Author response to community comment CC3 for preprint gchron-2021-22

Issler, D. R., McDannell, K. T., O'Sullivan, P. B., and Lane, L. S.: Simulating sedimentary burial cycles – Part 2: Elemental-based multikinetic apatite fission-track interpretation and modelling techniques illustrated using examples from northern Yukon, Geochronology Discuss. [preprint], https://doi.org/10.5194/gchron-2021-22, in review, 2021.

Duddy and Green take an extreme view that alleged serious data quality issues "irrevocably" compromise the previously demonstrated (Schneider and Issler, 2019; Powell et al., 2020) conclusions that r_{mt0} is a more general parameter for characterizing the AFT annealing kinetic behaviour than wt% Cl. They conclude that the thermal history modelling therefore has no basis. They utilize select plots and impose arbitrary "eyeball" trends on the data to try and lead readers to the conclusion that both the AFT and elemental data sets are rife with errors while they ignore strong trends that link independently determined AFT age populations (from mixture modelling) with kinetic populations (determined using r_{mr0} values derived from elemental data). In an attempt to further cement their views, they suggest that it is not even appropriate to use the age mixture modelling the way we have done and therefore it is meaningless that AFT age populations just happen to coincide with kinetic populations. There is no scientific justification for this view. However, it conflicts with their Cl-based method—an approach that rests on assumptions that are not supported by published AFT annealing experiments and some field studies (see reply to their community comment CC1). Their willingness to infer trends in the data that do not exist touches on the subject of unconscious bias and the cognitive predisposition for pattern recognition with regard to the application EDM versus LA-ICPMS AFT methods (see reply to reviewer 2 comment RC2 and community comment CC1). They place a lot of restrictions on how they believe this, and future research should be undertaken and, conveniently, their recommendations conform to the way they have done it. There seems to be no room for new technological advances (LA-ICPMS) or a different way of looking at kinetic populations other than their Cl-based continuum model. The problem with their weak arguments is that they are based on opinions and a superficial examination of the data that do not hold up under closer scrutiny. We prefer not to follow their advice because it leads to confirmation bias where you only collect the minimum data needed to "confirm" a model rather than to test it. Duddy and Green either fail to recognize, or they ignore that multiple parameters are used to support our interpretation, unlike their single parameter Cl method. Below we respond to their various criticisms.

EPMA data.

Duddy and Green state categorically that only elemental data with totals between 98-101 wt% are acceptable and that data outside of this range are unfit for the purpose we are using them for. They show simple plots of wt% total versus (Ca site):(P site) and rmr0/eCl. This comparison is irrelevant because they fail to investigate the data deeply enough to determine whether their assertion-that the data provide no useful information-is correct. They suggest that we violate step 7 of our own workflow by not restricting ourselves to using only ideal data. We do not agree with their assessment of "obviously poor data" but it is clear that we need to elaborate on what we mean by this term. All the data and detailed model results are in our GSC Open File report (Issler et al., 2021; 161 p.) which is available for download and is included as an asset for the paper. All elemental totals < 97 wt% are highlighted clearly in the raw elemental wt% data tables. We adhere to scientific principles of full disclosure and make all the data available for people to scrutinize unlike Duddy and Green with respect to their Cl-based interpretation and modelling scheme. We decided that it was better to publish the data as a separate report that includes descriptions of the data and methods. We will continue to follow this model for future studies so that all AFT data sets can be obtained online from the same government source. We would also like to clarify Duddy and Green's comment on the "unreviewed online assets link (Issler et al., 2021)." All GSC reports are subject to a formal review process before they can be published. In our case, the lead author forgot to thank the reviewer in the acknowledgements after making revisions, but this oversight was corrected, and the modified version was uploaded on the 6^{th} of October 2021.

Good quality EPMA data have elemental totals between 97-100 wt% according to two experienced university-based microprobe analysts that we spoke with. We can assess our data using similar criteria as Duddy and Green (but using 97-100 wt% range) and investigate the implications for calculated r_{m0} /eCl values. First, it should be noted that elemental totals could be underestimated if apatite grains contain elements that were not analyzed. For these cases, data should not be considered poor because it is not reasonable to expect that all elements should be accounted for in samples with mixed, heterogeneous natural apatite from widely varying sources. We do not know in advance the range of elements that we may encounter in different samples, so we have settled on a suite of commonly

encountered elements that have proven to be useful based on our experience. There is not a great incentive for measuring elements that were not used for calibrating the r_{mr0} model because we lack knowledge on how they would influence annealing. Also, OH is not accounted for in the wt% totals and estimated values are in the range of 0–1.9 wt% for the studied samples. We acknowledge that some totals are too low to be attributed solely to missing elements and that they can point to analytical issues such as track void space and inclusions in the beam excitation volume. These issues are not as easy to avoid, as Duddy and Green would have us believe. Very small apatite grains can be harder to analyse and this issue is not restricted to the LA-ICPMS AFT method. EDM AFT samples can also have small grains that have elemental totals < 97 wt%. We prefer not to skip over small grains as this leads to age-grain selection bias.

The more important consideration is the magnitude of the effect on r_{mr0} calculations if elements are missing and/or elemental totals are < 97 wt%. The question should be, "What do r_{mr0} values look like for grains with lower elemental totals?" and this should be evaluated with respect to all the multi-parameter data before deciding to throw out data based on rigid culling schemes. As emphasized in the preprint, absolute kinetic parameter determination for apatite compositions not encountered in annealing experiments is not a realistic expectation—and this applies especially to the scheme of Duddy and Green which forces a Cl composition on all samples. Instead, the goals of the r_{mr0} approach are to identify different kinetic populations based on relative annealing behaviour, and qualitatively assign grains to different kinetic parameters for the more exotic very high and very low retentivity populations we have encountered. Any small variations in r_{mr0} values related to less than ideal wt% totals are accommodated easily in our approach because they are within the kinetic parameter range for the studied population. We have plenty of good data for the "anchor" kinetic populations that conform to the well-studied apatite specimens in annealing experiments that provide an absolute reference for annealing calculations.

Samples from the northern Yukon were collected and analysed over an 11-year period and the LHA003 sample was among the initial set of samples to be analysed in 2011. AtoZ Inc. produced the AFT data, arranged for the elemental analysis, and provided us with the apfu (atoms per formula unit) and r_{mr0} values that were included with the AFT data and used for the multikinetic interpretation. In subsequent years we were sent raw wt% data and calculated our own apfu values using in-house software (Probecal) that was updated to include the stoichiometric calculations of Ketcham (2015). Prior to submitting the GSC Open file data report and the gchron-2021-22 preprint, we determined that the 2011 elemental data were generated at the University of Alaska, Fairbanks. We obtained the raw elemental data to include with the Open File report and recalculated apfu and r_{mr0} values using Probecal and found that new values were similar to the old values so there was no need to revise the interpretation and modelling. This was stated in the GSC report but not mentioned by Duddy and Green.

Figure 1A shows plots of elemental wt% total versus the ratio of the sum of apfu values at the Ca and P sites for the LHA003 sample. The points are colour-coded according to different ranges of wt% totals. The dark blue dots define a region with Ca site/P site ratios for the 59% of analyses with good elemental wt% totals and 88% of the data are within this range. Eleven analyses or 12% of the data with lower elemental wt% totals have higher Ca site/P site ratios outside this range but it is not possible to predict how r_{mr0} values may be affected on the basis of this figure alone. We are fully aware that low wt% totals do not represent ideal data but we retain these analyses for the reasons given below. Figure 1B shows the same data with respect to the Ca/P ratio, showing that these ratios will vary depending on variable elemental substitutions in the apatite mineral formula. Figures 1C and 1D show the same plots for the original AtoZ Inc. data and the narrower range of values. These plots are insufficient to assess whether the data are useful without taking a closer look.



Figure 1. Total elemental wt% versus (Ca site)/(P site) (A) and Ca/P (B) for LHA003 wt% oxide data recalculated using the Ketcham (2015) stoichiometric model. Total elemental wt% versus (Ca site)/(P site) (C) and Ca/P (D) for the original AtoZ Inc. apfu values. Note that the (Ca site)/(P site) will vary depending on the elemental substitutions in the apatite mineral formula. Blue points represent good wt% totals and define a range of (Ca site)/(P site) ratios that encompass 88% of the data (A). The AtoZ Inc. data were normalized and therefore have a narrower range of (Ca site)/(P site) ratios. Both sets of data produce similar r_{mo} values.

Figure 2A shows elemental wt% total versus eCl values derived using the r_{mr0} parameter for the LHA003 sample. The dark blue symbols (open circles – kinetic population one; filled circles – kinetic population two) are for analyses with good elemental wt% totals and they define the eCl ranges for each kinetic population. We consider the analysis with the 100.1 wt% total (light blue diamond) to define the upper eCl limit (~0.52 apfu) for population two based on "good data." Only two eCl values with lower elemental wt% totals fall outside of the eCl range defined using "good data." Duddy and Green suggest that "increasingly lower totals correlate with lower r_{mr0} and higher eCl values." Instead, our data demonstrate that their inferred trend is an illusory correlation based on their expectation of how "poor" data should behave. Almost all of the r_{mr0} values with lower elemental wt% totals are within the range of the good totals and don't show any obvious systematic trend with wt% totals than kinetic population one. We included analyses with lower elemental wt% data because they contain an elemental signature that is absent in kinetic population one and therefore they provide relevant information for assigning data to the appropriate kinetic population. Average D_{par} values (mean of four measurements each unless indicated otherwise) are shown beside data points with the lowest elemental wt% values. These high D_{par} values further confirm the eCl results indicating that these grains are highly track retentive and belong to kinetic population two. The grain with a D_{par} value of 1.74 (two

measurements) is in the range where population one and two D_{par} values overlap but it has a single grain age (246.6 Ma) that is well within one standard deviation of the pooled age for kinetic population two. Furthermore, we can see that kinetic populations one and two are almost completely separated at an eCl value of ~0.07 apfu whereas kinetic populations one and two overlap entirely when plotted with respect to measured Cl only. Only two age grains from population two overlap with kinetic population one but they are well within the typical eCl uncertainty range of ± 0.03 apfu.



Figure 2. Total elemental wt% versus eCl, derived from the r_{me} parameter using detailed elemental data (left panel), and Cl (right panel). Analyses with good elemental wt% totals are in blue. Open symbols are for kinetic population 1 analyses whereas solid fill symbols correspond to kinetic population 2. Kinetic populations are resolved using eCl data with good elemental wt% totals and data with totals <97 wt% show the same range of eCl values (left panel). Dpar values are listed for the points with the highest eCl values and lowest wt% elemental totals. Kinetic populations are not resolved using Cl alone (right panel).

An examination of the tabulated AFT age data for sample LHA003 further undermines the arguments of Duddy and Green (see table below). Effective Cl values are shown for the original AtoZ Inc. normalized apfu values and the apfu values recalculated using the Ketcham (2015) stoichiometric model (2021 Probecal columns). eCl values < 0.725 apfu are coloured-coded in yellow with larger values shown in green. Both sets of eCl values are very similar and kinetic population interpretations are unaffected with virtually identical average eCl values for populations one (0.035 versus 0.031 apfu) and two (0.200 versus 0.208 apfu). Replicate eCl analyses for age and length measurements show very good agreement with most values being within ± 0.03 apfu for both good elemental totals and those outside of the 97-100 wt% range (colour-coded in orange). Importantly a number of eCl values with < 97 wt% totals are associated with replicate measurements with good totals and the eCl values are very similar for both. OH values > 0.414 apfu are highlighted in blue and are confined to population 2. Dpar values ≤ 2.38 are highlighted in green (overlap region for kinetic populations one and two) and higher values are shown in blue. Cl values ≤ 0.072 apfu are marked in yellow and it is clear that a number of low Cl values in population two are associated with good wt% totals and high eCl values due to OH and other elements. If we relied on Cl to interpret the data, we would have older AFT ages associated with low retentivity grains. Samples used in other published studies (Schneider and Issler, 2019; Powell et al., 2020) show the same pattern. Clearly, a method that relies solely on Cl interpretation is untenable as a general model for the diverse range of apatite compositions that will be encountered in nature. Given the complementary nature of our multi-parameter data, we stand by our statement that, "The Devonian outcrop sample is of high quality with 39 single grain ages and 202 track lengths that clearly define two robust kinetic populations in eCl-space (Fig. 5a, b)."

					Age meas.					2021 Probecal				Age	Lenath	
	FT	one	#			meas.	calc.	AtoZ	Age	Lenath	Age	Length		EPMA	EPMA	
Grain	age	sigma	Etch	Dpar	F	CI	ОН	r _{mr0}	eCl	eCl	eCl	eCl	Comment	wt %	wt %	
No.	(Ma)	(Ma)	Figs.	(µm)	(apfu)	(apfu)	(apfu)		(apfu)	(apfu)	(apfu)	(apfu)		Total	Total	
				۲	(inetic p	op #1 (-	0.1 < eff	ective C	< 0.072	5 apfu)						
17	84.5	59.8	2	1.96	1.926	0.003	0.071	0.841	-0.003		-0.014			99.0		
14	0.0	597.4	1	1.97	1.771	0.009	0.218	0.838	0.006		-0.003			98.1		
1	30.0	30.0	1	2.00	1.961	0.006	0.028	0.835	0.017		0.019			100.0		
36	125.4	23.9	4	2.24	1.975	0.021	0.000	0.832	0.025		0.034			96.4		
40	104.0	26.5	4	2.07	1.556	0.029	0.414	0.828	0.036		0.031			96.5		
3	183.2	75.0	3	2.10	1.647	0.023	0.329	0.824	0.047		0.041			96.8		
26	134.8	67.6	3	2.38	1.621	0.004	0.375	0.822	0.050	0.017	0.043	0.007	len gr 28	97.7	98.6	
28	130.9	23.1	4	2.05	1.633	0.070	0.293	0.819	0.060	0.067	0.054	0.062	len gr 30	96.7	97.3	
24	115.1	36.8	4	1.92	1.893	0.013	0.093	0.814	0.072		0.072	· · · · · ·		97.3		
	119.3	12.2	-			0.020			0.035		0.031		Q=86%			
	122.8 12.4											disp = 0%				
Kinetic pop #2 (0.0725 < effective CI < 0.7 apfu)																
7	226.2	37.1	4	1.70	1.845	0.085	0.069	0.822	0.052		0.047			96.6		
13	229.1	57.8	4	2.04	1.898	0.005	0.096	0.821	0.053		0.048			97.3		
32	246.7	46.4	4	2.15	1.985	0.001	0.014	0.814	0.073	0.101	0.075	0.102	len gr 39	98.5	98.7	
11	293.6	46.1	4	2.36	1.430	0.027	0.540	0.804	0.098		0.096			94.1		
30	449.6	83.9	4	2.21	1.194	0.045	0.756	0.799	0.108	0.109	0.100	0.106	len gr 33	97.5	97.7	
6	313.6	46.7	4	2.21	1.884	0.116	0.000	0.796	0.116	0.114	0.123	0.117	len gr 4	98.6	99.6	
31	313.8	79.1	4	2.61	1.128	0.066	0.804	0.796	0.116	0.120	0.117	0.125	len gr 40	98.9	97.2	
39	175.0	49.1	4	2.42	1.205	0.073	0.722	0.793	0.124		0.121			97.6		
9	227.6	50.6	4	2.22	1.438	0.075	0.485	0.793	0.124	0.094	0.122	0.088	len gr 9	92.6	97.1	
19	232.2	55.3	4	1.80	1.586	0.054	0.359	0.792	0.126		0.127			99.2		
34	225.6	52.2	4	2.33	1.921	0.057	0.022	0.791	0.127		0.151			96.7		
22	217.0	36.3	4	2.25	1.446	0.207	0.338	0.775	0.163	0.167	0.166	0.167	len gr 22	95.3	96.6	
12	246.6	142.6	2	1.74	1.509	0.187	0.303	0.771	0.171		0.174			88.8		
15	246.5	45.7	4	2.18	1.595	0.081	0.323	0.764	0.185	0.174	0.197	0.182	len gr 17	94.2	98.9	
21	223.3	32.8	4	2.30	1.661	0.338	0.000	0.761	0.192		0.204			98.7		
37	159.2	159.3	1	2.07	0.976	0.028	0.994	0.759	0.196		0.205			97.1		
20	203.7	40.0	4	2.11	1.040	0.200	0.070	0.758	0.197		0.202			07.0		
233	316.7	1/2 1	4	2.00	1.7.54	0.076	0.100	0.757	0.200		0.213			97.0		
27	159.5	56.6	4	2.19	1.400	0.020	0.323	0.734	0.204		0.200			90.0		
10	615.9	252.9	4	2.23	1.501	0.120	0.263	0.743	0.227		0.223			95.2		
29	207.6	85.0	4	2.23	1.385	0.196	0.419	0.742	0.228		0.234			95.9		
4	316.5	82.5	4	2.58	1.376	0.126	0.496	0.717	0.272	0.277	0.288	0.289	len ar 6	95.3	99.3	
35	216.7	97.2	4	2.61	1,202	0.112	0.686	0.708	0.287	0.21	0.308	0.200	.o., g. o	97.5	00.0	
16	237.7	55.0	4	2.41	1.320	0.175	0.496	0.704	0.293	0.283	0.309	0.313	len ar 18	95.2	87.2	
25	236.1	68.9	4	2.48	0.970	0.211	0.816	0.672	0.341	0.303	0.352	0.314	len ar 29	96.2	95.6	
23	143.9	64.5	4	2.62	0.881	0.155	0.962	0.664	0.353	0.321	0.372	0.332	len gr 21	94.8	95.9	
8	303.3	74.1	4	2.19	0.968	0.233	0.799	0.625	0.405		0.427		3, 2,	93.0		
38	205.2	63.2	4	2.91	0.765	0.471	0.758	0.501	0.540	0.517	0.592	0.530	len ar 45	89.0	100.1	
5	370.8	99.8	4	3.43	0.772	0.652	0.576	0.310		0.694			len ar 7	90.7	92.8	
	252.4	11.6				0.1500		0.7348	*0.216		0.208		Q=31%			
	258.3	11.6							*0.200				disp = 7%			

Apatite fission track sample: 2009LHA003 (1148-06) Imperial Fm., Late Devonian

*Average eCl for population 2 including replicate eCl value for length grain 7

**Average eCl for population 2 excluding eCl value for length grain 7

Detailed examination of the data for sample P013-12 leads to the same conclusions. Figure 3 shows a much narrower range of Ca site/P site ratios for P013-12 compared with LHA003, with 96% of the data within the region defined by the analyses with good elemental wt% totals (left panel)—three points barely fall outside the region and are not significantly different. For reference, the Ca/P plot (right panel) shows a wider range of values, which indicates how different elemental substitutions affect the ratio. Duddy and Green have chosen to plot the P013-12 data at a different scale than for LHA003 and draw a dashed inferred trend on the data to indicate that there is a correlation between calculated ratios and elemental wt% totals. This is an illusion because the apparent shift to higher ratios with lower elemental totals coincides with a change in kinetic population with no apparent effect on calculated r_{mr0} values (left panel, Figure 4). Three kinetic populations are resolved using eCl with a few overlapping grains as discussed in preprint gchron-2021-22. In contrast, there is complete overlap of populations one and two when the data are plotted with respect to Cl (right panel). We believe that the criticism of Duddy and Green lacks merit and that our data do "*provide a reliable discrimination of differential AFT annealing.*"



Figure 3. Total elemental wt% versus (Ca site)/(P site) (A) and Ca/P (B) for P013-12 wt% oxide data calculated using the Ketcham (2015) stoichiometric model. Note that the (Ca site)/(P site) will vary depending on the elemental substitutions in the apatite mineral formula. Blue points represent good wt% totals and define a range of (Ca site)/(P site) ratios that encompass 96% of the data (A).



Figure 4. Total elemental wt% versus eCl, derived from the r_{m0} parameter using detailed elemental data (left panel), and Cl (right panel). Analyses with good elemental wt% totals are in blue. Open circles are for kinetic population 1, solid fill circles are for kinetic population 2, and open triangles correspond to kinetic population 3. Kinetic populations are resolved using eCl data with good elemental wt% totals and data with totals <97 wt% show the same range of eCl values (left panel). Kinetic populations are not resolved using Cl alone (right panel).

Duddy and Green cite unpublished data to support their assertions and methods. They claim to have 1057 full EPMA analyses and, on the basis of a relatively small set of data, proceed to give us advice on what "*should be the norm for any apatite EMPA study*." We have tens of thousands of EMPA analyses and have encountered far more situations than these commentators when it comes to acquiring and using complete EPMA data to characterize AFT kinetic populations. What Duddy and Green provide is hearsay "evidence" with little explanation on the conditions under which the data were acquired. For example, what apatite grain sizes were studied and how many elements were analysed? We get excellent EPMA data for many of our AFT samples, but low elemental totals associated with very small grains are not easily avoided unless you simply skip over the grains. Low EPMA wt% totals occur for some small grains with AFT data acquired using EDM (no laser and no Cf-irradiation). We already know that Duddy and Green generally only measure AFT age for about 25 grains per sample, which is too low (marginal at best) to properly characterize age distributions in multikinetic samples. Therefore, age bias exists. We don't know whether the authors also prefer to select larger grains to get good analyses but that would be consistent with a result where almost 100% of the data have good totals. We give a detailed discussion of our choice of analytical steps and discuss the pros and cons of different approaches in our reply to reviewer 1 comment RC1.

LA-ICPMS AFT Data

We are fully aware that Duddy and Green, as developers and promoters of the external detector method, do not believe that the LA-ICPMS method yields reliable AFT data in spite of published scientific studies to the contrary. This bias against new technologies is very clear in their community comments: "the willingness of many thermochronology labs to invest in expensive new machines to measure uranium for fission track studies by laser ablation, and to measure U-Th/He ages, when these methods are far from proven and have been shown to provide misleading results in many cases" and "so much money has been devoted to investment in other aspects of thermochronology which are far from proven." We find this perspective to be puzzling because (in our opinion) investigating new technologies should be a normal part of scientific research and an essential activity in pushing forward scientific knowledge. Having questioned the utility of the EPMA data, it is no surprise that their next line of attack is to try and claim that the AFT data are unreliable as well. Their line of criticism involves using tabulated information inappropriately to try and "demonstrate" that U zoning is an issue that compromises AFT ages, raising side issues that are not relevant to the preprint, and making unsupported statements concerning the accuracy of the method. Hopefully they can put aside their bias and conduct their current research into the LA-ICPMS method in an objective manner.

Despite what Duddy and Green claim, the GSC Open File data report contains all the information required to calculate single-grain and pooled ages for the tabulated data when using published age equations for the LA-ICPMS method. The information was extracted from Excel data sheets with embedded formulas that calculated ages directly using the data. Duddy and Green need to read the report more carefully. They complain that the vast amount of raw laser data that were processed using specialized software to yield the final parameters for age calculation are not included with the reduced data. This is consistent with reporting of EDM AFT data where only final summarized grain count data are reported and none of the "raw" images of grain mounts showing the selected count areas are included. Without such information, it is not possible to assess the potential role of age selection bias or to evaluate any single-grain data trends. Duddy and Green do not conform to these minimum EDM reporting standards in terms of including detailed tables of single-grain ages and individual length measurements as documentation for their own publications. In contrast, we believe our report sets a high standard for reporting data and provides enough information for readers to assess the method and replicate our model results.

Duddy and Green speculate that, "the data for sample P013-12 shows evidence for significant U-zoning in many grains although no mention is made of this in the paper under review." The reason U zoning is not mentioned in the paper is because we consider that zoning is not a significant issue for our data. We would expect a lot more age variation and a lack of correlation between age and elemental composition if zoning were a problem. Instead, we find that high sample age dispersion disappears when single-grain ages are sorted into different kinetic populations using independently acquired elemental data. The fact that each of the individual kinetic populations have low age dispersion, high χ^2 probabilities, and ages that are consistent with independently determined age populations from mixture modelling is compelling evidence for the existence of kinetic populations. These results are also consistent with the elevated thermal maturity of the samples, which indicates high degrees of thermal annealing—conditions that are conducive to the formation AFT kinetic populations. Duddy and Green would have us believe that poor quality AFT and EPMA analyses, collected independently of one another, have worked systematically in concert to

generate data that only give the appearance of representing kinetic populations. We would assign an extremely low probability that these purported errors affect two samples of different age from different locations and analyzed years apart, in exactly the same way. We think the probability is infinitesimally low when including results of other published studies for rocks of different age from different locations with data that were acquired using different labs and methods (Schneider and Issler, 2019; Powell et al., 2020).

Duddy and Green use the U values incorrectly even though it is stated in the GSC Open File data report that these values are tabulated but should not be used because they are inaccurate as discussed in Cogné et al. (2020). Only the ²³⁸U/⁴³Ca values should be used for age calculations. We report the raw, uncorrected U, Th and Sm values for the sake of completeness and transparency. It is interesting that Duddy and Green say, "In fact, Cogne et al. (2020) recommend against even reporting U ppm values (and presumably also Th and Sm) determined by LAICPMS as they are not accurate" and yet they ignore their own advice and use the same "inaccurate" data to claim that they provide "*clear evidence for U-zoning*." Plots of ²³⁸U/⁴³Ca versus U are not used as a quality control criterion because they can give misleading results. Data reduction involves auto-filtering and depth-averaging of laser data and therefore plots of ²³⁸U/⁴³Ca versus U will not necessarily show a consistent relation. Plots such as their Figure 4 (see community comment CC3) provide fodder for speculation and incorrect interpretation. When a laser intersects an inclusion, it can generate a sharp spike in the U, Th, and Sm depth profiles and produce extremely high values. We know that the effects of inclusions encountered vertically during ablation can be identified based on these types of signatures. The influence of inclusions on ²³⁸U/⁴³Ca values depends on the depth of the inclusion and the degree to which it is intersected by the laser. In mild cases, auto-filtered data yield AFT ages that are similar to other ages encountered in a sample. In more extreme cases, filtering alone cannot remove the effects of inclusions or other factors that contribute to high ²³⁸U/⁴³Ca values and artificially young AFT ages (see below). In such cases, the single-grain analysis is flagged, and removed from the final sample analysis. Duddy and Green have decided that they "feel" U-zoning is a more likely explanation with no evidence to support the claim. In any case, the extremely young ages have U profiles that do not pass quality control checks and therefore should be rejected. Fortunately, this is a relatively rare occurrence, and we find that all the age data presented as part of the final sample analyses can be used in most cases.

Green and Duddy point to the conference abstract of Seiler et al. (2013) where they compared AFT methods as proof that LA-ICPMS data are inaccurate at low U. For context the Seiler et al. abstract states: "*The comparison shows that, with a few exceptions, single grain fission track ages from LA-ICP-MS and EDM are concordant within analytical uncertainties and scatter symmetrically around the 1:1 correlation line. Although the relative difference in single grain ages varies significantly in either direction (up to 70%), there are no systematic variations between the two methods suggesting that this variation is simply due to random sampling effects. However, we did find systematic offsets in grains that have either very low or very high U concentration. At low U levels (less than a few ppm), apparent fission track ages obtained by LA-ICP-MS are consistently older than those from EDM. Analysis of an internal apatite standard with well-established and homogeneous U concentration indicates that are too old. By contrast, apatites with high U yield apparent LA-ICP-MS ages that are systematically younger than those determined by EDM. In this case, the EDM results are probably less robust as induced (mica) track densities are so high that tracks become difficult to resolve, which leads to an underestimate of the EDM derived U content (and hence an older apparent EDM age)."*

While these claims make sense from a first-order methodological perspective, those data were never published, and details of the analyses are unknown. By comparison, the apparent EDM ages that are 'too old' due to high N_s density could also be problematic and bias AFT results—but Duddy and Green ignore these issues. Another interesting and poorly known variable recently discussed by Gleadow et al. (2019) that may play a significant role in age calculation is the etchable length of a single fission track, and if this value deviates significantly between unknowns and age standards resulting in too old or too young AFT 'model' ages versus the 'true' age as expected from the age equation. That is to say, there are poorly quantified variables that can affect AFT ages derived from both the EDM and LA-ICPMS methods—so how can they be used reliably to say which method is "better" or "more correct"? All that can really be asserted at this time is that both methods usually agree most of the time (the main point of Seiler et al. 2013) and reiterated in Cogné et al. 2020).

LA-ICPMS AFT methods are well documented in the scientific literature and have been shown to produce results that are similar to EDM data (e.g., Cogné et al., 2020). We see no need for the requirement to rehash old arguments

and defend the method every time it is being used. The method yields consistent results within individual samples and for different samples across a study area. If someone wants to invoke unsupported zoning issues in the face of a consistent dataset then there is not much you will be able to do to convince them otherwise.

Duddy and Green question why we reject a small number of age grains (one out of 40 or 2.5% for LHA003 and three out of 64 or < 5% for P013-12). The simple reason is there is nothing to be gained by including data that fail quality control, as this would be poor scientific practice. We consider the very low rejection rate as a testament to the consistent quality of the LA-ICPMS method. The rejected grain for LHA003 has ${}^{238}U/{}^{43}Ca = 0.0003$ (estimated U ~ 0.09 ppm) which is clearly below the accuracy for U measurement, and it yields a 1.8 Ga age with an uncertainty range of 181 Ma to 10 Ga. We think that a result that encompasses an age older than the Earth should be rejected as "obviously poor" with no useful information content. Similarly, a grain with $^{238}U/^{43}Ca = 0.0034$ and a 1.9 Ga age with an uncertainty range of 735 Ma to 4.2 Ga was removed due to low precision on ²³⁸U/⁴³Ca. After examining thousands of LA-ICPMS analyses for Phanerozoic AFT samples, we have found that a useful cut-off value for ²³⁸U/⁴³Ca is ~ 0.003. Below this value, AFT ages increase rapidly in the gigayear range with up to gigayear uncertainties. We find that low-U grains with $^{238}U/^{43}Ca$ ratios above ~0.003 yield similar ages to grains with higher U concentrations but with larger uncertainty. Two other anomalously young ages (2.7 Ma and 10 Ma) were rejected for the P013-12 sample because they were completely 'blown out' by the laser, which is why they have no associated EPMA data. All the grain material went into the plasma for analysis, which yielded very high initial isotope values followed by no signal. This resulted in high ²³⁸U/⁴³Ca (0.31 and 0.49), U, Th, and Sm values, and very young AFT ages because all the U was treated as contributing to the formation of tracks near the surface. These two ages appear as a distinct separate age population on the radial plot (8 Ma versus 57 Ma). Inclusion of the two ages into population one increases age dispersion from 9% to ~40% and the χ^2 probability drops from 64% to 0 %. These two anomalous ages have a distorting effect on the central age, which decreases by 10 Ma if they are included. Based on these observations, they do not pass data quality control and are excluded from further discussion.

Following the logic of Duddy and Green, it seems that we should not report poor analyses, or we should not apply quality control procedures because all measurements should be perfect. It would seem the only appropriate reason for culling data is when it does not conform to the Cl model (see Figure 4 of Green and Duddy community comment CC1). Our criterion for rejecting a few age grains in this study is an obviously poor U analysis. It is important to include all data in the tables, even poor analyses, to ensure full transparency and objectivity. People are then able to assess whether poor analyses are a common occurrence. If everyone culls data prior to publication, a true assessment of potential analytical issues or data nuances are not possible if the published data always appear `perfect'. This may lead to a misconception that all data are in fact `perfect' and contribute to increased rates of data omission and repression during scientific publication due to fears of public derision or future reprisal if there are analyses that did not meet data standards. We have a low rejection rate for our samples and can use most or all of the data. These results are not consistent with the conviction of Duddy and Green that the LA-ICPMS method yields unreliable data. We also strongly disagree with the unsupported assertion of Duddy and Green that the LA-ICPMS method cannot provide accurate data at < 10 ppm. We routinely get similar AFT ages for low U grains and high U grains when the 238 U/ 43 Ca > ~0.003 which implies ≤ 1 ppm. Of course, if data are not properly interpreted within a multikinetic framework, then older ages may simply be rejected, if for example, they do not conform to a model (see Figure 4 in community comment CC1 and author reply AC3).

Duddy and Green inappropriately refer to a completely different study by McDannell et al. (2019) to suggest that data quality control is inconsistent. The conditions influencing these much younger Phanerozoic samples are not the same as those affecting the much older Precambrian samples in McDannell et al. (2019) and to equate the two completely different sample sets is misdirection. However, since it was mentioned by Duddy and Green, the phenomenon of "radiation-enhanced annealing," (REA) borne out of longstanding observations of negative AFT age-U trends in Precambrian basement AFT samples was revisited by McDannell et al. (2019) primarily to spur the geochronological and material science/nuclear physics communities to critically re-examine these empirical observations, rather than simply dismiss them. Following that publication and discussions with members of those communities, Li et al. (2021) experimentally validated that alpha-decay causes enhanced fission-track annealing in both apatite and zircon, yet the effects may only be an issue for zircon. However, questions remain regarding whether track annealing in apatite is affected by alpha-damage over *geologic time* and whether this may potentially reduce track thermal annealing resistance (Ketcham, 2019; McDannell et al., 2019). Recently, Cogné and Gallagher (2021) posited that apparent age-U correlations may be instead due to the collection of LA-ICPMS AFT data using

only a single ablation spot rather than REA, however this is uncertain since their multi-spot LA data show similar trends as the single-spot data and negative single-grain age-U trends also exist in vintage and newer cratonic EDM data. Regardless of these arguments and questions, the LHA003 and P013-12 single-grain ages show no correlation with ²³⁸U/⁴³Ca unlike the Precambrian samples in McDannell et al. (2019) and this topic is not relevant to Duddy and Green's comments on preprint gchron-2021-22.

We reiterate our view (see reply AC2 to reviewer 2 comment RC2; as well as the abstract by Seiler et al., that Duddy and Green cited) that LA-ICPMS AFT has certain advantages with respect to EDM—not that it is *better*. In addition to lowering the possibility for age selection bias, single-grain ages tend to have higher associated precision because uncertainties on U measurement are generally less than those determined using the proxy-counting method with EDM. This can be viewed by some as over-precision as expressed by Duddy and Green who state, "*the generally increased precision of U-determination by LAICPMS over EDM is at the expense of accuracy.*" We have a different perspective based on a comparison of EDM AFT data in the Mackenzie Delta region versus LA-ICPMS data for the northern Yukon. Age analysis using Binomfit (which includes an F-test for assessing whether multiple populations are present) consistently shows two kinetic populations are present in Paleogene strata of the Mackenzie Delta whether or not samples passed the χ^2 text. When age data are plotted with respect to eCl, two kinetic populations are visible in accordance with the results of age mixture modelling even for samples that pass the χ^2 test due to large errors on single-grain ages with low U. For northern Yukon LA-ICPMS AFT samples, single grain age errors tend to be smaller and kinetic populations are better resolved as a result. The issue of why EDM data have a better chance of passing the χ^2 test is briefly discussed by McDannell (2020), while the nuances of statistical model assumptions, which affect the results of the χ^2 test and mixture modelling are covered by Vermeesch (2019).

rmr0 calculations

Elemental values used for eCl/r_{mr0} are shown in the elemental apfu data tables in the report of Issler et al. (2021). Elements such as Ti, S, Zr, Si, P, Al, Ba, As, K, Br, and I were not used because they do not appear in the original Carlson et al. (1999) equation. Duddy and Green repeat cautionary statements about the use of r_{mr0} in Carlson et al. (1999) and Ketcham et al. (2007). They then go on to say, "Issler et al. dismiss these warnings (Lines 555-561) and proceed without regard to the limited constraints on chemical composition available in the original Carlson et al (1999) annealing data set, for which no follow-up studies that might confirm or otherwise the importance of elements other than Cl have been undertaken in almost the last 2 decades." We fully understand the cautionary statements and agree that r_{mr0} will not accurately predict kinetic parameters for natural samples outside of the range of experimental calibration data and that is clearly stated in preprint gchron-2021-22. That is not how we are applying rmr0. The majority of specimens in the Carlson et al. (1999) lab annealing experiments have rmr0 values between 0.73–0.84 (eCl of 0–0.25 apfu). This provides a very good calibration for apatites that commonly occur as a kinetic population within multikinetic samples. These populations provide a suitable absolute reference for estimating eCl/r_{mr0} values for other kinetic populations within samples that have not be studied in the lab. While we understand the need for the cautionary statements concerning rmr0, they have had the unfortunate effect of dissuading people from doing follow-up studies and from trying to use the method. We do not share the concerns of Duddy and Green that "no element that is significant to the r_{mr0} calculation is particularly abundant" or "These values are very much less than measured in the single high Fe, Mn and Sr apatites in the Carlson et al. (1999) data set." This type of thinking leads to an expectation that these elements should have no significant effect on annealing and yet this biased view is not supported by available evidence. Duddy and Green choose to casually dismiss the evidence as being unreliable and revert to their conviction that only Cl matters.

What is controlling r_{mr0} in the two samples?

Duddy and Green present another plot (Figure 5 in community comment CC3) that misleads readers. They claim their plot of r_{mr0} versus wt% Cl shows that "the major control on r_{mr0} is clearly the chlorine content above all other elements" and that "major excursions from the trend probably reflect the low quality of the EMPA analyses." These statements are made without checking to see if they are true. We agree that Cl is an abundant element and that it has a significant effect on thermal annealing. For many grains, r_{mr0} does increase with increasing Cl content. Our point is that it is not the sole element controlling annealing and that other elements will influence annealing significantly as can be demonstrated when the data are examined more closely. Furthermore, they quote Barbarand et al. (2003) to support their view that "none of the measured elements are outside the typical range of 'normal' apatite" and therefore Cl controls annealing and nothing else is important. Barbarand et al. (2003) studied much less

compositionally diverse, Cl-dominant apatite in comparison to Carlson et al. (1999). Therefore, it should be no surprise that Cl is seen as the major control on annealing. Given the very limited amount of data available, it is hard to make general statements concerning "normal" apatite and the range of compositional variation in natural samples (also see Ketcham comment RC3 on CC5). Duddy and Green make the logical leap that the small number of specimens used in annealing experiments represents the majority of apatites that will be encountered. This is an optimistic and unsupported assertion that is not consistent with our work or the original annealing studies.

Our Figure 5 (below) shows plots of eCl and r_{mr0} versus wt% Cl with colour-coded points grouped by total elemental wt%. We have already shown above that lower and higher elemental wt% totals yield similar eCl/ r_{mr0} values and therefore the admonishment of Duddy and Green that much of the data should be rejected is not supported. However, in keeping with the quality control recommendations of Duddy and Green, we can see that data with good elemental wt% totals (filled and open dark blue symbols) show the largest "excursions from the trend" that Duddy and Green casually attribute to the low quality of EPMA analyses. What these deviations from the trend unequivocally demonstrate is that elements other than Cl have a significant effect on AFT annealing. For example, Figure 5E and 5F show that Cl varies by up to 1.1 wt% (from 0.09 to 1.2 wt%) for eCl and r_{mr0} values of 0.2 apfu and 0.76, respectively. If we adopt the recommendations of Duddy and Green, then old age grains are grouped with young age grains, and we can either model them as χ^2 failures (ignoring the results of the radial plot) or reject the anomalous values (see their Figure 4 in community comment CC1).

Multikinetic annealing

Duddy and Green say "We have investigated the effect of apatite composition of AFT annealing since the early 1980s and have incorporated the chlorine content in our work since for over 30 years. Despite the overwhelming evidence of the importance of Cl (e.g., Green and Duddy, 2012), we are bemused at the reluctance of the community to take it on board." Our question is, "What is the overwhelming evidence and where is it?" Duddy and Green have released selected plots and model results over the years to support their ideas. Where are the underlying data sets? Where are the detailed descriptions of experimental procedures outlining the elements that were measured and the range of samples that were used to calibrate their model? Where are the details of their annealing experiments? Were all the samples used to develop the model from one location or do they represent a sampling of apatite from different regions and rocks of different ages? Frankly, we are not surprised that the community has chosen not to use the model. It is not a standard scientific approach to accept ideas at face value that have not undergone rigorous peer review. Duddy and Green go on to say, "the approach of collecting "complete" compositional data and calculating r_{mr0} is unnecessary. Simpler and better results can be achieved by determination of chlorine alone." It is an interesting position to say less data are better unless you are concerned that more data may reveal shortcomings with an existing method. If you only collect Cl data, then you are not able to say other elements have no influence. This is just a conviction that Duddy and Green seem to not want to be tested by others.

Further comments for the benefit of the authors

All the recommendations by Duddy and Green are self-serving and designed to limit further scientific investigation of multikinetic annealing. Duddy and Green suggest that the only acceptable way to undertake the research is the way that they have done it. Following their recommendations ensures confirmation bias. Their approach is to assume the correctness of the Cl-based model and have the data conform to their assumptions. Minimal data (Cl only) are collected, radial plot results are ignored because they believe that the "continuum model" is correct, and AFT data are sorted into bins which dilutes the information from age and length data and makes it easier to obtain some type of model fit. "Occasional" outliers (in the context of 25 grains being measured) can be safely discarded because they don't fit the model and there is no need to understand the reason why. We use a data-driven, rather than model-driven, approach that uses multiple parameters and independent lines of evidence to better understand what the data represent.



Figure 5. eCl and r_{m0} versus wt% Cl for LHA003 (A,B), P013-12 (C,D) and the combined data (E,F). Points are colour-coded by elemental wt% total. Data with good totals show a large spread in wt% Cl values for a given eCl/ r_{m0} value indicating that elements other than Cl are controlling AFT annealing kinetics.

Duddy and Green suggest that demonstrating r_{nr0} is a useful measure of annealing "can never be achieved with the analysis of further outcrop samples or well samples of the type used in this study." We completely disagree with this assertion based on looking at > 150 Phanerozoic core and outcrop samples with detailed elemental data from different areas of Canada. These samples have a broad range of apatite compositions, and much can be learned about how they behave in different tectonic environments with different thermal conditions. Duddy and Green suggest that field studies are of no utility unless undertaken under highly restricted conditions using core samples from wells that are at maximum temperature or that each field sample with different chemistry must be subject to heating experiments before we can come to any conclusions. Naturally, this would make the method cumbersome and impractical to use because we would be confined to studying apatite compositions associated with publicly available core samples from a limited number of sedimentary basins or having to do annealing experiments each time we encounter a new sample with different chemistry in order to use "absolute" kinetic parameters every time we do thermal history modelling. We already have a suite of multikinetic AFT core samples from across the Mackenzie Delta with high-quality temperature and thermal maturity data (including offshore wells that are at maximum temperature) that fit the criteria outlined by Duddy and Green and a subset of results have been published (Schneider and Issler, 2019; also see author reply AC3 to community comment CC1). These results further demonstrate the utility of our method, but they are for particular apatite compositions encountered in that study region and these can differ from apatite compositions found in northern Yukon.

While we welcome and encourage additional annealing experiments to improve the r_{mr0} calibration, we present a practical method for exploiting multikinetic behaviour that doesn't rely on an absolute calibration for all apatite compositions that may be encountered. Our method uses the same basic assumption of Duddy and Green and other workers that: (1) the same AFT annealing mechanism applies to all apatite grains but that, (2) the annealing parameters vary with composition. We acknowledge that annealing kinetics are well constrained by lab experiments for simple apatite compositions dominated by Cl and F. These are common in the less track-retentive component of many multikinetic samples, and they serve as an absolute reference for estimating parameters for poorly understood apatite compositions. Assumptions (1) and (2) are supported by annealing experiments and are fundamental to our approach of using relative annealing behaviour and observed AFT data to determine eCl values for other kinetic populations with a shared thermal history. Importantly, the results of additional annealing experiments and r_{mr0} recalibration in the future will not change our fundamental data interpretation approach by using radial plot mixture modelling and sorting/grouping data by kinetic parameter—new experimental results will only improve both the absolute and relative annealing framework on which these interpretations are based. Furthermore, the data do not have to conform to a rigid, ad hoc interpretive model, which will inevitably result in rejection of any nonconformist data points.

Duddy and Green claim that "the approach employed by Issler et al in defining age populations and then applying an arbitrary compositional boundary is backwards. If apatite composition is important, it is important in all samples, regardless of the range of AFT ages. Compositional boundaries should be defined based on the annealing kinetics of known compositions, and the ages in each group should be compared with model ages to derive a common thermal history." We disagree and think it is unwise to ignore statistical age populations that generally match those determined using elemental data. Of course, composition always matters, and the range of AFT ages encountered will be dependent upon the specific thermal history experienced by the rocks in question. Detrital AFT populations have heterogeneous compositions that do not entirely conform to the simple assumptions of a Cl-only based scheme so there is no reason to believe that they must conform to the idealized continuum model used by Duddy and Green. Again, our approach is informed by what is observed in the data rather than on preconceived notions of how natural samples are expected to behave.

In addition, Duddy and Green already have demonstrated a strong bias against using LA-ICPMS for U determination so their recommendation to avoid using it comes as no surprise. It is also unclear to us how a proxy method can be considered absolutely superior to a direct measurement analytical technique that is widely used by the geochronological community in a variety of applications. If modern LA-ICPMS techniques were available 50 years ago, would there have even been a need for development of the EDM? To be clear, there is nothing 'wrong' with the EDM, but to ignore, downplay, or outright reject new analytical methods for apparently no good reason other than personal bias, hearsay, or misunderstanding is unscientific. Obviously, there are specific analytical concerns that need to be accounted for with LA-ICPMS AFT methods (true for *any* analytical method), but if U concentrations (ratios) were unable to be measured at a suitable level of accuracy or precision, then the U–Pb community may also be in trouble. There are a fair number of places to look in the literature where U can be

measured by LA-ICPMS accurately and precisely for age standards and unknowns at the low ppm level with no difference in calculated ages for grains containing higher U concentrations (e.g., Hasebe et al., 2009; Soares et al., 2014; Soares et al., 2015; Pickering et al., 2020). For example, the Soares et al. (2014) LA-ICPMS results for Durango (7.4 μ g/g U) and Mud Tank apatite (6.8 μ g/g U) demonstrate that both AFT age standards (< 10 ppm in this case) show variation in uranium concentration within 3% (1RSD)—thereby obtaining AFT ages in total agreement with known ages for these lab age standards. This paper also shows that in some situations, instances of U-zoning can clearly be identified during ICP-MS analysis (analogous to what we described above regarding high U spikes or grain blowouts). Our own experience shows that we can define multikinetic populations using the EDM or the LA-ICPMS method and both methods can yield harmonious AFT results for the same samples (e.g., Cogné et al. 2020). Well-cuttings samples can be problematic when contamination and cavings issues can be demonstrated and we have samples where that is the case. However, for some regions, only cuttings samples are available, and it is worthwhile to determine whether they provide useful information before rejecting them out of hand. Geotrack did a lot of work for the petroleum industry, and we find it doubtful that they did not use cuttings samples given that cores are rarely taken during drilling of exploration wells.

-Dale Issler, Kalin McDannell, Paul O'Sullivan, and Larry Lane

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