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Dating lacustrine carbonate strata with detrital zircon U-Pb geochronology

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ABSTRACT

Carbonate lacustrine strata in nonmarine systems hold great potential for refining depositional ages through U-Pb dating of detrital zircons. The low clastic sediment flux in carbonate depositional environments may increase the relative proportion of zircons deposited by volcanic air fall, potentially increasing the chances of observing detrital ages near the true depositional age. We present U-Pb geochronology of detrital zircons from lacustrine carbonate strata that provides proof of concept for the effectiveness of both acid-digestion recovery and resolving depositional ages of nonmarine strata. Samples were collected from Early Cretaceous foreland basin fluvial sandstone and lacustrine carbonate in southwestern Montana (USA). Late Aptian-early Albian (ca. 115-110 Ma) maximum depositional ages young upsection and agree with biostratigraphic ages. Lacustrine carbonate is an important component in many types of tectonic basins, and application of detrital zircon U-Pb geochronology holds considerable potential for dating critical chemical and climatic events recorded in their stratigraphy. It could also reveal new information for the persistent question about whether the stratigraphic record is dominated by longer periods of background fine-grained sedimentation versus short-duration coarse-grained events. In tectonically active basins, lacustrine carbonates may be valuable for dating the beginning of tectonic subsidence, especially during periods of finer-grained deposition dominated by mudrocks and carbonates.

INTRODUCTION

Nonmarine strata are notoriously difficult to date because of poor preservation of marker beds, challenges to the recovery of palynomorphs, and biozones of generally long duration (e.g., Wainman et al., 2018). Another approach, detrital zircon U-Pb geochronology, is routinely used to obtain crystallization ages of zircons in fluvial sandstone. These data typically contain multiple age modes that are used to interpret sediment provenance. The youngest zircon or group of zircons is used to resolve the maximum depositional age (MDA) because, according to the law of inclusions, deposition must postdate the youngest constituent (Dickinson and Gehrels, 2009). MDAs most closely approximate true depositional ages when two conditions are met: (1) profuse contemporaneous volcanism to contribute zircons to the fluvial system either directly from source-area erosion or via air fall, and (2) retention of these zircons in the sedimentary system. In cases where volcanism is subdued, any signal from nominal volcanic grains may be masked by abundant detrital grains from other sources. Furthermore, incremental additions of volcanic populations via air fall may be carried out of the nonmarine system altogether by river currents.

To circumvent these issues, we target carbonate lacustrine strata because of their lowenergy, restricted depositional setting and ability to retain uninterrupted sedimentary records. The development of lacustrine carbonate is primarily controlled by tectonics and climate, and, when conditions permit, they are relatively common features in all types of sedimentary basins (Gierlowski-Kordesch, 2010). These systems hold much promise for recovering depositional-age zircons because clastic sediment flux is greatly reduced or completely absent, sedimentation rates are generally slower and permit the prolonged accrual of intermittent volcanic air fall, and retention and recovery of even trivial amounts of volcanic grains are much greater.

We conducted laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) U-Pb geochronology of detrital zircons from Early Cretaceous foreland basin fluvial sandstone and lacustrine carbonate in southwestern Montana (United States; Fig. 1A). These strata were deposited during an important time in the evolution of the western North American Cordillera when there was a significant transition in both depositional style and basin dynamics (Leier and Gehrels, 2011). We present the first example of retooling an existing rock-digestion method in order to extract minor quantities of detrital zircon from lacustrine carbonate. Previously, Tang et al. (2016) used traditional mineral-separation methods and found a few depositional-age detrital zircon grains in shallow marine metacarbonate rocks. However, marine strata, in contrast to nonmarine strata, are much easier to date biostratigraphically, better preserve volcanic ashes, and are far easier to correlate given their lateral uniformity. Here, we provide proof of concept for the effectiveness both of zircon recovery using an acid-digestion method and of resolving depositional ages of nonmarine strata.

BACKGROUND

The Kootenai Formation in southwestern Montana is an asymmetric sedimentary wedge that comprises conglomerate, sandstone, silt-stone, mudstone, and limestone (DeCelles, 1986). We collected samples from four mappable units that are informally referred to as the lower clastic, lower calcareous, upper clastic, and upper calcareous members (Kauffman, 1963; Table S1 in the Supplemental Material¹). Sample ZG-15 m was collected from a sandstone near the base of the lower clastic member; sample ZG-150 m was collected from a micritic limestone bed in the lower calcareous member;

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^{&#}x27;Supplemental Material. Detrital zircon U-Pb analytical data. Please visit https://doi.org/10.1130/GEOL.S.13076141 to access the supplemental material, and contact editing@geosociety.org with any questions.

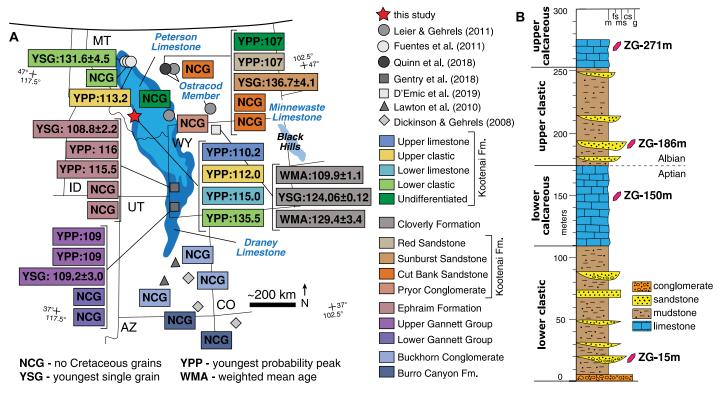


Figure 1. (A) Locations of detrital zircon samples and maximum depositional ages (in Ma) of Lower Cretaceous strata, western United States. MT—Montana; WY—Wyoming; ID—Idaho; UT—Utah; CO—Colorado; AZ—Arizona. (B) Measured stratigraphic section of the Kootenai Formation at Ziegler Gulch, Montana, with positions of detrital zircon samples from this study. m—mudstone; fs—fine-grained sandstone; ms—medium-grained sandstone; cs—coarse-grained sandstone; g—gravel.

sample ZG-186 m was collected from a prominent sandstone in the upper clastic member; and sample ZG-271 m was collected from a fossiliferous micritic limestone in the upper calcareous member (Fig. 1B). None of the sampled beds displayed any clear evidence for the presence of tephra or volcaniclastic detritus.

MINERAL-SEPARATION AND ANALYTICAL METHODS

Approximately 5-10 kg of sandstone or limestone was collected from each of the four members (Fig. 1B). Sandstones were processed following typical mineral-separation protocols of jaw crusher, pulverizer, and water-table, freefall, and barrier Frantz magnetic separations, followed by methylene iodide heavy liquid separation. The limestones were processed through a jaw crusher for reduction to gravel size and then digested in buffered formic acid to dissolve the carbonate matrix following procedures traditionally used for conodont extractions (see the Supplemental Material). The goal of acid digestion was to free any zircons of carbonate matrix that may affect their bulk densities and cause them to be removed during the mineralseparation process. Subsequent rinsing was performed with stacked disposable sieve screens of $32~\mu m$ and $425~\mu m$ to preserve the coarse-silt to medium-sand fraction. Sample ZG-150 m from the lower calcareous member produced >2 kg of sand, which was processed over the water table to remove low-density minerals before magnetic separation. Sample ZG-271 m from the upper calcareous member produced <500 g of sand that went directly to magnetic separation. Final heavy-mineral separates were obtained with a methylene iodide separation. U-Pb data were collected following the methods of Sundell et al. (2020) for the sandstones and Gehrels and Pecha (2014) for the limestones.

RESULTS

Zircon Yield and Characteristics

Zircon yield, shape, color, and proportion of young versus old grains contrast for the two different lithologies. Sandstone samples produced a typical quantity of clean heavy-mineral separate (0.065 g and 0.155 g) that amounts to thousands of individual zircons. Grain shapes range from euhedral to well rounded with mostly colorless to pale yellow grains and a smaller proportion of pink and purple grains (Fig. 2). Limestones yielded n = 32 and n = 63 zircons that were dominantly euhedral and colorless with few rounded and only one purple grain. The sandstones contain >90% grains that are not part of the youngest group, whereas the carbonates have <50% of these grains (Fig. 3).

Maximum Depositional Ages

Methods to determine the MDA of strata from detrital zircons are constantly being reevaluated (e.g., Dickinson and Gehrels, 2009; Coutts et al., 2019). Here, we modify the method of Spencer et al. (2016) and filter the youngest group that overlaps within their 1σ error for potential Pb loss by removing ages in the negatively skewed tail and then discard any remaining grains that do not overlap concordia within their 1σ error (Fig. 3). From this filtered group, we identify the youngest single concordant grain (YSCG), the youngest probability peak (YPP), the youngest statistical population (YSP), and the weighted mean age (WMA). For all samples except ZG-186 m, the YSP and WMA are identical. The mean square weighted deviation (MSWD) of the WMA was evaluated using the method of Wendt and Carl (1991) to determine whether the youngest group form a single statistical population. Three of the resultant MSWDs fall within their acceptable range, but the MSWD for sample ZG-186 m implies that there is <5% probability that the data form a single statistical population.

For this data set, we prefer MDAs based on the YPP or WMA-YSP rather than the YSCG. MDAs from both the YPP and WMA-YSP young upsection, suggesting that they likely represent the true depositional age (e.g., Schwartz et al., 2017). In contrast, the YSCG from the three upper samples yield an anomalously young age (ca. 105 Ma). Because recent Pb loss in young grains may still produce concordant ages, we do not use these ages to estimate MDA. Sample ZG-15 m from a sandstone in the lower clastic unit

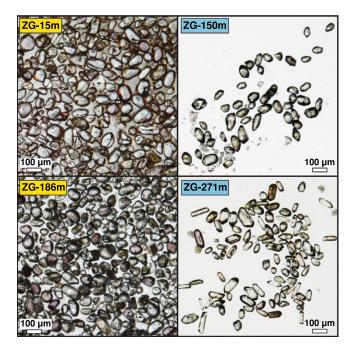


Figure 2. Representative transmitted-light microscope images of zircon crystals from four samples investigated in this study. Yellow boxes indicate sandstone separates; blue boxes are limestone separates.

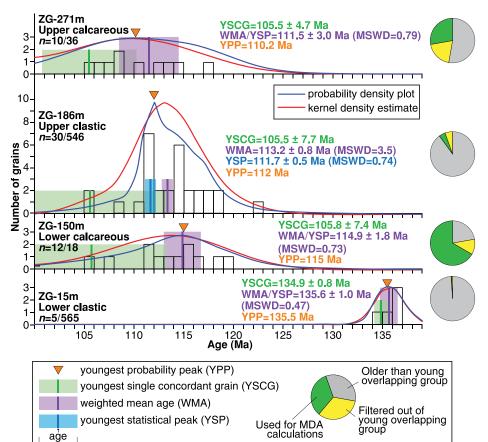


Figure 3. Probability density plots, kernel density estimates (KDEs), histograms, and maximum depositional ages (MDAs, with their 1σ errors) of the youngest groups of zircons from samples in this study. For all samples except ZG-186 m, weighted mean age (WMA) and youngest statistical population (YSP) are identical. Bandwidth for KDEs was optimized for the data set and is 4.5 m.y. MSWD—mean square weighted deviation.

near the base of the section produced a YSCG of 134.9 ± 0.8 Ma, a YPP of 135.5 Ma, and a WMA-YSP of 135.6 ± 1.0 Ma based on n = 5 with an

MSWD = 0.47 (Fig. 3). Sample ZG-150 m from the lower calcareous unit produced a YPP of 115 Ma and a WMA-YSP of 114.9 ± 1.8 Ma

based on n = 12 with an MSWD = 0.73. Sample ZG-186 m from a sandstone in the upper clastic unit yielded a YPP of 112 Ma, a WMA of 113.2 \pm 0.8 Ma based on n = 30 with an MSWD = 3.5, and a YSP of 111.7 \pm 0.5 Ma based on n = 15 with an MSWD = 0.74. Near the top of the section, sample ZG-271 m from the upper calcareous unit has a YPP of 110.2 Ma and a WMA-YSP of 111.5 \pm 3.0 Ma based on n = 10 with an MSWD = 0.79.

DISCUSSION AND CONCLUSIONS Lower Cretaceous Strata in the Western United States

The onset of deposition of Lower Cretaceous strata in the western United States, including not only the Kootenai Formation but also the correlative Gannett Group, Cloverly Formation, Lakota Formation, and Cedar Mountain and Burro Canyon Formations (Fig. 1A), has been a topic of debate for decades. Most previous biostratigraphic analyses have focused on nonmarine mollusks, ostracods, and charophytes from the interbedded carbonates that indicate Aptian to early Albian deposition (Yen, 1949, 1951; Eyer, 1969; Ostrom, 1970). In contrast, Sohn (1979) designated the Lakota Formation as pre-Aptian (Barremian or older) based on nonmarine ostracods. Furthermore, palynological analyses of interbedded mudstone have been used to interpret Berriasian or Barremian to early Albian deposition for the Cedar Mountain and Burro Canyon Formations, Kootenai Formation, and Cloverly Formation (Burden, 1984; Tschudy et al., 1984; DeCelles and Burden, 1992; Furer et al., 1997; Zaleha, 2006; Elliott et al., 2007; Fuentes et al., 2011).

A major quandary, therefore, is that although the biostratigraphy for the lowest parts of the section suggests predominantly Aptian and older deposition, detrital zircon U-Pb MDAs from those sandstones have for the most part yielded no Cretaceous grains or only rare grains <130 Ma and nearly all Albian ages (<110 Ma) from the upper parts of the section (Fig. 1A). As a result, much of the Early Cretaceous stratigraphic package lacks geochronologic data. Our results suggest late Aptian–early Albian (ca. 115–110 Ma) deposition for most of the Kootenai Formation, with deposition beginning as early as Valanginian time.

Lower Cretaceous foredeep packages in the western U.S. all have interbedded clastic and carbonate strata. For example, the lower limestone in the study area is in a similar stratigraphic position as the Ostracod Member and limestone beds in the lower Kootenai Formation in central and northwestern Montana, respectively (Fig. 1A). The lower and upper limestones in the Kootenai Formation contain similar biota to the Peterson and Draney Limestones in the Gannett Group, as well as the Minnewaste Limestone in the Black Hills of South Dakota and limestone

in the Cloverly, Cedar Mountain, and Burro Canyon Formations (Eyer, 1969; Sohn, 1979; Tschudy et al., 1984).

Based on these observations, most workers have directly correlated these various limestones and, as a result, proposed large carbonate lacustrine depocenters during the Early Cretaceous (Fig. 1A; Holm et al., 1977; Zaleha, 2006). If the correlations are accurate, an area of roughly 150,000 km² was characterized by lacustrine carbonate sedimentation in the foreland basin during two different intervals. This is not only greater than the world's largest freshwater lake today (Lake Superior, North America, ~82,000 km²), it is more than an order of magnitude greater than the largest modern alkaline lake (Lake Turkana, Kenya, ~6000 km²).

Recently, detrital zircon analyses of Lower Cretaceous strata in central Montana have produced MDAs from sandstones above and below the limestones that have challenged these correlations (Quinn et al., 2018). We demonstrate here that detrital zircons are present in and recoverable from the carbonates and, furthermore, that U-Pb MDAs most likely represent true depositional ages. Broader application of this method to the Lower Cretaceous strata, with the addition of high-precision age dating, would likely redefine our understanding of the current stratigraphic framework which would provide critical control on the timing of tectonic episodes in the North American Cordillera.

Significance of Lacustrine Carbonates in Refining Basin Models

Carbonate lake deposits develop in all climates and are found in many different types of tectonic basins, although they are most common in underfilled or balance-filled basins (Bohacs et al., 2003). Carbonate buildup in lakes is primarily dependent upon the degree of exposure of carbonates or calcium-rich rocks in the sediment source areas, including not only limestones, dolostones, and marble, but also basalt and carbonatite (Gierlowski-Kordesch, 2010). Prevalence of these zircon-poor lithologies in sediment source areas, especially in areas with active volcanism, means that fewer detrital zircons from older recycled sedimentary strata will be found in lacustrine carbonates compared to their clastic counterparts (Fig. 3). In retroforeland basins, the presence of lacustrine carbonate in the medial and distal parts of the foredeep has been inferred to represent a combination of thrusting in the fold-thrust belt, reduced clastic supply to that part of the basin, and erosion of carbonate rocks in the sediment source areas.

Lower Cretaceous strata in the western U.S. are the oldest strata definitively related to fore-deep deposition (e.g., DeCelles, 2004). However, a critical question that remains in Cordilleran geology is the timing of onset and regional pattern of crustal shortening in the retroarc re-

gion. Based on the interpretation of lacustrine carbonates in other retroforeland settings, their common occurrence in the Cordilleran foreland, and our detrital ages presented here, we could infer that sediment source areas were characterized by extensively exposed carbonate strata and that thrusting was active along much of the U.S. Cordillera during late Albian—early Aptian time (ca. 115–110 Ma). Yet direct evidence for this age of thrusting in the fold-thrust belt is sparse and mostly limited to Nevada and Utah (DeCelles, 2004).

An alternative explanation for lacustrine carbonate formation can be found in new models that demonstrate that worldwide neritic carbonate preservation rates reached maximum values between 125 Ma and 95 Ma with a peak at 110 Ma (Pohl et al., 2020). This is interpreted to reflect changes in continental configurations and volcanic CO₂ degassing, with geodynamics influencing accommodation and turnovers in dominant biota playing secondary roles. Therefore, at least in the mid-Cretaceous Cordilleran foredeep, late Aptian-early Albian lacustrine carbonate deposition may represent an interplay between regional factors, such as tectonic loading, and global influences, including plate tectonics and volcanic degassing.

Further Applications of Detrital Zircon Geochronology from Lacustrine Carbonate

Here we provide proof of concept for detrital zircon recovery and resolution of depositional ages for nonmarine strata using U-Pb geochronology from lacustrine carbonate. This technique can be broadly applied across many types of sedimentary basins and could aid in answering a diverse set of questions. For example, lacustrine carbonates archive Earth's chemical and climatic history, and this method could be used to date critical chemical and climatic events recorded in their stratigraphy. It could also help to constrain the duration and depositional rates of fine-grained sedimentation versus shortduration coarse-grained events. In tectonically active settings, the initiation of tectonic subsidence could be dated when it is recorded in finergrained strata such as mudrocks and carbonates.

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