

# 1 Intercalibration of the Hb3gr $^{40}\text{Ar}/^{39}\text{Ar}$ dating standard

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15

## 16 Abstract

17

18 The  $^{40}\text{Ar}/^{39}\text{Ar}$  dating technique is based on the knowledge of the age of neutron fluence  
19 monitors (standards). Recent investigations have improved the accuracy and precision of the  
20 ages of most of the Phanerozoic-aged standards (e.g. Fish Canyon Tuff sanidine (FCs), Alder  
21 Creek sanidine, GA1550 biotite and LP-6 biotite); however, no specific study has been  
22 undertaken on the older standards (i.e. Hb3gr hornblende and NL-25 hornblende) generally  
23 used to date Precambrian, high Ca/K, and/or meteoritic rocks.

24 In this study, we show that Hb3gr hornblende is relatively homogenous in age, composition  
25 (Ca/K) and atmospheric contamination at the single grain level. The mean standard deviation  
26 of the  $^{40}\text{Ar}^*/^{39}\text{Ar}_K$  (F-value) derived from this study is 0.49%, comparable to the most  
27 homogeneous standards. The intercalibration factor (which allows direct comparison between  
28 standards) between Hb3gr and FCs is  $R_{FCs}^{Hb3gr} = 51.945 \pm 0.167$ . Using an age of 28.02 Ma for  
29 FCs, the age of Hb3gr derived from the R-value is  $1073.6 \pm 5.3$  Ma ( $1\sigma$ ; internal error only)  
30 and  $\pm 8.8$  Ma (including all sources of error). This age is indistinguishable within uncertainty  
31 from the K/Ar age previously reported at  $1072 \pm 11$  Ma (Turner et al., 1971; [Turner G.,  
32 Huneke, J.C., Podosek, F.A., Wasserburg, G.J., 1971.  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  ages and cosmic ray exposure  
33 ages of Apollo 14 samples. *Earth Planet. Sci. Lett.* 12, 19-350]).

34 The R-value determined in this study can also be used to intercalibrate FCs if we consider the  
35 K/Ar date of 1072 Ma as a reference age for Hb3gr. We derive an age of  $27.95 \pm 0.19$  Ma  
36 ( $1\sigma$ ; internal error only) for FCs which is in agreement with the previous determinations.  
37 Altogether, this shows that Hb3gr is a suitable standard for  $^{40}\text{Ar}/^{39}\text{Ar}$  geochronology.

38

## 39 **1. Introduction**

40

41 The  $^{40}\text{Ar}/^{39}\text{Ar}$  technique is one of the most powerful methods, supplanting the conventional  
42 K/Ar method because it is possible to better evaluate the accuracy of an age (i.e. using age  
43 spectrum).

44 In return,  $^{40}\text{Ar}/^{39}\text{Ar}$  dating is based on the knowledge of the ages of standards (neutron fluence  
45 monitors) because of the irradiation of samples and is thus directly dependent on how  
46 accurately and precisely their ages are known. The age of a standard can be determined either  
47 directly by conventional K/Ar dating (primary standards such as GA1550 biotite, [McDougall  
48 and Roksandic \(1974\)](#) and NL-25 hornblende, [Schaeffer and Schaeffer \(1977\)](#)) or through  
49  $^{40}\text{Ar}/^{39}\text{Ar}$  intercalibration with a primary standard (secondary standards such as Fish Canyon  
50 sanidine (FCs); [Cebula et al. \(1986\)](#)). Secondary standards may be used, in turn, to calibrate  
51 “higher order” standards (e.g. Alder Creek sanidine (ACs) calibrated against FCs; [Nomade et  
52 al., 2005](#)). In this case, the age and error of the intermediate standard(s) should be propagated  
53 in the final result ([Renne et al., 1998](#); [Nomade et al., 2005](#)). Currently, analytical precision on  
54 isotopic ratios measured with high-resolution, low-background mass spectrometers may reach  
55  $\pm 0.1$ - $0.2$  %, whereas the ages of fluence monitors are generally considered to be known to  $\pm$   
56  $0.5$  % ( $1\sigma$ ) if we neglect the error on the decay constant (e.g. FCs, ACs, GA1550; [Renne et  
57 al., 1998](#)). Therefore, the overall uncertainty on the age of an unknown is partially limited by  
58 the uncertainty on the age of the fluence monitor. Additionally, recent studies ([Renne et al.,  
59 1998](#); [Min et al., 2000](#)) have focused on systematic errors including the  $^{40}\text{K}$  decay constant  
60 uncertainties (and possibly accuracy) which is the most significant error contributor to the  
61 global error on the age. The value of the decay constant is currently in question (e.g. [Min et  
62 al., 2000](#); [Kwon et al., 2002](#)) but will not be discussed here.

63 The uncertainty on the age of a  $^{40}\text{Ar}/^{39}\text{Ar}$  standard is also dependent of the chemical  
64 homogeneity of the mineral at the single grain level. This is crucial for  $^{40}\text{Ar}/^{39}\text{Ar}$  dating  
65 because standards are generally measured on single grains using a laser device. K/Ar  
66 measurement of a primary standard requires much larger amounts of material compared to  
67  $^{40}\text{Ar}/^{39}\text{Ar}$  dating and thus, homogenizes the  $^{40}\text{Ar}^*/^{40}\text{K}$  ratio of the standard. Studies

68 concerning the homogeneity of standards have recently been conducted on some of the most  
69 widely used standards (e.g. [Baksi et al, 1996](#); [Renne et al., 1998](#); [Spell & McDougall, 2003](#)).

70 Ideally, a standard should be chosen to approximate the age and/or composition of the  
71 unknown sample to minimize the range of isotopic ratios measured (c.f. [Renne et al., 1997](#),  
72 [1998](#); [Nomade et al., 2005](#)). In addition, a standard must be homogeneous in composition (i.e.  
73 without secondary inclusions) at single-grains scale ([Roddick, 1983](#)).

74 The dating of Precambrian and/or high Ca/K rocks mostly relies on two available standards -  
75 NL-25 hornblende (2660 Ma; [Schaeffer and Schaeffer, 1977](#); particularly used for meteorite  
76 study) and Hb3gr hornblende (1072 Ma; [Turner et al., 1971](#)). Nevertheless, the homogeneity  
77 of these standards at the single grain level has never been properly documented. Here, we  
78 investigate the age and composition of the Hb3gr standard.

79 The apparent age of the Hb3gr hornblende standard was determined to be  $1072 \pm 11^1$  Ma  
80 more than three decades ago using the K/Ar method ([Turner et al.; 1971](#)). The age of Hb3gr is  
81 therefore known to  $\pm 1\%$  and can be compared to other widely-used standards such as  
82 GA1550 and FCs, which are known to  $1\%$  (including error on the decay constant; [Renne et  
83 al., 1998](#)). The Hb3gr standard is currently in use in several laboratories and has been used in  
84 many studies for which a precision of the  $^{40}\text{Ar}/^{39}\text{Ar}$  ages better than  $1\%$  is desirable. (e.g.  
85 [Turner et al. 1971](#); [Roddick et al. 1983](#), [Hall et al., 1984](#); [McConville et al., 1988](#); [Courtillot  
86 et al., 2000](#), [Renne, 2000](#); [Nomade et al., 2001](#); [LeGall et al., 2002](#); [Whitby et al., 2002](#), [Yibas  
87 et al., 2002](#), [Roberts et al., 2002](#) [Lenoir et al., 2003](#), [Jourdan et al., 2004](#); [Burgess et al., 2004](#);  
88 [Verati et al., 2005](#)). Therefore, to take full advantage of the Hb3gr standard, it is crucial that  
89 (1) the age of Hb3gr currently adopted is re-assessed, (2) the precision on the age should be  
90 improved and (3) the homogeneity of the hornblende at the single grains level should be  
91 evaluated. In order to do so, we conducted 72 single grain  $^{40}\text{Ar}/^{39}\text{Ar}$  total fusion analyses on  
92 the Hb3gr hornblende monitor irradiated along with 87 grains of the FCs standards (28.02  
93 Ma; [Renne et al., 1998](#)). Additionally, we determined the intercalibration factor (R-value)  
94 which, in this case, is the  $^{40}\text{Ar}^*/^{39}\text{Ar}$  ratio of Hb3gr to that of FCs (see [Renne et al., 1998](#)).  
95 The intercalibration factor makes it possible to compare different standards. Finally, we  
96 propose a new age and uncertainty for Hb3gr hornblende using the full propagation error  
97 calculation of [Renne et al. \(1998\)](#).

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<sup>1</sup> All uncertainties concerning the ages of standards in this paper are given at the  $1\sigma$  level unless otherwise indicated.

## 100 2. Description and ages of the studied standards

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### 102 2.1. Fish Canyon sanidine (FCs)

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104 The Fish Canyon sanidine comes from the large Fish Canyon ash-flow tuff (~5000 km<sup>3</sup>;  
105 [Lipman, 1997](#)) located in San Juan Volcanic field, south-western Colorado. The tuff contains  
106 abundant phenocrysts (35-50%) of plagioclase, sanidine, biotite, hornblende, quartz, zircon,  
107 titanite and apatite ([Lipman et al., 1997](#)).

108 The Fish Canyon tuff has a relatively simple history, and therefore, the FCs was introduced as  
109 a potential <sup>40</sup>Ar/<sup>39</sup>Ar standard by [Cebula et al. \(1986\)](#). Since then, the FCs has become one of  
110 the most widely used neutron fluence monitors. Numerous studies attempted to determine its  
111 age either as (1) a primary standard (i.e. by K-Ar measurement; [Steven et al., 1967](#); [Hurford  
112 and Hammerschmidt, 1985](#)); however, it is difficult to extract all the <sup>40</sup>Ar\* from viscous  
113 sanidine melts ([Webb and McDougall, 1967](#)), or (2) a secondary standard (i.e. relative to a  
114 primary standard used as a fluence monitor; [Renne et al., 1994](#); [Hilgen et al., 1997](#); [Renne et  
115 al., 1998](#); [Villeneuve et al., 2000](#); [Lanphere & Baadsgaard, 2001](#); [Spell & McDougall, 2003](#);  
116 [Dazé et al., 2003](#)).

117 The precision and accuracy required for the <sup>40</sup>Ar/<sup>39</sup>Ar dating standard imply that the age used  
118 for a standard should be the same in every laboratory. If we discard the younger age of 27.57  
119 ± 0.18 Ma published by [Lanphere & Baadsgaard, \(2001\)](#) based on the poorly constrained SB-3  
120 biotite monitor (see [Spell & McDougall, 2003](#) and [Schmitz et al., 2003](#)), the various ages  
121 obtained from more recent measurements using the <sup>40</sup>Ar/<sup>39</sup>Ar technique are in good agreement  
122 within uncertainties. Using the decay constant of [Steiger and Jager \(1977\)](#) but without taking  
123 uncertainties on the decay constant into account, the ages are 28.15 ± 0.19 Ma ([Hilgren et al.,  
124 1997](#)), 27.98 ± 0.08 Ma ([Villeneuve et al.; 2000](#)), 28.10 ± 0.04 ([Spell and McDougall; 2003](#))  
125 and 28.02 ± 0.16 Ma ([Renne et al.; 1998](#)). Published <sup>40</sup>Ar/<sup>39</sup>Ar age spectra are flat ([Lanphere  
126 & Baadsgaard, 2001](#), and [Spell and McDougall, 2003](#)) highlighting the superior homogeneity  
127 of this standard.

128 In this study, we adopted the age published in [Renne et al. \(1998\)](#) for FCs as 28.02 ± 0.16 Ma  
129 based in turn on an age of 98.79 ± 0.5 Ma (K/Ar age) for the primary standard GA1550 (both  
130 age errors exclude uncertainties on the decay constant). We use this age because the K and  
131 <sup>40</sup>Ar\* concentrations of GA1550 have been determined recently ([Renne et al., 1998](#)) by the  
132 ‘first principles’ and can be assumed to be more reliable than the K and Ar\* concentration of

133 Hb3gr determined more than 34 years ago. The corresponding  $R_{GA-1550}^{FCs}$  value is  $0.27811 \pm$   
134  $0.000295$ .

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## 136 *2.2. Hb3gr hornblende*

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138 The Hb3gr hornblende was originally analyzed by [Zartman \(1964\)](#) and re-analyzed after  
139 purification by [Turner et al. \(1971\)](#). It was extracted from the Lone Grove Pluton (LGP)  
140 located in Texas (USA) ([Zartman, 1964](#); [Turner et al., 1971](#); [Rougvie et al., 1999](#)). The LGP  
141 has a simple cooling history with a titanite U/Pb age of  $1093 \pm 6$  Ma and hornblende and  
142 biotite  $^{40}\text{Ar}/^{39}\text{Ar}$  weighted-mean ages (from a different location than Hb3gr) of  $1076 \pm 8$  and  
143  $1079 \pm 8$  Ma, respectively ([Rougvie et al., 1999](#)). From these ages, [Rougvie et al \(1999\)](#)  
144 estimated a cooling rate of  $\sim 14^\circ\text{C}/\text{Myr}$  for the LGP.

145 The hornblende was originally chosen by [Turner et al. \(1971\)](#) as a neutron fluence monitor  
146 because of its relatively simple history with no subsequent metamorphism. Hornblende was  
147 preferred to biotite mostly because of its higher closure temperature ( $\sim 600^\circ\text{C}$ ; [Villa et al.,](#)  
148 [1996](#)) than biotite ( $\sim 300^\circ\text{C}$ ; [Harrison et al., 1985](#)). Subsequently, [Roddick \(1983\)](#)  
149 demonstrated that the Hb3gr hornblende bulk sample appeared homogenous in age at the  
150 0.1% level.

151 [Zartman \(1964\)](#) first provided a K-Ar age for the Hb3gr hornblende of  $1060 \pm 20$  Ma. [Turner](#)  
152 [et al. \(1971\)](#) refined this age, and [Roddick \(1983\)](#) recalculated it using the decay constant of  
153 [Steiger and Jager \(1977\)](#) to get an age of  $1072 \pm 11$  Ma. The age of  $1072 \pm 11$  Ma became the  
154 “official age” for the Hb3gr monitor in 1983. A more recent investigation of Hb3gr concerned  
155 the measurement of two sets of hornblende grains (a re-preparation of the Hb3gr standard  
156 referred to as PP-20). These hornblende crystals were co-irradiated with FCs with the purpose  
157 of dating the Acapulco meteorite ([Renne, 2000](#)). Both samples consisted of 38 grains each  
158 and yielded two ages of  $1074 \pm 4$  and  $1073 \pm 4$  Ma (analytical errors only; based on the FCs  
159 monitor with an age of 28.02 Ma; [Renne et al., 1998](#)). However, no detailed interpretation of  
160 these ages was provided.

161

## 162 **3. Analytical techniques**

163

164 We investigated pre-existing mineral separates of Hb3gr hornblende ( $n = 72$ ; 125-200  $\mu\text{m}$   
165 fraction) and FC sanidine ( $n = 87$ ; 200-315  $\mu\text{m}$  fraction). All standards were carefully

166 handpicked under a binocular microscope to avoid impurities or inclusions such as quartz or  
167 microcline (Turner et al., 1971; Roddick et al., 1983). This intercalibration was performed by  
168 using three distinct irradiations with two different durations, so as to address any issues  
169 related to irradiation conditions. For each irradiation, the Hb3gr and FCs standards were  
170 loaded separately in three square-shaped, flat packages (7-10 mm wide) made of aluminum  
171 foil. To ensure a minimum neutron flux variation during the irradiation, the packages were  
172 assembled as follows: one package including 30 grains of Hb3gr was framed by two packages  
173 including 15 grains of FCs each (irradiation MC36) and 30 grains of FCs framed by two  
174 packages of 15 grains of Hb3gr each (irradiations MC34 and MC37). For each irradiation, the  
175 three packages were placed side-by-side along with other unrelated samples in a 15 cm-long  
176 package made of aluminum foil. The standards were irradiated for 150h (MC34) and 70h  
177 (MC36 and MC37) in the Hamilton McMaster University nuclear reactor (Canada) in position  
178 5C (i.e. in the high flux area and receiving flux from all directions). The total neutron flux  
179 density during irradiation was  $1.9 \times 10^{19}$  neutron/cm<sup>2</sup> (MC34) and  $8.8 \times 10^{18}$  neutron/cm<sup>2</sup> (MC36  
180 and MC37). The flux gradient ranges from 0 to 0.1% per mm in length of the final package  
181 and is laterally negligible. Each grain of standard was analyzed separately in a single fusion  
182 experiment using a CO<sub>2</sub> Synrad 48-5 laser beam. The gas was purified in a stainless and glass  
183 extraction line using two Al-Zr getters (working at 400°C and ambient temperature,  
184 respectively) and a liquid nitrogen cold trap. Isotopic measurements were performed with a  
185 VG3600 mass spectrometer and a Daly-photomultiplier system.  $^{40}\text{Ar}^*/^{39}\text{Ar}_K$  values (Table 1)  
186 were corrected for blank, mass discrimination, radioactive decay and interference isotopes.  
187 Mass discrimination was monitored weekly using an online air pipette providing  $^{40}\text{Ar}/^{36}\text{Ar}$   
188 ratios ranging from 282.2 to 289.9 with error of  $\pm 0.5\%$ . This corresponds to corrections  
189 ranging from 1.01178 to 1.00483 per atomic mass unit. The correction factors for interfering  
190 isotopes were measured using Ca and K pure silicates and were  $(^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 7.06 \times 10^{-4}$  ( $\pm$   
191 4%),  $(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 2.79 \times 10^{-4}$  ( $\pm 3\%$ ) and  $(^{40}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 2.97 \times 10^{-2}$  ( $\pm 3\%$ ). The  $^{40}\text{Ar}/^{39}\text{Ar}$   
192 ages obtained for the Hb3gr monitor are based on the reference age of  $28.02 \pm 0.16$  Ma  
193 (Renne et al., 1998; neglecting the error on the decay constant). For each irradiation, the  
194 neutron fluence value (J) was determined using the  $^{40}\text{Ar}^*/^{39}\text{Ar}_K$  (F-value) weighted-mean  
195 ratio obtained for the FCs grains. The J-values are  $(3.5381 \pm 0.0033) \times 10^{-2}$ ;  $(1.6162 \pm 0.0019)$   
196  $\times 10^{-2}$  and  $(1.7268 \pm 0.0018) \times 10^{-2}$  for MC34, MC36 and MC37 suite respectively. Individual  
197 errors in Table 1 are given at the  $1\sigma$  level. The decay constants are those recommended by  
198 Steiger & Jager (1977).

199

## 200 4. Results / discussion

201

### 202 4.1. Fish Canyon monitor

203

204 The three F-value suites obtained for FCs from the three irradiations have been plotted as  
205 probability density distribution diagrams (Sircombe et al., 2004) in Fig. 1. The three  
206 probability curves show simple and symmetric Gaussian distributions. Their weighted-mean  
207 F-values (Table 2) are  $0.4424 \pm 0.0002$  (MSWD=1.19; n= 27),  $0.9686 \pm 0.0006$   
208 (MSWD=0.96; n=28), and  $0.9064 \pm 0.0005$  (MSWD=1.98; n=31) for irradiation MC34,  
209 MC36 and MC37, respectively. These values exclude discordant data deviating more than  $3\sigma$   
210 from the weighted-mean values. These values are not directly comparable as they depend of  
211 the irradiation duration and neutron flux; they can be converted in age after mathematical  
212 treatment using their associated J-values. The three mean F-value standard deviations ( $1\sigma$ )  
213 describe the overall dispersion of the data and range from 0.26% to 0.48%.

214 The standard deviation values must be compared with analytical errors alone because the  
215 errors from interference factors and mass discrimination corrections represent constant values  
216 for each irradiation. For each irradiation suite, the average analytical error values vary from  
217 0.26% to 0.38% and are similar to the F-value standard deviations. Altogether, close-to-1  
218 MSWD values and relatively low standard deviations obtained on the FCs standard are  
219 similar to the results of previous studies (e.g. Renne et al., 1998). These results confirm that  
220 the analyzed FCs crystals have strong between-analysis reproducibility and are homogenous  
221 at the single crystal level.

222

### 223 4.2. Hb3gr hornblende

224

#### 225 4.2.1. Hornblende reproducibility

226

227 The three different irradiation batches yield mean F-values standard deviations for Hb3gr  
228 hornblende of 0.33% (MC34; n=14), 0.62% (MC36; n=28) and 0.51% (MC37; n=28) (Table  
229 2) excluding two outlying data points (MC34-3 and MC36-i30) which lie greater than  $3\sigma$   
230 outside the weighted-mean value of a given irradiation. These values are roughly similar to  
231 those obtained from the FCs although the MC36 suite shows a higher standard deviation.  
232 MSWD values are varying alike the standard deviations, with relatively low MSWD for  
233 MC34 and MC37 batches (1.07 and 2.4 respectively) and a substantially higher MSWD-value

234 of 4.1 for the MC36 suite. The high MSWD of MC36 clearly shows that error cannot be  
235 attributed to analytical error alone because the three values obtained by averaging the  
236 analytical errors for the three irradiations vary from 0.30% to 0.34%. This discrepancy is  
237 mirrored by the probability curves of F-value of MC34 and MC37 suites which show well-  
238 defined Gaussian curves whereas MC36 exhibits an asymmetrical curve with 2 peaks and a  
239 relative difference between the two peaks equal to 0.6%. No correlation is apparent between  
240 the age (F-value) of the data and their respective  $^{37}\text{Ar}_{\text{Ca}}/^{39}\text{Ar}_{\text{K}}$  ratios (proxy for Ca/K) and  
241 atmospheric  $^{40}\text{Ar}$  content. The reason for the skewed distribution of the MC36 suite is not  
242 understood.

243 The estimated Ca/K composition derived from the  $^{37}\text{Ar}/^{39}\text{Ar}$  ratios ( $\text{Ca/K} = 1.79 \times$   
244  $^{37}\text{Ar}_{\text{Ca}}/^{39}\text{Ar}_{\text{K}}$ ) shows minor variation mostly from 5.0 to 6.5 and is in agreement with the  
245 chemical composition of the hornblende (P.R. Renne, unpublished data). Moreover,  $^{40}\text{Ar}/^{39}\text{Ar}$   
246 measurements show that the Hb3gr hornblende grains have a relatively low and constant  
247 atmospheric contamination (0.2-1.2%; Table 1), showing no correlation with the F-value.  
248 Therefore, the interference correction of  $^{36}\text{Ar}_{\text{Ca}}$  is almost negligible (despite a high CaO  
249 concentration) and the propagation of the uncertainties linked to this interference correction  
250 factor is minimized. Altogether, this suggests that the small, 7.5% younger, inclusions of  
251 microcline ( $K \approx 13\%$ ) reported in some Hb3gr hornblende (Zartman, 1964) have been  
252 successfully eliminated during grain selection, thus, these inclusions are probably not the  
253 factor responsible for the MC36 F-value variation. The two age populations from MC36 could  
254 reflect heterogeneity of the neutron flux from side to side of the 1 cm-wide package, but  
255 previous experiments done on McMaster irradiated samples have failed to demonstrate this  
256 effect (G.F. unpublished data). In addition, it would be hard to explain why this effect is not  
257 observed in the MC34 and MC37 suites.

258 The average standard deviation of the Hb3gr F-values at the single grain level calculated for  
259 the three suites is 0.49 % and is inversely proportional to the reproducibility. If we exclude  
260 younger additional outliers (MC34-1, MC34-2, MC36-I22, and MC37-X6; Table 1) which are  
261 inside the  $3\sigma$  confidence limit but “visually discordant” (i.e. ranging from 1.7 to  $2\sigma$ ) in F-  
262 value vs. analysis (not shown) and age vs. analysis plots (Fig. 2A), the global standard  
263 deviation becomes 0.38%. These values are on the same order than those derived from other  
264 commonly used standards such as: GA1550 (0.17-0.36%), TCs (0.30-0.51%) and MMhb-1  
265 (0.15-0.61%) determined by Renne et al. (1998), or GA1550 (0.39%), MMhb-1 (0.82%), 92-  
266 176 sanidine (0.60%), and LP-6 (1.45%) determined by Spell & McDougall (2003). This  
267 comparison therefore suggests that the Hb3gr hornblende is relatively homogenous at the



268 single crystal level and shows a good reproducibility although lower than FCs (s.d. = 0.34%;  
269 this study).

270

#### 271 4.2.2. Intercalibration value ( $R_{FCs}^{Hb3gr}$ )

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273 The intercalibration (R) value allows a direct comparison between standards of different ages.  
274 It is defined as the ratio of the F-value ( $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ) between two standards after their co-  
275 irradiation (Renne et al., 1998). This value is particularly useful for the recalculation of  
276 previously published ages using a different age for the fluence monitor. It also allows a full  
277 propagation of the errors when only analytical errors plus error on J-value are quoted (i.e.  
278 excluding uncertainties of the decay constant and the age of the monitor; e.g. Min et al.,  
279 2000).

280 The R-values for the three suites of irradiation (Table 3) are deduced from weighted-means of  
281 the F-values (Table 2). The overall R-value (Table 4) may be obtained by different statistical  
282 approaches using: (1) the weighted-mean (and weighted-mean error) of each R-value (e.g.  
283 Spell & McDougall, 2003), (2) the arithmetic mean with either the standard error of the mean  
284 (i.e. the standard deviation divided by the roots-squared of the number of analyses (e.g. Renne  
285 et al., 1998), or the standard deviation (Nomade et al., 2005) as uncertainties. Although the  
286 arithmetic and weighted-mean calculation provides similar values; the error values are  
287 substantially different (Table 4).

288 The overall mean  $R_{FCs}^{Hb3gr}$  values derived from  $R_{MC34}$ ,  $R_{MC36}$  and  $R_{MC37}$  calculated using the  
289 arithmetic mean and weighted-mean are 51.945 and 51.965, respectively, and are concordant  
290 within uncertainties. The weighted-mean error, the standard error of the mean and the  
291 standard deviation values are 0.025 [relative error (r.e.)= 0.05%], 0.167 [r.e.= 0.33%] and  
292 0.292 [r.e.= 0.56%], respectively.

293 The weighted-mean may not be the best calculation when using a small dataset (i.e. 3 R-  
294 values), but the choice between arithmetic and weighted-mean does not sensibly affect the  
295 final value. However, the error on the weighted-mean tends to minimize the uncertainties on  
296 the final results. The calculation of the weighted-mean error includes only the error associated  
297 with each measurement (here, R-values) and does not take into account the natural dispersion  
298 of the dataset due to compositional heterogeneities, crystal defects, etc... For instance, the  
299 calculated weighted-mean error (0.05%) on  $R_{FCs}^{Hb3gr}$  is lower than for  $R_{GA-1550}^{FCs}$  (0.11%; Renne  
300 et al., 1998) although we previously showed that Hb3gr has a grain to grain reproducibility

301 noticeably lower than FCs and GA1550. Similarly, the standard deviation provides good  
302 information on the dispersion of the data relative to the mean, but is not relevant to estimate  
303 the uncertainty on a mean value. It is more appropriate when one tries to describe the error  
304 associated with a mean calculated from a gradient. The standard error of the mean better  
305 describes our datasets as it combines information about the dispersion of the data relative to  
306 the mean and takes into account the number of analyses (i.e. increasing the number of  
307 measurements leads to better precision, e.g. Mandel, 1984).

308 For these reasons, we report hereafter the age calculated with the arithmetic mean and the  
309 standard error of the mean where  $R_{FCs}^{Hb3gr} = 51.945 \pm 0.167$  [0.33%]

310

#### 311 4.2.3. Age of Hb3gr

312

313 Apparent individual ages obtained for Hb3gr mostly range from  $1063 \pm 3$  to  $1085 \pm 3$  Ma  
314 (Fig. 2A, Table 1) based on an age of 28.02 Ma for FCs (Renne et al., 1998). Two analyses  
315 displaying much younger ages (1055 and 1043 Ma), clearly discordant from the range  
316 mentioned above, have been rejected. However, the ages are not equally distributed  
317 depending on the irradiation batch. The MC34 irradiation includes generally younger ages  
318 varying from  $1063 \pm 3$  to  $1071 \pm 2$  Ma (n=14), whereas MC36 and MC37 show a broader age  
319 spectrum varying from  $1064 \pm 3$  to  $1085 \pm 3$  Ma (n=29) and  $1064 \pm 2$  to  $1081 \pm 4$  Ma (n=28),  
320 respectively (Fig. 2).

321 The weighted-mean ages of the three suites are concordant, with values of  $1069 \pm 5$ ,  $1078 \pm$   
322  $10$  and  $1074 \pm 8$  Ma for MC34, MC36 and MC37, respectively. However, the Hb3gr global  
323 age is calculated by using the previously calculated overall  $R_{FCs}^{Hb3gr}$  value of  $51.945 \pm 0.167$   
324 instead of the average of all individual age measurements. As previously mentioned, this  
325 allows calculating and propagating all external sources of errors (Renne et al., 1998). The  
326 total uncertainty on the age of Hb3gr therefore includes errors on: (1) the GA-1550 primary  
327 standard  $^{40}\text{Ar}^*/^{40}\text{K}$  ratio ( $0.0059 \pm 0.0000332$ ), (2) the  $R_{GA-1550}^{FCs}$  intercalibration factor  
328 ( $0.27811 \pm 0.000295$ ), and (3) the calculated  $R_{FCs}^{Hb3gr}$  value ( $51.945 \pm 0.167$ ), in addition to the  
329 internal, J-value, and mass discrimination errors and the interference corrections. Depending  
330 on the purpose of the study, the total uncertainty may also include error on the decay constant.

331 Excluding the uncertainties on the decay constant, we obtain an age at  $1073.6 \pm 5.3$  Ma  
332 (Table 4). This age is similar to the age of  $1073.9 \pm 4.6$  Ma obtained by using the R-value  
333 calculated with the weighted-mean. On the other hand, using the weighted-mean error, the

334 standard error of the mean or the standard deviation for the calculation of R-value, yields  
335 substantially different uncertainties in the final results (respectively 4.6 Ma [r.e.=0.43%]; 5.3  
336 Ma [r.e.=0.50%] and 6.50 Ma [r.e.=0.60%]). Including the uncertainties on the decay constant  
337 into the calculation (Min et al., 2000) almost doubles the global uncertainties (+45%),  
338 yielding a mean age of  $1073.6 \pm 8.8$  Ma [0.82%].

339 A global (n=70) age-data probability diagram (Fig. 2B) shows a nearly Gaussian curve with  
340 the peak age at 1073 Ma, slightly lower by 0.15% than the weighted-mean age (although  
341 largely in agreement within uncertainties). This age discrepancy likely results from the slight  
342 departure of the probability curve from a perfect Gaussian plot (visible on the right side of the  
343 curve).

344 From a geological point of view, we can establish how the age and the precision of an  
345 unknown sample (measured using the Hb3gr monitor) will be affected by the results  
346 presented here. For instance, a sample measured with Hb3gr monitor and using an age of  
347  $1072.0 \pm 6.6$  Ma (Turner et al., 1971; analytical uncertainties only) yields an (arbitrary) age of  
348  $100.0 \pm 1.4$  Ma (including all uncertainties). Application of equations (1), (3), (4) and (7) of  
349 Renne et al. (1998) for age calculation and error propagation and using an age of  $1073.6 \pm 5.3$   
350 Ma for Hb3gr, yield a slightly older age of  $100.2 \pm 1.4$  Ma. The precision of the age is not  
351 significantly modified mainly because the contributions of the decay constant and analytical  
352 errors may represent a dominant source of errors relatively to the improvement of the age of  
353 Hb3gr standard.

354 In summary, the global age and internal error of  $1073.6 \pm 5.3$  Ma obtained in this study is in  
355 agreement within uncertainties with the previously reported K/Ar ( $1072 \pm 7$  Ma; analytical  
356 error only; Turner et al., 1971) and  $^{40}\text{Ar}/^{39}\text{Ar}$  ( $1074 \pm 4$  and  $1073 \pm 4$  Ma; Renne, 2000) ages.  
357 The age variation shown in the double-peak probability curve displayed for the MC36 suite  
358 results (Fig. 1) suggests that the Hb3gr hornblende monitor exhibits a slight departure from  
359 homogeneity. These variations contribute to decrease the precision on the age of the Hb3gr  
360 standard. Therefore, the total reproducibility (standard deviation) calculated over 70 single  
361 grains (0.49 %, and possibly 0.38% by removing  $\sim 2\sigma$  outliers) is similar to the most reliable  
362 standards (e.g. GA-1550; ACs; FCs) and suggests that the Hb3gr hornblende is relatively  
363 homogeneous at the single crystal level.

364

365 **5. Circular statement: the age of the FCs**

366

367 Hb3gr could also be considered as a primary standard for which the K-Ar age was determined  
368 by [Turner et al. \(1971\)](#). These authors measured K and  $^{40}\text{Ar}^*$  concentrations of  $1.247 \pm 0.008$   
369 % and  $7.09 \times 10^{-7}$  cc/gm standard temperature and pressure (STP), respectively using ‘first  
370 principles’ (i.e. the  $^{40}\text{Ar}^*$  has been calibrated relatively to a known volume of air) and  
371 isotopes dilution techniques. Using the decay constants suggested by [Steiger & Jäger \(1977\)](#),  
372 this composition yielded the widely used age of  $1072 \pm 11$  Ma (including errors on the decay  
373 constant). As mentioned above, the new  $^{40}\text{Ar}/^{39}\text{Ar}$  ages obtained for Hb3gr hornblende grains  
374 ( $1073.6 \pm 8.8$  Ma) is in agreement within uncertainties with the K/Ar age determined by  
375 [Turner et al. \(1971\)](#). If we assume that the K/Ar age determined on the Hb3gr standard has the  
376 same accuracy as the one obtained for GA-1550, we can use the Hb3gr hornblende as a  
377 fluence monitor to directly intercalibrate FCs (using a  $R_{\text{Hb3gr}}^{\text{FCs}}$  value of  $0.01925 \pm 0.00006$  ( $=1/$   
378  $R_{\text{FCs}}^{\text{Hb3gr}}$ )). We obtained an age of  $27.95 \pm 0.19$  Ma ([0.68%;] for FCs, excluding uncertainties  
379 on the decay constant; Table 4B). This age is in agreement with the most recent age  
380 determinations of  $28.15 \pm 0.19$  Ma ([Hilgren et al., 1997](#)),  $28.02 \pm 0.16$  Ma ([Renne et al.,](#)  
381 [1998](#)),  $27.98 \pm 0.08$  Ma ([Villeneuve et al., 2000](#)) and  $28.10 \pm 0.04$  ([Spell and McDougall,](#)  
382 [2003](#)) although it should be noted that the reported errors are (unfortunately) calculated using  
383 different statistical approaches.

384 A re-evaluation of the FCs age is out of the topic of this paper because it would require  
385 further detailed intercalibration using the most reproducible and well known primary  
386 standards. If the age of the FCs is modified by future investigations, the  $R_{\text{FCs}}^{\text{Hb3gr}}$  value  
387 provided in this study should allow a rapid recalculation of the age of the Hb3gr without  
388 further measurement.

389

## 390 **6. Conclusions**

391

392 We studied the age and suitability of the widely-used Hb3gr hornblende fluence monitor  
393 using 70 individual grains distributed over three irradiations and co-irradiated with the Fish  
394 Canyon sanidine standard. The major conclusions include:

395

- 396 1. The relatively low mean standard deviation (0.49% and possibly 0.38% if we exclude  
397 outliers) of the three F-values ( $= ^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ) of Hb3gr, the low atmospheric  
398 contamination and the relatively invariant Ca/K composition from grain to grain

399 altogether suggest that Hb3gr is age-homogeneous and is therefore suitable as a  
400 single-grain  $^{40}\text{Ar}/^{39}\text{Ar}$  dating standard.

401 2. The intercalibration factor ( $R_{FCs}^{Hb3gr}$  value) calculated from the arithmetic mean and  
402 standard error of the mean of three R-values obtained from three irradiations is  $51.947$   
403  $\pm 0.169$  (n=70). Considering an age of 28.02 Ma for FCs (Renne et al., 1998), the age  
404 calculated for Hb3gr is  $1073.6 \pm 5.4$  Ma (internal error only) and  $\pm 8.8$  Ma (including  
405 all sources of error). This age is indistinguishable (though more precise) from the  
406 K/Ar age of  $1072 \pm 11$  Ma determined by Turner et al. (1971) more than 3 decades  
407 ago.

408 3. Assuming that the K/Ar age determined by Turner et al. (1971) on Hb3gr is correct,  
409 and using the Hb3gr as a fluence monitor to intercalibrate FCs, we obtained a FCs age  
410 of  $27.95 \pm 0.19$  (internal error only) and  $\pm 0.30$  (full error propagation) in agreement  
411 with previous determinations. Nevertheless, more data from various primary standards  
412 are required to improve the age of FCs.

413

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415

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420

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532

533 **Figure and table captions**

534

535 Figure 1. Probability density distribution diagram of  $^{40}\text{Ar}^*/^{39}\text{Ar}_k$  ratios (F-value) of Hb3gr and  
536 FCs calculated using [Sircombe et al. \(2004\)](#). Numbers of analysis for each standard are  
537 indicated for each of the three irradiations. Arrows indicate the width at the middle-height of  
538 the peaks and percentages represent the relative width compared to the peak value allowing  
539 fast comparison between the different F-values.

540

541 Figure 2. A) Age vs. number of analysis for Hb3gr (n=72). Individual errors are quoted at  $1\sigma$ .  
542 Gray diamond: grains from irradiation MC34 (n=15); white diamond: MC36 (n=29) and black  
543 diamond: MC37 (n=28). Outliers outside the  $3\sigma$  confidence level of the weighted-mean (n=2)  
544 and pseudo-outliers at  $\sim 2\sigma$  and “visually discordant” (n=4) are indicated. B)  $^{40}\text{Ar}/^{39}\text{Ar}$  age-  
545 probability density distribution diagram for the three irradiations calculated excluding two  
546 ( $>3\sigma$ ) outliers. Dotted line: MC34 (n=14); Dotted-dashed line: MC36 (n=28); dashed line:  
547 MC37 (n=28). The solid line represents the calculation including all 70 Hb3gr grains. The age  
548 derived from the intercalibration-factor (see text) is indicated, and it includes all sources of  
549 error.

550

551 Table 1.  $^{40}\text{Ar}/^{39}\text{Ar}$  analytical data for Hb3gr and FCs.

552  $^{40}\text{Ar}^*$ = radiogenic Ar;  $^{37}\text{Ar}$  and  $^{39}\text{Ar}$  produced by neutron interference with Ca and K  
553 respectively. Decay constants are from [Steiger and Jager \(1977\)](#). Errors on the discrimination  
554 are not reported but are considered to be  $\pm 0.5\%$ . Ages obtained for Hb3gr are derived using  
555 an age of 28.02 Ma for FCs ([Renne et al, 1998](#)). J-value is indicated for each irradiation and is  
556 referenced to FCs. Correction factors for interfering isotopes were  $(^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 7.06 \times 10^{-4}$  ( $\pm$   
557 4%),  $(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 2.79 \times 10^{-4}$  ( $\pm$  3%) and  $(^{40}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 2.97 \times 10^{-2}$  ( $\pm$  3%). Analytical  
558 uncertainties on ages in this table are  $1\sigma$ .

559

560

561 Table 2. Summary of  $^{40}\text{Ar}^*/^{39}\text{Ar}_k$  (F-value) results for Hb3gr and FCs.

562 F-values were calculated using the weighted-mean and error of the weighted-mean.  
563 Irradiation duration, standard deviation ( $1\sigma$ ) and MSWD are indicated. n: number of analysis  
564 for each suite.

565

566

567 Table 3. Summary of R-values for each individual irradiation.

568 R values were calculated using the Hb3gr and FCs F-values ratios given in Table 2. Error on  
569 each R-value is the error of the weighted-mean on the F-values of Hb3gr and FCs propagated  
570 into the calculation.

571

572 Table 4. A) Summary of the global R-value and associated age proposed for Hb3gr relative to  
573 FCs (28.02 Ma).

574 Results from different calculations based on the 3 R-values of Table 3 are shown. These  
575 calculations include arithmetic and weighted-mean with, error of the weighted-men, standard  
576 error of the mean and standard deviation ( $1\sigma$ ). Relative errors are indicated in brackets. Ages  
577 calculated from the corresponding R-value are indicated ( $1\sigma$ ) both excluding and including  
578 error on the decay constant ( $\sigma\lambda$ ). B). similar as Table 4A, but using Hb3gr as fluence monitor  
579 (1072 Ma; [Turner et al., 1971](#)) and FCs as the intercalibrated standard.

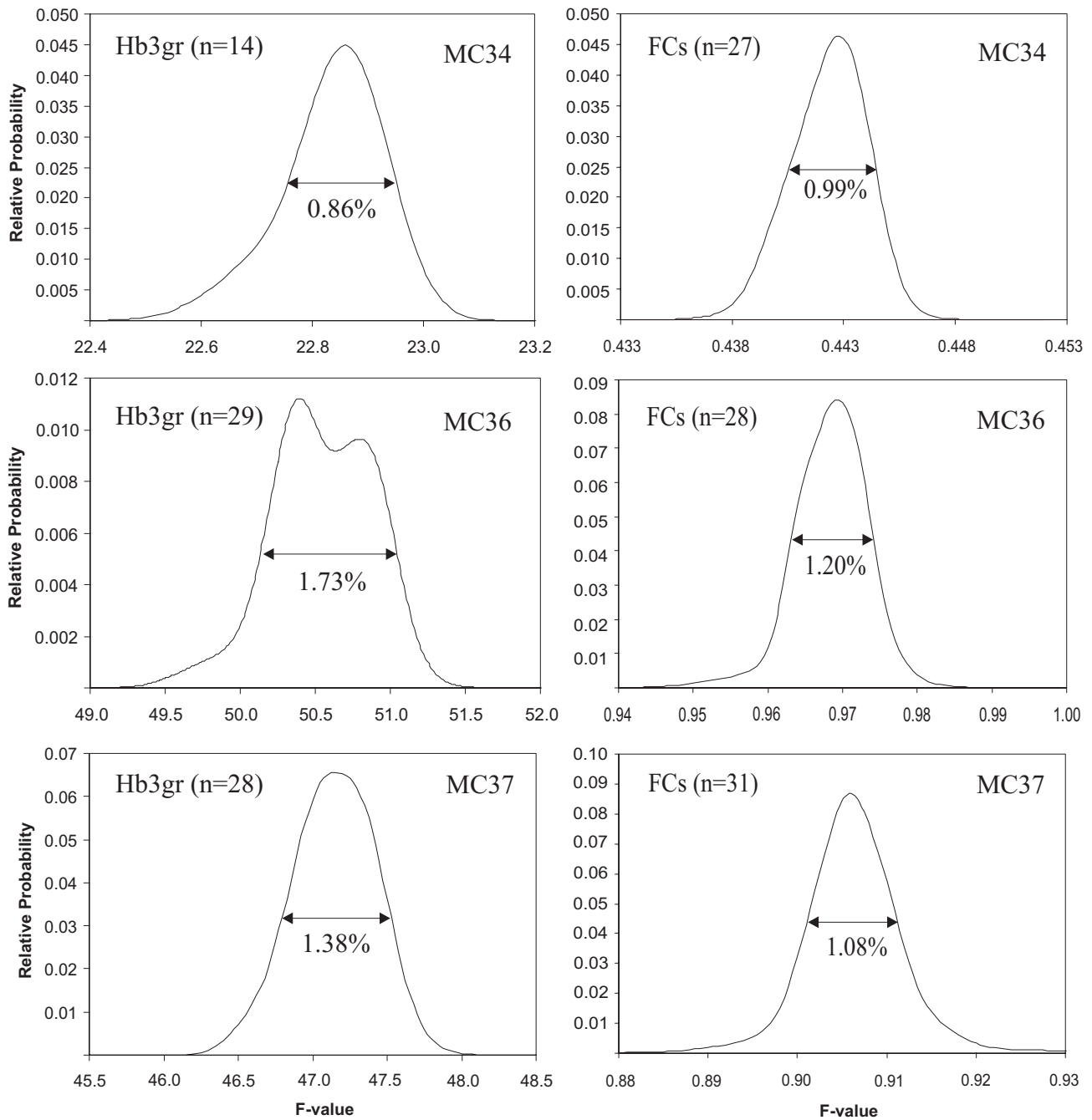


Fig. 1 : Jourdan et al.

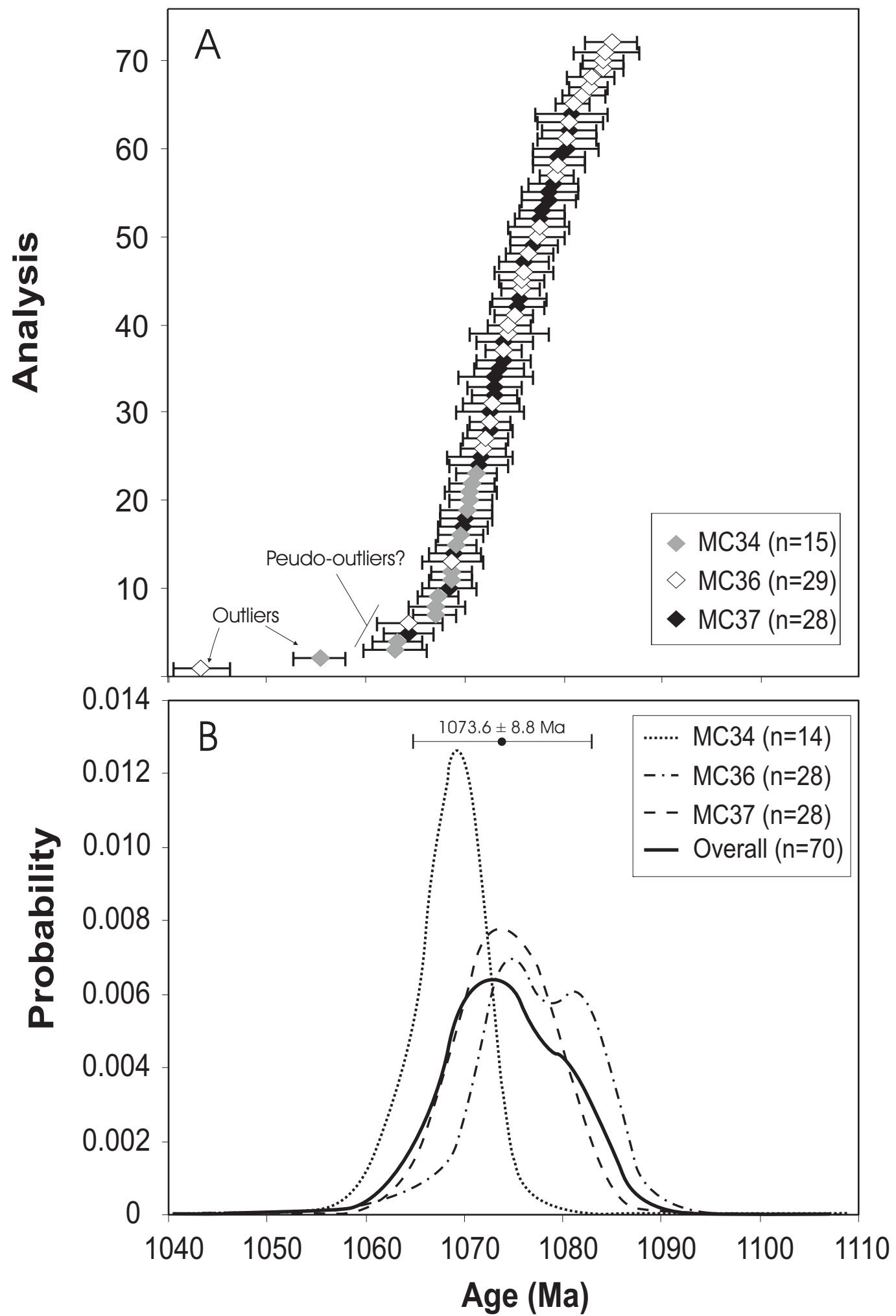


Table1

[Click here to download Table: Table 1.pdf](#)

Standard	Package number	Sample name	Discrimination value	Atmospheric contamination (%)	$^{37}\text{Ar}_{\text{Ca}}/^{39}\text{Ar}_{\text{K}}$	$^{40}\text{Ar}^*/^{39}\text{Ar}$	Error	Age (Ma)	Error (1 $\sigma$ )
<i>MC34 (J=0.035381 ± 0.000033)</i>									
Hb3gr	1	1	1.00624	0.79	3.205	22.689	0.070	1063.2	2.5
Hb3gr	1	2	1.00624	0.92	3.146	22.681	0.090	1062.9	3.2
Hb3gr	1	3	1.00624	1.12	3.309	22.466	0.072	1055.3	2.6
Hb3gr	1	4	1.00624	0.59	3.168	22.888	0.074	1070.2	2.6
Hb3gr	1	5	1.00624	0.79	3.156	22.857	0.056	1069.1	2.0
Hb3gr	1	6	1.00624	0.84	3.416	22.841	0.059	1068.6	2.1
Hb3gr	1	7	1.00624	0.79	3.282	22.801	0.083	1067.2	2.9
Hb3gr	1	8	1.00624	0.67	3.163	22.904	0.064	1070.8	2.3
Hb3gr	1	9	1.00624	0.52	3.416	22.898	0.073	1070.6	2.6
Hb3gr	1	10	1.00624	0.62	3.292	22.870	0.067	1069.6	2.4
Hb3gr	1	11	1.00624	0.60	3.023	22.844	0.060	1068.7	2.2
Hb3gr	1	12	1.00624	0.45	2.922	22.898	0.062	1070.6	2.2
FCs	2	e1	1.00624	0.73	0.007	0.443	0.001	-	-
FCs	2	e2	1.00624	0.31	0.008	0.443	0.001	-	-
FCs	2	e4	1.00624	0.66	0.008	0.441	0.001	-	-
FCs	2	e5	1.00714	0.90	0.007	0.441	0.002	-	-
FCs	2	e6	1.00714	0.58	0.007	0.442	0.001	-	-
FCs	2	e7	1.00714	0.40	0.009	0.443	0.001	-	-
FCs	2	e8	1.00714	1.08	0.009	0.441	0.002	-	-
FCs	2	e9	1.00714	0.85	0.008	0.440	0.001	-	-
FCs	2	e10	1.00714	0.76	0.008	0.442	0.002	-	-
FCs	2	e11	1.00714	1.04	0.009	0.444	0.001	-	-
FCs	2	e12	1.00714	0.85	0.008	0.442	0.001	-	-
FCs	2	e13	1.00714	0.63	0.008	0.442	0.001	-	-
FCs	2	e14	1.00714	1.08	0.007	0.443	0.001	-	-
FCs	2	e15	1.00714	0.49	0.007	0.444	0.001	-	-
FCs	2	e16	1.00714	0.84	0.007	0.441	0.001	-	-
FCs	2	e17	1.00714	0.42	0.008	0.444	0.001	-	-
FCs	2	e18	1.00714	0.22	0.007	0.444	0.001	-	-
FCs	2	e19	1.00714	0.97	0.008	0.440	0.001	-	-
FCs	2	e20	1.00714	1.61	0.008	0.443	0.001	-	-
FCs	2	e21	1.00714	0.39	0.006	0.443	0.001	-	-
FCs	2	e22	1.00714	0.41	0.008	0.443	0.002	-	-
FCs	2	e23	1.00714	0.51	0.008	0.442	0.001	-	-
FCs	2	e24	1.00714	1.00	0.008	0.442	0.001	-	-
FCs	2	e25	1.00714	0.58	0.011	0.442	0.001	-	-
FCs	2	e26	1.00714	0.53	0.008	0.443	0.001	-	-
FCs	2	e27	1.00714	0.52	0.007	0.443	0.001	-	-
FCs	2	e28	1.00714	0.53	0.007	0.442	0.001	-	-
Hb3gr	3	D1	1.00827	0.90	3.101	22.804	0.060	1067.3	2.1
Hb3gr	3	D2	1.00827	0.63	3.1	22.916	0.060	1071.2	2.1
Hb3gr	3	D3	1.00827	0.29	3.099	22.796	0.061	1067.0	2.2
<i>MC36 (J=0.016162 0.000019)</i>									
FCs	1	X1	1.00776	0.86	0.007	0.968	0.003	-	-
FCs	1	X2	1.00776	0.52	0.006	0.973	0.002	-	-
FCs	1	X3	1.00776	0.50	0.007	0.969	0.002	-	-
FCs	1	X4	1.00776	0.76	0.008	0.969	0.003	-	-
FCs	1	X5	1.00776	0.89	0.005	0.964	0.002	-	-
FCs	1	X6	1.00776	2.50	0.011	0.964	0.002	-	-
FCs	1	X7	1.00921	1.08	0.007	0.970	0.004	-	-
FCs	1	X8	1.00921	1.54	0.006	0.958	0.006	-	-
FCs	1	X9	1.00921	0.55	0.008	0.971	0.005	-	-
FCs	1	X10	1.00921	0.78	0.007	0.970	0.004	-	-
FCs	1	X11	1.00921	0.67	0.008	0.968	0.003	-	-
FCs	1	X12	1.00921	2.33	0.007	0.970	0.006	-	-
FCs	1	X13	1.00921	1.62	0.008	0.969	0.003	-	-
FCs	1	X14	1.00921	0.63	0.006	0.972	0.004	-	-
FCs	1	X15	1.00921	3.37	0.007	0.972	0.002	-	-
Hb3gr	2	Y1	1.00948	0.86	3.357	50.016	0.194	1068.8	3.1
Hb3gr	2	Y2	1.00948	0.56	3.275	50.248	0.126	1072.5	2.0
Hb3gr	2	Y3	1.00948	0.56	3.129	50.559	0.193	1077.5	3.1
Hb3gr	2	Y4	1.00948	0.56	3.087	50.465	0.189	1076.0	3.0
Hb3gr	2	i6	1.01188	0.98	3.109	50.368	0.246	1074.4	4.0
Hb3gr	2	i7	1.01132	0.57	3.301	50.259	0.174	1072.7	2.8
Hb3gr	2	i8	1.01132	0.37	3.154	50.887	0.156	1082.7	2.5
Hb3gr	2	i9	1.01132	0.53	3.13	50.370	0.131	1074.5	2.1
Hb3gr	2	i10	1.01132	0.68	3.118	50.225	0.146	1072.1	2.4
Hb3gr	2	i12	1.01132	0.45	3.187	50.773	0.111	1080.9	1.8
Hb3gr	2	i13	1.01132	0.81	3.173	50.962	0.130	1084.0	2.1
Hb3gr	2	i14	1.01132	0.31	3.162	50.961	0.138	1083.9	2.2

Standard	Package number	Sample number		Atmospheric contamination (%)	$^{37}\text{Ar}_{\text{Ca}}/^{39}\text{Ar}_{\text{K}}$	$^{40}\text{Ar}^*/^{39}\text{Ar}$	Error	Age (Ma)	Error (1 $\sigma$ )
Hb3gr	2	i15	1.01132	0.25	3.325	50.984	0.210	1084.3	3.4
Hb3gr	2	i16	1.01113	0.78	2.959	50.456	0.143	1075.8	2.3
Hb3gr	2	i17	1.01113	0.72	3.175	50.681	0.162	1079.5	2.6
Hb3gr	2	i18	1.01113	0.65	3.156	50.444	0.122	1075.7	2.0
Hb3gr	2	i19	1.01113	0.51	3.231	50.209	0.141	1071.9	2.3
Hb3gr	2	i20	1.01113	0.90	3.253	50.546	0.169	1077.3	2.7
Hb3gr	2	i21	1.01113	0.46	3.075	50.341	0.113	1074.0	1.8
Hb3gr	2	i22	1.01113	1.22	3.385	49.747	0.207	1064.4	3.4
Hb3gr	2	i23	1.01113	0.69	3.047	50.401	0.126	1075.0	2.0
Hb3gr	2	i23	1.01113	0.83	3.305	50.736	0.190	1080.3	3.0
Hb3gr	2	i25	1.01113	0.54	3.219	50.842	0.136	1082.0	2.2
Hb3gr	2	i26	1.01113	0.44	3.323	51.016	0.161	1084.8	2.6
Hb3gr	2	i27	1.01113	0.48	3.075	50.874	0.117	1082.5	1.9
Hb3gr	2	i28	1.00919	0.28	3.468	50.497	0.145	1076.5	2.3
Hb3gr	2	i29	1.00919	0.29	3.209	50.669	0.107	1079.3	1.7
Hb3gr	2	i30	1.00919	0.52	2.797	48.451	0.170	1043.4	2.8
Hb3gr	2	i31	1.00919	0.78	3.323	50.756	0.211	1080.7	3.4
FCs	3	Z1	1.00483	0.57	0.007	0.970	0.004	-	-
FCs	3	Z2	1.00483	0.46	0.007	0.968	0.004	-	-
FCs	3	Z3	1.00483	0.58	0.007	0.971	0.005	-	-
FCs	3	Z4	1.00483	0.68	0.007	0.967	0.005	-	-
FCs	3	Z5	1.00483	1.34	0.007	0.968	0.004	-	-
FCs	3	Z6	1.00483	0.69	0.007	0.966	0.003	-	-
FCs	3	Z7	1.00915	0.76	0.007	0.970	0.002	-	-
FCs	3	Z8	1.00915	1.28	0.007	0.966	0.004	-	-
FCs	3	Z9	1.00915	0.52	0.007	0.972	0.004	-	-
FCs	3	Z10	1.00915	1.24	0.007	0.962	0.005	-	-
FCs	3	Z11	1.00915	0.85	0.006	0.966	0.004	-	-
FCs	3	Z12	1.00705	0.36	0.007	0.971	0.004	-	-
FCs	3	Z13	1.00705	1.46	0.008	0.966	0.004	-	-
FCs	3	Z14	1.00705	0.52	0.006	0.971	0.005	-	-
<b>MC37 (J=0.0172168±0.000018)</b>									
Hb3gr	1	X1	1.00885	0.24	3.176	47.435	0.152	1079.5	2.6
Hb3gr	1	X2	1.00885	0.32	3.462	47.200	0.160	1075.5	2.8
Hb3gr	1	X3	1.00885	0.42	3.137	46.871	0.141	1069.8	2.4
Hb3gr	1	X4	1.00885	0.94	3.281	47.509	0.214	1080.8	3.7
Hb3gr	1	X5	1.00885	0.47	3.101	46.966	0.174	1071.5	3.0
Hb3gr	1	X6	1.00885	0.83	3.156	46.552	0.140	1064.3	2.4
Hb3gr	1	X7	1.00885	0.45	3.614	47.115	0.163	1074.0	2.8
Hb3gr	1	X8	1.00885	0.61	3.288	46.889	0.154	1070.1	2.7
Hb3gr	1	X9	1.00885	0.92	3.266	46.972	0.192	1071.6	3.3
Hb3gr	1	X10	1.00885	1.06	3.489	46.789	0.162	1068.4	2.8
Hb3gr	1	X11	1.00885	0.48	3.152	47.029	0.198	1072.6	3.4
Hb3gr	1	X12	1.00885	0.42	3.365	47.381	0.170	1078.6	2.9
Hb3gr	1	X13	1.00885	0.71	3.205	47.062	0.221	1073.1	3.8
Hb3gr	1	X14	1.00885	0.68	3.373	47.476	0.188	1080.2	3.2
Hb3gr	1	X15	1.00885	0.69	3.166	47.108	0.164	1073.9	2.8
Fcs	2	Y1	1.00770	0.62	0.008	0.904	0.003	-	-
Fcs	2	Y2	1.00770	0.84	0.007	0.907	0.002	-	-
Fcs	2	Y3	1.00770	1.00	0.008	0.906	0.002	-	-
Fcs	2	Y4	1.00770	1.66	0.008	0.897	0.004	-	-
Fcs	2	Y5	1.00770	0.67	0.007	0.904	0.002	-	-
Fcs	2	Y6	1.00770	1.02	0.008	0.918	0.010	-	-
Fcs	2	Y7	1.00770	0.91	0.008	0.905	0.003	-	-
Fcs	2	Y8	1.00770	0.62	0.007	0.903	0.003	-	-
Fcs	2	Y9	1.00770	1.06	0.007	0.902	0.002	-	-
Fcs	2	Y10	1.00770	1.27	0.007	0.900	0.002	-	-
Fcs	2	Y11	1.00770	0.31	0.007	0.910	0.002	-	-
Fcs	2	Y12	1.00770	0.07	0.007	0.915	0.003	-	-
Fcs	2	Y13	1.00770	0.37	0.008	0.909	0.003	-	-
Fcs	2	Y14	1.00770	1.66	0.008	0.906	0.002	-	-
Fcs	2	Y15	1.00770	0.82	0.007	0.905	0.003	-	-
Fcs	2	Y17	1.00770	0.68	0.009	0.906	0.003	-	-
Fcs	2	Y18	1.00770	0.31	0.009	0.910	0.002	-	-
Fcs	2	Y19	1.00770	0.29	0.007	0.908	0.002	-	-
Fcs	2	Y20	1.00770	0.49	0.009	0.910	0.004	-	-
Fcs	2	Y21	1.00770	0.25	0.007	0.911	0.002	-	-
Fcs	2	Y22	1.00770	0.67	0.007	0.907	0.003	-	-
Fcs	2	Y23	1.01141	0.99	0.008	0.903	0.002	-	-
Fcs	2	Y24	1.01141	0.65	0.009	0.912	0.004	-	-
Fcs	2	Y25	1.01141	0.83	0.008	0.905	0.004	-	-
Fcs	2	Y26	1.01141	0.58	0.008	0.906	0.004	-	-
Fcs	2	Y27	1.01141	1.88	0.012	0.899	0.009	-	-

Standard	Package number	Sample number		Atmospheric contamination (%)	$^{37}\text{Ar}_{\text{Ca}}/^{39}\text{Ar}_{\text{K}}$	$^{40}\text{Ar}^*/^{39}\text{Ar}$	Error	Age (Ma)	Error (1 $\sigma$ )
Fcs	2	Y28	1.01141	0.75	0.007	0.906	0.002	-	-
Fcs	2	Y29	1.01141	0.69	0.007	0.905	0.003	-	-
Fcs	2	Y30	1.01141	0.56	0.007	0.905	0.005	-	-
Fcs	2	Y31	1.01141	1.10	0.008	0.906	0.005	-	-
Fcs	2	Y32	1.01141	0.15	0.007	0.911	0.002	-	-
Hb3gr	3	Z1	1.00885	0.80	3.327	46.822	0.156	1069.0	2.7
Hb3gr	3	Z2	1.00885	0.69	3.117	47.026	0.129	1072.5	2.2
Hb3gr	3	Z3	1.00885	0.60	3.067	47.059	0.162	1073.1	2.8
Hb3gr	3	Z4	1.00885	0.51	3.328	47.493	0.163	1080.5	2.8
Hb3gr	3	Z5	1.00885	0.22	3.213	47.337	0.138	1077.8	2.4
Hb3gr	3	Z6	1.00885	0.27	3.105	47.082	0.142	1073.5	2.4
Hb3gr	3	Z7	1.00885	0.29	2.925	47.051	0.131	1072.9	2.3
Hb3gr	3	Z8	1.00770	0.31	3.141	47.371	0.160	1078.4	2.7
Hb3gr	3	Z9	1.00770	0.34	3.385	47.194	0.160	1075.4	2.7
Hb3gr	3	Z10	1.00770	0.38	3.077	47.230	0.143	1076.0	2.5
Hb3gr	3	Z11	1.00770	0.27	3.181	47.322	0.148	1077.6	2.6
Hb3gr	3	Z12	1.00770	0.47	3.34	47.284	0.139	1076.9	2.4
Hb3gr	3	Z13	1.00770	0.62	3.136	47.401	0.149	1078.9	2.6

**Table2**[Click here to download Table: Table 2.pdf](#)

Table 2

Irradiation	Standard	n	Duration (h)	F-value ( <sup>40</sup> Ar*/ <sup>39</sup> Ar <sub>K</sub> )	Error of the weighted mean	Standard deviation (1σ)	Relative Standard Deviation(%)	MSWD
MC34	FCs	27	150	0.4424 ± 0.00020 (± 0.05%)		0.001144	0.26%	1.19
MC34	Hb3gr	14	150	22.842 ± 0.017 (± 0.08%)		0.074712	0.33%	1.07
MC36	FCs	28	70	0.9686 ± 0.00058 (± 0.06%)		0.002750	0.28%	0.96
MC36	Hb3gr	28	70	50.566 ± 0.028 (± 0.05%)		0.315936	0.62%	4.13
MC37	FCs	31	70	0.9064 ± 0.00046 (± 0.05%)		0.004396	0.48%	1.98
MC37	Hb3gr	28	70	47.134 ± 0.030 (± 0.06%)		0.240521	0.51%	2.36



**Table3**[Click here to download Table: Table 3.pdf](#)

Calculation	$R_{FCs}^{Hb^{3gr}}$	Error	Standard deviation	Relative standard deviation
MC34	51.632	0.046	0.215	0.42%
MC36	52.204	0.042	0.358	0.69%
MC37	51.999	0.042	0.366	0.70%

Table 3

Table 4A

Calculation	$R_{FCs}^{Hb3gr}$	Error	Age Hb3gr (Ma)	Error (1 $\sigma$ ; excluding $\sigma\lambda$ )	Error (1 $\sigma$ ; including $\sigma\lambda$ )
Arithmetic mean ( $\pm$ standard deviation)	51.945 $\pm$ 0.290 ( $\pm$ 0.56%)		1073.6 $\pm$ 6.5 ( $\pm$ 0.60%)		$\pm$ 9.5 ( $\pm$ 0.88%)
( $\pm$ standard error of the mean)	$\pm$ 0.167 ( $\pm$ 0.33%)		$\pm$ 5.3 ( $\pm$ 0.49%)		$\pm$ 8.8 ( $\pm$ 0.82%)
Weighted mean ( $\pm$ weighted mean error)	51.962 $\pm$ 0.025 ( $\pm$ 0.05%)		1073.9 $\pm$ 4.6 ( $\pm$ 0.43%)		$\pm$ 8.4 ( $\pm$ 0.78%)

Table 4B

Calculation	$R_{Hb3gr}^{FCs}$	Error	Age Fcs (Ma)	Error (1 $\sigma$ ; excluding $\sigma\lambda$ )	Error (1 $\sigma$ ; including $\sigma\lambda$ )
Arithmetic mean ( $\pm$ standard deviation)	0.01925 $\pm$ 0.00011 ( $\pm$ 0.56%)		27.95 $\pm$ 0.23 (0.83%)		$\pm$ 0.32 (1.16%)
( $\pm$ standard error of the mean)	$\pm$ 0.00006 ( $\pm$ 0.33%)		$\pm$ 0.19 (0.68%)		$\pm$ 0.30 (1.06%)
Weighted mean ( $\pm$ weighted mean error)	0.01924 $\pm$ 0.00001 ( $\pm$ 0.05%)		27.94 $\pm$ 0.17 (0.61%)		$\pm$ 0.28 (1.02%)