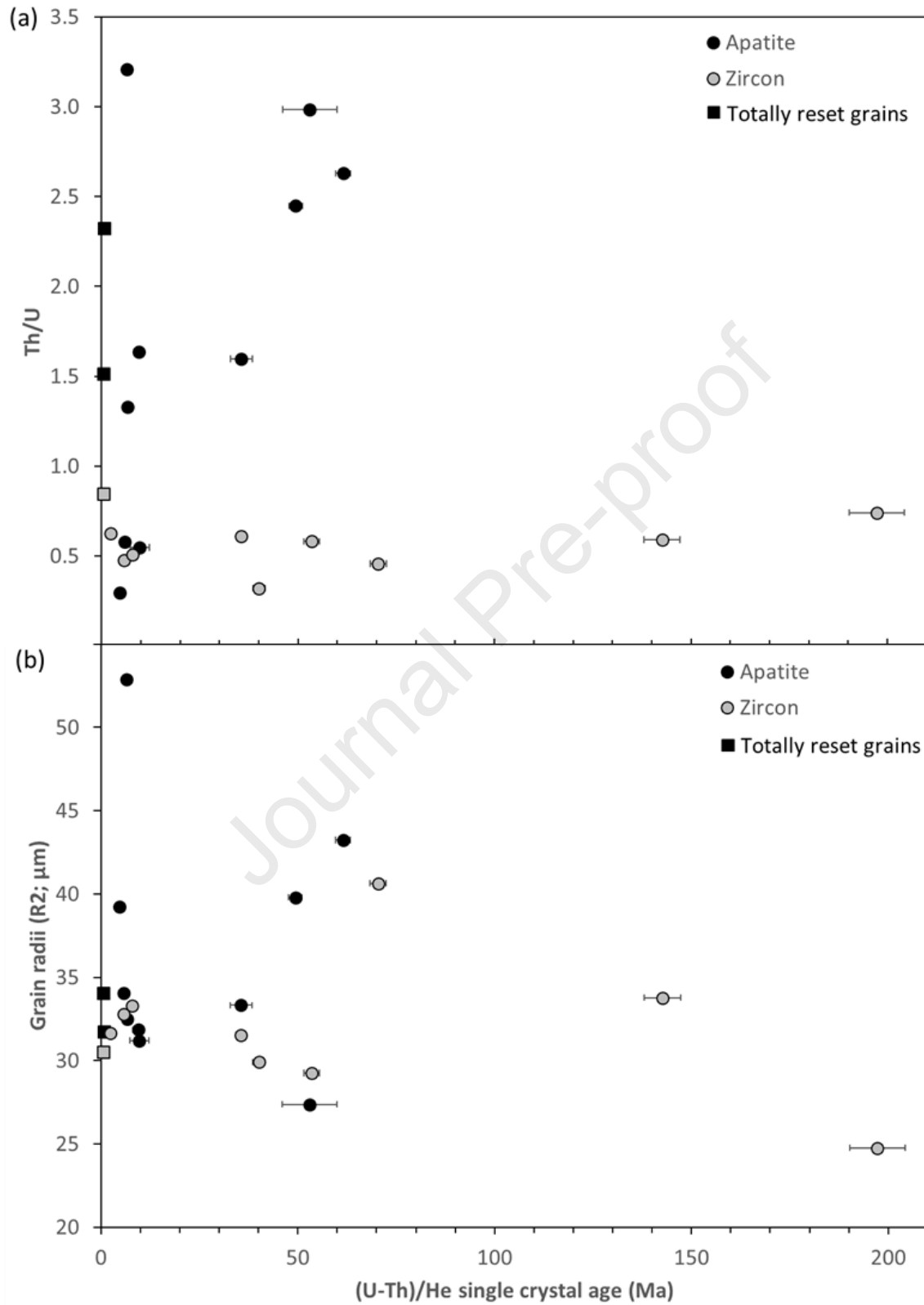


Fig. 6

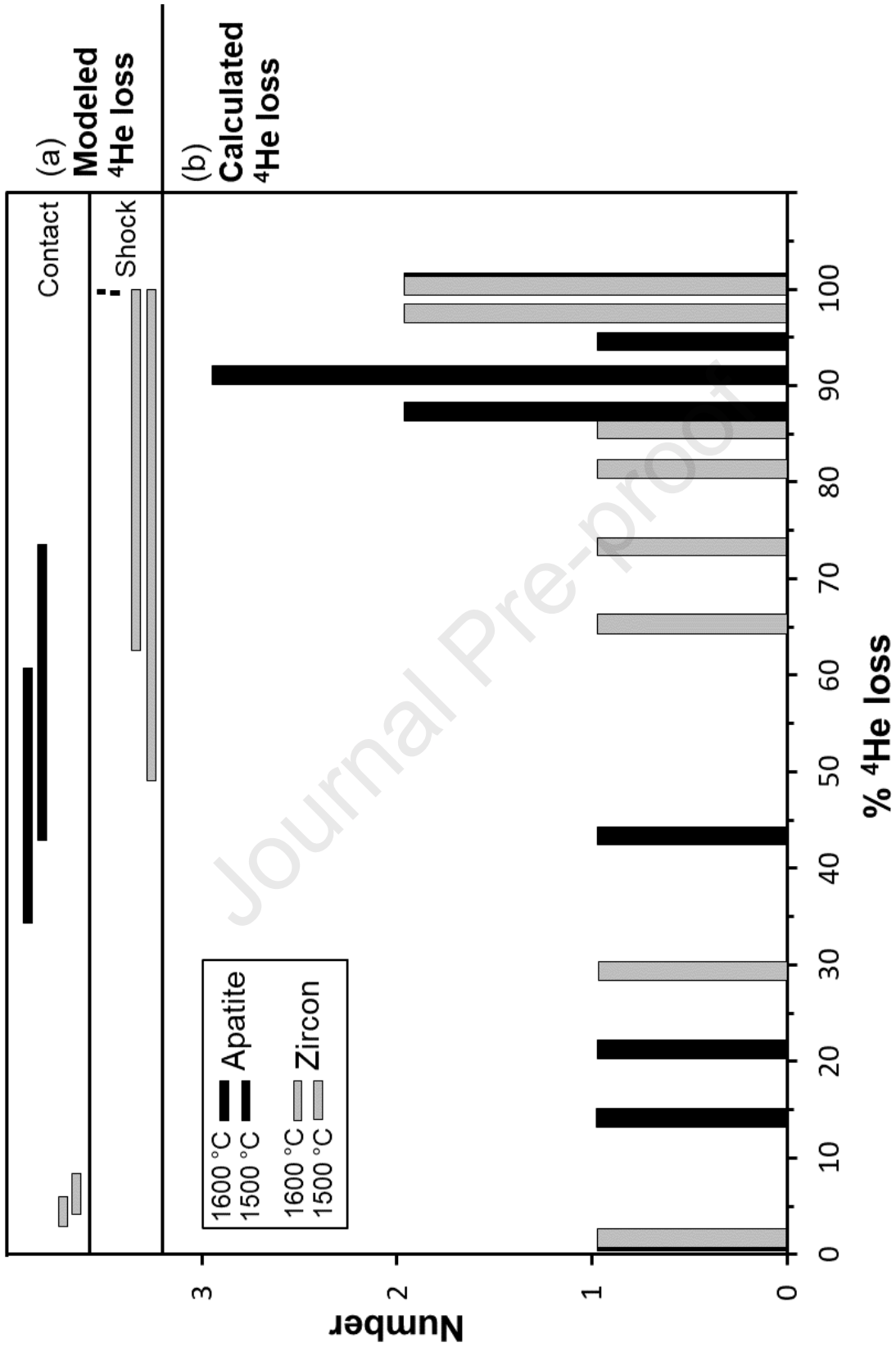


Fig. 7



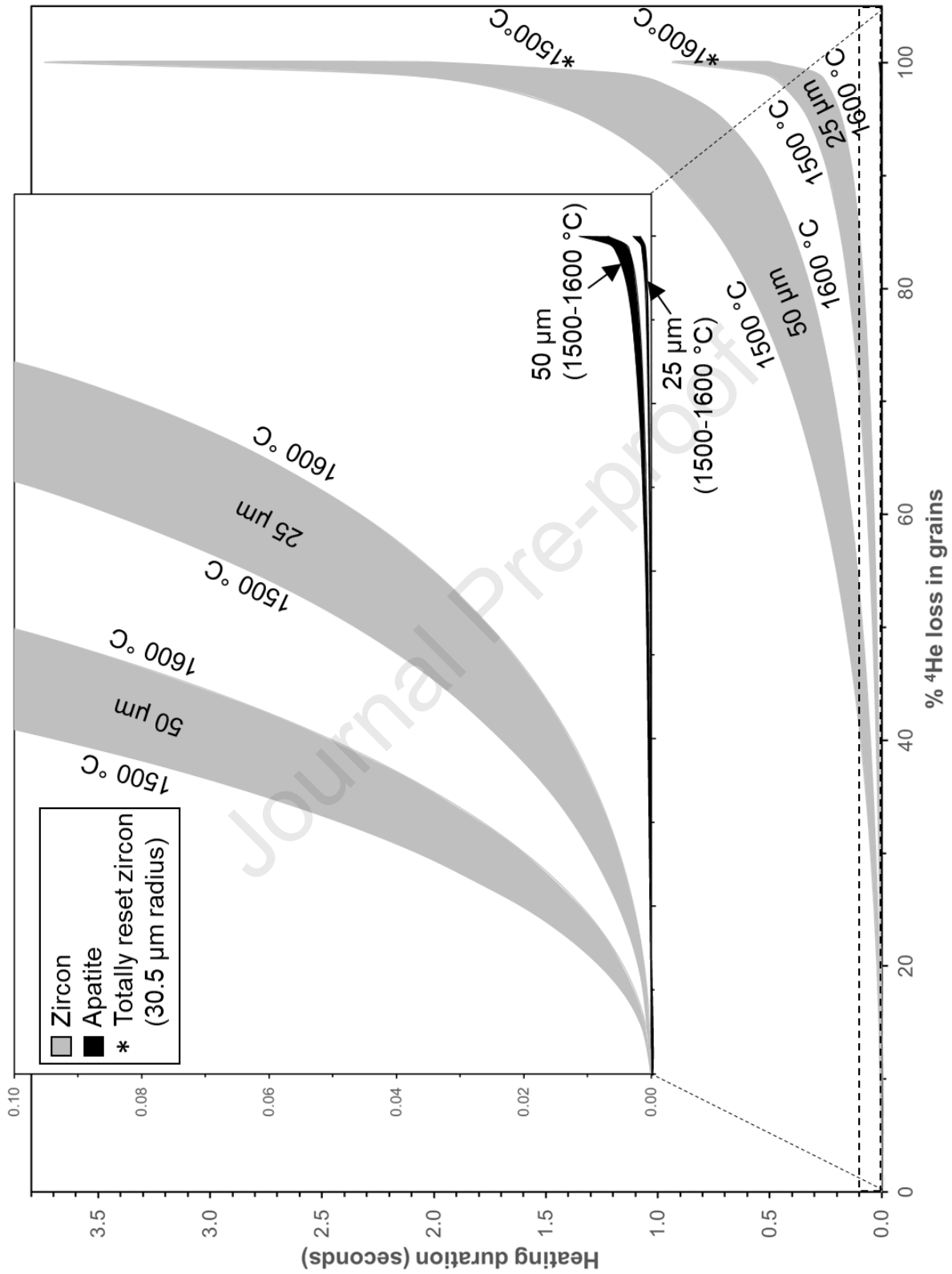


Fig. 8

Table 1: (U-Th)/He geochronological analyses of apatites and zircons from the Monturaqui impact crater

Sample	Grain# <sup>a</sup>	[ <sup>238</sup> U] <sup>b</sup>	Error <sup>c</sup>	[ <sup>232</sup> Th] <sup>b</sup>	Error <sup>c</sup>	Th/U	[ <sup>4</sup> He] <sup>b,d</sup>	Error <sup>c</sup>	Age <sub>raw</sub> <sup>e</sup>	Ma	Error <sup>c</sup>	R1 <sup>f</sup>	R2 <sup>g</sup>	L <sup>g</sup>	T1 <sup>h</sup>	T2 <sup>h</sup>	β <sup>i</sup>	F <sub>T</sub>	Mean <sup>k</sup>	Age <sub>corr.</sub> <sup>k</sup>	Error <sup>c</sup>	Calculated	
		pmol	1σ	pmol	1σ	pmol	fmol/pmol	1σ	Ma	1σ	Ma	μm	μm	μm	μm	μm	(1/μm)		Ma	2σ	% <sup>l</sup>	He loss	
<b>Apatites</b>																							
CIUP 08099	a004	0.0860	0.0015	0.2091	0.0035	2.45	0.510	0.016	2.96	0.10	-	39.2	114.6	-	-	-	0.0763	0.63	4.69	0.31	93.4		
	a008	0.2116	0.0028	0.0623	0.0019	0.30	1.04	0.02	3.58	0.09	-	34.1	127.2	-	-	-	0.0835	0.61	5.83	0.28	91.5		
	a001	0.1386	0.0023	0.3616	0.0053	2.63	9.24	0.12	32.37	0.56	-	39.8	163.7	-	-	-	0.0703	0.66	49.36	1.71	20.0		
	a005	0.5148	0.0054	0.2959	0.0048	0.58	31.65	0.38	42.17	0.65	-	43.2	161.8	-	-	-	0.0658	0.69	61.51	1.90	-		
CIUP 08100	a012	0.1428	0.0069	0.3291	0.0046	2.32	0.104	0.008	0.370	0.032	-	34.0	128.0	-	-	-	0.0835	0.60	0.616	0.107	100.1		
	a001	0.1047	0.0022	0.3103	0.0086	2.98	0.109	0.012	0.480	0.052	-	31.7	110.7	-	-	-	0.0909	0.57	0.845	0.183	99.7		
	a014	0.0868	0.0052	0.1145	0.0021	1.33	0.655	0.018	4.50	0.24	-	52.9	102.7	-	-	-	0.0631	0.69	6.50	0.70	90.4		
	a005	0.0607	0.0025	0.0985	0.0019	1.63	0.416	0.017	3.89	0.20	-	32.5	108.4	-	-	-	0.1012	0.58	6.70	0.69	90.1		
	a006	0.0362	0.0028	0.0573	0.0027	1.60	0.322	0.013	5.08	0.36	-	31.9	69.8	-	-	-	0.1012	0.53	9.51	1.35	85.5		
	a008	0.0226	0.0027	0.0123	0.0012	0.548	0.175	0.010	5.36	0.66	-	31.2	80.2	-	-	-	0.099	0.55	9.74	2.40	85.1		
	a007	0.0788	0.0037	0.1184	0.0038	1.51	2.761	0.045	20.25	0.79	-	33.3	87.1	-	-	-	0.0923	0.57	35.57	2.78	42.6		
	a002	0.0144	0.0014	0.0457	0.0026	3.21	0.872	0.017	27.2	1.8	-	27.4	100.7	-	-	-	0.1043	0.51	52.98	6.93	14.0		
<b>Zircons</b>																							
CIUP 08099	z001	10.37	0.12	4.917	0.068	0.478	0.05841	0.00068	3.960	0.061	32.5	32.8	145.6	27.9	28.8	0.0751	0.70	5.67	0.18	97.5			
	z003	7.418	0.075	3.367	0.047	0.457	0.05763	0.00066	5.482	0.081	30.9	33.3	142.4	32.2	29.1	0.0771	0.69	7.93	0.23	96.3			
	z004	8.77	0.10	4.412	0.053	0.507	0.3103	0.0036	24.67	0.39	28.9	31.5	203.9	32.2	37.1	0.0759	0.70	35.49	1.13	82.3			
	z002	28.15	0.27	17.02	0.20	0.609	2.173	0.025	52.60	0.76	34.8	40.6	226.0	43.0	32.9	0.0619	0.75	70.43	2.02	64.5			
	z005	4.314	0.051	2.527	0.034	0.590	0.6214	0.0073	98.1	1.6	26.3	33.8	188.2	31.1	28.2	0.0777	0.69	142.64	4.56	27.8			
CIUP 08100	z003	2.614	0.037	1.628	0.027	0.627	0.001664	0.00029	0.4341	0.0093	29.8	30.5	105.6	24.1	23.7	0.0866	0.66	0.662	0.029	100.0			
	z001	13.47	0.14	7.772	0.096	0.581	0.03041	0.00037	1.553	0.024	28.7	31.7	126.1	20.4	22.7	0.0819	0.67	2.31	0.07	99.2			
	z005	0.793	0.014	0.663	0.017	0.842	0.03081	0.00038	25.36	0.50	26.1	29.9	116.7	34.1	31.0	0.0925	0.63	40.07	1.57	80.0			
	z002	0.975	0.016	0.717	0.017	0.741	0.04675	0.00056	31.87	0.59	23.3	29.3	84.3	19.9	21.0	0.1033	0.60	53.48	1.98	73.1			
	z004	1.943	0.027	0.619	0.016	0.321	0.3300	0.0039	122.1	2.2	27.9	24.8	114.4	33.0	31.3	0.0977	0.62	197.25	6.99	-			

<sup>a</sup>The missing grain numbers represent grains that were determined to contain inclusions, which were not detected during mineral picking. These were identified by the helium re-extraction failing to yield blank levels, which would be the case for inclusion-free apatites.

<sup>b</sup>Absolute concentrations measured for <sup>4</sup>He, <sup>238</sup>U, and <sup>232</sup>Th are used to calculate a "raw age" that does not reflect <sup>4</sup>He loss due to a-ejection.

<sup>c</sup>The propagated analytical uncertainty.

<sup>d</sup><sup>4</sup>He yields are in femto-mol for the apatite analyses and in pico-mol for the zircon analyses.

<sup>e</sup>The "raw age" was calculated with an iterative approach to solving the age equation.

<sup>f</sup>R2 values for the apatites are the half-widths of equi-dimensional hexagonal prisms, and R1 and R2 values for the zircons are half-widths for tetragonal prisms.

<sup>g</sup>L represents the total crystal length.

<sup>h</sup>T1 and T2 represent the length of each pyramidal termination in the zircons.

<sup>i</sup>β represents the surface-area-to-volume ratio for the crystals.

<sup>j</sup>The mean F<sub>T</sub> (alpha-ejection correction) was calculated assuming a bipyramidal tetragon prism geometry for the zircons (Hourigan et al. 2005), and an equi-dimensional hexagon prism geometry for the apatites (Farley et al., 1996).

<sup>k</sup>A F<sub>T</sub> correction was applied to the "raw age" following the procedures of Farley et al. (1996).

<sup>l</sup>% Calculated <sup>4</sup>He loss determined using equation [4], assuming the oldest apatite (61.51 Ma) and zircon (197.25 Ma) (U-Th)/He ages (shown by - symbols) and an impact event age of 0.663 Ma.

Table 1

## Contact and compression event

Mineral	Grain		Time (seconds) <sup>a</sup>	Modeled	
	radius ( $\mu\text{m}$ )	Temperature ( $^{\circ}\text{C}$ )		% <sup>4</sup> He loss <sup>b</sup>	Notes <sup>c</sup>
Apatite	27.4 (min.)	1500	0.000273	60.9	4.91 m impactor (min.)
Apatite	32.9 (av. reset)	1500	0.000273	52.3	4.91 m impactor (min.)
Apatite	52.9 (max.)	1500	0.000273	34.4	4.91 m impactor (min.)
Apatite	27.4 (min.)	1600	0.000273	79.4	4.91 m impactor (min.)
Apatite	32.9 (av. reset)	1600	0.000273	70.0	4.91 m impactor (min.)
Apatite	52.9 (max.)	1600	0.000273	47.6	4.91 m impactor (min.)
Apatite	27.4 (min.)	1500	0.000431	72.4	7.76 m impactor (max.)
Apatite	32.9 (av. reset)	1500	0.000431	81.6	7.76 m impactor (max.)
Apatite	52.9 (max.)	1500	0.000431	42.2	7.76 m impactor (max.)
Apatite	27.4 (min.)	1600	0.000431	89.8	7.76 m impactor (max.)
Apatite	32.9 (av. reset)	1600	0.000431	63.1	7.76 m impactor (max.)
Apatite	52.9 (max.)	1600	0.000431	57.7	7.76 m impactor (max.)
Zircon	24.8 (min.)	1500	0.000273	4.9	4.91 m impactor (min.)
Zircon	30.5 (reset)	1500	0.000273	4.0	4.91 m impactor (min.)
Zircon	40.6 (max.)	1500	0.000273	3.0	4.91 m impactor (min.)
Zircon	24.8 (min.)	1600	0.000273	7.6	4.91 m impactor (min.)
Zircon	30.5 (reset)	1600	0.000273	6.2	4.91 m impactor (min.)
Zircon	40.6 (max.)	1600	0.000273	4.7	4.91 m impactor (min.)
Zircon	24.8 (min.)	1500	0.000431	6.1	7.76 m impactor (max.)
Zircon	30.5 (reset)	1500	0.000431	5.0	7.76 m impactor (max.)
Zircon	40.6 (max.)	1500	0.000431	3.8	7.76 m impactor (max.)
Zircon	24.8 (min.)	1600	0.000431	9.5	7.76 m impactor (max.)
Zircon	30.5 (reset)	1600	0.000431	7.7	7.76 m impactor (max.)
Zircon	40.6 (max.)	1600	0.000431	5.8	7.76 m impactor (max.)

## Adiabatic decompression and shock metamorphism event

Mineral	Grain		Time (seconds)	Modeled	
	radius ( $\mu\text{m}$ )	Temperature ( $^{\circ}\text{C}$ )		% <sup>4</sup> He loss <sup>b</sup>	% <sup>4</sup> He loss <sup>b</sup>
Apatite	27.4 (min.)	1,500	1 (max.)	100.0	
Apatite	27.4 (min.)	1,500	0.1 (min.)	100.0	
Apatite	27.4 (min.)	1,600	1 (max.)	100.0	
Apatite	27.4 (min.)	1,600	0.1 (min.)	100.0	
Apatite	32.9 (av. reset)	1,500	1 (max.)	100.0	
Apatite	32.9 (av. reset)	1,500	0.1 (min.)	100.0	
Apatite	32.9 (av. reset)	1,600	1 (max.)	100.0	
Apatite	32.9 (av. reset)	1,600	0.1 (min.)	100.0	
Apatite	52.9 (max.)	1,500	1 (max.)	100.0	
Apatite	52.9 (max.)	1,500	0.1 (min.)	100.0	
Apatite	52.9 (max.)	1,600	1 (max.)	100.0	
Apatite	52.9 (max.)	1,600	0.1 (min.)	100.0	
Zircon	24.8 (min.)	1,500	1 (max.)	71.2	
Zircon	24.8 (min.)	1,500	0.1 (min.)	100.0	
Zircon	24.8 (min.)	1,600	1 (max.)	90.8	
Zircon	24.8 (min.)	1,600	0.1 (min.)	100.0	
Zircon	30.5 (av.)	1,500	1 (max.)	61.5	
Zircon	30.5 (av.)	1,500	0.1 (min.)	99.6	
Zircon	30.5 (av.)	1,600	1 (max.)	82.5	
Zircon	30.5 (av.)	1,600	0.1 (min.)	100.0	
Zircon	40.6 (max.)	1,500	1 (max.)	49.1	
Zircon	40.6 (max.)	1,500	0.1 (min.)	96.6	
Zircon	40.6 (max.)	1,600	1 (max.)	69.0	
Zircon	40.6 (max.)	1,600	0.1 (min.)	99.9	

Abbreviations: min. = minimum, av. = average, max. = maximum, E = activation energy, and  $D_0$  = frequency factor.

<sup>a</sup> Time (seconds) calculated using equation [1].

<sup>b</sup> Modeled % <sup>4</sup>He loss calculated using equations [2-3], using the following He diffusion parameters: apatite (E = 32.9 kcal/mol,  $D_0$  = 31.62 cm<sup>2</sup>/s with cylindrical diffusion geometry; Farley, 2000), and zircon (E = 40.4 kcal/mol,  $D_0$  = 0.45 cm<sup>2</sup>/s with spherical diffusion geometry; Reiners et al., 2004).

<sup>c</sup> Impact size calculated using *Crater* (Melosh and Beyer, 2002) and *Impact and Explosion Effects* (Holsapple, 2020) online software.

Table 2

Table 1: (U-Th)/He geochronological analyses of apatites and zircons from the Monturaqui impact crater

Sample	Grain# <sup>a</sup>	[ <sup>238</sup> U] <sup>b</sup> pmol	Error <sup>c</sup> 1σ	[ <sup>232</sup> Th] <sup>b</sup> pmol	Error <sup>c</sup> 1σ	Th/U	[ <sup>4</sup> He] <sup>b,d</sup> fmol/pmol	Error <sup>c</sup> 1σ	Age <sub>raw</sub> <sup>e</sup> Ma	Error <sup>c</sup> 1σ	R1 <sup>f</sup> μm	R2 <sup>f</sup> μm	L <sup>g</sup> μm	T1 <sup>h</sup> μm	T2 <sup>h</sup> μm	β <sup>i</sup> (1/μm)	F <sub>T</sub> Mean <sup>j</sup>	Age <sub>corr.</sub> <sup>k</sup> Ma	Error <sup>c</sup> 2σ	Calculated % <sup>4</sup> He loss <sup>l</sup>
<b>Apatites</b>																				
CIUP 08099	a004	0.0860	0.0015	0.2091	0.0035	2.45	0.510	0.016	2.96	0.10	-	39.2	114.6	-	-	0.0763	0.63	<b>4.69</b>	<b>0.31</b>	93.4
	a008	0.2116	0.0028	0.0623	0.0019	0.30	1.04	0.02	3.58	0.09	-	34.1	127.2	-	-	0.0835	0.61	<b>5.83</b>	<b>0.28</b>	91.5
	a001	0.1386	0.0023	0.3616	0.0053	2.63	9.24	0.12	32.37	0.56	-	39.8	163.7	-	-	0.0703	0.66	<b>49.36</b>	<b>1.71</b>	20.0
	a005	0.5148	0.0054	0.2959	0.0048	0.58	31.65	0.38	42.17	0.65	-	43.2	161.8	-	-	0.0658	0.69	<b>61.51</b>	<b>1.90</b>	-
CIUP 08100	a012	0.1428	0.0069	0.3291	0.0046	2.32	0.104	0.008	0.370	0.032	-	34.0	128.0	-	-	0.0835	0.60	<b>0.616</b>	<b>0.107</b>	100.1
	a001	0.1047	0.0022	0.3103	0.0086	2.98	0.109	0.012	0.480	0.052	-	31.7	110.7	-	-	0.0909	0.57	<b>0.845</b>	<b>0.183</b>	99.7
	a014	0.0868	0.0052	0.1145	0.0021	1.33	0.655	0.018	4.50	0.24	-	52.9	102.7	-	-	0.0631	0.69	<b>6.50</b>	<b>0.70</b>	90.4
	a005	0.0607	0.0025	0.0985	0.0019	1.63	0.416	0.017	3.89	0.20	-	32.5	108.4	-	-	0.1012	0.58	<b>6.70</b>	<b>0.69</b>	90.1
	a006	0.0362	0.0028	0.0573	0.0027	1.60	0.322	0.013	5.08	0.36	-	31.9	69.8	-	-	0.1012	0.53	<b>9.51</b>	<b>1.35</b>	85.5
	a008	0.0226	0.0027	0.0123	0.0012	0.548	0.175	0.010	5.36	0.66	-	31.2	80.2	-	-	0.099	0.55	<b>9.74</b>	<b>2.40</b>	85.1
	a007	0.0788	0.0037	0.1184	0.0038	1.51	2.761	0.045	20.25	0.79	-	33.3	87.1	-	-	0.0923	0.57	<b>35.57</b>	<b>2.78</b>	42.6
	a002	0.0144	0.0014	0.0457	0.0026	3.21	0.872	0.017	27.2	1.8	-	27.4	100.7	-	-	0.1043	0.51	<b>52.98</b>	<b>6.93</b>	14.0
<b>Zircons</b>																				
CIUP 08099	z001	10.37	0.12	4.917	0.068	0.478	0.05841	0.00068	3.960	0.061	32.5	32.8	145.6	27.9	28.8	0.0751	0.70	<b>5.67</b>	<b>0.18</b>	97.5
	z003	7.418	0.075	3.367	0.047	0.457	0.05763	0.00066	5.482	0.081	30.9	33.3	142.4	32.2	29.1	0.0771	0.69	<b>7.93</b>	<b>0.23</b>	96.3
	z004	8.77	0.10	4.412	0.053	0.507	0.3103	0.0036	24.67	0.39	28.9	31.5	203.9	32.2	37.1	0.0759	0.70	<b>35.49</b>	<b>1.13</b>	82.3
	z002	28.15	0.27	17.02	0.20	0.609	2.173	0.025	52.60	0.76	34.8	40.6	226.0	43.0	32.9	0.0619	0.75	<b>70.43</b>	<b>2.02</b>	64.5
	z005	4.314	0.051	2.527	0.034	0.590	0.6214	0.0073	98.1	1.6	26.3	33.8	188.2	31.1	28.2	0.0777	0.69	<b>142.64</b>	<b>4.56</b>	27.8
CIUP 08100	z003	2.614	0.037	1.628	0.027	0.627	0.001664	0.000029	0.4341	0.0093	29.8	30.5	105.6	24.1	23.7	0.0866	0.66	<b>0.662</b>	<b>0.029</b>	100.0
	z001	13.47	0.14	7.772	0.096	0.581	0.03041	0.00037	1.553	0.024	28.7	31.7	126.1	20.4	22.7	0.0819	0.67	<b>2.31</b>	<b>0.07</b>	99.2
	z005	0.793	0.014	0.663	0.017	0.842	0.03081	0.00038	25.36	0.50	26.1	29.9	116.7	34.1	31.0	0.0925	0.63	<b>40.07</b>	<b>1.57</b>	80.0
	z002	0.975	0.016	0.717	0.017	0.741	0.04675	0.00056	31.87	0.59	23.3	29.3	84.3	19.9	21.0	0.1033	0.60	<b>53.48</b>	<b>1.98</b>	73.1
	z004	1.943	0.027	0.619	0.016	0.321	0.3300	0.0039	122.1	2.2	27.9	24.8	114.4	33.0	31.3	0.0977	0.62	<b>197.25</b>	<b>6.99</b>	-

<sup>a</sup> The missing grain numbers represent grains that were determined to contain inclusions, which were not detected during mineral picking. These were identified by the helium re-extraction failing to yield blank levels, which would be the case for inclusion-free apatites.

<sup>b</sup> Absolute concentrations measured for <sup>4</sup>He, <sup>238</sup>U, and <sup>232</sup>Th are used to calculate a “raw age” that does not reflect <sup>4</sup>He loss due to a-ejection.

<sup>c</sup> The propagated analytical uncertainty.

<sup>d</sup> <sup>4</sup>He yields are in femto-mol for the apatite analyses and in pico-mol for the zircon analyses.

<sup>e</sup> The “raw age” was calculated with an iterative approach to solving the age equation.

<sup>f</sup> R2 values for the apatites are the half-widths of equi-dimensional hexagonal prisms, and R1 and R2 values for the zircons are half-widths for tetragonal prisms.

<sup>g</sup> L represents the total crystal length.

<sup>h</sup> T1 and T2 represent the length of each pyramidal termination in the zircons.

<sup>i</sup>  $\beta$  represents the surface-area-to-volume ratio for the crystals.

<sup>j</sup> The mean  $F_T$  (alpha-ejection correction) was calculated assuming a bipyramidal tetragon prism geometry for the zircons (Hourigan et al. 2005), and an equi-dimensional hexagon prism geometry for the apatites (Farley et al., 1996).

<sup>k</sup> A  $F_T$  correction was applied to the “raw age” following the procedures of Farley et al. (1996).

<sup>l</sup> % Calculated  $^4\text{He}$  loss determined using equation [4], assuming the oldest apatite (61.51 Ma) and zircon (197.25 Ma) (U-Th)/He ages (shown by - symbols) and an impact event age of 0.663 Ma.

Table 2. Modeling of  $^4\text{He}$  loss from apatite and zircon grains during Monturaqui impact crater-forming stages.**Contact and compression event**

Mineral	Grain radius ( $\mu\text{m}$ )	Temperature ( $^{\circ}\text{C}$ )	Time (seconds) <sup>a</sup>	Modeled % $^4\text{He}$ loss <sup>b</sup>	Notes <sup>c</sup>
Apatite	27.4 (min.)	1500	0.000273	60.9	4.91 m impactor (min.)
Apatite	32.9 (av. reset)	1500	0.000273	52.3	4.91 m impactor (min.)
Apatite	52.9 (max.)	1500	0.000273	34.4	4.91 m impactor (min.)
Apatite	27.4 (min.)	1600	0.000273	79.4	4.91 m impactor (min.)
Apatite	32.9 (av. reset)	1600	0.000273	70.0	4.91 m impactor (min.)
Apatite	52.9 (max.)	1600	0.000273	47.6	4.91 m impactor (min.)
Apatite	27.4 (min.)	1500	0.000431	72.4	7.76 m impactor (max.)
Apatite	32.9 (av. reset)	1500	0.000431	81.6	7.76 m impactor (max.)
Apatite	52.9 (max.)	1500	0.000431	42.2	7.76 m impactor (max.)
Apatite	27.4 (min.)	1600	0.000431	89.8	7.76 m impactor (max.)
Apatite	32.9 (av. reset)	1600	0.000431	63.1	7.76 m impactor (max.)
Apatite	52.9 (max.)	1600	0.000431	57.7	7.76 m impactor (max.)
Zircon	24.8 (min.)	1500	0.000273	4.9	4.91 m impactor (min.)
Zircon	30.5 (reset)	1500	0.000273	4.0	4.91 m impactor (min.)
Zircon	40.6 (max.)	1500	0.000273	3.0	4.91 m impactor (min.)
Zircon	24.8 (min.)	1600	0.000273	7.6	4.91 m impactor (min.)
Zircon	30.5 (reset)	1600	0.000273	6.2	4.91 m impactor (min.)
Zircon	40.6 (max.)	1600	0.000273	4.7	4.91 m impactor (min.)
Zircon	24.8 (min.)	1500	0.000431	6.1	7.76 m impactor (max.)
Zircon	30.5 (reset)	1500	0.000431	5.0	7.76 m impactor (max.)
Zircon	40.6 (max.)	1500	0.000431	3.8	7.76 m impactor (max.)
Zircon	24.8 (min.)	1600	0.000431	9.5	7.76 m impactor (max.)
Zircon	30.5 (reset)	1600	0.000431	7.7	7.76 m impactor (max.)
Zircon	40.6 (max.)	1600	0.000431	5.8	7.76 m impactor (max.)

**Adiabatic decompression and shock metamorphism event**

Mineral	Grain radius ( $\mu\text{m}$ )	Temperature ( $^{\circ}\text{C}$ )	Time (seconds)	Modeled % $^4\text{He}$ loss <sup>b</sup>
Apatite	27.4 (min.)	1,500	1 (max.)	100.0
Apatite	27.4 (min.)	1,500	0.1 (min.)	100.0
Apatite	27.4 (min.)	1,600	1 (max.)	100.0
Apatite	27.4 (min.)	1,600	0.1 (min.)	100.0
Apatite	32.9 (av. reset)	1,500	1 (max.)	100.0
Apatite	32.9 (av. reset)	1,500	0.1 (min.)	100.0
Apatite	32.9 (av. reset)	1,600	1 (max.)	100.0
Apatite	32.9 (av. reset)	1,600	0.1 (min.)	100.0
Apatite	52.9 (max.)	1,500	1 (max.)	100.0
Apatite	52.9 (max.)	1,500	0.1 (min.)	100.0
Apatite	52.9 (max.)	1,600	1 (max.)	100.0
Apatite	52.9 (max.)	1,600	0.1 (min.)	100.0
Zircon	24.8 (min.)	1,500	1 (max.)	71.2
Zircon	24.8 (min.)	1,500	0.1 (min.)	100.0
Zircon	24.8 (min.)	1,600	1 (max.)	90.8
Zircon	24.8 (min.)	1,600	0.1 (min.)	100.0
Zircon	30.5 (av.)	1,500	1 (max.)	61.5
Zircon	30.5 (av.)	1,500	0.1 (min.)	99.6
Zircon	30.5 (av.)	1,600	1 (max.)	82.5
Zircon	30.5 (av.)	1,600	0.1 (min.)	100.0
Zircon	40.6 (max.)	1,500	1 (max.)	49.1
Zircon	40.6 (max.)	1,500	0.1 (min.)	96.6
Zircon	40.6 (max.)	1,600	1 (max.)	69.0
Zircon	40.6 (max.)	1,600	0.1 (min.)	99.9

Abbreviations: min. = minimum, av. = average, max. = maximum, E = activation energy, and  $D_0$  = frequency factor.

<sup>a</sup> Time (seconds) calculated using equation [1].

<sup>b</sup> Modeled %  $^4\text{He}$  loss calculated using equations [2-3], using the following He diffusion parameters: apatite (E = 32.9 kcal/mol,  $D_0$  = 31.62  $\text{cm}^2/\text{s}$  with cylindrical diffusion geometry; Farley, 2000), and zircon (E = 40.4 kcal/mol,  $D_0$  = 0.45  $\text{cm}^2/\text{s}$  with spherical diffusion geometry; Reiners et al., 2004).

<sup>c</sup> Impact size calculated using *Crater* (Melosh and Beyer, 2002) and *Impact and Explosion Effects* (Holsapple, 2020) online software.

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**Declaration of interests**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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