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Detrital zircon and apatite (U-Th)/He geochronology of intercalated baked sediments: A new approach to dating young basalt flows

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[1] Simple numerical models suggest that many basaltic lava flows should sufficiently heat the sediments beneath them to reset (U-Th)/He systematics in detrital zircon and apatite. This result suggests a useful way to date such flows when more conventional geochronological approaches are either impractical or yield specious results. We present here a test of this method on sediments interstratified with basalt flows of the Taos Plateau Volcanic Field of New Mexico. Nineteen zircons and apatites from two samples of baked sand collected from the uppermost 2 cm of a fluvial channel beneath a flow of the Upper Member of the Servilleta Basalt yielded an apparent age of 3.487 ± 0.047 Ma (2 SE confidence level), within the range of all published $^{40}Ar/^{39}Ar$ dates for other flows in the Upper Member (2.81–3.72 Ma) and statistically indistinguishable from the $^{40}Ar/^{39}Ar$ dates for basal flows of the Upper Member with which the studied flow is broadly correlative (3.61 ± 0.13 Ma). Given the high yield of ^{4}He from U and Th decay, this technique may be especially useful for dating Pleistocene basalt flows. Detailed studies of the variation of (U-Th)/He detrital mineral dates in sedimentary substrates, combined with thermal modeling, may be a valuable tool for physical volcanologists who wish to explore the temporal and spatial evolution of individual flows and lava fields.

Components: 5100 words, 3 figures, 1 table.

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1. Introduction

[2] Placing precise and accurate age constraints on young basalt flows can be challenging. U-Th-Pb dating is commonly hindered in basaltic rocks by a lack of U- and Th-bearing phenocrysts such as zircon and apatite. This leaves $^{40}Ar/^{39}Ar$ dating as the favored method, one that provides highly precise and robust dates for many samples. However, uncertainties regarding $^{40}Ar/^{36}Ar$ initial ratios and variable amounts of xenocrystic contamination, combined with generally low potassium contents for most basalts, can render some $^{40}Ar/^{39}Ar$ dates imprecise and unreliable [*Kelley*, 2002; *McDougall and Harrison*, 1999, and references therein]. Such issues have stimulated considerable interest in test-

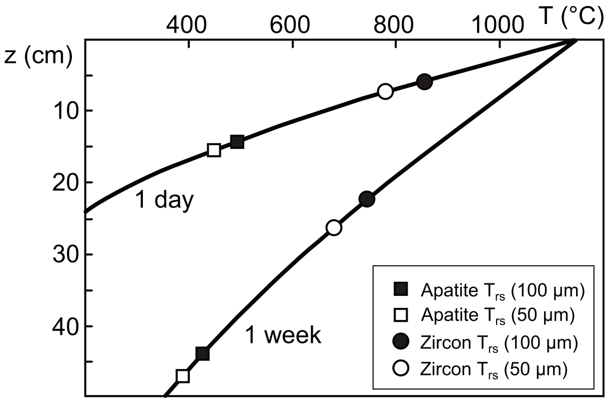

Figure 1. Illustrations of the theoretical temperature distribution beneath a basalt flow (based on a 1-D conductive heat transfer model) and approximate resetting temperatures for (U-Th)/He thermochronometers in subjacent sediments. Curves for continuous flow durations of 1 day and 1 week are shown. Resetting temperatures (T_{rs}) for zircons (circles) and apatites (squares) were calculated using the equations presented by *Gardés and Montel* [2009], kinetic data for He diffusion in apatite [*Farley*, 2000] and zircon [*Reiners et al.*, 2004], a radial-cylindrical diffusion geometry for apatite, a spherical diffusion geometry for zircon, and heating rates at various levels beneath the basalt as derived from the thermal models for 1 day and 1 week flow durations. Open squares and circles are for a_A and a_Z equal to 50 μm; solid symbols represent 100 μm grain half sizes.

ing alternative geochronometers for young basalts. Several of these alternatives have been based on the (U-Th)/He method because the high production rate of ^{4}He by radioactive decay of U, Th, and (to a lesser extent) Sm leads to the accumulation of large and precisely measurable quantities of that isotope, even in very young samples. For example, *Aciego et al.* [2003, 2007, 2010], *Blackburn et al.* [2007], and *Min et al.* [2006] demonstrated that low-U + Th phenocrysts such as garnet, magnetite, olivine, and pyroxene can yield reliable dates for young volcanic rocks, although these techniques require relatively large sample aliquots and extensive sample preparation in order to minimize the effects of recoil implantation of ^{4}He from surrounding higher U + Th material. *Blondes et al.* [2007] employed a different tactic, dating zircons in felsic xenoliths extracted from young basalts and finding that the high temperature of the basaltic magma effectively reset the (U-Th)/He zircon chronometer to the ages of the basalts. We report here on yet another approach that may be widely applicable: the (U-Th)/He dating of reset detrital zircons and apatites in sediments that have been baked by overlying basalt flows.

2. Conceptual Basis

[3] The short-term thermal structure of a substrate beneath a volcanic flow is described adequately for our purposes by the well-established mathematics of one-dimensional heat conduction [e.g., *Jaeger*, 1968]. Curves in Figure 1 illustrate the hypothetical temperature profiles beneath a 7 m thick, 1150°C lava flow that has flowed continuously over its substrate for a period of a day and a week. (For simplicity, we assumed no heat production within the substrate and an initial temperature of 0°C.) For the present study, we are particularly interested in the thermal structure that would be established in a substrate of unconsolidated fluvial sediments, so these curves were constructed assuming a reasonable thermal diffusivity for dry sand (1.8 × 10^{-7} m^2/s) [*Bristow et al.*, 1994].

[4] The (U-Th)/He systematics of detrital apatite and zircon crystals in a sedimentary substrate can be completely reset if the crystals are subjected to temperatures high enough and long enough for bulk diffusive loss of previously accumulated ^{4}He. As shown by *Gardés and Montel* [2009], the effective bulk resetting temperature (T_{rs}) of a (U-Th)/He thermochronometer is a function of the diffusion parameters for ^{4}He in the mineral of interest, the effective diffusion dimension (or half-grain size), an assumed diffusion geometry, and an assumed heating rate. Also shown in Figure 1 are (U-Th)/He T_{rs} values for 50 μm and 100 μm half-grain sizes of zircon (a_Z) and apatite (a_A) given the conductive heating rates caused by the overlying flow after a day or a week. After a day of continuous flow of the overlying basalt, detrital apatites with a_A = 50–100 μm would be expected to completely reset in the sedimentary substrate within about 15 cm of the basalt contact, whereas detrital zircons of the same sizes would be reset within about 7 cm of the contact. Longer durations of flow would produce resetting at greater depths; after a week, 50–100 μm apatites and zircons would be reset down to about 45 and 25 cm, respectively. After a month of continuous flow, all (U-Th)/He dates obtained from a_A or a_Z = 50–100 μm apatites and zircons in sediments up to 50 cm below the basalt would be completely reset. The downward extent of resetting for all three scenarios would increase if the sediments were wet, and consequently had higher

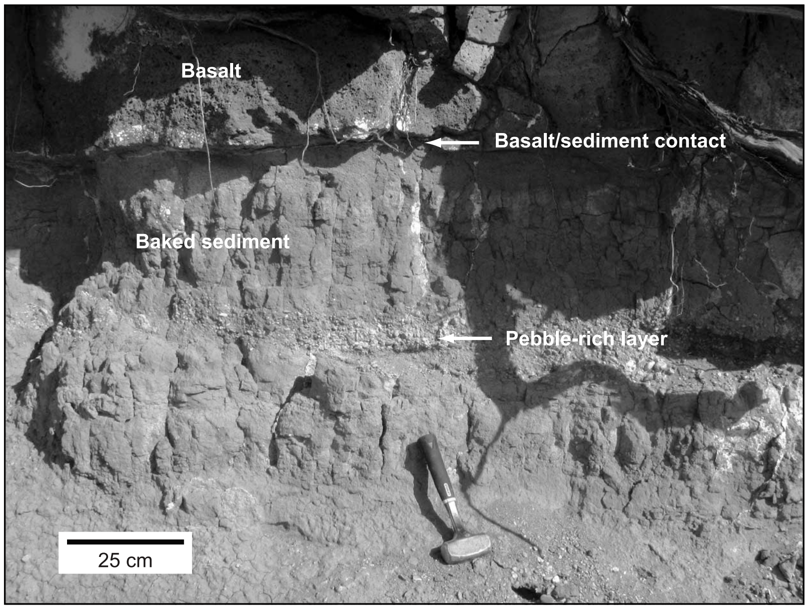

Figure 2. Contact between vesicular basalt flow above and baked fluvial sediments below. The basalt shows a chilled margin but is not glassy. Sample FT05 was collected from the upper 2 cm of sediment beneath the flow here.

thermal diffusivity, or if convective processes played a significant role in the transfer of heat. Zircon and apatite crystals that might be unusually retentive of radiogenic ^{4}He, for example, those displaying significant radiation damage [*Shuster et al.*, 2006], may only be reset in close proximity to the basalt contact. Regardless, it seems likely that detrital mineral (U-Th)/He geochronology of sediments a few centimeters beneath a basalt flow of sufficient thickness (a few meters or more) and having a sufficient duration of activity (a day or more) should yield reset ages equivalent to the eruptive age of the flow.

3. Proof of Concept

[5] Encouraged by our numerical experiments, we conducted a proof-of-concept study in the Rio Grande Rift near Taos, New Mexico, where the Rio Grande gorge presents spectacular exposures of the volcanic and sedimentary stratigraphy of the Pliocene Taos Plateau Volcanic Field (TPVF). The dominant eruptive lithology of the TPVF is the Servilleta Basalt, a sequence of voluminous (>200 km^3), low- to medium-K_2O tholeiitic lavas. The Servilleta Basalt is informally divided into three members, lower, middle, and upper, each of which comprises numerous 1–15 m thick pahoehoe flows [*Dungan et al.*, 1986]. These are interbedded with and underlain by laterally extensive and locally thick fluvial and alluvial fan sediments described as either the Pliocene Cieneguilla Member of the Santa Fe Group [*Lipman and Mehnert*, 1975, 1979; *Dungan et al.*, 1984, 1986] or the Pliocene Servilleta Formation [*Kelson et al.*, 2008].

[6] Emplacement of each of the three Servilleta Basalt members likely occurred rapidly relative to the intervening periods of inactivity, with major eruptive episodes lasting several hundred years to produce several individual flows. The intervening periods of inactivity allowed sediment to accumulate before onset of the next eruptive episode [*Dungan et al.*, 1986]. We know of no published estimates for the duration of activity of individual Servilleta Basalt flows, but studies of other flow fields suggest durations for comparable flows of weeks to months [e.g., *Hon et al.*, 1994; *Self et al.*, 1997].

[7] For our study, we collected two samples of baked sediment from a fluvial channel beneath a 7 m thick flow of the Upper Member of the Servilleta Basalt (geographic coordinates: 36.50978°N; 105.71983°W). In this area, the flow represents the basal flow of the Upper Member. Both samples (FT05 and FT15) were collected immediately beneath the flow, within the upper 2 cm of baked sediment. Sample FT15 comprises well-sorted sand, whereas sample FT05 was collected from the same stratigraphic level but from a more poorly sorted facies a short distance away (Figure 2).

[8] Although the flow directly above these samples has not been dated, *Appelt* [1998] reported $^{40}Ar/^{39}Ar$ ages derived from total fusion of groundmass concentrates for Upper Member Servilleta flows along the Rio Grande Gorge ranging from 2.81 ± 0.26 Ma to 3.72 ± 0.22 Ma (2σ). Three flows near the base of the section yielded statistically indistinguishable dates with an error-weighted mean of 3.61 ± 0.13 Ma (2 SE; MSWD, or Mean Squared Weighted Deviation = 0.77). Based on reasonable correlations along strike from our study location, we anticipated that the flow we intended to date indirectly would be of approximately this age.

4. Methods

[9] In order to avoid selecting grains that may have been thermally shielded within pebbles, particularly in sample FT05, the samples were not crushed. Instead, each sample was placed in a 1 L beaker containing Milli-Q 18.2 MegaOhm polished water and ultrasonicated for ~30 min until the sediment was completely disaggregated. Zircon and apatite grains were then separated using conventional sieving, magnetic and heavy liquid mineral separation techniques. A total of 19 crystals from the two samples were handpicked and dated by the (U-Th)/He method: 4 zircons and 5 apatites from FT15, and five crystals of each mineral from FT05. Grains were selected on the basis of size, euhedral habit, clarity, apparent lack of inclusions (in the case of apatite), and the presence of as few inclusions as possible (in the case of zircon). Helium isotopic analyses of individual grains were accomplished by diode laser gas extraction and quadrupole mass spectrometry in the Noble Gas, Geochronology, and Geochemistry Laboratories (NG^3L) at Arizona State University (ASU). U and Th measurements involved inductively coupled plasma-source mass spectrometry (ICPMS) on dissolved samples in the W. M. Keck Foundation Laboratory for Environmental Geochemistry at ASU. (U-Th)/He dates calculated from the measurements were then corrected for alpha particle ejection using previously measured grain dimensions and the correction algorithms of *Farley et al.* [1996] for apatite and *Hourigan et al.* [2005] for zircon. More complete descriptions of the analytical and data reduction procedures used in the ASU laboratories are given by *Schildgen et al.* [2009a, 2009b] and in the auxiliary material.[1] Results are shown in Table 1. Dates for individual single-crystal analyses are quoted in the text and Table 1 (and illustrated in Figure 3) at the 2σ uncertainty level. Error-weighted means for groups of analyses are reported as two standard errors of the mean (2 SE).

[1]Auxiliary materials are available in the HTML. doi:10.1029/2011GC003650.

5. Results and Interpretations

[10] All nineteen detrital grains yielded (U-Th)/He dates that are consistent with their having been fully reset to a single age by emplacement of the overlying flow (Figure 3 and Table 1). Zircon dates for FT05 ranged from 3.28 ± 0.14 Ma to 3.99 ± 0.12 Ma, whereas apatites from the same sample yielded dates between 3.09 ± 0.19 Ma and 3.94 ± 0.29 Ma. For FT15, zircons ranged from 2.99 ± 0.11 to 4.08 ± 0.13 Ma and apatites from 3.28 ± 0.20 to 3.97 ± 0.41 Ma. Taking analytical uncertainties into consideration for our data as well as those of *Appelt* [1998], all FT05 and FT15 (U-Th)/He dates lie within the range in $^{40}Ar/^{39}Ar$ dates for the Upper Member of the Servilleta Basalt. Since we expect all zircon and apatite dates to reflect complete resetting of the (U-Th)/He chronometer to the age of the overlying flow, we elected to treat them as a single population. We calculated an error-weighted mean date for all nineteen grains and employed the common practice of multiplying the propagated analytical uncertainty on the weighted mean by the square root of the mean squared weighted deviation (MSWD) [e.g., *Wendt and Carl*, 1991]. This gives us an error-weighted mean date of 3.49 ± 0.21 Ma (2σ, MSWD = 17.8) or, more appropriately, 3.487 ± 0.047 Ma using 2 standard errors of the mean. We regard this as the best (U-Th)/He estimate for the eruptive age of the overlying basalt flow.

[11] Our error-weighted mean (U-Th)/He age is within uncertainty of the error-weighted mean $^{40}Ar/^{39}Ar$ age for basal Upper Member Servilleta Basalt flows dated by *Appelt* [1998]. The precision on our individual zircon and apatite dates generally exceeds that of the $^{40}Ar/^{39}Ar$ groundmass dates reported by *Appelt* [1998], although the greater dispersion in our data gives rise to total errors that are similar for the two data sets. We note that a few of the single crystal zircon and apatite dates are slightly older or slightly younger than would be expected based on analytical precision alone (Table 1). Excess scatter of this sort is frequently observed in (U-Th)/He data sets and has several possible explanations. Undetected microinclusions of zircon in apatite could lead to erroneously old dates because the protocols used for preparing apatites for

Table 1. Zircon and Apatite (U-Th)/He Data

Sample	[^{4}He][a] (fmol)	1σ (fmol)	[^{238}U][a] (fmol)	1σ (fmol)	[^{232}Th][a] (fmol)	1σ (fmol)	Th/U[b]	Raw Age[c] (Ma)	1σ[d] (Ma)	R1[e] (μm)	R2[e] (μm)	L[e] (μm)	T1[e] (μm)	T2[e] (μm)	F_T,[f] Mean	Corr. Age[g] (Ma)	2σ[d] (Ma)	^{4}He F_T[h] (fmol)	1σ (fmol)
								FT05 Apatite											
a001	0.592	0.013	176.5	6.1	238.3	5.9	1.35	1.99	0.07		35.3	134	–	–	0.62	3.22	0.23	0.958	0.021
a002	0.785	0.016	219.5	6.6	392.7	6.1	1.79	1.97	0.06		36.6	171	–	–	0.64	3.09	0.19	1.228	0.025
a003	0.451	0.012	96.2	3.7	222.8	2.4	2.32	2.38	0.09		33.0	155	–	–	0.60	3.94	0.29	0.747	0.020
a004	0.529	0.012	96.8	4.8	414.8	6.9	4.28	2.14	0.07		38.2	187	–	–	0.65	3.31	0.23	0.818	0.019
a005	0.226	0.011	46.9	1.8	142.3	3.3	3.03	2.21	0.12		32.9	98	–	–	0.57	3.88	0.42	0.397	0.019
								FT15 Apatite											
a001	0.490	0.013	78.2	2.6	314.6	4.9	4.02	2.53	0.08		42.4	171	–	–	0.67	3.78	0.25	0.733	0.020
a002	0.453	0.013	101.5	1.5	324.9	3.9	3.20	2.00	0.06		39.9	132	–	–	0.64	3.12	0.19	0.706	0.020
a003	0.684	0.013	139.8	2.6	524.4	7.2	3.75	2.04	0.05		37.4	126	–	–	0.62	3.29	0.15	1.103	0.022
a004	0.491	0.013	77.5	1.5	372.0	4.1	4.80	2.34	0.06		32.7	143	–	–	0.59	3.98	0.22	0.835	0.021
a005	0.165	0.0077	27.8	1.1	117.0	1.9	4.21	2.34	0.12		41.6	179	–	–	0.67	3.51	0.36	0.247	0.012
								FT05 Zircon											
z001	20.18	0.25	5401	65	3614	55	0.67	2.52	0.04	36.7	36.9	209	38.9	30.5	0.74	3.41	0.11	27.26	0.33
z002	11.55	0.14	3221	42	2278	32	0.71	2.40	0.04	30.6	30.1	150	33.0	27.2	0.68	3.53	0.12	16.97	0.21
z003	14.43	0.16	3847	47	2479	34	0.64	2.55	0.04	39.5	31.8	184	43.7	40.4	0.72	3.53	0.11	20.02	0.23
z004	44.13	0.53	9162	109	12400	171	1.35	2.86	0.04	38.6	33.9	168	34.0	49.1	0.72	3.99	0.12	61.61	0.74
z005	7.235	0.093	2158	33	1984	119	0.92	2.16	0.05	26.5	28.6	172	30.2	45.3	0.66	3.28	0.14	11.02	0.14
								FT15 Zircon											
z001	4.107	0.060	1438	18	1349	25	0.94	1.83	0.03	25.6	23.9	129	29.0	33.8	0.61	2.99	0.11	6.717	0.098
z002	12.70	0.16	3255	38	2635	32	0.81	2.56	0.04	36.1	35.3	145	40.6	28.7	0.71	3.61	0.12	17.88	0.22
z003	7.187	0.092	1847	21	1411	27	0.76	2.58	0.04	26.0	24.9	143	28.3	26.3	0.63	4.08	0.13	11.37	0.15
z004	4.283	0.059	1331	21	1143	18	0.86	2.09	0.04	28.5	29.2	121	30.9	42.3	0.64	3.28	0.13	6.705	0.092

[a]Absolute measured ^{4}He, ^{238}U, and ^{232}Th concentrations used to calculate the "raw age," which was not corrected for the effects of ^{4}He loss due to alpha particle recoil.

[b]The Th/U ratio of the analyzed crystal. For this calculation the ^{235}U has been accounted for by dividing the measured ^{238}U by 137.88.

[c]The "raw age" was calculated with an iterative approach to solving the age equation.

[d]Based on propagated analytical uncertainties.

[e]R1 and R2 describe the perpendicular half widths of the zircon crystal and in the case of apatite R2 describes the average radius measured in at least two directions perpendicular to the c axis of the crystal. L describes the total length of the zircon or apatite crystal, and T1 and T2 describe the height of the pyramidal terminations of the zircon crystals.

[f]The mean F_T correction calculated following *Farley et al.* [1996] for apatite and *Hourigan et al.* [2005] for zircon.

[g]The F_T corrected age of the crystal. The F_T correction was applied to the raw age following *Farley et al.* [1996].

[h]The F_T corrected [^{4}He] in femtomoles; since individual crystals were not weighed prior to analysis, this number was calculated using the respective specific densities for apatite (3.20 g/cm^3) and zircon (4.65 g/cm^3) and volume calculations based on a hexagonal prism geometry for apatite and for zircon the bipyramidal prism geometry from *Hourigan et al.* [2005].

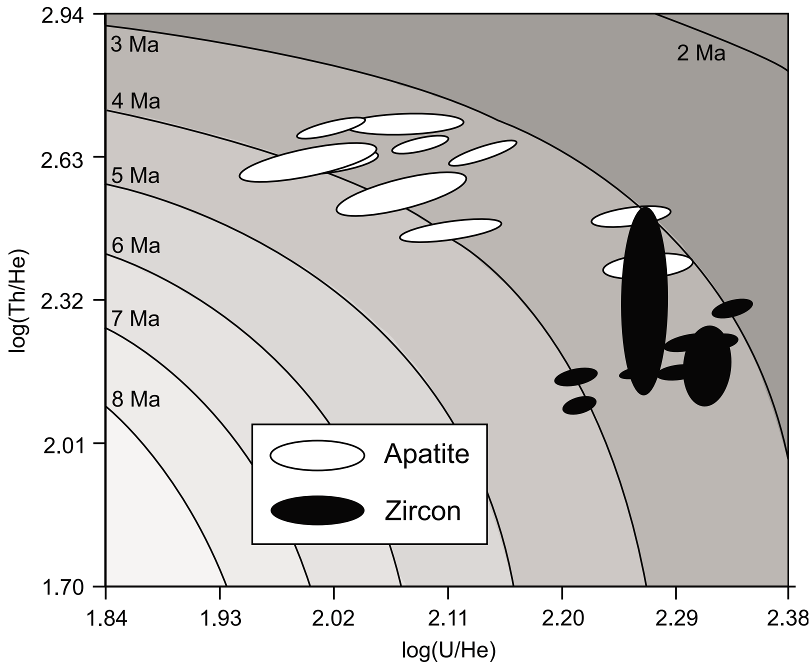

Figure 3. Log ratio plot showing individual (U-Th)/He dates as 2σ error ellipses for the individual apatite (white) and zircon (black) crystals from both samples. The plot was made with Helioplot [*Vermeesch*, 2010] (http://pvermees.andropov.org/helioplot/). Together, the data yield an error-weighted mean date of 3.487 ± 0.047 Ma (2 SE confidence level), which we interpret as the eruptive age of the overlying basalt flow.

U+Th ICPMS analysis would not dissolve zircon in the analyte and thus U and Th concentrations would be underestimated. However, unless the concentrations of U and Th in the microinclusions are particularly high, the large disparity in volume between the grain and the microinclusions should result in a minimal effect on the (U-Th)/He date [*Vermeesch et al.*, 2007]. Additionally, incomplete or variable resetting of apatite and zircon can result from crystal radiation damage. This has been shown to affect He diffusivity in both apatite [e.g., *Shuster et al.*, 2006; *Flowers et al.*, 2009; *Gautheron et al.*, 2009], and zircon [e.g., *Nasdala et al.*, 2004], but in an opposite manner (zircons become less retentive with radiation damage while apatites become more retentive). However, this would lead to preferentially older apatite dates and younger zircon dates, a pattern that is not observed in our data set. Younger lava flows in the Taos field may have reheated the FT05 and FT15 samples sufficiently to cause slight ^{4}He loss after emplacement of the overlying flow. The degree of such partial resetting could vary from grain to grain if the grains are variably retentive of radiogenic ^{4}He. However, we think the most likely reason for dispersion in this and many other (U-Th)/He data sets is grain-specific undercorrection or overcorrection for alpha ejection due to a lack of understanding of the degree and character of U and Th zoning in individual crystals [cf. *Hourigan et al.*, 2005]. Grain-to-grain variations in U and Th zoning tend to be more significant in detrital populations, and thus we may expect greater zoning-related apparent age dispersions for the results of detrital (U-Th)/He studies, including those aimed at dating overlying volcanic flows.

6. Discussion

[12] Despite such complications, our results overall suggest that the (U-Th)/He method can be applied successfully to date young basalts when the upper few centimeters of baked sediment directly beneath the lava flow is selected for study. This method could be particularly powerful for dating Pleistocene volcanism because, compared with ^{40}K to ^{40}Ar decay, a much larger number of radiogenic ^{4}He isotopes is produced for every radioactive parent isotope decay. For example, given the analytical capabilities of most modern (U-Th)/He facilities, it would be possible to date zircons, with U and Th concentrations similar to those encountered in this study, as young as circa 100 ka with 2σ uncertainties of 5%–10%. Even younger flows could be dated using detrital minerals higher in U and Th or with multigrain rather than single-grain aliquots.

[13] With larger data sets, this method also offers the potential to explore the depth to which the thermal effect of the lava flow continues down into the sediment below. If the depth of the transition from reset to unreset thermochronometers can be established, such information has important implications regarding the lava temperature and flow duration. However, care should be taken to fully characterize the history of the grains in the sediment in order to distinguish between partial resetting due to distance from the lava flow and partial resetting due to complications such as those discussed above.

7. Conclusions

[14] Simple heat conduction calculations suggest that lava flows can sufficiently alter the thermal structures of their substrate to cause partial or full resetting of low-temperature (U-Th)/He thermochronometers. Reset single crystal (U-Th)/He dates for detrital zircons and apatites collected from fluvial sediments beneath a flow of the Upper Member of the Servilleta Basalt from the Taos Plateau Volcanic Field confirm that detrital mineral (U-Th)/He geochronology provides a useful tool for dating basaltic flows with precision and accuracy approaching (or, for very young materials, potentially exceeding) those of $^{40}Ar/^{39}Ar$ geochronology. A similar approach should prove equally valuable for dating basaltic flows with bedrock substrates containing apatite and/or zircon.

[15] Our study also suggests that applications of this indirect dating technique will be most successful if detrital minerals are collected very near (≤2 cm) to the sediment-flow interface. Beneath relatively thin flows, temperatures sufficient to fully reset (U-Th)/He chronometers may persist only a few centimeters below the contact, and samples collected from deeper levels in a subjacent fluvial channel consequently may yield partially or unreset grains that are difficult to interpret. Typical U and Th concentrations in detrital apatites and (especially) zircons would permit the use of this technique to date Pleistocene basalt flows with relatively high precision, providing important information regarding patterns of volcanism in the recent geologic past.

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