



New zircon (U-Th)/He and U/Pb eruption age for the Rockland tephra, western USA



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ABSTRACT

Eruption ages of a number of prominent Quaternary volcanic deposits remain inaccurately and/or imprecisely constrained, despite their importance as regional stratigraphic markers in paleo-environment reconstruction and as evidence of climate-altering eruptions. Accurately dating volcanic deposits presents challenging analytical considerations, including poor radiogenic yield, scarcity of datable minerals, and contamination of crystal populations by magma, eruption, and transport processes. One prominent example is the Rockland tephra, which erupted from the Lassen Volcanic Center in the southern Cascade arc. Despite a range in published eruption ages from 0.40 to 0.63 Ma, the Rockland tephra is extensively used as a marker bed across the western United States. To more accurately and precisely constrain the age of the Rockland tephra-producing eruption, we report U/Pb crystallization dates from the outermost $\sim 2 \mu\text{m}$ of zircon crystal faces (surfaces) using secondary ion mass spectrometry (SIMS). Our new weighted mean $^{238}\text{U}/^{206}\text{Pb}$ age for Rockland tephra zircon surfaces is $0.598 \pm 0.013 \text{ Ma}$ (2σ) and MSWD = 1.11 (mean square weighted deviation). As an independent test of the accuracy of this age, we obtained new (U-Th)/He dates from individual zircon grains from the Rockland tephra, which yielded a weighted mean age of $0.599 \pm 0.012 \text{ Ma}$ (2σ , MSWD = 5.13). We also obtained a (U-Th)/He age of $0.628 \pm 0.014 \text{ Ma}$ (MSWD = 1.19) for the Lava Creek Tuff member B, which was analyzed as a secondary standard to test the accuracy of the (U-Th)/He technique for Quaternary tephra, and to evaluate assumptions made in the model-age calculation. Concordance of new U/Pb and (U-Th)/He zircon ages reinforces the accuracy of our preferred Rockland tephra eruption age, and confirms that zircon surface dates sample zircon growth up to the time of eruption. We demonstrate the broad applicability of coupled U/Pb zircon-surface and single-grain zircon (U-Th)/He geochronology to accurate dating of Quaternary tephra, and highlight the challenges and opportunities of this technique.

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1. Introduction

Quaternary and older volcanic eruptions and associated tephra deposits define isochronous stratigraphic markers over broad regions, and are used extensively to link geological sequences to radiometric dates. Tephrochronology requires accurate dating and correlation of geographically discontinuous tephra horizons, which is critically important for paleoclimate, palaeoecological, volcanic hazard, and stratigraphic studies (e.g., Mulch et al., 2008; Dean et al., 2015; Maier et al., 2015). Although tephrochronology is

often cited to offer high temporal precision (e.g., Lowe, 2011), the confidence of any correlation is dependent on the accuracy and precision of the radiometric date(s) on which the correlation is based. Obtaining accurate and precise radiometric ages for volcanic eruptions associated with Quaternary tephra remains a major analytical challenge in geochronology (e.g., Danišik et al., 2017). Intermediate to silicic calc-alkaline melts characteristic of subduction settings are particularly challenging to date using K-Ar and $^{40}\text{Ar}/^{39}\text{Ar}$ methods due to their characteristically low-K plagioclase phenocrysts and the frequent absence of high-K phases such as sanidine that can be reliably dated. Similarly, while zircon is a nearly ubiquitous accessory phase in calc-alkaline arc magmas, it generally has relatively low uranium content (typically <500 ppm U, although large compositional diversity has been observed within

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individual grains and crystal populations; [Claiborne, 2011](#); [Klemetti and Clynne, 2014](#)). Moreover, zircons often exhibit heterotemporal crystallization histories over 10^3 – 10^6 yr intervals that reflect the duration of magma chamber assembly and/or assimilation processes (e.g., [Simon et al., 2008](#); [Schmitt et al., 2010](#); [Klemetti and Clynne, 2014](#)).

In an effort to accurately determine eruption ages for zircon extracted from Quaternary tephra, we measured U/Pb isotopes using SIMS by sputtering through the outermost ~ 2 μm of zircon prism faces. Based on the notion that zircon in calc-alkaline magmas reaches saturation at intermediate temperatures between the liquidus and solidus (e.g., [Watson and Harrison, 1983](#); [Fu et al., 2008](#); [Boehnke et al., 2013](#)), and thus continues to crystallize in an evolved rhyolitic magma, measuring zircon crystal faces has the highest likelihood of sampling the final stage of zircon crystallization that most immediately precedes eruption. Recent experimental results indicate that the outer 2 μm of zircon growth can represent a crystallization interval as short as ca. 2–20 years ([Zhang and Xu, 2016](#)). Thus, SIMS measurements on zircon surfaces may provide a final snapshot of the crystallization history, whereas analyses of polished zircon interiors are more likely to sample material crystallized during protracted zircon growth within a magma system (e.g., [Simon et al., 2008](#)). Complementing zircon U/Pb crystallization ages measured on crystal faces, the (U–Th)/He geochronometer applied to single zircon crystals dates the timing of zircon closure to the diffusive loss of radiogenic He, which occurs at temperatures below ~ 200 °C ([Reiners, 2005](#)). Thus, whereas U/Pb surface analysis captures the timing of final zircon crystallization and therefore represents a maximum estimate for the eruption age, (U–Th)/He analysis captures the timing of zircon cooling upon eruption if undisturbed by subsequent heating events. Coupled, these two chronometers offer the ability to accurately determine the eruption age of Quaternary and older tephra ([Danišik et al., 2017](#)). The zircon (U–Th)/He technique also has the advantage that pre-eruptive zircon ages will be reset at magmatic temperatures, which can provide a consistency check for silicic systems where the U/Pb or U–Th crystallization ages from zircon rim significantly predate the eruption (e.g., [Klemetti and Clynne, 2014](#); [Barboni et al., 2016](#)).

Distal deposits of the Rockland tephra have been recognized throughout the western United States ([Fig. 1](#)), including in offshore sediment cores ([Sarna-Wojcicki et al., 1987](#)), making the Rockland tephra an important chronostratigraphic marker for correlating Quaternary sequences. The Rockland tephra was the product of the largest eruption of the Lassen Volcanic Center (LVC) ([Fig. 1](#)). Identification of the Rockland tephra has been used to interpolate ages for undated tephra and sedimentary packages based on sediment accumulation-rate curves (e.g., [Sarna-Wojcicki et al., 1987, 1991](#)). These curves are generally constructed by interpolation between radiometric ages of the Rockland tephra and/or other dated tephra(s), augmented with magnetostratigraphic, biostratigraphic, or other age constraints. Thus, the Rockland tephra age is critical in Quaternary stratigraphic interpretations, including investigation of tephra and climate records at International Ocean Discovery Program (IODP) and Deep Sea Drilling Project (DSDP) sites in the northeast Pacific Ocean (e.g., [Sarna-Wojcicki et al., 1987](#)), lacustrine sediments at Tulelake, northern California ([Sarna-Wojcicki et al., 1988, 1991](#); [Adam et al., 1989](#); [Rieck et al., 1992](#)), and fluvial sedimentation rates in the Sacramento–San Joaquin Delta, California ([Maier et al., 2015](#)). Each of these studies used different model ages for the Rockland tephra, ranging from 0.400 to 0.575 Ma, to calculate sedimentation-rate curves, highlighting inconsistency in the literature about the eruption age of the Rockland tephra and difficulty in correlating between different datasets.

Radiometric dating and correlation of the Rockland tephra has

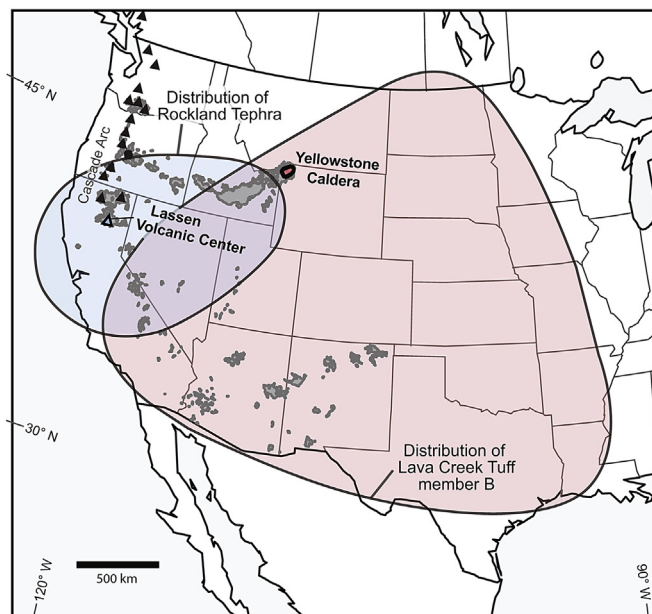


Fig. 1. Map of known distributions of the Rockland tephra and Lava Creek Tuff member B, modified from [Lanphere et al. \(2004\)](#) and [Matthews et al. \(2015\)](#). Shown in gray is the distribution of Quaternary volcanic rocks and the modern Cascade Arc volcanoes (triangles), highlighting the areas of recent volcanic activity and potential volcanic hazards.

been the focus of a number of studies over the past five decades ([Gilbert, 1969](#); [Meyer et al., 1980, 1991](#); [Sarna-Wojcicki et al., 1985, 1987, 1988](#), [Sarna-Wojcicki, 2000](#); [Berger, 1992](#); [Alloway et al., 1992](#); [Lanphere et al., 1999, 2004](#)). However, the Rockland tephra has been notoriously difficult to date via radiometric methods, due in part to the prevalence of contaminants, notably lithic rock fragments and crystals incorporated during eruption, xenocrysts assimilated from country rock during magma genesis, and crystal contamination during reworking. As a result, interpretation of the eruption age of the Rockland tephra ([Fig. 2](#)) has been controversial (e.g., [Lanphere et al., 1999](#); c.f., [Sarna-Wojcicki, 2000](#)). Widespread use of unpublished ages based on personal communications or tephrostratigraphic ages have further complicated interpretation of the Rockland tephra eruption age (e.g., [Mulch et al., 2008](#); [Maier et al., 2015](#)).

To more accurately and precisely constrain the timing of the Rockland tephra-producing eruption, we applied coupled U/Pb and (U–Th)/He zircon geochronometry on two proximal Rockland tephra samples. We also analyzed the Lava Creek Tuff member B as a secondary standard of known age, which erupted from Yellowstone caldera at ~ 0.63 Ma ([Fig. 1](#)), and is the youngest “super-eruption” sourced from calderas defining the Snake River Plain–Yellowstone hotspot trend (e.g., [Pierce and Morgan, 1992](#); [Matthews et al., 2015](#)). The Rockland tephra is stratigraphically younger than the Lava Creek Tuff (e.g., [Sarna-Wojcicki et al., 1991](#)), and they can occur together in the same stratigraphic successions ([Fig. 1](#)).

2. Geologic background

2.1. Previous studies of Rockland tephra

The Rockland tephra erupted from the Rockland caldera complex, part of the LVC ([Fig. 1](#)), one of only three Quaternary calderas identified in the Cascade arc ([Hildreth, 2007](#)). It represents the

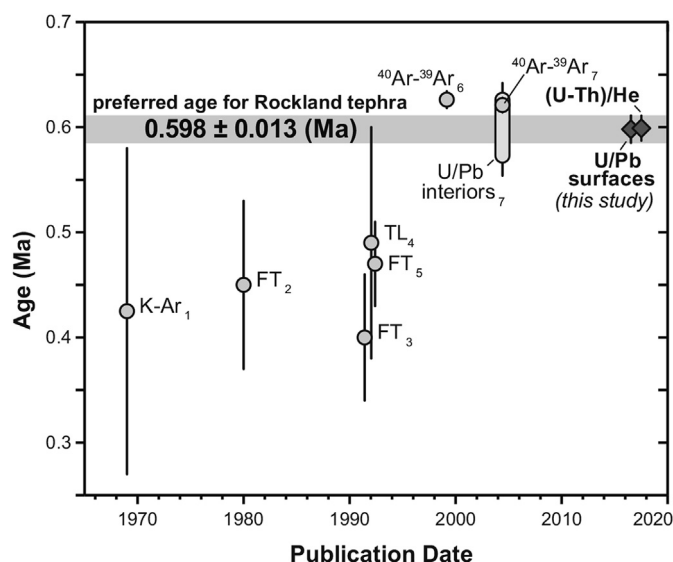


Fig. 2. Age of the Rockland tephra versus publication date, highlighting the range of published ages for this regional tephra. New U/Pb and (U-Th)/He zircon ages from this study shown as gray bar (2σ uncertainty). Data from (1) Gilbert (1969); (2) Meyer et al. (1980); (3) Meyer et al. (1991); (4) Berger (1992); (5) Alloway et al. (1992); (6) Lanphere et al. (1999); (7) Lanphere et al. (2004). Previous U/Pb zircon ages are shown as a range from the weighted mean for all grains and the youngest subset (see text). Method abbreviations: FT = fission-track; TL = additive dose thermoluminescence. Unpublished ages previously reported as personal communication are excluded.

largest eruption in the ~0.825 Ma history of the LVC (Clynne and Muffler, 2010), with an estimated erupted volume between 50 km^3 (Sarna-Wojcicki et al., 1985) and 248 km^3 (Pouget et al., 2014). The Rockland tephra ranges from rhyodacite to rhyolite (69–75 wt% SiO_2), with a mineral assemblage dominated by plagioclase, and minor amounts of hornblende, hypersthene, and quartz (Clynne and Muffler, 2010).

Previously published $^{40}\text{Ar}/^{39}\text{Ar}$ dates (shown in angle brackets below) are recalculated to Fish Canyon sanidine fluence monitor age of 28.172 Ma (Rivera et al., 2011; $R_{\text{FC}}^{\text{CS}} = 1.0112 \pm 0.001$ from Renne et al., 1998; Coble et al., 2011) to be consistent with recent published $^{40}\text{Ar}/^{39}\text{Ar}$ ages for Lava Creek Tuff (Matthews et al., 2015). Two aliquots of plagioclase from a proximal pumice sample of Rockland tephra near the quarry at Manton, California were analyzed by Lanphere et al. (1999) yielding plateau ages of 0.619 ± 0.009 (607) Ma and 0.635 ± 0.009 (622) Ma, with a pooled age of 0.626 ± 0.008 (614) Ma. Lanphere et al. (2004) resampled and analyzed plagioclase separated from pumice collected at the same locality (sample RPT25), yielding a $^{40}\text{Ar}/^{39}\text{Ar}$ plateau age of 0.599 ± 0.012 (587) Ma. Based on the results from both studies, Lanphere et al. (2004) interpreted a pooled age of 0.621 ± 0.007 (609) Ma (Fig. 2) as the best estimate for the eruption. Note that the originally published $^{40}\text{Ar}/^{39}\text{Ar}$ ages increase on average by $\Delta +0.012$ Ma (or 2%) due to recalculated of fluence monitor, which is greater than the quoted 2σ analytical uncertainties for individual or pooled ages, but is within the $\text{ca.} \pm 0.03$ Ma (~5%) uncertainty if error in the ^{40}K decay constants and the irradiation parameter J are fully propagated.

In addition to plagioclase $^{40}\text{Ar}/^{39}\text{Ar}$ dates, Lanphere et al. (2004) also obtained zircon U/Pb dates measured by SIMS using the SHRIMP-RG (sensitive high-resolution ion microprobe with reverse geometry) on polished crystals (interiors) separated from the same proximal Rockland tephra samples. Individual U/Pb analyses range from 0.45 to 0.81 Ma, yielding a weighted mean age of 0.626 ± 0.016 Ma for all grains (MSWD = 3.2, $n = 33$) whose

elevated MSWD indicate the presence of multiple population and/or protracted crystallization (Lanphere et al., 2004). A subset of 17 analyses defined a younger population at 0.573 ± 0.019 Ma (MSWD = 1.7) (Fig. 2). An evaluation of the plagioclase $^{40}\text{Ar}/^{39}\text{Ar}$ and zircon U/Pb analyses led Lanphere et al. (2004) to conclude the best estimate for the age of Rockland tephra is in the range of 0.570–0.610 Ma. Thus, a contradiction remains between the $^{40}\text{Ar}/^{39}\text{Ar}$ and U/Pb zircon geochronology (Lanphere et al., 2004) leaving ambiguity as to what eruption age should be assigned to the Rockland tephra and what uncertainty to assign to this age.

The Rockland tephra is found stratigraphically above the Lava Creek Tuff member B, which has been dated at 0.629 ± 0.004 Ma by ID-TIMS on zircon (Wotzlaw et al., 2015), $^{40}\text{Ar}/^{39}\text{Ar}$ in sanidine at 0.631 ± 0.004 Ma and SIMS analyses of zircon crystal faces at 0.634 ± 0.008 ka (Matthews et al., 2015). Both the Rockland and Lava Creek member B tephra occur within the normal polarity Brunhes magnetostratigraphic zone (Meyer et al., 1991).

3. Methods

3.1. Zircon U/Pb measurements and calculated ages

U/Pb surface analyses of zircon were performed on source-proximal Rockland tephra samples RPT25 and LV09. RPT25 is coarse pumice collected from a quarry near Manton, California (40.4369° N , $-121.8621^\circ \text{ W}$). The zircon concentrate analyzed in this work is the same used by Lanphere et al. (2004). This sample was chosen to facilitate an unbiased comparison to previous Rockland zircon analyses. Sample LV09 is pumice lapilli (1–3 cm) collected from the same pumice quarry ~0.7 km east of Manton, California (40.4366° N , $-121.8621^\circ \text{ W}$).

Analyses were performed using the USGS/Stanford SHRIMP-RG operated with a O_2^- primary beam intensity ranging from 6.0 to 7.2 nA to sputter material (~5 ng) from a spot approximately $35 \times 32 \times 2 \text{ } \mu\text{m}$ focused onto euhedral crystal faces of zircons pressed into soft indium metal, following the approach described by Matthews et al. (2015). Calculated model ages for zircon were standardized relative to Temora-2 (416.8 Ma; Black et al., 2004), which were analyzed repeatedly throughout the duration of the analytical session. New $^{238}\text{U}/^{206}\text{Pb}$ dates were corrected for common Pb using measured $^{207}\text{Pb}/^{206}\text{Pb}$ (Ireland and Williams, 2003) assuming common $^{207}\text{Pb}/^{206}\text{Pb} = 0.8357$ (modern value; Stacey and Kramers, 1975) and ^{230}Th disequilibria using the method of Schärer (1984). For Rockland tephra, we used an initial $^{232}\text{Th}/^{238}\text{U}_{\text{melt}}$ value of 3.15 ± 0.15 (1σ) based on a compilation of published U and Th composition for Rockland tephra (see Suppl. Material Table 1) assumed to be in equilibrium with initial $^{230}\text{Th}/^{232}\text{Th}$. The model weighted mean age includes an error term constrained by the external reproducibility of zircon age standard Temora-2.

3.2. Zircon (U-Th)/He measurements and model age parameters

(U-Th)/He dates were measured from individual zircon crystals in the Thermochronology Laboratory at Stanford University. Euhedral Rockland tephra zircon grains were extracted from the indium mounts for sample RPT25 and LV09. Zircon from Lava Creek Tuff member B were taken from the same zircon concentrate analyzed by Matthews et al. (2015). The a_1 , b_2 , and c crystal axes and tip heights were measured using an optical microscope, and individual zircon were encapsulated in niobium (Nb) tubes. Radiogenic ^4He was extracted from individual zircon grains by heating to 900–1100 $^\circ\text{C}$ for 240 s. Three to four isothermal extractions were performed until the cumulative extracted ^4He was >98%. Extracted gas was purified and analyzed using a Nu-Instruments Noblesse operated using a single discrete dynode secondary electron

Table 1
Calculated (U-Th)/He and U/Pb zircon ages from the Rockland tephra and Lava Creek Tuff member B. The combined age is the calculated weighted mean of grains from Rockland tephra samples LV09 and RPT25, and is our preferred age (bold).

Sample	Unit	^{230}Th -corr. U/Pb Surface Age (Ma)	n	MSWD	^{230}Th -corr. (U-Th)/He Age (Ma)	n	MSWD
LV09	Rockland tephra	0.592 ± 0.017	41 of 43	1.19	0.586 ± 0.014	14 of 19	5.30
RPT25		0.607 ± 0.019	19 of 20	0.88	0.646 ± 0.025	7 of 11	2.71
Combined	Rockland tephra	0.598 ± 0.013	60 of 63	1.11	0.599 ± 0.012	21 of 30	5.13
12NMYC04	Lava Creek Tuff member B	0.634 ± 0.008^a	35 of 35	0.73	0.628 ± 0.014	21 of 21	1.19

^a U-Pb age from Matthews et al. (2015) are the weighted mean age of two proximal and one distal samples of Lava Creek Tuff member B.

multiplier operated in pulse-counting mode. The high sensitivity of the Noblesse (Coble et al., 2011) and signal-to-noise ratio of EM detectors make high-precision He measurements of single zircons grains from Quaternary volcanic rocks possible.

Following He extractions, the encapsulating Nb was dissolved in ~80 μL concentrated 50% HF and 50% nitric (HNO_3) acid at room temperature for 30 s. Individual zircons were rinsed with distilled water and hydrothermally dissolved at 220 °C in an 80:20 ratio of concentrated HF and HNO_3 . Parent ^{235}U , ^{238}U and ^{232}Th isotopes were measured by isotope dilution using a Finnigan X-Series ICP-MS (inductively coupled plasma mass spectrometry) at the University of California Santa Cruz Keck laboratory. U and Th concentrations were calculated relative to a ~10 ppb $^{229}\text{Th}/^{236}\text{U}$ spike added during zircon dissolution.

Individual zircon (U-Th)/He ages were calculated following the methods described by Reiners (2005). The accuracy of calculated (U-Th)/He ages is partly dependent upon model assumptions, including crystal geometry and parent isotope distribution, alpha ejection (Ft) correction, initial crystallization age, and degree of initial ^{230}Th disequilibrium. We briefly discuss our effort to quantify each model assumption below, and discuss the impact of these corrections on the calculated age presented in section 5.2.

The surface area and volume for each zircon crystal was calculated using a tetrahedral prism geometry with pyramidal terminations and He retention factors from Hourigan et al. (2005), which best represents the crystal geometry of the Rockland tephra and Lava Creek Tuff member B zircons. The magnitude of the internal compositional zoning for the zircon U and Th parent distribution ($\delta_{\text{R/C}}$) is approximated by the ratio of U concentration of the rim (R), measured during U-Pb surface analyses, relative to core (C) concentration, calculated from whole-grain dissolution (see Supplemental Material). We assume the Th/U is fixed for the entire grains, and use the Th/U based on the solution ICP-MS measurements. The $\delta_{\text{R/C}}$ is calculated for each grain to account for the unique internal composition variation. Samples are corrected for α -ejection using Monte Carlo simulation (FZAC; Hourigan et al., 2005), which incorporates grain dimensions, Th/U, and assumes a step function gradient for the core and rim compositional variation ($\delta_{\text{R/C}}$) and a rim thickness based on a compilation of CL profiles across selected polished zircons aiming to be representative of each sample (Fig. 3). The uncertainty in the α -ejection correction (Ft and FZAC) was calculated assuming a 2% (1σ) uncertainty in grain-dimension measurements, and is fully propagated into calculated ages. Because the zircon CL profiles were measured on a different subset of grains than those analyzed for (U-Th)/He, we used the average CL-defined rim thickness for Rockland tephra (Fig. 3) and Lava Creek Tuff member B, which was estimated to be 10 μm and 8 μm , respectively. We consider the approach of approximating the rim thickness based on CL characteristics to be reasonable based on previous studies (e.g., Chamberlain et al., 2014; Matthews et al., 2015), which document a correlation between zircon age populations and CL zoning textures.

As independent confirmation of a correlation between U content and CL intensity, we calibrated CL brightness by measuring U and Th concentrations on SHRIMP-RG (Fig. 3) using a 12 μm diameter analytical spot to target portions of the polished grain interiors characterized by homogeneous CL intensity over length scales of 15–20 μm . Zircon CL intensity is expected to be inversely correlated with crystal lattice defects attributed to cumulative alpha dose (α -decay events/mg) from radioactive decay of U and Th nuclides (Palenik et al., 2003), although other factors such as instrument conditions or rare earth element content (e.g., Nasdala et al., 2003) may also be important. See Supplemental Material Text for a full discussion of analytical methodology.

The α -ejection corrected (U-Th)/He ages for Rockland tephra were adjusted for initial ^{230}Th excess using the Monte Carlo computational routine MCHCalc (Schmitt et al., 2010) assuming $^{232}\text{Th}/^{238}\text{U}_{\text{melt}} = 3.15 \pm 0.15$, the same values applied to corrections of U/Pb ages, and an initial zircon crystallization age of 0.740 ± 0.040 Ma for Rockland tephra based on the 7 oldest interior ages from Lanphere et al. (2004). For Lava Creek Tuff member B, the same corrections were applied using $^{232}\text{Th}/^{238}\text{U}_{\text{melt}} = 4.7 \pm 0.2$ and initial zircon crystallization age of 0.700 ± 0.025 Ma based on the 5 oldest U/Pb interior ages from Matthews et al. (2015).

4. Results

4.1. $^{238}\text{U}/^{206}\text{Pb}$ zircon surface ages

The calculated weighted mean ^{230}Th -corrected $^{238}\text{U}/^{206}\text{Pb}$ ages for sample RPT25 and LV09 overlap at 2σ confidence, and are 0.607 ± 0.019 (MSWD = 0.88; $n = 19$ of 20) and 0.592 ± 0.017 Ma (MSWD = 1.19; $n = 41$ of 43), respectively (Table 1). Both samples yield reproducible, unimodal populations (Fig. 4). Combining all new zircon surface dates yields a weighted mean age of 0.598 ± 0.013 Ma (MSWD = 1.11; $n = 60$ of 63 Table 1), which we prefer as the eruption age of the Rockland tephra. Three points were excluded from the preferred age due to (1) relatively high common-Pb and being younger than the weighted average (2) >2 standard deviation difference in Th/U relative to sample average, or (3) older ages due to analysis of inherited portions of the grains. The U/Pb measurement conditions, data reduction, common-Pb, and ^{230}Th disequilibria corrections used for Rockland tephra zircon dates were the same as those previously applied by Matthews et al. (2015), who obtained a ^{230}Th -corrected weighted mean $^{238}\text{U}/^{206}\text{Pb}$ age for Lava Creek Tuff member B of 0.634 ± 0.008 Ma (MSWD = 0.73; $n = 35$).

Measured simultaneously with U/Pb ages, U and Th concentration of Rockland tephra zircon surfaces (outermost ~2 μm) from samples RPT25 and LV09 average 177 ± 37 ppm U and 75 ± 27 ppm Th (1σ standard deviation), which is a relatively restricted range compared to analyses of zircon interiors, which range from 90 to 2600 ppm U (Fig. 3; see Supplemental Material Fig. 2). In comparison, Lava Creek Tuff member B zircon surfaces

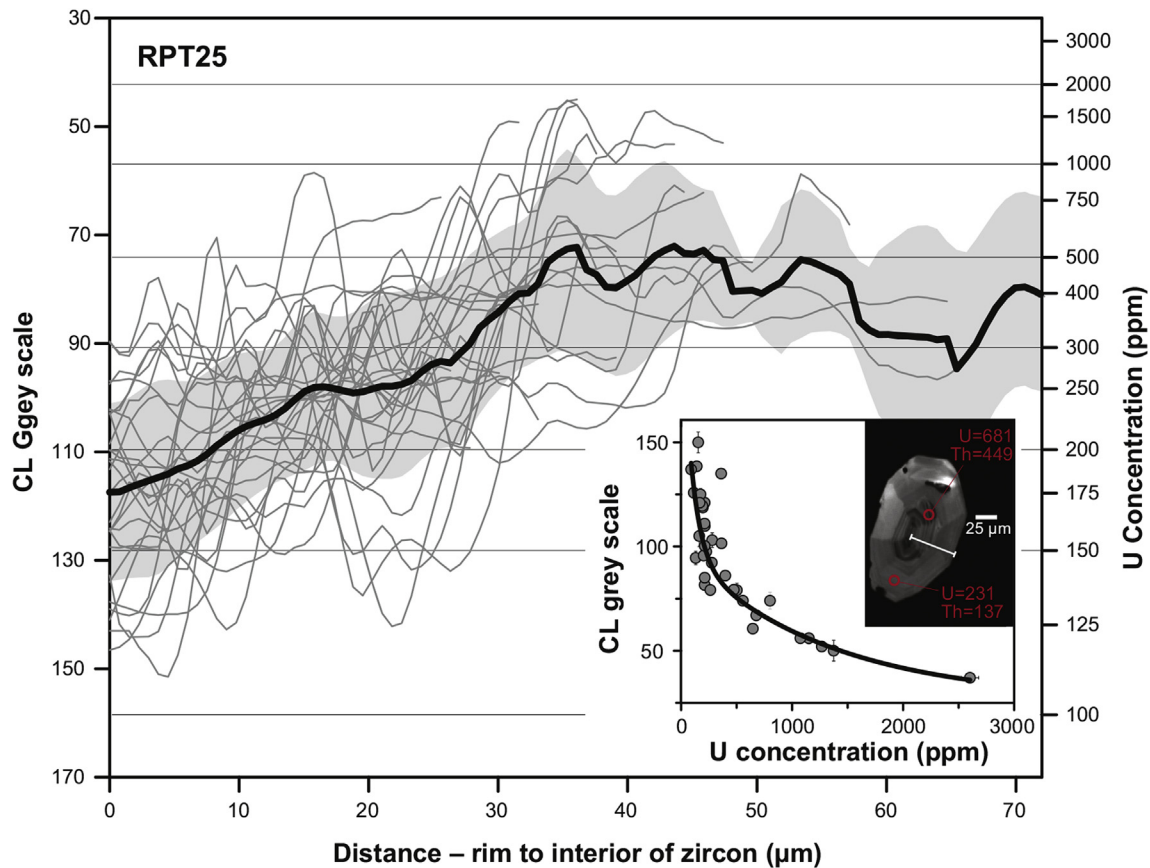
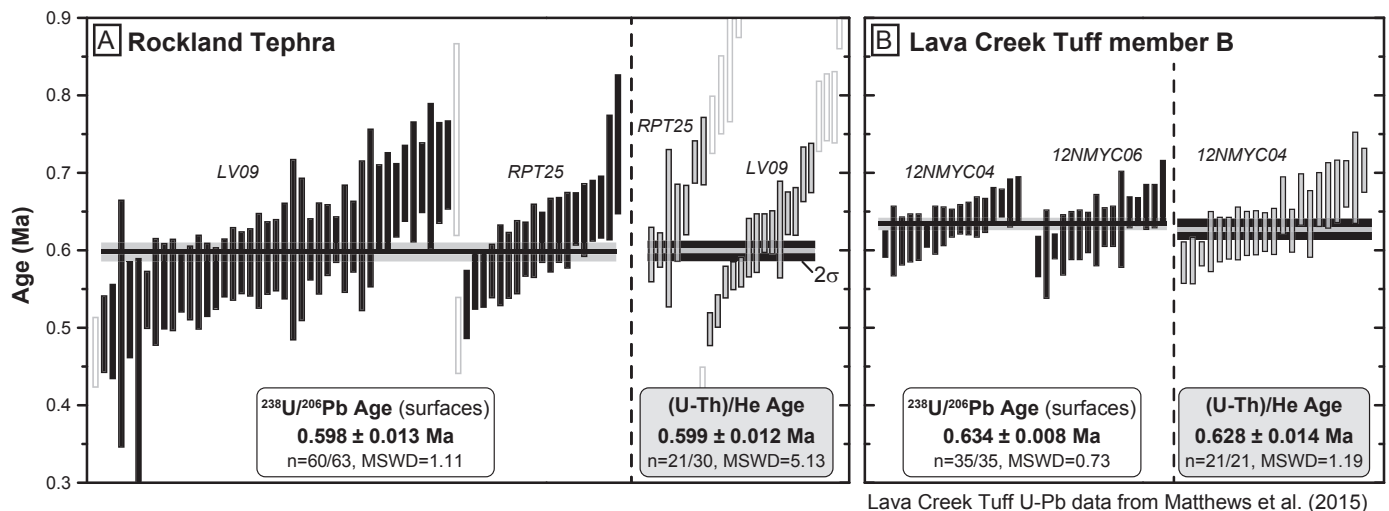


Fig. 3. Plot showing zoning profiles of Rockland tephra zircon for sample RPT25. Each thin gray line is the cathodoluminescence (CL) gray scale intensity profile of an individual zircon measured rim to interior (center) for 29 crystals (resolution was $0.75 \mu\text{m}$). Gray scale intensity increases with increasing brightness. The bold black line and shaded gray area are the average and 1σ standard deviation of these measurements. The inset shows the calibration of CL gray scale intensity to U concentration, based on trace element measurements of RPT25 zircon on SHRIMP-RG from $20 \times 20 \mu\text{m}^2$ areas that were homogeneous in CL intensity. Example of a representative zircon CL image, trace element analytical spots (red), and rim to core profile (white) is also shown. The uranium concentration (ppm) y-axis is based on the inset calibration and shows that RPT25 zircons are generally CL-dark and U-enriched towards their interiors. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)



Lava Creek Tuff U-Pb data from Matthews et al. (2015)

Fig. 4. U/Pb zircon surface dates and single-grain zircon (U-Th)/He dates for (A) Rockland tephra and (B) Lava Creek Tuff member B. Dates for individual analyses are shown at 1σ and are ranked by age. Calculated weighted mean ages and 2σ standard errors (boxes) are shown graphically as horizontal lines and gray boxes; analyses omitted in calculated age are unfilled bars (see text).

(12NMYC04) average 700 ± 146 ppm U (Matthews et al., 2015). Thus, the slightly larger uncertainty in the Rockland tephra age

occurs because Rockland tephra zircon grains contain $\sim 75\%$ less ^{238}U than does Lava Creek Tuff member B zircon grains. A

discussion of the sensitivity of the calculated U/Pb age to model parameters (e.g., initial ^{230}Th disequilibrium, common Pb, etc.) is provided in section 5.1.

4.2. Crystal zonation and (U-Th)/He ages

A compilation of CL zoning profiles from 29 individual RPT25 grains indicates that the majority of Rockland tephra zircons are compositionally zoned, with interiors that are enriched in U (90–2600 ppm) and Th (45–4200 ppm) relative to rims which have an average of 177 ± 37 ppm U (Fig. 3). Sample RPT25 zircons have interiors that are on average 4 times more enriched in U (115–725 ppm) and ~7 times more enriched in Th (45–590 ppm) compared to LV09. This may suggest the composition of zircon changed during the eruption and different deposits preserve slightly different chemical populations. Taking into account a 10 μm rim with 177 ppm U for Rockland tephra zircons, sample RPT25 has $\delta_{\text{R/C}}$ ranging from 0.037 to 0.235, with an average of 0.117, whereas $\delta_{\text{R/C}}$ for LV09 ranges from 0.159 to 3.807, with an average of 1.042 (Supplemental Material Table 2). The $\delta_{\text{R/C}}$ was calculated for each individual grain assuming a 10 μm rim with 177 ppm U for Rockland tephra zircons, and used in the FZAC α -ejection-correction (Hourigan et al., 2005).

The α -ejection- and ^{230}Th -corrected (U-Th)/He weighted mean zircon age for the Rockland tephra sample LV09 is 0.586 ± 0.014 Ma ($n = 14$ of 19; MSWD = 5.30). One younger grain at 0.41 Ma and four older grains ranging from 0.77 to 0.89 Ma were excluded from the calculated age (Fig. 4). The MSWD is higher than expected for a homogeneous population (Mahon, 1996). Older, inherited zircon grains will have lost radiogenic ^4He because magmatic temperatures greatly exceeded the zircon closure temperature of approximately $<200^\circ\text{C}$. Therefore older (U-Th)/He dates and excess scatter is attributed to inaccuracy of model assumptions (i.e., α -ejection- and ^{230}Th -corrections), or underestimation of the uncertainty in the α -ejection correction, which dominates the total uncertainty budget. Although scattered, individual grain dates are reproducible, we interpret the data to represent a single population and argue additional uncertainty based on the higher MSWD is unjustified. The (U-Th)/He dates for sample RPT25 are more scattered than sample LV09, with a population defining an age of 0.646 ± 0.025 Ma ($n = 7$ of 11; MSWD = 2.71), and four older grains ranging from 0.76 Ma to 0.94 Ma that were excluded from the weighted mean calculation (Fig. 4).

Combining the zircon (U-Th)/He results from samples LV09 and RPT25 yields a weighted mean age of 0.599 ± 0.012 Ma ($n = 21$; MSWD 5.13), which overlaps the 0.598 ± 0.013 Ma U/Pb age from the outermost rims of Rockland tephra zircon at 2σ confidence (Table 1).

For Lava Creek Tuff member B zircons, $\delta_{\text{R/C}}$ was calculated for individual grains assuming an 8 μm thick rim with 700 ± 146 ppm U. The $\delta_{\text{R/C}}$ for 12NMYC04 ranges from 0.041 to 0.468, with an average of 0.089. The α -ejection- and ^{230}Th -corrected (U-Th)/He weighted mean age for Lava Creek Tuff member B is 0.628 ± 0.014 Ma ($n = 21$ of 21; MSWD = 1.19) (Fig. 4). The calculated (U-Th)/He age for Lava Creek Tuff is sensitive to the modeled rim thickness utilized in the α -ejection correction due to the fact that the average $\delta_{\text{R/C}}$ for 12NMYC04 corresponds to a ~11 times enrichment of U in the interiors of Lava Creek Tuff member B zircons relative to the rims. The MSWD of the calculated (U-Th)/He age for Lava Creek Tuff member B is reasonable for a homogeneous population (e.g., Mahon, 1996), suggesting sources of external error are not underestimated, compared to the MSWD for the Rockland tephra which suggests more dispersion in individual (U-Th)/He dates. We attribute the low MSWD for the Lava Creek Tuff to the zircon population having

zoning profiles that are very comparable crystal-to-crystal (Matthews et al., 2015), thus the α -ejection correction based on average CL-profile is accurate. In comparison, there is more variability in the Rockland tephra zircon profiles (Fig. 3; Supplemental Material Fig. 1), suggesting the α -ejection correction using an average CL-profile is causing additional scatter.

5. Discussion

5.1. Influence of model parameters on the calculated U/Pb zircon surface ages for Rockland tephra

Our calculated crystallization age for the Rockland tephra is largely insensitive to model assumptions relative to the ± 0.013 Ma uncertainty in the calculated age. The largest correction accounts for initial ^{230}Th disequilibrium, which, using a $^{232}\text{Th}/^{238}\text{U}_{\text{melt}}$ value of 3.15 ± 0.15 (see Supplemental Material Table 1), increases the calculated age by an average of +0.094 Ma. Using the lowest published $^{232}\text{Th}/^{238}\text{U}_{\text{melt}}$ value of 2.98 (Bullen and Clynne, 1990) decreases the calculated age by –0.001 Ma. The second-most impactful correction is for common-Pb, which we assume to be a zero-age composition ($^{207}\text{Pb}/^{206}\text{Pb} = 0.8357$) for anthropogenic-Pb. This composition is chosen due to the likely incorporation of common-Pb resulting from the difficulty of sufficiently cleaning indium-mounted grain surfaces. Lanphere et al. (2004) used a $^{207}\text{Pb}/^{206}\text{Pb} = 0.823$ based on whole rock Pb-isotope values from Bullen and Clynne (1990). Recalculating our new U/Pb ages with the whole rock common-Pb value decreases the calculated weighted mean age by –0.003 Ma. The last major assumption affecting the model age is the assumed age for the primary U/Pb standard. We calibrated measured Pb/U values for Temora-2 to a value of $^{206}\text{Pb}^*/^{238}\text{U} = 0.0668$ (416.8 Ma; Black et al., 2004). Using a more recent age of 418.6 Ma for Temora-2 (Mattinson, 2010) increases the calculated weighted mean age by +0.002 Ma. Finally, although three points were excluded from the preferred age, including all 63 zircon surface analyses yields an age that is < 0.002 Ma older than the preferred age, a change well within the analytical uncertainty.

Although these assumptions and associated corrections are important, each can only change the calculated age by 0.16–0.50%. Therefore, relative to the 2.2% uncertainty in the calculated age of 0.598 ± 0.013 Ma, the overall impact is insignificant. Other factors, such as uncertainty in the U and Th decay constants or ^{231}Pa initial disequilibrium (e.g., Schmitt, 2007), have less impact on the calculated age than those discussed above.

5.2. Sensitivity of the (U-Th)/He age to the α -ejection correction and U-zoning

For sample LV09, the difference between the age calculated assuming homogeneous U and Th distribution compared to a model using a 10 μm rim is –0.023 Ma (both corrected for ^{230}Th excess). The average $\delta_{\text{R/C}}$ for LV09 is close to unity, which confirms the lack of significant U and Th zoning in LV09 zircons, and thus provides additional confidence in the accuracy of the calculated (U-Th)/He age. In contrast, zircon from RPT25 have U enriched interiors relative to rims (average $\delta_{\text{R/C}} = 0.119$), which makes the calculated dates sensitive to the distribution of parent isotopes within individual crystals. The calculated model age assuming homogeneous U and Th changes the age by –0.157 Ma compared to a model using a 10 μm rim.

The interiors of Lava Creek Tuff member B zircon are enriched in U by an average of 11x relative to the rims (average $\delta_{\text{R/C}} = 0.089$), and therefore the calculated (U-Th)/He age is very sensitive to the assumed rim thickness. If, for example, we assume a 5 μm rim with

700 ppm U, rather than the 8 μm rim used in this study, the average $\delta_{\text{R/C}}$ will increase by 22.4%, which in turn decreases the α -ejection correction parameter (FZAC) and the calculated model age by 8.7% and -0.072 Ma, respectively.

Because the calculated (U–Th)/He is sensitive to zoning of U and Th within individual zircon grains, especially for sample RPT25 and 12NMYC04, we prefer the U/Pb surface ages as the most accurate measurement of eruption age. The (U–Th)/He ages measured on the same zircon samples confirm the U/Pb surface ages are concordant with the timing of eruption. If the U/Pb dates were less reproducible and older than (U–Th)/He ages, which is observed in other studies (e.g., Klemetti and Clynne, 2014; Barboni et al., 2016; Zimmerer et al., 2016), the (U–Th)/He age would be interpreted as the eruption age. The (U–Th)/He dates in this study could be further improved by laser-ablation depth profiling prior to He analysis and zircon dissolution in order to better quantify of U, Th, and Sm parent distribution within individual crystals (e.g., Hourigan et al., 2005).

5.3. Revised eruption age for the Rockland tephra

Our new eruption age for the Rockland tephra of 0.598 ± 0.013 (2 σ) Ma improves on both the precision and accuracy of the previous range of 0.570–0.610 Ma (Lanphere et al., 2004), although this range must be revised to 0.570 to 0.625 Ma based on recalculation of $^{40}\text{Ar}/^{39}\text{Ar}$ ages to updated fluence monitor age. Our new eruption age for the Rockland tephra (Table 1) is statistically younger (beyond 2 σ uncertainty) compared to the pooled $^{40}\text{Ar}/^{39}\text{Ar}$ plagioclase ages of 0.626 ± 0.008 (614) Ma (Lanphere et al., 1999) and 0.621 ± 0.007 (609) Ma (Lanphere et al., 2004), the two dates most frequently cited for the Rockland tephra in the literature.

Statistical overlap of the U/Pb ages measured from the outermost surface of Rockland tephra zircon with the (U–Th)/He cooling ages (Table 1) suggests our preferred age is accurate and represents the timing of eruption. This interpretation is supported by previous studies where zircon crystallization ages measured by SIMS on grain surfaces agree with (U–Th)/He and/or $^{40}\text{Ar}/^{39}\text{Ar}$ cooling (eruption) ages within analytical uncertainty (e.g., Holocene eruptions at Salton Buttes, CA by Schmitt et al., 2012; Wright et al., 2015; Pleistocene eruptions at Yellowstone Caldera by Stelten et al., 2015).

Our new U/Pb surface ages for Rockland zircons (Table 1) yield a less dispersed distribution than previous U/Pb SIMS results by conventionally sectioned and polished zircons (Lanphere et al., 2004). The older ages (>0.810 Ma) are interpreted by Lanphere et al. (2004) to represent pre-eruptive zircon growth, and the broader range of zircon dates (~ 0.37 Ma) likely represents overlap of the 20–30 μm diameter spot on polished zircons resulting in mixing of older and younger age domains. The average uncertainty of individual analyses in our study is ± 0.055 (1 σ) compared to ± 0.071 for analyses by Lanphere et al. (2004), which reinforces the interpretation that our preferred weighted mean age 0.598 ± 0.013 (2 σ) Ma (MSWD = 1.11) is derived from a reproducible, homogenous population of zircon crystals surfaces.

Our dataset demonstrates that coupled application of U/Pb SIMS analysis of zircon crystal faces (non-sectioned surfaces) and (U–Th)/He measurements using a sector noble gas mass spectrometer can yield an accurate date of the timing of tephra eruption. Limitations to the “double-dating” approach include (1) the assumption that zircon crystallized up to the time of eruption, and/or (2) the accuracy of model (U–Th)/He dates require knowledge of U and Th zonation within the crystal. Samples where internal zonation is minimal ($[U_{\text{rim}}]/[U_{\text{core}}] \approx 1$; e.g., LV09) or can be constrained independently (see suppl. material; Hourigan et al., 2005) offer better potential for accurate (U–Th)/He eruption ages.

5.4. Implications of the revised Rockland tephra eruption age

Widespread tephra deposits are frequently used to provide stratigraphic age-equivalent constraints in reconstruction of geologic sequences, paleoenvironments, and volcanic hazards (e.g., Lowe, 2011). Integrating accurate radiometric ages from tephras with Quaternary marine oxygen isotope records can aid in correlation of local stratigraphic sequences with global glacial-interglacial climate oscillations. For example, a large amplitude deglacial episode is well correlated with the Lava Creek super-eruption (>1000 km³) at the end of marine isotope stage 16 (MIS16, Fig. 5) (Dean et al., 2015; Matthews et al., 2015; Wotzlaw et al., 2015). The ability to directly link MIS16 with an accurate eruption age (Matthews et al., 2015; Wotzlaw et al., 2015) is important because undated sedimentary sections that identify the Lava Creek Tuff can be directly linked into a robust time-scale and thus a larger global context of climate datasets (e.g., marine $\delta^{18}\text{O}$).

Similarly, the Rockland tephra, which has a maximum estimated eruptive volume of ~ 250 km³ (Pouget et al., 2014), represents a regionally significant deposit that has been identified in outcrop and drill cores (e.g., Sarna-Wojcicki et al., 1991, 1997) across the western United States (Fig. 1). Based on our revised U/Pb age for the Rockland tephra (Table 1), the eruption occurred at 0.598 ± 0.013 Ma, coincident with interglacial MIS15 (Fig. 5). Our new age of the Rockland tephra confirms the correlation by Lanphere et al. (2004) that eruption occurred during MIS15. This new date permits revision of previous studies employing the Rockland tephra as a chronostratigraphic marker, and allows accurate recasting of geologic interpretations by integrating a revised chronology with global ice and marine climate records (e.g., Lisiecki and Raymo, 2005; Dean et al., 2015). Identification of the Rockland, Lava Creek Tuff, and/or the Bishop Tuff tephras together within stratigraphic sections (e.g., Sarna-Wojcicki et al., 1987; Rieck et al., 1992) will provide more accurate linkage of these sections to MIS15, MIS16, and MIS19 (Fig. 5), respectively (Zeeden et al., 2014).

Several undated tephra deposits have been identified in core and outcrop sections in stratigraphic proximity to the Rockland

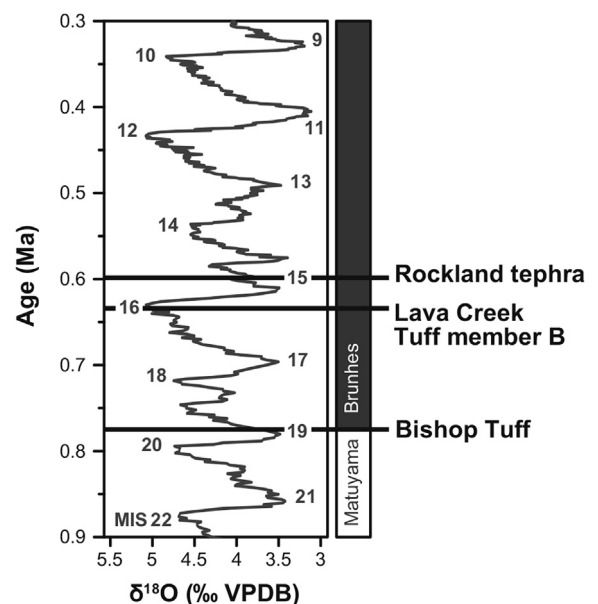


Fig. 5. $\delta^{18}\text{O}$ composition as recorded in benthic foraminifera (Lisiecki and Raymo, 2005), marine isotope stages (MIS), and the geomagnetic polarity time scale (Singer, 2014) for the late Pleistocene. Horizontal black lines designate the ages of tephras dated in this study, as well as the Bishop Tuff, California (BT; Chamberlain et al., 2014).

tephra, including the stratigraphically older Dibekulewe Bed (e.g., Adam et al., 1989; Rieck et al., 1992) and stratigraphically younger Loleta ash (Sarna-Wojcicki et al., 1987, 1988). Of particular importance is the Loleta ash, which has been identified widely in northern California and southern Oregon (e.g., Sarna-Wojcicki et al., 1987; Maier et al., 2015), and has been chemically correlated to the Bend Pumice (Sarna-Wojcicki et al., 1987). The age for the Loleta ash had been interpolated to between 0.38 and 0.40 Ma, based on sedimentation rates constrained by a Rockland tephra age of 0.40 Ma (Sarna-Wojcicki et al., 1987, 1988). Our new age and uncertainty for the Rockland tephra constrains the age of the Loleta to 0.50 ± 0.11 Ma. New high-precision radiometric dating is needed to more accurately constrain the age of the Loleta ash. Like many tephra sourced from the Cascade arc, the Loleta ash lacks potassium feldspar but contains zircon and is thus a good candidate for radiometric dating by U/Pb of zircon surfaces combined with (U-Th)/He zircon measurements.

The Dibekulewe ash is identified regionally in Oregon, Nevada, and California, and occurs stratigraphically between the Rockland tephra and the Lava Creek Tuff in the Tulelake core (Sarna-Wojcicki et al., 1991). The Dibekulewe has an interpolated age of ca. 0.51 Ma based on previous sediment accumulation rates (Sarna-Wojcicki et al., 1991, 1997). Our new age for the Rockland tephra constrains the age of the Dibekulewe ash between ca. 0.60 and 0.63 Ma (or 0.615 ± 0.15 Ma), which is a substantial revision, moving eruption from MIS13 to MIS15. This reinterpretation is consistent with the older age of ~0.63 Ma proposed by Hildreth (2007) for the Dibekulewe ash, and suggests revision to the timing/rates of tectonism-induced sedimentation in Death Valley (e.g., Knott et al., 1999) and Owen Lake (Sarna-Wojcicki et al., 1997), California.

6. Conclusions

Based on $^{238}\text{U}/^{206}\text{Pb}$ zircon surface and single-grain zircon (U-Th)/He dates, we suggest an eruption age of 0.598 ± 0.013 Ma (2σ) for the Rockland tephra, which improves on the precision and accuracy of the previous radiometric dating. The Rockland tephra epitomizes problems encountered in accurately dating Quaternary eruptions of tephra and/or lavas from many arc volcanoes, where eruptive products lack suitable minerals for radiometric dating and prolonged crystal residence in long-lived magmatic systems is common (Lanphere et al., 1999, 2004; Klemetti and Clynne, 2014). Application of zircon (U-Th)/He eruption ages and in-situ U/Pb crystallization dates of zircon crystal faces (rims), when integrated together, can accurately and precisely date the timing of eruption, although careful consideration must be made to accurately correct for U and Th distribution of parent isotopes within individual zircon.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at <http://dx.doi.org/10.1016/j.quascirev.2017.08.004>.

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