



# On etching, selection and measurement of confined fission tracks in apatite

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## Abstract

This work investigates the selection of horizontal confined tracks for fission-track modelling. It is 1 carried out on prism sections of Durango apatite containing induced tracks with mean lengths of 2  $\sim$ 16,  $\sim$ 14,  $\sim$ 12, and  $\sim$ 10  $\mu$ m. Suitable tracks are identified during systematic scans in transmitted 3 light. The explicit selection criteria are that the tracks are horizontal and measurable. We meas-4 ure the length, width, orientation, and cone angle of each selected track and in some cases other 5 dimensions. 6 The confined track selection is in the first place dependent on a threshold width and in the second 7 place on the requirement that the tracks are etched to their ends. In most cases the first condition 8 implies the second, which decreases in importance as the tracks are shortened following anneal-9 ing. The widest confined tracks, which must also be the shallowest, come to intersect the surface 10 and are excluded. In general, the selection is dominated by the width of the etched tracks. This, in 11 turn, depends on their orientation relative to the *c*-axis and the apatite etch rates, and their effec-12 tive etch times. Despite the different geometrical configuration of the unetched host tracks and con-13 fined tracks, neither the angular distribution nor the etch time distribution of the confined track 14 sample depends on the degree of annealing. This illustrates the general principle that those tracks 15 are selected that have the right properties for being selected. In this case etching-related factors 16 determining the track width are the most important, while the known geometrical biases are sec-17 ond order. The track etch rate exhibits no demonstrable variation along the track, but significant 18 differences from track to track. Moreover, although the track etch rate of induced tracks is not 19 correlated with the extent of partial annealing, it is on average twice as high as the value for fossil 20 tracks. 21

Our length measurements are in good agreement with the annealing models for this apatite and etch protocol. We submit that this is not fortuitous and that it is possible to select a representative confined track sample, and perform reproducible and meaningful confined track length measurements. Deliberate or inadvertent biasing, carelessness or inexperience will of course give different results, but these should be treated as statistical outliers, not as an indication that track lengths are fluid.

Keywords: apatite, fission-track, confined track etching, selection and measurement, track etch
 rate

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## 1. Introduction

Fission-track analysis is a method for determining the ages of rocks and retracing their thermal 30 histories. It is based on the trails of lattice damage left in suitable minerals by the fragments of 31 fissioned uranium nuclei. The tools for interpreting fission-track data have evolved apace but 32 33 fundamental questions have remained unanswered. An important problem has to do with the relationship between the actual damage trails and the fission tracks that are counted and meas-34 ured after etching the mineral grains. Galbraith (2005) observed that "Inferring thermal histo-35 36 ries from track measurements involves two steps. The first is to relate measurements made on a sample to the true length distribution f(l), and the second is to relate f(l) to the thermal his-37 tory". Several studies have addressed observational biases connected with measurements of 38 (horizontal) confined tracks. These are of a geometrical nature and their numerical treatment 39 is based on the so-called line-segment model. They include length bias, orientation bias, inter-40 section (fracture and host track thickness) bias, and edge and surface proximity biases (Laslett 41 et al., 1982; 1984; Galbraith et al., 1990; Galbraith, 2002; 2005; Ketcham, 2003; 2005; Ketcham 42 et al., 2007). 43

In contrast to the geometrical biases, biases related to the actual etching of confined tracks are 44 less well understood. It was soon clear that the etching conditions (etchant, concentration, du-45 ration and temperature) affected the lengths of confined tracks in apatite. Several experiments 46 with different etching protocols, listed in Jonckheere et al. (2017), showed that the mean length 47 of confined tracks first increased at a rapid rate up to a point where the tracks were considered 48 fully etched, following which it further increased at the much slower apatite etch rate (Laslett 49 et al., 1984). Watt et al. (1984) observed that the greater mean length of TinCLE's compared to 50 51 that of TinT's in the same sample could be explained by the time required for the host track to etch, as compared to the almost immediate penetration of the etchant through a crack to reveal 52 the TinCLE's. Ketcham (2003) found that geometrical biases alone cannot account for the prop-53 erties of confined track samples and proposed that "under-etching bias" was also an important 54 selection criterion. 55

Laslett et al. (1984) drew attention to the factors causing individual confined tracks to be etched 56 for a different time: "most confined tracks are over-etched in order to ensure that the etchant can 57 percolate down fractures and other tracks to etch all intersecting tracks (access time). Also, the 58 shortest tracks must be over-etched in order to ensure that the longest ones are fully revealed 59 60 (length)". These factors were integrated in numerical models (Rebetez et al., 1988; Ketcham and Tamer, 2021), which showed that the lengths of etched confined tracks span the range from 61 zero length to that of the longest track. This implies that the measured track length distribution 62 is to some extent the product of an active selection based on a geometrical criterion or operator 63 bias (Ketcham and Tamer, 2021). Tamer et al. (2019) demonstrated the practical importance 64 of track selection; two operators mutually rejected  $\sim 14\%$  of each other's selections. Ketcham 65 and Tamer (2021) guardedly concluded that the importance of selection "affects the fidelity of 66 thermal history modelling". 67

Step-etching of individual confined tracks in apatite showed that their lengths increase in erratic 68 fashion, with single 10s increments between  $\sim 0$  and >1 µm, decreasing overall as the etch time 69 increases (5.5 M HNO<sub>3</sub> at 21 °C; Jonckheere et al., 2017). This underscores the importance of the 70 etch times of individual tracks for modelling of the confined track length distribution. Aslanian et 71 al. (2021) and Jonckheere et al. (2022) proposed a new etch model and measured the rates for 72 Durango apatite etched in 5.5 M HNO<sub>3</sub> at 21 °C. This permits to calculate the true duration ( $t_E$ ) for 73 74 which an individual confined track has been etched from its orientation, shape and thickness. On the one hand, it offers a practical criterion for selecting tracks for thermal history modelling, that 75 is independent of a person's judgement, and independent of the factors that caused a specific 76 measured track to have been etched for a given length of time. On the other, it presents a tool for 77 investigating the factors controlling  $t_E$  or the  $t_E$ -distribution of the measured population. In the 78 next sections we investigate the influences of the track density and length using Durango apatites 79





annealed to nominal mean lengths of ~16, ~14, ~12 and ~10  $\mu$ m (Ketcham et al., 2015; Aslanian et al., 2022).

#### 2. Materials and Methods

To investigate the effects of partial annealing on the composition and properties of the confined 82 track population we carried out measurements on four prism sections of Durango apatite dis-83 tributed for inter-laboratory comparisons. The samples had been pre-annealed, neutron-irra-84 diated and - all except one - annealed again to create induced-track populations with nominal 85 mean lengths of ~16  $\mu$ m (sample 21-2), ~14  $\mu$ m (sample 21-4), ~12  $\mu$ m (sample 21-1) and ~10 86  $\mu$ m (sample 21-3). Details of the irradiation and annealing conditions are given in Ketcham et 87 88 al. (2015). The track densities measured in transmitted and reflected light are reported in Aslanian et al. (2021). 89

90 The samples were mounted in resin, ground on SiC papers, polished with 6-, 3-, and 1-µm diamond suspensions, and given a final polish with 0.04-µm silica suspension, until the surface 91 was free of scratches under reflected light, and only faint scratches reappeared after etching. 92 All samples were etched for 20 s in 5.5 M HNO<sub>3</sub> at 21 C° (Carlson et al., 1999). The confined 93 94 track imaging was carried out with a motorized Zeiss AxioImager Z2m microscope connected to a desktop computer running the Autoscan program. The samples were systematically scanned 95 in transmitted light at an optical magnification of  $250 \times (100 \times \text{Epiplan Neofluar dry objective})$ 96 and 2.5× optovar). We made image stacks of each confined track considered suitable for meas-97 urement. This meant that both its ends were free, distinct and well etched, and that a sharp 98 image of the entire track was contained within a single stack of six frames with a fixed spacing 99 of 0.25  $\mu$ m. For an 8- $\mu$ m track this corresponds to a dip angle of arcsin(1.25/8)  $\approx$  9° and an error 100 of <2%. The advantage of this criterion is that it is clear and amenable to mathematical descrip-101 tion. The disadvantage is that it introduces a bias in favour of short tracks proportional to their 102 reciprocal length, which acts counter to the conventional length bias (Laslett et al., 1982; 1984; 103 Galbraith et al., 1990). 104

Depending on the case, we extracted the best image from a stack or compressed a part of it to a 105 single image, converted it to eight-bit and loaded it in CorelDraw, cutting out a square frame con-106 taining a single track to add to the database (Figure 1a-d). For calculating its effective etch time 107  $(t_E)$  we measured its maximum width  $(r_0)$  at the intersection with its host track (TinT; Figure 108 **1e**) or, in rare cases, with a crack (TinCLE). For calculating the track etch rate  $(v_T)$ , we measured 109 a second width  $(r_1)$  at some distance  $(s_1)$  from the first along a straight section of track. This 110 works for tracks at most angles to the *c*-axis ( $\phi \lesssim 80^\circ$ ) but not for those at the highest ( $\phi \gtrsim$ 111 80°), where part of the channel is obscured by a diamond-shaped etch figure (Figure 1f; Jonck-112 heere et al., 2022). As this figure is bounded by the fastest etching faces, a precise  $t_E$ -estimate is 113 obtained from measurements of the distances between opposing sides ( $d_1$  and  $d_2$ ). A less precise 114 lower estimate can still be obtained from the width of the channel outside the diamond shape 115  $(r_0).$ 116

117 Some tracks at  $\geq$ 45° to the *c*-axis in the most annealed sample have an atypical shape due to "interrupted" etching at a "gap" in the latent track ("unetchable gap", Green et al., 1986). We 118 distinguish between "stepped" tracks where the gap was pierced during etching (Figure 1c) 119 and "gapped" tracks where it was not (Figure 1d; Galbraith, 2005). The latter are characterized 120 by their short lengths and irregular endings. We divided stepped tracks in three segments for 121 measurement (Figure 1g). The first, from the host track intersection to the gap, has length  $l_{1}$ ; 122 the second, from the intersection in the opposite direction, has length  $l_2$ ; the third, from the gap 123 to the other end, has length  $l_3$ . The maximum width at the intersection with the host track is  $r_0$ ; 124 those on either side of the gap are  $r_1$  and  $r_3$ ; the widths along segments 2 and 3 at a distance 125 from  $r_0$  and  $r_3$  are  $r_2$  and  $r_4$ . 126

From these measurements, we calculated the effective etch time  $(t_E)$  of each confined track as:

$$t_E(s) = 30 \frac{r_0(\mu m)}{v_R(\mu m/min)}$$
 (\$\phi \le 80°\$) (1a)







Figure 1. Etched confined tracks distinguished in this work: (a) continuous track at a low to mod-128 erate angle to the *c*-axis; (**b**) continuous track at a high angle to the *c*-axis, with a diamond shaped 129 etch figure at its intersection with the host track; (c) stepped track showing narrowing (arrow) 130 due an interruption of along-track etching; (d) gapped track with an abnormal termination (ar-131 row); (e) length (*l*), *c*-axis angle ( $\phi$ ) and width ( $r_0$ ) of track (a); the width at  $r_1$  and its distance ( $s_1$ ) 132 to  $r_{\theta}$  are used to calculate the cone angle ( $\theta$ ) and the etch rate ( $v_T$ ) of the track (equations 3a and 133 b); (f) measurement of the distances  $(d_1 \text{ and } d_2)$  between facing sides of the diamond shape at 134 track (b), bounded by the fastest etching apatite faces, used for calculating its effective etch time 135 (*t<sub>E</sub>*; equation 1b); (g) measurements of widths (*r*<sub>0</sub>, *r*<sub>1</sub>, *r*<sub>2</sub>, *r*<sub>3</sub>, and *r*<sub>4</sub>) of track (c) aimed at determin-136 ing its effective etch time  $(t_E)$ , the etch delay  $(t_D)$  at the constriction and the etch rates of the dif-137 ferent track sections; (h) measurement of the length (l), width ( $r_0$ ) and orientation ( $\phi$ ) of the gap-138 ped track in (d). 139





$$t_E(s) = 15 \frac{(d_1 + d_2)(\mu m)}{V_{R,MAX}(\mu m/min)} \qquad (\phi \gtrsim 80^{\circ})$$
(1b)

140 wherein  $v_R$  is calculated from the model of Aslanian et al. (2021), with  $\phi_W = 90 - \phi$ , and  $v_{R,MAX} \approx$ 141 3.0 µm/min:

 $v_R(\mu m/min) = -0.0071 \phi_W^2 + 0.2807 \phi_W + 0.2495 \quad (\phi_W \leq 20^\circ)$  (2a)

$$v_R(\mu m/min) = 0.00025 \phi_W^2 - 0.0633 \phi_W + 4.2500 \quad (\phi_W \gtrsim 20^\circ)$$
 (2b)

142 The track etch rate  $v_T$  is calculated as:

$$v_T(\mu m/min) = \frac{v_R(\mu m/min)}{\sin(\theta/2)}$$
(3a)

143 wherein  $\theta$  is the angle between facing straight margins of the track, calculated from  $r_0$ ,  $r_1$  and  $s_1$ 144 as:

$$\theta = 2 \arcsin\left(\frac{(r_0 - r_1)(\mu m)}{2 s_1(\mu m)}\right)$$
(3b)

The expected minimum and maximum widths for each track orientation are calculated from  $v_R$ (eqs. 2a and 2b) as:

$$r_{MAX}(\phi) \ (\mu m) = \left(\frac{2}{3}\right) (min) \ v_R(\phi_w) \ (\mu m/min) \tag{4a}$$

$$r_{MIN}(\phi) \ (\mu m) = \left(\frac{1}{5}\right) (min) v_R(\phi_w) \ (\mu m/min) \tag{4b}$$

where  $r_{MAX}$  refers to tracks etched for the full 20 s immersion time  $t_i$ , and  $r_{MIN}$  to the minimum time required for a track to be etched from its midpoint to both ends ( $\approx 7.5(\mu m)/75(\mu m/min)$ ; Aslanian et al., 2021). We also estimated the maximum ( $t_{E,MAX}$ ) and minimum ( $t_{E,MIN}$ ) effective etch times. The maximum assumes that a track etches from the moment that the sample is immersed in the etchant till it is taken out and rinsed. The minimum assumes that a track must be etched for long enough to reach a width of ~0.3 µm, in order to be selected for measurement (Aslanian et al., 2021)

$$t_{E,MAX}(\phi)(s) = t_I \tag{5a}$$

$$t_{E,MIN}(\phi) (s) = 30 \ \frac{0.3 \ (\mu m)}{v_R(\phi_w) \ (\mu m/min)}$$
(5b)

#### 3. Results and Discussion

From 2170 track images taken by one participant the other rejected 20 as not suitable for measurement. This <1% rejection rate is much lower than the averages of both participants in the investigation of Tamer et al. (2019; ~14%). However, the present is a one-way rate concerning a single set of images taken using one set of etching and observation conditions. Also in contrast to the present, the latter investigation was of in general lower densities of fossil tracks in <sup>252</sup>Cf-irradiated samples.

#### 3.1 Track lengths

Plots of the measured (*l*) and the *c*-axis projected lengths ( $l_P$ ; Donelick et al., 1999) of horizontal confined tracks against angles to the *c*-axis ( $\phi$ ) illustrate the known length shortening and increasing anisotropy with increasing annealing (**Figure 2**; Green et al., 1986; Donelick, 1991). The mean lengths ( $l_M$ ) are close to the values predicted by the annealing equations for Durango ap-





atite etched 20 s in 5.5 M HNO<sub>3</sub> at 21 °C (Ketcham et al., 1999; Table 1). The maximum differ-164 ence  $(0.15 \,\mu\text{m})$  is that between the measured mean length  $(10.45 \,\mu\text{m})$  and that predicted by 165 the fanning rectilinear model for the most annealed sample (21-3; 10.30 μm). All other differ-166 ences between measured and predicted mean lengths are  $<0.05 \ \mu m$ . The standard deviations 167 of the length distributions  $(s_M)$  are also in agreement with model predictions, with a maximum 168 difference between the measurements and models of 0.08 µm for sample 21-1 (Table 1). The 169 170 relationship between the standard deviations  $(s_M)$  and means  $(l_M)$  of the track length distributions as well as that between the *c*-axis (*lc*) and *a*-axis intercepts (*l*<sub>4</sub>) of ellipses fitted to the length vs. 171 orientation data are consistent with the equations of Donelick et al. (1999; Figure 3a). The agree-172 173 ment between our data and those reported in Carlson et al. (1999) shows that independent scientists working two decades apart on samples annealed at different conditions, using different 174 equipment and measurement methods, but the same etching protocol, nevertheless produce con-175 sistent results. We believe that this is not without significance and return to the issue later in the 176 discussion. 177

Figure 2 plots the *c*-axis-projected length of each track against its actual angle to the axis (Donelick 178 et al., 1999). The agreement between the mean c-axis-projected lengths ( $l_P$ ) and the annealing 179 models (Ketcham et al., 1999) is somewhat worse than for the non-projected lengths. The calcu-180 lated values are 0.21-0.33 µm above the predictions of both the rectilinear and curvilinear models 181 (Table 1). In contrast, the standard deviations ( $s_P$ ) are all within 0.05  $\mu$ m of their predicted values. 182 183 The relationship between  $s_P$  and  $l_P$  is again consistent with the equation of Donelick et al. (1999; **Figure 3b**). Except for a small offset of 0.2-0.5  $\mu$ m, the *c*-axis (*l<sub>PC</sub>*) and *a*-axis (*l<sub>PA</sub>*) intercepts of 184 regression lines fitted to the  $(l_P, \phi)$ -data are almost identical, indicating that *c*-axis projection ef-185 fectively eliminates the anisotropy of the track lengths in the annealed and unannealed samples 186 (Figure 3a). 187

Although it is most noticeable in the case of sample 21-3 (**Figure 2h**), *l<sub>P</sub>* is more tightly distributed 188 about the local mean at greater angles to the *c*-axis in all four samples. This is not related to the 189 measurements but to the *c*-axis projection, which funnels tracks at higher angles to the *c*-axis into 190 191 a narrower length interval than those at lower angles (Donelick et al., 1999). The *c*-axis projection thus trades the unequal means  $l_{PM}$  of the length distributions at different *c*-axis angles for unequal 192 standard deviations *s<sub>PM</sub>*. The effect is quite pronounced; the standard deviations of all confined 193 tracks lengths at  $<15^{\circ}$  to the *c*-axis are on average twice as high as those of tracks at  $>75^{\circ}$  to the 194 *c*-axis (Figures 3b and B1 of Appendix B). In the case of sample 21-3, *spm* decreases from ~0.8 195  $\mu$ m at ~0° to the *c*-axis to ~0.2  $\mu$ m at ~90°. It is not clear how this affects thermal histories mod-196 elled using *c*-axis projection, but it would appear that careful accounting for the orientations of 197 198 the measured tracks is essential to avoid artefacts.

**Figure B2** of appendix B shows the length histograms of tracks at 15° angular intervals, projected 199 200 onto the *c*-axis while preserving length differences, i.e., keeping the distance from each data point to the fitted ellipse fixed. This eliminates length anisotropy without affecting the distributions 201 about the means. However, because it applies to tracks annealed under identical conditions and 202 not to variable-temperature histories it is of no use for modelling. It is nevertheless interesting to 203 contrast both projections. The latter understands each length measurement as an estimate of the 204 mean length in a given orientation but departing from it due to accidents of track formation and 205 etching, but not due to annealing. The former interprets each single length measurement as the 206 mean of a population, whose value is a direct reflection of its annealing history, excluding random 207 effects. As we understand it, the former is assumption more correct but the latter a condition for 208 modelling. 209

However the assumption implicit in the common *c*-axis projection has interesting implications.
In contrast to individual track lengths, two mean track lengths projected onto the *c*-axis differ due
to their thermal histories, not due to accidents of track formation or etching, which do not affect
the means. Since annealing unidirectionally lowers the mean lengths, a population with a shorter







Figure 2. Measured (a-d) and c-axis projected (e-h) lengths of induced horizontal confined tracks 214 in the four studied samples plotted against angle to the *c*-axis: (**a**, **e**): unannealed; (**b**, **f**): annealed 215 10 h at 240 °C; (**c**, **g**): annealed 10 h at 288 °C and (**d**, **h**) annealed 10 h at 310 °C (Ketcham et al., 216 2015). White circles: continuous tracks; grey circles: stepped tracks; black circles: gapped tracks. 217 The solid lines in **a-d** are ellipses fitted to the data (excluding data for stepped and gapped tracks 218 219 in d); those in e-h are linear regression lines; n: number of measured lengths; l<sub>M</sub>: mean track length;  $s_M$ : standard deviation of the length distribution;  $l_c$ : c-axis intercept of the fitted ellipse;  $l_A$ : 220 *a*-axis intercept of the ellipse; *l<sub>PM</sub>*: mean *c*-axis projected length; *s<sub>PM</sub>*: standard deviation of the *c*-221 axis projected lengths;  $l_{PC}$ : *c*-axis intercept of the fitted regression line;  $l_{PA}$ : *a*-axis intercept of the 222 223 regression line.







Figure 3. (a) Relationship between the *c*-axis and *a*-axis intercepts of ellipses fitted to the measured 224 lengths (white circles) and regression lines fitted to the *c*-axis projected lengths (grey circles) of 225 confined tracks in the studies samples; solid lines represent published equations (RD: Donelick 226 et al., 1999; 1:1: isotropic trend), dashed lines have been fitted to the measurements in this work; 227 (b) relationship between the standard deviations and means of the distributions of the measured 228 (white circles) and *c*-axis projected lengths (grey circles) of horizontal confined tracks in the stud-229 230 ies samples; the orange circles represent the standard deviations of tracks at <15° and >75° to the 231 apatite *c*-axis.





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| Sample | 1     | φ(°) | <i>l</i> (μm) | <i>l</i> <sub>P</sub> (μm) | <i>r</i> ₀ (μm) | <i>r</i> 1 (μm) | <i>s</i> 1 (μm) | θ (°) | <i>d</i> (µm) | $t_E(s)$ | VT<br>(µm/min) |
|--------|-------|------|---------------|----------------------------|-----------------|-----------------|-----------------|-------|---------------|----------|----------------|
| 21-2   | Count | 637  | 637           | 637                        | 629             | 553             | 553             | 500   | 139           | 712      | 500            |
|        | Mean  | 53.1 | 16.1          | 16.4                       | 0.68            | 0.53            | 7.56            | 2.92  | 1.00          | 11.0     | 208            |
|        | Error | 0.89 | 0.03          | 0.02                       | 0.01            | 0.01            | 0.10            | 0.07  | 0.02          | 0.12     | 22             |
|        | S.Dev | 22.5 | 0.82          | 0.62                       | 0.23            | 0.18            | 2.33            | 1.63  | 0.21          | 3.22     | 118            |
| 21-4   | Count | 374  | 372           | 372                        | 337             | 337             | 337             | 335   | 49            | 366      | 335            |
|        | Mean  | 54.2 | 14.6          | 15.4                       | 0.79            | 0.63            | 6.04            | 3.53  | 1.05          | 11.9     | 165            |
|        | Error | 1.10 | 0.04          | 0.03                       | 0.01            | 0.01            | 0.16            | 0.10  | 0.03          | 0.17     | 18             |
|        | S.Dev | 21.2 | 0.82          | 0.57                       | 0.22            | 0.19            | 2.86            | 1.80  | 0.21          | 3.26     | 84             |
| 21-1   | Count | 320  | 320           | 320                        | 308             | 281             | 281             | 213   | 66            | 249      | 209            |
|        | Mean  | 53.5 | 12.5          | 13.9                       | 0.76            | 0.63            | 5.96            | 3.14  | 1.33          | 12.0     | 201            |
|        | Error | 1.23 | 0.05          | 0.03                       | 0.02            | 0.01            | 0.11            | 0.13  | 0.03          | 0.21     | 14.0           |
|        | S.Dev | 21.9 | 0.94          | 0.53                       | 0.27            | 0.23            | 1.87            | 1.89  | 0.28          | 3.37     | 115            |
| 21-3   | Count | 821  | 821           | 821                        | 724             | 713             | 713             | 684   | 165           | 826      | 684            |
|        | Mean  | 53.2 | 10.4          | 12.6                       | 0.67            | 0.52            | 4.93            | 4.03  | 1.00          | 10.6     | 159            |
|        | Error | 0.77 | 0.05          | 0.02                       | 0.01            | 0.01            | 0.07            | 0.11  | 0.02          | 0.12     | 26             |
|        | S.Dev | 22.1 | 1.53          | 0.54                       | 0.20            | 0.16            | 1.92            | 2.94  | 0.23          | 3.37     | 91             |

**Table 1A.** Average *c*-axis angles, lengths and widths of horizontal confined induced tracks in prism faces of Durango apatite and calculated track effective etch times  $t_E$  and track etch rates  $v_T$ . Etching conditions: 20 s in 5.5 M HNO<sub>3</sub> at 21 °C; the different measured lengths and widths are shown in Figure 1.

| Sample          |       | φ(°) | <i>l</i> (μm) | <i>l</i> <sub>P</sub> (μm) | <i>r</i> <sub>0</sub> (μm) | <i>r</i> 1 (μm) | <i>s</i> 1 (μm) | θ (°) | <i>d</i> (μm) | $t_E(s)$ | νı<br>(μm/min |
|-----------------|-------|------|---------------|----------------------------|----------------------------|-----------------|-----------------|-------|---------------|----------|---------------|
| Continu-<br>ous | Count | 731  | 731           | 732                        | 668                        | 658             | 658             | 629   | 103           | 735      | 629           |
|                 | Mean  | 50.6 | 10.7          | 12.7                       | 0.66                       | 0.52            | 4.86            | 3.56  | 0.97          | 10.7     | 82            |
|                 | Error | 0.80 | 0.05          | 0.02                       | 0.01                       | 0.01            | 0.07            | 0.08  | 0.02          | 0.13     | 1.9           |
|                 | S.Dev | 21.7 | 1.26          | 0.54                       | 0.20                       | 0.16            | 1.92            | 2.00  | 0.25          | 3.48     | 48            |
| Stepped         | Count | 78   | 78            | 61                         | 50                         | 49              | 49              | 49    | 52            | 79       | 49            |
|                 | Mean  | 73.9 | 8.93          | 12.6                       | 0.89                       | 0.42            | 5.90            | 10.1  | 1.05          | 10.3     | 39            |
|                 | Error | 1.31 | 0.17          | 0.04                       | 0.02                       | 0.02            | 0.21            | 0.77  | 0.03          | 0.23     | 3.0           |
|                 | S.Dev | 11.6 | 1.50          | 0.31                       | 0.17                       | 0.14            | 1.44            | 5.41  | 0.18          | 2.04     | 21            |
| Gapped          | Count | 12   | 12            | 12                         | 6                          | 6               | 6               | 6     | 10            | 12       | 6             |
|                 | Mean  | 76.6 | 5.02          | 11.9                       | 0.58                       | 0.49            | 4.14            | 3.51  | 1.00          | 8.52     | 152           |
|                 | Error | 3.76 | 0.27          | 0.11                       | 0.08                       | 0.08            | 1.30            | 1.09  | 0.07          | 0.82     | 49            |
|                 | S.Dev | 13.0 | 0.95          | 0.39                       | 0.20                       | 0.19            | 3.20            | 2.67  | 0.21          | 2.84     | 119           |

Table 1B. Breakdown of the data for sample 21-3 in Table 1A according to the appearance of

the etched track (Figure 1; continuous, stepped or gapped).





238 mean length has experienced more severe annealing than one with a longer mean length. Thus, 239 the order of projected lengths corresponds to the order of formation of the tracks. The number and 240 lengths of confined tracks thus divide a sample's history in a corresponding number of time-intervals to each of which corresponds a specific mean length. This allows to convert its age and length 241 distribution directly to a Tt-path, without the need to search Tt-space. Allowance can be made for 242 biases, e.g., the fact that shorter tracks represent a larger population and time interval than longer 243 tracks. The uncertainties concerning the exact time of formation of each population can also be 244 taken into consideration (Jonckheere and Ratschbacher, 2010). The single solution represents 245 the core of the set of T,t-solutions consistent with the data and measurement uncertainties 246 (Rebetez et al., 1994). 247

248 Sample 21-3 is characterized by the occurrence of gapped and stepped tracks (Figure 1) at >45° to the *c*-axis (**Figure 2d**). This is less than the calculated value for Durango apatite, both 249 for the predicted ( $\phi_{alr}$  >60°;  $\phi_{alr}$  = angle of accelerated length reduction; Donelick et al., 1999) 250 251 and the measured lengths ( $\phi_{alr}$  >80°). The occurrence of stepped and gapped tracks increases 252 with increasing angle to the *c*-axis (Figure 4a). This correlates with a rapid drop of the apatite etch rate along the track axis from <2  $\mu$ m/min at 45° to ~0.5  $\mu$ m/min perpendicular to the *c*-253 axis (Figure 4b). This, together with the simultaneous appearance of gapped and stepped 254 tracks, argues for the formation of non-etchable gaps (Green et al., 1986) rather than for a more 255 256 general form of accelerated length reduction (Donelick et al., 1999), and therefore for a breakup of the tracks and a discontinuous structure of latent tracks (Paul and Fitzgerald, 1992; Paul, 257 1993). It appears that *c*-axis projection converts the lengths of gapped and stepped tracks to 258 values within a narrow length interval that depends little on their original lengths and orienta-259 tions. It thus seems that less precise measurements of these lengths or orientations are not a 260 great concern. 261

The gapped tracks in sample 21-3 have lengths  $<7 \mu m$  (Figure 1h; Green et al., 1986), which is 262 somewhat shorter that the main segments of the stepped tracks, intersected by the host tracks 263 264 (**Figure 1g**;  $l_1 + l_2$ ). The total lengths of the stepped tracks, including the section after the gap (**Figure 1g**;  $l_3$ ), are on average ~0.2 µm shorter than continuous tracks with the same orienta-265 tions (**Figure 4a**;  $l_t$ ) due to the delay caused by the gap. The lengths of continuous tracks ( $l_t$ ), the 266 final lengths of stepped tracks ( $l_1 + l_2 + l_3$ ), that of their main sections ( $l_1 + l_2$ ), and even the lengths 267 of gapped tracks  $(l_q)$  appear to exhibit a similar linear decrease with increasing angle to the *c*-268 axis. In contrast,  $l_3$  exhibits no visible dependence on orientation and no discernible preferred 269 length between zero and half the total track length (**Figure 4c**). This suggests that non-etchable 270 gaps can form more or less along the entire length of the tracks at this stage of annealing. Substi-271 tuting  $r_1$  and  $r_3$  for  $r_0$  in equation (1a) we calculated the difference of the effective etch time  $\Delta t_{E1,3}$ 272 273 on either side of the gap and interpreted it as the time required to pierce the gap at the etch rate of undamaged apatite  $v_R$  in the direction of the track in order to estimate the size of the gaps. The 274 results range from <10 nm to >100 nm, with nevertheless a pronounced mode at  $\sim30$  nm, which 275 is consistent with TEM observations on latent tracks (Figure 4d; Paul and Fitzgerald, 1992; Paul, 276 277 1993) (Table 3).

## 3.2 Track widths

Figure 5 plots the widths of the confined track against their angles to the *c*-axis. The data for all 278 samples define a similar boomerang shape, with minima parallel and perpendicular to the *c*-axis, 279 and the greatest widths at 60-75°. The solid curve (1) defines the maximum achievable widths 280 after 20s immersion in 5.5 M HNO<sub>3</sub>, calculated with the etch rates of Aslanian et al. (2021). Few 281 282 tracks attain that width due to the variable but finite access times required for the etchant to travel (etch) down the host tracks and across to the confined tracks (Rebetez et al., 1988; 283 284 Ketcham and Tamer, 2021). For illustration, the shaded band under (1) represents access times 285 of up to 4 seconds. There also appears to be a distinct lower limit (2), which we interpret as the 286 minimum width for a track to be selected for measurement. Our lower limit at  $\sim 0.3 \ \mu m$  is lower 287 than the value of Aslanian et al. (2021;  $\sim$ 0.5 µm), perhaps due to their longer immersion time or







288 **Figure 4**. (a) Measurements of stepped and gapped tracks plotted against angle to the *c*-axis ( $\phi$ ); 289 white circles: lengths of the main sections of stepped tracks (Figure 1g;  $l_1 + l_2$ ); grey: full lengths of stepped tracks  $(l_1 + l_2 + l_3)$ ; black: lengths of gapped tracks  $(l_1)$ ; grey-edged circles and dashed 290 line show the full lengths of continuous tracks  $(l_T)$  in the same interval, for comparison; the col-291 our-coding along the horizontal axis refers to angular intervals with decreasing apatite etch rates 292 in the etch rate plot (b); (b) apatite etch rate plot from Aslanian et al. (2021), colour-coded to 293 294 correspond with (a); (c) frequency distribution of the lengths of the distal segments of stepped tracks (13; past the constriction); (d) frequency distribution of the sizes of perforated gaps be-295 tween the main and distal segments of stepped tracks, calculated from the difference of the widths 296 at  $r_1$  and  $r_3$  (Figure 1g). 297







Figure 5. (a-d) Widths of horizontal confined tracks (Figure 1e; ro; excluding high-angle tracks) 298 plotted against their angles to the *c*-axis ( $\phi$ ), and the main factors that guided their selection; (1) 299 maximum width of confined tracks that started etching between 0 and 4 seconds after immer-300 sion; (2) overall minimum width of tracks accepted for measurement unless condition (3) ap-301 plies; (3) minimum width of a track etched to both ends, calculated using equation (6); (4) ap-302 proximate limit above which tracks close to the surface become too wide to remain confined; (e-303 h) distributions of track angles to the c-axis for the four studied samples; white: continuous 304 tracks; grey: stepped tracks; black: gapped tracks; the short-dashed line is a polynomial fit to the 305 combined data for the four samples (including high-angle tracks); the long-dashed line is a poly-306 nomial fit to the range of widths at each orientation delineated by constraints (1)-(4) (i.e., exclud-307 ing high-angle tracks). 308





to different track selection criteria. Over a broad range of orientations ( $\leq 45^{\circ}$  to  $\geq 75^{\circ}$ ), the minimum track widths are above this limit. This is due to the minimum time required for a track to etch to its ends, so that it is suitable for measurement. For the sake of calculation, we assume that both ends need to attain a width of ~0.2 µm for a track to be measurable. Assuming that a track of length *l* etches from the middle toward both ends, we find that  $t_{E,MIN} = \frac{1}{2} (l/v_T + 0.2/v_R)$ . The corresponding minimum width is:

$$w_{MIN}(\mu m) = \left(\frac{l(\mu m)}{2}\right) \left(\frac{v_R(\mu m/min)}{v_T(\mu m/min)}\right) + 0.2 (\mu m)$$
(6)

This estimate depends on both the length and orientation of the tracks, and is shown as curve (3) 315 in **Figure 5**. Despite the fact that these limits are diffuse, i.e., dependent on variable access times 316 and intersection points, they appear to constrain the data rather well, at least in a qualitative 317 318 manner. Within these bounds, there is nevertheless a conspicuous lack of tracks wider than  $\sim 1.25$ 319  $\mu$ m at 60-75° to the *c*-axis (4). To attain greater widths the access times must be short, which means that the tracks must be close to the surface, which is itself lowered at a slow rate during 320 the immersion of the sample. We conclude that the lack of tracks wider than  $\sim$ 1.25 µm is due to 321 the fact that broad, shallow tracks come to intersect the surface as etching proceeds. This is a 322 form of surface proximity bias (Galbraith et al., 1990), although there is in our data no obvious 323 correlation with the track length. 324

The distance  $\Delta w$  between the lower of the upper limits (1 and 4) and the higher of the lower limits 325 (2 and 3) is proportional to the range of track widths consistent with these four criteria at a given 326 orientation. **Figures 5e-h** plot the average  $\Delta w$  for our four samples against angle to the *c*-axis, 327 ignoring the small differences between them. The result is a good fit to the combined and individ-328 ual distributions of the track orientations. The agreement with the measured distributions is in 329 fact better than shown in **Figure 5e-h** since the latter include high-angle tracks not included in 330 Figure 5a-d because their effective etch times were calculated as in Figure 1f. This is an indica-331 332 tion that etching- and observation-related factors have as least as much influence on the angular frequencies of confined tracks as geometrical factors (Ketcham, 2003). The angular distributions 333 334 of our four samples are indistinguishable when the stepped and gapped tracks are included in the measurements of the most annealed sample. This extends the range of constant angular bias 335 to within the domain of track break-up, i.e., somewhat beyond the limit assumed by Galbraith et 336 al. (1990) and Ketcham (2003). 337

Thus, in samples with low to moderate annealing, confined tracks are in the first place selected 338 based on their width, in the form of a minimum width independent of their length and orientation 339 (2) and of the rate of width increase (etch rate) in a given orientation, which determines the frac-340 tion of tracks above the threshold (1). This selection is modified by factors depending on width 341 and length (3, 4), i.e., surface proximity bias (Galbraith et al., 1990) and the fact that longer tracks 342 343 on average attain a greater width before they are etched to their ends (Laslett et al., 1984). However, the influence of the track length on the angular frequencies of horizontal tracks is not no-344 ticeable within the resolution of our measurements and the annealing range of our samples ( $I_M$  = 345 16.1 -10.5 μm). It seems fitting to describe these as "(under-)etching biases" (Ketcham 2003). At 346 347 advanced stages of annealing, track selection comes to be controlled by "track loss" (Green et al., 348 1986; Ketcham, 2003).

## 3.3 Effective etch time

**Figures 6a-d** plot the effective etch times  $t_E$  of individual confined tracks against their angles to the *c*-axis;  $t_E$  is calculated from the widths in **Figure 5a-d** using equations (1a) and (2a-b) and the apatite etch rates of Aslanian et al. (2021). The effective etch times of the high-angle tracks ( $\phi$ >80°), not included in **Figure 5a-d**, are calculated with equation (1b) and plotted with a different symbol; the two estimates are consistent with each other. The selection boundaries (1-4) are converted to effective etch time limits in the same manner. Together the data again define boomerang shapes, although inverted compared to those in **Figure 5a-d**, with the thinnest tracks at low







**Figure 6.** (a-d) Effective etch times of horizontal confined tracks ( $t_E$ ) in the four studied samples 356 plotted against their angles to the c-axis ( $\phi$ ), and the main factors, (1)-(4), that guided track se-357 lection, taken from Figure 5; white: low and moderate angle tracks (Figure 1e); grey: high-angle 358 tracks (Figure 1f); (e-h) effective etch time distributions for the four studied samples; white: con-359 tinuous tracks; grey: stepped tracks; black: gapped tracks; the short-dashed line is a polynomial fit 360 to the combined data for the four samples;  $t_{E,M}$  and  $s_{t_E}$ : arithmetic mean and standard deviation of 361 the etch-time distribution (i-l) *c*-axis projected lengths (*l*<sub>P</sub>) of confined tracks plotted against their 362 effective etch times (*t<sub>E</sub>*); white: low and moderate angle tracks (**Figure 1e**); grey: high-angle tracks 363 (Figure 1f); the solid black line is a linear regression line fitted to the low and moderate angle 364 365 data (white); the dashed line is a regression line fitted to the high-angle data (grey).





366 and high angles to the *c*-axis having the longest etch times and the broadest tracks the shortest. This illustrates the fact that those tracks are selected that have the right properties for being se-367 lected and that the dominant selection criterion across all samples, lengths and orientations is 368 width. Longer low-angle tracks and shorter high-angle tracks widen at a comparable slow rate 369 (Aslanian et al., 2021) and are both measured after etch times close to the total immersion time, 370 371 while tracks in intermediate orientations, which widen at a faster rate, are selected after a shorter 372 time. High-angle tracks like that in **Figure 1b** are an exception because their effective etch time 373 is calculated from the diamond shaped figure at its intersection with the host track (Jonckheere et al., 2022). 374

375 The etch time distributions are all right skewed, with similar means and standard deviations (Fig**ure 6e-h**). This might be unexpected, given the different track lengths and densities of the sam-376 377 ples (Aslanian et al., 2022), which affect the number and length of the host tracks, their distances to the nearest confined tracks, and therefore the access time and the etch time distributions. The 378 distributions in **Figure 6e-h** are however those of *selected* tracks, not of the sampled population 379 as such, and as long as it contains tracks meeting fixed selection criteria their distribution remains 380 381 the same. The etch time distribution is controlled by the threshold width (2) and the immersion 382 time (1). This does not exclude geometrical biases; apart from through length bias (Laslett et al., 383 1982), track length influences the selection through constraints (3) and (4). However, their effect 384 on samples with simple length distributions, like those investigated here, appears to be small. The operator influences track selection through the threshold width (2) and the assessment 385 whether tracks are etched to their ends (3). These decisions are not independent as a high thresh-386 old width implies that most selected tracks are etched to their ends. E.g., Figure 6a-d suggests 387 388 that a threshold width of  $\sim 0.7 \,\mu m$  would ensure that all selected track lengths are suitable for measurement. The actual value is somewhat higher because (3) refers to tracks etched from the 389 middle towards both ends. A track that etches from one end towards the other requires a longer 390 etch time, during which it acquires a greater thickness; equation (6) is also based on rather ad 391 392 *hoc* estimates but the conclusion stands. **Figure 6e-h** shows, based on the same reasoning and 393 with the same proviso, that most tracks etched for an effective duration of  $\sim 10$  seconds are 394 etched to their ends, which lends support to the (20 + 10)-s etching protocol proposed by Tamer and Ketcham (2023). 395

We cannot, at this stage, estimate the precise effects of each constraint on the confined track se-396 397 lection, but we can make a distinction between hard constraints (1) and (2) and soft constraints (3) and (4), which depend on the track length, but have less effect on the selection. The threshold 398 399 width (2) can shift over a fraction of a micrometre depending on the operator, but the maximum width (1) is a function of the immersion time and the anisotropic apatite etch rate. In conse-400 quence, the confined track sample selected for measurement will depend on the immersion time 401 and etch rate, and thus on the etchant and apatite compositions. This means that the anisotropic 402 apatite etch rate is the main cause of the different angular distributions of confined tracks in ap-403 404 atites with different Dper/Dpar ratios (Barbarand et al., 2003; Ketcham, 2003; Ravenhurst et al., 2003). However, not because of a target-projectile relationship (Galbraith et al., 1990; Galbraith, 405 2002; Ketcham, 2003) but because of the variation of the apatite etch rate with orientation 406 (Aslanian et al., 2021). 407

A series of recent studies throw light on the influence of the etching protocol, experimental con-408 ditions (transmitted vs. reflected light) and selection criteria on measurements of step-etched 409 confined tracks in Durango apatite (Tamer et al., 2019; Tamer and Ketcham, 2020; 2023; 410 411 Ketcham and Tamer, 2021). The overall conclusion is that substantial differences of the measured mean track length arise from the etching protocol and selection but less from the actual measure-412 ments. Individual mean lengths 10-20 standard errors from the overall sample means (Ketcham 413 et al., 2015) are interpreted as due to differences in the evaluation of the roundedness of the 414 etched track ends. This contrasts with the excellent agreement of our current measurements of 415 416 the same samples with the annealing models based on the calibration data of Carlson et al. (1999). 417 We submit that if track selection is indeed for the most part controlled by constraints (1) and (2), 418 with modifications from (3) and (4), as our single-track data indicate, then it must be possible to perform reproducible and meaningful confined track length measurements which are suitable 419





for modelling. An unbiased systematic search for horizontal confined tracks using transmitted
light is adequate for this purpose. Deliberate or inadvertent biasing, carelessness or inexperience
will affect the results, but these should be treated as statistical outliers, not as an indication that
track lengths are fluid.

Figure 6i-l shows a weak positive correlation between the *c*-axis projected lengths and effective etch times of individual confined tracks. Precise rates of increase are difficult to estimate because of the weak correlation and large scatter of  $t_E$  and  $l_P$ . The best estimate is based on the high-angle tracks because their etch times are calculated from the greatest widths and a single etch rate (**Figure 1f**; equation 1b). The result (2.3 µm/min) is somewhat lower than the value for fossil tracks, which drops from 3.4 µm/min at 5 s to 2.9 µm/min at 20 s (Aslanian et al., 2021). It is closer to the rate for the non-projected lengths of fossil and induced tracks reported by Tamer

431 and Ketcham (2020; 2.6 μm/min).

#### 3.4 Track etch rate

432 The calculated track etch rates span an order of magnitude (60-600 µm/min) in all samples and 433 the v<sub>T</sub>-distributions are right-skewed in each case (Figure 7a-d). This can be a measurement ef-434 fect because  $v_T$  is obtained by from the apatite etch rate  $v_R$  perpendicular to the track margins and the subtended angle  $\theta$  (equation 3a). Because  $\theta$  averages 3-4° and is difficult to measure, signifi-435 cant errors of 1° and more cannot be excluded. In that case, the harmonic mean  $v_T$  are less biased 436 central estimates than the arithmetic means. These range from  $\sim 160 \,\mu$ m/min for the unannealed 437 sample to  $\sim 120 \,\mu$ m/min for the most annealed. There is however no connection between the ex-438 439 tent of partial annealing and the track etch rate. Given the uncertainties it is reasonable to suppose that there is no demonstrable effect of annealing on the etch rate of induced tracks. The 440 overall mean (harmonic or arithmetic) for the four studied samples is ~140 µm/min. This is twice 441 the value for unannealed fossil tracks of Aslanian et al. (2021). Their arithmetic mean is 75 442  $\mu$ m/min; the harmonic mean for the same data is ~63  $\mu$ m/min. Even considering the uncertain-443 ties, it appears that fossil tracks etch at a slower rate than comparable, partially annealed induced 444 tracks, which can be related to their different annealing behaviour (Wauschkuhn et al., 2015a). 445 Step-etch experiments also revealed differences between fossil and unannealed induced tracks 446 (Jonckheere et al., 2017). 447

Our calculation assumes that a single v<sub>T</sub> value characterizes the entire track (equation 3a; Figure 448 **1e**). This implies a constant-core  $v_T$ -model rather than a linear model (Tamer and Ketcham, 2020; 449 Ketcham and Tamer, 2021), one in which the core extends over most of the etchable track length, 450 451 except for perhaps  $\sim 1 \, \mu m$  at either end, where the average etch rate drops as the damage becomes intermittent (Paul and Fitzgerald, 1992; Paul, 1993; Toulemonde et al., 1994; Li et al., 452 2011; 2012). This is at variance with the view that  $v_T$  reflects the variation of the damage or di-453 ameter along the track, resulting from its formation (Fleischer et al., 1969; Price et al., 1973). It is 454 nonetheless reasonable to consider that depleted, amorphous or porous track cores (Fleischer et 455 al., 1965; Szenes, 1996a; b; Li et al., 2010) could act as conduits along which the etchant advances 456 at a rate that does not depend on the degree of material depletion, disorder, or porousness of the 457 track core, but on the rate of fluid flow, the reaction rate, or the exchange rate of acid and reaction 458 products at the solid-fluid interface. This could explain the different (etchant-strength/etch-time) 459 protocols used for revealing fission tracks in apatite over much the same length, the revelation of 460 ion tracks from different particles with the same etchant, and the different shapes of etched tracks 461 produced with low and high acid concentrations (references in Jonckheere et al., 2017; Jonck-462 heere and Van den haute, 1996). 463

464 Constant-core models avoid a number of problems of linear models, and those with variable  $v_T$  in 465 general. Due to the "motorboat effect", linear  $v_T$  models generate different-shaped confined tracks 466 depending on whether they are etched with the  $v_T$ -gradient, from their centre towards both ends, 467 or against the gradient, from one end towards the centre (Fleischer et al., 1969; 1975; Paretzke 468 et al., 1973). The track outlines in consequence exhibit concave and convex curvature not ob-469 served in actual samples (**Figure 8**). The fact that confined tracks and surface tracks are straight







Figure 7. (a-d) Etch-rate (VT-) distributions of horizontal confined tracks in the four studied sam-470 ples; white: continuous tracks; grey: stepped tracks; black: gapped tracks; the dashed line is a fit to 471 the combined data; *v<sub>TM</sub>* and *s<sub>vT</sub>*: arithmetic means and standard deviations of the etch-rate distri-472 butions; *v*<sub>TH</sub> and *v*<sub>TG</sub>: harmonic and geometric means; (e-h) *c*-axis projected lengths (*l*<sub>P</sub>) plotted 473 against the etch rates ( $v_T$ ) of horizontal confined tracks; white: low and moderate angle tracks (Fig-474 ure 1e); grey: high-angle tracks (Figure 1f); the solid black lines are linear regression lines fitted 475 to the combined data; (i-l) c-axis projected lengths ( $l_P$ ) plotted against the apatite etch rates ( $v_R$ ) 476 in the direction of the tracks; the solid black lines are linear regression lines fitted to the combined 477 data. 478





-edged irrespective of whether etching started from the centre or from the end is in itself a strong 479 indication of a constant  $v_T$  (Figure 9). A linear  $v_T$  model creates an excess of underetched tracks, 480 e.g., whenever etching starts at the end of a track or its effective etch time is less than the immer-481 sion time (Figure 8). It is possible that underetched confined tracks are not observed or not se-482 lected for measurement (Ketcham and Tamer, 2021), but this is not the case for surface tracks, 483 which are not selected. Nevertheless, all measurement of surface tracks reveal a deficit of short 484 485 tracks rather than an excess (Dakowski, 1978; Grivet et al., 1993; Jonckheere et al., 1993; Jonckheere and Van den haute, 2002). Surface tracks with lengths approaching those of confined tracks 486 also could not exist (Figure 8d). On reflection, there are other difficulties with linear v<sub>T</sub> models. 487 They predict that a substantial fraction of the confined tracks exhibit a rapid length increase at 488 the end of a standard immersion time, which is not observed in step-etch experiments (Jonckheere 489 et al., 2017; Tamer et al., 2019; Tamer and Ketcham, 2020, 2023). A linear model also implies that 490 a latent track cannot be etched over its entire length at the track etch rate  $v_T$  in less than infinite 491 492 time as  $v_T \rightarrow 0$  at its ends.

Our findings support a constant-core model extending over most of the etchable length of a track. 493 Nevertheless, v<sub>T</sub> must decrease towards the ends (Figure 8 of Laslett et al., 1984; Figure 1 of Watt 494 495 and Durrani, 1985; Figures 7c and 8c of Aslanian et al., 2021). In a constant core model, it is not obvious that this decrease correlates with a gradual change of the latent-track properties. Latent-496 track and etched-track studies (Paul and Fitzgerald, 1992; Paul, 1993; Li et al., 2011; 2012; 497 Wauschkuhn et al., 2015b; Jonckheere et al., 2017) indicate an intermittent structure, made up of 498 499 damaged sections etching at a rate comparable to  $v_T$ , alternating with recrystallized sections etching at a rate comparable to the apatite etch rate  $v_R$  (unetchable gaps; Green et al., 1986). In that 500 case, the rate of length increase  $v_L$  is an average of  $v_T$  and  $v_R$  over a short section of track. A de-501 502 creasing  $v_L$  towards the track tip causes it to become rounded (motorboat effect; Fleischer et al., 1969; Ketcham and Tamer, 2021), until a gap cannot be breached and the end of the etched track 503 becomes bounded by basal and prism faces (Aslanian et al., 2021). The transition of rounded to 504 polygonal track tips would be the point at which an individual track is fully etched (Tamer and 505 506 Ketcham, 2023). Our results, however, reveal no correlation between the *c*-axis projected lengths and the track (Figure 7e-h) or the apatite etch rate (Figure 7i-l). This can mean that no correla-507 tion exists or that it is weak and obscured by the statistical variation of the length and etch-rate 508 509 measurements.

510 Considering that a constant-core model implies that  $v_T$  is insensitive to the properties of the latent 511 track, it is surprising that substantial differences appear to exist between individual tracks. At this stage, we cannot exclude that most of the  $v_T$ -variation is related to its measurement and cal-512 culation, and that induced fission tracks in apatite in fact have the same or a narrow range of v<sub>T</sub>-513 values. Even in that case, the harmonic means of our measurements provide valid first-order v<sub>T</sub>-514 estimates indicating that the cores of fossil and induced tracks etch at different rates. Price et al. 515 516 (1973) ascribed the different etching behaviour of "old" and "new" tracks in meteoritic minerals to a rearrangement of the damage. In apatite, this phenomenon could be related to ageing (Gleadow 517 and Duddy, 1981) or seasoning (Durrani and Bull, 1987; Wauschkuhn et al., 2015a). However, the 518 significance of the core etch rates for the lengths of etched tracks is at present unclear (Ketcham 519 and Tamer, 2021). 520

## 4. Conclusions

Our investigation is concerned with the factors affecting the selection of confined tracks for meas-521 urement and T,t-modelling. It was conducted on induced tracks in four prism sections of Durango 522 apatite etched for 20 s in 5.5 M HNO<sub>3</sub> at 21 °C. Their nominal mean track lengths are  $\sim$ 16,  $\sim$ 14, 523  $\sim$ 12, and  $\sim$ 10  $\mu$ m (Ketcham et al., 2015), and their transmitted-light track densities are between 524  $\sim$ 2.9 and  $\sim$ 1.9 10<sup>6</sup> cm<sup>-2</sup> (Aslanian et al., 2022). The tracks were selected by scanning the samples 525 in transmitted light using two conscious criteria: (1) the entire track could be captured in an im-526 age stack with a depth of  $1.25 \,\mu m$ ; (2) both ends of the track were distinct. One operator collected 527 528 the images; the second vetted them, without deliberating with the first, and performed the measurements. In order to characterize each selected track as fully as possible, these included length, 529 orientation, width, cone angle and other dimensions needed for calculating their individual effec-530 tive etch times. 531







Figure 8. Geometries of unannealed induced confined tracks in apatite etched 20 s in 5.5 M HNO<sub>3</sub> at 21 °C, calculated with the linear  $v_T$ -model of Ketcham and Tamer (2021; Figure 15 and Table 2) (a) track etched from the middle towards both ends; (b) confined track intersected at  $\frac{1}{4}$  length from one end; (c) at  $\frac{1}{8}$  length; (d) at the end; brighter grey shades indicate increasing etch times from 5 to 20 s.







Figure 9. Microscope images and contours of confined tracks with a host track intersections close to the middle (**a**, **c**) and close to the end (**b**, **d**), both showing straight edges indicative of a constant  $v_{T7}$  (**e**) compressed image stack of straight-edged surface tracks in a prism face of Durango apatite etched 20 s in 5.5 M HNO<sub>3</sub> at 21 °C, showing no excess of short tracks or deficit of long tracks; the fact that all confined tracks, however thin compared to surface tracks with the same orientations, are etched over something approaching their full lengths also argues for a constant high track etch rate (arrows).





In contrast to the large variation reported in Ketcham et al. (2015), our results are in good agree-544 ment with the annealing equations and original data for this apatite and etchant (Carlson et al., 545 1999; Ketcham et al., 1999). This leads us to question whether all variation between analysts can 546 be reduced to individual judgements as to which tracks are suitable for measurement (Ketcham 547 and Tamer, 2021). Our data show that *c*-axis projection (Donelick et al., 1999) eliminates all track 548 length variation with orientation but leads to a narrowing of the length distribution with increas-549 ing angle to the c-axis, with consequences for T,t-modelling that merit thorough consideration. 550 Our sample annealed to a mean length of  $\sim 10 \,\mu m$  contains segmented tracks, whose number in-551 creases and whose length decreases with their angle to the *c*-axis, and thus with a decrease of the 552 553 apatite etch rate. Their appearance is due to a local obstruction delaying the progress of the etch-554 ant, i.e., to unetchable gaps (Green et al., 1986) rather than accelerated length reduction (Donelick, 1991). Our measurements show that the gaps are between  $\sim 10$  and  $\sim 100$  nm, with a mode at 555 556  $\sim$ 30 nm, confirming estimates based on transmission electron microscope observations (Paul and Fitzgerald, 1992; Paul, 1993). 557

The confined track selection is in the first place determined by a threshold width and in the sec-558 ond place by the requirement that the tracks are etched to their ends. In most cases the first con-559 dition implies the second, which decreases in importance are the tracks are shortened by anneal-560 ing. The remaining cases correspond to orientations in which the track width increases fastest 561 compared to its length. The selection is further limited by the fact that the widest confined tracks, 562 563 which are also the shallowest, come to intersect the surface, which eliminates them from consid-564 eration. This is a surface proximity bias with the emphasis on the width rather than on the length of the tracks (Galbraith et al., 1990; Galbraith, 2005). The number of suitable confined tracks at 565 each angle to the c-axis is proportional to the difference between the minimum and maximum 566 widths corresponding to these constraints. The angular distribution of the selected tracks is there-567 fore a close reflection of the range of possible track widths, with little influence from geometric 568 biases. Because the confined track selection depends on the track widths rather than lengths, it 569 570 is almost unaffected by annealing, at least up to the point that selective track loss occurs. A change 571 of the etch rates, due to the etchant or apatite composition would in contrast have a definite effect 572 (Ketcham, 2003).

573 We calculated the true duration for which each individual confined track has been etched (effective etch time) from its width and the apatite etch rate perpendicular to its axis (Aslanian et al., 574 2021). The results remain consistent with the constraints, converted to etch times in the same 575 manner. These are somewhat indistinct because they depend to an extent on the length of each 576 confined track and on where along its length the host track intersects it. In favourable orienta-577 578 tions, an unannealed track intersected in the middle can etch in five seconds. This places a lower limit 100 µm/min on the track etch rate. The etch time distributions are right-skewed with a 579 mode at just over half the immersion time. The thinnest tracks at low and high angles to the *c*-580 axis need the longest etch times and also appear to be less affected by surface proximity bias than 581 the wider tracks. The etch time distribution shows no demonstrable dependence on the extent of 582 annealing despite the different geometrical relationship between the unetched host tracks and 583 confined tracks. This illustrates a principle known from other selection processes, nl. that those 584 entities are selected that have the right qualities for being selected, in this case the track width is 585 the overriding condition. 586

Our calculated track etch rates span an order of magnitude, with arithmetic means of 160 to 200 587  $\mu$ m/min and harmonic means of 120 to 160  $\mu$ m/min. The  $v_T$ -distributions are right-skewed with 588 values ranging up to 600  $\mu$ m/min, which are thought to be overestimates because the  $v_T$ -calcula-589 tion is vulnerable to measurement error. In contrast to earlier reports (Tamer and Ketcham, 590 591 2020; Ketcham and Tamer, 2021), we find no evidence for an increase of  $v_T$  with annealing or for 592 variation of  $v_T$  along the tracks. We favour a constant etch rate over most of the track length because it seems inevitable that a linear model creates a great excess of short tracks, which even if 593 excluded from the confined track sample (Ketcham and Tamer, 2021), would produce a concave-594 upwards distribution of the projected lengths of surface tracks (Laslett et al., 1982; Rebetez et al., 595





1990), which is not supported by measurements (Dakowski, 1978; Grivet et al., 1993; Jonckheere 596 et al., 1993; Jonckheere and Van den haute, 2002). Calculation also shows that for a linear vr-597 598 model the etchant will often fail to reach the ends of the confined tracks within the standard im-599 mersion time (Figure 10). A constant-core model does not have this drawback; for the mean etch rates determined in this work (140 µm/min), it takes under seven seconds to etch the longest 600 track from end to end. A high  $v_T$  would also account for the common experience that almost all 601 the confined tracks in a sample have close to their full lengths, and for the fact that the measured 602 lengths exhibit no correlation with  $v_T$ . A high  $v_T$  would also limit the effect of several host tracks 603 intersecting a confined track, which we estimate to be negligible unless it is intersected at distant 604 points at the same time. This implies that the dependence of the track length on effective etch 605 606 time is a limited effect due to etching at its endpoints at a rate  $v_L$  intermediate between the track and the apatite etch rates (Aslanian et al., 2021). The question of the track etch rate nevertheless 607 presents challenges as it seems reasonable that if  $v_T$  exhibits no variation along a track, it would 608 also show little variation from track to track. Our measured  $v_T$  also do not correlate with the ex-609 tent of annealing of the induced tracks, but are nevertheless twice as high as the value for fossil 610 tracks (75 µm/min; Aslanian et al., 2021). 611

## Supplement link

## Author contribution

- RJ designed the experiment, collected the microscope images, analysed the data and prepared the 612
- manuscript. Dr. M.T. Tamer made a substantial contribution to the measurements but desires not 613
- to be listed as co-author. 614

# **Competing interests**

The author declares that he has no conflict of interest. 615

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Figure 10. Comparison of etchant progress along an unannealed induced track-in-track-in-track 619 in Durango apatite etched 20 s in 5.5 M HNO<sub>3</sub> at 21 °C. Yellow: seconds after immersion based on 620 the linear model of Ketcham and Tamer (2021); green: seconds after immersion based on the 621 effective etch times and track etch rates calculated from the measured widths using the etch rates 622 of Aslanian et al. (2021). The etchant takes ~6 seconds to advance down the host track and across 623 to intersect  $\alpha$ ; both models are in rough agreement about the time taken to progress from there 624 to the intersection of  $\alpha$  and  $\beta$ , and somewhat less along the main section of  $\beta$ . However, the calcula-625 tions diverge towards the ends of both tracks due to the rapid decrease of the track etch rate in 626 the linear model. Because of the expenditure of  $\sim$ 6 seconds of etch time before the etchant can 627 begin to etch  $\alpha$ , it is unable to reach the ends of  $\alpha$  or  $\beta$  within the immersion time of the sample. 628 According to the calculation based on Aslanian et al. (2021) the etchant reaches the furthest end 629 of  $\beta$  with three seconds to spare, which is enough to widen it to ~0.2  $\mu$ m and for it to be observed 630 631 and measured (Appendix A).



632

633 634



# Appendix A: etch time calculation



**Figure A1.** Linear etch rate model for unannealed induced tracks (after Ketcham and Tamer, 2021; Figure 15); (1) etching against the etch-rate gradient, (2) etching with the gradient.

 $\frac{dv}{dx} = c \tag{A1}$ 

$$\frac{dv}{dt}\frac{dt}{dx} = c \tag{A2}$$

$$\frac{dv}{v} = c \ dt \tag{A3}$$

 $\ln(v) = c t + u \tag{A4}$ 

$$t = 0 \rightarrow x = a \rightarrow v = v_a$$

$$\ln(v_a) = u \tag{A5}$$

$$\ln\left(\frac{v}{v_a}\right) = c t \tag{A6}$$

$$v = v_a \, e^{ct} \tag{A7}$$

$$\frac{dx}{dt} = v_a e^{ct} \tag{A8}$$

$$dx = v_a \, e^{ct} \, dt \tag{A9}$$

$$x = \frac{v_a}{c} e^{ct} + z \tag{A10}$$

$$t=0 \to x=a$$

 $a - \frac{v_a}{c} = z \tag{A11}$ 

$$x = \frac{v_a}{c} e^{ct} + \left(a - \frac{v_a}{c}\right) \tag{A12}$$

$$x = a + \frac{v_a}{c} (e^{ct} - 1)$$
 (A13)

(1)  $v_a = c a$ 

$$x = a + \frac{c a}{c} (e^{ct} - 1)$$
 (A14)

$$x = a \ e^{ct} \tag{A15}$$

$$t = \frac{1}{c} \ln\left(\frac{x}{a}\right) \tag{A16}$$

(2) 
$$v_a = c \left(\frac{l}{2} - a\right)$$

$$x = a + \left(\frac{l}{2} - a\right) (e^{ct} - 1)$$
 (A17)

 $t = \frac{1}{c} \ln\left(1 + \frac{2(x-a)}{(l-2a)}\right)$ (A18)









**Figure A2.** A track-in-track-in-track in an apatite prism face etched 20 s in  $5.5 \text{ M HNO}_3$  at 21 °C, with the orientations and lengths of different sections indicated.





Figure A3. Calculated etch-time profiles of the tracks in Figure A2.





# Appendix B: c-axis projections

This appendix shows a comparison of alternative *c*-axis projections of confined track lengths. **Figure B1** shows the results of the known method (Donelick et al., 1999), which is implemented
in programs for modelling thermal histories. It assumes that each measured track length
represents the mean of a different population. The projection in **Figure B2** assumes that each
measured length represents a different track from the same population distributed about a single
ellipse.



Figure B1. Normalized frequency distributions of the *c*-axis-projected track lengths in the four studied samples for populations within different angular intervals (15°) to the apatite *c*-axis. The different populations have consistent mean lengths but the standard deviations of the distributions show a marked decrease with increasing angle to the *c*-axis. This trend is clearest for samples 21-2 and 21-3, which are based on a greater number of measurements; *l<sub>PM</sub>*: mean; *s<sub>PM</sub>*: stand-ard deviation.







Figure B2. Normalized frequency distributions of the *c*-axis-projected track lengths in the four
 studied samples for populations within different angular intervals (15°) to the apatite *c*-axis. For
 comparison with Figure B1, the projection preserves the absolute differences between individual
 lengths.





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