

Application of U–Pb detrital zircon geochronology to drill cuttings for age control in hydrocarbon exploration wells: A case study from the Rukwa Rift Basin, Tanzania

Hannah Hilbert-Wolf, Eric Roberts, Bob Downie, Cassy Mtelela, Nancy J. Stevens, and Patrick O'Connor

ABSTRACT

Precise dating and correlation of drilled wells through continental successions is challenging for hydrocarbon exploration, especially where preservation and recovery of age-diagnostic fossils is poor. As a complement or alternative to biostratigraphic dating we demonstrate the effectiveness of U–Pb geochronology via laser ablation–inductively coupled plasma–mass spectrometry on detrital zircon from well cuttings. In basins with syndepositional volcanic input, the youngest zircons in a stratigraphic interval can refine and serve as a proxy for the age of deposition. We demonstrate the reliability of this technique when applied to hydrocarbon exploration wells by analyzing drill cuttings through a continental interval of the Galula-1 well in the Rukwa Rift Basin, East African rift system, Tanzania, which previously yielded conflicting biostratigraphy results. The lower third of the well section reveals a late Miocene to Pliocene up-hole younging trend in the youngest detrital zircon populations, which matches new radioisotopic ages on volcanic tuffs from a correlative outcrop section. This is followed by an interval with recycled young zircons, followed by a zircon-free interval, interpreted to correspond to changes in magma composition of the nearby Rungwe volcanic province. This study provides the first radioisotopic age constraints for the Lake Beds in the Rukwa rift

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and demonstrates that sedimentation in the basin began by 8.7 Ma, critical for burial and thermal history modeling and establishing the probability of a working hydrocarbon system. Correspondence in age and zircon preservation between well and outcrop samples from the same intervals provides strong support for applying U–Pb detrital zircon geochronology to well cuttings, as a rapid, inexpensive approach for hydrocarbon exploration.

INTRODUCTION

Critical decisions made during exploration and field development of prospective hydrocarbon bearing plays are underpinned by accurate stratigraphic control and correlation within and between wells. Biostratigraphy, the conventional method for such dating and correlation of sedimentary rocks, is not always possible or reliable. For example, oxidized red-bed successions and other, typically continental, intervals commonly yield depauperate microfossil and/or pollen assemblages and in some cases lack preservation altogether. Accurate dating and correlation of sequences where microflora and fauna are limited or absent is now more critical than ever, as global hydrocarbon exploration moves to consider biostratigraphically lean frontier basins.

As one of the next hydrocarbon frontiers, East Africa hosts a suite of prospective rift basins with characteristics amenable to hydrocarbon generation, including deep depocenters with rapid sediment accumulation and high heat flow, promoting early thermal maturation (petroleum kitchens; Tiercelin et al., 2012). However, an absence of reliable index fossils in these nonmarine, intracratonic rift basins commonly limits the use of traditional biostratigraphic dating methods. Assessing essential elements of the petroleum system such as basin structure, composition and architecture of sedimentary successions, and thermal history (Cohen, 1989; Nelson et al., 1992; Morley, 1995) hinges on the accurate dating of sedimentary successions, and where biostratigraphy is poor, alternative means of dating are important. Rift settings, in particular, require temporal resolution for understanding their complex tectonic histories and the timing between such key events as rift lake development, volcanism, and uplift, all of which have a major effect on hydrocarbon prospectivity. Recent onshore successes, such as hydrocarbon discoveries in the Turkana (Lokichar) and Albertine Rift Basins, have renewed interest in exploring other rift segments along the East African rift system (EARS). In particular, the western branch of the EARS has seen considerable hydrocarbon exploration since 2007, when the Kingfisher-1 discovery in the Albertine Graben demonstrated the commercial hydrocarbon potential of these rift basins (Logan et al., 2009).

Other innovative approaches to correlation and depositional age approximation have been used successfully, such as chemostratigraphic techniques (Ratcliffe et al., 2015), samarium–neodymium isotope analysis (Dalland et al., 1995), and fission track dating (Carter et al., 1995), though not all of these approaches provide age constraint. Radioisotopic dating of ash beds in core samples, or correlation of wells to dated ash beds in outcrop, is the most common alternative for dating sedimentary units where biostratigraphy is problematic or greater precision is required. In recent years the increasing throughput and decreasing costs of laser ablation–inductively coupled plasma–mass spectrometry (LA–ICP–MS) U–Pb detrital zircon geochronology have opened this technique to a much greater number of applications. In particular, recent papers have highlighted the potential of dating large numbers of detrital zircons from sedimentary units with the express intent of identifying the youngest population of grains as a means of constraining the maximum depositional age (Dickinson and Gehrels, 2009; Lawton et al., 2010; Tucker et al., 2013). Although studies have focused on the application of detrital zircons and other minerals for maximum depositional age constraint, it is noteworthy that in petroleum geology, a field for which this approach has great potential, there has been limited application of this technique to date. This is perhaps because of the uncertainty of applying the technique to well cuttings (for which sample sizes are typically limited) and concerns about possible contamination issues, as well as issues associated with proprietary data.

A primary concept in the use of detrital zircons for dating the deposition of sediments is that the radiometric ages of zircons derived from volcanic sources record the date of the eruptive event(s). The technique is most useful where eruptive volcanic events occur in proximity to active sedimentary basins, and there is an expectation that the erupted zircons are essentially coeval in age to the sediments into which they are deposited. This assumption is likely to widely pertain to much of the EARS. Thus, when the ages of zircon populations within the sediments are determined, the older populations, although useful in provenance studies, can be excluded, and the youngest population can be used to define the maximum age of sediment deposition. In such cases, the maximum depositional age (i.e., the age of the youngest zircon population, which the sediments cannot be older than) is a proxy for the depositional age of the sedimentary unit from which the zircons derive (Dickinson and Gehrels, 2009; Lawton et al., 2010; Tucker et al., 2013).

Here we apply detrital zircon geochronology to systematically investigate maximum depositional ages from well cuttings through the Miocene–Holocene Lake Beds succession in the Galula-1 well in the Rukwa Rift of Tanzania (Figure 1). This work has been undertaken in combination with stratigraphic

DATASHARE 76

Tables S1–S12 are available in electronic versions on the AAPG website (www.aapg.org/datashare) as Datashare 76.

EDITOR'S NOTE

Color versions of Figures 1–3 can be seen in the online version of this paper.

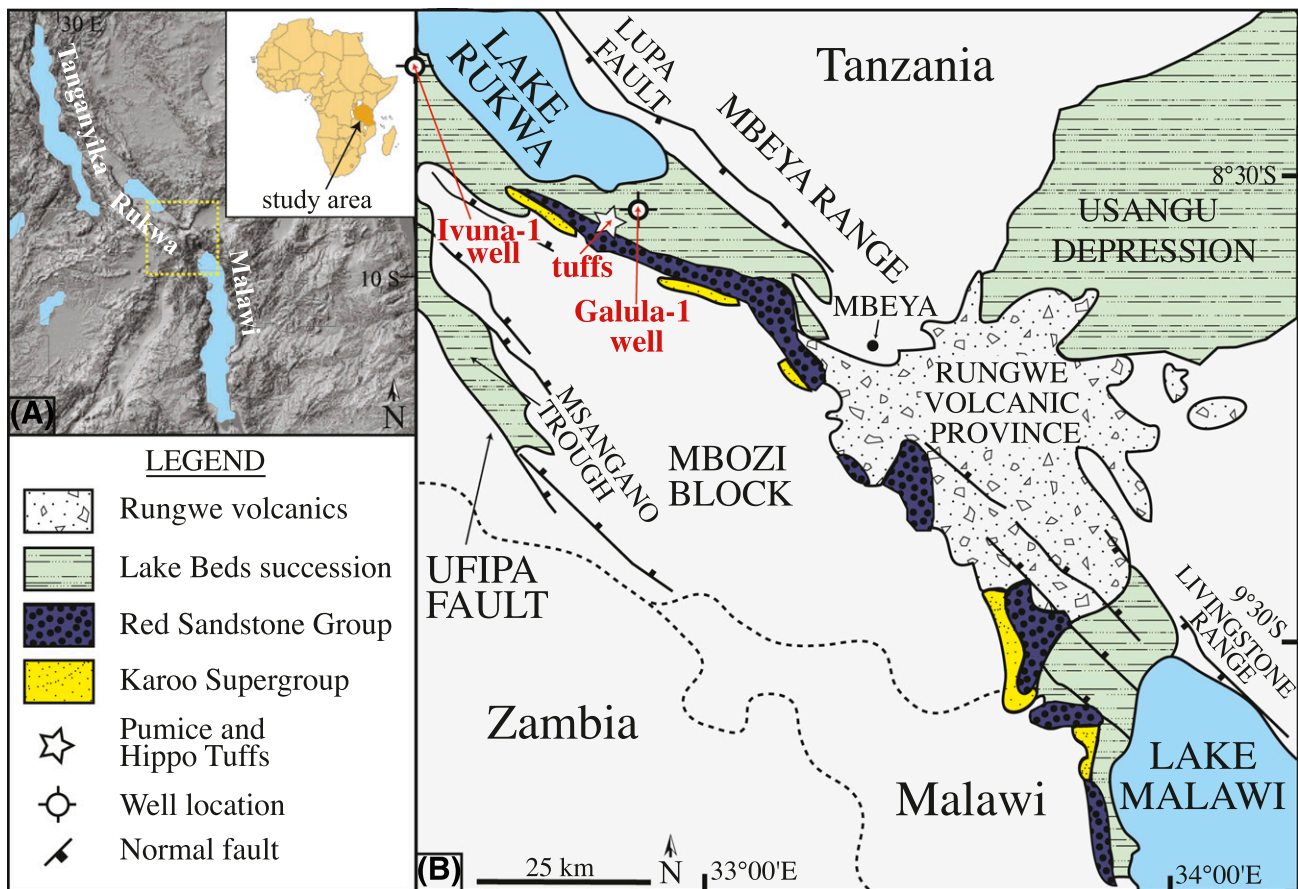


Figure 1. (A) Map of the Tanganyika–Rukwa–Malawi rift segment (western branch, East African rift system), modified from the National Aeronautics and Space Administration Shuttle Radar Topography Mission collection. Inset map shows the location of the study area within Africa, and the yellow dotted box shows the location of the map in (B). (B) Geologic map of Phanerozoic sedimentary deposits in the southern Rukwa Rift and northern Malawi Rift Basins. The sample sites are labeled. Scale: 25 km = 15.5 mi. Note: A color version can be seen in the online version.

investigations and radioisotopic dating of volcanic tuffs from a control section logged through the Lake Beds succession in outcrop. This approach is designed to provide an independent test of this method via correlation between the subsurface and outcrop. This paper evaluates the application of U–Pb detrital zircon geochronology for age control of stratigraphic units and correlation in petroleum exploration wells, which has great potential for refining depositional ages of continental sedimentary basins around the world that are commonly notorious for ambiguous biostratigraphy and poor age constraint. Using these ages in combination with absolute ages dated herein from newly discovered volcanic tuffs in outcrop, we are able to delineate the timing of sedimentation (and, therefore, late Cenozoic rifting) in this part of the western branch of the EARS for the first time. These results are highly encouraging and strongly

support the potential for applying U–Pb detrital zircon geochronology via LA–ICP–MS to well cuttings in certain basin types as an effective exploration tool in petroleum geology for improving temporal resolution in wells through poorly constrained intervals.

RUKWA RIFT BASIN GEOLOGY

The western branch of the EARS is characterized by a series of linked, elongate sedimentary basins that typically follow preexisting Paleoproterozoic basement structures. The Rukwa Rift Basin in southwestern Tanzania is an approximately 300-km (186-mi)-long by approximately 50-km (31-mi)-wide, half-graben basin occupying the middle of the Tanganyika–Rukwa–Malawi (TRM) rift segment, which forms the northwest-trending southern part of the western

branch (Figure 1). The TRM rift segment has been affected by repeated rifting cycles and fault reactivation from the Paleozoic to Holocene (Delvaux et al., 2012). Initiation of Cenozoic rifting in the western branch of the EARS began by 25 Ma with the opening of the Rukwa Rift (Roberts et al., 2012), followed by development of the Tanganyika Rift between 12 and 9 Ma (Cohen et al., 1993) and the Malawi Rift by at least 8.6 Ma (Ebinger et al., 1989, 1993).

The Rukwa Rift Basin preserves a major sedimentary succession (at least 8 km [5 mi] thick), representing a rich archive of tectonic and environmental change (Roberts et al., 2012, and references therein). Paleozoic to Holocene rifting and sedimentation are recorded in the basin by a suite of sedimentary deposits, which are subdivided into the (1) Permian–Triassic Karoo Supergroup, (2) Cretaceous Galula Formation, (3) Oligocene Nsungwe Formation, and (4) Miocene–Holocene Lake Beds succession. The last of these depositional sequences, the Miocene–Holocene Lake Beds succession, is the focus of the case study presented herein and is the primary target for ongoing hydrocarbon exploration in the rift. The Lake Beds succession is best exposed on the basin margins, where it is composed of variably consolidated conglomerates, sandstones, siltstones, and mudstones, along with intercalated volcanic tuffs. Volcanic-rich fluvial, alluvial, and deltaic deposits are common at the peripheral basin exposures, and the depocenter is modeled as being composed of finer-grained, deeper water lacustrine sequences (Kilembe and Rosendahl, 1992). The Miocene–Holocene Rungwe volcanic province on the southern margin of the basin covers greater than 1500 km² (932 mi²) (Figure 1), where it developed along a complex tectonic junction between multiple rift branches, including the Rukwa Rift, Usangu Basin, and northern Malawi Rift. The sedimentary strata of the Lake Beds are largely derived from the uplifted metamorphic basement rock and from the Rungwe volcanics.

This study presents the first absolute age constraints on the deposition of the Lake Beds succession. A long history of conflicting age interpretations for strata in the rift exists, though it has generally been accepted that the Lake Beds are Neogene to Pleistocene in age (Stockley, 1938; Spence, 1954). Quenell et al. (1956) suggested the Lake Beds were dominantly Pleistocene strata, whereas Pentelkov (1979) assigned the lower part of the Lake Beds to

the Cretaceous. Published geological survey maps over the area (e.g., Van Loenen and Kennerley, 1962) ascribe a Neogene age to the Lake Beds. More recently, outcrop investigations of the uppermost part of the Lake Beds have proposed a latest Quaternary to Holocene age for the upper 50–100 m (164–328 ft) of the formation (Cohen et al., 2013; Hilbert-Wolf and Roberts, 2015; Mtelega et al., 2016), and further biostratigraphic analyses are underway by our research group. In 1987 the Amoco Tanzania Oil Company drilled two exploration wells in the Rukwa Rift Basin, Ivuna-1 and Galula-1. In the published biostratigraphic analysis of these wells, Wescott et al. (1991) were only able to assign broad ages, suggesting that the Lake Beds are for the most part Pliocene to Holocene, based on a limited range of palynomorphs and diatoms. These workers also assigned Miocene to Holocene ages to the underlying Red Sandstone Group, which has now been dated as Cretaceous to Oligocene based on extensive vertebrate biostratigraphy and high-precision radioisotopic dating (Roberts et al., 2010, 2012; Stevens et al., 2013). Stimulated by the significant Neogene discoveries in Turkana (Lokichar) and Albertine Basins, the Rukwa Rift Basin is once again a focus for investigation of its hydrocarbon potential. A critical element in the determination of the Lake Beds prospectivity, particularly source rock maturation, is clear resolution of the formations age(s). Thus, an unconventional approach for dating the well sections was sought, and the possibility of using detrital zircon LA-ICP-MS U–Pb geochronology to refine the temporal resolution of this unit was investigated.

METHODS

Laser Ablation–Inductively Coupled Plasma–Mass Spectrometry U–Pb Geochronology

The supply of cuttings from the Galula-1 and Ivuna-1 wells was very limited, so sample sizes were increased by aggregating several individual cuttings samples over depth ranges from 46 to 100 m (150 to 360 ft; average 69 m [226 ft]) to enhance zircon yield. Twelve composited samples were selected from the Lake Beds succession over the interval 960–27 m (3150–90 ft) in the Galula-1 well (0489729

E 9053938.8 N; Universal Transverse Mercator [UTM] zone 36, ARC 1960 datum), with each sample initially weighing between 150 and 500 g. Two samples from the Ivuna-1 well (0423732.4 E 9087313.4 N; UTM zone 36, ARC 1960 datum) were also investigated for a pilot study, but zircon yield was low, likely reflecting the dominance of fine-grained lacustrine shale facies closer to the paleodepocenter. In outcrop, tuffs of the Lake Beds succession are distinctive and were identified by their clay-dominated composition, including relict pumice shapes and textures (now devitrified). The tuffs range in color from white, to gray, to brown, to purple. The basal, phenocryst-rich horizons were targeted for sampling.

Samples were crushed and milled (when necessary) in a tungsten carbide disc mill and sieved at various size fractions, with all grains less than 500 μm recovered to enhance the possibility of sampling the widest range of zircon sizes, including distal volcanic ash derived zircons. Heavy minerals were separated using lithium polytungstate adjusted to a specific gravity of 2.85. Mineral separates were then washed and dried, and a hand magnet was used to remove strongly magnetic minerals, followed by a Frantz magnetic separator at progressively higher magnetic currents of 0.5, 0.8, 1.2, and 1.4 A, set at a constant 10° side slope. The nonmagnetic heavy mineral separates were treated according to hybrid selection protocols that involved handpicking zircons as randomly as possible from the greater population within a defined field of view. Following this, the remainder of each sample was handpicked a second time with the intention of selecting the clearest, most euhedral grains remaining in each sample, to increase the likelihood of analyzing age-constraining young zircons. Depending on sample size and zircon yield, approximately 60–100 grains (or as many as were recovered, if less than this) were mounted in a 25-mm (1-in.) epoxy resin puck, polished to expose their midsections, and imaged using a Jeol JSM5410LV scanning electron microscope with attached cathodoluminescence detector to document microstructures, cracks, inclusions, inherited cores, and other zircon grain complexities.

All LA-ICP-MS U-Pb analyses were conducted at the Advanced Analytical Centre of James Cook University, using a Coherent GeolasPro 193-nm argon fluoride Excimer laser ablation system connected to a Bruker 820-MS (formerly Varian 820-MS). Zircons were analyzed using the same optimized LA-ICP-MS

method outlined in Tucker et al. (2013). Laser spot sizes ranged between 32 and 44 μm according to the size and morphology of sample and standard zircons. However, for a given set of standard and unknown analyses, spot size remained constant. The total measurement time was set at 70 s per analysis. The first 30 s were for gas blank measurement, and during the final 40 s the shutter was opened to allow for sample ablation and measurement. At the beginning and end of each sample, as well as after every 10 or so unknown grains, at least two analyses each of the primary zircon standard GJ1 (608.5 ± 0.4 Ma, 2σ ; Jackson et al., 2004) and the secondary zircon standards Temora-2 JCU (416.8 Ma ± 1.1 Ma, 2σ ; Black et al., 2003) and Fish Canyon Tuff (28.5 ± 0.03 Ma, 2σ ; Schmitz and Bowring, 2001) were completed to monitor down-hole fractionation and for age and instrument drift corrections. All secondary standard analyses were within 1%–2% of the expected ages. Because of instrumental drift during the course of a day, different calibrations were used to correct for this, including either linear or average fitting of the standards. National Institute of Standards and Technology Standard Reference Material 610 or 612 was analyzed at the beginning, middle, and end of each session for the purpose of calibrating thorium and uranium concentrations. Data reduction and age calculations based on measured isotope ratios were carried out using the Glitter software (Van Achterbergh et al., 2001). All time-resolved isotope signals were filtered for signal spikes or perturbations related to inclusions and fractures, and then background and sample intervals were selected based on evaluation of the most stable and representative isotope ratios. Isoplot/Ex version 4.15 (Ludwig, 2012) was used for extracting ages from multigrain populations (i.e., calculating weighted means). The $^{206}\text{Pb}/^{238}\text{U}$ ages have been reported for zircon grains younger than 1.0 Ga, and $^{207}\text{Pb}/^{206}\text{Pb}$ ages are preferred for zircon grains older than 1.0 Ga. Where applicable, $^{206}\text{Pb}/^{238}\text{U}$ ages were corrected for common Pb based on the Pb isotope evolution model of Stacey and Kramers (1975). Zircon grains with an age discordance greater than or equal to 30% were omitted from the samples and from this study as a whole. However, in young zircon grains (e.g., <10 Ma) the production of radiogenic Pb is typically low, and hence, the $^{207}\text{Pb}/^{206}\text{Pb}$ age can be discordant, so the above criterion was not applied.

Recommendations for Sampling

Sampling a well or borehole for LA-ICP-MS U-Pb zircon geochronology is dictated by the availability of well cuttings or cores and by the project-specific aims of a dating campaign. A few general strategies may maximize the likelihood of obtaining reliable maximum depositional ages. Sandy intervals are sometimes more likely to contain zircon than muddy intervals, and similarly, volcanoclastic sandstones and tuffs are preferred because if the volcanic source was zircon-fertile, these units are likely to contain depositional age-defining volcanic zircons. A potential limitation to applying this approach to cuttings from existing wells is the small sample sizes generally available (commonly <200 g per sample per depth interval), in comparison to outcrop detrital zircon samples, where approximately 2–5 kg (4.4–11 lb; or more) of a sample is typically collected. The retention and collection of larger well cutting samples can be prearranged when detrital zircon dating is planned prior to drilling. A large sample from each targeted depth would be preferable to combining smaller-volume well cuttings over longer intervals, to minimize mixing and to increase dating resolution. However, in studies involving legacy wells and well cuttings, large sample sizes may not be possible, and the approach used in this study to increase sample size by combining cuttings over a few hundred feet may still be the best option. We also recommend that care is taken in the selection and recording of the drilling fluids that are used, to avoid or recognize potential contamination issues. If possible, it is best not to prewash well cuttings before the mineral separation process, because this may lead to the unnecessary loss of very fine-grained zircon crystals. All mineral separates should be retained for alternative dating approaches (e.g., U-Pb on titanite and Ar/Ar on detrital sanidine). In some cases, through careful mineral separation, this approach may be a gateway to discovering other datable radiogenic minerals capable of recording depositional age, such as detrital apatite, titanite, monazite, or sanidine.

Calculating Maximum Depositional Ages

Because of the uncertainties associated with U-Pb isotopic systematics, multiple approaches are commonly

used to define the youngest detrital zircon age population (Dickinson and Gehrels, 2009). When working with detrital minerals, it is important to compare and evaluate results from each approach and to assess maximum depositional ages by correlation with existing biostratigraphy, well-dated tuffs, or other means if available. We applied four robust approaches to each sample for determining maximum depositional ages, including (1) youngest single grain (YSG), (2) weighted mean of the three youngest grains (WM), (3) weighted mean age of the youngest cluster of two or more grains overlapping in age at 1σ (standard deviation) (YCL 1σ ; Dickinson and Gehrels, 2009), and (4) weighted mean age of the youngest cluster of three or more grains overlapping in age at 2σ (YC2 σ ; Dickinson and Gehrels, 2009). Other calculations may also be applied to determine maximum depositional ages (e.g., TuffZirc algorithm of Ludwig and Mundil, 2002); however, here we have chosen robust approaches that are most suitable to the size and nature of our data set. Approaches 2, 3, and 4 produced similar maximum depositional ages where there were enough grains to calculate a mean age, and all revealed a younging trend in the bottom three to four Galula-1 well samples (Figure 2). Similarly, the youngest single grain ages through the approximately 950–625 m (3117–2051 ft) intervals in the Galula-1 well show a zircon younging trend (i.e., a depositional age younging trend), although these ages are less than or equal to 1 Ma younger than calculated maximum depositional ages using methods 2–4. Although it may be best practice to consider the youngest population ($n \geq 2$) of zircons when estimating depositional age, as shown in Figure 2, the youngest single grains throughout the Galula-1 well are also reliable indicators of depositional age because it is unlikely these crystals experienced Pb loss because of their young crystallization ages and the lack of late Cenozoic metamorphic activity affecting this region. However, maximum depositional ages based on the youngest populations of zircons, instead of on single grain ages, are more consistently compatible with depositional ages (Dickinson and Gehrels, 2009). In all cases, biostratigraphy; dating tuffs where available; and knowledge of the age of contemporaneous, nearby volcanic deposits are ideal for evaluating such maximum depositional ages (Figure 3).

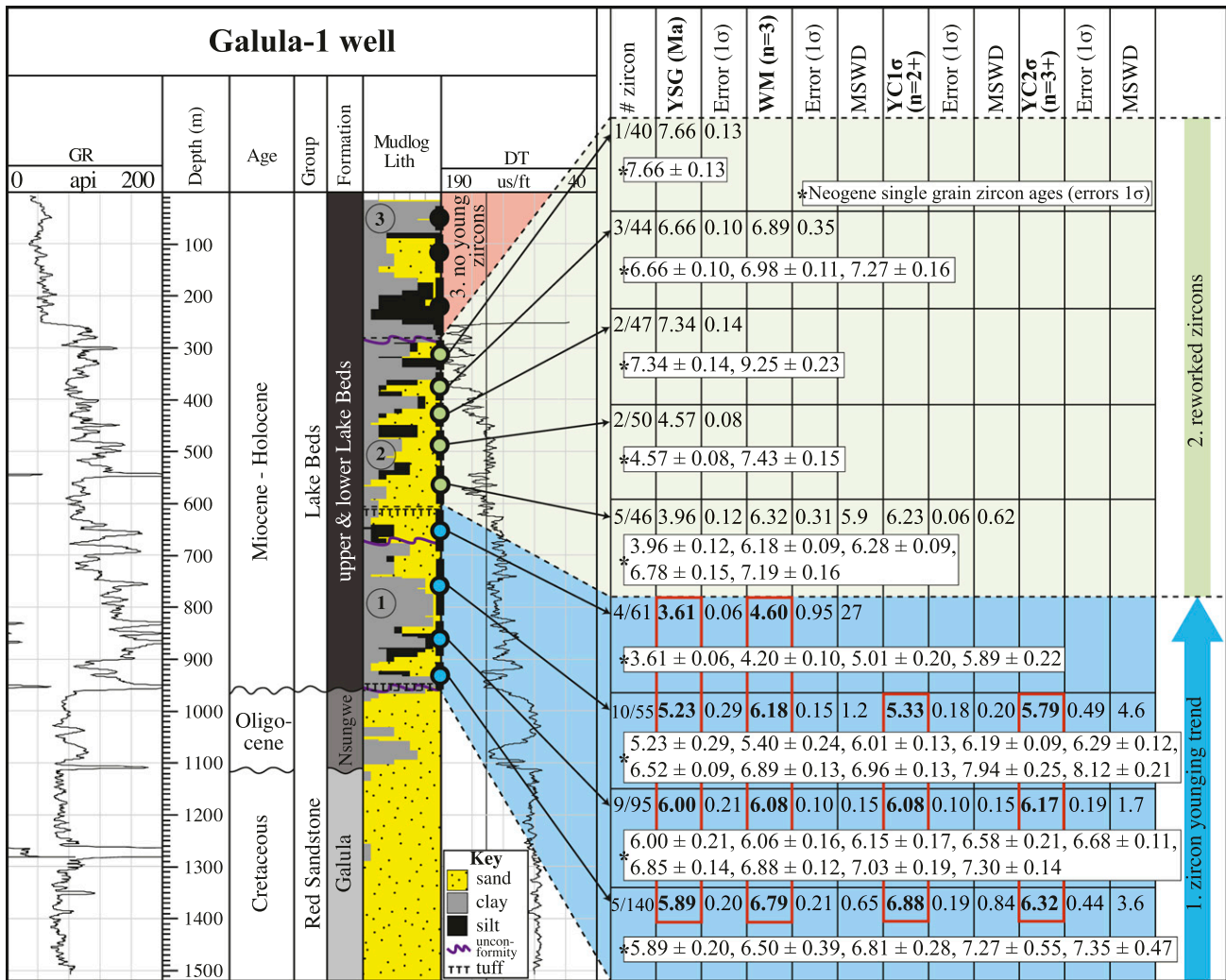


Figure 2. Stratigraphically ordered display of all Neogene zircons recovered from the Galula-1 well cuttings (single grain ages are listed in white boxes). The results of four different methods for calculating maximum depositional age for each sample interval are shown, and up-hole younging trends are highlighted by rectangles (red). Galula-1 well log data were provided by Heritage Rukwa Tanzania Limited. Stratigraphic interpretations are based on Roberts et al. (2004, 2010) and all available well data sets (not all presented here). Dashed lines through the mudlog lithology are suggested unconformities, based on three distinct detrital zircon zones: (1) up-hole zircon younging trend (zircons of Neogene age), (2) reworked Neogene zircons, and (3) no Neogene zircons. Wavy lines (purple) represent correlated unconformities recognized in outcrop, and rows of T symbols show the correlated stratigraphic positions of tuffs from outcrop (see Figure 3). # zircon = number of Neogene zircon ages/total number of concordant zircon ages acquired; DT = sonic; GR = gamma ray; MSWD = mean square weighted deviation; WM = weighted mean; YC1σ (2+) = weighted mean of youngest zircon cluster of greater than or equal to two grains overlapping in age at 1σ; YC2σ (3+) = weighted mean of youngest zircon cluster of greater than or equal to three grains overlapping in age at 2σ; YSG = youngest single grain. Note: A color version can be seen in the online version.

RELIABLE DEPOSITIONAL AGE CONSTRAINTS ACQUIRED FROM WELL CUTTINGS

Geochronology Results

Twelve samples from the Galula-1 exploration well yielded 638 concordant U–Pb detrital zircon ages.

The dominant detrital zircon age populations for the Lake Beds include (1) 3100–2500 Ma, (2) 2200–1800 Ma, (3) 1050–950 Ma, (4) 900–500 Ma, and (5) 8.7–3.5 Ma. Young, Neogene zircons (<10 Ma) are present in the nine deepest Lake Beds samples and constitute 2.5%–18.2% of the dated zircons from each interval. All Neogene zircon ages are reported in Table 1 and in Figure 2 in the white boxes

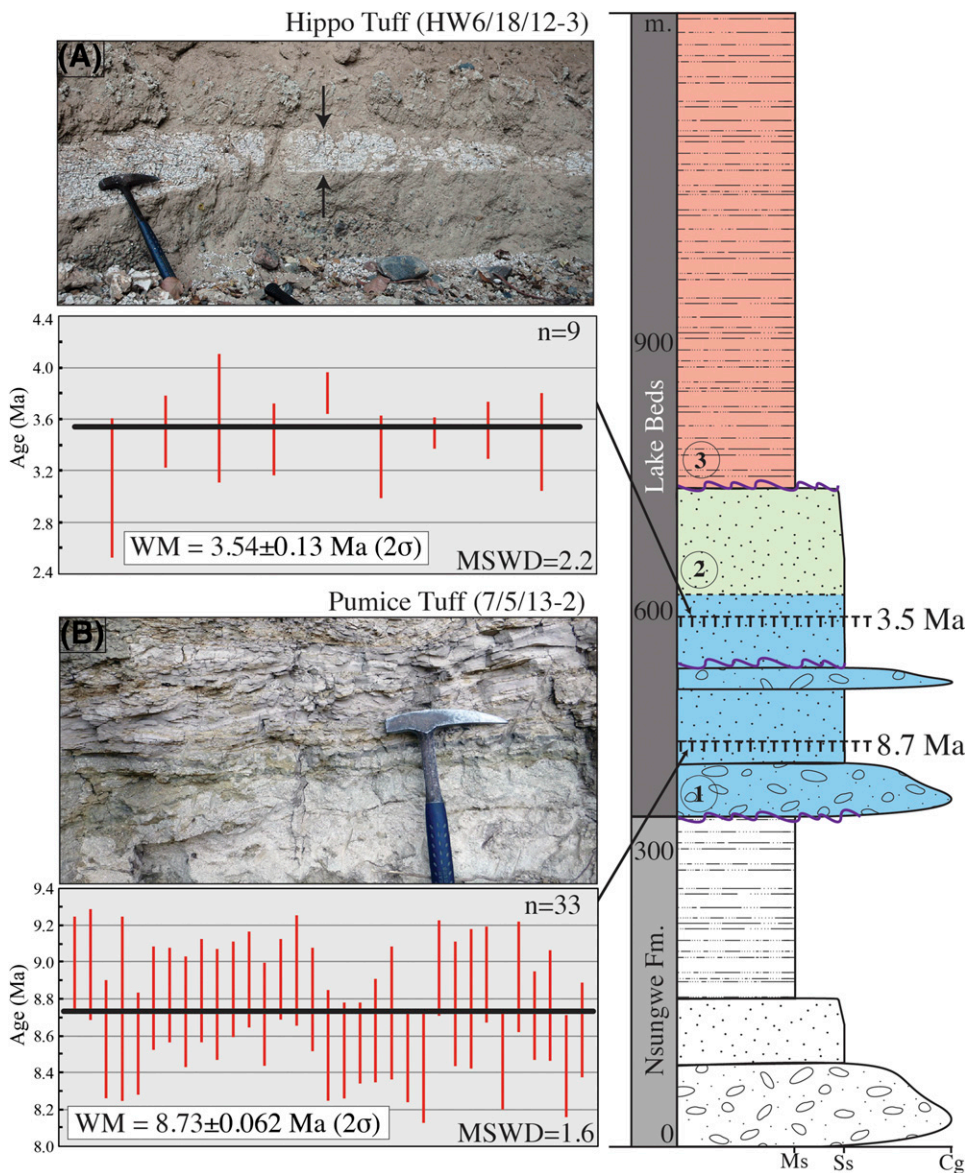


Figure 3. Laser ablation–inductively coupled plasma–mass spectrometry U–Pb ages of two newly dated volcanic tuffs from the Lake Beds in outcrop, located within a composite stratigraphic section (positions of tuffs denoted by rows of T symbols). The three indicated zones correlate with the detrital zircon zones ([1] up-hole zircon younging trend [zircons of Neogene age], [2] reworked Neogene zircons, and [3] no Neogene zircons) distinguished in Figure 2. Wavy lines (purple) represent unconformities recognized in outcrop. Cg = conglomerate; Fm. = Formation; Ms = mudstone; MSWD = mean square weighted deviation; Ss = sandstone; WM = weighted mean. Note: A color version can be seen in the online version.

corresponding to each sampled well interval. For the purposes of this study, only the youngest detrital zircon populations are reported in Figure 2 and Table 1. However, the full detrital zircon data sets for each of the 12 samples from the Galula-1 well are available in Tables S1–S12 (supplementary material available as AAPG Datashare 76 at www.aapg.org/datashare). All tuff zircon ages acquired from this study are reported in Figure 3 and Table 2. Zircon recovery decreased significantly from the deepest (960 m [3150 ft]) to shallowest (27.5 m [90 ft]) intervals sampled from the Lake Beds in the Galula-1 well.

Maximum depositional ages for Neogene zircon-bearing samples were calculated via the four methods

described above (YSG, WM, YC1σ, and YC2σ). The deepest four Lake Beds samples reveal maximum depositional ages that young from approximately 6.9 to 4.6 Ma toward the top of the Galula-1 well (Figure 2), which correlate well with the ages of two tuffs recognized in outcrop, also from the lower part of the Lake Beds strata. These two tuffs from the Lake Beds were identified in the field along the Hamposia River section (Figures 1, 3). Volcanic zircons from each tuff were sampled and dated using the same LA–ICP–MS U–Pb methodology as applied to the Galula-1 well cuttings (Figure 3). The lower tuff (Pumice Tuff: 7/5/13-2; 0480150 E 9045105 N; UTM zone 36, ARC 1960 datum), 29 m (95 ft) from the

Table 1. The $^{206}\text{Pb}/^{238}\text{U}$ Ages of Youngest Detrital Zircon Populations from the Galula-1 Well Cuttings

Galula-1 Well Samples	283–341 m (930–1120 ft) (N = 1)		347–399 m (1140–1310 ft) (N = 3)		405–451 m (1330–1480 ft) (N = 2)		457–515 m (1500–1690 ft) (N = 2)		527–600 m (1730–1970 ft) (N = 5)		613–692 m (2010–2270 ft) (N = 4)		704–814 m (2310–2670 ft) (N = 10)		826–893 m (2710–2930 ft) (N = 9)		905–960 m (2970–3150 ft) (N = 5)	
	$^{206}\text{Pb}/^{238}\text{U}$ Age (Ma)	Error (1 σ) (Ma)	$^{206}\text{Pb}/^{238}\text{U}$ Age (Ma)	Error (1 σ) (Ma)	$^{206}\text{Pb}/^{238}\text{U}$ Age (Ma)	Error (1 σ) (Ma)	$^{206}\text{Pb}/^{238}\text{U}$ Age (Ma)	Error (1 σ) (Ma)	$^{206}\text{Pb}/^{238}\text{U}$ Age (Ma)	Error (1 σ) (Ma)	$^{206}\text{Pb}/^{238}\text{U}$ Age (Ma)	Error (1 σ) (Ma)	$^{206}\text{Pb}/^{238}\text{U}$ Age (Ma)	Error (1 σ) (Ma)	$^{206}\text{Pb}/^{238}\text{U}$ Age (Ma)	Error (1 σ) (Ma)	$^{206}\text{Pb}/^{238}\text{U}$ Age (Ma)	Error (1 σ) (Ma)
Youngest (Ma)	7.66	0.13	6.66	0.10	7.34	0.14	4.57	0.08	3.96	0.12	3.61	0.06	5.23	0.29	6.00	0.21	5.89	0.20
			6.98	0.11	9.25	0.23	7.43	0.15	6.18	0.09	4.20	0.10	5.40	0.24	6.06	0.16	6.50	0.39
			7.27	0.16					6.28	0.09	5.01	0.20	6.01	0.13	6.15	0.17	6.81	0.28
									6.78	0.15	5.89	0.22	6.19	0.09	6.58	0.21	7.27	0.55
									7.19	0.16			6.29	0.12	6.68	0.11	7.35	0.47
													6.52	0.09	6.85	0.14		
													6.89	0.13	6.88	0.12		
													6.96	0.13	7.03	0.19		
													7.94	0.25	7.30	0.14		
													8.12	0.21				
<i>WM (Ma) N = 3</i>	<i>n/a</i>		<i>6.89</i>	<i>0.35</i>	<i>n/a</i>		<i>n/a</i>		<i>6.32</i>	<i>0.31</i>	<i>4.6</i>	<i>0.95</i>	<i>6.18</i>	<i>0.15</i>	<i>6.08</i>	<i>0.10</i>	<i>6.79</i>	<i>0.21</i>
<i>YC1σ (2+; Ma)</i>	<i>n/a</i>		<i>n/a</i>		<i>n/a</i>		<i>n/a</i>		<i>6.23</i>	<i>0.06</i>	<i>n/a</i>		<i>5.33</i>	<i>0.18</i>	<i>6.08</i>	<i>0.10</i>	<i>6.88</i>	<i>0.19</i>
<i>YC2σ (3+; Ma)</i>	<i>n/a</i>		<i>n/a</i>		<i>n/a</i>		<i>n/a</i>		<i>n/a</i>		<i>n/a</i>		<i>5.79</i>	<i>0.49</i>	<i>6.17</i>	<i>0.19</i>	<i>6.32</i>	<i>0.44</i>

Maximum depositional ages are calculated (italics). The full detrital zircon data sets are available in Tables S1–S12 (supplementary material available as AAPG DataShare 76 at www.aapg.org/datasshare).
Abbreviations: WM = weighted mean; YC1 σ = weighted mean of youngest cluster of two or more grains overlapping in age at 1 σ ; YC2 σ = weighted mean of youngest cluster of three or more grains overlapping in age at 2 σ .

base of the Lake Beds, yields a U–Pb zircon age of 8.7 ± 0.03 Ma (1σ ; Figure 3, Table 2). The Hippo Tuff (HW6/18/12-3; 0480513 E 9047045 N; UTM zone 36, ARC 1960 datum), approximately 75 m (246 ft) above the Pumice Tuff, yields a U–Pb zircon age of 3.5 ± 0.07 Ma (1σ ; Figure 3, Table 2).

Interpretation of Zircon Depositional Ages for the Lower Lake Beds

The U–Pb zircon ages we report here are the first radioisotopic ages for the lower Lake Beds succession. The Pumice Tuff, which sits 29 m (95 ft) above the base of the Lake Beds, yields a depositional age of 8.73 ± 0.06 Ma (2σ), which we interpret to record the age of the initiation of the last major phase (Miocene–Holocene) of basin development and sedimentation in the Rukwa Rift. The age of this tuff corresponds to the initiation of the Rungwe volcanic province and is possibly correlative with the early Rungwe volcanic activity, such as the 8.60 ± 0.01 and 8.15 ± 0.02 Ma (1σ) Songwe phonolitic tuffs in the Karonga Basin of the northern Lake Malawi Rift (Ebinger et al., 1993) or to the 9.2 ± 0.4 Ma trachyte dome in the Usangu Basin (Ivanov et al., 1999).

Subdivision of the Lake Beds in the Galula-1 well is possible based on the presence or absence of Neogene, depositional age–constraining zircons. The Lake Beds in the Galula-1 well can be divided into three zones based on patterns of Neogene-aged detrital zircons (Figure 2): (1) zone of maximum depositional age–constraining zircons that progressively young upward (blue zone 1), (2) zone of reworked Neogene zircons (i.e., no younging trend; green zone 2), and (3) zone containing no Neogene zircons (red zone 3). A zircon younging trend recorded in the four deepest sampled Lake Beds intervals (Figure 2, zone 1) establishes maximum depositional ages from 6.88 ± 0.19 Ma to 4.60 ± 0.95 Ma (1σ) in the well. A combination of zircon zonal partitioning, lithostratigraphy, and zircon-based maximum depositional ages allows for a strong correlation between the Galula-1 well and the two dated tuffs and stratigraphic unconformities identified in outcrop (Figures 2, 3). The two dated tuffs bracket zone 1 stratigraphically (Figures 2, 3). Zone 1 is underlain by the 8.73 ± 0.06 Ma (2σ) Pumice Tuff and capped by the 3.54 ± 0.13 Ma (2σ) Hippo Tuff (Figure 3),

which nicely fits the upward younging trend of depositional age–defining detrital zircons from approximately 6.9 to 4.6 Ma that is recorded in the well samples that correlate stratigraphically to between these two tuffs. This provides corroborating evidence to support the accuracy of maximum depositional ages recorded by detrital zircon in the Galula-1 well, strongly supporting the application of this approach.

After approximately 3.5 Ma, zircon-generating volcanism capable of reaching the southern end of the Rukwa Rift Basin apparently ceased in the Rungwe volcanic province because the middle of the Lake Beds succession recorded in the Galula-1 well contains only reworked approximately 9–4 Ma Neogene zircons, and no tuffs were identified in outcrop from this level (Figure 2, zone 2). An increase in the proportion of sandy facies in Galula-1 also occurs at the point where the younging zircon trend shuts off (between zones 1 and 2; Figure 2), suggesting a period of volcanic inactivity characterized instead by fluvial reworking within the basin. At the top of the well, in units characterized by clay- and silt-rich lacustrine facies, no Neogene-age zircons were recovered, a trend that was also observed in detrital zircon samples collected from outcrops through this interval (Figure 2, zone 3). Radiocarbon analyses from the uppermost Lake Beds succession suggest an age of $27,750 \pm 110$ calibrated radiocarbon dating before present (Cal BP) to 7270 ± 60 Cal BP (Cohen et al., 2013; Hilbert-Wolf and Roberts, 2015; Mtelega et al., 2016) for the uppermost Lake Beds succession. The presence or absence of syndepositional zircon age populations in the sedimentary record is coincident with changing magma compositions within the Rungwe volcanic province between trachytes, phonolites, and basalts (Ebinger et al., 1989, 1993). The Rungwe volcanic province evolved from dominantly phonolitic magmas between 9.2 and 5.4 Ma to dominantly zircon-free olivine basalts and trachytes from 3 to 1.5 Ma and 0.6 Ma to the present (Ebinger et al., 1989; Fontijn et al., 2012), although each of these compositions is recognized to some extent in each of the three major Rungwe volcanic province phases. First-cycle, approximately 8.7- to 3.4-Ma zircons occur in zone 1, the most zircon-fertile interval that coincides with the earliest, phonolitic dominated magmas (9.2–5.4 Ma; Fontijn et al., 2012). Above this stratigraphic interval, reworked 9- to 4-Ma zircon grains (zone 2) and an absence of zircons in

Table 2. The $^{206}\text{Pb}/^{238}\text{U}$ Ages of Tuff Zircons from Outcrop Samples 7/5/13-2 (Pumice Tuff) and HW6/18/12-3 (Hippo Tuff)

Spot Identification	$^{206}\text{Pb}/^{238}\text{U}$	
	Age (Ma)	Error (1σ) (Ma)
Pumice Tuff (7/5/13-2)		
7-5-13-2-1	8.99	0.13
7-5-13-2-2	8.98	0.15
7-5-13-2-4	8.58	0.16
7-5-13-2-5	8.75	0.25
7-5-13-2-6	8.56	0.14
7-5-13-2-7	8.80	0.14
7-5-13-2-8	8.82	0.13
7-5-13-2-9	8.73	0.15
7-5-13-2-10	8.84	0.14
7-5-13-2-11	8.77	0.15
7-5-13-2-14	8.85	0.13
7-5-13-2-15	8.90	0.13
7-5-13-2-16	8.72	0.14
7-5-13-2-17	8.91	0.11
7-5-13-2-18	8.95	0.15
7-5-13-2-19	8.79	0.14
7-5-13-2-20	8.55	0.15
7-5-13-2-21	8.52	0.13
7-5-13-2-22	8.56	0.11
7-5-13-2-23	8.62	0.14
7-5-13-2-24	8.72	0.18
7-5-13-2-25	8.48	0.12
7-5-13-2-26	8.43	0.15
7-5-13-2-28	8.97	0.13
7-5-13-2-29	8.77	0.17
7-5-13-2-30	8.80	0.19
7-5-13-2-31	8.93	0.13
7-5-13-2-32	8.46	0.13
7-5-13-2-34	8.92	0.15
7-5-13-2-35	8.71	0.12
7-5-13-2-36	8.76	0.15
7-5-13-2-39	8.44	0.14
7-5-13-2-40	8.63	0.13
<i>Weighted mean</i>	<i>8.73</i>	<i>0.03</i>
Hippo Tuff (HW6/18/12-3)		
HW6/18/12-3-5	3.06	0.27
HW6/18/12-3-8	3.50	0.14
HW6/18/12-3-19	3.61	0.25
HW6/18/12-3-31	3.44	0.14
HW6/18/12-3-32	3.80	0.08
HW6/18/12-3-33	3.30	0.16
HW6/18/12-3-36	3.49	0.06
HW6/18/12-3-45	3.51	0.11

(continued)

Table 2. Continued

Spot Identification	$^{206}\text{Pb}/^{238}\text{U}$	
	Age (Ma)	Error (1σ) (Ma)
HW6/18/12-3-50	3.42	0.19
<i>Weighted mean</i>	<i>3.54</i>	<i>0.07</i>

zone 3 suggest deposition during a period of predominantly olivine basalt-producing volcanism with very low zircon fertility.

DISCUSSION

Suitability of Detrital Zircon Approach

In a pioneering study on the relationship between detrital zircon age populations in sedimentary strata and basin types, Cawood et al. (2012) found that the crystallization age of the youngest detrital zircon population tends to very closely approximate the timing of sediment deposition in certain basin types. For example, convergent margin basins are typically associated with felsic-to-intermediate, arc-related volcanism and tend to produce sedimentary sequences with a high proportion of young, near-syn depositional detrital zircon populations. An implication of this and other recent detrital zircon studies (e.g., DeCelles et al., 2007; Barbeau et al., 2009; Park et al., 2010; Tucker et al., 2013; Gehrels, 2014) is that certain basins are suited for the application of detrital zircon geochronology, such as foreland, fore-arc, and back-arc, as well as volcanic retro-arc foreland basins and rift basins, where this technique has the potential to be used as a stratigraphic tool for refining the age of successive stratigraphic units.

However, collisional and extensional basins, which include peripheral (pro) foreland basins, passive margins, and intracratonic basins, as well as rifts, tend to record a proportionally smaller volume of young, age-constraining syn depositional zircons (Cawood et al., 2012). Nonetheless, with a statistically significant number of detrital zircon grains (Vermeesch, 2004), one should expect to recover a population of depositional age-constraining detrital zircons, even if the population is a very minor constituent of the total zircon volume. Although recovering greater than 100 detrital zircons may not be

possible with small-volume samples from well cuttings, this study makes it evident that other basin types (in addition to convergent margins) associated with active volcanism (such as the intracratonic Rukwa Rift Basin) can be rich sources of syndepositional volcanic zircons for depositional age constraint and do not require 100+ detrital zircons for the detection of young syndepositional zircon grains. Because zircon populations are not equally abundant (*sensu* Andersen, 2005), in the right tectonic setting, dating fewer total detrital zircon grains (here, for example, as few as 46) can still successfully reveal depositional age-constraining populations. This being said, if possible, it would still be advantageous to plan for detrital zircon work prior to drilling and collect larger cutting samples for this work.

For provenance studies, to be certain that all zircon populations are sampled, recommendations range from dating 35–117 zircon grains (Vermeesch, 2004; Andersen, 2005) to 300–1000 zircon grains (Pullen et al., 2014). With the 150–500 g well cutting samples from Galula-1, we were able to recover 40–140 concordant zircon grains from each sample (full detrital zircon data sets available in Tables S1–S12 [supplementary material available as AAPG Datashare 76 at www.aapg.org/datashare]). Because we are concerned only with the presence of one particular zircon population (*i.e.*, that which constrains the sediment depositional age), the number of zircons dated per sample is not an imperative statistic. The presence of a zircon population in a very small data set suggests that the origin of such zircons is a significant sediment source. When seeking a maximum depositional age via detrital zircon in any volcanically influenced basin, the presence of just greater than or equal to two to three young zircons is typically admissible for constraining or greatly refining the depositional age of sediments (*e.g.*, Dickinson and Gehrels, 2009), providing it is a geologically and tectonically sensible age.

No matter the size of the data set, of greatest importance when deciding to use this method for maximum depositional age control and temporal refinement of stratigraphic intervals is the likelihood that a syndepositional magmatic source (zircon fertile) contributed significantly to the zircon content of the sediment (Sláma and Košler, 2012). Although extensional basins tend to have a high proportion of reworked zircons derived from the basement

(Cawood et al., 2012), volcanism is commonly present along rift zones such as in the EARS, and these basin types are potential targets for applying detrital zircon geochronology for maximum depositional age constraint of their stratigraphic successions. If the youngest sediment source is greatly overshadowed by zircon input from older sources caused by drainage patterns, the depositional age population is likely to be overlooked even with a large number of detrital zircon analyses (Andersen, 2005). Similarly, many volcanic centers, particularly those that are rift-related, tend to evolve through time, meaning that zircon fertility changes. As a result of this, a modified approach to dating well cuttings may yield even better results, particularly one in which radioisotopic dating of other mineral phases, such as titanite, monazite, apatite, or rutile, is applied alongside detrital zircon geochronology.

One caveat of the approach applied here is an explicit understanding that the maximum depositional age of detrital zircons (or other detrital minerals) is not necessarily the same as depositional age. In many volcanically active settings, it has been clearly demonstrated that the maximum depositional age from zircons does closely correspond to the depositional age of the sediments from which the zircons were recovered (*e.g.*, Dickinson and Gehrels, 2009; Cawood et al., 2012; Tucker et al., 2013). However, a good understanding of tectonics and other supporting geologic data is critical to the accurate interpretation of the results. Correspondence to known stratigraphic superposition in the well and other associated geologic data, such as dated tuffs or volcanic strata and well-constrained biostratigraphic intervals, is an excellent indication that the youngest population of detrital zircon grains was generated concurrently with strata deposition. Similarly, younging maximum depositional ages upward through a well supports such interpretations. Knowledge of the ages of nearby zircon sources (particularly volcanic sources), as well as assessing mineral shape for signs of weathering and reworking, can also contribute significantly to assigning a depositional age based on the youngest zircon population. In addition to the likelihood that a basin collects and preserves syndepositional volcanic sediment, consideration must be made for analytical uncertainties and for the possibility of disturbance of the U–Pb system in zircons (*e.g.*, Pb loss), which may result in age underestimations.

Implications for the Development of the East African Rift

The Rukwa Rift Basin is an important segment of the EARS, in the sense that it preserves sedimentary rocks from the Permian–Triassic to Holocene, and because of its superposition on top of a structurally weak shear zone, the Rukwa Rift Basin has been subject to rifting, subsidence, and uplift repeatedly throughout its history. The Oligocene onset of rifting in the western branch of the EARS has been determined through the dating of tuffs from the Nsungwe Formation, which underlies the Lake Beds in the Rukwa Rift Basin (Roberts et al., 2012). Until this study, accurate age constraints on the initiation of the most recent phase of sedimentation (i.e., the Lake Beds) were absent because of conflicting biostratigraphic age information. The U–Pb zircon ages from the basal part of the Lake Beds succession reported here are significant for placing age constraints on this most recent phase of late Cenozoic rifting, demonstrating that renewed sedimentation commenced at approximately 8.7 ± 0.06 Ma (2σ). Significantly, these dates show that initiation of late Cenozoic sedimentation was coeval with the first phase of late Cenozoic volcanism in the Rungwe volcanic province. This demonstrates that reactivation of the Rukwa Rift Basin was heralded by both volcanism and sedimentation (i.e., rifting and basin filling).

The characteristics of sedimentary fill in a rift basin depend on climate, provenance, and subsidence rates. Because extensional events in the EARS are marked by sedimentation and magmatism, the age and provenance of both detrital and tuffaceous zircons from the Rukwa Rift strata provide reliable insight into the timing of rift development and likely regional uplift in the western branch. Our new constraints on the timing of this most recent phase of rifting are consistent with demonstrated rifting and uplift in Uganda (Bauer et al., 2010) and Malawi (Ebinger et al., 1993), as well as with the initiation of the Rungwe volcanic province at the southern end of the Rukwa Rift (Ebinger et al., 1993; Ivanov et al., 1999) and the estimated development of Lake Tanganyika to the north (Cohen et al., 1993).

Constraining the timing of rifting and deposition of the Lake Beds also allows us to investigate links between critical climate events, landscape changes, and evolutionary patterns. Specifically, understanding the timing of the Lake Beds deposits

has the potential to help understand the tempo and extent of late Cenozoic uplift. Along the East African Rift, as uplift of the rift shoulders commenced, the climate of eastern Africa changed, putatively driving patterns of vertebrate evolution (e.g., Sepulchre et al., 2006). Recently, a large number of previously unknown species have been discovered in the Rukwa Rift Basin. These discoveries reveal that this part of the East African Rift was an important setting for the evolution of unique flora and fauna (e.g., Feldmann et al., 2007; Stevens et al., 2013; Gorscak et al., 2014; McCartney et al., 2014). The radioisotopic ages presented here allow us to confirm a late Miocene–Pliocene age for the lower Lake Beds and place critical constraints on newly discovered vertebrate-bearing deposits in the Rukwa Rift. This discovery is significant because it represents the only known fossil-bearing deposit of this age exposed in the western branch of the EARS between lakes Edward and Malawi. Therefore, the detrital and tuff-derived zircons dated here provide important temporal context for the rich vertebrate record described from the East African Rift and help to illuminate the tectonic backdrop and timing of important large-scale faunal shifts in East Africa.

SUMMARY

The ages of sedimentary basins along the Cenozoic EARS, the world's archetypal continental rift system, figure prominently into our understanding of the timing of rift formation, evolution of topography and climate, hydrocarbon generation, and other fundamental geologic questions. Detrital zircon geochronology of the Galula-1 well cuttings, combined with U–Pb tuff dating from correlative outcrops, has provided the first radioisotopic age constraints on deposition of the lower Lake Beds succession of the Rukwa Rift Basin, from approximately 8.7 ± 0.06 Ma to 3.5 ± 0.13 Ma (2σ), suggesting that Neogene rift reactivation, volcanism, and sedimentation began coevally. These results strongly support the application of detrital zircon geochronology in hydrocarbon exploration, specifically where small-volume well cuttings may limit samples. The U–Pb LA–ICP–MS detrital zircon geochronology is economic and now widely available and should be considered as a standard complementary tool to

biostratigraphy for refining the depositional age of the strata in wells, particularly through intervals with limited or conflicting biostratigraphy. This approach has the potential to (1) closely approximate the depositional age of stratigraphic units, (2) estimate and provide constraints on sedimentation rates, and (3) provide intrabasinal correlation of units using detrital zircon fingerprinting, thereby resolving a variety of stratigraphic, burial, time, and thermal history questions critical to hydrocarbon exploration and the potential development of these resources.

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