Carbon, oxygen, and sulfur isotope excursions in a Neoproterozoic cap carbonate from the Otavi Group, Namibia, Africa

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ABSTRACT

This essay records and explains the isotope excursions of carbon, oxygen, and sulfur over a 30m outcrop from a cap dolostone from the Otavi group in Namibia, Africa. The Cryogenian Period in the Neoproterozoic (1000-542 Ma) was plagued by alternating periods of mass if not total glaciation. Icehouse conditions followed by extreme greenhouse and weathering lead to a variety of situational rock formations such as Banded Iron Formation, glacial deposits, and cap carbonates. Cap carbonates are indicators of mass-terrigenous weathering and large influx of these sediments into the oceans, which settled and developed in thick packets. These carbonates, which can be limestone or dolostone, record paleo isotopes of a variety of elements, including but not limited to carbon, oxygen, sulfur, manganese, and other trace elements, that can act as proxies for conditions during deposition. The purpose of this project is not to conduct revolutionary research, but rather to contribute data to an ongoing scientific discussion of snowball Earth and provide more evidence for significant isotope excursions in oxygen, carbon, and sulfur.

Keywords: Neoproterozoic; cap-carbonate; sulfur isotopes; carbon isotopes; oxygen isotopes
INTRODUCTION

During the Neoproterozoic (1000-542 Ma), the Earth experienced at least two mass-glaciations in which ice sheets reached low latitudes and even possibly encapsulated the entire planet. This period, which is predicted to have had continents concentrated at sub-tropical to tropical latitudes, is called snowball Earth and was caused by two main driving forces: albedo effect and widely fluctuating atmospheric carbon dioxide levels. The albedo, a positive feedback system (Hoffmann & Schrag, 2002), became so intense with no strong competition to stop it that surface temperatures dropped so low that ice caps began to encroach on the tropics. Synchronously, the earth was experiencing unstable atmospheric carbon dioxide levels, and this negative feedback loop meant that when carbon dioxide was high, temperatures rose and therefore weathering of the continents increased. However, this increase in silicate weathering caused atmospheric carbon dioxide to fall, and temperatures plummeted for such a long period that the oceans, which are normally resistant to cold periods, were helpless. One of these usually long cold periods is when the ice sheets began to form at the poles and the albedo effect began to take control. Depending on sea-ice albedo, ice could reach more than a kilometer thick (albedo 0.6 would result in about 1.4km thick ice) (Hoffman & Schrag, 2002).

Whatever the case, this period of glacial freeze was interrupted by thaws due to ongoing tectonic and volcanic activity. Because volcanoes are not impacted by surface events, they continued to introduce carbon dioxide and sulfur gases, which eventually melted holes locally in the ice. These gases accumulated in the atmosphere and because the sinks for CO$_2$ such as silicate weathering and photosynthesis were unavailable (Kirschvink, 1992), the Earth rapidly shifted to a greenhouse. Sea ice thinned but continental ice actually expanded due to a stronger hydrological cycle. Studies indicate that it would have taken a few million years for enough CO$_2$
to accumulate to raise temperatures enough for tropical ice to melt perennially, and once this occurred, the greenhouse effect was runaway and the Earth deglaciated in possibly as little as 2000 years (Hurtgen et al., 2006) (Fig. 1) due to reverse ice-albedo feedback (Caldeira & Kasting, 1992; Crowley et al., 2001).

Following these meltings, the surface was very vulnerable to erosion. Rocks would have been severely cracked by freeze-thaw action, and encroaching and receding ice sheets probably had torn up the surface. This, as well as a rapid increase in temperature and precipitation, would
introduce very large amounts of terrigenous sediment into the oceans which were deposited in large bundles called cap carbonates. That, coupled with the fact that the large amounts of atmospheric carbon dioxide would precipitate calcium carbonate in warm surface waters (Hoffman et al., 1998), produced these cap carbonates globally. These rocks “capped” locally formed Banded Iron Formations and limestones composed of sea floor cements (Hoffmann & Schrag, 2002). The occurrence of BIFs suggests that ice sheets restricted supply of oxygen to the oceans, and the dissolved iron that was able to accumulate in oxygen-poor seas precipitated once the ice melted and oxygen was able to return to the seas (Hoffman & Schrag, 2002).

The process of mass glaciation and then rapid thaw occurred on different scales, but planet-wide at least two times based on Neoproterozoic diamictites on every paleocontinent (Pruss et al., 2010). Carbon, oxygen, sulfur, and manganese isotopes as well as trace elements can be analyzed in order to give clues as to the paleo conditions under which these sediments were deposited.

GEOLOGICAL SETTING

The studied cap carbonate samples, which are dolostone, were collected in Northwest Namibia (Fig. 2) on the southwestern margin of the Congo craton and was a carbonate platform at time of deposition (Hurtgen et al., 2006). The 19 samples span a total of a 30m section of the Otavi Group at about 1.5 m intervals. The Keilberg Member is a cap dolostone found everywhere on the Otavi platform and is the transgressive base of the cap sequence. It ranges from 10-25 m thick on the northern platform to 50-75 m thick in the south (Hurtgen et al., 2006). The Keilberg was deposited during post-glacial transgression and contains a few rare sedimentary structures that are common in Marinoan cap dolostones, such as microbial bioherms
and megaripple structures toward the top of the formation (Allen & Hoffman, 2005). These ripples suggest deposition at depths between 200 and 400 m under oscillatory flow which were possibly due to strong, persistent winds because of high atmospheric pressure gradients following glaciation (Allen & Hoffman, 2005). This group is Cryogenian in age, and paleomagnetic data suggests it occurred at about 12° paleolatitude at 743 ±30 Ma, and about 39°S at 547±4 Ma (Hurtgen et al., 2006), at the end of the Neoproterozoic.

METHODS

Dr. Matthew Hurten collected 19 samples from the Otavi Group in Namibia, seen in Figure 2. Each sample was cut in order to remove weathered surfaces and veins, and then pulverized in a shatterbox.

Carbon and Oxygen Isotopes

Both the carbon and oxygen isotope ratios are presented in per mil (‰) deviations relative to VPDB standard in delta notation (δ^{13}C, δ^{18}O). Once the samples were prepared as explained above, isotope deviations were determined using a scanning electron microscope for the 19 samples. One in ten samples were tested twice in order to calibrate the machine and ensure that the values it was measuring were consistent. Data are presented in Appendix A-1.

Sulfur Isotopes

During deposition of cap carbonates, some of the carbonate in dolostone is replaced with sulfate because of their similar size and charge. This is the type of sulfur that I am aiming to analyze. Sulfur isotope ratios are expressed as per mil (‰) deviations from the S isotope composition of Canon Diablo Triolite (CDT) using delta notation (δ^{34}S). Carbon associated
sulfur (CAS) analysis involves many steps of filtering and soaking the rock powders in liquids in order to dissolve out all carbonates and silica, leaving only sulfate and other trace elements. After rocks are cut and pulverized, the next step is to measure out about 80g of rock powder and put it in a beaker with approximately 300mL of sodium hypochlorite, or basically household bleach. This is done in order to dissolve out FeS because the sulfur in FeS and SO$_4^{2-}$ have different isotopic signatures, and we want those from the sulfate only. The solution is mixed for about 10 minutes using a mixing plate and magnetic stir bar then sits for a few days in order to dissolve all unwanted substances.

Next, the bleach is drained with a Buchner funnel and pump using 150 mm diameter Whatman brand filter paper. Samples are rinsed with deionized water, then mixed with about 650mL of 3N HCl (or enough to bring the reaction to completion so that the solution no longer aggressively fizzes when more HCl is added). This is mixed for one day using the same apparatus as when mixing the bleach and sediment. The purpose of this step is to dissolve all carbonates so that only the sulfate and other miscellaneous minerals like quartz remain.

After a day of mixing, the solution is run through a filter with a Buchner funnel. Once completely drained, the solution must be filtered again, this time using a finer fitted glass filter apparatus covered with 47 mm diameter and 0.45 µm thickness filter paper to remove practically all remaining sediment. Approximately 25 mL of BaCl$_2$ is added to the remaining substance in the 3 N HCl. The barium in the barium chloride reacts with powdered sulfates in the solution, which precipitates and forms barite that is filtered out one final time. About 2 to 10 mg of the BaSO$_4$ is mixed with V$_2$O$_5$ and combusted in an Elemental Analyzer at about 1000°C. Total S mass was determined after SO$_2$ was isolated cryogenically on a vacuum line, and this purified
Fig. 2: Geological map of outcrop, overlain on maps of Namibia and Africa for locational reference.

Geological map adapted from Pruss et al., 2010.
SO$_2$ was sealed in pyrex tubing and analyzed on a VG Prism Series II isotope ratio mass spectrometer. Data are presented in Appendix 1.

RESULTS

The studied outcrop was 30m with samples taken roughly every 1.5m. Isotope excursion data for $\delta^{13}$C and $\delta^{18}$O were taken from each of these 19 samples, but due to technical issues, data for $\delta^{34}$S were only measured for 12 samples and therefore not as consistently spaced. Data can be found in Appendix A1 and A2.

Carbon and Oxygen Isotope Excursions

The $\delta^{13}$C data (Appendix A2) in the lower 23 meters falls between $-2.52$ and $-2.97\%$ and form a fairly smooth profile, with the exception of sample G3001.1.5A at 1.5m from the base of the outcrop. Above 23m, $\delta^{13}$C compositions become fairly more negative up section with the lowest peak at 27.9m measuring at $-5.09\%$. The highest point at 30m then increases back to $-4.28\%$. The notable negative shift may be an indicator of a dolostone to limestone transition (Hurtgen et al., 2006). $\delta^{18}$O compositions (Appendix 2B) roughly mirror the pattern seen for that of $\delta^{13}$C, with fairly stable range between $-5.41$ to $-6.43\%$, an outlier at 1.5m with $-7.25\%$, and then another sharp decrease higher up section, especially at 27.9m, which reaches $-8.13\%$.

Sulfur Isotope Excursions

$\delta^{34}$S composition values (Appendix 2C) did not produce as stable of a curve as those of carbon and oxygen. The range for data is much wider, from 22.5 to 34.0\%, and the widest difference between neighboring samples is $12.6\%$ between 21.7m and 24.8m. However, extreme
fluctuations in data like this are not to be ignored because similar studies on other sections have found similar compositions (Hurtgen et al., 2006), perhaps due to secondary processes that will be explained later.

**DISCUSSION**

**Opposition to snowball and slushball Earth**

The theory that Earth was covered almost entirely in glaciers for millions of years did not seem practical to many scientists when the idea was first proposed by Joe Kirschvink and his student Dawn Sumner in 1992. For example, Williams and Tonkin (1985) argued that the Earth was just experiencing a large obliquity greater than °54, and this would cause average annual temperatures at the equator to be colder than those at the poles. However, there were still very hot periods at the equinoxes due to insolation that would cause rapid melting. Richard Shelton (1984) thought that large orbiting ice rings episodically collapsed into the atmosphere, transiently shielding sunlight and giving rise to glaciers at low latitudes. Furthermore, climate physicists in general believed that such a runaway feedback would be irreversible.

However, Kirschvink came up with three tests based on carbon isotopes, cap carbonates, and iron formations that would support his theory following on a few assumptions that must be true for snowball Earth to have been as large-scale as he believed. While going into detail of these tests is beyond the scope of this paper, their credible explanations helped scientists understand many rather puzzling pieces of Neoproterozoic Earth history that were previously unexplained.
Data Analysis

The previously stated data come from only one outcrop from the Otavi platform, so I will be comparing these values to those from a similar study by Hurtgen et al. (2006), and I am using his explanations of fluctuation in isotopic values to understand what was happening during the deposition of my cap dolostone. To begin, these cap carbonates have unusual geochemical, sedimentological, and isotopic characteristics not found in other Neoproterozoic and Phanerozoic carbonates, and they occur even in successions otherwise lacking carbonates (Hoffman & Schrag, 2002). We rely on Late Neoproterozoic glacial deposits (LNGD) to understand the mechanisms of snowball Earth because not only are they found on all continents, making them reliable and easy to compare, they have sharp contact with cap carbonates- which are more commonly dolostone than limestone- which suggests a quick transition from icehouse to greenhouse as the snowball theory states.

When it comes to isotopic excursion analysis of these units, it is commonly understood that $\text{SO}_4^{2-}$ can substitute for $\text{CO}_3^{2-}$ in the carbonate crystal lattice due to comparable size and charge. Therefore, carbonate-associated sulfur (CAS) is commonly used as a proxy for the $\delta^{34}\text{S}$ of ancient seawater sulfate. These levels give insight into oceanic conditions during deposition (Hurtgen et al., 2002). It is important to note that the large variations seen not only in my $\delta^{34}\text{S}$ data but those in other studies suggests that secondary processes like diagenesis can affect the sulfur-isotope composition of ancient carbonates. In fact, burial diagenesis can decrease CAS concentrations (Hurtgen et al., 2006).

Dolomite samples have a greater CAS concentration than limestone samples, which means that carbonates might incorporate pore-water sulfate during dolomitization (Hurtgen et al., 2006), a process that limestones do not undergo and therefore are not susceptible to that
additional sulfate. Based on this, I can predict that the ~9‰ decrease in $\delta^{34}$S in the top 12m of outcrop may correspond to a transition from dolostone to limestone carbonate. Such a lithologic shift is also observed by Hurtgen et al. in many locations where there is a sharp contact between a cap dolostone and an overlying limestone rhythmite (Fig 3.). The rather high 38.7‰ value at 24.8 m may be an outlier, but regardless of this large value, that 9‰ decrease corresponds to a lithologic change in a similar isotopic study by Hurtgen et al. (2006).

![Stratigraphic columns of the Maieberg Formation showing a paleographic cross-section from the northern Otavi platform to the Outjo basin in the south. Note the bottommost contact between cap dolostone and overlying limestone rhythmite. Adapted from Hurtgen et al., 2006.](image)

Hurtgen et al. (2006) predicts that since oceanic sulfate concentration would have probably been lower during a snowball event, the sulfur isotope that is measured in CAS is a result of several situational sources such as volcanically derived SO$_2$, sulfur species that were trapped in the ice and released during melting, and the weathering of sulfides and the release of trace sulfate during the dissolution of platform carbonates which were exposed during a glacial period.
Furthermore, the decrease by 10‰ in the lower majority and then increase at the top of the dolostone of the Keilberg Member that is present in all of Hurtgen et al.’s measured sections (2006) as well as in my section suggests that the cap dolostone precipitated from a water mass that had about the same sulfate composition throughout. The noted increase probably means that initial \([\text{SO}_4^{2-}]\) was low, but then an input of \(\text{34S}\)-enriched sulfate to or the removal of \(\text{34S}\)-depleted sulfur to the surface ocean (or some combination of the two) (Hurtgen et al., 2006). Additionally, the increase could be due to the removal of isotopically light sulfur through bacteria sulfate reduction (BSR) and sulfide burial. Another source was the mantle supplying sulfide through hydrothermal vents, which was exaggerated because the riverine influx of sulfate was cut off during glaciation (Hurtgen et al., 2006).

The lithology of the outcrop—whether it is dolostone or limestone—provides a clue as to the conditions under which the unit was deposited. In open ocean conditions that are closer to the main source of sulfate, for example, sulfate concentrations were high enough to prevent dolomite precipitation. However, in more restricted, shallower areas where sulfate conditions were very low (CAS<25 ppm), primary or early dolomite precipitation took place over the cap dolostone (Hurtgen et al., 2006).

Hurtgen suggests that a negative shift for \(\delta^{13}\text{C}\) and \(\delta^{18}\text{O}\) are consistent with an equilibrium isotope fractionation between dolostone and limestone because carbon and oxygen are isotopically heavier in dolomite. The carbon-isotopic difference could be preserved either if the dolomite was primary, or if it formed at the same time as deposition while still in equilibrium with seawater, assuming that the limestone was precipitated from seawater and that the dolostone could take on the same \(\delta^{13}\text{C}\) values as the calcite it came from if the dolomite was not primary (Hurtgen et al., 2006). They also suggest (2006) that if the decline in \(\delta^{34}\text{S}\) was the result of
oxidation of deep-water sulfide, then maybe the isotopically light carbon came from upwelling from the deep ocean as well by oxidation of organic carbon to bicarbonate via BSR.

However, while we see this trend for both sulfur and carbon, the opposite is true for oxygen— that is, open ocean settings are usually characterized by lighter $\delta^{18}O$. Granted, none of this would have been possible without ocean stratification between cold, dense, deep water in the open ocean and warm, oxic shallow water throughout glaciation (Hurtgen et al., 2006).

As stated before, $\delta^{13}C$ values range from about -2.5 to -5‰, $\delta^{18}O$ from -5.1 to -8.1‰, and $\delta^{34}S$ from 22.5 to 38.7‰. While these values are relatively insignificant and unremarkable by themselves, they correspond very closely with those from Measured Section 8 in the study by Hurtgen et al. (2006), and is located far to the east of the cross section in Fig. 3. Hurtgen et al. predicts that this particular section was deposited near the shelf edge, as was Measured Section 5. Similar values were also recorded in the equivalent Nuccaleena cap dolostone in South Australia (Hurtgen et al., 2005).

The decrease in $\delta^{34}S$ roughly corresponds to a decrease in $\delta^{13}C$. This pattern can also be seen in the data presented by two different studies by Hurtgen (2005 & 2006). This suggests that the transition from dolostone to limestone effects $\delta^{13}C$ concentrations as well as those for $\delta^{34}S$. Unfortunately, no clear pattern exists for $\delta^{18}O$ values like there is from Hurtgen et al. (2006). Nevertheless, because data aligns with at least some of the preexisting geologic data, I believe it can be used to strengthen any arguments for its implications in depositional location near a shelf edge or for what biology was present at that time.
CONCLUSIONS

The geochemical and isotopic excursion gradients present in this 30m outcrop from the Keilberg Member of the Otavi platform, when compared to other similar studies of not only the same location but also from South Australia, suggest that waters during deposition of this unit were stratified following glaciation. Dolostones, such as the one that I studied, most likely formed in shallower waters with low sulfate conditions and warm, oxic waters. On the other hand, where water was cold and euxinic, limestones dominated. The transition between these rock types is always sharp, suggesting a fairly rapid change in conditions including but not limited to temperature and ion concentrations.

The predicted contact in the studied outcrop is indicated by a decrease in both $\delta^{34}$S and $\delta^{13}$C concentrations. The former can be explained by the location and depth that the carbonate was deposited in- low $\delta^{34}$S\textsubscript{sulfate} values are indicative of open ocean settings closer to the shelf margin, whereas high values that existed in restricted settings reflect the ongoing BSR in shallow, isolated waters with low sulfate concentrations. The later suggests a transition from dolostone to limestone because dolomite favors isotopically heavy carbon and oxygen. So, as the section approaches the transition from the dolostone of the Keilberg Member to the overlying limestone rhythmites, the decline in $\delta^{13}$C is an indicator both an equilibrium isotope effect between coeval dolomite and calcite as well as the input of $^{13}$C- depleted deep-water dissolved inorganic carbon to the surface from the deep ocean at the shelf margin.

Based on the findings from Hurtgen et al. (2006), I believe that this outcrop was deposited on a shelf rim where it was susceptible to upwelling $^{13}$C and $^{34}$S\textsubscript{sulfate} that inland surface waters would not have access to, while deep ocean waters would not be conducive to the precipitation of dolomite for reasons previously described. The sharp contact between dolostone
and limestone suggests a quick turnover from icehouse to greenhouse and therefore a change in ocean conditions. However, more data from subsequent glacial deposits and cap carbonates are necessary to determine more general global conditions such as sulfur isotopic composition before and after the Cryogenian. Nevertheless, it can be concluded that the Earth was very susceptible to change during this period, and marine sulfur, oxygen, and carbon were very responsive to these changes as seen through isotopic excursions in cap carbonates from the late Neoproterozoic around the globe.

ACKNOWLEDGEMENTS

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The data from Hurtgen et al., 2006 can be found online at doi:10.1016/j.epsl.2006.03.026.

REFERENCES CITED


APPENDIX A. Geochemical Isotopic Data

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1. Values of $\delta^{18}$O_{carbonate}, $\delta^{13}$C_{carbonate}, and $\delta^{34}$S_{sulfate} in standard delta notation represented in per mille notation.
2A. Geochemical data for $\delta^{13}$C in per mille notation.
$\delta^{18}O$ for G3001.00 to G3001.30.0

2B. Geochemical data for $\delta^{18}O$ in per mille notation.
2C. Geochemical data for δ³⁴S in per mille notation.