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Spring 2016

Testing of the late-ordovician Pre-GICE warm water carbonate hypothesis in Alabama

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Testing of the Late-Ordovician Pre-GICE Warm

Water Carbonate Hypothesis in Alabama

An Honors Program Project Presented to

the Faculty of the Undergraduate

College of Science and Mathematics

James Madison University _______________________

by Brandon Christopher Euker and Stacey Elizabeth Law

May 2016

Accepted by the faculty of the Department of Geology and Environmental Science, James Madison University, in partial fulfillment of the requirements for the Honors Program.

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PUBLIC PRESENTATION

This work is accepted for presentation, in part or in full, at the Southeastern Section of the Geological

Society of America Meeting in Columbia, South Carolina on April 1, 2016.

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Acknowledgements

We would like to thank our research Advisor Dr. Stephen Leslie for his constant advice and guidance throughout the course of this project. We would also like to thank Dr. Steve Baedke and Dr. John Haynes for acting as readers for this thesis and providing valuable feedback. Finally, we would like to express our gratitude to Paige Quinton and Dr. Ken MacLeod of the University of Missouri for providing their facilities, their hospitality, and their time to aid in the completion of this thesis; their assistance was integral to its success.

This research was funded by NSF EAR 1323444 to Kenneth G. MacLeod, NSF EAR 1324577 to Stephen A. Leslie, and NSF EAR 1324954 to Achim D. Herrmann.

Abstract

The Guttenberg Carbon Isotope Excursion (GICE) (uppermost Sandbian-lower Katian, Late Ordovician) has been suggested to represent the transition from a Cambrian-Ordovician greenhouse world to Late Ordovician icehouse world. This transition is thought to coincide with a proposed shift from deposition of warm water carbonate rocks to cool water carbonate rocks in the North American midcontinent. We used oxygen isotopes $(\delta^{18}O)$ of conodonts to test the idea that the rocks below the GICE interval represent a consistently warm environment. Conodonts were isolated from samples of the Chickamauga Group collected at the Tidwell Hollow section in Blount County, AL, from the Red Mountain Expressway section in Birmingham, AL, and from Dickeyville, WI. These successions underlie strata that biostratigraphically correlate with the GICE interval. Conodonts at Tidwell Hollow include *Appalachignathus* sp. and *Phragmodus flexuosus* near the base of the Chickamauga Group and *Oulodus oregonia* and *Aphelognathus kimmswickensis* near its top. These taxa indicate that the interval studied ranges from the *Plectodina aculeata* Zone to at least the *Plectodina tenuis* Zone, spanning from below and through the GICE. Conodonts from the Red Mountain Expressway section were collected only to 22m below the Millbrig K-bentonite, where *Belodina compressa* and *Curtognathus* spp. dominate the fauna. At 10m below the Millbrig, *Polyplacognathus ramosus* occurs and the top of the section contains a fauna that includes *Phragmodus undatus* and *Icriodella superba*. Results indicate that the sampled interval ranges from the *Belodina compressa* Zone to the *Phragmodus undatus* Zone, an interval which is below and up to the GICE. The conodont $\delta^{18}O$ values indicate that the rocks below the GICE in Alabama represent a consistently warm environment throughout the Chickamauga Group below the Millbrig K-bentonite Bed, supporting the idea that

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the pre-GICE environment was relatively stable, and that the GICE, along with the changes in δ^{18} O in the GICE interval, could reflect a perturbation in the Ordovician climate.

Introduction

The purpose of this thesis was to test the null hypothesis that the rock records at Red Mountain, AL; Tidwell Hollow, AL; and Dickeyville, WI represent a consistently warm environment before the Guttenburg Carbon Isotope Excursion (GICE).

Figure 1. This figure from Pope and Read (1998) displays the stratigraphy and frequency of glacial deposits during the Ordovician. The main focus of the figure as it pertains to this study is the increase in glacial deposits shown occurring in the middle Ordovician. This change in lithology was how the shift from a warm to cool depositional environment was first suggested for this time period (Pope and Read, 1998). This shift was later correlated to the existence of the GICE, which occurs at the same time (Young and Saltzman, 2005).

The GICE has been suggested to correlate with a shift from a warm water depositional environment to a cool water depositional environment (Young and Saltzman, 2005). The idea that a shift from warm water deposition to cool water deposition occurs in this time frame, as depicted in Figure 1, was suggested by Pope and Read in 1998. This shift was suggested based on a change in limestone deposition from micrites to phosphatic rich grainstones and wackestone that correspond to an increase in glacial deposits in Gondwana. After the change in depositional environments was proposed, the GICE

was discovered to occur at the same time as the shift (Young and Saltzman, 2005).

The GICE is a positive carbon isotope excursion that occurs during the late Ordovician (late Sandbian, early Katian). The Hirnantian Carbon Isotope Excursion (HICE) is a significant positive δ^{13} C excursion that occurred later in the Ordovician and is widely recognized as the beginning of the Ordovician glaciation. Young and Saltzman (2005) suggested the GICE may actually be the first indication of changing climatic conditions. The temperature constrained by δ^{18} O for the GICE is not as well constrained as it is for the HICE, as can be seen on Figure 2 below. The HICE is defined by multiple $\delta^{18}O$ data points that exhibit a clear curve deflecting to higher δ^{18} O and therefore cooler temperatures. For the interval containing the GICE however, there are only two $\delta^{18}O$ data points that do not exhibit a significant trend. Because the $\delta^{13}C$ and

Figure 2

Figure from Bergström et al. (2009) that spans the entirety of the Ordovician. Displayed are the conodont zones, the $\delta^{13}C$ record and the $\delta^{18}O$ record. The $\delta^{13}C$ record is well documented whereas the δ^{18} O record is weaker, in particular around the GICE. The goal of this study was to analyze the area around the GICE in greater resolution.

 δ^{18} O data in the HICE both exhibit excursions, one hypothesis is that the δ^{13} C data for the GICE would indicate a similar $\delta^{18}O$ excursion, which could be seen with greater sample density. This thesis tests whether the time period prior to the GICE is indeed a stable warm environment as suggested by the rock record.

To test the warm water hypothesis, rock samples were collected at Red Mountain, Tidwell Hollow and Dickeyville. These locations would have been equatorial during the Ordovician, between 15º and 30º S latitude, supporting the idea that the paleoclimate was warm

Figure 3. A reference map of the sampling sites for this study ($DV =$ Dickeyville, $RM = Red$ Mountain, $TH = Tidwell$ Hollow). In addition, numbered sites correspond to sections in Fig. 8 that are included to illustrate the correlation of data with Quinton et al., in review.

(Rosenau et al., 2012). Figure 3 shows the three sampling sites at their present-day latitudes. These locales can be correlated using K-bentonite beds, which are layers of altered volcanic ash deposited originally as tephra during a volcanic event. K-bentonite beds are valuable for

Figure 4. This diagram depicts the regional correlation of the K-bentonite beds with late Middle Ordovician stratigraphy in North America. The sampling sites for this paper are the Chickamauga Group in Alabama and the Guttendburg Formation in Wisconsin, which were correlated using the Millbrig and Deicke as seen on the diagram (Leslie, S. A., & Bergstrom, S. M., 1997).

stratigraphic correlation because ash layers are deposited as a single geologic event throughout a large region, allowing the stratigraphy to be understood through relative time as shown below in Figure 4 (Leslie and Bergstrom, 1997). In addition, these sections can be correlated using sequence stratigraphy.

The M4/M5 boundary is a third order sequence boundary placed between the Millbrig and Deicke in the Upper Mississippi Valley and Minnesota. Some workers place it above the Millbrig in Oklahoma, Alabama, Virginia and Kentucky. This boundary must be either between the Millbrig and the Deicke or above them; it cannot exist in both places or else it would be diachronous. This is a discrepancy which must be resolved, a feat that is difficult to achieve due to the fact that there is an extremely small rock record present between the Millbrig and the Deicke and

we are correlating across outcrops separated by hundreds of miles.

In these sections, we analyzed oxygen isotopes $(\delta^{18}O)$ extracted from conodonts, which are ancient soft-bodied organisms with microfossil phosphatic teeth like those seen in Figure 5. These teeth have the chemical formula $Ca₅(PO₄, CO₃, F)₃(OH, F, CL, CO₃),$ which contains the strongly covalently bonded phosphate ion. The oxygen in the phosphate ion can be analyzed for $\delta^{18}O$, which is the ratio between the oxygen isotopes ${}^{16}O$ and

Figure 5. Ordovician conodont species as photographed by Dr. Stephen Leslie of James Madison University

 18 O, and is tied to temperature of the water where the organism lived (Quinton and MacLeod, 2014). The $\delta^{18}O$ ratio is inversely correlated to changes in temperature. This is due to the fact that during warmer time periods the higher temperatures allow more of the heavier ^{18}O isotope to evaporate, leaving more of the lighter ¹⁶O isotope and thus decreasing the ratio between the two. This ratio is reflected in the phosphatic conodont teeth, which are formed using oxygen in the ocean waters; when more of the ^{18}O isotope is evaporated there is less of it available for development. In this way, the composition of the teeth depends on the $\delta^{18}O$ ratio at the time of formation.

All three areas record carbonate deposition in shallow marine waters, but Red Mountain continues to a shelf slope. The deeper conditions at Red Mountain lead us to consider the idea that we may get slightly cooler temperature data from those samples

Methods

Samples were collected from Dickeyville, WI in October 2013, and samples from Tidwell Hollow and Red Mountain were collected in December 2013; two of these outcrops are pictured below in Figures 6 and 7. Using the Millbrig and Deicke K-bentonite volcanic ash beds present in all three sections as datums, we sampled for conodonts every five meters along each section, filling a bag with many fist-sized or smaller rocks hewn from the outcrop using picks and rock hammers. Individual samples to be used for carbon isotope analysis were taken in conjunction with the conodont samples.

Figure 6. An outcrop photograph from Dickeyville, WI, taken in October 2013. Only part of the section is pictured here.

Figure 7. Field work at the Tidwell Hollow outcrop in Alabama in late December 2013. Pictured to the left is Dr. Achim Herrmann with an LSU student.

In total, 34 samples were fully processed. The samples were initially crushed to gravel size using the Bico Chipmunk rock crusher. 3-4 kg samples were then placed in a mixture of 14 L water, 310 g CaCO₃, 18.6 g Tri-Calcium-Phospate, and 1.5 L formic acid and allowed to dissolve for 24-48 hours. Once most of the rock material had dissolved the samples were run through ASTM No. 35, No. 140, and No. 200 sieves. The sample which caught on the No. 200 sieve was taken to the lab for further processing.

The sieved samples were then suspended in LST heavy liquid. LST is a tungstate heavy liquid with a specific gravity of 2.85 that allowed only parts of the sample with a specific gravity of greater than 2.85 to sink in the liquid. Since conodonts have a specific gravity of greater than 2.85 they sink in LST. The mixture was stirred by hand using a glass rod to ensure homogenization and was then run through a centrifuge at 3000 RPM for seven minutes. Following the centrifuge run, the bottom of the sample container was exposed to liquid nitrogen until frozen. The top of the sample was poured off and run through the vacuum filtration system pictured in Figure 8 to preserve the light fraction and the conserve heavy liquid. The frozen

portion of the sample was placed in the oven at low temperature $(< 100$ ºC) and allowed to melt. Following the melting, the heavy fraction of the sample was filtered through silkscreen material to save the sample and the heavy liquid. The filtering process was aided by vacuum pressure to remove the maximum volume of liquid from the sample. The dried heavy fractions of the samples were rinsed and set out to dry after vacuuming was complete.

Figure 9. Conodonts on microscope slides, separated by species. The paintbrushes seen in the upper righthand corner were used to pick individual conodonts out of the samples.

Once completely dry, these heavy fraction portions run through sieves of 40,60,80, and 100 microns in diameter. The material that was less than 100 microns was collected in the pan at the bottom of the sieve stack and

Figure 8. Sample is placed on filter paper and vacuumed to remove high density LST, which can be seen collecting in the bottom of the container.

stored for future examination. The material that was retained in all of the other sieves (greater than 100 microns in size) was saved for picking, due to the higher probability of containing conodont elements. The useful portions of the samples were then poured out onto picking trays and the individual elements were gradually removed

by hand using a microscope and a fine-tipped wet paintbrush. The elements were placed on slides, as seen in Figure 9, and separated by species for the purpose of biostratigraphic analysis. Once complete, the slides were taken to Missouri to undergo the trisilver phosphate methods for oxygen isotope analysis.

Trisilver Phosphate Methods

Samples were prepared for analysis by isolating the $PO₄⁻³$ anion from the highly crystalline bioapatite with the chemical formula $Ca₅(PO₄, CO₃, F)₃(OH, F, CL, CO₃)$. This was done using a modified technique from LaPorte et al. (2009) . The PO₄⁻³ anion was isolated due to its strong covalent bond which holds the original oxygen isotope and phosphorous together from the time of formation. This means that the oxygen isotope which is analyzed is truly representative of the isotopic composition of the water during the Ordovician.

 Samples ranging in weight from 250 to 600 μg (10-60 elements, not species specific) were placed in 1.5 mL centrifuge tubes with 1 mL of 4-6% sodium hypochlorite (NaOCl) for 24 hours to clean the surface and remove any residual glue from the elements. The samples were then rinsed 4-5 times with deionized water and placed in an oven at a temperature $≤50$ °C until dry (approximately 24 hours).

In order to dissolve the apatite, 0.1mL of 0.5M nitric acid (HNO₃) was added. Dissolution was complete after 24 hours and the PO_4^{-3} anion was present in solution as H_3PO_4 . Samples were centrifuged at 10,000 rpm for two minutes, thereby consolidating residual organic matter or other insoluble materials at the base of the tube.

Figure 10 below shows columns filled with a cation resin (AG 50W-X8) that were prepared and conditioned with a rinse of 20 mL of 6.0M HCl followed by approximately 60 mL of deionized water to elute any residual chloride ions. The dissolved apatite was transferred to the column via pipetting, as shown in Figure 11, where the H_3PO_4 was eluted in 1 mL of

deionized water and collected in 2 mL centrifuge tubes. Cations present in solution (e.g. Ca, Sr and REEs) were bound to the resin.

Figure 10. The columns at University of Missouri sit awaiting sample introduction. Sample is filtered through the columns, labeled with letters, as the orange resin captures cations. As the sample filters through, the desired product is captured in the 2 mL centrifuge tubes (pink, at forefront of figure).

Figure 11. The samples, now in the form of dissolved apatite, are individually transferred to the columns via 100 μ L pipette.

Next, 0.15 mL of 2M ammonium hydroxide (NH4OH) was added to the samples in order to increase the pH of the solution. This was followed by the introduction of 0.15 mL of a silver amine solution (comprised of $0.4M$ AgNO₃ and $0.7M$ NH₄NO₃). An additional $0.10mL$ of ammonium hydroxide was added to the samples if a yellow foam formed in the vials; this would have inhibited the ability for crystals to grow. All of the Tidwell Hollow and Red Mountain samples were subjected to the supplementary amount of ammonium hydroxide. Samples were placed in an oven at temperatures $\leq 50^{\circ}$ C. The pH of the solution decreased as NH₃ slowly

evaporated, thereby promoting the growth of silver phosphate crystals (Ag₃PO₄) as seen below in Figure 12.

Figure 12. Silver Phosphate Crystals are inspected under the microscope to ensure a lack of contamination by fibers, etc., before being packed in silver boats

Figure 13. Dried crystals weighed and packed in 3.5 x 5 mm pressed silver capsules, then transferred to a plastic storage container to await introduction to the mass spectrometer.

The samples were removed after 24 hours and centrifuged at 10,000 rpm for three minutes. The supernatant was removed and the crystals were rinsed with deionized water four to five times before being returned to the oven.

The dried crystals were weighed out and packed in 3.5 x 5 mm pressed silver capsules to await isotopic analysis, a process which is seen in Figure 13 above. The initial IRMS run was completed using NS-120c standards; the data collected from this run was averaged to provide a correction value of 22.6. Samples were analyzed using a TC-EA (high-temperature conversionelemental analyzer) at 1400°C and analyzed by a continuous flow Finnegan Delta-Plus XL gas source mass spectrometer.

High-temperature reduction (HTR) methods reduce the needed sample size significantly (0.2-1.0mg of Ag3PO4) while maintaining a high degree of accuracy (0.3‰ standard deviation) with the use of reference standards during analyses (Vennemann et al., 2002). This method involves the thermal reduction of Ag3PO⁴ within a high-temperature conversion-elemental analyzer in a glassy carbon tube filled with glassy carbon chips and a graphite reaction cup (Vennemann et al., 2002). The sample is held at a temperature of 1400°C and the resulting gases (CO) are transported by a stream of helium through a gas chromatograph under continuous flow (Vennemann et al., 2002). The relative simplicity of this method and the small sample requirements makes the HTR method especially attractive for studies using conodont elements as the source of biogenic phosphate.

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Data and Results

Tables 1, 2, and 3 display the data from conodonts from Red Mountain, Tidwell Hollow, and Dickeyville, respectively. The sample IDs are arbitrary names used to identify the samples once removed from the field. The description 'mixed' under the Sample heading indicates a variety of species analyzed from the sample. In samples where a particular species was numerous enough, the species in question is designated in that column. Table 1 contains data that was procured by Paige Quinton of the University of Missouri.

Discussion

Our results tentatively support the null hypothesis that the rock records at Red Mountain, AL; Tidwell Hollow, AL; and Dickeyville, WI represent a consistently warm environment pre-GICE. Unfortunately, due to the lack of conodonts recovered in some samples and poor crystal growth while processing using the trisilver phosphate methods the data density was not as robust as originally planned. However our data combined with data from Dr. Leslie, Dr. Herrmann, Dr. MacLeod and Quinton Page presents a more compelling argument that the time period prior to the GICE was a relatively stable warm depositional environment. Sparse results from Tidwell Hollow can be attributed to low conodont recovery and small element sizes, which led to poor crystal formation. Similar problems were experienced with the Red Mountain samples; therefore, the Red Mountain data displayed in this paper was previously generated by Page Quinton of the University of Missouri.

A complication to the comparison among locations is the difference in conodont alteration index (CAI) between sample locations. CAI is an estimate of the maximum temperature to which a sedimentary rock has been exposed after deposition. The Dickyville samples have a low CAI, around 1, while the Red Mountain and Tidwell Hollow conodonts have higher CAIs of around 4. While conducting this study, we found that the samples with higher conodont alteration indices tended to required more elements to yield sizeable crystals, though a more thorough study would be required to determine if this occurrence can be seen throughout the rock record. High CAI was indicated by the notably dark, opaque color of these conodont elements. This is a possible explanation for the lack of usable data from Red Mountain samples, in conjunction with small elements that produce powdery crystals that more often grow on the sides of the vial instead of the surface.

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The data sets at each of the six sampling sites, indicated by the transparent red arrows in Figure 14, show fairly consistent trends. In the Upper Mississippi Valley, the data initially shows consistent $\delta^{18}O$ values that soon trend to the left into and through the GICE before trending back towards their original measures (Quinton et al., in review).

Figure 14. Data from this study in Alabama (Tidwell Hollow and Red Mountain) and Dickeyville, Wisconsin, represented by light gray circles, is correlated with similar data sets from three other studies, represented by black circles, to illustrate the conditions surrounding the proposed location of the GICE in each section. This study looked at temperatures pre-GICE, and these six locations were chosen to see if there was lateral change. The locations were correlated using the K-bentonite beds (Millbrig and Deicke) represented on this diagram by red lines and through biostratigraphy. The M4/M5 boundary, which is a third order sequence boundary, is indicated by a gray line across all locations. The data points are plotted with the measured $\delta^{18}O$ values along the bottom axis and their relative corresponding temperature along the top. Numerical values were not used to due to some discrepancies that exist between different calculation methods.

The data in Minnesota shows a nearly identical trend; data in the lower part of the section exhibits δ^{18} O values between 19.5 to 21, but shifts to between 18 and 19.5 just before the GICE interval before shifting back towards and remaining at 20 (Buggisch et al, 2010). In the Oklahoma section, the oxygen data shifts from $\delta^{18}O$ values of 19-20 towards values between 18-19.5; the leftward trend persists into the GICE (Rosenau et al., 2012). The data from this study helped define this trend in Alabama, where $\delta^{18}O$ values were initially measured between 19-20.5 in the lower part of the section before shifting dramatically towards 17 across the M4/M5 boundary. The one site where this trend is not evident is Virginia, where the few data points that have been collected show a constant trend of $\delta^{18}O$ values falling between 19 and 20.5 throughout the section (Quinton et al., in review). Finally, the trend can be seen in Kentucky as the $\delta^{18}O$ values shift from 19.5-20.5 at the bottom of the section to towards 18.5 at the top of the section (Buggish et al., 2010). This overall inclination towards lower $\delta^{18}O$ values actually suggests a warming trend through the GICE interval, which is unexpected because it is not consistent with the trend indicated by the δ^{13} C values that correlate with the GICE.

A possible reason that a large shift in $\delta^{18}O$ values was not seen at any of these sampling sites was due to the fact that they were located at a similar equatorial paleolatitude (between 15- 30 degrees) during the Ordovician. This means that during a glaciation event or an icehouse world, the temperature changes in these areas might not have been large enough to cause a shift in the isotopic record. To further investigate on this possible cause, another project was started where samples were collected in Sweden during the summer of 2014. Sweden was at a higher paleolatitude during the Ordovician and thus could reveal a shift in temperature that is not seen at these sites. The results of those samples are not part of this study, however, and have yet to be completed.

Conclusion:

In this study, we conducted field work in Alabama at the Tidwell Hollow section in Blount County and the Red Mountain Expressway section in Birmingham, as well as in Dickeyville, Wisconsin. Our data was supplemented with data from the Upper Mississippi Valley, Minnesota, Oklahoma, Alabama, Virginia, and Kentucky that was collected as part of a larger study of the GICE interval (Quinton et al., in review). Despite processing 34 samples, we had sparse data because of poor conodont recovery and smaller than average elements, as well as a possible inverse correlation with CAI values. However, the data we were able to generate in conjunction with the data collected by others does tentatively support the null hypothesis that the rock records at Red Mountain, AL, Tidwell Hollow, AL, and Dickeyville, WI reflect a consistently warm environment prior to the GICE.

In addition to the stable warm environment prior to the GICE, there appears to be a warming trend both into and during the GICE at a majority of the sample locations. This is not entirely surprising because these locations were equatorial at the time of formation; although cooling may have been occurring at higher latitudes, it is possible that this was not reflected in the tropics. This creates the desire for sampling locations at different latitudes and thus we await the results of the fieldwork in Sweden.

In the future, it would be recommended to collect larger sample sizes at Red Mountain and Tidwell Hollow to ensure adequate conodont yield and thus adequate crystal growth. To see if there is a quantifiable change in isotopic trends at non-equatorial paleolatitudes, work must be done to process the samples collected in Sweden and complete isotopic analysis.

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