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## Standardless fission-track dating of the Durango apatite age standard

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### ABSTRACT

Five dating strategies were used for determining the standardless fission-track age of the Durango apatite. These use the same fossil-track densities but differ in the manner in which the induced-track densities are determined. A conventional age calculation, without correction for experimental factors, gives inconsistent ages with method-related differences >15%. Correcting for these factors brings the ages in line with each other and with the reference age but leaves no room for a partial-annealing correction based on the confined-track lengths. Three further reasons suggest that a length correction is not appropriate. (1) The evidence for length-based corrections is inconclusive. (2) The plateau age of the Durango apatite is consistent with its apparent fission-track age to within 1%. (3) The calculated effective etchable length of the fossil tracks agrees within error with that of the induced tracks; both are further consistent with the measured mean length of confined induced tracks. The circumstance that the (U,Th)/He ages of the accepted and proposed apatite age standards are consistent with their reference ages leaves no margin for a lowered fission-track age resulting from partial annealing, although the case of the Durango apatite itself is inconclusive because of its exceptional crystal size. It is conjectured that the shortening of the fossil tracks in the Durango apatite is due to a lowering of the track etch rate over time. In this case, annealing equations fitted to induced-track data underestimate the extent of confined-track-length reduction in geological samples.

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### 1. Introduction: premises

The progress of the fission-track dating method can be divided into a time before and an almost equal time since the recommendation of the  $\zeta$ -calibration (Hurford, 1990a, b). During the first period, it was dogged by problems associated with the  $^{238}\text{U}$ -fission constant, neutron-fluence measurement and experimental factors related to the dating techniques (Bigazzi, 1981). Since the acceptance of the  $\zeta$ -calibration and the development of apatite (T,t)-path modelling (Crowley, 1985; Green et al., 1989), it has met with great practical success. A decade before the  $\zeta$ -recommendation, Bigazzi (1981) proposed two strategies for overcoming the problems of the fission-track method. One comes down to what became the  $\zeta$ -calibration. The other involves solving the methodological problems in three steps: (1) achieving accurate neutron-fluence measurements, (2) eliminating errors associated with the dating techniques, and (3) establishing a reliable correction for partial annealing of the fossil tracks. The question of the  $^{238}\text{U}$ -fission constant would then resolve itself.

Our contribution sets out from the premise that neutron-fluence measurements using the recommended metal monitors (Au, Co) and well-

thermalized irradiation facilities are no longer an issue (Bigazzi et al., 1990; Curvo et al., 2013; De Corte et al., 1991, 1995; Van den haute et al., 1988, 1998). Our second assumption is that the matter of the fission constant has been settled following recent redeterminations (Eikenberg et al., 1993; Guedes et al., 2000, 2003; Suzuki, 2005; Yoshioka et al., 2005) and reassessments (Holden, 1989; Holden and Hoffman, 2000). Our work concentrates instead on steps (2) and (3) of Bigazzi's (1981) research program by using five dating methods, requiring method-specific corrections for experimental factors, for determining the standardless fission-track age of the Durango apatite.

### 2. Adjusted age equation

Natural apatite contains trace amounts of uranium. A fraction of the isotope  $^{238}\text{U}$  undergoes spontaneous fission over geological time. The nuclear fragments, moving in opposite directions through the lattice, produce a single ca. 20- $\mu\text{m}$  long (Bhandari et al., 1971; Jonckheere, 2003a, b), <10-nm diameter (Afra et al., 2011; Jaskierowicz et al., 2004; Lang et al., 2015; Li et al., 2014; Miro et al., 2005; Paul and Fitzgerald, 1992; Schauries et al., 2014) fossil fission track. The number of tracks per unit volume ( $N_S$ ) is proportional to the age ( $t$ ) and  $^{238}\text{U}$  content ( $U_{238}$ ) of the sample and the spontaneous-fission rate ( $\lambda_F$ ) of  $^{238}\text{U}$  (Eq. 1).

$$N_S = \lambda_F t U_{238} \quad (1)$$

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Fission tracks also form when apatite is irradiated in a reactor. The thermal neutrons in the reactor spectrum cause a fraction of the  $^{235}\text{U}$  isotope to fission, producing induced fission tracks. The number of induced tracks per unit volume ( $N_I$ ) is proportional to the concentration of  $^{235}\text{U}$  ( $U_{235}$ ) and the fission rate  $R_F = \sigma \phi$ , wherein  $\phi$  is the thermal-neutron fluence and  $\sigma$  the effective cross-section for  $^{235}\text{U}$ -fission by thermal-neutron capture (Eq. 2).

$$N_I = \sigma \phi U_{235} \quad (2)$$

It follows from Eqs. (1) and (2) that:

$$t = (\lambda_F)^{-1} (N_S/N_I) (U_{235}/U_{238}) \sigma \phi \quad (3)$$

Eq. (1) neglects the depletion of  $^{238}\text{U}$  by spontaneous fission and  $\alpha$ -disintegration. An exact equation derived by Price and Walker (1963) implies that Eq. (1) is accurate to within 1% for  $t < 10^{10}$  a. Eq. (2) does not account for burn-up of  $^{235}\text{U}$  or for the production of induced tracks by epithermal- and fast-neutron fission of  $^{235}\text{U}$ . Eqs. (1) and (2) furthermore do not consider spontaneous or induced fission of other isotopes. In practice, these are negligible for the neutron fluences required for dating apatite and for irradiations in well-thermalized reactor channels (Tagami and Nishimura, 1992; Wagner and Van den haute, 1992). Eq. (3) is adequate for dating samples up to 100 Ma.

There is a complication related to the  $^{235}\text{U}$ -fission rate  $R_F = \sigma \phi$  in Eq. (3), when  $\phi$  is calculated from the  $\gamma$ -activities of co-irradiated metal-activation monitors (Au, Co). This calculation is in practice based on the “simple” Høgdahl formalism, which can cope with non-ideal epithermal-neutron spectra, and gives the conventional sub-cadmium neutron fluence  $\phi_S$ . The Høgdahl formalism is however not suited for non- $(1/\nu)$  reactions such as thermal-neutron induced  $^{235}\text{U}$  fission. The appropriate formalism for non- $(1/\nu)$  reactions is the more sophisticated Westcott convention, which includes the  $g(T_n)$  correction factor (Westcott  $g$ -factor;  $T_n$ : Maxwellian neutron temperature) and uses the conventional total (Westcott) neutron fluence  $\phi_W$ . It can be shown that in well-thermalized irradiation channels  $\phi_W \approx \phi_S$  ( $\phi_W = \beta \phi_S$ ). In channel 8 of the Thetis nuclear reactor (Universiteit Gent; thermal to epithermal fluence ratio:  $f = 155$ ; epithermal spectrum parameter:  $\alpha = 0.10$ ), used for the irradiations in this work,  $\beta = 1.0025$  (Jonckheere, 1995; Verheijke, 1994) and  $g(T_n) = 0.9832$  (Holden, 1999;  $T_n = 27$  °C; Wagemans et al., 1988). Another consideration concerns the rare-earth-element content of the Durango apatite, in particular the Gd concentration of up to 200 ppm. Its high neutron-absorption cross-section (ca. 49,000 b) can cause fluence depression in the samples of up to ca. 4% (Naeser and Fleischer, 1975). The calculated correction factor for thermal-neutron shielding ( $G_{th}$ ; De Corte et al., 1991) in the Durango sections dated here is  $G_{th} = 0.986 \pm 0.001$  (Bellemans, pers. com.; Jonckheere, 2003b). Combining these factors, the fission rate  $R_F = \sigma \phi$  in Eq. (3) can be rewritten as:

$$R_F = (g(T_n) \sigma_0) (G_{th} \beta \phi_S) = 0.972 (\sigma_0 \phi_S) \quad (4)$$

$\sigma_0$  is the conventional (2200 m/s) fission cross-section of  $^{235}\text{U}$  ( $\sigma_0 = 586 \pm 3$  b; Holden and Holden, 1989) and  $\phi_S$  the conventional subcadmium neutron fluence calculated from the measured  $\gamma$ -activities of the co-irradiated metal-activation monitors using the Høgdahl formalism.

The natural isotopic ratio  $I = \theta_{235}/\theta_{238}$  ( $7.253 \cdot 10^{-3}$ ; Cowan and Adler, 1976) is substituted for  $(U_{235}/U_{238})$  in Eq. (3) for calculating the age of a sample. This is accurate if  $U_{238}$  and  $U_{235}$  in Eqs. (1)–(3) refer to the same elemental uranium concentration, i.e. if the fossil and induced fission-track densities,  $N_S$  and  $N_I$ , have been produced by the same concentration of uranium atoms. Natural apatite minerals often exhibit within-grain and between-grain uranium inhomogeneities. For this reason, the estimates of  $N_S$  and  $N_I$  are either each averaged over

representative grain samples (multi-grain or population methods) or both measured in the same grains (single-grain or matched-areas methods; Gleadow, 1981; Galbraith, 1981).

The external-detector method (Gleadow, 1981) is the single-grain method most used for dating. The fossil tracks are counted in a polished internal apatite surface and the induced tracks in an external detector irradiated in contact with it. Thus, the fossil tracks result from uranium fission on both sides of the etched apatite surface ( $4\pi$ -geometry) whereas the induced tracks result from uranium fission on one side of the surface of the external detector ( $2\pi$ -geometry). The ratio of the fossil to induced-track densities ( $\rho_S/\rho_I$ ) is therefore multiplied by a geometry factor  $G (= 2\pi/4\pi)$ . This also applies to the re-etch method (Gleadow, 1981), where the induced tracks are counted in an external apatite surface.

The Durango apatite dated in this work consists of cm-sized crystals, which were cut into ca. 1/2-mm thick sections for the specific purpose of comparing the fission-track ages of basal and prism faces. Part of the analyses was carried out with a population method. In our case this did not involve different aliquots but different sections from a single crystal (Section 3). One section was annealed and irradiated for counting the induced tracks, while the adjoining section, which was neither annealed nor irradiated, was used for counting the fossil tracks. In the presence of uranium-concentration gradients, this presents a risk of systematic error. To avoid it, both sections were re-irradiated in contact with muscovite external detectors. A correction factor  $U$  can then be calculated from the induced-track densities  $\rho_{ED}$  in both external detectors and the respective neutron fluences  $\phi_S$ :

$$U = (\rho_{ED}/\phi_S)_I / (\rho_{ED}/\phi_S)_S \quad (5)$$

The subscripts outside the brackets refer to the irradiations of the sections with fossil (S) and induced (I) tracks. The single-grain dating methods present a similar problem. For the external-detector and re-etch methods (Gleadow, 1981), it is assumed that half the uranium that produced the fossil tracks is not available for producing induced tracks during neutron irradiation. In the presence of significant short-range uranium variation perpendicular to the apatite surface, this assumption is invalid. We have no means of accounting for it, and assume that it is negligible ( $U = 1$ ). With the repolish method (Gleadow, 1981), there is a small offset between the sections used for counting the fossil and induced tracks due to the intervening repolish. It is again assumed that no correction is required.

$N_S$  and  $N_I$  in Eq. (3) are volumetric track densities. In practice, the fossil and induced tracks intersecting the detector surface are enlarged by etching and their areal densities ( $\rho_S$  and  $\rho_I$ ) are determined by counting under a microscope. Because of etching and observation effects, the track-counting efficiencies  $[\eta q]_S$  and  $[\eta q]_I$  (Jonckheere, 1995, 2003b; Wagner and Van den haute, 1992) are in general  $< 1$ . For dating methods where  $\rho_S$  and  $\rho_I$  are determined in different track-registration geometries, it cannot be assumed that  $[\eta q]_S = [\eta q]_I$  (Jonckheere and Van den haute, 1998, 1999, 2002). This is accounted for by introducing a procedure factor  $Q = [\eta q]_I / [\eta q]_S$  and substituting  $(N_S/N_I) = Q (\rho_S/\rho_I)$  in Eq. (3).

It is expedient to introduce an etch-time factor  $T$  for the re-etch method (Gleadow, 1981), in which the fossil tracks are etched before ( $\rho_S(t_E)$ ;  $t_E$ : etch time) and after irradiation ( $\rho_S(2t_E) + \rho_I(t_E)$ ). Because of the etch-time difference, it should not be assumed that the fossil tracks are revealed and identified the same in both counts ( $[\eta q]_S(2t_E) \neq [\eta q]_S(t_E)$  or  $\rho_S(2t_E) \neq \rho_S(t_E)$ ).  $T$  can be determined from step-etch experiments ( $T = \rho_S(t_E)/\rho_S(2t_E)$ ).  $T$  is important for correcting for the effect of residual etch figures, i.e. etch pits that persist after the lower track end-points have been overtaken by surface etching (Jonckheere and Van den haute, 1996).

A fission fragment crossing from mineral to external detector leaves a damage trail in both. However, not every etched track in the mineral corresponds to one in the external detector (Iwano and Danhara,

1998; Iwano et al., 1992, 1993; Jonckheere, 1995, 2003b) due to their different track-revelation thresholds (range deficits; Fleischer et al., 1975). A fraction of the fission fragments crossing the contact between an apatite sample and a muscovite external detector leaves an etchable track in the mica but not in the apatite (Jonckheere, 2003b). This requires an additional correction factor  $R$  ( $= 1.21 \pm 0.03$ ; range deficit factor) in Eq. (3).

The number of etched tracks intersecting a unit surface ( $\rho$ ) is a function of their etchable length ( $l$ ; Fleischer et al., 1975). Due to geological annealing, the etchable lengths of fossil tracks ( $l_s$ ) in natural apatites are shorter than those of induced tracks ( $l_i$ ). A correction factor  $L$  can be included in Eq. (3) for dating the onset of track retention, i.e. for calculating the age of the oldest surviving track, allowing for subsequent partial annealing. The relationship between the mean length ( $l/l_0$ ) and density ( $\rho/\rho_0$ ) of induced tracks has been investigated in annealing experiments (Green, 1988; Laslett et al., 1984; Naeser et al., 1989; Watt and Durrani, 1985; Watt et al., 1984). The equations fitted to the data (Crowley, 1993; Guedes et al., 2004; Ketcham, 2005; Ketcham et al., 2000; Lutz and Omar, 1991; Naeser et al., 1989; Tello et al., 2006; Willett, 1997) agree on an approximate 1:1 relationship between ( $\rho/\rho_0$ ) and ( $l/l_0$ ) down to  $l/l_0 \leq 0.7$ . The fossil tracks in the Durango apatite are ca. 10% shorter than the induced tracks (Gleadow et al., 1986; Green, 1988; Green et al., 1986). The length-correction factor is thus given by:  $L \approx l_i/l_s$ , although its exact value would be slightly higher due to the broadening of the fossil-track-length distribution, and somewhat dependent on the annealing equations and (T,t)-history (Wauschkuhn et al., 2015a).

The lengths used for age correction are those of the fossil ( $l_s$ ) and induced ( $l_i$ ) confined tracks. However, with the aid of the track-counting efficiencies ( $\eta q$ ) and known physical constants, it is possible to calculate the effective lengths of the fossil ( $l_{s,0}$ ) and induced ( $l_{i,0}$ ) tracks from the corresponding track densities ( $\rho_s, \rho_i$ ). This requires a known reference age, an independent estimate of its uranium concentration, and that the thermal-neutron fluence has been measured with metal-activation monitors. The equations for calculating the uranium content from the track densities ( $\rho_{ED}$ ) in co-irradiated external detectors (Enkelmann et al., 2005a; Hoffmann et al., 2008), the standardless fission-track ages ( $\phi$ -ages; Jonckheere, 2003b) and the effective length

of the fossil and induced tracks (Jonckheere, 1995) are summarized in Table 1, with the requisite numerical values.

### 3. Samples and experiments

The experiment was carried out on the Durango apatite from the Oligocene Cerro de Mercado iron-ore deposit (Durango, Mexico), where it occurs in association with martite within a volcanic series (Alva-Valdivia et al., 2001; Corona-Esquivel et al., 2007, 2009; Foshag, 1929; Lyons, 1988; McDowell et al., 2005; McDowell and Keizer, 1977; Swanson et al., 1978). The distinctive cm-sized transparent yellow crystals contain inclusions and exhibit chemical zonation (Hasebe et al., 2013; Johnstone et al., 2013; Marks et al., 2012; Pellas and Perron, 1984). Its crystal properties ( $a = b = 9.3955 \text{ \AA}$ ;  $c = 6.8801 \text{ \AA}$ ; Stock et al., 2015; Young et al., 1969) and chemical composition are well established, with substantial substitutions of LREE at the  $\text{Ca}^{2+}(2)$ -site and HREE at the  $\text{Ca}^{2+}(1)$ -site, charge balanced by  $\text{Na}^+/\text{Ca}^{2+}$  and  $\text{Si}^{4+}/\text{P}^{5+}$  substitutions (Rønso, 1989) and a F:Cl-ratio of ca. 8:1. (Barbarand and Pagel, 2001a, b; Barbarand et al., 2003a; Belousova et al., 2002; Carlson et al., 1999; Goldoff et al., 2012; Marks et al., 2012; McCubbin et al., 2010; Morishita et al., 2008; Patiño Douce et al., 2011; Roeder et al., 1987; Rogers et al., 1984; Teiber et al., 2015; Yang et al., 2014; Young et al., 1969). The actinide concentrations are ca. 7–22 ppm U and 150–380 ppm Th (Abdullin et al., 2014; Boyce and Hodges, 2005; Chew et al., 2014; Johnstone et al., 2013; Kimura et al., 2000; Morishita et al., 2008; Soares et al., 2014; Young et al., 1969).

Table 2 summarizes some geochronological data relating to the Durango apatite; classical fission-track ages are not included. The Fission Track Working Group of the I.U.G.S. Subcommittee on Geochronology recommended the Durango apatite as one of two apatite age standards for fission-track dating (Hurford 1990a, b). It has since also been adopted as an age-reference material for apatite (U,Th)/He dating and for other dating methods as well. Although the reference age of the Durango apatite is not specified in the recommendation, it is understood that the recommended value is that based on the K/Ar ages of ignimbrites bracketing the Mercado Iron Member, determined by F.W. McDowell (McDowell and Keizer, 1977), reported by Naeser and Fleischer (1975) and recalculated by Green (1985) with the constants

**Table 1**  
Equations, physical constants and numerical values for calculating the uranium concentrations, the fission-track ages and plateau age of the Durango apatite and the effective lengths of the fossil and induced tracks. Reactor-specific values refer to channel 8 of the Thetis reactor (Universiteit Gent). Values specific to the track counts and length measurements refer to the etching conditions in this work. Errors are  $1\sigma$ .

Equation	Factor	Value	Reference
(6) Uranium concentration:	$d_A$	$3.2 \pm 0.1 \text{ g cm}^{-3}$	McConnel (1973)
	$N_A$	$6.022 \cdot 10^{23}$	Avogadro's number
	$A_U$	238.08	mass uranium atom
	$\theta_{235}$	0.00720	Steiger and Jäger (1977)
	$l$	$7.253 \cdot 10^{-3}$	Cowan and Adler (1976)
(7) FT age (no subtraction):	$\lambda_\alpha$	$(1.551 \pm 0.007) \cdot 10^{-10} \text{ a}^{-1}$	Jaffey et al. (1971); Holden (1989)
	$\lambda_F$	$(8.45 \pm 0.10) \cdot 10^{-17} \text{ a}^{-1}$	Holden and Hoffman (2000)
	$\sigma_0$	$586 \pm 3 \text{ barn}$	Holden and Holden (1989)
(8) FT age (w. subtraction):	$g(T_n)$	0.9832	Holden (1999)
	$G_{th}$	$0.986 \pm 0.001$	Bellemans (pers. com.)
	$\beta$	1.0025	Jonckheere (1995)
	$G, g_{IS}, g_{ED}$	$\frac{1}{2}$ or 1	Gleadow (1981); Jonckheere (2003b)
	$Q = [\eta q]_{IS/ES/ED}/[\eta q]_{IS}$		Method-dependent procedure factor
(9) Length of induced tracks:	$[\eta q]_{IS}$	$0.91 \pm 0.01$	Jonckheere and Van den haute (2002)
	$[\eta q]_{ES}$	$1.01 \pm 0.01$	Jonckheere and Van den haute (2002)
	$[\eta q]_{ED}$	$0.91 \pm 0.01$	Jonckheere and Van den haute (2002)
	$[2R]_A/[2R]_M$	$0.962 \pm 0.017$	Jonckheere (2003b)
	$R$	$1.21 \pm 0.03$	Jonckheere (2003b)
(10) Length of fossil tracks:	$U$	Table 3	Enkelmann et al. (2005a)
	$T$	Table 5	Jonckheere and Van den haute (1996)
	$l_s$	$14.5 \pm 0.1 \text{ \mu m}$	Jonckheere and Van den haute (2002)
	$l_i$	$16.3 \pm 0.1 \text{ \mu m}$	Jonckheere and Van den haute (2002)
	$l_M$	$20.5 \pm 0.3 \text{ \mu m}$	Bigazzi (1967); Nagpaul et al. (1974); Guedes et al. (2008)
	$t_R$	$31.4 \pm 0.3 \text{ Ma}$	McDowell and Keizer (1977); Green (1985)

**Table 2**

Published geochronological and induced and fossil track-length data for the Durango apatite. *N*: number of measurements; method: decay process on which the age determination is based; (U,Th)/He does not mean that Sm is not considered (see references for methodological details);  $t_E$  (s): etching time;  $C_E$  (M HNO<sub>3</sub>): etchant concentration;  $T_E$  (°C): etchant temperature; RT: room temperature; <sup>(1)</sup> <sup>207</sup>Pb-corrected <sup>238</sup>U/<sup>206</sup>Pb-age; <sup>(2)</sup> uncorrected <sup>232</sup>Th/<sup>208</sup>Pb-age; <sup>(3)</sup> Concordia age (65 μm spot); <sup>(4)</sup> Concordia age (110 μm spot); <sup>(5)</sup> <sup>207</sup>Pb-corrected mean age; <sup>(6)</sup> <sup>204</sup>Pb-corrected Concordia age; <sup>(7)</sup> calculated with (U,Th)-concentrations from Zeitler et al. (1987); <sup>(8)</sup> calculated with (U,Th)-concentrations from Young et al. (1969) and Naeser and Fleischer (1975); <sup>(9)</sup> laser degassing; <sup>(10)</sup> furnace degassing; <sup>(11)</sup> Durango apatite uranium standard; <sup>(12)</sup> Mud Tank apatite uranium standard; <sup>(13)</sup> number of dated units; <sup>(14)</sup> tracks etched 10 min after irradiation; <sup>(15)</sup> tracks etched 41 days after irradiation. Errors recalculated to 1σ.

Age (Ma)	<i>N</i>	Method	Reference	
<i>Individual measurements</i>				
30.6 ± 1.2 <sup>(1)</sup>  32.5 ± 0.6 <sup>(2)</sup>	19 19	U/Pb Th/Pb	Chew et al. (2011)	
33.0 ± 1.2	45	U/Pb	Chew and Donelick (2012)	
31 ± 2	18	U/Pb	Li et al. (2012)	
32.2 ± 2.7 <sup>(3)</sup>  32.0 ± 1.5 <sup>(4)</sup>	10 5	U/Pb	Thomson et al. (2012)	
32.0 ± 0.3 <sup>(5)</sup>  31.8 ± 0.2 <sup>(6)</sup>	36 36	U/Pb	Chew et al. (2014)	
31.1 ± 0.3	12	U/Pb	Cochrane et al. (2014)	
31.4 ± 0.3	14	K/Ar	McDowell and Keizer (1977); Green (1985)	
31.4 ± 0.1	4 <sup>(13)</sup>	Ar/Ar	McDowell et al. (2005)	
43.2 <sup>(7)</sup>  32.2 <sup>(8)</sup>	1	(U,Th)/He	Zeitler et al. (1987)	
33.9	1	(U,Th)/He	Wolf et al. (1996)	
27.5 ± 1.5	1	(U,Th)/He	Warnock et al. (1997)	
32.3 ± 0.2	25	(U,Th)/He	House et al. (1999)	
32.1 ± 1.7 <sup>(9)</sup>  32.1 ± 1.7 <sup>(10)</sup>	11 16	(U,Th)/He	House et al. (2000)	
31.0 ± 0.2	24	(U,Th)/He	McDowell et al. (2005)	
31.4 ± 0.3	34	(U,Th)/He	Solé and Pi (2005)	
32.8 ± 1.8	11	(U,Th)/He	Foeken et al. (2006)	
30.9 ± 0.9	17	(U,Th)/He	Spiegel et al. (2009)	
34.1 ± 0.6	5	(U,Th)/He	Gong et al. (2013)	
32.2 ± 1.0	15	(U,Th)/He	Johnstone et al. (2013)	
30.5 ± 2.2	–	(U,Th)/He	Mahéo et al. (2013)	
31.0 ± 0.3	61	(U,Th)/He	Shan et al. (2013)	
30.6 ± 0.7	3	(U,Th)/He	Zeng et al. (2013)	
32.6 ± 1.5	6	(U,Th)/He	Legrain et al. (2014)	
32.6 ± 0.7	3	(U,Th)/He	Cox et al. (2015)	
33.7 ± 1.8	9	(U,Th)/He	Hoke et al. (2015)	
31.7 ± 9.4	2	(U,Th)/Ne	Gautheron et al. (2006)	
34.5 ± 3.3	9	(U,Th)/Ne	Cox et al. (2015)	
34.2 ± 8.6	1	(U,Th)/(Ne,He)	Gautheron et al. (2006)	
29.7 ± 1.9	1	FT ICP-MS	Hasebe et al. (2004)	
31.2 ± 0.2	104	FT ICP-MS	Abdullin et al. (2014)	
31.4 ± 2.5 <sup>(11)</sup>  31.8 ± 2.4 <sup>(12)</sup>	1 1	FT ICP-MS	Soares et al. (2014)	
<i>Lab averages</i>				
32.1 32.0	>30	(U,Th)/He	Caltech (Farley, 2000, 2002)	
31.0 ± 0.9	70	(U,Th)/He	CSIRO (Evans et al., 2005)	
31.9 ± 0.1	169	(U,Th)/He	Arizona (Reiners and Nicolescu, 2006)	
32.1 ± 1.1	156	(U,Th)/He	Yale (Min et al., 2006)	
31.7 ± 0.3	59	(U,Th)/He	Tübingen (Danišik et al., 2008)	
31.1 ± 0.9	23	(U,Th)/He	Curtin (Danišik et al., 2012)	
30.8 ± 1.4	38	(U,Th)/He	Waikato (Piotraschke et al., 2015)	
Track length (μm)	$t_E$ (s)	$C_E$ (M)	$T_E$ (°C)	Reference
<i>Induced tracks</i>				
15.2 ± 0.1	180	0.8	RT	Green (1981)
15.0 ± 0.1–15.2 ± 0.2	45	0.8	RT	Watt et al. (1984)
15.2 ± 0.1–17.1 ± 0.1	45–180	0.8	RT	Watt and Durrani (1985)
15.9 ± 0.1–16.5 ± 0.1	20	5.0	20	Gleadow et al. (1986); Green et al. (1986)
15.9 ± 0.1	20	5.0	20	Green (1988)
16.2 ± 0.1–16.6 ± 0.1 <sup>(14)</sup>	25	5.0	23	Donelick et al. (1990)
16.2 ± 0.1–16.3 ± 0.1 <sup>(15)</sup>	25	5.0	23	Donelick et al. (1990)
16.2 ± 0.1–17.5 ± 0.1	15–60	5.5	21	Carlson et al. (1999)
15.3 ± 0.8	55	0.8	22	Barbarand and Pagel (2001b)
16.3 ± 0.2	50	0.8	20	Iunes et al. (2002)
16.3 ± 0.1	60	0.4	25	Jonckheere and Van den haute (2002)
15.8 ± 0.1–16.1 ± 0.1	20	5.0	20	Barbarand et al. (2003b)
15.8 ± 0.1–16.0 ± 0.1	20	5.0	20	Barbarand et al. (2003a)
17.0 ± 0.1–17.3 ± 0.1	47	5.0	20	Ravenhurst et al. (2003)
16.1 ± 0.1–16.3 ± 0.1	50	1.3	20	Tello et al. (2006)
16.1 ± 0.1–16.2 ± 0.1	20–40	5.5	21	Jonckheere et al. (2007)
16.2 ± 0.1–16.4 ± 0.1	15–35	4.0	25	Jonckheere et al. (2007)
16.2 ± 0.1–16.5 ± 0.1	20	5.5	21	Chew and Donelick (2012)
15.8 ± 0.1	20	5.0	20	Schmidt et al. (2014)
<i>Fossil tracks</i>				
14.1 ± 0.1–14.8 ± 0.1	20	5.0	20	Gleadow et al. (1986)
14.2 ± 0.1	20	5.0	20	Green (1988)
14.9 ± 0.1	65	0.4	25	Jonckheere et al. (1993)
13.9 ± 0.2–14.9 ± 0.2	50	0.8	20	Iunes et al. (2002)

(continued on next page)



Table 2 (continued)

Track length ( $\mu\text{m}$ )	$t_E$ (s)	$C_E$ (M)	$T_E$ ( $^{\circ}\text{C}$ )	Reference
$14.5 \pm 0.1$ Fossil tracks	60	0.4	25	Jonckheere and Van den haute (2002)
$14.3 \pm 0.1$	70	0.4	25	Enkelmann and Jonckheere (2003)
$14.3 \pm 0.1$	20	5.5	21	Enkelmann and Jonckheere (2003)
$13.7 \pm 0.1$ – $14.7 \pm 0.1$	20	4.0	25	Jonckheere et al. (2007)

recommended by Steiger and Jäger (1977):  $31.4 \pm 0.5$  Ma ( $2\sigma$ ; comment (5) to Tables 2 in Hurford, 1990a, b). This value was confirmed and specified by Ar/Ar dating of the same units:  $31.4 \pm 0.2$  Ma ( $2\sigma$ ; McDowell et al., 2005). The other radiometric ages in Table 2 have been determined on the Durango apatite itself, but are less precise or have been obtained with methods susceptible to methodological issues (low age; low U content; high Th/U-ratio; radiation damage effects) that have only begun to be addressed, or both. The recommended reference age is therefore adopted here:  $31.4 \pm 0.5$  Ma ( $2\sigma$ ).

Sixteen sections were cut from four Durango apatite crystals with a Struers Accutom 50 instrument, four from each crystal. Two crystals were cut perpendicular to the  $c$ -axis, giving eight basal sections; the two remaining crystals were cut parallel to the  $c$ -axis, giving eight prism sections. Four basal sections and four prism sections were annealed for 10 h at  $450^{\circ}\text{C}$  to erase the fossil tracks, and mounted in resin. After curing for 24 h at  $45^{\circ}\text{C}$ , the mounts were ground and polished with  $6\text{-}\mu\text{m}$ ,  $3\text{-}\mu\text{m}$  and  $1\text{-}\mu\text{m}$  diamond pastes on a Struers DPU-4 apparatus. The thickness of the mounts was reduced to 2 mm before irradiation. A  $1\text{-cm}^2$  muscovite external detector was attached to each mount. The samples were stacked in an irradiation can, together with Au, Co and Cu monitors for determining the thermal-neutron fluence (Au, Co) and axial and radial fluence gradients (Cu), and irradiated in channel 8 of the Thetis-reactor of the Universiteit Gent. All but one basal and one prism section with induced tracks were again cut out of the resin after the irradiation (Fig. 1).

Each section was paired with one which had adjoined it in the crystal and that had not been annealed or irradiated. Four sets were assembled; each set comprised a pair of basal sections and a pair of prism sections. Each such pair consisted of a section with fossil tracks and one with induced tracks. One set was annealed for 1 h at  $250^{\circ}\text{C}$  in a Naber N3 muffle oven, another set at  $300^{\circ}\text{C}$  and a third at  $350^{\circ}\text{C}$ ; the fourth set

was not annealed. The purpose of these annealing steps was to determine the plateau age of the Durango apatite. Because of a transient  $5^{\circ}\text{C}$  temperature drop following insertion of the samples in the oven and  $5^{\circ}\text{C}$  oscillations during the experiment, the cited temperatures are nominal. This has no consequences for the plateau age because the pairs of sections with fossil and induced tracks were annealed in close contact. The basal and prism sections with induced tracks of the unannealed set had been polished before irradiation and not unmounted or repolished after. The induced tracks were thus registered in external apatite surfaces ( $2\pi$ ) while the fossil tracks in the corresponding unirradiated sections were registered in internal surfaces. This made it possible to date the Durango apatite with a variant of the population method, not used for dating applications (method B). It has methodological interest, however, because, in contrast to the familiar population method (method A), it involves a geometry factor of  $\frac{1}{2}$  and  $\eta q$ -factor for induced-track counts in an external apatite surface,  $[\eta q]_{ES}$ , resulting in a  $Q$ -factor like that for the re-etch method (method D).

The fourteen unmounted apatite sections were thereupon mounted in resin, ground and polished as before. All apatite sections were etched for 60 s in  $2.5\%$   $\text{HNO}_3$  at  $25.0 \pm 0.1^{\circ}\text{C}$  in a forced-circulation thermostatic bath. The etchant temperature was recorded with a Julabo TD-300 digital thermometer with a Pt-100 sensor. After etching and rinsing in p.a. ethanol, the eight sections with fossil tracks were covered with  $1\text{-cm}^2$  muscovite external detectors, stacked in an irradiation can with Au, Co and Cu monitors for measuring the thermal-neutron fluence (Au, Co) and axial and radial fluence gradients (Cu), and irradiated in channel 8 of the Thetis reactor. After irradiation, the external detectors were detached and etched, together with those from the first irradiation, for 20 min in  $40\%$  HF at  $25.0 \pm 0.1^{\circ}\text{C}$ .

At this stage, there were sixteen etched apatite sections and their corresponding external detectors (Fig. 1). Four apatite sections, the basal and

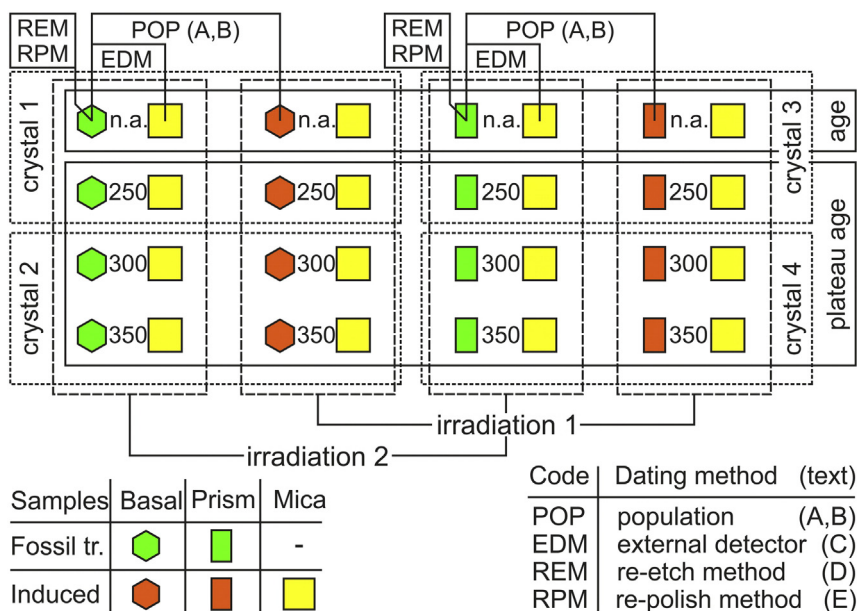


Fig. 1. Schematic organization of the experiment, showing the 8 basal and 8 prism sections of Durango apatite and their external detectors, the two irradiations, and the samples used for dating with different methods and for the determination of the plateau age. See text for details.

prism sections with fossil tracks and those with induced tracks that had not been annealed after the first irradiation were used for the age determinations. The remaining twelve sections that had been partially annealed, i.e. a pair of basal sections and a pair of prism sections annealed for 1 h at 250 °C and the corresponding pairs annealed at 300 °C and 350 °C were used for determining the plateau age. The external-detector counts were used for calculating the uranium content of all sections.

The weights of the co-irradiated metal-activation monitors were measured on electronic and mechanical scales to a precision of 0.01%; the maximum deviation between the instruments was <0.1%. The  $\gamma$ -activities were measured on a single-open-ended Ge(Li) detector linked to a Canberra-S40 4000-channel analyser. The wireform monitors were wound into tight spirals with approximate point source geometries and measured at the reference distance of 15 cm, for which a peak-detection-efficiency calibration curve ( $\log \varepsilon_p$  vs.  $\log E_\gamma$ ) had been established by reactor staff with the aid of calibrated point sources. The measurements were continued until >50,000 counts had been collected under each peak. Accidental and true-coincidence losses are negligible for the source-detector distance and low count rates of the measurements (Moens, 1981). The peak areas were calculated assuming a sigmoidal background for the 411.8 keV  $^{198}\text{Au}$   $\gamma$ -peak and the 1173.2 keV and 1332.5 keV  $^{60}\text{Co}$   $\gamma$ -peaks (Sequal program; Op de Beeck, 1976) and a trapezoidal background for the 511.0 keV  $^{64}\text{Cu}$  annihilation peak (Trap program). The fluence calculations were carried out in the Høgdahl convention using the formulae and nuclear constants in Van den haute et al. (1988) and De Corte et al. (1991). The unweighted mean of the fluences calculated from the 1173.2 keV and 1332.5 keV  $^{60}\text{Co}$   $\gamma$ -peaks was taken as the final Co-fluence, and averaged with the value calculated from the 411.8 keV  $^{198}\text{Au}$   $\gamma$ -peak to obtain the final estimate of the sub-cadmium neutron fluence in the Høgdahl formalism ( $\phi_s$ ). Fluence gradients were calculated from the 511.0 keV activities of the Cu monitors (Van den haute and Chambaudet, 1990) and assumed linear over the dimensions of the sample stack. The fluence for each sample was calculated by interpolation.

The track counts in apatite were carried out in transmitted light with an Olympus BH-2 microscope, equipped with a dry no-cover 100 $\times$  objective and 10 $\times$  eye pieces. The nominal magnification, including a drawing tube factor (1.25 $\times$ ), was 1250 $\times$ . The size of the 10 $\times$  10 counting grid was calibrated with a Graticules (50 $\times$  2)- $\mu\text{m}$  stage micrometre and amounted to 78.5  $\mu\text{m}$   $\times$  78.5  $\mu\text{m}$  (6.16  $10^{-5}$   $\text{cm}^2$ ); the actual overall magnification was 1274 $\times$ . The track counts in the external detectors were carried out with cover glass and a 100 $\times$  oil-immersion objective and 10 $\times$  eye pieces. The size of the counting grid was 82.9  $\mu\text{m}$   $\times$  82.9  $\mu\text{m}$  (6.87  $10^{-5}$   $\text{cm}^2$ ) and the actual magnification 1206 $\times$ . The cover glass served to protect the mica, which is more easily scratched and more difficult to clean than the apatite mounts.

These and the following experimental steps had four goals: (1) ascertaining the uranium concentration of the apatite sections; (2) dating basal and prism sections of the Durango apatite with the population method (A), a variant of the population method (B), the external-detector method (C), the re-etch method (D) and the repolish method (E) (Gleadow, 1981); each method requires specific corrections for experimental factors (Section 2); (3) determining the plateau age of the Durango apatite, and (4) establishing the need, extent and appropriate method of age correction for partial annealing of the fossil tracks. The sequence of experimental steps makes it more practical to discuss population method (B) before (A).

**Uranium concentration.** The uranium concentration of the apatite sections was calculated from the induced-track densities in the external detectors covering the mounts during the first and second irradiations, and the corresponding thermal-neutron fluences. The equation (Table 1; Eq. 6) is that of Jonckheere (1995), Enkelmann et al. (2005a) and Hoffmann et al. (2008). Enkelmann et al. (2005a) reported partial results. Table 3 reports full details of the track counts. The concentrations were calculated with the more recent value of  $g(T_n)$  (0.9832; Holden, 1999) instead of the value used before (0.977; Wagemans et al., 1988; Section 2).

**Population method (B).** In this case, the induced tracks were etched in an external apatite surface. This is the result of polishing the induced-track mounts before their irradiation. This method differs from the familiar population method (A; Gleadow, 1981) in that each population consists of an apatite section, within which random fields were counted. It nevertheless has the essential feature of a population method, i.e. counting of the fossil and induced tracks in separate apatite aliquots (sections), one of which was annealed before neutron irradiation. In contrast to the familiar population method (A), it requires a correction factor ( $Q = [\eta q]_i / [\eta q]_s$ ) for the different etching and counting efficiencies of the fossil and induced tracks. The relevant  $\eta q$ -values are from Jonckheere and Van den haute (2002) and Jonckheere (2003b) and summarized in Table 1. The age calculation also requires a correction factor ( $U$ ) for the difference between the uranium concentrations of the apatite sections with fossil and induced tracks.  $U$  was calculated from the uranium concentrations in Table 3. The age calculation is summarized in Tables 4 and 5.

**Population method (A).** The fossil-track counts for this dating method are the same as for the above method (B). The sections with the induced tracks were repolished and re-etched under the same conditions as before. This obviates the need for the factor  $Q$  as the fossil and induced tracks are now both counted in an internal apatite surface. Population method (A) is equivalent to the familiar method from a methodological perspective. In spite removing 50  $\mu\text{m}$  from the surface of the sections with induced tracks, it is assumed that the same uranium correction applies as for method (B). The results are listed in Tables 4 and 5.

**External-detector method (C).** The fossil-track counts are the same as before. The induced tracks were counted in external detectors covering the sections with fossil tracks during the second irradiation. This method differs from the familiar external-detector method in that no effort is made to count the fossil and induced tracks in matching areas. From the perspective of the methodological factors (Section 2), it is nevertheless equivalent. The age calculation involves  $G$ ,  $Q = [\eta q]_i / [\eta q]_s$ , and  $R$ . In this case,  $[\eta q]_i$  refers to the track counts in the mica external detectors, in contrast to the population method (B) and the re-etch method (D), where it refers to track counts in the external apatite surface. The relevant correction factors (Jonckheere, 2003b) and external-detector ages are summarized in Tables 4 and 5.

**Re-etch method (D).** The fossil-track counts are the same as before. The basal and prism section with fossil tracks were re-etched for 60 s in 2.5%  $\text{HNO}_3$  at 25 °C, revealing the induced tracks of the second irradiation and increasing the total etch time of the fossil tracks to 120 s. The induced-track densities are given by:  $\rho_i = \rho_{i+s} - T \rho_s$  ( $T$ : etch-time factor). Because the induced tracks were etched in an external surface,  $G$  and  $Q$  are needed (Tables 4 and 5).

**Repolish method (E).** The fossil-track counts are the same as before. The basal and prism sections were repolished and re-etched (60 s in 2.5%  $\text{HNO}_3$  at 25 °C), revealing the combined fossil and induced tracks in internal surfaces. The induced-track densities were calculated by subtraction:  $\rho_i = \rho_{i+s} - \rho_s$ . The age calculations do not require  $G$ ,  $T$ ,  $Q$  or  $R$  (Tables 4 and 5).

**Plateau age.** The age of the unannealed reference sample is that obtained before with the population method (A). The data for the age plateau were obtained on the pairs of basal and prism sections with fossil and induced tracks annealed together between the two irradiations (Fig. 1). The age calculation is as for population method (A), including a correction ( $U$ ) for the uranium concentrations of sections with fossil and induced tracks (Table 6).

## 4. Results and discussion

### 4.1. Uranium concentration

The uranium concentrations calculated with Eq. (6) in Table 1 are listed in Table 3. The differences with an earlier equation

**Table 3**  
Calculation of the uranium concentrations of the Durango apatite samples;  $N$ : number of counted tracks;  $n$ : number of counted fields (1 field =  $6.87 \cdot 10^5 \text{ cm}^2$ );  $\rho_{\text{ED}}$  ( $10^5 \text{ cm}^{-2}$ ): induced-track densities in the mica external detectors;  $\sigma/\sigma_p$ : ratio of the standard deviation of the track density distribution to that of a Poisson distribution;  $\phi$  ( $10^{15} \text{ cm}^{-2}$ ): thermal-neutron fluence;  $\rho_{\text{ED}}/\phi$  (tracks/ $10^{10}$  neutrons);  $[U]_{\text{FT}}$  (ppm): uranium concentration calculated from  $\rho_{\text{ED}}/\phi$  using Eq. (6);  $[U]_{\text{NA}}$  (ppm): uranium concentrations determined by epithermal-neutron-activation analysis. Errors are  $1\sigma$ .

Mica ED	Section	$N$	$n$	$\rho_{\text{ED}}$	$\sigma/\sigma_p$	$\phi_s$	$\rho_{\text{ED}}/\phi_s$	$[U]_{\text{FT}}$	$[U]_{\text{NA}}$
MSB 025	Basal	2929	255	$1.67 \pm 0.03$	1.01	$1.22 \pm 0.02$	$1.37 \pm 0.04$	$9.22 \pm 0.45$	$9.17 \pm 0.07$
MIB 025	Basal	2990	229	$1.90 \pm 0.03$	0.97	$1.32 \pm 0.03$	$1.44 \pm 0.04$	$9.68 \pm 0.47$	$9.35 \pm 0.15$
MSB 250	Basal	3309	258	$1.87 \pm 0.04$	1.12	$1.21 \pm 0.02$	$1.54 \pm 0.05$	$10.38 \pm 0.50$	
MIB 250	Basal	3128	234	$1.95 \pm 0.04$	1.07	$1.31 \pm 0.03$	$1.48 \pm 0.04$	$9.99 \pm 0.48$	
MSB 300	Basal	3464	212	$2.38 \pm 0.04$	0.96	$1.20 \pm 0.02$	$1.98 \pm 0.05$	$13.33 \pm 0.64$	
MIB 300	Basal	4175	230	$2.64 \pm 0.04$	1.03	$1.30 \pm 0.03$	$2.03 \pm 0.05$	$13.67 \pm 0.65$	
MSB 350	Basal	3543	216	$2.39 \pm 0.04$	1.06	$1.18 \pm 0.02$	$2.02 \pm 0.05$	$13.61 \pm 0.65$	
MIB 350	Basal	4062	232	$2.55 \pm 0.04$	1.09	$1.30 \pm 0.03$	$1.96 \pm 0.05$	$13.19 \pm 0.63$	
MSP 025	Prism	3860	254	$2.21 \pm 0.04$	1.11	$1.22 \pm 0.02$	$1.81 \pm 0.05$	$12.20 \pm 0.59$	$12.18 \pm 0.18$
MIP 025	Prism	3860	237	$2.37 \pm 0.04$	1.01	$1.31 \pm 0.03$	$1.81 \pm 0.05$	$12.17 \pm 0.58$	$12.05 \pm 0.31$
MSP 250	Prism	3297	211	$2.27 \pm 0.04$	1.09	$1.20 \pm 0.02$	$1.89 \pm 0.05$	$12.75 \pm 0.62$	
MIP 250	Prism	3512	212	$2.41 \pm 0.04$	0.96	$1.31 \pm 0.03$	$1.84 \pm 0.05$	$12.38 \pm 0.59$	
MSP 300	Prism	2536	207	$1.78 \pm 0.04$	1.09	$1.19 \pm 0.02$	$1.50 \pm 0.04$	$10.08 \pm 0.50$	
MIP 300	Prism	2900	223	$1.89 \pm 0.04$	1.05	$1.30 \pm 0.03$	$1.46 \pm 0.04$	$9.79 \pm 0.48$	
MSP 350	Prism	2526	222	$1.66 \pm 0.04$	1.09	$1.18 \pm 0.02$	$1.40 \pm 0.04$	$9.44 \pm 0.47$	
MIP 350	Prism	3091	227	$1.98 \pm 0.04$	0.96	$1.29 \pm 0.03$	$1.54 \pm 0.04$	$10.33 \pm 0.50$	

(Enkelmann et al., 2005a; Hoffmann et al., 2008; Jonckheere, 1995) are the new Westcott  $g$ -factor (0.9832; Holden, 1999) and the correction ( $\beta = 1.0025$ ; Jonckheere, 1995) for the difference between the Westcott and Høgdahl formalisms. Their combined effect is  $<1\%$ . Table 3 also reports the uranium concentrations of four sections determined with epithermal-neutron-activation analysis (ENAA; Bellemans, Institute for Nuclear Sciences, Universiteit Gent). These are on average 1.3% lower, half of which is attributable to one sample. The good agreement supports the value of the  $\eta q$ -factor in Eq. (6) ( $[\eta q]_{\text{ED}} = 0.91 \pm 0.01$ ; Table 1), which implies that the efficiency of induced-track counts in muscovite external detectors is far lower than 100%. This indicates that  $\eta q$  is not a function of the bulk-etch rate but of the fraction of shallow tracks ( $z < 0.5 \mu\text{m}$ ; Jonckheere and Van den haute, 2002) approaching the optical contrast limit of the microscope. These results further endorse that the induced-track density in an external detector is not proportional to the etchable track length in the irradiated mineral but to that in the detector, corrected for the length deficit in both (Jonckheere, 2003b). The uranium results can also be interpreted as evidence for accurate neutron-fluence determinations with the aid of dilute metal-activation monitors (Au, Co). It is important that this evidence is not affected by the interdependence of  $\phi$  and  $\lambda_F$  ( $\phi/\lambda_F$ -problem), which has long been considered an unsurmountable obstacle to standardless dating, and heralded the  $\zeta$ -calibration (Hurford, 1990a, b, 1998; Hurford and Green, 1981, 1983).

**Table 4**  
Conventional calculation of the fission-track age of the Durango apatite. A: population method with induced-track counts in an internal apatite section; B: population method with induced-track counts in an external apatite surface; C: external-detector method; D: re-etch method; E: repolish method;  $[RG]_s$ ,  $[RG]_i$ : registration geometries of the fossil tracks (S) and induced (I) tracks (IS: internal section; ES: external surface; ED: external detector);  $N_s$ ,  $N_i$ : fossil (S) and induced (I) tracks counts;  $n_s$ ,  $n_i$ : number of fields counted for the fossil (S) and induced (I) tracks (IS and ES: 1 field =  $6.16 \cdot 10^{-5} \text{ cm}^2$ ; ED: 1 field =  $6.87 \cdot 10^{-5} \text{ cm}^2$ );  $\rho_s$ ,  $\rho_i$  ( $10^5 \text{ cm}^{-2}$ ): surface-track densities of the fossil (S) and induced (I) tracks (\*:  $\rho_s + \rho_i$ );  $\phi_s$  ( $10^{15} \text{ cm}^{-2}$ ): Høgdahl thermal-neutron fluence;  $[t_{\text{FT}}]_c$  (Ma): conventional fission-track age. Errors are  $1\sigma$ .

Method	Section	$[RG]_s$	$N_s$	$n_s$	$\rho_s$	$[RG]_i$	$N_i$	$n_i$	$\rho_i$	$\phi_s$	$[t_{\text{FT}}]_c$
A	Basal	IS	2205	246	$1.45 \pm 0.03$	IS	4265	219	$3.16 \pm 0.05$	$1.32 \pm 0.03$	$29.6 \pm 1.0$
A	Prism	IS	3172	267	$1.93 \pm 0.04$	IS	4610	190	$3.94 \pm 0.06$	$1.31 \pm 0.03$	$31.3 \pm 1.0$
B	Basal	IS	2205	246	$1.45 \pm 0.03$	ES	2500	225	$1.80 \pm 0.04$	$1.32 \pm 0.03$	$26.0 \pm 1.0$
B	Prism	IS	3172	267	$1.93 \pm 0.04$	ES	2907	219	$2.15 \pm 0.04$	$1.31 \pm 0.03$	$28.6 \pm 1.0$
C	Basal	IS	2205	246	$1.45 \pm 0.03$	ED	2929	255	$1.67 \pm 0.03$	$1.22 \pm 0.02$	$25.9 \pm 0.9$
C	Prism	IS	3172	267	$1.93 \pm 0.04$	ED	3860	254	$2.21 \pm 0.04$	$1.22 \pm 0.02$	$25.9 \pm 0.9$
D	Basal	IS	2205	246	$1.45 \pm 0.03$	ES	2226	123	$*2.94 \pm 0.07$	$1.22 \pm 0.02$	$29.2 \pm 1.1$
D	Prism	IS	3172	267	$1.93 \pm 0.04$	ES	3264	116	$*4.57 \pm 0.08$	$1.22 \pm 0.02$	$21.8 \pm 0.7$
E	Basal	IS	2205	246	$1.45 \pm 0.03$	IS	5293	204	$*4.21 \pm 0.06$	$1.22 \pm 0.02$	$31.4 \pm 1.1$
E	Prism	IS	3172	267	$1.93 \pm 0.04$	IS	3837	105	$*5.93 \pm 0.10$	$1.22 \pm 0.02$	$28.7 \pm 1.0$

The uranium concentrations in Table 3 give an indication of the uranium distribution at different scales. The ratios of the standard deviations of the track-density distributions to those of the corresponding Poisson distributions ( $\sigma/\sigma_p$ ) are on average somewhat higher than 1 (mean: 1.04), indicating the absence of substantial uranium inhomogeneities at the scale of the counting grid ( $>0.01 \text{ mm}^2$ ). The ratio of the standard deviations of the values for the four sections from each crystal to their average statistical uncertainties is of the same order (mean: 1.11), allowing a similar conclusion concerning uranium inhomogeneities between sections at the scale of the areas counted within each ( $>1 \text{ mm}^2$ ). In contrast, the differences between crystals (up to 30%) are much larger than the variation within each (standard-deviation ratio  $>10$ ), as noted by Naeser and Fleischer (1975).

#### 4.2. Apparent fission-track age

Table 1 lists the equations and the physical and empirical constants used for calculating the fission-track ages without (Eq. 7; population and external-detector methods) and with subtraction (Eq. 8; re-etch and repolish methods). No annealing correction is involved at this stage; the results are thus apparent ages, not corrected for shortening of the fossil tracks ( $L = 1$ ). Table 4 summarizes the track counts and neutron fluences and lists the results of a conventional age calculation, i.e. leaving out the method-specific corrections for experimental factors

discussed in Section 2. In practice this means that  $G = \frac{1}{2}$  or 1, depending on the method,  $Q = 1$ ,  $R = 1$ , and  $T = 1$ . Although  $U$  is a sample-specific factor and not a fundamental methodological one, it was also assumed that  $U = 1$  for simplicity. The conventional ages exhibit a much greater spread ( $21.8 \pm 0.7$  to  $31.4 \pm 1.1$  Ma; Table 4; Fig. 2) than their errors permit. Their mean ( $27.8 \pm 0.9$  Ma) is  $>10\%$  lower than the reference age ( $31.4 \pm 0.5$  Ma; Green, 1985; Hurford, 1990a, b). It is striking that the common population (A; mean  $30.5 \pm 0.8$  Ma) and repolish methods (E; mean  $30.0 \pm 1.4$  Ma), neither of which requires methodological corrections, give ages within  $<2\sigma$  of the reference age.

Table 5 lists the apparent fission-track ages based on the same track counts (Table 4), but calculated with the correction factors discussed in Section 2, together with their values calculated from the data in Tables 1 and 3.  $G$  depends on the method used ( $\frac{1}{2}$  or 1).  $R$  is constant for apatite dated using muscovite external detectors, but takes a different value for other minerals or other external detectors.  $Q$  and  $T$ , in contrast, are specific to the etching and observation conditions and the track identification criteria of the microscope operator.  $Q$  and  $T$  are also expected to be different for other apatite sections than basal and prism faces.  $U$  is specific to the samples and experimental procedure in this work. In contrast to the conventional ages, the corrected apparent ages determined with the different dating methods are consistent with each other and the reference age (Table 5). The results range from  $28.7 \pm 1.0$  Ma to  $32.0 \pm 1.7$  Ma with a mean of  $30.9 \pm 0.3$  Ma,  $<2\%$  lower than the reference age. Their standard deviation (0.9 Ma) is less than the calculated uncertainties of the individual ages. This is expected because the fossil-track counts are the same for all dating methods and some correction factors are common to two or more age calculations. The same applies to the neutron fluences because a single fluence, albeit corrected for minor fluence gradients, was determined for each of the two sample irradiations.

Earlier studies (Bellemans, 1996; Bigazzi et al., 1990; Curvo et al., 2013; De Corte et al., 1991, 1995; Jonckheere, 1995; Van den haute et al., 1988, 1998) give reason for trusting that the calculated neutron fluences are accurate. The agreement of the uranium concentrations calculated with Eq. (6) with independent data (Enkelmann et al., 2005a; Hoffmann et al., 2008; Section 4.1) supports this assumption. It is reasonable to conclude from re-evaluations of the  $^{238}\text{U}$ -fission constant (Holden, 1989; Holden and Hoffman, 2000) that the value used is also accurate. The fact that the correction factors introduced in Section 2 remove the inconsistencies between the ages obtained with different dating methods is a convincing argument for concluding that these are needed and accurate within their uncertainties. It is reasonable to conclude that the agreement of the mean apparent age ( $30.9 \pm 0.6$  Ma;  $2\sigma$ ) with the reference age ( $31.4 \pm 0.5$  Ma;  $2\sigma$ ) is not fortuitous. However, this runs counter to the current consensus, implemented in all modelling algorithms, which requires that a shortening of the fossil-

track length of 10% or more, relative to the length of the induced tracks, implies a proportional lowering of the apparent fission-track age (Crowley, 1993; Green, 1988; Guedes et al., 2004; Jones and Dokka, 1990; Ketcham, 2005; Laslett et al., 1984; Lutz and Omar, 1991; Tello et al., 2006; Watt and Durrani, 1985; Willett, 1997).

### 4.3. Length-corrected age

The mean lengths of fossil and induced confined tracks etched in prism faces of the Durango apatite are  $l_s = 14.5 \pm 0.1 \mu\text{m}$  and  $l_i = 16.3 \pm 0.1 \mu\text{m}$  (Jonckheere and Van den haute, 2002), in good agreement with most published data, including for different etching conditions (Table 2). In this case,  $L = l_i/l_s (= 1.12 \pm 0.01)$  is a close lower estimate of the correction factor entered in Eqs. (7) and (8) (Table 1) for calculating the length-corrected ages. For approximate isothermal holding, this estimate is almost independent of the annealing model and the length-bias model ( $\rho/\rho_0$  vs.  $l/l_0$ ) (Wauschkuhn et al., 2015a). The length-corrected ages, including the corrections for methodological factors, are listed in Table 5. The ages obtained with different dating methods are still consistent with each other, but none is in agreement with the reference age of the Durango apatite. The mean length-corrected age ( $34.7 \pm 0.3$  Ma) overestimates the reference age by almost 10 standard errors, whereas the mean uncorrected age underestimates it by less than two.

It appears that the apparent fission-track age of the Durango apatite requires no, or at most a small, correction. This is also the case for the Fish Canyon tuff standard (Enkelmann et al., 2005b). Green (1988) made the same observation for apatites with “volcanic type” length distributions, rapidly cooled apatites, most but not all of volcanic origin, which were not reheated. The means (14.0–15.7  $\mu\text{m}$ ) and standard deviations (0.8–1.3  $\mu\text{m}$ ) of their fossil-track-length distributions indicate moderate ambient-temperature annealing (Gleadow et al., 1986). Green (1988) ascribed the fact that their apparent fission-track ages permitted no length correction to possible dating errors related to the efficiencies of the fossil and induced-track counts rather than to the effects of the neutron fluence or fission constant. This is not an adequate explanation of our results, considering the explicit corrections for these effects and their, at least partial, success at bringing the results of different dating methods and those for basal and prism sections in line with each other and with the reference age.

Gleadow and Duddy (1981) and Gleadow et al. (1983) proposed an explanation based on their investigation of borehole samples from the Australian Otway Basin. Their “ageing” concept holds that fossil tracks in apatite undergo length reductions up to 15–20% under geological annealing at  $<70^\circ\text{C}$  without attendant lowering of its apparent fission-track age. Although the concept was abandoned, ageing could account for our dating results. Ageing lost favour because Green

**Table 5**

Calculation of the fission-track age of the Durango apatite, corrected for experimental factors. A: population method with induced-track counts in an internal section; B: population method with induced-track counts in an external surface; C: external-detector method; D: re-etch method; E: repolish method;  $[t_{\text{FT}}]_{\text{C}}$  (Ma): conventional fission-track age (Table 3);  $[t_{\text{FT}}]_{\text{CL}}$  (Ma): length-corrected conventional age;  $Q = [\eta q]_{\text{I}}/[\eta q]_{\text{S}}$ : correction factor for the different counting efficiencies of the fossil (S) and induced (I) tracks (calculated from  $\eta q$ -values in Jonckheere and Van den haute, 2002; Table 1);  $R$ : correction factor for the different length deficit of induced tracks in apatite and muscovite (Jonckheere, 2003b);  $T$ : correction factor for the different etch times of the fossil and induced tracks (Jonckheere and Van den haute, 1996);  $U$ : correction factor for the different uranium contents of the apatite sections with fossil and induced tracks (Table 3);  $L$ : correction factor for the different etchable lengths of fossil and induced tracks (confined-track lengths in Jonckheere and Van den haute, 2002);  $[t_{\text{FT}}]_{\text{A}}$  (Ma): apparent fission-track age;  $[t_{\text{FT}}]_{\text{AL}}$  (Ma): length-corrected age. Errors are  $1\sigma$ .

Meth.	Sect.	$[t_{\text{FT}}]_{\text{C}}$	$[t_{\text{FT}}]_{\text{CL}}$	$G$	$Q$	$R$	$T$	$U$	$L$	$[t_{\text{FT}}]_{\text{A}}$	$[t_{\text{FT}}]_{\text{AL}}$
A	Basal	$29.6 \pm 1.0$	$33.3 \pm 1.2$	1	1	1	1	$1.05 \pm 0.04$	$1.12 \pm 0.01$	$31.1 \pm 1.6$	$35.0 \pm 1.8$
A	Prism	$31.3 \pm 1.0$	$35.2 \pm 1.2$	1	1	1	1	$0.99 \pm 0.04$	$1.12 \pm 0.01$	$31.2 \pm 1.5$	$35.1 \pm 1.7$
B	Basal	$26.0 \pm 1.0$	$29.2 \pm 1.1$	$\frac{1}{2}$	$1.12 \pm 0.02$	1	1	$1.05 \pm 0.04$	$1.12 \pm 0.01$	$30.6 \pm 1.7$	$34.4 \pm 1.9$
B	Prism	$28.6 \pm 1.0$	$32.1 \pm 1.1$	$\frac{1}{2}$	$1.12 \pm 0.02$	1	1	$0.99 \pm 0.04$	$1.12 \pm 0.01$	$32.0 \pm 1.7$	$35.9 \pm 1.9$
C	Basal	$25.9 \pm 0.9$	$29.1 \pm 1.1$	$\frac{1}{2}$	$1.00 \pm 0.02$	$1.21 \pm 0.02$	–	1	$1.12 \pm 0.01$	$31.3 \pm 1.5$	$35.2 \pm 1.7$
C	Prism	$25.9 \pm 0.9$	$29.2 \pm 1.0$	$\frac{1}{2}$	$1.00 \pm 0.02$	$1.21 \pm 0.02$	–	1	$1.12 \pm 0.01$	$31.4 \pm 1.4$	$35.3 \pm 1.6$
D	Basal	$29.2 \pm 1.1$	$32.8 \pm 1.2$	$\frac{1}{2}$	$1.12 \pm 0.02$	1	$0.94 \pm 0.03$	1	$1.12 \pm 0.01$	$30.9 \pm 1.6$	$34.7 \pm 1.8$
D	Prism	$21.8 \pm 0.7$	$24.4 \pm 0.8$	$\frac{1}{2}$	$1.12 \pm 0.02$	1	$1.26 \pm 0.04$	1	$1.12 \pm 0.01$	$30.1 \pm 1.5$	$33.8 \pm 1.7$
E	Basal	$31.4 \pm 1.1$	$35.3 \pm 1.2$	1	1	1	1	1	$1.12 \pm 0.01$	$31.4 \pm 1.1$	$35.2 \pm 1.2$
E	Prism	$28.7 \pm 1.0$	$32.2 \pm 1.1$	1	1	1	1	1	$1.12 \pm 0.01$	$28.7 \pm 1.0$	$32.2 \pm 1.1$
Mean:		$27.8 \pm 0.9$	$31.3 \pm 1.1$							$30.9 \pm 0.3$	$34.7 \pm 0.3$



(1988) argued that it was an artefact. The ages of the borehole samples ( $t$ ) had indeed been normalized to that of a surface sample ( $t_0$ ), that exhibited a length reduction ( $l_{50} < l_i$ ) relative to induced tracks ( $l_i$ ). After renormalizing ( $[t/t_0] \rightarrow [l_{50}/l_i] \times [t/t_0]$ ), the Otway data were found to be in agreement with the ( $\rho/\rho_0$  vs.  $l/l_0$ )-trend of induced tracks. An objection to Green's (1988) renormalization is that it assumed what it proved. Renormalization is acceptable if ageing is indeed an artefact. However, if it is not, then the renormalization itself is invalid, and the data remain unchanged, corroborating that ageing is real. A second objection is that the observation that the renormalized ( $\rho/\rho_0$  vs.  $l/l_0$ )-trend for fossil tracks coincides with that for induced tracks does not permit to conclude that the same annealing mechanism operates in both cases. The two trends cannot be assumed to coincide for the same reason that the trend for multicompositional apatites lies above that for monocompositional apatites (Green, 1988). A third argument against renormalization is that the renormalized apatite ages of outcrop samples are ca. 10% older than the titanite fission-track ages of the same volcanoclastic rocks. Barring unknown systematic errors of the right magnitude affecting one but not the other (excluding  $\phi$  and  $\lambda_F$ ), this is as strong an argument for ageing as the former, establishing that Green's (1988) case is inconclusive, but not that it is invalid.

#### 4.4. Plateau-age experiment

Our length measurements confirm the shortening of the fossil tracks in the Durango apatite (Table 2) but theory is inconclusive as to whether or not an age correction is required. An attempt was therefore made to determine the corrected age of the Durango apatite with the plateau method of Storzer and Poupeau (1973). This method is almost never used but can be shown to be valid within the current paradigm of the fission-track method. Numerical calculations based on the Markov assumption (equivalent time; Duddy et al., 1988) and an annealing model (e.g. Laslett et al., 1987) indicate that, if the age correction implied by the confined-track lengths is needed, then an age plateau exists at 35.3 Ma at temperatures upward of ca. 250 °C (for 1 h annealing; Jonckheere, 2003a). Moreover, the plateau method does not depend on a specific, or known, ( $\rho/\rho_0$  vs.  $l/l_0$ )-relationship or on the assumption that the same relationship holds for geological annealing of fossil fission tracks and lab annealing of induced tracks. Instead, it rests on the lesser condition that, when the fossil and induced tracks have been annealed together to the same length in the lab, the corresponding surface-track densities have also been reduced to the same fraction.

The plateau ages were calculated with Eq. (7) (Table 1), with  $G = 1$ ,  $Q = 1$ ,  $R = 1$ , and  $U$  based on the uranium concentrations in Table 3, and are listed in Table 6 and plotted in Fig. 3. There is a clear distinction between the ages of the samples annealed at 350 °C and at those annealed

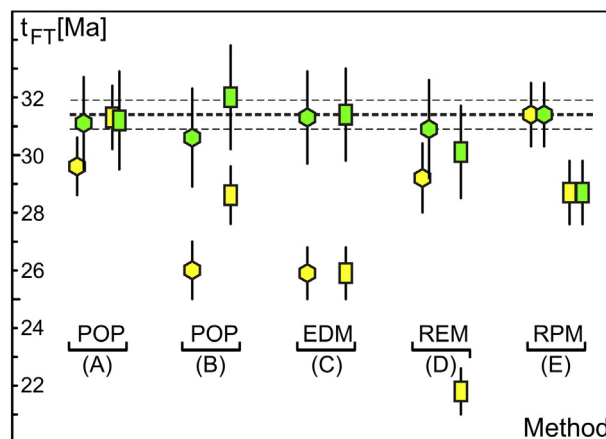


Fig. 2. Dating results obtained on basal and prism sections of the Durango apatite with the population (A, B; see text), external detector (C), re-etch (D) and repolish methods (E). Hexagons: basal sections; rectangles: prism sections; yellow: conventional age calculation ( $[t_{FT}]_A$ : Table 4); green: ages corrected for experimental factors but not for partial annealing of the fossil tracks ( $[t_{FT}]_A$ : Table 5); error bars are  $1\sigma$ . Dashed lines: reference age and  $2\sigma$  confidence interval. A conventional age calculation gives inconsistent results, of which only a fraction is in agreement with the reference age. Corrections for method-related experimental factors bring the fission-track ages in line with each other and with the reference age.

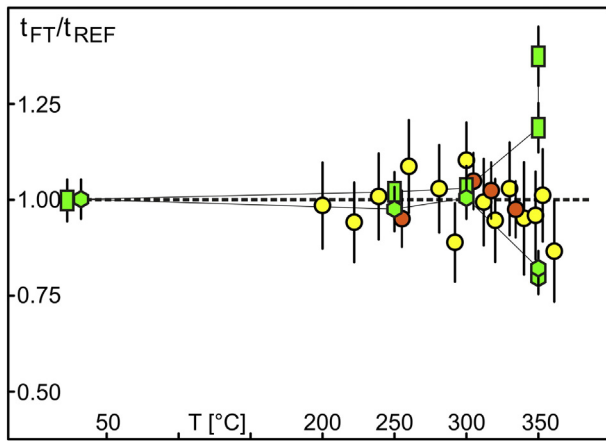
250 and 300 °C. This is also so for the data of Naeser and Fleischer (1975) and for the annealing data of Green (1988) for the Renfrew and Otway apatites, although not at exactly 350 °C. Annealing for 1 h at 350 °C brings the tracks to the point at which unetchable gaps appear (Green et al., 1986) or where tracks at high angles to the  $c$ -axis undergo accelerated length reduction (Donelick, 1991; Donelick et al., 1999). The distinction is not crucial because the relationship between the volumetric and surface-track densities breaks down in either case. Projected-length measurements of the fossil and induced tracks in the samples annealed at 350 °C revealed an excess of short tracks that is reduced but not eliminated by an additional etch (25 s; 2.5% HNO<sub>3</sub>; 25 °C; Jonckheere, 1995). This has unpredictable effects on the surface-track densities and results in erratic ages.

The mean age of the samples annealed at 250 and 300 °C (plateau age;  $31.4 \pm 0.7$  Ma) is indistinguishable from that of the unannealed samples (Table 6;  $31.2 \pm 0.7$  Ma), the mean of all dating methods (Table 5;  $30.9 \pm 0.3$  Ma) and from the reference age ( $31.4 \pm 0.5$  Ma;  $2\sigma$ ). This suggests that no, or at most a small (<2%), age correction is required and excludes the much larger correction (>12%) required by the measured mean confined-track lengths.

Table 6

Calculation of the fission-track age plateau of the Durango apatite.  $T$  (°C): 1 h annealing temperature (\*: samples etched additional 25 s);  $N_S$ ,  $N_I$ : fossil (S) and induced (I) tracks counts;  $n_S$ ,  $n_I$ : number of fields counted for the fossil (S) and induced (I) tracks (1 field =  $6.16 \cdot 10^{-5}$  cm<sup>2</sup>);  $\rho_S$ ,  $\rho_I$  ( $10^5$  cm<sup>-2</sup>): surface-track densities of the fossil (S) and induced (I) tracks;  $U$ : correction factor for the different uranium concentrations of the apatite sections with fossil and induced tracks;  $\phi_S$  ( $10^{15}$  cm<sup>-2</sup>): Høgdahl thermal-neutron fluence;  $[t_{FT}]_A$  (Ma): apparent fission-track age. Errors are  $1\sigma$ .

$T$	Section	$N_S$	$n_S$	$\rho_S$	$N_I$	$n_I$	$\rho_I$	$U$	$\phi_S$	$[t_{FT}]_A$
~25	Basal	2205	246	$1.45 \pm 0.03$	4265	219	$3.16 \pm 0.05$	$1.05 \pm 0.04$	$1.32 \pm 0.03$	$31.1 \pm 1.7$
~25	Prism	3172	267	$1.93 \pm 0.04$	4610	190	$3.94 \pm 0.06$	$0.99 \pm 0.04$	$1.31 \pm 0.03$	$31.2 \pm 1.6$
250	Basal	1499	159	$1.52 \pm 0.04$	2020	106	$3.09 \pm 0.07$	$0.96 \pm 0.04$	$1.31 \pm 0.03$	$30.4 \pm 1.8$
250	Prism	3118	262	$1.93 \pm 0.03$	4894	211	$3.76 \pm 0.06$	$0.98 \pm 0.04$	$1.31 \pm 0.03$	$31.8 \pm 1.6$
300	Basal	2225	214	$1.69 \pm 0.04$	2307	107	$3.50 \pm 0.07$	$1.02 \pm 0.04$	$1.30 \pm 0.03$	$31.3 \pm 1.7$
300	Prism	2626	330	$1.29 \pm 0.03$	3479	228	$2.48 \pm 0.04$	$0.97 \pm 0.05$	$1.30 \pm 0.03$	$32.1 \pm 1.8$
									Mean:	$31.4 \pm 0.7$
350	Basal	1615	255	$1.03 \pm 0.03$	2281	150	$2.47 \pm 0.06$	$0.98 \pm 0.03$	$1.30 \pm 0.03$	$25.6 \pm 1.4$
350	Prism	2318	404	$0.93 \pm 0.02$	2884	270	$1.73 \pm 0.03$	$1.08 \pm 0.04$	$1.29 \pm 0.03$	$37.0 \pm 2.0$
350*	Basal	1896	306	$1.01 \pm 0.02$	3210	209	$2.49 \pm 0.05$	$0.98 \pm 0.03$	$1.30 \pm 0.03$	$24.8 \pm 1.3$
350*	Prism	1843	270	$1.11 \pm 0.03$	2216	202	$1.78 \pm 0.04$	$1.08 \pm 0.04$	$1.29 \pm 0.03$	$42.8 \pm 2.4$



**Fig. 3.** Plateau-age results for the Durango apatite normalized to the reference age. Green hexagons: basal sections, green rectangles: prism sections (this work). Orange circles: data of Naeser and Fleischer (1975) for <350 °C; yellow circles: plateau ages calculated from the annealing data of Green (1988). The error bars are 1σ. Excluding the erratic ages at 350 °C, which are due to accelerated length reduction or break-up of the tracks, the plateau ages of the Durango apatite are consistent with its reference age, indicating that no length correction is required.

4.5. Effective track lengths

It appears that there is a contradiction between the mean confined-track lengths (*l*) and the dating results based on surface-track densities ( $\rho$ ). These are not independent of each other but related through the equation of Price and Walker (1963):  $\rho = \frac{1}{2} l N$ , applicable to both fossil and induced tracks etched in an internal apatite section. The volumetric track densities (*N*) can be calculated for the four samples for which we have independent estimates of the uranium concentration (Table 3). Converting the ppm-concentrations to atoms per volume and factoring in the isotopic abundances ( $\theta_{235}$ ;  $\theta_{238} = 1 - \theta_{235}$ ; Table 1) gives the isotope concentrations, *U*<sub>238</sub> and *U*<sub>235</sub>, in Eqs. (2) and (3). These equations then give the volumetric densities of fossil (*N*<sub>S</sub>) and induced (*N*<sub>I</sub>) tracks, if the sample age and neutron fluence are known. From *N*<sub>S</sub> and *N*<sub>I</sub> and the surface-track densities  $\rho_S$  and  $\rho_I$ , we can then calculate the effective etchable lengths of the fossil (*l*<sub>S0</sub>) and induced (*l*<sub>I0</sub>) tracks:  $l_{S0} = 2\rho_S/N_S$  and  $l_{I0} = 2\rho_I/N_I$ . The full equations, with corrections for the Høgdahl neutron fluence ( $\phi_S$ ) and the track registration ( $g_{IS}$ ), etching and counting efficiencies ( $[1\eta q]_{IS}$ ), are given in Table 1 (Eqs. 9 and 10). The calculation assumes that *l*<sub>S0</sub> and *l*<sub>I0</sub> are isotropic. The equations of Ketcham et al. (2007) confirm that this is a good approximation for fossil ( $l_A/l_C = 0.94$ ;  $l_A$  and  $l_C$ : mean track lengths perpendicular and parallel to the apatite *c*-axis) and induced tracks ( $l_A/l_C = 0.99$ ) in the Durango apatite.

Table 7 lists the calculated effective lengths. Their ratio  $L = l_{I0}/l_{S0}$  is not independent of the dating results as it is that which gives the exact

reference age when substituted in the age equation (Section 2). The values of *l*<sub>S0</sub> and *l*<sub>I0</sub> are not fixed by the dating results. The mean effective length of the induced tracks ( $l_{I0} = 16.8 \pm 0.4 \mu\text{m}$ ) agrees within error with most confined-track-length measurements using our and other etching conditions (Table 2). It agrees somewhat better with values obtained after longer etch times or for tracks etched minutes after irradiation (Donelick et al., 1990). The mean effective length of the fossil tracks ( $l_{S0} = 16.3 \pm 0.4 \mu\text{m}$ ) is ca. 0.5  $\mu\text{m}$  less than that of the induced tracks but consistent with it within error. It is however not consistent with values based on confined-track-length measurements, which are on average ca. 2  $\mu\text{m}$  lower (Table 2). This confirms that the shortening of the fossil confined tracks in the Durango apatite and, by extension, in age standards and apatites with volcanic-type length distributions has no, or at most a small, effect on their fossil-track densities, and hence on their fission-track ages. This supports the interpretation of Gleadow and Duddy (1981) and Gleadow et al. (1983) and contradicts the current consensus implemented in the fission-track modelling programs.

4.6. Independent evidence

The standardless fission-track ages of the Durango apatite, determined with different dating procedures, with proper corrections for the associated experimental factors, but without correction for the shortening of the fossil confined tracks, are in near-agreement with its reference age. It is nevertheless difficult to dispel the suspicion that there exist unknown methodological factors causing these ages to be overestimated by the exact amount required by the fossil and induced confined-track lengths (Green, 1988). Independent confirmation is provided by considerations unrelated to the complexities of fission-track dating. It is known that the apatite (U,Th)/He system is, in general, less retentive than the fission-track system. Although the order is reversed below ca. 35 °C, depending on the diffusion and annealing model, the difference is small. Moreover, the track-annealing models refer to the shortening of the confined-track length, which, according to our findings, must not involve an equal lowering of the fission-track age. Therefore, the fact that a sample's (U,Th)/He age has not been lowered relative to that determined with high-temperature thermochronometers is a strong indication that its fission-track age has not been lowered either. Although this is the case for the Durango apatite (Table 2), its exceptional crystal size implies that significant ( $\geq 5\%$ ) He loss requires temperatures in excess of ca. 75 °C (Durango diffusion kinetics; equivalent spherical radius 5 mm; 30 Ma isothermal holding). The argument is therefore inconclusive in the specific case of the Durango apatite.

The case is instead proved by the apatite (U,Th)/He ages of the Fish Canyon Tuff, which are concordant with U/Pb and Ar/Ar ages dating its eruption at ca. 28 Ma (Gleadow et al., 2015, and references therein). At the classic sampling site of this age standard, the (U,Th)/He age ( $20.8 \pm 0.4 \text{ Ma}$ ) is lowered due to protracted cooling in the temperature range 40–60 °C but the LA-ICP-MS fission-track age ( $28.8 \pm 0.8 \text{ Ma}$ ) is

**Table 7**

Calculation of the effective lengths of induced (*l*<sub>I0</sub>;  $\mu\text{m}$ ) and fossil (*l*<sub>S0</sub>;  $\mu\text{m}$ ) tracks in the Durango apatite; *N*<sub>I</sub>: number of induced and fossil tracks; *n*<sub>I</sub>: number of fields counted for the induced and fossil-track counts;  $\rho_{IS}$  ( $10^5 \text{ cm}^{-2}$ ): areal densities of induced and fossil tracks; [U]<sub>ENAA</sub> (ppm): uranium concentration determined with epithermal-neutron-activation analysis;  $\phi_S$  ( $10^{15} \text{ cm}^{-2}$ ): Høgdahl subcadmium neutron fluence calculated from the activities of co-irradiated metal-activation monitors; [*t*]<sub>REF</sub> (Ma): reference age of the Durango apatite. Errors are 1σ.

Induced tracks								
Sample	Section	<i>N</i> <sub>I</sub>	<i>n</i> <sub>I</sub>	$\rho_I$	[U] <sub>ENAA</sub>	$\phi_S$	<i>l</i> <sub>I0</sub>	
DIB 025	Basal	4265	219	$3.16 \pm 0.05$	$9.35 \pm 0.15$	$1.32 \pm 0.03$	$17.0 \pm 0.6$	
DIP 025	Prism	4610	190	$3.94 \pm 0.06$	$12.05 \pm 0.31$	$1.31 \pm 0.03$	$16.5 \pm 0.7$ $16.8 \pm 0.4$	
Fossil tracks								
Sample	Section	<i>N</i> <sub>S</sub>	<i>n</i> <sub>S</sub>	$\rho_S$	[U] <sub>ENAA</sub>	[ <i>t</i> ] <sub>REF</sub>	<i>l</i> <sub>S0</sub>	
DSB 025	Basal	2205	246	$1.45 \pm 0.03$	$9.17 \pm 0.07$	$31.4 \pm 0.3$	$16.3 \pm 0.6$	
DSP 025	Prism	3172	267	$1.93 \pm 0.04$	$12.18 \pm 0.16$	$31.4 \pm 0.3$	$16.3 \pm 0.5$	

not. The latter is consistent with the mean LA-ICP-MS fission-track age ( $28.2 \pm 0.5$  Ma; Gleadow et al., 2015) and standardless external-detector ages ( $27.7 \pm 0.5$  Ma; Enkelmann et al., 2005b;  $27.3 \pm 0.5$  Ma; Iwano, pers. com.). This establishes that the apatite (U,Th)/He method is more temperature sensitive than the fission-track method, in accord with the small grain size of the Fish Canyon Tuff compared to Durango. Therefore, except at the classic sampling site, the fission-track ages cannot be lower than the emplacement age dated with the (U,Th)/He and other thermochronometers. This conflicts with the mean fossil confined-track length ( $14.84 \pm 0.04$   $\mu\text{m}$ ; Gleadow et al., 2015) requiring a correction of ca. 10%, giving a length-corrected age  $> 31$  Ma, which is irreconcilable with the emplacement age. The same reasoning and conclusion apply to the Limberg t3 proposed apatite age standard (mean sanidine Ar/Ar age:  $16.3 \pm 0.2$  Ma; mean titanite (U,Th)/He age:  $16.5 \pm 0.5$  Ma; mean apatite (U,Th)/He age:  $16.8 \pm 1.0$  Ma (grain size 300–500  $\mu\text{m}$ ); mean fossil-track length:  $15.2 \pm 0.1$  ( $1\sigma$ )  $\mu\text{m}$ , theoretically requiring a age correction of ca. 7%; Kraml et al., 2006).

## 5. Summary and conclusions

Five dating strategies were used for determining the standardless fission-track age ( $\phi$ -age) of the Durango apatite, differing in the manner in which the induced-track densities are measured. A conventional calculation, ignoring experimental factors, gives inconsistent ages with method-related differences  $> 15\%$ . Correcting for these method-specific experimental factors brings them in line with each other and with the reference age, but leaves no margin for a correction based on the mean fossil confined-track length. Three observations suggest that such a correction is indeed inappropriate. (1) The evidence for age corrections based on the mean confined-track lengths is shown to be inconclusive. (2) The plateau age of the Durango apatite is consistent with its apparent age to within 1%. (3) The calculated effective etchable length of the fossil fission tracks agrees within error with that of the induced tracks; both are also in agreement with the measured length of induced confined tracks. Moreover, although the specific case of the Durango apatite is inconclusive, the fact that the (U,Th)/He ages of dated age standards are consistent with their reference ages, leaves no margin for a lowered fission-track age resulting from partial annealing.

The corrections for experimental factors specific to the dating method and analyst imply that – like the  $\zeta$ -calibration – standardless fission-track dating, in general, requires a personal empirical calibration factor. However, for the population method (A) and repolish method (E), it is reasonable to assume that the overall experimental factor is equal 1 for samples homogenous in uranium. For all dating methods, the calibration factor can be determined using age standards, in which case the method ceases to be standardless but becomes a deconvoluted  $\zeta$ -method (Hurford, 1998). On the other hand, it is also possible to construct the calibration factor from its components (G, Q, R, T; Jonckheere, 2003b; Enkelmann and Jonckheere, 2003; Enkelmann et al., 2005b; Soares et al., 2013) or to combine both these approaches in a single multiple calibration ( $\zeta_0$ -method; Jonckheere, 2003b). The  $\zeta$ -method has the advantage of eliminating the need for neutron-fluence measurements but the disadvantage that the substituting track counts ( $\rho_D$ ) in micas irradiated against standard uranium glasses lack the resolution for detecting all but large neutron-fluence gradients. The  $\varepsilon$ -method (Hasebe et al. 2004) and  $\xi$ -method (Gleadow et al., 2015), standardless dating methods in which the neutron irradiation and induced-track counts are replaced by mass-spectrometric uranium measurements, require the same attention to the track-counting efficiencies as the standardless ages determined in this work, and the question of the effective length of the fossil tracks cannot be ignored. These experimental factors can be sidestepped by calibration against age standards ( $\zeta_{\text{MS}}$ -method; Hasebe et al., 2013). The  $\zeta$ -method still appears to be the only accepted option for dating titanite, zircon and minerals more sensitive to irradiation damage than apatite. However, Danhara and Iwano (2013) and Iwano (pers. com., in part based on Iwano and Danhara, 1998) reported

standardless fission-track dating of zircon and titanite with the external-detector method, despite the added complications caused by radiation damage effects.

The recommendation of the  $\zeta$ -calibration by the Fission Track Working Group of the I.U.G.S. Subcommittee on Geochronology (Hurford, 1990a, b) has thus served as much to circumvent experimental factors related to track registration, revelation and counting as to circumvent the  $\phi/\lambda_F$ -problem (Hurford and Green, 1981, 1983). This meant that these problems remained unresolved but supported countless geological applications. The fact that the fission-track ages of the age standards need no correction for partial annealing explains that the  $\phi$ - and  $\zeta$ -ages of geological samples are consistent within error (De Grave and Van den haute, 2002; Enkelmann et al., 2005b, 2006; Grimmer et al., 2002; Wauschkuhn et al., 2015a). It also lends meaning to  $\zeta$ -ages, which must otherwise be interpreted as part-corrected ages (Green, 1985, 1988), with an uncertain relationship to the apatite closure temperature.

The available data indicate that the conclusion that the shortened etchable length of the fossil confined tracks in the Durango apatite has no effect on its apparent fission-track age applies to other age standards (Enkelmann et al.; 2005b; Enkelmann and Jonckheere, 2003; Iwano, pers. com., in part based on data of Iwano and Danhara, 1998; Kraml et al. 2006) and apatites with a volcanic-type length distribution (Green, 1988). It is probable that it also applies to samples with a substantial component of tracks formed at low temperatures. Recent evidence shows that terminal track sections that are unetchable in the apatite grain interiors are revealed by surface-assisted sub-threshold etching at their intersection with the surface (Wauschkuhn et al., 2015b). In consequence, the etchable lengths of the confined fission tracks must not be the same as those of the surface tracks. Step-etching results (Tamer, 2012) further suggest that the apparent shortening of fossil confined tracks in the Durango apatite could be due to a lowering of the track-etch rate in geological (radiation damaged) samples. In that case, models fitted to lab-annealing data will underestimate the length reduction of the fossil confined tracks in geological samples etched under the same conditions as induced tracks. In consequence, apatite fission-track modelling programs overstretch their (T,t)-paths and overestimate palaeotemperatures in their later parts. The familiar worldwide recent exhumation is then an artefact due to a methodological imperfection rather than a consequence of selecting the annealing model of Laslett et al. (1987). It also implies that normalizing fossil-track lengths to a personal initial length (zero-length), either that of induced tracks (Ketcham et al., 2009; 2015) or that of fossil tracks considered not to have experienced significant geological annealing (Gunnell et al., 2003; Kohn et al., 2002; 2005), has unpredictable effects on the thermal histories.

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