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When Dinosaurs Walked Through Diamonds: Constraining the Age of Early Cretaceous Footprints in Volcanic Crater Sediments

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ABSTRACT

The volcanic rocks of the Catoca Diamond Mine, northeastern Angola, were formed in an eruption ~118 million years ago. Above these rocks, researchers discovered Early Cretaceous mammalian, crocodylian, and sauropod dinosaur footprints in crater lake sediments. These footprints are among the relatively few vertebrate fossils from the Cretaceous Period (145-66 million years ago) found in Sub-Saharan Africa. The ~118 million-year-old age, which is the maximum age of the footprints, is provided by the uranium and lead isotope ratios in the zircon crystals from these volcanic rocks. The presence of dinosaur footprints limits the minimum age to 66 million years ago. Detrital zircons from the sediments were collected for uranium-lead dating because these lake sediments may contain not only the ~118 million-year-old zircons but also zircons from younger eruptions, which could more precisely constrain the maximum age of these sediments. Forty zircons have been analyzed from this sediment sample, yielding an age range of 2.9 billion years ago to 150 million years ago. None of these zircons was ~118 million years old. Work is currently underway to find more zircons and to determine why no ~118 million-year-old zircons have been found in the lake bed sample.

1. INTRODUCTION

The Catoca Diamond Mine in northeastern Angola is the fourth largest diamond mine in the world (Figure 1). A massive, rapid eruption brought the Catoca diamonds to the surface [1]. This eruption created the pipe structure of the mine and allowed for volcanic rocks called kimberlites to crystallize at the surface. Kimberlites contain the mineral zircon, which concentrates uranium. Uranium decays to lead at a regular rate, allowing researchers to calculate an age of crystallization for a specific mineral. Robles-Cruz et al. (2012) measured the U-Pb isotope ratios in these zircons and obtained an average age of ~118 million years ago (Ma). This eruption also formed a volcanic crater, which subsequently filled with water to form a lake. The sediments deposited in that lake are the target of this study.

Mateus et al. (2017) reported fossilized footprints preserved in crater lake sediments found inside the mine (Figure 2). These footprints mark the only known Cretaceous fossil locality found in the interior of Angola and suggest that mammals, crocodiles, and sauropod dinosaurs likely coexisted with each other and left their tracks in the mud of a lacustrine environment. It is also the first site in Angola to feature mammalian footprints. The size of the mammal prints is similar to that of modern raccoons [2]. These footprints are the only evidence of this animal's existence because skeletal remains have not been found. One of the sauropod footprints at this locality has a preserved skin

impression, which provides evidence for the skin structure on this sauropod.

U-Pb radiometric dating of zircons can be applied to sedimentary rocks. Because the crater lake sediments are a collection of detrital grains and contain no primary igneous minerals, an absolute age cannot be obtained for these strata. However, if the detrital grains include igneous minerals with radioactive elements, then the age of crystallization for these grains can be acquired. The age of the youngest grain can set the maximum age for these strata.

Mateus et al. (2017) argued that the crater lake sediments must have been deposited between the volcanic eruption ~118 Ma and the extinction of the dinosaurs 66 Ma. Because the current age range is ~52 million years of geologic time, the goal of this work is to further constrain the age of these sediments by ascertaining the ages of the detrital zircons. A more precise age range will provide a more accurate context in which to study these footprints and to relate the Catoca locality to the events and structures associated with the rifting of South America and Africa. Because this is a sedimentary sample, Precambrian-Paleozoic-age zircons from regional geologic features should be present, but there is an expectation of finding zircons with the age of ~118 Ma.

2. GEOLOGIC SETTING

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The Catoca Diamond Mine is a kimberlite pipe, or diatreme, that erupted 117 ± 0.7 Ma [1]. Kimberlites are ultramafic igneous rocks that form as magmas from partial mantle melt at 150-200 km depth [4 & 5]. Volatile gases propel the resulting magmas upwards. As these magmas rise to the surface at a rate of ~ 400 m/s, the internal pressures of the magmas and the volatile gases inside them become so large that the magmas become explosive, ripping pieces off the surrounding rock and erupting when they reach the water table. The resulting eruption excavates a crater at the top of the pipe. This pipe has intracrater epiclastic and volcanoclastic rocks that have been mapped by Pervov et al. (2011) (Figure 3). The pipe-filling pyroclastic rocks in Figure 3 contain the 117 ± 0.7 Ma zircons and represent the transition zone between the crater and the diatreme [3].

The Catoca Mine sits in the Lucapa Fault System, which was active due to the rifting of Africa and South America and allowed for the emplacement of deeply-sourced magmas [1]. The Lucapa Fault is a 50-90 km wide, $>1,200$ km long, Paleoproterozoic, NE-SW trending fault system that created a basin in northeastern Angola along a line that extends southwest to a transform fault in the Mid-Atlantic Ridge [1 & 3]. Most of the diamondiferous kimberlites in Angola are found in this fault system, where it is superimposed on the Kasai Shield, defined as the Precambrian basement. These faults created local extension and compression and allowed for kimberlite magmas to rise to the surface and erupt [1].

One of the minerals that crystallize in kimberlite magmas is zircon [1]. Zircons are minerals that are found in many igneous rocks and concentrate uranium, which is a radioactive element that decays into lead [6]. The isotopic ratios of uranium and lead can be measured to obtain an age for the rocks in which they crystallized. Robles-Cruz et al. (2012) used this technique to obtain the 117 ± 0.7 Ma age for the kimberlite pipe.

Currently, the age of the footprints at Catoca is constrained by the 117 ± 0.7 Ma eruption and by the overlying Calonda Formation, deposited 100.5-93.9 Ma [2], and the Cretaceous-Paleogene (K-Pg) extinction event 66 Ma [2]. This age range can be further constrained and quantified if researchers collect the appropriate detrital zircons from the crater lake sediments. We expected to find the 117 ± 0.7 Ma zircons because these zircons should be eroding out of the surrounding kimberlite rocks and be deposited in the sediments. We also hypothesized that we would find younger zircons because three concordant zircons that have a mean age of $\sim 79.0 \pm 6.8$ Ma have been reported in the southern Congo Basin, which confirms that volcanism did continue after the 117 ± 0.7 Ma eruption [7].

3. FOOTPRINTS

After the eruption, a section of the resulting crater collapsed. This collapsed section was filled with water and sediment, forming a lake that was 500-600 meters in diameter [2 & 8]. Various animals came to this lake and left their footprints in the sediments [2]. These sediments were soft enough for the animals to leave their footprints along the lakeshore but were also firm enough for the imprints not to be washed away by water.

When the volcanic crater collapsed, it was initially filled in by coarse-grained sediments from the crater walls,

described as silicified and carbonated tuff [3]. Later, when the collapsed section was submerged, fine-grained lacustrine sediments, described as tuffaceous sandstones, siltstones, and mudstones, were deposited on top of the coarse-grained tuff. These fine-grained sediments contain the fossilized footprints. Figure 4 shows the fining-up gradient found in the crater lake sediments.

The fossilized footprints are found on the southern side of the crater in a 10-20 m-thick sequence of sedimentary and pyroclastic rocks that interfinger with "dark brown, cherry red-brown to gray and pink tuffaceous sandstones, siltstones, and mudstones" [3]. The footprints are in the purple-red, shaly mudstones located at the top of the sequence. This sequence has no primary contacts between the underlying intracrater volcanoclastic rocks and the overlying intracrater epiclastic rocks. It has been broken up by faults, interpreted to have occurred along ring dislocations associated with the subsidence of the central part of the pipe [2 & 3]. This faulting has caused the sedimentary sequence to dip 60° - 70° toward the crater center [2] (Figure 5).

4. METHODS

Octávio Mateus (Institutions: GeoBioTec, Faculdade de Ciências e Tecnologia, Universidade Nova de Lisboa, 2829-516 Caparica, Portugal and Museu da Lourinhã, Rua João Luís de Moura 95, 2530-158 Lourinhã, Portugal) provided a sample from the footprint-containing sequence for this study. This sample is comprised of purple-red, fine-grained sediments (Figure 6).

We broke the sample into small chunks using a rock hammer and crushed it to an average size of ~ 250 - 500 microns with a rock miller. We then sieved the crushed material and divided it into these size fractions: >500 microns, 250-500 microns, 125-250 microns, and <125 microns. Afterward, we thoroughly cleaned the rock hammer, the counters, the rock miller, and the collection container inside the rock miller before processing the sample to avoid contamination.

Then, we separated the zircons and other high-density minerals from those with lower densities using high-density liquids. First, we poured Methylene Iodide-Diiodomethane (MI), which has a density of 3.3 g/cm³ into an open-top flask while the stopcock was in the closed position. Then, we drained the Catoca sediments into the flask and vigorously stirred them. Ten to fifteen minutes after stirring, the heavy minerals, including the zircons, settled to the bottom while the light minerals remained suspended. We then opened the stopcock and segregated the minerals from the heavy liquids using a filter-paper-covered funnel so that the clean MI would flow into the beaker below. Acetone was then used to clean the grains and filter paper of any leftover MI. After we cleaned the filter paper, we transferred the zircons to a glass petri dish for further work.

Sticking mounting tape onto a circular metallic plate, we picked 300 minerals we suspected were zircons with a needle and placed them onto the tape. We labeled this sample mount "YJ-1." Later, we placed sixty standardized zircons from Sample AS3 onto the mounting tape next to the 300 Catoca minerals, one standard for every five unknowns. The AS3 standards come from the Duluth Gabbro in Duluth,

Minnesota [9]. We then stuck an epoxy ring onto mount YJ-1 (Figure 7) and poured epoxy resin into the ring, ensuring that no air bubbles formed at the bottom. Soon after, we put the mount into an oven at ~ 50 °C to heat overnight.

The next day, we removed the epoxy ring off the pucks that formed from the cured resin (Figure 8) and peeled the mounting tape and excess epoxy off the metallic plate. We ground down the lip of the epoxy puck with 600-grit polishing paper. After we placed the puck into a lathe and shaped it to the appropriate size, we used water and 1200-grit polishing paper to begin polishing the sample. Then, we sprayed a 3-micrometer-sized, water-based diamond abrasive onto computer paper and polished the grain mount by rubbing it over the water-based solution eight times. We repeated this polishing process with the 1-micrometer-sized diamond abrasive. After polishing the mount, we rinsed it with water to remove the polishing solutions.

We then took the sample to the Scanning Electron Microscope (SEM) laboratory to determine which of the grains we picked were zircons. In the SEM, we attached the sample to a pin wrapped with copper tape to enhance conductivity. Under the back-scatter electrons mode (BSE), we could see the defining shape and luminescence of a zircon and could take chemical analyses of the grains using an Energy Dispersive Spectrometer. The spectrometer confirms the identity of the zircons with the presence of a zirconium element peak (Figure 9). We then took SEM images of the zircons under BSE (Figure 10), numbered them, and placed them into groups. We counted a total of 24 zircons for this grain mount. After that, we examined and photographed these zircons under cathodoluminescence (CL), which reveals zoning and internal structures in zircon crystals (Figure 11).

Due to the small number of zircons in YJ-1, we made another sample mount, named it "YJ-2," and repeated the steps we did for YJ-1 before examining it under the SEM. SEM examination revealed 16 additional zircons that we could use in our U-Pb isotope analysis. We photographed these zircons and analyzed them under CL.

We then cleaned mounts YJ-1 and YJ-2 with soap, deionized water, and low-concentration HCl to remove any residual lead from the mount's surface. The mounts were then coated in gold to create a conductive surface before placing them in the ion microprobe.

When the sample was in the ion microprobe, we selected one spot on each zircon for analysis, based on the desire to avoid any cracks and irregularities visible in the CL images. Then, we set up the analyses to run in a continuous chain of one standard and four unknowns per hour.

U-Pb isotope ratios of zircons are measured using a mass spectrometer. To measure these ratios, we need to separate all of the elements in the zircons so that we can pick out the uranium and lead isotopes. In the ion microprobe, we measure elements as ions with a charge of +1 so that we can move the atoms through the instrument with charged plates. A beam of oxygen atoms is emitted and hits the selected grain, generating secondary ions out of the sample. These secondary ions are then organized into a beam, and this beam takes a right turn into the magnetic sector of the instrument, with the lighter elements making a tighter turn than the heavier elements. In this way, the magnet splits the beam, with the heavier atoms separating from the lighter ones. As

the masses spread out, they form peaks at the end of the sector, and we can position the detectors to collect the uranium and lead isotopes [10, references therein]. We refer to this separation as the mass spectrum of secondary ionization mass spectrometry (SIMS).

We analyze standards in the ion microprobe because elements ionize differently under the +1 charge. To calculate accurate ages, we need to determine the relative sensitivity factor (RSF) to accommodate the error associated with the difference in ionization. To calculate the RSF between uranium and lead, we ran the standard zircons from Sample AS3 along with zircons of unknown ages. We ran one standard after every four or five unknowns to get useful statistics on our RSF and to monitor for any instrumental changes during our analysis.

5. RESULTS

Contrary to our expectation, none of the forty zircons analyzed gave an age of ~ 118 Ma or younger. The youngest zircon analyzed had a discordant age of over 150 Ma while the oldest zircon had a concordant age of 2.9 billion years ago (Ga).

Figure 12 is a "Concordia plot," made in Isoplot [11], that shows all of the ages we obtained from this project. Because zircons have two isotopes of uranium (235 and 238) that decay to two isotopes of lead (207 and 206), we can plot the expected decay over time of these two clocks against each other as X and Y axes. The dark blue line indicating agreement between the two clocks (Figures 12, 14, and 15) is referred to as "Concordia" and zircons that fall along this line are considered "concordant," meaning that the two uranium-lead clocks give the same age. These grains are considered to be the most reliable since they have not been disturbed by later geologic events. Concordia plots, therefore, illustrate the accuracy of the zircon ages, where the ages are represented as ellipses. If these ellipses are large and do not plot on the Concordia, then the ages have more substantial errors, or the two ages disagree. These ages are defined as "discordant." The depicted ellipses represent 2-sigma error (95% probability) [12].

Figure 13 is a histogram that displays the distribution of the best ages for the forty zircons. Figure 14 is a Concordia plot that clearly illustrates the discordant ages. Zircons have discordant ages when they lose lead due to metamorphism or a later volcanic heating event. Because of lead loss, the zircon yields a U-Pb ratio that suggests a younger age than the actual age of the grain. We can fit a line through the discordant ages to determine the old intercept (the age of crystallization) and the young intercept (the age of lead loss) of the curve. If we have many discordant ages, then the intercepts will be more accurate. We fit a line through two of the discordant ages on our plot because these are only the ages we could use to determine the old and young intercepts on the curve (Figure 15). The old intercept was 2590 ± 39 Ma, and the young intercept was 101 ± 16 Ma. Although these intercepts imply a young age that could be associated with the Catoca eruption or a later volcanic event, this interpretation is speculative, and only a concordant young age would definitively date the sedimentation age of these rocks.

6. DISCUSSION

A broad distribution of zircon ages is expected in sedimentary rocks, such as the crater lake sediments at Catoca. Sedimentary grains, such as the zircon in Figure 10, are weathered out of their source rock and transported a considerable distance before being deposited in the crater lake sediments. Judging by its well-rounded shape and dull exterior, this zircon is probably ~2 Ga and most likely originated in the igneous rocks that make up the Precambrian basement of the Kasai Shield, along with the eleven other zircons that date to this age.

The 150 Ma zircon and the other discordant grains suggest that more data would reveal a younger age, such as the 118 Ma age of the Catoca volcanic deposits. However, without a concordant zircon at this age, we cannot confirm its accuracy, but it prompts a search for more zircons.

Surprisingly, we did not find any ~118 Ma zircons out of the forty we analyzed because these zircons should have been eroded out of the kimberlite rocks that are near the crater lake sediments. There may be ~118 Ma zircons in the 10-20 m sedimentary sequence, and we have to process more materials and analyze more zircons. It is also probable that our sample was not sufficient since it was composed of fine-grained sediments. Since zircons are dense minerals, we may have a higher chance of finding the ~118 Ma or younger zircons in the coarse-grained sediments at the bottom of the sedimentary sequence.

7. CONCLUSIONS

The Catoca Diamond Mine is an active mine where Mateus et al. (2017) reported mammalian, crocodylian, and sauropod dinosaur footprints in crater lake sediments. Robles-Cruz et al. (2012) obtained an age of 117 ± 0.7 Ma for the Catoca volcanic units by measuring the U-Pb isotope ratios in the zircons found inside the kimberlite rocks next to the crater lake sediments. Mateus et al. (2017) estimated that the sediments and the fossilized footprints have an age range between 117 ± 0.7 Ma and 66 Ma.

The U-Pb age dating technique can be applied to zircons found in the crater lake sediments. The ages obtained from this analysis ranged from a concordant age of 2.9 Ga to a discordant age of 150 Ma. None of the zircons gave the expected age of ~118 Ma or younger. The 2 Ga zircons came from the surrounding Precambrian basement of the Kasai Shield. The discordant 150 Ma age could be younger, but there is not enough evidence to confirm the younger age. We expected to find ~118 Ma zircons because they should be eroding out of the local kimberlite rocks. We predict that we will find these younger zircons by processing additional coarse-grained material. Therefore, we plan to process and analyze more Catoca samples.

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9. ACKNOWLEDGEMENTS

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10. APPENDIX



Figure 1. **A.** Map showing the location of the Catoca Diamond Mine (Modified from Mateus et al., 2017, By courtesy of Jacobs, L.L. and Polcyn, M.J.). **B.** Aerial photograph of the Catoca Diamond Mine from Google Earth.

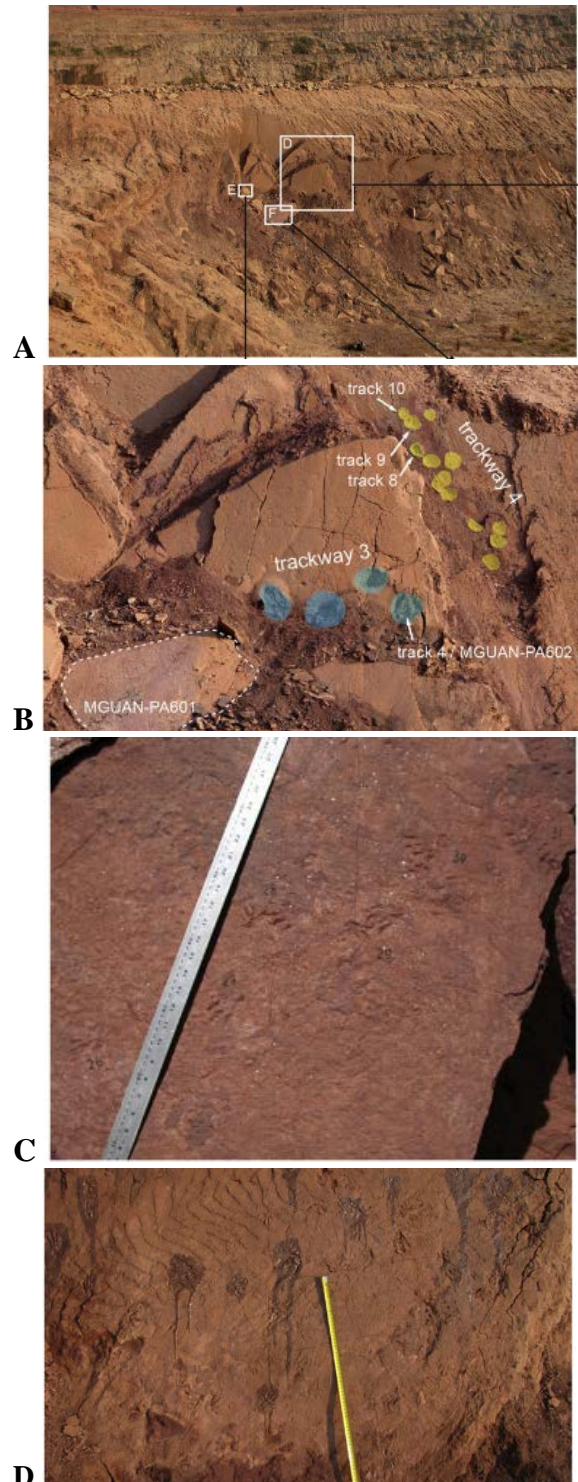


Figure 2. **A.** Early Cretaceous sedimentary sequence including mammalian, crocodilian, and sauropod dinosaur footprints. **B.** The annotated overview perspective of the sauropod dinosaur trackways 3 and 4. **C.** Overview perspective of the crocodilian trackway 1. **D.** Overview perspective of a trampled surface bearing mammalian footprints and crocodilian trackway 2 with ripple marks on

the top left (Modified from Mateus et al., 2017, By courtesy of Jacobs, L.L., and Polcyn, M.J.).

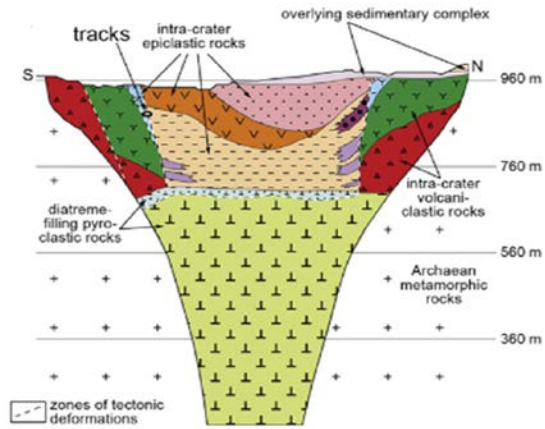


Figure 3. Stratigraphy of the Catoca kimberlite pipe. The dated unit at 117 ± 0.7 Ma is the core of the pipe, which is pale green in the diagram. The sampled unit is pointed out as the light blue section labeled "tracks." (Modified from Mateus et al., 2017, By courtesy of Jacobs, L.L. and Polcyn, M.J.).

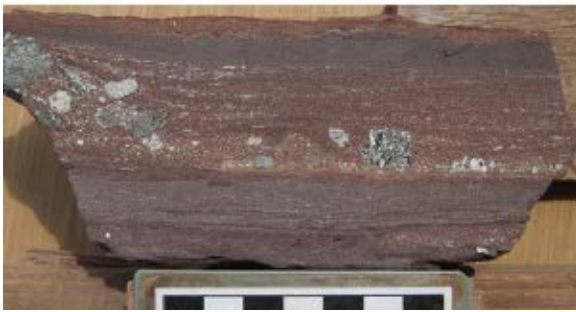


Figure 4. Cross-sectional view of a sample from the mine with mammalian footprints on its surface. Sample contains intercalation of tuffaceous sandstones, siltstones, and mudstones. The sandstone contains metamorphic rock clasts and phlogopite flakes. Desiccation cracks are found at the top of upper mudstone layer (Modified from Mateus et al., 2017, By courtesy of Jacobs, L.L. and Polcyn, M.J.).



Figure 5. The sedimentary sequence containing fossilized mammalian, crocodylian, and sauropod footprints. It is tilted due to subsidence of central part of the pipe. (Modified from Mateus et al., 2017, By courtesy of Jacobs, L.L. and Polcyn, M.J.).



Figure 6. The Catoca Sample with scale.



Figure 7. YJ-1 mount with epoxy ring on top.



Figure 8. Epoxy puck made from dried epoxy resin.

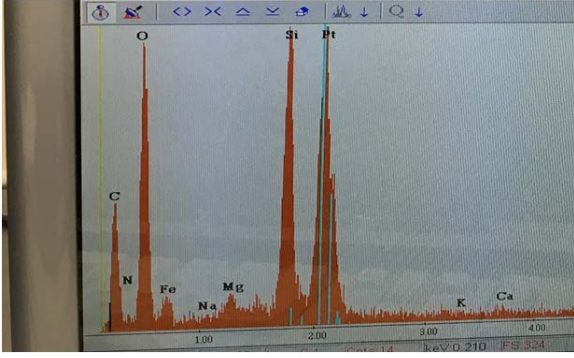


Figure 9. Chemical analysis taken of a zircon. High spikes in the elements silicon, oxygen, and zirconium (blue lines) confirm the presence of a zircon.

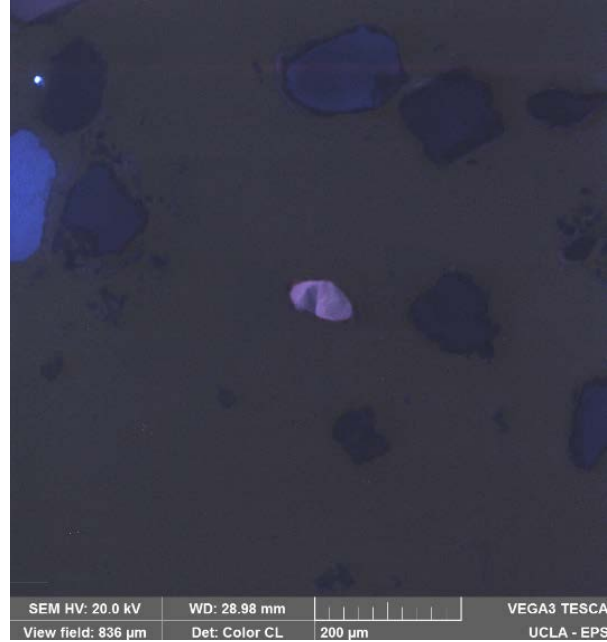


Figure 11. Catoca zircon crystal under a cathodoluminescence ray.

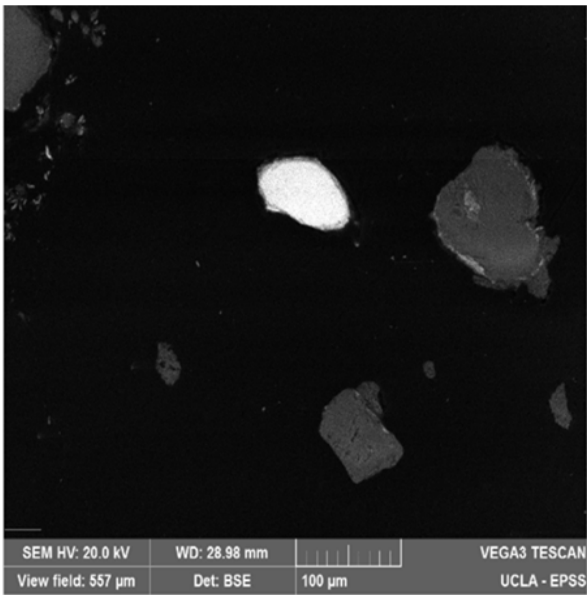


Figure 10. Catoca zircon crystal under the scanning electron microscope (SEM). The zircon is the highly luminescent, well-rounded mineral in the center.

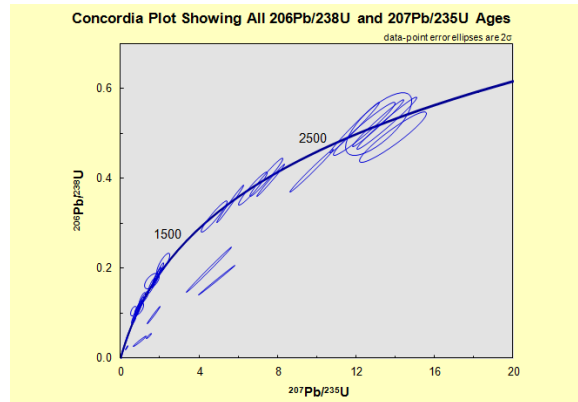


Figure 12. Concordia plot of all zircons ages. Ages are represented by ellipses. Small ellipses that plot right on the curve are very accurate and concordant whereas large ellipses that do not plot on the curve are less reliable and are discordant.

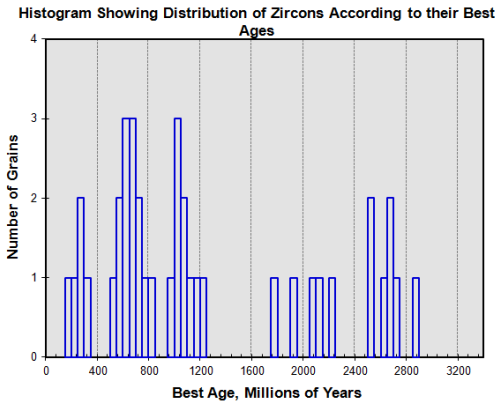


Figure 13. Histogram showing age distribution for forty zircons analyzed. The oldest zircon age is more than 2.9 Ga, and the youngest zircon is 150 Ma. For ages older than 1.7 Ga, the best age is defined as the age given by the $^{206}\text{Pb}/^{238}\text{U}$ isotope ratios, and for ages younger than 1.7 Ga, the best age is defined as the age given by the $^{207}\text{Pb}/^{206}\text{Pb}$ isotope ratios.

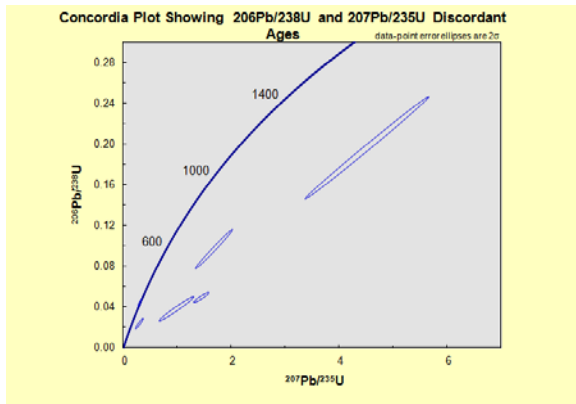


Figure 14. Concordia plot that clearly demonstrates all discordant ages.

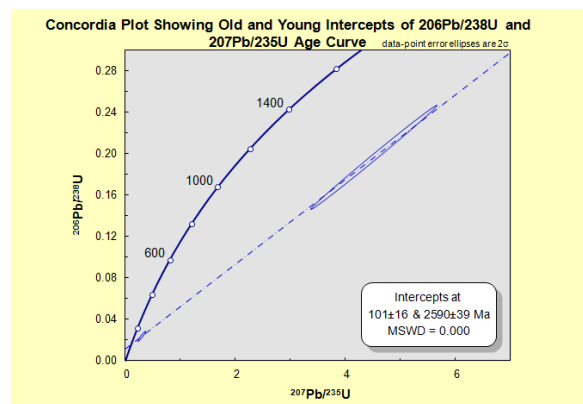


Figure 15. Concordia plot that shows the two discordant ages that were used to fit a line to determine the old and young intercepts.