

# Geochronological and isotopic records of crustal storage and assimilation in the Wolverine Creek–Conant Creek system, Heise eruptive centre, Snake River Plain

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**Abstract** Understanding the processes of differentiation of the Yellowstone–Snake River Plain (YSRP) rhyolites is typically impeded by the apparent lack of erupted intermediate compositions as well as the complex nature of their shallow interaction with the surrounding crust responsible for their typically low O isotopic ratios. A pair of normal- $\delta^{18}\text{O}$  rhyolitic eruptions from the Heise eruptive centre in eastern Idaho, the Wolverine Creek Tuff and the Conant Creek Tuff, represent unique magmatic products of the Yellowstone hotspot preserving abundant vestiges of the intermediate differentiation steps leading to rhyolite generation. We address both shallow and deep processes of magma generation and storage in the two units by combining high-precision ID–TIMS U–Pb zircon geochronology, trace element, O and Hf isotopic studies of zircon, and Sr isotopic analyses of individual high-Mg# pyroxenes inherited from lower- to mid-crustal differentiation stages. The zircon geochronology confirms the derivation of both tuffs from the same rhyolitic magma reservoir erupted at  $5.5941 \pm 0.0097$  Ma, preceded by at least  $92 \pm 14$  ky of

continuous or intermittent zircon saturation approximating the length of pre-eruptive magma accumulation in the upper crust. Some low-Mg# pyroxenes enclosing zircons predate the eruption by at least  $45 \pm 27$  ky, illustrating the co-crystallisation of major and accessory phases in the near-liquidus rhyolitic melts of the YSRP over a significant period of time. Coeval zircon crystals are isotopically heterogeneous (two populations at  $\epsilon\text{Hf} \sim -5$  and  $-13$ ), requiring the assembly of isotopically distinct melt pockets directly prior to, or during, the eruption. The primitive Mg# 60–90 pyroxenes are out of isotopic equilibrium with the host rhyolitic melt ( $^{87}\text{Sr}/^{86}\text{Sr}_i = 0.70889$ ), covering a range of  $^{87}\text{Sr}/^{86}\text{Sr}_i = 0.70705\text{--}0.70883$  corresponding to ratios typical of the most radiogenic YSRP basalts to the least radiogenic YSRP rhyolites. Together with the low  $\epsilon\text{Hf}$  in zircon, the Sr isotopic ratios illustrate limited assimilation dominated by radiogenic Archean crustal source materials incorporated into variably evolved YSRP melts as they progress towards rhyolitic compositions by assimilation–fractional crystallisation.

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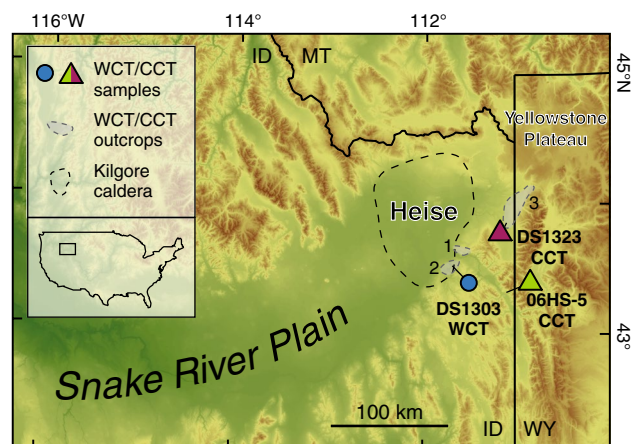
**Keywords** Heise · Yellowstone hotspot · Zircon · U–Pb geochronology · Sr isotopes · Pyroxene

## Introduction

The geological processes and timescales of generation and accumulation of large, ‘super-eruptive’ volumes of highly evolved magma remain topics of intense debate, with marked differences in apparent magma accumulation timescales recognised between eruptive products of broadly subduction-related and intraplate tectonic settings, associated with contrasts in e.g. crystallinity, magmatic temperatures, or volatile contents (Bachmann and Bergantz 2008;

Christiansen and McCurry 2008; Wolff et al. 2015). It is increasingly observed in arc settings that the geochronological record of zircon crystals found in evolved melts preserves evidence for an extended period of crystallisation of up to a few hundred thousand years (e.g. Reid et al. 1997; Brown and Fletcher 1999; Claiborne et al. 2010; Wotzlav et al. 2013; Guillong et al. 2014). Such longevity is likely related to long-term thermal buffering of these systems near or below the zircon saturation temperature by repeated inputs of recharge magma, balanced by conduction-dominated cooling (Mahood 1990; Bachmann and Bergantz 2004; Gelman et al. 2013; Cooper and Kent 2014). In contrast, within intraplate volcanic centres, such as the iconic Yellowstone–Snake River Plain (YSRP) volcanic province in the north-western USA, the apparent spread of individual zircon ages appears to be limited to a few tens of thousands of years (Rivera et al. 2014; Wotzlav et al. 2014, 2015). It remains unclear whether such a zircon record always indicates a true rapid crystallisation/assembly timescale in the Yellowstone hotspot system, or whether such data represent the last cooling events whereas most older zircon crystals are efficiently resorbed during preceding reheating events. While cool, wet, crystal-rich super-eruptions are thought to mobilise near-solidus crystal mushes providing an effective long-term insulation of the magmatic reservoir (Marsh 1981; Bachmann and Bergantz 2004; Hildreth 2004), the evidence of such a mechanism increasing the survivability of the hot, crystal-poor magmas of the YSRP is less obvious (Ellis et al. 2014).

In addition to the discussion of the longevity of YSRP rhyolites, the details of the magmatic evolution responsible for generating large volumes of YSRP rhyolite with subordinate basalt yet no erupted intermediate composition magmas remain contentious. The trace element and radiogenic isotope records indicate that the rhyolites likely originate from mantle-derived basaltic parents with important assimilation of Archean basement rocks (e.g. 7–40%, preferred 12%, McCurry and Rodgers 2009; 45–65%, Hildreth et al. 1991 or >60%, Nash et al. 2006). Moreover, the light oxygen isotope compositions of many YSRP rhyolites have been used to argue for an important contribution from melting of shallow, young, and hydrothermally altered crust (Bindeman and Valley 2001; Boroughs et al. 2005; Bindeman et al. 2007; Watts et al. 2011; Boroughs et al. 2012; Simakin and Bindeman 2012). Such massive amounts of assimilation of cold crustal rocks seem at odds with thermal constraints during assimilation–fractional crystallisation (e.g. Thompson et al. 2002; Bohrsen et al. 2014). While it appears that a large part of the radiogenic isotope record may be sourced already from the interaction of the Yellowstone plume with deep crust or lithospheric mantle (Doe et al. 1982; Hanan et al. 2008; McCurry and Rodgers 2009; Jean et al. 2014), the exact crustal evolutionary path of YSRP melts from primitive basalts to the



**Fig. 1** Location of the Heise eruptive centre in the eastern Snake River Plain. The *black dashed line* illustrates the approximate position of the Heise caldera (Kilgore Tuff caldera of Morgan and McIntosh 2005). The *grey shading* schematically represents outcrop areas of both studied units in the Big Hole Mountains (1), northern Caribou Range (2), and the north-western part of the Teton Range (3). *Symbols* indicate localities of Wolverine Creek Tuff (WCT) and Conant Creek Tuff (CCT) samples used in this study (for coordinates, see Supplementary material)

erupted rhyolites remains enigmatic. Geophysical evidence suggests the existence of low-melt content, intermediate magmatic storage levels throughout the crust underneath the YSRP (Sparlin et al. 1982; Peng and Humphreys 1998; DeNosaquo et al. 2009; Farrell et al. 2014; Huang et al. 2015), but direct sampling of these lithologies parental to the rhyolites has been scarce to absent.

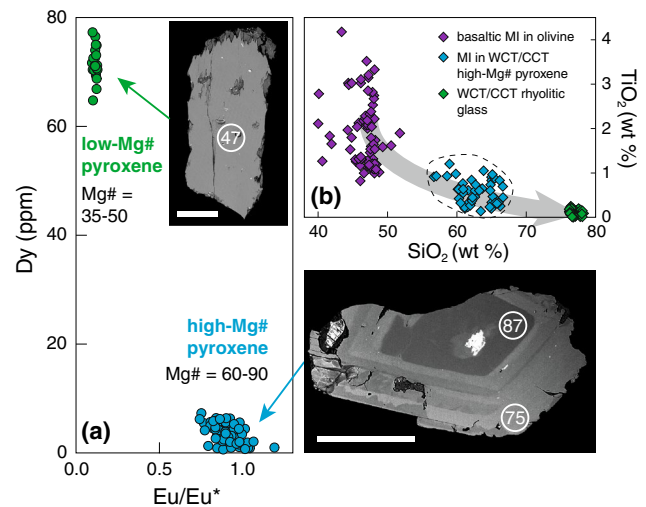
In this contribution, we focus on two normal- $\delta^{18}\text{O}$  or slightly  $^{18}\text{O}$ -depleted ( $\delta^{18}\text{O}_{\text{melt}} = 5.9$  and  $6.5\text{‰}$ ; Watts et al. 2011) rhyolitic ignimbrites from the Heise volcanic field in Idaho, the Wolverine Creek Tuff and Conant Creek Tuff, previously shown to preserve vestiges of intermediate composition magmas inferred to complete the basalt–rhyolite fractionation sequence (Szymanowski et al. 2015). The choice of normal- $\delta^{18}\text{O}$  units with O isotopic ratios characteristic for purely mantle-derived melts is of particular importance as it limits the probability of shallow-crustal melting of hydrothermally altered crust in generating these rhyolites, and provides a rare glimpse into the deeper processes of rhyolite genesis. We combine high-precision ID–TIMS zircon U–Pb geochronology with radiogenic isotope studies of zircon (Hf) and pyroxene (Sr) crystals to address both shallow and deep processes of magma generation and pre-eruptive storage.

## Geological background

The Heise volcanic field in the eastern Snake River Plain, Idaho (Fig. 1), active between 6.6 and 4 Ma (Morgan and

McIntosh 2005; Bindeman et al. 2007; Watts et al. 2011), is the second-youngest eruptive centre of the Yellowstone hotspot track, preceding the main period of activity of the Yellowstone Plateau volcanic field by approximately 2 million years (Christiansen 2001). The eruptive record at Heise is dominated by a series of voluminous (100–1000s km<sup>3</sup>) explosive eruptions of high-silica rhyolite followed by effusions of basaltic lavas filling the topographical low of the Snake River Plain. The rhyolitic magma produced within the whole YSRP system ('Snake River-type' rhyolites of Branney et al. 2008) is typically hot ( $T > 800$  °C, e.g. Cathey and Nash 2004; Watts et al. 2011), dry ( $\leq 2.5$  wt% H<sub>2</sub>O, e.g. Bolte et al. 2015), and crystal-poor with an anhydrous mineral assemblage of plagioclase  $\pm$  sanidine  $\pm$  quartz + augite  $\pm$  pigeonite  $\pm$  orthopyroxene  $\pm$  fayalitic olivine  $\pm$  ilmenite  $\pm$  magnetite + accessory zircon, apatite, allanite/chevkinite (Christiansen 2001; Ellis et al. 2013). Most notably, the vast majority of YSRP rhyolites, including some Heise units, have a low  $\delta^{18}\text{O}$  signature ( $\delta^{18}\text{O}_{\text{magma}} < 5.5\text{‰}$ ) requiring an involvement of hydrothermally altered crust (Hildreth et al. 1984; Bindeman and Valley 2001; Bindeman et al. 2007; Watts et al. 2011).

Within the Heise sequence, two of the normal- $\delta^{18}\text{O}$  rhyolitic ignimbrites, the Wolverine Creek Tuff (WCT) and the Conant Creek Tuff (CCT), share a number of features hinting at their common provenance. Both units were previously dated at around 5.5 Ma (Morgan and McIntosh 2005; Bindeman et al. 2007; Anders et al. 2014), but despite the multiple existing <sup>40</sup>Ar/<sup>39</sup>Ar and U–Pb dates, the relative timing of the eruptive events producing both deposits is not fully resolved. The two ignimbrites are distinct in the outcrops owing to different degrees of welding; the WCT occurs as a non-welded, crystal-poor ignimbrite dominantly composed of microvesicular pumice clasts and bubble wall shards as well as angular to sub-rounded clasts of black glass; the CCT bears resemblance to other 'SR-type' rhyolites being crystal-poor and densely welded with a dominant devitrified zone associated with a basal vitrophyre (Morgan and McIntosh 2005). The two units only occur in stratigraphic contact at one locality where the CCT overlies the WCT with no clear depositional break (Phillips et al. 2016). However, despite the macroscopic differences, the mineral and glass major and trace element chemistry of both units is indistinguishable, suggesting that they derive from a single magma reservoir (see Szymanowski et al. 2015 for a detailed discussion). In addition to the typical rhyolitic mineral cargo, both WCT and CCT contain a proportion of relatively primitive clinopyroxene of Mg# ~ 60–90 (Fig. 2a) and plagioclase of An<sub>38–63</sub>, often found within crystal aggregates, interpreted to represent parts of a cumulate body that was remobilised and partly incorporated into the rhyolitic magma chamber. The primitive pyroxene crystals enclose intermediate (57–67 wt% SiO<sub>2</sub>) melt inclusions (Fig. 2b)



**Fig. 2** Summary of the bimodal mineral chemistry within the Wolverine Creek and Conant Creek tuffs (Szymanowski et al. 2015). **a** Two kinds of clinopyroxene found within WCT/CCT differing in Mg#, REE contents (here Dy), Eu/Eu\* and internal compositional zonation patterns illustrated with backscatter electron images of representative pyroxene crystals from both groups (*numbers in circles* denote Mg#). The *white bars* in both images are 250  $\mu\text{m}$  long. **b** TiO<sub>2</sub>–SiO<sub>2</sub> concentrations in melt inclusions (MI) hosted by the high-Mg# WCT pyroxene crystals compared to the host WCT volcanic glass and melt inclusions in olivine from Snake River Plain basalts (Stefano et al. 2010). The *grey arrow* illustrates a trend in melt composition consistent with (assimilation–) fractional crystallisation of SRP basalts to produce rhyolitic melts of the WCT/CCT

recording magmatic liquids that bridge the compositional gap between basalts and rhyolites and thus support the fractional crystallisation component of magmatic differentiation within the two units. In addition, bulk isotopic data with <sup>87</sup>Sr/<sup>86</sup>Sr ratios of 0.7089 for CCT and 0.7095 for WCT (Watts et al. 2011), as well as  $\epsilon\text{Nd}$  between  $-5.2$  and  $-6.8$  (Nash et al. 2006; Watts et al. 2011), require a significant amount of assimilated material to account for the shift from the primitive plume-derived basalts ('Imnaha component') at <sup>87</sup>Sr/<sup>86</sup>Sr ~0.704 and  $\epsilon\text{Nd}$  ~+4 (Wolff et al. 2008).

## Methods

### Zircon geochemistry

A total of 27 zircon crystals from the Wolverine Creek Tuff ( $n = 14$ ) and Conant Creek Tuff ( $n = 13$ , including three crystals liberated from clinopyroxene separates used for Sr isotope determination) were analysed for U–Pb isotopes by isotope dilution-thermal ionisation mass spectrometry (ID–TIMS) at the University of Geneva (CCT; Wotzlaw et al. 2015) and ETH Zürich (WCT, px-hosted CCT; von Quadt et al. 2016). Prior to analysis, ~30 crystals per sample were

separated following standard procedures and annealed by heating to 900 °C for 48 h in a muffle furnace. Individual zircons were then selected under a binocular microscope and mounted in epoxy resin, polished, and imaged using cathodoluminescence and backscattered electron microscopy, then analysed for trace elements by LA-ICPMS (WCT, CCT) and by SIMS for O isotopes (CCT). LA-ICPMS analyses included U–Pb isotopes in order to detect inherited cores, but no xenocrysts were found.

Trace element concentrations were determined by LA-ICPMS using a 193 nm ArF excimer laser coupled to a Thermo Element ICP mass spectrometer at the University of Lausanne (CCT) or ETH Zürich (WCT), using NIST 612/610 glass as the primary standard. Both cores and rims were analysed in each crystal with a spot size of 20/25 µm. Repeat analyses of zircon standards yielded external reproducibility (2 RSD) between 2.9% (Hf) and 23.9% (Nd) with the low-abundance Pr reproducible within 57%. Oxygen isotope analyses of CCT zircons were performed at the Canadian Centre for Isotopic Microanalysis, University of Alberta, employing a Cameca IMS1280 secondary ion mass spectrometer (see Wotzlaw et al. 2015 for details). Repeat analyses of primary reference zircons (Mud Tank zircon,  $\delta^{18}\text{O}_{\text{VSMOW}} = +4.87\text{‰}$ ) yielded within-session reproducibility between  $\pm 0.14$  and  $0.20\text{‰}$  ( $2\sigma$ ) that was propagated into the uncertainty of samples yielding an average single-spot uncertainty of  $\pm 0.25\text{‰}$ . Analyses of secondary reference zircon Temora-2 ( $\delta^{18}\text{O}_{\text{VSMOW}} = +8.20\text{‰}$ ; Black et al. 2004) yielded a weighted mean  $\delta^{18}\text{O}_{\text{VSMOW}}$  of  $+8.260 \pm 0.061\text{‰}$  (MSWD = 1.6;  $n = 18$ ).

After LA-ICPMS/SIMS analyses, selected individual crystals were extracted from the grain mount, transferred into 3 ml Savillex beakers, rinsed with 3 N  $\text{HNO}_3$ , and loaded into 200 µl Savillex microcapsules for partial dissolution in HF + trace  $\text{HNO}_3$  at 180 °C for 12 h ('chemical abrasion (CA)'; Mattinson 2005). The abraded crystals were transferred back into 3 ml beakers for cleaning with 6 N HCl and 3 N  $\text{HNO}_3$ , then reloaded into their respective 200-µl microcapsules, spiked with 3–9 mg of the EARTH-TIME  $^{202}\text{Pb}$ – $^{205}\text{Pb}$ – $^{233}\text{U}$ – $^{235}\text{U}$  tracer solution (Condon et al. 2015; McLean et al. 2015), and dissolved in HF + trace  $\text{HNO}_3$  at 210 °C for ~60 h. After dissolution, samples were dried down and redissolved in 6 N HCl at 180 °C for ~12 h to convert to chlorides, then dried down again and taken up in 3 N HCl. Pb and U were separated from the matrix using an HCl-based single-column anion exchange chemistry modified from Krogh (1973); the remaining eluted fraction ('wash') was collected for Hf isotopic analyses. The U–Pb fractions were dried down with a drop of 0.035 M  $\text{H}_3\text{PO}_4$  and loaded on single outgassed Re filaments with a silica gel emitter modified from Gerstenberger and Haase (1997). U and Pb measurements were performed with a

Thermo TRITON (UNIGE) or TRITON Plus (ETH) thermal ionisation mass spectrometer; Pb was measured in dynamic mode on a MasCom secondary electron multiplier and U was measured as  $\text{UO}_2$  in static mode on Faraday cups equipped either with  $10^{12} \Omega$  resistors (UNIGE) or  $10^{13} \Omega$  resistors (ETH; von Quadt et al. 2016). Pb mass fractionation factors were derived from the measured  $^{202}\text{Pb}/^{205}\text{Pb}$  ratio normalised to the true value of 0.99924 (Condon et al. 2015). U isotopic ratios were corrected for isobaric interferences of  $^{233}\text{U}^{18}\text{O}^{16}\text{O}$  on  $^{235}\text{U}^{16}\text{O}_2$  using an  $^{18}\text{O}/^{16}\text{O}$  of  $0.00205 \pm 0.00005$  and for mass fractionation using the measured  $^{233}\text{U}/^{235}\text{U}$  ratio relative to the true value of 0.99506 (Condon et al. 2015) and sample  $^{238}\text{U}/^{235}\text{U}$  of  $137.818 \pm 0.045$  (Hiess et al. 2012). All common Pb in zircon analyses was attributed to laboratory blank and corrected with the average composition of total procedural blank measurements. Data reduction was performed using the Tripoli and U–Pb\_Redux software packages (Bowring et al. 2011) using data reduction and uncertainty propagation algorithms of McLean et al. (2011). U–Pb ratios and dates were calculated relative to a tracer  $^{235}\text{U}/^{205}\text{Pb}$  ratio of  $100.23 \pm 0.046\%$  ( $2\sigma$ ) and using the decay constants of Jaffey et al. (1971).  $^{206}\text{Pb}/^{238}\text{U}$  ratios and dates were corrected for initial  $^{230}\text{Th}$  disequilibrium using Th/U of  $3.39 \pm 0.12$  ( $2\sigma$ ,  $n = 124$ ) measured in WCT/CCT glass by Szymanowski et al. (2015); whole-grain Th/U of zircon crystals (Table 1) is relatively homogeneous at  $0.58 \pm 0.22$  ( $2\sigma$ ), similar to rim values of  $0.51 \pm 0.30$  (Fig. 5) considered to be in equilibrium with the glass. All uncertainties are reported at the  $2\sigma$  level and ignore systematic uncertainties associated with the tracer calibration and decay constants (Jaffey et al. 1971; McLean et al. 2015).

Hf isotopic compositions of single zircons were analysed in eluted fractions remaining from U–Pb anion exchange chemistry. Hf was either separated using an HCl–HF-based column chemistry using the Ln-spec resin and analysed on a Nu Plasma II MC-ICPMS at ETH Zürich (WCT, px-hosted CCT), or analysed on a Thermo Neptune Plus MC-ICPMS at the University of Geneva without chemical separation (CCT; see D'Abzac et al. 2016 for details). All  $^{176}\text{Hf}/^{177}\text{Hf}$  isotope ratios were normalised to the JMC475 standard (recommended value of  $^{176}\text{Hf}/^{177}\text{Hf} = 0.282160$ ; Vervoort and Blichert-Toft 1999), which returned highly reproducible values over the course of three sessions at  $0.282186 \pm 10$  ( $2\sigma$ ,  $n = 29$ ),  $0.282153 \pm 12$  ( $n = 11$ ), and  $0.282153 \pm 22$  ( $n = 35$ ). A minimal correction for in situ ingrowth of  $^{176}\text{Hf}$  due to  $^{176}\text{Lu}$  decay was calculated using the  $^{176}\text{Lu}$  decay constant of Scherer et al. (2001), with  $^{176}\text{Lu}/^{177}\text{Hf}$  directly measured (CCT) or derived from Lu/Hf measured via LA-ICPMS (WCT). All reported uncertainties include the within-run precision of each measurement and the reproducibility of the JMC475 standard.

**Table 1** CA-ID-TIMS U–Pb isotopic data for WCT/CCCT zircon

Sample	Composition				Isotopic ratios						Dates (Ma)					
	Th/U <sup>a</sup>	Pb* (pg) <sup>b</sup>	Pb <sub>c</sub> (pg) <sup>c</sup>	Pb*/Pb <sub>c</sub> <sup>d</sup>	<sup>206</sup> Pb/ <sup>204</sup> Pb <sup>e</sup>	<sup>206</sup> Pb/ <sup>238</sup> U <sup>f</sup>	$\pm 2\sigma$ (%)	<sup>207</sup> Pb/ <sup>235</sup> U <sup>f</sup>	$\pm 2\sigma$ (%)	<sup>207</sup> Pb/ <sup>206</sup> Pb <sup>f</sup>	$\pm 2\sigma$ (%)	Corr. coef.	<sup>206</sup> Pb/ <sup>238</sup> U <sup>g</sup>	$\pm 2\sigma$ (abs)	<sup>207</sup> Pb/ <sup>235</sup> U <sup>g</sup>	$\pm 2\sigma$ (abs)
<i>Conant Creek Tuff</i>																
z1	0.52	1.16	0.13	9.04	534	0.0008591	0.18	0.00544	3.99	0.0459	3.86	0.712	5.5357	0.0098	5.5281	0.0095
z2	0.59	1.75	0.09	20.46	1164	0.0008592	0.09	0.00565	1.73	0.0477	1.68	0.671	5.5364	0.0048	5.6266	0.0047
z3	0.67	1.22	0.14	8.68	494	0.0008603	0.23	0.00535	4.67	0.0451	4.52	0.699	5.544	0.013	5.631	0.012
z4	0.50	2.26	0.23	10.01	592	0.0008599	0.17	0.00553	3.54	0.0467	3.42	0.689	5.5408	0.0094	5.6338	0.0092
z5	0.58	0.77	0.10	8.03	468	0.0008620	0.22	0.00574	4.41	0.0483	4.25	0.719	5.555	0.012	5.645	0.012
z6	0.50	0.71	0.09	8.04	478	0.0008606	0.22	0.00556	4.43	0.0468	4.28	0.708	5.545	0.012	5.638	0.012
z7	0.55	1.88	1.45	1.30	90	0.0008594	1.02	0.00690	21.34	0.0583	20.59	0.752	5.538	0.056	5.629	0.054
z8	0.63	0.47	0.77	0.61	51	0.0008647	2.24	0.00817	39.62	0.0686	37.97	0.752	5.57	0.12	5.66	0.12
z9	0.52	2.99	0.41	7.29	433	0.0008624	0.19	0.00583	4.50	0.0490	4.37	0.726	5.557	0.011	5.649	0.010
z10	0.48	0.23	0.13	1.78	121	0.0008539	0.82	0.00514	21.12	0.0436	20.51	0.754	5.502	0.045	5.596	0.044
<i>Pyroxene-hosted Conant Creek Tuff</i>																
Px12-1	0.55	1.31	0.62	2.11	141	0.0008580	0.39	0.00602	6.78	0.0509	6.54	0.638	5.529	0.022	5.620	0.021
Px11-1	0.98	2.36	0.71	3.34	194	0.0008623	0.35	0.00575	5.10	0.0484	4.93	0.506	5.557	0.020	5.634	0.019
Px12-2	0.59	1.19	0.74	1.61	111	0.0008612	0.47	0.00613	8.52	0.0517	8.24	0.615	5.549	0.026	5.639	0.026
<i>Wolverine Creek Tuff</i>																
z3	0.86	0.67	0.17	3.98	234	0.0008557	0.36	0.00568	5.91	0.0482	5.63	0.774	5.514	0.020	5.596	0.020
z6	0.55	0.33	0.28	1.21	89	0.0008624	0.65	0.00630	11.51	0.0530	11.11	0.641	5.557	0.036	5.649	0.035
z7	0.52	0.87	0.16	5.40	337	0.0008591	0.18	0.00573	3.06	0.0484	2.94	0.663	5.5360	0.0099	5.6283	0.0097
z10	0.49	0.67	0.20	3.35	217	0.0008558	0.30	0.00581	4.95	0.0492	4.75	0.711	5.515	0.016	5.608	0.016
z11	0.53	0.35	0.15	2.27	152	0.0008618	0.48	0.00607	8.10	0.0511	7.75	0.740	5.553	0.026	5.645	0.026
z13	0.61	0.46	0.18	2.58	167	0.0008577	0.63	0.00564	10.21	0.0477	9.70	0.817	5.527	0.035	5.616	0.034
z14	0.68	0.68	0.13	5.28	317	0.0008690	0.18	0.00591	3.81	0.0493	3.69	0.651	5.5992	0.0099	5.6866	0.0096
z15	0.58	5.94	0.21	28.56	1678	0.0008648	0.10	0.00568	0.74	0.0477	0.67	0.687	5.5724	0.0055	5.6629	0.0054
z16	0.48	0.81	0.16	5.23	330	0.0008536	0.18	0.00575	3.04	0.0489	2.92	0.682	5.501	0.010	5.5941	0.0097
z17	0.52	0.17	0.20	0.85	69	0.0008678	1.77	0.00723	33.64	0.0605	32.28	0.779	5.592	0.099	5.684	0.095
z18	0.50	0.65	0.18	3.68	237	0.0008617	0.29	0.00590	4.65	0.0497	4.46	0.694	5.552	0.016	5.645	0.016
z21	0.52	0.86	0.12	7.48	460	0.0008631	0.14	0.00578	2.40	0.0486	2.31	0.682	5.5617	0.0080	5.6541	0.0078

Table 1 continued

Sample	Composition				Isotopic ratios						Dates (Ma)						
	Th/U <sup>a</sup>	Pb* (pg) <sup>b</sup>	Pb <sub>c</sub> (pg) <sup>c</sup>	Pb*/Pb <sub>c</sub> <sup>d</sup>	<sup>206</sup> Pb/ <sup>204</sup> Pb <sup>e</sup>	<sup>206</sup> Pb/ <sup>238</sup> U <sup>f</sup>	±2σ (%)	<sup>207</sup> Pb/ <sup>235</sup> U <sup>f</sup>	±2σ (%)	<sup>207</sup> Pb/ <sup>206</sup> Pb <sup>f</sup>	±2σ (%)	<sup>206</sup> Pb/ <sup>238</sup> U <sup>g</sup>	±2σ (abs)	<sup>206</sup> Pb/ <sup>238</sup> U <sup>h</sup>	±2σ (abs)	<sup>207</sup> Pb/ <sup>235</sup> U <sup>g</sup>	±2σ (abs)
z25	0.67	0.77	0.19	4.07	249	0.0008573	0.30	0.00572	4.72	0.0484	4.52	0.016	0.016	5.612	0.016	5.80	0.27
z31	0.49	0.24	0.22	1.08	82	0.0009211	1.08	0.00703	16.93	0.0554	16.08	0.064	0.062	6.028	0.062	7.1	1.2

<sup>a</sup> Th contents calculated from radiogenic <sup>208</sup>Pb and the <sup>230</sup>Th-corrected <sup>206</sup>Pb/<sup>238</sup>U date of the sample, assuming concordance between the U–Pb and Th–Pb systems

<sup>b</sup> Total mass of radiogenic Pb

<sup>c</sup> Total mass of common Pb

<sup>d</sup> Ratio of radiogenic Pb (including <sup>208</sup>Pb) to common Pb

<sup>e</sup> Measured ratio corrected for fractionation and spike contribution only

<sup>f</sup> Measured ratios corrected for fractionation, tracer, and blank. Blank was corrected using the average composition of total procedural blank measurements taken throughout the course of the study in both laboratories. At University of Geneva (CCT): <sup>206</sup>Pb/<sup>204</sup>Pb = 17.618 ± 0.368, <sup>207</sup>Pb/<sup>204</sup>Pb = 14.730 ± 0.450, <sup>208</sup>Pb/<sup>204</sup>Pb = 35.77 ± 1.07 (*n* = 10), at ETH Zurich (WCT): <sup>206</sup>Pb/<sup>204</sup>Pb = 18.407 ± 0.197, <sup>207</sup>Pb/<sup>204</sup>Pb = 15.191 ± 0.194, <sup>208</sup>Pb/<sup>204</sup>Pb = 36.926 ± 0.453 (*n* = 18). All uncertainties are 1σ

<sup>g</sup> Isotopic dates calculated using the decay constants  $\lambda(^{238}\text{U}) = 1.55125 \times 10^{-10}$  and  $\lambda(^{235}\text{U}) = 9.8485 \times 10^{-10} \text{ year}^{-1}$  (Jaffey et al. 1971)

<sup>h</sup> Corrected for initial Th/U disequilibrium using radiogenic <sup>208</sup>Pb and Th/U<sub>meit</sub> of 3.39 ± 0.12 (2σ)

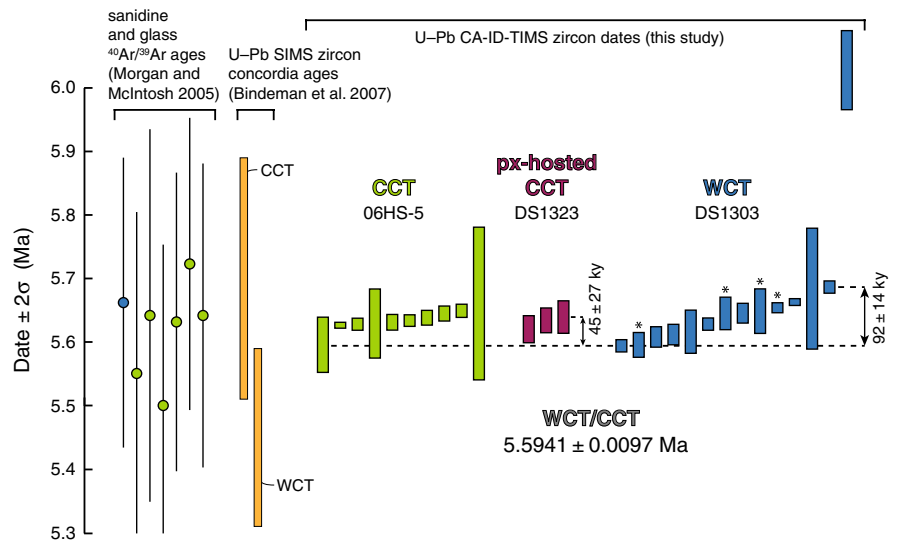
## <sup>87</sup>Sr/<sup>86</sup>Sr in pyroxene and glass

The high-Mg# clinopyroxenes from the Wolverine Creek Tuff are sufficiently large and rich in Sr (50–260 ppm) that single pyroxenes could be analysed for <sup>87</sup>Sr/<sup>86</sup>Sr using thermal ionisation mass spectrometry (TIMS); the evolved (low-Mg#, <15 ppm Sr) clinopyroxene separated from other Heise units required bulk mineral separates of 10–20 grains (2–4 mg). For glass, large separates of 7.1 and 9.4 mg were hand-picked from crushed samples. The separates were digested with a concentrated HF/HNO<sub>3</sub> mixture in Teflon beakers for 3 days (glass) or 24 h (pyroxene); after evaporation, they were redissolved in 6 N HCl for 12 h. All solutions were evaporated, and the dry residues were converted in 2.5 N HNO<sub>3</sub>, followed by Sr separation in PP ion exchange columns with Sr- and TRU-spec resins, employing the chromatography technique of Pin et al. (1994). Strontium isotope ratios were measured on a Thermo TRITON Plus multicollector TIMS at ETH Zürich in static mode. The Sr isotope ratios are mass fractionation corrected to <sup>88</sup>Sr/<sup>86</sup>Sr = 8.375209; the NBS 987 standard measurements returned <sup>87</sup>Sr/<sup>86</sup>Sr = 0.7102460 ± 34 (2 se; *n* = 14) during the period of analysis. Rb and Sr concentrations of most samples were determined by isotope dilution using a highly enriched <sup>87</sup>Rb/<sup>84</sup>Sr spike. All <sup>87</sup>Sr/<sup>86</sup>Sr ratios are reported as initial values using Rb/Sr measured by TIMS, LA-ICPMS (Szymanowski et al. 2015) or assumed based on similarity to other samples; all corrected to <sup>40</sup>Ar/<sup>39</sup>Ar ages of Morgan and McIntosh (2005).

## Results

### The zircon record

Three kinds of zircon crystals were analysed in this study: bulk-rock-separated WCT zircons, bulk-rock-separated CCT zircons, and pyroxene-hosted CCT zircons recovered from low-Mg# clinopyroxene samples dissolved for Sr isotopic analyses (Px11, Px12, Px19). All three samples return overlapping zircon age distributions (Fig. 3) and the youngest grains dated in each of the samples overlap in age at 5.596 ± 0.044 Ma (2σ, CCT z10), 5.620 ± 0.021 Ma (Px12-1), and 5.5941 ± 0.0097 Ma (WCT z16; Table 1). Both CCT samples display a narrow range of individual zircon crystallisation ages, while the WCT preserves zircons both younger and older than that range (including one antecryst at 6.028 ± 0.062 Ma). The overlap of all three samples supports their cogenetic identity and allows the derivation of a common maximum estimate of the WCT/CCT eruption age. We consider the crystallisation age of the single youngest zircon (WCT z16) as our best estimate of WCT/CCT eruption age at 5.5941 ± 0.0097 Ma (Fig. 3).



**Fig. 3** ID-TIMS U-Pb zircon geochronology of Conant Creek Tuff (CCT) and Wolverine Creek Tuff (WCT). Age-ranked individual zircon  $^{206}\text{Pb}/^{238}\text{U}$  dates are reported with their  $2\sigma$  analytical uncertainty. The WCT/CCT eruption age estimate of  $5.5941 \pm 0.0097$  Ma (dashed line) is based on the date of the youngest WCT/CCT zircon (WCT z16) reported with its  $2\sigma$  analytical uncertainty, not including tracer and decay constant contributions. Asterisks indicate grains of

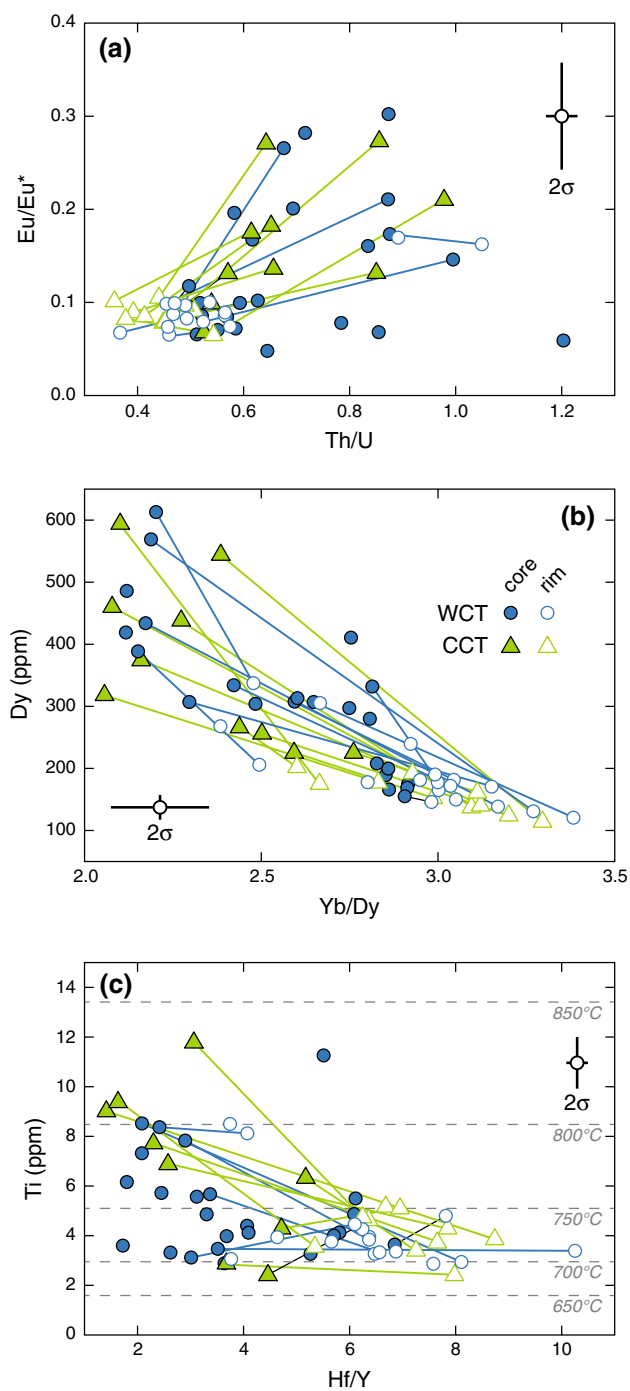
$\varepsilon\text{Hf} < -10$ . Shown next to pyroxene-hosted CCT zircon dates is the maximum period of pre-eruptive crystallisation of the pyroxenes; next to WCT dates is a minimum estimate of the duration of pre-eruptive zircon crystallisation. Also shown for comparison are U-Pb zircon concordia ages from Bindeman et al. (2007) and individual  $^{40}\text{Ar}/^{39}\text{Ar}$  eruption ages for both units from Morgan and McIntosh (2005) recalculated using the calibration of Kuiper et al. (2008)

This result is in good agreement with previously published U-Pb SIMS zircon concordia ages of  $5.45 \pm 0.14$  Ma (WCT) and  $5.70 \pm 0.19$  Ma (CCT; Bindeman et al. 2007) and it overlaps within uncertainty with published  $^{40}\text{Ar}/^{39}\text{Ar}$  eruption ages of both units (Morgan and McIntosh 2005) recalculated utilising the calibration of Kuiper et al. (2008) (Fig. 3). Subtracting this ID-TIMS zircon eruption age estimate from the age of the oldest autocrystic zircon (WCT z14) defines the minimum time of pre-eruptive zircon crystallisation as  $92 \pm 14$  ky. Additionally, the recognition that some of the zircons were fully included in CCT clinopyroxene crystals requires that those pyroxenes crystallised not earlier than  $45 \pm 27$  ky before the eruption.

Zircon crystals from both eruptive units are compositionally similar, and both populations exhibit a consistent, age-independent core-rim compositional relationship (Fig. 4). Crystal cores are enriched in all rare earth elements (REE), Y, Ti and depleted in Hf relative to the rims that also display a decrease in Th/U and a more pronounced negative Eu anomaly. The steepness of the heavy element-enriched zircon REE pattern (as depicted by e.g. Yb/Dy ratio for heavy REEs, Fig. 4b) increases from core to rim, which requires a successive depletion of the melt in light and middle REEs relative to HREE. The decrease in Eu/Eu\* can be related to the fractionation of feldspar, while the decrease in Th/U and the REE signatures requires the fractionation of apatite or rare thorium- and light REE-enriched accessory phases such as allanite and/or chevkinite.

Ti-in-zircon temperatures obtained using the formulation of Ferry and Watson (2007) show a down-temperature evolution from cores to rims over  $\sim 100$  °C, between  $\sim 800$  °C (zircon saturation temperature; Watts et al. 2011) and  $\sim 700$  °C, with a wide range of temperature in the cores and a limited, lower temperature range in the rims (Fig. 4c). As model inputs,  $a_{\text{SiO}_2} = 1$  was assumed based on the presence of quartz, while the melt  $a_{\text{TiO}_2} = 0.5$  was derived using the  $\text{TiO}_2$  solubility model of Hayden and Watson (2007) using a major element compositional parameter  $\text{FM} = 1.54$  (based on glass compositions), average WCT/CCT glass Ti content (769 ppm; Szymanowski et al. 2015) and a zircon saturation temperature of 800 °C (Watson and Harrison 1983; Watts et al. 2011). Varying  $a_{\text{TiO}_2}$  by 0.1 results in changing the computed temperatures by  $\leq 25$  °C.

In situ oxygen isotope ratios measured in CCT zircons range between  $\delta^{18}\text{O} = 3.20$  and  $4.93\text{‰}$  with the majority of data clustering at  $\delta^{18}\text{O} = 3.9\text{--}4.5\text{‰}$  (Fig. 5a). The O isotopic composition does not vary systematically between crystal cores and rims, nor does it correlate strongly with trace element contents. The in situ data are consistent with previous laser fluorination determinations on bulk CCT zircon separates ( $\delta^{18}\text{O} = 3.89, 4.03\text{‰}$ ), WCT/CCT sanidine crystals ( $\delta^{18}\text{O} = 5.19\text{--}6.06\text{‰}$ ), and WCT glass ( $\delta^{18}\text{O} = 6.35\text{--}6.36\text{‰}$ , all Watts et al. 2011), indicating that all mineral phases are close to high-temperature (e.g. 750 °C) oxygen isotopic equilibrium with a normal- $\delta^{18}\text{O}$  to



**Fig. 4** Trace element (Th/U, Eu/Eu\*, Dy, Yb/Dy, Ti, and Hf/Y) variability in WCT/CCT zircons. Where available, analyses of paired cores and rims are joined with lines. Eu/Eu\* is controlled by the fractionation of feldspar while Th/U, Yb/Dy, Hf/Y are sensitive to fractionating accessory phases, e.g. apatite, allanite, or chevkinite. Ti is used as a proxy for zircon crystallisation temperature following Ferry and Watson (2007). Typical analytical uncertainties are based on the reproducibility of natural zircon standards. All trace element data are given in the Supplementary material

slightly ( $\sim 1\%$ ) depleted rhyolitic melt ( $\delta^{18}\text{O} = 5.7\text{--}6.3\%$ ; Loewen and Bindeman 2016).

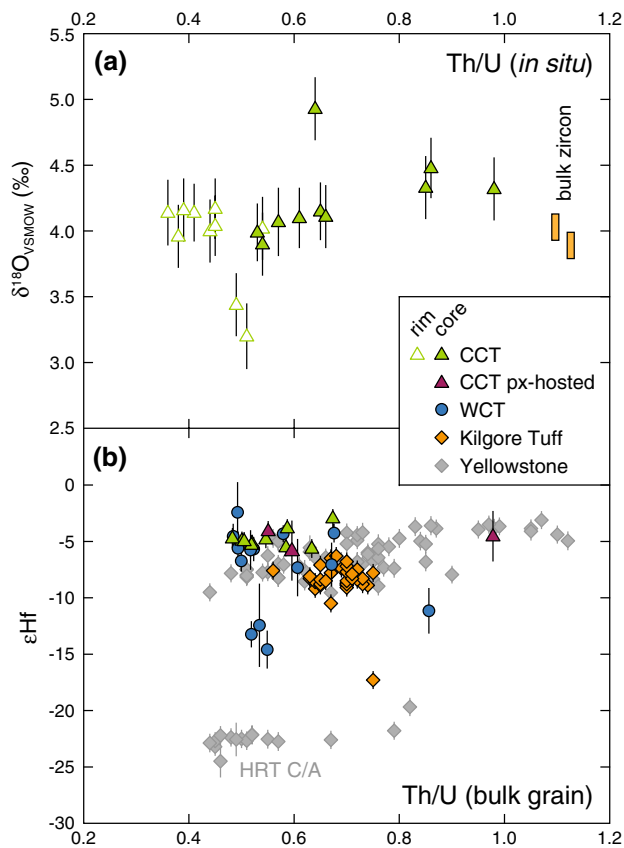
Hf isotope ratios in zircon from both CCT samples are homogeneous, while the WCT zircons cluster into at least two distinct populations in  $\epsilon\text{Hf}\text{--Th/U}$  space (Fig. 5b). Most WCT/CCT zircons belong to a main group at  $\epsilon\text{Hf} = -5.0 \pm 2.4$  ( $2\sigma$ ), the second WCT population averages  $\epsilon\text{Hf} = -12.9 \pm 2.5$  and includes three grains (WCT z6, z11, z21) that appear to be limited in age to  $\sim 5.65$  Ma as well as a high-Th/U grain (WCT z3) overlapping the eruption age (Table 1; Fig. 3). While no previous Hf isotope values are available for WCT/CCT, the Hf isotopic ratios of most zircons appear consistent with glass  $\epsilon\text{Nd}$  values determined by Nash et al. (2006) when related to  $\epsilon\text{Hf}$  using the ‘terrestrial array’ approximation (Vervoort et al. 1999) returning  $\epsilon\text{Hf} = -4.1$  for WCT and  $-4.9$  for CCT. The Hf isotopic ratios correlate neither with zircon trace element compositions nor age and are in broad agreement with  $\epsilon\text{Hf}$  values of most Yellowstone and Heise zircons measured previously (Wotzlaw et al. 2014, 2015). The presence of multiple zircon populations with distinct Hf isotope compositions in single eruptive units has been noted before in the Huckleberry Ridge Tuff (Wotzlaw et al. 2015; Fig. 5b) and may represent a common feature of Yellowstone–Snake River Plain magmas.

### Sr isotopic systematics

Single high-Mg# clinopyroxenes sampled from the Wolverine Creek Tuff, representing the previously described intermediate population remaining in major and trace elemental disequilibrium with the surrounding rhyolitic glass and mineral assemblage (Szymanowski et al. 2015), are also in Sr isotopic disequilibrium with WCT glass measured here and whole rock reported previously (Watts et al. 2011) (Fig. 6; Table 2). Two WCT glass aliquots analysed have  $^{87}\text{Sr}/^{86}\text{Sr}_i = 0.708893$  and  $0.708896$ , which is lower than whole-rock CCT ( $0.70894$ ) and WCT ( $0.70947$ ; Watts et al. 2011), both representing some of the least radiogenic YSRP rhyolites so far reported (Fig. 6). The glass has also lower  $^{87}\text{Sr}/^{86}\text{Sr}_i$  than the coexisting WCT/CCT plagioclase previously shown to be in elemental equilibrium (Szymanowski et al. 2015). We note, however, that the LA–MC–ICPMS method used to obtain the plagioclase  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios may overestimate the ratio in comparison with our preferred TIMS data.

The Sr isotopic ratios obtained from nine single high-Mg# WCT pyroxene grains and three multi-grain aliquots of CCT pyroxene range from  $0.707047$  to  $0.708827$  (grey band in Fig. 6). The mixed CCT pyroxene samples (Px11,





**Fig. 5** Trace elemental and isotopic compositions of WCT/CCT zircons. **a** In situ  $\delta^{18}\text{O}$  and Th/U of CCT zircon cores and rims compared to  $\delta^{18}\text{O}$  of zircon separates of Watts et al. (2011). **b** Bulk crystal  $\varepsilon\text{Hf}$  and Th/U of WCT and CCT zircons. Kilgore Tuff data are from Wotzlaw et al. (2014); Yellowstone zircon data including Snake River Butte, Huckleberry Ridge Tuff (HRT), Mesa Falls Tuff, and Lava Creek Tuff eruptions from Wotzlaw et al. (2015). All O and Hf isotopic data are provided in the Supplementary material

Px12, Px19) include both high-Mg# (50–260 ppm Sr) and low-Mg# rhyolitic pyroxenes (3–5 ppm Sr) at variable proportions (in Px19, mass balance requires 4–24% of high-Mg# pyroxenes); therefore, their  $^{87}\text{Sr}/^{86}\text{Sr}_i$  can be considered a mixture of two different signals strongly biased towards the high-Sr, high-Mg# intermediate pyroxene compositions. All  $^{87}\text{Sr}/^{86}\text{Sr}_i$  values measured in WCT/CCT mineral separates are significantly less radiogenic than the host glass, indicating a variable Sr isotopic disequilibrium between these pyroxene crystals and their surrounding melt at the time of eruption. Indeed, the WCT/CCT pyroxene measured here is less radiogenic than any YSRP rhyolites so far reported and has  $^{87}\text{Sr}/^{86}\text{Sr}_i$  values similar to the most radiogenic YSRP basalts found (Fig. 6b).

As a comparison, four multi-grain separates of low-Mg# clinopyroxene from other Heise rhyolites were analysed: the 6.6 Ma Blacktail Creek Tuff (BCT, 3 samples), the 6.2 Ma Lidy Hot Springs lava (LHS, 1), and the 4.5 Ma

Kilgore Tuff (KT, 1). Pyroxene from these units is universally near-homogeneous in major and trace elements, with compositions in equilibrium with the rhyolitic melt. The  $^{87}\text{Sr}/^{86}\text{Sr}_i$  values obtained (Table 2) are within the range of YSRP rhyolites (Fig. 6) and in good agreement with the whole-rock ratios of Watts et al. (2011). The only exception to this is the Blacktail Creek Tuff, where the mixed pyroxene separates bracket the bulk  $^{87}\text{Sr}/^{86}\text{Sr}_i$  value of 0.71238 with a spread of 0.71148–0.71299, indicating a possible isotopic disequilibrium within the pyroxene crystal cargo. Overall, the agreement between the isotopic ratios of pyroxene and whole rock from these rhyolites confirms previous results indicating a dominantly equilibrated nature of crystals residing in ‘typical’ YSRP magmas (Wolff et al. 2011) and reinforcing the unusual nature of the WCT pyroxenes.

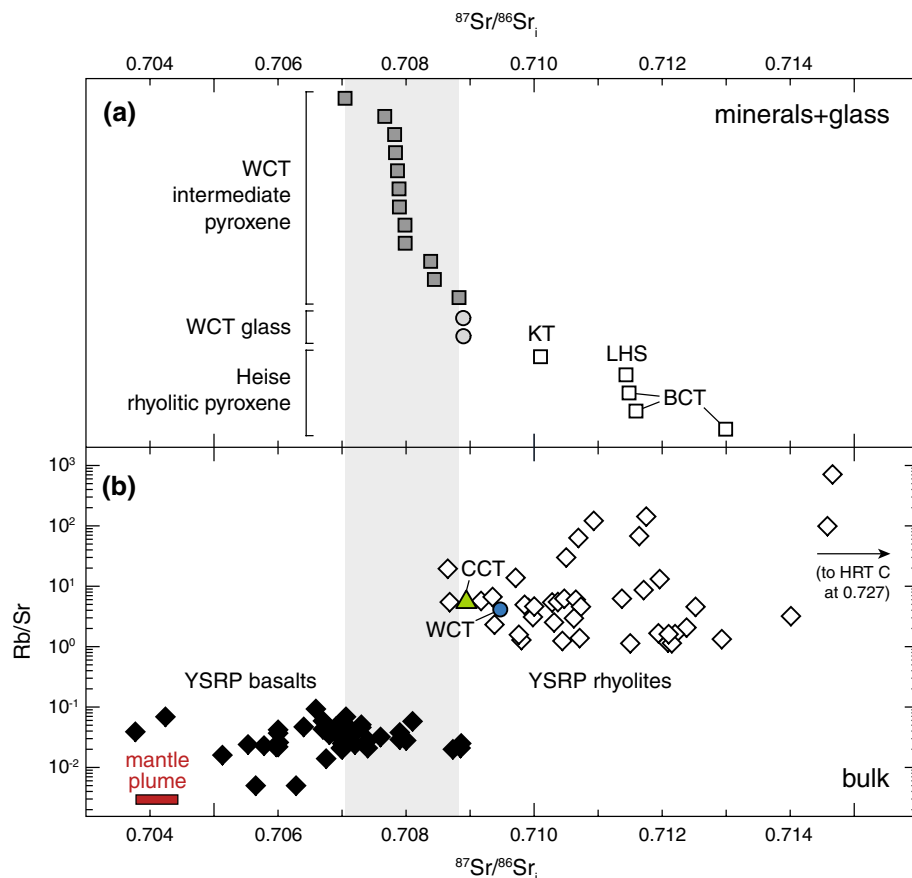
## Discussion

### Common Wolverine Creek–Conant Creek magma reservoir

Despite the differences in the field appearance of the two studied ignimbrites, the strong similarities observed in zircon and pyroxene data presented here indicate provenance of the CCT and the WCT from the same magma reservoir. The correspondence in individual zircon ages (Fig. 3), trace elements (Fig. 4), and Hf isotopic compositions of most zircon crystals (Fig. 5) from both eruptive units requires that all the autocrystic zircons crystallised from a similar (and similarly evolving) rhyolitic melt. While the zircon data set does record inter-crystal Hf isotopic heterogeneities, the age equivalence of the isotopically distinct populations supports the conclusion of a common petrogenesis. The new data support previous interpretations of the equivalence of the WCT and CCT magma reservoirs using glass, feldspar, and pyroxene compositions (Szymanowski et al. 2015), further reinforced by bulk isotope data (Watts et al. 2011) and the overlapping  $^{40}\text{Ar}/^{39}\text{Ar}$  eruption ages (Morgan and McIntosh 2005).

An important petrologic difference between the two units remains in the variable proportions of rhyolitic (low-Mg#) and intermediate, cumulate-derived, high-Mg# pyroxene crystals (Fig. 2), with the WCT being dominated by the intermediate component and the CCT hosting mostly low-Mg# pyroxene (Szymanowski et al. 2015). Reconciling such variations with the uniform melt composition in the WCT and the CCT requires that a recharge bringing the intermediate crystal cargo into the rhyolitic melt is not fully mixed into it, but rather erupted first as the cumulate material-dominated, precursor WCT, followed by the (more voluminous?) rhyolitic material-dominated CCT. We

**Fig. 6** Strontium isotopic results for pyroxene and glass compared with whole-rock isotopic ratios of YSRP basalts and rhyolites. **a** Ranked  $^{87}\text{Sr}/^{86}\text{Sr}_i$  for pyroxene and glass analysed by TIMS (data presented in Table 2). *KT* Kilgore Tuff, *LHS* Lidy Hot Springs rhyolite, *BCT* Blacktail Creek Tuff. **b** Compilation of whole-rock isotopic data for YSRP basalts and rhyolites; SRP olivine tholeiite data are from Leeman and Manton (1971), Yellowstone basalts and rhyolites from Hildreth et al. (1991), Heise rhyolites from Watts et al. (2011), central SRP rhyolites from Bonnichsen et al. (2008). Note that a few very radiogenic outliers in the rhyolite field reach higher ratios outside of the plot and up to 0.72685 (Huckleberry Ridge Tuff C; Hildreth et al. 1991). Uncertainties are smaller than symbol sizes. The ‘mantle plume’ represents the most primitive ‘Imnaha component’ approximating the composition of the Yellowstone mantle plume (Wolff et al. 2008)



propose the WCT may be viewed as an early first phase of a dual WCT/CCT eruption where the welding differences are mainly due to variable fragmentation, transport, and deposition conditions.

### Timescales of shallow-crustal magma accumulation

High-precision U–Pb geochronology of single zircons from the WCT/CCT system reveals a history of protracted zircon crystallisation prior to eruption (Fig. 3). We consider the youngest  $^{206}\text{Pb}/^{238}\text{U}$  date of  $5.5941 \pm 0.0097$  Ma as the best estimate for the eruption age of both units. The apparent range of individual zircon crystallisation ages within the larger WCT data set spans  $92 \pm 14$  ky, while the bulk-rock-separated CCT zircons record an apparent spread of  $23 \pm 11$  ky when excluding two relatively imprecise data points at both ends of the age spectrum (CCTz10, z8; Fig. 3). However, taking all the analysed crystals as representative of the same magmatic system, we can consider the larger range of  $92 \pm 14$  ky as our best (minimum) estimate of overall time the rhyolitic melt spent continuously or intermittently zircon-saturated. During this time, the magma crystallised pyroxene, feldspar, and accessory apatite, allanite, and/or chevkinite as indicated by the consistent core–rim trace

element zoning profiles in zircon (Fig. 4). The dating of pyroxene-hosted zircons provides an additional clue about the timescales of pyroxene crystallisation in the system; the zircon dates require that many low-Mg# pyroxenes crystallised not earlier than  $45 \pm 27$  ky prior to eruption. This result shows that in Snake River-type systems, zircon may co-crystallise with or predate major phases such as pyroxene, consistent with near-liquidus zircon saturation.

The minimum estimate of  $92 \pm 14$  ky pre-eruptive magma accumulation lends itself to comparison with results from similar systems, such as the Kilgore Tuff erupted from the same Heise eruptive centre ( $10^3$ – $10^4$  years; Wotzlaw et al. 2014) and the super-eruptions of Yellowstone ( $10^3$ – $10^4$  years; Rivera et al. 2014; Wotzlaw et al. 2015). In these cases, the authors interpreted the apparent spreads in individual zircon ages as representing a rapid magma assembly and short storage of upper crustal silicic reservoirs. While such interpretation is plausible also for the WCT/CCT, we note that the  $92 \pm 14$  ky estimate is necessarily a minimum; with zircon a near-liquidus phase, the system may have undergone multiple heating events cyclically pushing it into zircon undersaturation (Cooper and Kent 2014), thus repeatedly crystallising and dissolving zircon crystals (e.g. Bindeman and Melnik 2016).

**Table 2** Strontium isotopic compositions of pyroxene and glass analysed by TIMS

Sample	Unit	No. grains/mass	Rb (ppm)	Sr (ppm)	<sup>87</sup> Rb/ <sup>86</sup> Sr	<sup>87</sup> Sr/ <sup>86</sup> Sr	±2σ	Age (Ma)	<sup>87</sup> Sr/ <sup>86</sup> Sr <sub>i</sub>
<i>Rhyolitic glass</i>									
WCT-GL3	Wolverine Creek Tuff	7.1 mg	159.2	15.6	27.92	0.711113	0.000042	5.6	0.708893
WCT-GL4	Wolverine Creek Tuff	9.4 mg	131.8	12.7	30.09	0.711289	0.000062	5.6	0.708896
<i>Rhyolitic (low-Mg#) pyroxene</i>									
BCT-1	Blacktail Creek Tuff	multiple	<1	9 ± 1	0.50	0.711638	0.000074	6.6	0.711591
BCT-2	Blacktail Creek Tuff	multiple	2.0	10.8	0.52	0.713041	0.000035	6.6	0.712992
BCT-3	Blacktail Creek Tuff	multiple	1.7	10.8	0.46	0.711526	0.000028	6.6	0.711483
LHS	Lidy Hot Springs rhyolite	multiple	<1	8 ± 2	0.50	0.711479	0.000016	6.2	0.711435
KT	Kilgore Tuff	multiple	<1	8 ± 1	0.50	0.710132	0.000009	4.5	0.710100
<i>WCT/CCT pyroxene</i>									
Px7	Wolverine Creek Tuff	single	<1	106 ± 36	0.10	0.707055	0.000039	5.6	0.707047
Px9	Wolverine Creek Tuff	single	<1	106 ± 36	0.10	0.708392	0.000033	5.6	0.708384
Px10	Wolverine Creek Tuff	single	<1	106 ± 36	0.10	0.707874	0.000031	5.6	0.707866
Px13	Wolverine Creek Tuff	single	3.0	71.8	0.12	0.708837	0.000021	5.6	0.708827
Px14	Wolverine Creek Tuff	single	7.5	215.6	0.10	0.707994	0.000021	5.6	0.707986
Px15	Wolverine Creek Tuff	single	6.3	236.1	0.08	0.707902	0.000013	5.6	0.707896
Px16	Wolverine Creek Tuff	single	6.8	173.1	0.11	0.708451	0.000049	5.6	0.708442
Px17	Wolverine Creek Tuff	single	3.6	87.4	0.13	0.707677	0.000006	5.6	0.707667
Px18	Wolverine Creek Tuff	single	3.8	100.2	0.11	0.707830	0.000008	5.6	0.707821
Px11	Conant Creek Tuff	multiple	–	–	0.70	0.707891	0.000034	5.6	0.707835
Px12	Conant Creek Tuff	multiple	–	–	0.70	0.708039	0.000038	5.6	0.707983
Px19	Conant Creek Tuff	multiple	3.5	14.4	0.71	0.707947	0.000010	5.6	0.707890

For WCT samples with high-Sr contents of pyroxene, single grains were analysed; rhyolitic (low-Mg#) pyroxene as well as mixed low-/high-Mg# pyroxene samples of CCT (Px11, Px12, Px19) were larger aliquots of 10–20 grains amounting to a mass of 2–4 mg. Rb and Sr were measured by ID-TIMS for spiked samples; where not available, average concentrations from LA-ICPMS analyses of pyroxene from the same population are given in italics. No estimate is given for Px11 and Px12 as these samples are mixtures of low- and high-Mg# pyroxene. For the purpose of age correction, the <sup>87</sup>Rb/<sup>86</sup>Sr of some samples is also assumed based on similarity to other samples and given in italics. Initial <sup>87</sup>Sr/<sup>86</sup>Sr ratios (<sup>87</sup>Sr/<sup>86</sup>Sr<sub>i</sub>) are calculated using the measured or assumed <sup>87</sup>Rb/<sup>86</sup>Sr and eruption ages of Morgan and McIntosh (2005) with the decay constant  $\lambda(^{87}\text{Rb}) = 1.42 \times 10^{-11} \text{ year}^{-1}$  (Steiger and Jäger 1977)

The range in zircon ages could record either (1) the entire lifetime of the erupted magma chamber, or (2) only the last cooling event in an extended life of a heterogeneous upper crustal magma reservoir. Long storage (>10<sup>5</sup> years) of large volumes of magma has been demonstrated for continental arc-related silicic eruptions where magma reservoirs are thought to be stored as near-solidus crystal-rich mush bodies (Vazquez and Reid 2004; Bachmann et al. 2007; Wotzlaw et al. 2013). We speculate that YSRP rhyolitic magmas, while being in many respects different from such arc-related systems (e.g. mineralogy, crystal content of erupted material, volatile contents), may be characterised by broadly comparable storage/crystallisation times. In the case of YSRP, such extended residence in the upper crust may be facilitated by the formation of refractory and mechanically strong mush zones (in places with cumulate characteristics) around the crystal-poor, eruptible melt pockets, partly insulating them from the cold pre-existing wall rocks (Ellis et al. 2014).

### Isotopic heterogeneities and the upper crustal architecture of the magma reservoir

Zircons from the WCT/CCT cluster into at least two populations of contrasting Hf isotopic compositions, while overlapping in age within the resolution of our high-precision geochronology. Such relations require the existence of two or more isotopically distinct rhyolite reservoirs that co-existed at the time of eruption and derived their isotopic compositions from variable amounts of assimilation of radiogenic Archean crust during crustal differentiation. A simple two-component mixture model assuming an asthenospheric mantle plume source (Wotzlaw et al. 2015) requires ca. 20 and >30% assimilation of radiogenic Archean crust for the two groups of WCT/CCT zircons with  $\epsilon\text{Hf} \sim -5$  and  $-13$ , respectively. The consistency in trace element compositions between the zircon populations of variable  $\epsilon\text{Hf}$  suggests similar melt evolution paths with consistent fractionating mineral assemblages, and points

towards similar shallow storage conditions of multiple physically separated batches of melt that either became connected before the eruption or were mixed only during the eruption. Such small-scale sub-chambers can be imagined as multiple comparably sized melt batches or a single dominant melt region with a range of small storage volumes adjacent to chamber roof or walls that would also display variable responses to recharge, crystallisation and wall-rock assimilation. Similar complex multi-batch magmatic architectures of variable proportions have been extensively documented in the Yellowstone–Snake River Plain province based on multiple distinct co-erupted glass or mineral compositional groups (Cathey and Nash 2004; Ellis and Wolff 2012; Ellis et al. 2014; Wotzlaw et al. 2014, 2015) and may represent a common feature of many large-volume volcanic systems worldwide (e.g. Reubi and Nicholls 2005; Shane et al. 2008; Cooper et al. 2012; Bégué et al. 2014).

### Isotopic record of crustal assimilation

Evaluating the extent of crustal assimilation in the WCT/CCT recorded by both pyroxenes and zircons requires knowledge of the isotopic characteristics of both the crust and the initial mantle source regions. The bulk of the crust underlying much of the Snake River Plain belongs to old Precambrian basement domains, with the eastern SRP crossing into the Archean Wyoming craton (Foster et al. 2006; McCurry and Rodgers 2009). Sampling of xenoliths within YSRP mafic lavas reveals a spectrum of granulite facies metamorphic lithologies yielding zircon dates between 2.5 and >3.5 Ga (Wolf et al. 2005), variably radiogenic  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios between 0.703 and 0.893,  $\epsilon\text{Nd}$  of  $-52$  to  $-23$  (Leeman et al. 1985; McCurry and Rodgers 2009), and ‘normal’ oxygen isotope signatures of  $\delta^{18}\text{O} = 6\text{--}9\text{‰}$  (Watts et al. 2010). In contrast, most of the uppermost crust is thought to be made up of young volcanic rocks produced by the Yellowstone hotspot itself, providing limited leverage in radiogenic isotope ratios (Fig. 6) but exposed to hydrothermal circulation and as such having the potential of delivering large amounts of  $^{18}\text{O}$ -depleted material that can be assimilated by new magmas (e.g. Friedman et al. 1974; Hildreth et al. 1984; Bindeman and Valley 2001; Watts et al. 2011; Boroughs et al. 2012). The isotopic composition of the mantle-derived melts can be approximated by the compositions of YSRP olivine tholeiites (Fig. 6); however, their relatively radiogenic nature ( $^{87}\text{Sr}/^{86}\text{Sr} = 0.704\text{--}0.709$ ) suggests that much of the radiogenic isotope signal in the YSRP melts may be derived already from deep plume–lithosphere interaction (Doe et al. 1982; Hanan et al. 2008; Jean et al. 2014).

As presented above, the WCT/CCT has one of the lowest bulk and glass  $^{87}\text{Sr}/^{86}\text{Sr}$  and  $\epsilon\text{Nd}$  among YSRP rhyolites (one of the most primitive YSRP rhyolites; Fig. 6). It also

displays a normal to moderately light O isotope composition (Watts et al. 2011). Both these facts suggest relatively minor assimilation of both Archean source materials and previously erupted (and possibly hydrothermally altered) volcanic materials (cf. Watts et al. 2011), assuming an initial melt similar to the relatively radiogenic YSRP basalts. The WCT/CCT magma therefore seems to have evolved dominantly through polybaric fractional crystallisation, with the limited isotopic shift more likely achieved by assimilating small amounts of fertile Archean crust rather than young, shallow volcanic rocks. Localised portions of melt at  $\epsilon\text{Hf} < -10$  (as recorded by WCT/CCT zircons) additionally support a prevailing role of Archean source material as assimilant.

A chemical evolution dominantly controlled by fractional crystallisation is further supported by the unique record of ‘intermediate’ WCT pyroxenes, which span a range of compositions between Mg# 60–90 corresponding to equilibrium crystallisation from mafic to intermediate melts (Szymanowski et al. 2015) while covering a range of  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios typical for the most radiogenic YSRP basalts to the least radiogenic YSRP rhyolites. The single-grain Sr isotopic record thus indicates dominantly lower- to mid-crustal assimilation consistent with previous petrological conclusions about intermediate storage and differentiation levels (Szymanowski et al. 2015) as well as a growing amount of geophysical evidence for high-crystallinity, low-melt fraction magmatic bodies occurring throughout the crustal column in association with eruptive centres of the Yellowstone–Snake River Plain volcanic province (Sparlin et al. 1982; Peng and Humphreys 1998; DeNosaquo et al. 2009; Farrell et al. 2014; Huang et al. 2015).

### Conclusions

Our combined study of zircon and pyroxene crystals in the Wolverine Creek–Conant Creek system reveals a complex record of magma–crust interaction in a crustal-scale magmatic system and helps to paint a picture of the evolution of Yellowstone–Snake River Plain magmas when unaffected by the shallow-crustal influence of hydrothermally altered materials. The compositions of the high-Mg# pyroxene and melt inclusions support the predominance of fractional crystallisation processes in producing the rhyolites, which is consistent with geophysical imaging of high-crystallinity intermediate storage levels underlying the YSRP eruptive centres throughout the whole crust. The new data set of Sr isotopes in WCT/CCT pyroxene presented here shows that (1) the amount of assimilation of Archean crust into the YSRP melts is limited, and (2) it may occur dominantly in the lower- and mid-crust while the melt evolves from primitive basalts towards more silicic compositions.

Variably radiogenic Hf isotope ratios in zircon between  $\epsilon_{\text{Hf}} = -2$  and  $-15$  indicate that the WCT/CCT was derived by amalgamation of at least two isotopically distinct melt domains derived by assimilating slightly different amounts of Archean crust. Such melts evolved towards rhyolitic compositions finally stored at a shallow-crustal level where they retained zircons for a minimum of  $92 \pm 14$  ky. The final WCT/CCT eruption sampled an upper crustal magma reservoir in which isotopically distinct but compositionally similar rhyolitic liquids were stored separately prior to mixing upon eruption. Such multi-batch architectures of magmatic reservoirs appear to be a common feature of high-temperature rhyolites of the Yellowstone–Snake River Plain area and possibly other volcanic systems in similar tectonic settings.

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