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**KECK GEOLOGY CONSORTIUM
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2014-2015 PROJECTS

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Students: BRIANNA BERG, University of Montana, AMAR MUKUNDA, Amherst College, REBECCA BLAND, Mt. Holyoke College, JACOB HUGHES, Western Kentucky University, LUIS RODRIGUEZ, Universidad de Puerto Rico-Mayaguez, MARIAH ARMENTA, University of Arizona, CLEMENTINE HAMELIN, Smith College

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CLÉMENTINE HAMELIN, Smith College
Research Advisor: John B. Brady

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CALCITE-GRAPHITE STABLE ISOTOPE THERMOMETRY IN MARBLES OF THE RUBY RANGE, SW MONTANA

REBECCA BLAND, Mount Holyoke College

Research Advisor: Steven R. Dunn

INTRODUCTION

Today, the Ruby Range of southwest Montana provides exposure to rocks that are the result of a 1.8-1.7 Ga Proterozoic orogeny, the Big Sky orogeny (Brady et al. 2004). The Precambrian lithologies of this range include amphibolite, pelite, marble, banded iron formation, and other metaigneous and metasedimentary rocks. The rocks experienced metamorphic events both at 2.45 Ga and 1.8 Ga (Harms et al., 2015). This study focuses on marbles from the western portion of the Ruby Range in the Christensen Ranch and Dillon groups (James, 1990) in order to understand the history of this belt of rocks and the temperatures of their metamorphism

and deformation. This study applies the calcite-graphite carbon isotope thermometer in order to determine metamorphic temperatures and uses mineral assemblages to constrain metamorphic fluid composition in selected samples.

UNIT DESCRIPTION

The marble of the metasedimentary Christensen Ranch suite of the Ruby Range exists in layers up to half a kilometer in thickness, with outcrop exposures <10 m wide (James, 1990). Individual outcrop are often bounded by pelite in the Christensen Ranch group, and a mylonitic garnet leucogneiss, or characteristic quartzofeldspathic gneiss in the Dillon group (Fig. 1). The marbles from these outcrops are fine to medium grained and sugary in texture. Coarser grained marbles are generally more pure calcite, with grains ~1 cm in diameter. These coarser grained marbles are also very weathered and crumbly with a brown crust. Marbles appear both with layering and more massive and granular (Fig. 2). Where layering is present, it distinguishes as bands richer in silicates or graphite.

Across the Christensen Ranch group, marbles are less commonly a pure calcite marble, but range to a mix of calcite and dolomite, to pure dolomitic marble. In addition to calcite and dolomite, diopside is common, with grains subhedral, ranging from 1-3 mm in size and composing up to ~15% of the rock. Graphite in these rocks is distinct, although not common. Flakes are generally up to 1 mm in diameter, and where present compose 1-3% of the rock. Other common minerals include K-feldspar, hornblende and biotite with local plagioclase, quartz, clinozoisite (or epidote), scapolite and garnet (no sample contains all of these

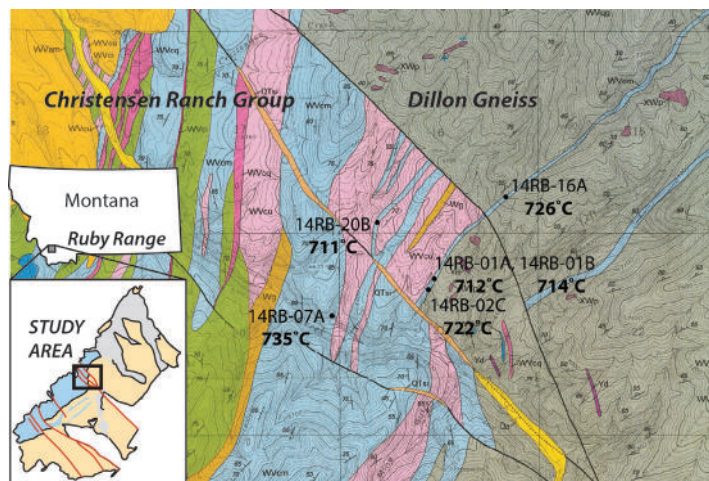


Figure 1. The location of the Ruby Range in southwest Montana with an inset showing the study area within the Ruby Range. Sample locations and the calculated temperatures from those locations are shown within the Christensen Ranch Group and the Dillon Gneiss. Areas in light blue are mapped marble units, light pink is an undifferentiated unit, and the green to grey color represents characteristic Dillon quartzofeldspathic gneiss. Figures modified from Harms et al. (2015, this volume) and James (1990).

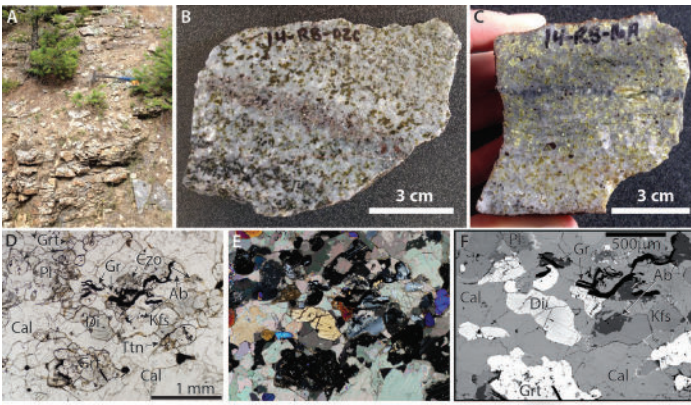


Figure 2. A. Typical exposure of marble unit. B and C: Representative textures of marbles from within the Christensen Ranch Group. 14RB-02C is shown in B depicting the layered fabric that occurs in some samples. The bands in 14RB-02C are formed with greater graphite density. C shows sample 14RB-16A with a more granular texture made visible by the abundance of serpentine throughout the sample. D and E: Photomicrograph of sample 14RB-07A in plane light and crossed polars, respectively. F: Backscattered image of same area as D and E.

phases). Accessory titanite and sulfides are common. Evidence for hydrothermal alteration exists in the form of talc and serpentine, talc appears in marbles located near talc mines in the field area, mostly in small blebs, ~5 mm in diameter, and is a greenish white color. Several marble exposures contain rounded grains of serpentine, presumably pseudomorphs after

olivine (Fig. 2c). Complete mineralogy is presented in Table 1.

CALCITE-GRAPHITE THERMOMETRY

The temperature dependent fractionation of carbon isotopes in calcite and graphite provides a reliable isotopic geothermometer (referred to as cal-gr). Because of the extraordinarily limited diffusion rates of carbon in graphite, this geothermometer is particularly good at retaining peak metamorphic temperatures (Dunn and Valley, 1992). Resetting of the isotopic fractionations most likely requires complete recrystallization of the graphite (Dunn and Valley, 1992). Calcite will exchange carbon much more readily at metamorphic temperatures, however, generally there are no additional carbon-bearing minerals for the calcite to exchange with, so the cal-gr system is relatively free of retrograde modification. The refractory nature of graphite makes experimental study of graphite and calcite carbon exchange difficult, nevertheless two empirically-calibrated versions of the cal-gr geothermometer have been widely applied: Dunn and Valley (1992) and Kitchen and Valley (1995). Dunn (2005) shows that Dunn and Valley (1992) is very accurate for the temperature range of 525°C-650°C, but above 650°C the Kitchen and Valley

Sample	Unit	Rock Type	Cal	Gr	Dol	Di	Am	Kfs	Pl	Ep	Qtz	Ttn	Bt	Srp	Tlc	Phl	Scp	Ap	Chl	Grt
14RB-01A	CR	M	80	x		x	x	x	x	x	x	x							x	
14RB-01B	CR	M	70	x	x				x				x	s						
14RB-02B	CR	M		x	80	x														
14RB-02C	CR	M	70	x	x									s						
14RB-02D	CR	M	x	x		x								s						
14RB-04	CR	CS	x	x		50	x	x	x						x					x
14RB-05	CR	CS	x				x	x	x		x					x				
14RB-07A	CR	CS	x	x		x	x	x	x	x		x	x				x	x	x	x
14RB-07B	CR	CS	x	x		x	x	x				x					x			
14RB-07C	CR	CS	x	x		x	x	x				x			x		x			x
14RB-12A	DG	CS	x	x		x														
14RB-12D	DG	CS				x	x													
14RB-14C	DG	M	x		x									s						
14RB-16A	DG	M	x	x	x									s						
14RB-20A	CR	M	93	x			x		x		x									
14RB-20B	CR	M	65	x					x		x									x
14RB-21B	DG	M	95	x		x														
14RB-24A	CR	CS	x	x		x	x	x	x	x	x	x						x		
14RB-26	CR	M	50				x	x		x			x		x					x

Table 1. Mineralogy of marble (M) and calc-silicate (CS) samples from the Christensen Ranch group (CR) and Dillon gneiss (DG). Cal = calcite; Gr = graphite; Dol = dolomite; Di = diopside; Am = amphibole; Kfs = potassium feldspar; Pl = plagioclase feldspar; Ep = epidote; Qtz = quartz; Ttn = titanite; Bt = biotite; Srp = serpentine (usually secondary); Tlc = talc; Phl = phlogopite; Scp = scapolite; Ap = apatite; Chl = chlorite; Grt = garnet.

(1995) calibration is preferable and was the one used throughout this study.

METHODS

In the field, sampling occurred across the Christensen Ranch group as well as more limitedly within the Dillon. Attempts were made to collect samples from all stratigraphic levels within the Christensen Ranch, specifically along the contact with the Dillon gneiss. While sampling, care was taken to avoid dolomite. Samples are of both calcite marble with graphite inclusions that are otherwise as pure as possible, and calc-silicate rocks that include graphite.

Graphite for stable isotope analysis was gathered largely using a flotation technique, wherein $\sim 3 \text{ cm}^3$ of sample was crushed and powdered, and stirred in a beaker of water. The floating minerals were scooped out, rinsed with HCl and purified water, and dried. When this method failed to produce sufficient graphite, $\sim 2 \text{ cm}^3$ of sample was dissolved in 200 ml strong HCl, rinsed, and dried. From this dissolution residue, flakes of graphite were then hand picked from the resulting rock sand. The collected graphite was then combined with excess CuO, placed in a pre-fired quartz tube, evacuated, sealed, and heated overnight at 950°C . CO_2 was collected from these samples in 6 mm pyrex tubes after using standard cryogenic techniques to remove any H_2O or SO_2 (Dunn, 2005) and these were taken to the University of Massachusetts – Amherst for mass spectrometer analysis. Additionally, a small ($\sim 2 \text{ cm}^3$) piece of each of the analyzed samples was dissolved separately in strong HCl in order to assess whether the graphite flakes in each sample were armored by silica, given that silica armoring results in isotopically lighter grains of graphite (Wada, 1988; Dunn and Valley, 1992).

Stable isotope analysis of carbonate was performed on crushed and ground whole-rock powder of the same samples as the graphite separates. Calcite powder was analyzed with an automated carbonate preparation system connected to a Finnegan Delta XL mass spectrometer at the University of Massachusetts – Amherst. The results of these carbon and oxygen analyses are presented in Table 2 using standard delta notation, and are presented compared to the Vienna Standard Mean Ocean Water (VSMOW) and Vienna Pee Dee Belemnite (VPDB) standards for oxygen

and carbon, respectively. The precision for these analyses is 0.1‰ for both calcite and graphite based on repeated measurements of standards as well as graphite duplicates.

Calcite-graphite temperatures were obtained following Kitchen and Valley (1995): $\Delta^{13}\text{C}_{\text{cal-gr}} = 3.56 \times 10^6 \times \text{T}^{-2}(\text{K})$

Petrographic analysis was conducted on standard thin sections cut from 23 samples across the Christensen Ranch and Dillon Gneiss suite. These thin sections were stained with alizarin for ease in distinguishing between calcite and dolomite. Six of these samples, 14RB-01A, 14RB-02C, 14RB-05, 14RB-07A, 14RB-07C, and 14RB-24A were polished and carbon coated for EDS analysis, which was carried out at Mount Holyoke College using an FEI Quanta 50 scanning electron microscope to confirm mineral assemblages and textures.

Because samples 14RB-02C and 14RB-16A contain sufficient amounts of dolomite it is necessary to correct their $\delta^{13}\text{C}$ values so as to more accurately represent the calcite. The fractionation of carbon isotopes between calcite and dolomite is 0.4‰ in the range of 600°C – 700°C , with dolomite having a heavier isotopic weight than calcite (Friedman and O’Neil, 1977). At most a correction of 0.15 is thus needed for visual estimates of 20% dolomite and 55% calcite in sample 14RB-16A. A correction of 0.04 is used for visual estimates of 8% dolomite and 70% calcite in sample 14RB-02C. These corrections raised the temperature by 20°C and 5°C respectively (Friedman and O’Neil, 1977).

Sample #	$\delta^{13}\text{C}_{\text{gr}}$	$\delta^{13}\text{C}_{\text{cal}}$	$\delta^{18}\text{O-SMOW}$	$\text{D}_{\text{cal-gr}}$	Temp ($^\circ\text{C}$)
14RB-01A	-5.86	-2.18	13.75	3.67	712
	-5.84				
14RB-01B	-5.81	-2.13	16.12	3.65	714
	-5.75				
14RB-02C	-6.76	-3.17	15.76	3.59	722
	-6.85				
14RB-07A	-9.27	-5.82	14.27	3.50	735
	-9.39				
14RB-16A	-5.09	-1.27	14.15	3.57	726
	-4.89				
14RB-20B	-5.32	-1.65	11.75	3.67	711

Table 2. Data and resulting temperatures calculated using the Kitchen and Valley (1995) calibrations. Stable isotope compositions in ‰ of calcite and graphite. $\delta^{13}\text{C}$ values are highly reproducible, and duplicates of each sample were run except for 14RB-20B given a lack of sufficient graphite. Temperatures range from 711°C to 735°C .

RESULTS

Results from cal-gr thermometry are presented in Table 2. Calculated temperatures range from 711°C to 735°C, with an average temperature of 720°C. Carbon values are generally within the normal range for marine limestone (-2 to +4‰). Oxygen values are predominantly lighter than those of typical marine limestones (22 to 32‰) (Keith and Weber, 1964; Hoefs, 2009).

Uncertainty in the calculated temperature can result from both analytical uncertainty and from possible error in the calibration. Assessing any possible error in the calibration is beyond this study because I have no independent quantitative thermometry. Analytical uncertainty in the $\delta^{13}\text{C}$ values is about $\pm 0.1\text{‰}$ in both calcite and graphite. A 0.2‰ uncertainty in the $\Delta\text{cal-gr}$ value [$\delta^{13}\text{C}_{(\text{cal})} - \delta^{13}\text{C}_{(\text{gr})}$] would result in temperature uncertainty of 53°C, which would be a minimum uncertainty.

DISCUSSION

The carbon isotope values are more-or-less within a normal range except for sample 14RB-07A, which is quite light. With respect to oxygen, all are lighter than normal marine limestone. These lower isotopic compositions could be partly attributed to the loss of isotopically heavier CO_2 during metamorphic reactions, but exchange with isotopically light, externally-derived fluids is probably necessary to explain the marble $\delta^{18}\text{O}$ values (Bowman et al., 1994).

Mineral assemblages in some of the samples allow the fluid composition to be constrained. This is done with the use of T-X(CO_2) diagrams. The endmember positions of selected mineral reactions were determined for the chemical system $\text{CaO-MgO-Al}_2\text{O}_3\text{-SiO}_2\text{-CO}_2\text{-H}_2\text{O}$. A T-X(CO_2) diagram was created using the PTX program (GEØ-CALC) and database of Berman (1988) (Fig. 3). In order to accurately calculate the locations of mineral reactions within T-X space, one must specify the pressure of the T-X diagram. Clémentine Hamelin (2015, this volume) estimates the metamorphic pressure to be about 8 kbar (800 MPa) based on mineral assemblages within the KFMASH system for pelites within the Christensen Ranch group. Thus this study used 8 kbar for the T-X diagram.

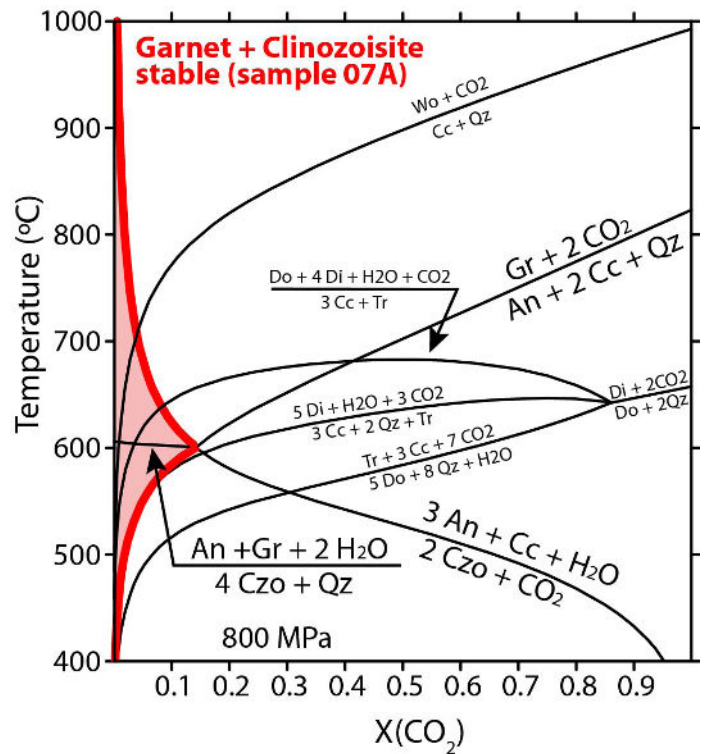


Figure 3. T-X(CO_2) diagram for selected reactions given mineral assemblages in sample 14RB-07A adjusted for solid solution as follows: $\alpha_{(\text{Gr})} = 0.09$, $\alpha_{(\text{An})} = 0.75$, $\alpha_{(\text{Tr})} = 0.06$, $\alpha_{(\text{Czo})} = 0.64$ and $\alpha_{(\text{Di})} = 0.50$. The presence of garnet, anorthite, clinozoisite, and calcite constrain reactions to the portion of the diagram highlighted in red. Cal-gr geothermometry indicates peak temperatures at $\sim 720^\circ\text{C}$, requiring very H_2O -rich fluid conditions.

Solid solutions in some of the minerals leads to changes in the mineral reaction positions in T-X space. To account for this, mineral compositions were determined on the SEM (Fig. 3). The “activity” of the endmember in the solid solutions was calculated assuming ideal solutions and mixing on cation sites. GEØ-CALC allows mineral activities to be used for the placement of reactions. Sample 14RB-07A, having the most restrictive mineral assemblage therefore provides the most well-constrained estimate of T-X(CO_2). The presence of anorthite, clinozoisite, garnet, and calcite in 14RB-07A allow us to constrain the fluid composition on the diagram. Incorporating the temperature range of $720 \pm 10^\circ\text{C}$, we find that over the course of metamorphism, these rocks came into interaction with fluids that were highly water rich, with $\leq 5\%$ CO_2 . The presence of garnet and epidote/clinozoisite in various samples from across the field area support this assertion and provide evidence for water rich fluid infiltration throughout the marbles of the Christensen Ranch suite.

Because the mineral reactions assumed to have taken place produce CO₂-rich fluid, the only way to explain the T-X(CO₂) relations of sample 14RB-07A, and other similar assemblages in the marble units is infiltration of an H₂O-rich fluid. This would also potentially explain the δ¹⁸O values that are lighter than normal limestones. Pelitic rocks are likely to expel H₂O-rich fluid during metamorphism as minerals such as chlorite and muscovite break down. The δ¹⁸O values of such fluid is expected to be much lighter than that of marble and could very well be responsible for lowering the δ¹⁸O values of the marble (Dunn, personal communication).

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