### Datashare 71

# Microanalysis of carbonate cement $\delta^{18}$ O in a CO<sub>2</sub>-storage system seal: Insights into the diagenetic history of the Eau Claire Formation (Upper Cambrian), Illinois Basin

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### **APPENDIX 1: ANALYTICAL METHODS**

### **Sample Preparation**

Samples for in situ  $\delta^{18}$ O analysis were cast into 25-mmdiameter epoxy mounts (Buehler EpoxyCure), taking care to position areas of interest within a 5-mm radius of the geometric center; this was done to minimize any potential fractionation effects associated with a sample's position relative to the focusing axis within the analysis chamber (Kita et al., 2009; Valley and Kita, 2009). Several grains of a running standard (end-member dolomite UW6220,  $\delta^{18}$ O = 22.60% Vienna standard mean ocean water [VSMOW], -8.1% Vienna Peedee belemnite [VPDB]: Śliwiński et al., 2015) were embedded in the center of each mount, which was then polished to a 0.25-µm finish using oil-based polycrystalline diamond suspensions (Buehler MetaDi Supreme) and Allied TECH-Cloth pads. Care was taken to keep polishing relief between mineral phases of contrasting hardness (e.g., carbonate cements in quartz-rich sandstones) to less than a few micrometers. Prior to analysis, sample mounts were cleaned with deionized water and ethanol and coated with carbon (25-nm thickness) to make sample surfaces electrically conductive.

### Oxygen Isotope Analyses by Secondary Ion Mass Spectrometry

In situ  $\delta^{18}O$  measurements were performed using a CAMECA IMS 1280 large-radius multicollector ion microprobe at the Wisconsin Secondary Ion Mass Spectrometer (WiscSIMS) Laboratory (Department of Geoscience, University of Wisconsin–Madison). The sample data presented here were collected during four analytical sessions throughout the course

of 1 yr: two 10-µm-diameter spot-size sessions (session S1, September 23–25, 2013, and session S7, May 13–16, 2014) and two 3-µm spot-size sessions (session S4, February 24–27, 2014, and session S8, May 22–25, 2014). The full data set is provided in Appendices 4 and 5. The analytical conditions employed were the same as those described in Śliwiński et al. (2015).

The analytical accuracy of oxygen-isotope ratio measurements by secondary ion mass spectrometry (SIMS) is affected by instrumental mass fractionation (referred to here as "SIMS  $\delta^{18}$ O bias"; Hervig et al., 1992; Kita et al., 2009; Valley and Kita, 2009), a component of which is systematically related to the chemical composition and crystal structure of a sample. A calibration scheme for correcting SIMS  $\delta^{18}$ O bias for carbonate mineral compositions along the dolomite-ankerite solid solution series was reported by Śliwiński et al. (2015) and employed here for reducing sample data. The calibration relates the magnitude of SIMS  $\delta^{18}$ O bias to the Fe number [=Fe/(Mg + Fe) molar ratio] of dolomite-ankerite using the three-parameter Hill equation (e.g., review of Goutelle et al., 2008), which elegantly describes empirical relationships of the "component concentration" versus "measured effect" type in systems that behave nonlinearly and reach saturation; it is characterized by two curve-shape parameters (n and k) and an analytical session-specific calibration scaling factor (bias\*<sub>max</sub>). Although it is common in SIMS analyses for the magnitude of instrumental  $\delta^{18}$ O bias to vary from session to session, the overall distribution of calibration standard data points in relation to one another remains remarkably consistent, with the values of both Hill shape parameters (n and k) remaining invariant for the IMS 1280 instrument and tuning protocols at the WiscSIMS lab. Thus, it is best in every analysis session to analyze a series of standards between dolomite and ankerite to construct a working curve, but even if only two standards from the dolomite-ankerite solid solution series are measured (end-member dolomite in addition to one ankerite from the opposite end of the series), a calibration curve can still be established, albeit with a slight increase in the analytical uncertainty (by an additional ~0.3‰). The analytical precision is typically  $\pm 0.3\%$  (two standard deviations [2SD]) for 10-µm-diameter sample spot analyses and  $\pm 0.7\%$  (2SD) for 3-µm spots, based on the spotto-spot reproducibility (number of analysis [n] = 8) of a running standard (end-member dolomite, UW6220) that "brackets" each set of 10 sample analyses. The accuracy of sample analyses is, in part, determined by the calibration residual, which is a measure of how well the SIMS  $\delta^{18}$ O bias correction scheme reproduces standard data in relation to the certified reference material (RM) NBS-19; for 10-µm spot-size sessions, the residual is constrained to 0.3%for a suite of 13 dolomite-ankerite standards, whereas it is within 0.4‰ when performing analyses using a 3-μm spot (Śliwiński et al., 2015).

Measured isotope ratios are reported using the conventional  $\delta$  notation expressed in per mil relative to the VSMOW and VPDB  $\delta^{18}O$  scales.

## Chemical Analysis (by Electron Probe Microanalysis)

To correct each in situ carbonate  $\delta^{18}$ O analysis for SIMS instrumental bias, it is necessary to have knowledge of the cation composition of the sample material sputtered from each sample pit. Chemical analyses in the immediate vicinity of each SIMS pit (detailed below) were performed by electron probe microanalysis (EPMA) using a CAMECA SX-51 at the Cameron Electron Microprobe Laboratory (Department of Geoscience, University of Wisconsin–Madison).

The electron microprobe was operated at 15 keV and 10 nA; care was taken to minimize beam damage to carbonate samples by performing analyses with the electron beam preferentially defocused to a 5- $\mu$ m diameter where possible. However, the narrow thickness of certain compositional zones at times necessitated the use of a 2- $\mu$ m-diameter beam. Monte Carlo simulations using Casino software (v2.41; Drouin et al., 2007) indicate that under these

conditions, a 2-µm-diameter beam produces an interaction volume in dolomite and ankerite with a surface footprint approximately 3-4 µm in diameter. Thus, to avoid any potential edge effects, EPMA analyses were performed at least 5 µm away from 3-µm SIMS pits (using a 2-µm electron beam) and approximately 10 µm away from 10-µm SIMS pits (using a 5-µm electron beam). Characteristic x-ray intensities were measured for 10 s on each spectral peak and on two background positions (one on either side of each peak). During the course of a point analysis, the intensities of characteristic x-rays fluorescing from electron beam-sensitive materials can drift; this is corrected by a feature in Probe for EPMA software (Donovan et al., 2007) called TDI (timedependent intensity), where data plotted in "measured x-ray intensity" versus "time" space are first detrended before the application of ZAF corrections (i.e., corrections for sample matrix effects, based on mean atomic number [Z], x-ray absorption [A], and x-ray fluorescence [F]).

The electron microprobe was standardized with the following RMs: Delight Dolomite (RM for Ca, analyzed using a pentaerythritol [PET] crystal, and RM for Mg, using a thallium acid phthalate [TAP] crystal), Callender Calcite (alternate RM for Ca), USNM 460 Siderite (RM for Fe, using a lithium fluoride [LiF] crystal), rhodochrosite (RM for Mn, using a LiF crystal), and strontianite (RM for Sr, using a TAP crystal). Data were collected during five analytical sessions throughout the course of a year (Fall 2013-Summer 2014). Based on replicate measurements (n = 5-10) of the above RMs during each session, the elemental concentrations determined for each of the cations are precise to within 2.5% relative (relative standard deviation), whereas deviations from accepted values are constrained to within 5% relative.

### Sample Imaging by Scanning Electron Microscope

Extensive characterization of carbonate cements in sandstone samples by backscattered electron (BSE) and cathodoluminescence (CL) imaging (using a Hitachi S3400-N scanning electron microscope [SEM]) was employed to identify areas of interest prior to in situ  $\delta^{18}$ O analyses by SIMS and then again after SIMS analysis to verify that sample pits were

indeed placed where intended. High-resolution secondary electron images were acquired prior to SIMS analysis to identify characteristic features on sample surfaces (e.g., cracks, cavities, etc.) to aid in navigating and to allow for triangulating target positions in the reflected light optics of the SIMS sample viewer; secondary electron imaging was used again after analysis to assess the overall quality of each sputtered SIMS pit by revealing its surface texture. Data points were flagged and excluded if pits were found to partially overlap epoxy or showed any of the following features: cracks, epoxy, cavities (larger than  $\sim 1-2 \,\mu m$ ), inclusions of other minerals, or any "shelves" or "ledges" that detract from an otherwise regular appearance of the pit sputtered by the ion beam. Cathodoluminescence imaging was employed to identify and to image the earliest formed, commonly luminescent generations of carbonate cement. Following the method of Reed and Milliken (2003), a broadband, short-wavelength (ultraviolet-blue range) filter was employed to avoid streaking in SEM-CL images that results from carbonate phosphorescence.

### Bulk Mineralogy and Clay Speciation Analyses by X-Ray Diffraction

Five Eau Claire Formation shale samples from different burial depths across the Illinois Basin were submitted to ActLabs (Ancaster, Ontario, Canada) for clay mineral analysis by x-ray diffraction. A part of each powdered sample was mixed with 10 wt. % corundum powder and loaded into a standard back-fill holder. Corundum was used as an internal standard to determine the x-ray amorphous content of the samples. The abundances of the crystalline mineral phases were determined using the Rietveld refinement method, which is based on reproducing an observed diffraction pattern through modeling with crystallographic parameters. A part of each sample was dispersed in distilled water and clay minerals in the less than 2-µm fraction separated by gravity settling of particles in suspension. Oriented slides of the less than 2-µm fraction were prepared by placing a part of the suspension onto a glass slide. To identify expandable clay minerals, the oriented slides were analyzed after (1) air drying, (2) saturation with ethylene glycol, and (3) subsequent heating at 375°C (~710°F) for 1 hr. The semiquantitative amounts of the different clay minerals in the less than  $2-\mu m$  fraction were determined based on calculations of basal-peak areas.

The x-ray diffraction analysis was performed on a Panalytical X'Pert Pro diffractometer equipped with a Cu x-ray source and an X'Celerator detector. The operating conditions were as follows: 40 kV and 40 mA, range 4°–70° 2 $\theta$  for random powder mounts and 3°–35° 2 $\theta$  for oriented clay mineral preparations, step size 0.017° 2 $\theta$ , time per step 50.165 s, fixed divergence slit (1/4°), and sample rotation 1 rev/s.

The abundances of illite and smectite in clay mineral separates can be assessed using Środoń's (1984) intensity ratio (IR). This parameter compares x-ray IRs of the illite 001 and 003 diffraction peaks measured in oriented clay mounts before and after glycolation and is very sensitive to the presence of swelling (smectite) layers (IR =  $[001/003]_{air}$  dried/ $[001/003]_{glycolated}$ ). Swelling has occurred if IR > 1.0; values less than approximately 1.5 are generally consistent with less than 15% smectite (Środoń, 1984).

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### APPENDIX 2: ABBREVIATED CORE DESCRIPTIONS

### Eau Claire Formation Interval, Illinois Basin

The purpose of the following is to provide a lithostratigraphic context to the samples that were collected and analyzed for this study. Sandstone beds within the predominantly silty–shaly Eau Claire Formation were sampled at three cored localities (refer to Figure 1 and Table 1 in main text) representing different sediment burial depths: (1) on the Wisconsin arch (sample SS-1, maximum burial depth <0.5 km (~1500 ft); see discussion in the Significance of Paleodepth-Related Differences in Carbonate Cement  $\Delta^{18}$ O [Early–Late] section), (2) on the basin margin in northern Illinois (SS-2, maximum burial depth ~1 km [3500 ft]), and (3) in the central Illinois Basin (SS-3, maximum burial depth ~2 km [6500 ft]).

Historical coring to the basal Mt. Simon Sandstone of the Illinois Basin often did not aim for a recovery of the overlying Eau Claire Formation interval (R. Mumm, 2013, personal communication). Thus, only a relatively small number of cores exist with the Eau Claire Formation intact or present altogether, hindering a thorough characterization of the stratigraphy and limiting the wherewithal to laterally correlate specific lithofacies across the basin (Neufelder et al., 2012).

Legacy porosity and permeability data are provided where available.

## Core C131467 (Dane County, Wisconsin; API Number: Not Applicable)

Depth of sandstone bed sampled for this study: 277.9 ft (84.7 m) (sample SS-1).

Aswasereelert et al. (2008) divided the Eau Claire Formation of west to southcentral Wisconsin into five distinct lithofacies (A–E; see table 1 therein) that represent different paleowater depths and depositional environments (outer shelf to shore face) of an epeiric shelf. Only the finer-grained, outer shelf facies (A and B) are present at this cored locality; an overview of the lithostratigraphy is shown in Figure A.1 (see figure 13 in Aswasereelert et al., 2008 for a more comprehensive description).

- Lithofacies A: This facies consists of mudstone and siltstone with minor, very thin-bedded (0.1–1 cm [~0.0625–0.375 in.]) sandstone beds. This designation was given to cored intervals comprised of at least one 1-ft (~30-cm) interval of very thin-(0.1–1 cm [~0.0625–0.375 in.]) to thin-bedded (1–10 cm) mudstone and/or siltstone.
- Lithofacies B: This facies consists of thin-bedded sandstone beds (1–10 cm [~0.375–4 in.], generally <5 cm [~2 in.]) with very thin siltstone and mudstone interbeds (0.1–1 cm [~0.0625–0.375 in.]).
- In southern Wisconsin, the Eau Claire Formation is an important regional aquitard (Aswasereelert, 2005; Aswasereelert et al., 2008).

### Core C12996 (UPH-3) (Stephenson County, Illinois; API Number: 121772131700)

Depth of sandstone bed sampled for this study: 1217.3 ft (371 m) (sample SS-2).

The following lithostratigraphic overview pertains to core UPH-1 (API number: 121772131500), drilled approximately 4.5 mi (~7 km) north of the core sampled for this study (C12996/UPH-3). The depth of the sandstone bed sampled in core C12996/UPH-3 (1217.3 ft [371 m]) was correlated to within several feet with the depth of the equivalent bed in core UPH-1 (position indicated in Table A.1 at 1101 ft [335.6 m]) based on the following reference horizons: (1) the depth of the contact between the Mt. Simon and the Eau Claire Formations (UPH-1: 1165 ft [355.1 m]; UPH-3: 1308 ft [398.7 m]) and (2) the depth of unique carbonate beds (UPH-1: ~1046.7–1047 (319–319.1 m) and 1050.5–1051.5 ft [320.2–320.5 m]; UPH-3: ~1191.6 ft [363.2 m]).

# Core C4006 (Champaign County, Illinois; API Number: 120190012800)

Depth of sandstone bed sampled for this study: 3857 ft (1175.6 m) (sample SS-3).

The sources of the following information are core descriptions from a well folder, provided by the Geological Records Unit of the Illinois State Geological Survey.

The Eau Claire Formation interval (3305–3993 ft [1007–1217.1 m]) was cored from 3751–3992 ft (1143.3–1216.8 m). The core is no longer complete; it has been reduced to "chips" that represent approximately 15% of the original core material. Table A.2 below provides a summarized overview of the lithologies present and the range of porosity and permeability values for the different depth intervals (measurements performed by Core Laboratories, Inc., Oklahoma City, Oklahoma, 1959). The position of the sandstone bed sampled for this study (sample SS-3, 3857 ft [1175.6 m]) is indicated.

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### Table A.1. Core UPH-1

Depth (ft [m])	Interval Thickness	Lithology
846-851 (257.9-259.4)	5 ft (1.5 m)	Sandstone with shale streaks
851-864 (259.4-263.3)	13 ft (4 m)	Shale
864-926 (263.3-282.2)	62 ft (18.9 m)	Interbedded siltstone-shale (centimeter scale)
926–927 (282.2–282.5)	1 ft (0.3 m)	Sandstone, heavily iron stained (thoroughly maroon)
927–930 (282.5–283.5)	3 ft (0.9 m)	Interbedded siltstone-shale (centimeter scale)
930–933 (283.5–284.4)	3 ft (0.9 m)	Sandstone, heavily iron stained (thoroughly maroon)
933–974 (284.4–296.9)	41 ft (12.5 m)	Clean sandstone
974–986 (296.9–300.5)	12 ft (3.7 m)	Shale and very shaly siltstone
986–993 (300.5–302.7)	7 ft (2.1 m)	Clean sandstone
993–1035 (302.7–315.5)	42 ft (12.8 m)	Sandstone with siltstone interbeds (centimeter–decimeter scale) and shale streaks
1035–1040 (315.5–317)	5 ft (1.5 m)	Clean sandstone
1040–1041 (317–317.3)	1 ft (0.3 m)	Carbonate (dolomite) bed
1041–1046 (317.3–318.8)	5 ft (1.5 m)	Sandstone, heavily dolomitized? (not clean in appearance)
1046.7–1047 (319.0–319.1)	0.3 ft (0.1 m)	Carbonate (dolomite) bed
1047–1048.5 (319.1–319.6)	1.5 ft (0.5 m)	Sandstone, heavily dolomitized? (not clean in appearance)
1048.5–1049.5 (319.6–319.9)	1 ft (0.3 m)	Carbonate (dolomite) bed; several inches of siltstone and/or shale near bottom
1049.5–1050.5 (319.9–320.2)	1 ft (0.3 m)	Sandstone, heavily dolomitized? (not clean in appearance)
1050.5-1051.5 (320.2-320.5)	1 ft (0.3 m)	Carbonate (dolomite) bed
1051.5–1054 (320.5–321.3)	2.5 ft (0.8 m)	Sandstone (not clean in appearance); some shale streaks
1054–1075 (321.3–327.7)	21 ft (6.4 m)	Mixed sandstone and siltstone with shale laminations
1075–1094 (327.7–333.5)	19 ft (5.8 m)	Mostly clean sandstone with some shale streaks and gray sandstone beds
1094–1107 (333.5–337.4)	13 ft (4 m)	Clean sandstone*
1107–1136 (337.4–346.3)	29 ft (8.8 m)	Mixed clean gray sandstone with some siltstone laminations and shale streaks
1136–1142 (346.3–348.1)	6 ft (1.8 m)	Interbedded sandstone, siltstone, and shale
1142–1165 (348.1–355.1)	23 ft (7 m)	Sandstone (not clean in appearance); some shale streaks

\*Sandstone bed sampled at depth of 1217.3 ft (371 m) (sample SS-3) in core C12996/UPH-3, equivalent in this core (UPH-1) to a depth of approximately 1101 ft (335.6 m).

Interval Identification	Depth (ft [m])	Interval Thickness	Depth (ft [m])	Lithology	Porosity (%)	<i>K</i> max (mD)	(Dm) 06 <i>X</i>	Kvert (mD)
Core 4	3751-3801 (1143.3-1158.5)	50 ft (15.2 m)	I	Black shale	8.4-10.3	<0.1	I	<0.1
Core 5	3810-3823 (1161.3-1165.3)	13 ft (4 m)	I	Interbedded shale	3.3-8.9	<0.1-16	<0.1–3.5	<0.1
				and siltstone				
Core 6	3823-3847 (1165.3-1172.6)	24 ft (7.3 m)	I	Missing	I	I	I	I
Core 7	3847-3870 (1172.6-1179.6)	23 ft (7 m)	I		3.5-8.9	<0.1-4.1	<0.1–3.8	<0.1-0.2
Core 7	3847-3870 (1172.6-1179.6)	23 ft (7 m)	3847-3856 (1172.6-1175.3)	Interbedded shale	4.8-7.7	<0.1-0.1	<0.1	<0.1
				and siltstone in beds up				
				to 3 in. thick (~50:50)				
Core 7	3847-3870 (1172.6-1179.6)	23 ft (7 m)	3856-3863 (1175.3-1177.4)	Interbedded sandstone	3.5-7.7	<0.1-4.1	<0.1–3.8	<0.1-0.2
				and shale in beds 3–12				
				in. thick; shale represents				
				approximately 20% of				
				interval and is concentrated				
				in beds 1–6 in. thick $*$				
Core 7	3847–3870 (1172.6–1179.6)	23 ft (7 m)	3863–3870 (1177.4–1179.6)	Silty sandstone with thin	5.5-8.9	<0.1-1.4	<0.1-1.2	<0.1-0.1
				wavy shale streaks; 90%				
				sandstone, 10% shale				
Core 8	3870-3940 (1179.6-1200.9)	49 ft (14.9 m)						
Core 8	3870-3940 (1179.6-1200.9)	49 ft (14.9 m)	3870–3880 (1179.6–1182.6)	Silty sandstone as above	5.6-10.3	<0.1-13	<0.1-13	<0.1–4.8
Core 8	3870-3940 (1179.6-1200.9)	49 ft (14.9 m)	3880-3896 (1182.6-1187.5)	Interbedded siltstone	5.5-10.9	<0.1-3.7	<0.1–2.6	<0.1-3.6
				and sandstone; shaly				
				zone in lower 2 ft				
Core 8	3870-3940 (1179.6-1200.9)	49 ft (14.9 m)	3896-3910 (1187.5-1191.8)	Mostly sandstone	5.5-10.2	<0.1-31	4.9–29	<0.1-10
Core 8	3870-3940 (1179.6-1200.9)	49 ft (14.9 m)	3910-3925 (1191.8-1196.3)	Interbedded siltstone	5.8-7.6	<0.1-18	<0.1-17	<0.1-4.2
				and sandstone				
Core 8	3870-3940 (1179.6-1200.9)	49 ft (14.9 m)	3925-3933 (1196.3-1198.8)	Mostly sandstone	6.2–9.2	0.1–28	0.1–25	<0.1-5.5
Core 8	3870-3940 (1179.6-1200.9)	49 ft (14.9 m)	3933-3940 (1198.8-1200.9)	Interbedded siltstone and	4.3–5.8	<0.1-2.5	<0.1–2.4	<0.1–2.5
				sandstone; numerous shale				
				layers 0.125–0.5 in. thick				
Core 8	3870-3940 (1179.6-1200.9)	49 ft (14.9 m)	3940 (1200.9)	Mostly sandstone	7.4-10.7	1.0–2.9	0.9–2.2	<0.1-0.8
Abbreviations: Kn	nax = maximum permeability of core me	easured in the horizontal	l direction; Kvert = permeability of core m	he vertical direction; $K90 = p$	bermeability measu	red after 90° rotat	tion from the dir	ection of Kmax.

Table A.2. Core C4006

\*Sandstone bed sampled at depth of 3857 ft (1175.6 m) (sample SS-3).



Figure A.1. Lithostratigraphy of core C131467 (Dane County, Wisconsin). Modified after Aswasereelert et al. (2008).

APPENDIX 3: PETROGRAPHIC DOCUMENTATION OF ALL SAMPLE REGIONS ANALYZED BY SECONDARY ION MASS SPECTROMETRY, WITH INDIVIDUALLY ANNOTATED ANALYSES PITS



Plate 1. (A) Shaly sandstone sample SS-2 (northern Illinois): cathodoluminescence-scanning electron microscopy (CL-SEM) image that corresponds to the backscattered electron (BSE) image of Figure 4A in the main text (see also Plate 4A herein). (B) Shaly sandstone sample SS-3 (central Illinois): CL-SEM image that corresponds to the BSE image of Figure 4D in the main text (see also Plate 4A herein). Ank = ankerite; Cal = calcite; DF = detritalK-feldspar; Dol = dolomite; DQ =detrital quartz; OF = overgrowth K-feldspar; QO = quartz overgrowth; V = void space.





**Plate 2.** Characteristic morphologies of calcite and dolomite cements in shaly sandstone sample SS-1 (Wisconsin arch). (A, C) Photomicrographs in plane-polarized light with (B, D) corresponding cross-polarized light images. (E, G) Backscattered electron (BSE) images with annotated secondary ion mass spectrometry (SIMS) analysis pits ( $\delta^{18}$ O Vienna standard mean ocean water [VSMOW]) and (F, H) corresponding cathodoluminescence (CL) images. Each analysis pit has a unique designation, such as "S8 (@205)," followed by the SIMS bias-corrected  $\delta^{18}$ O (VSMOW) value and the two standard deviations (2SD) value in parentheses; the "S8" is an analytical session designator, whereas the "(@205)" is a unique sample pit identification number for the corresponding session. Refer to Table 2 in the main text and Appendices 1 and 2. Cal = calcite; DF = detrital K-feldspar; Dol = dolomite; DQ = detrital quartz; ksp = K-feldspar (DF+OF); OF = overgrowth K-feldspar; V = void space.



**Plate 3.** Shaly sandstone sample SS-1 (Wisconsin arch). (A) Backscattered electron (BSE) image of a representative sample region where the  $\delta^{18}$ O Vienna standard mean ocean water (VSMOW) of carbonate cements (calcite and dolomite) was analyzed in situ by secondary ion mass spectrometry (SIMS). (B) Corresponding cathodoluminescence (CL) image. Each analysis pit has a unique designation, such as "S8 (@205)," followed by the SIMS bias-corrected  $\delta^{18}$ O (VSMOW) value and the two standard deviations value in parentheses; the "S8" is an analytical session designator, whereas the "(@205)" is a unique sample pit identification number for the corresponding session. Refer to Table 2 in the main text and Appendices 1 and 2. Cal = calcite; DF = detrital K-feldspar; Dol = dolomite; OF = overgrowth K-feldspar.



**Plate 4.** Shaly sandstone sample SS-2 (northern Illinois). (A) Backscattered electron (BSE) image of a representative sample region (Area 1) where the  $\delta^{18}$ O Vienna standard mean ocean water (VSMOW) of all dolomite–ankerite cement generations was analyzed in situ by secondary ion mass spectrometry (SIMS). Refer to Plate 1A for corresponding cathodoluminescence (CL) image. (B) The BSE and (C) corresponding CL images of early dolomite cement and the placement of 3-µm SIMS spots within the innermost crystal cores. (D) The BSE image of a supplementary, representative region (Area 2) where dolomite–ankerite cements were analyzed. Each analysis pit has a unique designation, such as "S8 (@205)," followed by the SIMS bias-corrected  $\delta^{18}$ O (VSMOW) value and the two standard deviations value in parentheses; the "S8" is an analytical session designator, whereas the "(@205)" is a unique sample pit identification number for the corresponding session. Refer to Table 2 in the main text and Appendices 1 and 2. Ank = ankerite; Dol = dolomite; DQ = detrital quartz; QO = quartz overgrowth; Sr = subregion.







Plate 4. Continued.



**Plate 5.** Shaly sandstone sample SS-3 (central Illinois). (A) Backscattered electron (BSE) image of a representative sample region (Area 9) where the  $\delta^{18}$ O Vienna standard mean ocean water (VSMOW) of dolomite–ankerite cements was analyzed in situ by secondary ion mass spectrometry (SIMS). Refer to Plate 1B for corresponding cathodoluminescence image. (B, C) Additional regions analyzed (Areas 2 and 6, respectively). Each analysis pit has a unique designation, such as "S8 (@205)," followed by the SIMS bias-corrected  $\delta^{18}$ O (VSMOW) value and the two standard deviations value in parentheses; the "S8" is an analytical session designator, whereas the "(@205)" is a unique sample pit identification number for the corresponding session. Refer to Table 2 in the main text and Appendices 1 and 2. Ank = ankerite; Dol = dolomite; DQ = detrital quartz; OQ = quartz overgrowth.



Plate 5. Continued.

# APPENDIX 4: COMPLETE SECONDARY ION MASS SPECTROMETRY DATA TABLE: 10- µM SPOT-SIZE SESSIONS

SIMS pit quality

Notes	File	Comment	δ <sup>18</sup> Ο (‰, VSMOW)	2SD 6 <sup>18</sup>	O bias 6	<sup>8</sup> O raw 5 <sup>18</sup>	0 (2SE) <sup>16</sup> C	(E9 cps) IF	(nA) Yield (	E9cps/nA)	date time	( microns	Y microns	DTFA-X	DTFA-Y	<sup>16</sup> O <sup>1</sup> H/ <sup>16</sup> O Sam	ple spot Fe# Sa	mple spot Ca#
	SIMS session	ı S7 (10-um spot-size): 13-	16th May, 2014	(J20)												_		(1001-1001)
	Calibration standar	rd mount: WI-STD-80																
	20140513@240.asc 20140513@241.asc 20140513@242.asc 20140513@243.asc 20140513@243.asc	<ul> <li>UWC-3 Grain 4</li> <li>UWC-3 Grain 1</li> <li>UWC-3 Grain 1</li> <li>UWC-3 Grain 2</li> <li>US-res= 183</li> <li>US-3 Grain 2</li> <li>SD</li> </ul>	12.49		4.61	8.02 7.88 7.74 7.66 <b>7.82</b>	0.39 0.36 0.42 0.31	1.937 1.915 2.045 2.060	.190 .174 .234 .244	.628 .631 .657	5/14/2014 20:31 5/14/2014 20:34 5/14/2014 20:40 5/14/2014 20:44	869 -45 796	726 596 -365 -330	-21 -19 -19	œ, ē, Ę, ē,	0.000878209 0.000916312 0.000890493 0.000877657		
	20140513@244.asc 20140513@245.asc 20140513@246.asc 20140513@247.asc	<ul> <li>UWO-1 grain 1</li> <li>UWO-1 grain 2</li> <li>UWO-1 grain 2</li> <li>UWO-1 grain 4</li> <li>average and 2 SD</li> </ul>	12.33	·	5.39	6.79 7.06 6.81 6.84 6.87	0.24 0.44 0.46 <b>0.25</b>	1.522 1 1.518 1 1.515 1 1.507 1	241 238 234 228	1.226 1.226 1.228	5/14/2014 20:49 5/14/2014 20:53 5/14/2014 20:57 5/14/2014 21:00	484 338 198 10	1811 2527 3378 4385	-26 -28 -28	4 9 4 4	0.000319328 0.000293101 0.000206439 0.000293573		
			bias *(STD-UW6220) Prop. error (2SE):		4.62 0.26													
	20140513@248.asc 20140513@249.asc 20140513@250.asc 20140513@251.asc 20140513@251.asc	<ul> <li>UW6220 dolomite Grain 1</li> <li>UW6220 dolomite Grain 3</li> <li>UW6220 dolomite Grain 4</li> <li>UW6220 dolomite Grain 2</li> <li>average and 2 SD</li> </ul>	22.60	·	9.96	12.40 12.17 12.61 12.48 <b>12.41</b>	0.28 0.36 0.31 0.37	2.180 2.167 2.138 2.128 2.128	219 205 194	1.788 1.786 1.775 1.775	5/14/2014 21:05 5/14/2014 21:09 5/14/2014 21:12 5/14/2014 21:16	-1488 -2532 -3954 -2908	759 1518 1202 591	22 24 24	φ ⊢ φ φ	0.000880465 0.000918037 0.000983065 0.00090259		
			bias*(STD-UW6220) Prop. error (2SE):		0.00													
	20140513@252.asc 20140513@253.asc 20140513@254.asc 20140513@254.asc 20140513@255.asc	<ul> <li>UWAnk1 Grain 3</li> <li>UWAnk1 Grain 5</li> <li>UWAnk1 Grain 2</li> <li>UWAnk1 Grain 2</li> <li>UWAnk1 Grain 3</li> <li>average and 2 SD</li> </ul>	15.88		1.92	13.86 13.76 13.95 14.13 <b>13.93</b>	0.17 0.18 0.22 0.32	2.210 2.181 2.188 2.163 2.163	176 162 154	.879 .878 .883 .874	5/14/2014 21:23 5/14/2014 21:27 5/14/2014 21:31 5/14/2014 21:37	-1215 -1221 -1853 -1359	-2275 -4085 -2446 -2446	6 4	9 5 7 9	0.001079286 0.001118702 0.001080029 0.001093807		
			bias*(STD-UW6220) Prop. error (2SE):		8.12 0.28													
	20140513@256.asc 20140513@257.asc 20140513@258.asc 20140513@259.asc 20140513@259.asc	<ul> <li>UWC-3 Grain 4</li> <li>UWC-3 Grain 1, Cs-res=184</li> <li>UWC-3 Grain 2</li> <li>UWC-3 Grain 3</li> <li>average and 2 SD</li> <li>bracket average and 2 SD</li> </ul>	12.49 12.49		4.60 4.60	8.09 7.62 7.64 <b>7.83</b>	0.47 0.43 0.38 0.38 0.38 0.38	1.964	.151 187 187	.619 .651 .641 .643	5/14/2014 21:41 5/14/2014 21:46 5/14/2014 21:50 5/14/2014 21:53	712 -70 962	849 377 -300 -300		÷ ÷ ÷ ÷	0.001160749 0.000970959 0.00118142 0.00096037		
			bias*(STD-UW6220) Prop. error (2SE):		5.41 0.23													
	SIMS 6 <sup>18</sup> O hias con	rection model for sample composition	ns along the dolomite-anke	rite solid soluti	on series													
	Bandard Standard UW620 dolomite UMAnk1 bitas a(STD–UW6 Session-specific con: bitas <sup>*</sup> <sup>ma</sup>	$F_{64} = \left[ \frac{F_{64}(k_{2} + k_{2})}{0.022} \right]$ $F_{64} = \left[ \frac{F_{64}(k_{2} + k_{2})}{0.022} \right]$ (220) $= \frac{0.022}{k^{11} + x^{11}}$ relatints: $= \frac{(2100 + k_{2} + k_{3})}{1.000}$ $= \frac{0.228}{0.000}$ $= \frac{0.228}{0.000}$	8.12 8.12 8.12															
	x = Fe# [= Fe/(Mg+F	k 0.10 Fe) molar ratio] of analyzed sample spot																
	SIMS 5 <sup>18</sup> O bias cor	rrection model for sample compositio	ns along the calcite-dolomi	te solid solutio	e													
	Standard UW6220 dolomite UWC-3 calcite	Ca/(Mg+Ca) molar ratio 0.501 0.976	bias*(STD-UW6220) 0 5.41															
	bias*(STD-UW6220, x = Ca/(Ma+Ca) mol	l) = 11.389x -5.706 lar ratiol of analyzed sample spot																
	Data lines 20140513	3@577-633: sample data that is not the	subject of this manuscript (A	DM CCS#1 551:	3.2 aka DVW#	1 5513.2')												
	20140513@634.asc 20140513@634.asc 20140513@635.asc 20140513@636.asc 20140513@637.asc	000 3637 C 4006 3857 UW6220 dol C 4006 3857 UW6220 dol C 4006 3857 UW6220 dol C 4006 3857 UW6220 dol				12.08 11.89 12.07	0.33 0.34 0.28	2.323 1 2.323 1 2.296 1 2.291		1.829 1.823 1.814 1.815	5/16/2014 0:39 5/16/2014 0:39 5/16/2014 0:42 5/16/2014 0:46 5/16/2014 0:49	-943 943 943	1105 1085 1065	14 - 14 18 14 14 14 14 14 14 14 14 14 14 14 14 14	E 51 65 65	0.001976297 0.002036046 0.002085866 0.002115155		
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1.891 1.878 1.879 1.879 1.858 1.858 1.858 1.858 1.924 1.1216 1.238	1.771 1.761 1.789 1.788	1,896 1,886 1,889 1,889 1,875 1,875 1,875 1,875 1,875 1,893 1,900 1,904 1,900	1.833 1.825 1.840 1.838		1.897 1.900 1.898 1.897	1.858 1.856 1.865 1.865 1.865 1.870 1.878 1.878 1.619 1.619 1.619	1.882 1.877 1.912 1.912	1.872 1.869 1.875 1.875 1.858 1.858	1.897 1.890 1.893 1.888			1.649 1.643 1.638 1.638	1.826 1.820 1.823 1.816	
1.280 1.280 1.280 1.272 1.272 1.250 1.250 1.250 1.250 1.250 1.250 1.250	1.203 1.200 1.266	1.278 1.276 1.277 1.277 1.267 1.267 1.268 1.268 1.268 1.258 1.258 1.258	1.210 1.206 1.266		1.269 1.271 1.271 1.272	1.271 1.275 1.265 1.265 1.263 1.263 1.263 1.266 1.246 1.246 1.246 1.246 1.246	1.219 1.216 1.269	1.268 1.268 1.268 1.268	1.268 1.268 1.268 1.268			1.137 1.132 1.127 1.121	1.111 1.105 1.100 1.095	
2,423 2,404 2,405 2,359 2,359 2,359 2,359 2,359 2,342 2,342 2,342 2,342 2,342 2,342 1,456 1,459 1,456	2.131 2.113 2.264	2,422 2,407 2,376 2,376 2,376 2,376 2,367 2,367 2,367 2,367 2,367 2,367 2,367 2,367 2,367 2,367 2,367 2,367 2,367 2,367 2,367 2,367 2,367 2,367 2,376 2,377 2,376 2,377 2,376 2,3777 2,3777 2,37777777777777777777777	2.218 2.321 2.327		2.408 2.416 2.413 2.413	2,362 2,365 2,365 2,365 2,368 2,358 2,368	2.295 2.283 2.425 2.426	2.374 2.367 2.378 2.378 2.358 2.358	2.406 2.397 2.399 2.395			1.875 1.860 1.846 1.836	2.028 2.012 2.004 1.989	
0.25 0.25 0.26 0.28 0.23 0.23 0.23 0.23 0.23 0.23 0.23 0.23	0.31 0.27 0.34 <b>0.36</b> <b>0.36</b>	0.19 0.27 0.27 0.28 0.28 0.25 0.23 0.23 0.23 0.23 0.23 0.23 0.23	0.42 0.35 0.34 0.31 0.33		0.37 0.35 0.36 0.46 <b>0.33</b>	0.35 0.34 0.34 0.37 0.30 0.30 0.30 0.37 0.30 0.30 0.48 0.48 0.48 0.48 0.48	0.31 0.37 0.35 0.34 0.34 0.35	0.40 0.37 0.45 0.24 0.40	0.41 0.34 0.30 0.37 0.06 0.27			0.31 0.36 0.32 0.34 <b>0.27</b>	0.24 0.37 0.27 0.27 0.33	
17.89 22.136 21.36 15.41 15.41 15.41 14.95 14.95 14.93 16.93 16.61	12.22 12.49 12.19 <b>12.24</b> <b>12.24</b>	13.22 15.28 18.10 17.67 17.67 19.84 19.82 19.22 19.22 15.60 15.50	12.18 12.53 12.05 12.22		12.61 12.30 12.35 <b>12.3</b> 5	13.55 13.75 13.75 13.76 13.78 13.70 13.70 13.70 13.29 13.29 20.05 20.05 20.05	12.35 12.70 12.22 <b>12.44</b> <b>12.40</b>	15.55 13.17 14.61 17.72 17.72 15.71	12.48 12.46 12.47 <b>12.49</b> <b>12.49</b>			9.12 9.31 9.45 <b>9.29</b>	13.33 13.50 13.10 <b>13.32</b> <b>13.31</b>	
4,71 3,46 3,46 5,232 4,42 5,53 5,53 5,63 5,63 5,63 5,63 5,63	-10.13		-10.15	<b>34.5'</b> )	-10.00	- 8.94 8.57 8.57 8.53 8.57 8.53 8.53 8.53 4.74 4.74 4.74 4.82 4.82	-9.94 -9.97	-9.19 -9.37 -9.06 -9.13	-9.89 1.91				90.6-	0.00 0.20
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	2.077 2.078 2.063 2.048			1.387 1.390 1.387 1.368 1.368		1.823 1.810 1.803 1.793														1.314 1.305 1.298 1.289 1.275 1.275 1.275	1.875 1.880 1.866 1.849 1.917	1.788 1.763 1.785 1.886 1.834 1.822	1.176 1.286 1.295 1.295	1.986 1.911 1.902 1.906 1.906 1.890 1.880	1.870 1.865 1.865 1.951 1.945 1.845
	0.25 0.23 0.36 0.17 0.17			0.29 0.41 0.32 0.32		0.39 0.42 0.32 0.31 0.31														0.36 0.45 0.40 0.31 0.37 0.37 0.36 0.36	0.31 0.21 0.22 0.27	0.37 0.33 0.24 0.25 0.35	0.36 0.42 0.39 0.32 <b>0.21</b>	0.20 0.29 0.24 0.21 0.21	0.24 0.26 0.29 0.35 0.28
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	20130923@31.asc 20130923@32.asc 20130923@33.asc 20130923@34.asc			20130923@38.asc 20130923@39.asc 20130923@40.asc 20130923@41.asc 20130923@41.asc 20130923@42.asc		20130923@43.asc 20130923@44.asc 20130923@45.asc 20130923@46.asc		SIMS 5 <sup>18</sup> O bias corr	Standard UW6220 dolomite	bias *(STD-UW6.	dession-specific cont	bias*m +	x = Fe# [= Fe/(Mg+F	SIMS 5 <sup>18</sup> O bias con	Standard	UW6220 dolomite UWC-3 calcite	bias*(STD-UWQ-1) =	x = Ca/(Mg+Ca) moli	Sample mount: C12	20130924@51.asc 20130924@55.asc 20130924@55.asc 20130924@55.asc 20130924@55.asc 20130924@55.asc 20130924@55.asc 20130924@56.asc 20130924@57.asc	20130924@58.asc 20130924@59.asc 20130924@60.asc 20130924@61.asc 20130924@61.asc 20130924@62.asc	20130924@63.asc 20130924@64.asc 20130924@65.asc 20130924@66.asc 20130924@67.asc 20130924@67.asc 20130924@68.asc	20130924@69.asc 20130924@70.asc 20130924@71.asc 20130924@72.asc	20130924@73.asc 20130924@74.asc 20130924@75.asc 20130924@76.asc 20130924@76.asc 20130924@76.asc 20130924@78.asc 20130924@78.asc	20130924@80.asc 20130924@81.asc 20130924@82.asc 20130924@83.asc 20130924@84.asc 20130924@84.asc
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1.264 1.250 1.259	1.253	1.916 1.916 1.898 1.892 1.898 1.928	1.944 1.861 1.899 1.908 1.908 1.891	1.259 1.264 1.266		1.247 1.248 1.245 1.247	1,432 1,435 1,414 1,717 1,771 1,771 1,771 1,771 1,771 1,771 1,771 1,771 1,771	1.256 1.250 1.248 1.245	1.243 1.243 1.244 1.246	1.712 1.741	1.248 1.249 1.245	1.777 1.779 1.779 1.778 1.778 1.773 1.773 1.755 1.773 1.755 1.773	1.240 1.237 1.224 1.237	1.274 1.277 1.272 1.275	1.957 1.923 1.901 1.872 1.880
0.957 1.087 1.072 1.072	1.069	1.049 1.044 1.036 1.036 1.031 1.022 1.018	1.013 1.009 1.000 1.000 0.997 0.986 0.986	0.977 0.972 1.010 1.023		1.010 1.008 1.006 1.006	0.998 0.998 1.002 1.009 1.012 1.013 1.015 1.016 1.016	1.011 1.009 1.012	1.008 1.002 0.993 0.987	0.978 0.972	0.958 0.958 1.041	1.042 1.038 1.036 1.035 1.029 1.029 1.022 1.022	1.019 1.017 1.009	0.991 0.985 0.982	0.979 0.979 0.987 0.993 0.993
1.210 1.359 1.358	1.339	2.010 1.996 1.975 1.960 1.980 1.980	1.970 1.898 1.903 1.908 1.908 1.865	1.229 1.229 1.279		1.259 1.258 1.252 1.252	1.429 1.422 1.408 1.798 1.798 1.798 1.738 1.738 1.738 1.752	1.270 1.262 1.266	1.253 1.246 1.235 1.229	1.673	1.200 1.196 1.293	1.852 1.859 1.845 1.845 1.852 1.852 1.761 1.761 1.796 1.718	1.264 1.259 1.240	1.262 1.257 1.253	1.917 1.883 1.875 1.858 1.858
0.40 0.33 0.22 0.40	0.34 0.31 0.29	0.26 0.27 0.28 0.28 0.28 0.28	0.21 0.34 0.28 0.28 0.33 0.33	0.30 0.26 0.32 0.32 0.32		0.34 0.37 0.42 0.36 <b>0.23</b>	0.26 0.17 0.19 0.25 0.25 0.25 0.25 0.26 0.25	0.38 0.29 0.44 0.38 0.38	0.38 0.28 0.46 <b>0.25</b>	0.27 0.29	0.38 0.40 0.41 0.41 0.40	0.25 0.35 0.37 0.37 0.35 0.34 0.33 0.34 0.33	0.38 0.33 0.48 0.48 0.42	0.32 0.35 0.50	0.13 0.30 0.25 0.32 0.32
7.25	7.26 7.28 7.16	18.52 17.71 14.28 14.28 16.08 19.85 19.12	20.96 15.94 14.18 16.16 15.15 14.91 14.91 13.45	7.14 7.14 7.03 7.40 7.23	(1094'))	6.89 7.16 7.02 7.08 7.04	22.15 22.38 14.78 15.85 16.67 16.67 16.54 16.54 16.12	7.42 7.01 7.10 7.14 7.14	7.17 7.36 7.08 7.29 <b>7.23</b>	17.19 18.09	7.22 7.70 7.43 7.46 7.34	15.76 15.23 15.44 17.38 17.04 14.29 14.29 14.29 16.07 16.56	7.57 7.91 7.70 7.75 7.59	7.68 7.67 7.80	7.70 16.05 20.35 20.67 16.42 18.07
7.443 7.047	-4.99	-5.85 -6.61 -7.28 -1.12 - 1.12		-5.09	I.1' and C12996	-5.23	0, 0,0,0,0,0,0,0, 0, 0, 0, 0, 0, 0, 0, 0	-5.12 -5.17	-5.04	-8.28	4.8 192	-7.74 -7.74 -7.74 -7.74 -7.76 -7.86 -7.86   	4.56 868		-4.57 -0.88 -2.55 -1.59 -1.10
at checked)		0.24 0.24 0.24 0.24			t (C12996 100-		0.31 0.31 0.31 0.31 0.31 0.31			- 0.40		0.42 0.42 0.42 0.42 0.42 0.42 0.42 0.42			0.29 0.29 0.29 0.29
. not checked) 39 (beam pos. n	12.33 12.33	23.71 21.03 19.83 20.99		90 12.33 12.33	of this manuscrip	12.33	25,56 25,79 24,46 24,46 25,28 25,45 25,45 25,83	12.33	12.33	- 26.58	12.33	23.68 23.15 23.36 25.31 25.09 24.79 -	12.33 12.33		12.33 16.94 23.39 23.28 18.03 19.19
C12996 1217.3 UWQ-1 lower grain (beam pos C12996 1217.3 UWQ-1 lower grain CS-Rea=10 C12996 1217.3 UWQ-1 lower grain C12996 1217.3 UWQ-1 lower grain	C12996 1217.3 UWQ-1 lower grain C12996 1217.3 UWQ-1 lower grain average and 2 SD bracket average and 2SD	C 12396 1217.3 Area R C0 5 Sport C 12396 1277.3 Area R C0 5 Sport	C 12396 1217,3 AreaH ROI 6 Sport C 12396 1217,3 AreaH ROI 6 Sport	C 12996 12/13 UWC-1 lower grain C 22996 12/13 UWC-1 lower grain C 12996 12/13 UWC-1 lower grain C 12996 12/13 UWC-1 lower grain C 12996 21/13 UWC-1 lower grain proverage and 2 SD	@110-180: sample data that is not the subject t 4667 277 or A1	C131487 2779 A1 UWO-1 C131487 2779 A1 UWO-1 C131487 2779 A1 UWO-1 C131487 2779 A1 UWO-1 C131487 2779 A1 UWO-1 average and 2 SD	C 131467 2777 9 A Avaid ROII Spot 1 C 131467 2777 9 A Avaid ROII Spot 2 C 131447 2779 A Avaid ROII Spot 2 C 131447 2779 A Avaid ROII Spot 2 C 131447 2779 A Avaid ROII Spot 9 C 131447 277	C131467 277.9 A1 UWO-1 C131467 277.9 A1 UWO-1 C131467 277.9 A1 UWO-1 C131467 277.9 A1 UWO-1 C131467 277.9 A1 UWO-1 Daraket average and 2 SD	C131467 277.9 A1 UWQ-1 NMR=1007664 C131467 277.9 A1 UWQ-1 C131457 277.9 A1 UWQ-1 C131457 277.9 A1 UWQ-1 average and 2 SD	C131467 277.9 A1 Area1 ROI4 Spot 1 C131467 277.9 A1 Area1 ROI4 Spot 2	C131467 277.9A1 UWQ-1 C131467 277.9A1 UWQ-1 C131467 277.9A1 UWQ-1 C131467 277.9A1 UWQ-1 C131467 277.9A1 UWQ-1 C314457 277.9A1 UWQ-1 C814487 277.9A1 UWQ-1 C814487 277.9A1 UWQ-1 D40444 aWU4g8 and 2 SD	C C C 31467 277 364 Avea RO C S 2001 1 2001 2001 2001 2001 2001 2001 20	C131467 277.9A1 UMQ-1 C131467 277.9A1 UMQ-1 C131467 277.9A1 UMQ-1 C131467 277.9A1 UMQ-1 C131467 277.9A1 UMQ-1 Saverage and 2 SD	06 3857 C4006 3857 UWQ-1 C4008 3857 UWQ-1 C4008 3857 UWQ-1 C4008 3857 UWQ-1 C4008 3857 UWQ-1	average and 2 SD C-2006 3857 Area6 RO 11 Spol 1 C-2006 3857 Area6 RO 11 Spol 2 C-2006 3857 Area6 RO 11 Spol 2 C-2006 3857 Area2 RO 12 Spol 1 C-2006 3857 Area2 RO 12 Spol 1
20130924@86.asc 20130924@87.asc 20130924@87.asc 20130924@88.asc 20130924@89.asc	20130924@90.asc 20130924@91.asc	20130324@92.asc 20130924@93.asc 20130924@94.asc 20130924@94.asc 20130924@96.asc 20130924@98.asc 20130924@98.asc	20130924@99.asc 20130924@100.asc 20130924@101.asc 20130924@102.asc 20130924@103.asc 20130924@104.asc 20130924@105.asc 20130924@105.asc	20130924@106.asc 20130924@107.asc 20130924@108.asc 20130924@109.asc 20130924@109.asc	Data lines 20130924 Samole mount: C13	20130925@286.asc 20130925@286.asc 20130925@288.asc 20130925@288.asc	20130925@289.asc 20130925@291.asc 20130925@291.asc 20130925@292.asc 20130925@292.asc 20130925@294.asc 20130925@294.asc 20130925@294.asc 20130925@294.asc 20130925@297.asc	20130925@299.asc 20130925@300.asc 20130925@301.asc 20130925@302.asc 20130925@302.asc	20130925@303.asc 20130925@304.asc 20130925@305.asc 20130925@306.asc	20130925@307.asc 20130925@308.asc	20130925@309.asc 20130925@310.asc 20130925@311.asc 20130925@312.asc 20130925@312.asc	20130925@313.asc 20130925@314.asc 20130925@316.asc 20130925@316.asc 20130925@316.asc 20130925@318.asc 20130925@318.asc 20130925@319.asc 20130925@319.asc 20130925@321.asc	20130925@323.asc 20130925@334.asc 20130925@322.asc 20130925@326.asc	Sample mount: C40 20130925@327.asc 20130925@328.asc 20130925@329.asc 20130925@330.asc	20130925@331.asc 20130925@331.asc 20130925@333.asc 20130925@334.asc 20130925@334.asc 20130925@335.asc
		Mixed analysis <sup>10</sup> , exclude Mixed analysis <sup>10</sup> , exclude Mixed analysis <sup>10</sup> , exclude	Mored analysta <sup>10</sup> , coclude Mored analysta <sup>10</sup> , coclude				Ecolode, Kopar Intergrowth(s) < 1 um cavities < 1 um cavities < 1 um cavities = 1 um cavities Ecolode, longer in pt Ecolode, cavities lenger than 1 um			Exclude: pit overlaps crack(s) < 1 µm cavities		<ul> <li>4 um cavites</li> <li>5 un cavites</li> <li>8 un cavites</li> <li>9 un</li></ul>			oldenu, " <sup>11</sup> bitylene boxM oldenu " <sup>11</sup> bitylene boxM oldenu " <sup>11</sup> bitylene boxM oldenu " <sup>11</sup> bitylene boxM
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0.466		,	0.442	- 0.238 0.136 0.143		0.464   0.141 0.238	
2.963E-03 3.140E-03 5.136E-03 2.985E-03 3.401E-03	5.761E-04 5.877E-04 5.921E-04 5.997E-04	5.687E-04	4.544E-03 3.469E-03 3.276E-03 3.626E-03 2.945E-03	3.129E-03 4.202E-03 3.429E-03 3.310E-03 2.965E-03 3.288E-03	6.008E-04 5.999E-04 5.970E-04 5.963E-04	3.259E-03 3.368E-03 3.286E-03 3.286E-03 3.395E-03 3.3112E-03 3.3112E-03 3.3112E-03 3.3145E-03 3.389E-03	5.930E-04 5.981E-04 6.337E-04 6.230E-04 6.200E-04
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1729 1732 1737 -1992 -1978	330 330 330	355	-1965 -1950 -550 -536 -526	-510 -482 -467 -467 -467 -482	254 254 254 254	2205 2175 2175 2134 2126 2126 2126 2126 2126 2165	236 231 160 160
16:54 16:59 17:03 17:12 17:15	17.21 17.24 17.28 17.28 17.31	17:41	17:45 17:50 17:55 17:55 18:03	18:07 18:11 18:15 18:20 18:24 18:27	18:33 18:36 18:40 18:43	18:52 18:56 19:01 19:05 19:13 19:13 19:23	19:29 19:33 19:37 19:41 19:45
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1.914 1.887 1.919 1.888 1.880	1.257 1.258 1.259	1.257	1.921 1.938 1.898 1.885	1.873 1.876 1.888 1.903 1.912	1.273 1.266 1.268	1.999 1.934 1.934 1.936 1.919 1.919 1.919 1.919	1.246 1.249 1.249 1.249
0.986 0.986 0.983 0.977 0.974	0.967 0.960 0.955 0.955	1.017	1.026 1.029 1.032 1.033	1.029 1.026 1.009 0.999 0.999	0.985 0.987 0.983 0.980	0.972 0.973 0.977 0.966 0.986 0.987 0.981 0.979	0.981 0.980 0.979 0.976 0.976
1.887 1.860 1.886 1.845 1.832	1.216 1.208 1.203	1.278	1.971 1.994 1.966 1.960	1.928 1.932 1.905 1.902 1.893	1.254 1.250 1.245	1.944 1.879 1.875 1.855 1.855 1.865 1.864 1.862	1.222 1.223 1.216 1.219 1.218
0.23 0.32 0.31 0.28 0.30	0.37 0.38 0.53 0.43 0.42 0.29	0.49	0.17 0.25 0.28 0.28	0.28 0.29 0.25 0.25 0.25	0.40 0.47 0.54 0.31	0.23 0.25 0.26 0.21 0.21 0.23 0.23	0.51 0.39 0.33 0.33 0.33 0.33 0.33 0.53
15.11 16.85 15.06 14.82 17.52	7.78 7.41 7.52 7.64 7.67	7.85	15.39 15.70 15.37 16.92 18.80	19.96 21.44 21.20 17.79 20.53	7.82 7.62 7.47 7.66 7.65	14.54 18.64 22.89 20.64 20.31 19.01 22.54	8.04 8.29 8.08 7.89 <b>8.07</b> 7.87
-0.86	4.63				-4.61 -4.62	-0.67 	8.560 -4.20 -4.41
0.29			0.34	0.34 0.34 0.34 0.34		0.53	
15.99	12.33 12.33		- - 16.33 -	- 23.23 24.07 21.14 23.76	12.33 12.33	15.22 - - 23.39 - 24.34	12.33 12.33
26@3363 asc C4006 3857 Area2 RO1 2 Spot 3 26@337 asc C4006 3857 Area2 RO1 2 Spot 4 26@3338 asc C4006 3857 Area2 RO1 2 Spot 5 26@338 asc C4006 3857 Area8 RO1 5 Spot 1 56@340 asc C4006 3857 Area8 RO1 5 Spot 2	26@341.asc C4066.3857 UWQ-1 26@342.asc C4066.3857 UWQ-1 26@344.asc C4066.3857 UWQ-1 26@344.asc C4066.3857 UWQ-1 26@344.asc c4066.3857 UWQ-1 bradeka tworage and 250	25@345.asc C4006 3857 UWQ-1 Cs-Res=203	560346 asc C 4006 3857 Area8 ROI 5 Spot 3 560347 asc C 4006 3857 Area8 ROI 5 Spot 4 560348 asc C 4006 3857 Area8 ROI 5 Spot 4 560348 asc C 4006 3857 Area8 ROI 5 Spot 1 560348 asc C 4006 3857 Area9 ROI 6 Spot 3	26@351 as C4006 3877 Mae8 ROI 5501 4 6@352.as C4006 3877 Mae8 ROI 5501 5 26@353.as C4006 3877 Mae8 ROI 5501 5 26@353.as C4006 3877 Mae8 ROI 5501 7 56@355.as C4006 3877 Mae8 ROI 5501 7 56@355.as C4006 3857 Mae8 ROI 5501 9	26@357.ac C4006.3857.UWQ-1 26@395.ac C4006.3857.UWQ-1 26@3959.ac C4006.3857.UWQ-1 26@390.ac C4006.3857.UWQ-1 25@390.ac c4006.3857.UWQ-1 bracket average and 2SD	Ereggisti are. C4008: 3857 Annal Roll 3 Spri 1 Ereggisti are. C4008: 3857 Annal Roll 3 Spri 2 Ereggisti are. C4008: 3877 Annal Roll 3 Ereggisti are. C4008: 3877 Annal Roll 3 Ereggisti are. C4008: 3877 Annal 8 Ereggisti are. C4008: 3877 Annal 8 Ereggist	25@370.asc C406 3857 UWQ-1 25@377.asc C406 3857 UWQ-1 25@377.asc C406 3857 UWQ-1 25@377.asc C406 3857 UWQ-1 25@374.asc C406 3857 UWQ-1 25@374.asc C406 3857 UWQ-1 briefek tworing and 28D
<ul> <li>2013095</li> <li>(is) 2013093</li> <li>(is) 2013093</li> <li>(is) 2013094</li> <li>2013095</li> <li>sige cavity 2013095</li> </ul>	2013090 2013090 2013090 2013090	2013092	(s) 2013002 (s) 2013002 (s) 2013002 (s) 2013002 e 2013002	(s) 2013095 (s) 2013095 2013095 2013095 2013095 2013095 2013095	201309( 201309( 201309( 201309(	a 2013095 (a) 2013095 (b) 2013095 (c) 201	2013095 2013095 2013095 2013095
Mixed analysis <sup>411</sup> , usable Exclude; pit overtaps crack Exclude; pit overtaps crack Mixed analysis <sup>411</sup> , excludi Exclude, pit overtaps crack(s) + Ia			Exclude: pit overlaps crack Exclude: pit overlaps crack Exclude: pit overlaps crack Mixed analysis <sup>21</sup> , exclude	Exclude, pit overlaps crack Exclude, pit overlaps crack		Mored analysis <sup>44</sup> , usable Mored analysis <sup>46</sup> , usable Ecoludio; pit overlage crass Ecoludio; pit overlage crass Intergrowths + more analysis <sup>44</sup> , Ecoludio; pit overlage crack	
Regular Irregular Regular Irregular			Irregular Irregular Regular Regular	Irregular Irregular Regular Regular Regular		Regular Regular Irregular Irregular Regular Irregular Irregular Regular	

<sup>10</sup> Mrcot analyse: In the *in-sult* analysis of 8<sup>1</sup>O in chemically zoned embouate minerals by SIMS. It is preferable that each sample pit hes placed within a single compositional zone transmission of the importance reportance minerals by SIMS. It is preferable that each sample pit hes placed within a single compositional zone transmission for a mixed many set of the matrix minimized method. The amplication of SIMS 8<sup>1</sup>O has corrections that its infinite method method. The method met

# APPENDIX 5: COMPLETE SECONDARY ION MASS SPECTROMETRY DATA TABLE: 3-µM SPOT-SIZE SESSIONS

SIMS pit quality

Notes	File	Comment	δ <sup>18</sup> Ο (‰, VSMOW)	2SD 5 <sup>18</sup> O bis	s $\delta^{18}$ O raw	δ <sup>18</sup> Ο (2SE)	<sup>16</sup> O (E6 cps)	date time	X micron	Y microns	DTFA-X	DTFA-Y	0 <sup>16</sup> 0 <sup>14</sup>	Sample spot Fe#	Sample spot Ca#
				(%°, VSMC	(M)									(Dol-Ank)	(Cal-Dol)
	SIMS session :	S4 (3-µm spot-size): 25-27th Fe	bruary, 2014												
	Calibration standard	I mount: WI-STD-80													
	20140224@16.asc 20140224@17.asc 20140224@18.asc 20140224@19.asc 20140224@20.asc 20140224@20.asc	WI-STD-80, UWC-3 shufter open Wi-STD-80, UWC-3 shufter open WI-STD-80, UWC-3 shufter open WI-STD-80, UWC-3 shufter open WI-STD-80, UWC-3 shufter open	12.49	-	1.523 -0.85 -0.91 -0.46 -0.73 <b>-0.73</b>	1.05 0.64 0.59 0.57 0.51	45.287 44.688 45.508 44.426 43.529	2/25/2014 8:46 2/25/2014 8:53 2/25/2014 9:02 2/25/2014 9:10 2/25/2014 9:10 2/25/2014 9:17	-327 -332 -328 -328 -328 -319	660 668 671 678 685	0000×	r- r- 9 8 8	1.639E-03 1.685E-03 1.785E-03 1.748E-03 1.788E-03 1.788E-03		
	20140224@21.asc 20140224@22.asc 20140224@23.asc 20140224@24.asc	WI-STD-80, UWC-1 gr.1 WI-STD-80, UWC-1 gr.2 WI-STD-80, UWC-1 gr.3 WI-STD-80, UWC-1 gr.4 average and 2 SD	12.33	-11.86	0.01 0.35 0.48 0.48	0.67 0.48 0.48 0.46 <b>0.42</b>	38.498 38.809 38.459 38.611	2/25/2014 9:27 2/25/2014 9:36 2/25/2014 9:44 2/25/2014 9:52	590 649 578	1295 2906 3602 4567	v 4 v o	φ ο	9.176E-04 9.325E-04 8.830E-04 8.692E-04		
			bias(STD-UW6220) Prop. error (2SE):	7.80 0.42											
	20140224@25.asc 20140224@26.asc 20140224@27.asc 20140224@28.asc	WI-STD-80, UWC-3 gr.1 WI-STD-80, UWC-3 gr.1 WI-STD-80, UWC-3 gr.1 WI-STD-80, UWC-3 gr.1 MI-STD-80, UWC-3 gr.1 Avg. and 2 SD	12.49	-13.68	-1.02 -1.50 -1.27 -1.27	0.70 0.64 0.55 0.55	45.577 44.161 44.860 45.051	2/25/2014 10:01 2/25/2014 10:08 2/25/2014 10:27 2/25/2014 10:35 2/25/2014 10:35	-309 -310 -298	684 674 664 681	00040	0 <sup>-</sup> 6 6 6	1.767E-03 1.874E-03 2.130E-03 1.865E-03		
	20140224@29.asc 20140224@30.asc 20140224@30.asc	Diacket: average and 2 50 WI-STD-80, UW6220 dol gr.1 WI-STD-80, UW5220 dol gr.3 WI-STD-80, UW6520 dol gr.3	8+ 77	10.01- 1	2.35 2.14 2.14	0.64	45.040 45.541 44 778	2/25/2014 10:45 2/25/2014 10:55 2/25/2014 10:55	-1202 -1833 -2916	921 1923 2682	1000	տ, տ, պ	1.554E-03 1.512E-03 1.424E-03		
	20140224@32.asc	WI-STD-80, UW6220 dol gr.4 average and 2 SD	22.60	-19.86	2.45 2.30	0.53 0.27	44.045	2/25/2014 11:10	-3484	1982	9	? 17	1.413E-03		
			bias(STD-UW6220) Prop. error (2SE):	0.00											
			bias(STD-UWQ-1) Prop. error (2SE):	-7.74 0.42											
	20140224@33.asc 20140224@34.asc 20140224@35.asc 20140224@36.asc	WI-STD-80. Ank-1 gr.1 WI-STD-80, Ank-1 gr.3 WI-STD-80, Ank-1 gr.2 WI-STD-80. Ank-1 gr.5 average and 2 SD	15.88	-10.71	4.74 4.91 5.10 5.24 5.00	0.39 0.37 0.29 <b>0.44</b>	59.048 58.785 57.351 58.620	2/25/2014 11:19 2/25/2014 11:27 2/25/2014 11:35 2/25/2014 11:43	-423 -1331 -481 -513	-1077 -1931 -2339 -3401	~ 6 ~ 6	- 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	1.466E-03 1.292E-03 1.309E-03 1.298E-03		
			bias(STD-UW6220) Prop. error (2SE):	9.33 0.36											
	SIMS 5 <sup>18</sup> O bias corre	ection model for sample compositions along	the dolomite-ankerite	solid solution seri	80										
	Standard UW6220 dolomite UWAnk1 bias #(STID-UW62: Session-specific const	$\begin{aligned} & \text{Feff} = \left[ \text{Fel(NQ+Fe}) \right] \\ & 0.004 \\ 0.522 \\ & 0.522 \\ & 20) = \frac{(b(as +_{max})x^n}{k^n + x^n} \end{aligned}$	bias *(STD-UW6220) 0.00 9.33												
	bias*,	max n k 0.10 k 0.10													
	x = Fe# [= Fe/(Mg+Fe	<ul> <li>molar ratio] of analyzed sample spot</li> </ul>													
	Data lines 20140224@ Data lines 20140224@	337-78: analyses of other non-dolomite-ankerit 3339-378: sample data that is not the subject of	e standards f this manuscript (ADM	CCS#1 5513.2 aka	DVW#1 5513 2'1										
	Sample mount: C129	996 1217.3'	-												
	20140224@379.asc 20140224@380.asc 20140224@381.asc 20140224@382.asc 20140224@383.asc 20140224@384.asc 20140224@385.asc	C12996 1273 UWQ-1 C12996 1273 UWQ-1 C12996 1273 UWQ-1 C12996 1277 UWQ-1	30V) 12.33	2 8 8 7 7 7 7 7 8	7,203 7,598 -6.13 -6.07 -6.07 -6.07 -6.07	0.60 0.73 0.55 0.55 0.65 0.46 0.40	36.466 36.243 36.539 36.446 35.571 35.571 35.509	2/27/2014 16:18 2/27/2014 16:25 2/27/2014 16:36 2/27/2014 16:45 2/27/2014 16:45 2/27/2014 16:52 2/27/2014 16:53	130 137 151 130 137	474 474 474 474 467 467	1 + + + + + + + + + + + + + + + + + + +	6 6 6 6 6 F	5.292E-04 6.597E-04 6.891E-04 7.222E-04 7.223E-04 7.243E-04 8.243E-04 8.255E-04		
Exclude; abnormal 2SE	20140224@386.asc 20140224@387.asc 20140224@388.asc	C12996 1217.3 A1 R1 Spot1 Dol-Ank C12996 1217.3 A1 R1 Spot2 Dol-Ank C12996 1217.3 A1 R1 Spot3 Dol-Ank	20.66 24.31 19.80	0.78 -18.08 0.78 -24.10 0.78 -16.62	2.21 -0.38 2.86	14.70 0.49 0.43	43.402 42.249 53.972	2/27/2014 17:19 2/27/2014 17:27 2/27/2014 17:35	-811 -809 -892	-1753 -1757 -1818	မှ မှ မှ	-19 -21	2.824E-03 4.237E-03 2.769E-03	- 0.024 0.513	
	20140224@389.asc 20140224@390.asc 20140224@391.asc 20140224@392.asc	C1296 1217,3 UWQ-1 C12996 1217,3 UWQ-1 C12996 1217,3 UWQ-1 C12996 1217,3 UWQ-1 AVG-and 2 SD Bracket: average and 2 SD	12.33 12.33	-17.95	-6.09 -6.99 -6.39 -5.85 -5.85	0.55 0.51 0.70 0.70 0.78	35.821 35.199 35.096 34.737	2/27/2014 17:45 2/27/2014 17:53 2/27/2014 18:00 2/27/2014 18:08	150 129 136	471 464 464	r, r, r, r,	7799	7.819E-04 8.125E-04 8.549E-04 8.348E-04 8.348E-04		
Missed larget	20140224@393.asc 20140224@394.asc 20140224@395.asc 20140224@396.asc 20140224@397.asc 20140224@398.asc 20140224@398.asc	C12996 1217.3 A1 R1 Spot4 Dol-Ank C12996 1277.3 A1 R1 Spot6 Dol-Ank C12996 1277.3 A1 R1 Spot6 Dol-Ank C12996 1277.3 A1 R1 Spot6 Dol-Ank C12996 1277.3 A1 R1 Spot9 Dol-Ank C12996 1277.3 A1 R1 Spot9 Dol-Ank	20.79 20.53 21.94 19.33 19.75	0.93 -16.31 0.93 -16.38 0.93 -17.93 0.93 -23.54 0.93 -22.84	4.14 3.82 3.62 -3.54 -3.54 -6.83	0.48 0.59 0.47 0.46 0.47 1.45	63.410 58.787 53.707 45.581 48.573 48.573	2/27/2014 18:18 2/27/2014 18:28 2/27/2014 18:37 2/27/2014 18:45 2/27/2014 18:53 2/27/2014 18:53	-920 -896 -874 -874 -917	-1824 -1798 -1792 -1779 -1769 -1728	ei <u>t</u> t t t ei ei	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	2.848E-03 2.812E-03 2.721E-03 4.654E-03 2.803E-03 3.133E-02	0.676 0.633 0.252 0.033 0.045	

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2.802E-03 5.671E-03	7.915E-04 8.294E-04 8.378E-04 8.039E-04	2.953E-03 2.913E-03 2.835E-03 2.825E-03 2.8071E-03 3.090E-03 3.090E-03 3.149E-03 3.149E-03	7.872E-04 7.969E-04 8.195E-04 7.894E-04 7.552E-04			0.001916663 0.001891053 0.001912977 0.001865505	0.00202446 0.001902186 0.001842399 0.001842399				0.001917978 0.001986835 0.001958207 0.001893924		0.002079631 0.002004771 0.002109825 0.001961048			0.002418117 0.002167842 0.003004554 0.002201979		0.001157633 0.001005096 0.000986626 0.001014232		0.002293631 0.002180405 0.00216137	0.002540689
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-1795	460 460 460	-2792 -2796 -2795 -2791 -2788 -2775 -2775 -2803 -1792	450 450 450 450			-481 311 -139	938 1537 1670 2487				-1321 -2579 -2710 -3742		-477 302 624 -135			-475 313 624 -129		1117 2507 3383 4461		-493 301 615	050
-876 -808	132 137 144 151	-1468 -1453 -1451 -1451 -1451 -1437 -1401 -1401 -1465 -1477 -787	131 138 152 152			68 -301 883 672	-1422 -2434 -3653 -3527				-487 -1440 -459 -447		81 -303 893 657			88 -295 901 649		510 321 277 251		74 -296 896 641	#80
2/27/2014 19:12 2/27/2014 19:22	2/27/2014 19:32 2/27/2014 19:40 2/27/2014 19:47 2/27/2014 19:54	2/27/2014 20:06 2/27/2014 20:13 2/27/2014 20:21 2/27/2014 20:28 2/27/2014 20:38 2/27/2014 20:43 2/27/2014 20:52 2/27/2014 20:52 2/27/2014 21:00 2/27/2014 21:00	2/27/2014 21:20 2/27/2014 21:27 2/27/2014 21:34 2/27/2014 21:41 2/27/2014 21:48			7/22/2014 17:49 7/22/2014 17:57 7/22/2014 18:05 7/22/2014 18:13	7/22/2014 18:21 7/22/2014 18:29 7/22/2014 18:36 7/22/2014 18:36				7/22/2014 18:55 7/22/2014 19:03 7/22/2014 19:10 7/22/2014 19:18		7/22/2014 19:26 7/22/2014 19:34 7/22/2014 19:43 7/22/2014 19:51			7/22/2014 21:05 7/22/2014 21:13 7/22/2014 21:22 7/22/2014 21:30		7/22/2014 22:09 7/22/2014 22:17 7/22/2014 22:24 7/22/2014 22:33		7/22/2014 22:41 7/22/2014 22:48 7/22/2014 23:01 7/22/2014 23:08	01;22 +102/22//
53.087 43.316	35.221 34.559 34.581 34.581	44.582 46.153 50.386 52.210 52.210 52.760 58.572 46.590 47.199 44.088	34.155 33.725 34.079 34.488 34.488 34.552			45.191 44.952 44.698 44.420	43.136 43.737 43.203 44.306				55.376 55.286 55.619 55.142		42.659 42.350 42.650 41.648			36.727 37.521 36.671 37.324		27.862 27.655 27.448 27.129		36.686 36.306 36.417 36.078	175.06
0.39 0.37	0.50 0.57 0.49 0.65 0.57	0.51 0.53 0.48 0.48 0.46 0.46 0.41 0.60	0.55 0.57 0.55 0.55 0.65 0.65 0.73			0.53 0.61 0.58 0.59	0.46 0.57 0.49 0.39	0.52			0.51 0.59 0.43 0.60		0.60 0.67 0.44 <b>0.25</b>			0.63 0.76 0.51 0.81 0.37		0.75 0.63 0.76 0.73 <b>0.96</b>		0.69	1.00 1.34
3.56 -2.13	6.09 6.04 6.14 6.14 6.14	-5.02 -1.63 -1.63 -2.41 -2.86 -0.19 -0.19 -3.78	6,12 6,575 6,09 6,28 6,21 6,18			-7.92 -8.09 -7.46 -8.08 -7.89	-3.74 -3.93 -3.33	-3.69			-1.41 -0.93 -0.69 -0.69		-7.66 -7.76 -7.96 -7.85 -7.81			-7.67 -7.87 -8.12 -7.84 -7.84 -7.84		-3.30 -2.78 -3.89 -3.24		-6.89 -6.69 -7.63	-0.38 -6.81 -7.28
-17.93 -24.12	-18.25 -18.10	-24,24 -21,18 -18,77 -17,88 -16,66 -16,66 -16,66 -22,0,7 -21,44 -24,92	-7.308 -18.31 -18.28				-7.55	-25.71	0.00 0.30	-9.93 0.43	-16.64	9.31 0.45	-20.09	5.77		-20.08		-15.38	10.03 0.73		-19.53
0.93		0.73 0.73 0.73 0.73 0.73 0.73 0.73 0.73																			
21.89 22.54	12.33 12.33	19.69 19.97 20.56 21.26 21.26 21.23 22.32 22.32 21.68	12.33 12.33	ly, 2014				22.60	bias(STD-UW6220) Prop. error (2SE):	bias(STD-UWQ-1) Prop. error (2SE):	15.88	bias(STD-UW6220) Prop. error (2SE):	12.49	bias(STD-UW6220)	e standards	12.49	e standards	12.33	bias(STD-UW6220) Prop. error (2SE):	ecked	12.49
C12996 1217.3 A1 R1 Spot10 Dol-Ank C12996 1217.3 A1 R1 Spot11 Dol-Ank	C12996 1217.3 UWQ-1 C12996 1217.3 UWQ-1 C12996 1217.3 UWQ-1 C12996 1217.3 UWQ-1 C12996 1217.3 UWQ-1 Avg. and 2 SD Bracket: average and 2SD	C12966 1271 3 A2 R2 Sport Dol-Avia C12966 1271 3 A2 R2 Sport Dol-Avia C12966 1271 3 A2 R2 Sport Dol-Avia C12960 1271 3 A1 R1 Sport Dol-Avia C12960 1271 3 A1 R1 Sport Dol-Avia C12960 1271 3 A1 R1 Sport Dol-Avia	C12986 12/13 UWQ-1 C12986 12/13 UWQ-1 C12986 12/13 UWQ-1 C12986 12/13 UWQ-1 C12986 12/13 UWQ-1 C12986 12/13 UWQ-1 Bracket: werage and 25 D	i8 (3-µm spot-size): 22-25th Ju	mount: WI-STD-80	WI-STD-80 UWC-3 SW gr WI-STD-80 UWC-3 gr.1 WI-STD-80 UWC-3 gr.4 WI-STD-80 UWC-3 gr.3 average and 2SD	WI-STD-80 UW6220 gr.1 WI-STD-80 UW6220 gr.3 WI-STD-80 UW6220 gr.4 WI-STD-80 UW6220 gr.5	average and 2SD			WI-STD-80 Ank-1 gr.1 WI-STD-80 Ank-1 gr.3 WI-STD-80 Ank-1 gr.2 WI-STD-80 Ank-1 gr.5 average and 2SD		WI-STD-80 UWC-3 SW gr WI-STD-80 UWC-3 SW gr WI-STD-80 UWC-3 gr,4 HV no change WI-STD-80 UWC-3 gr,5 WI-STD-80 UWC-3 gr,5 UWC-3 Reference bracket average and UWC-3 Reference bracket average and		23-30: analyses of other non-dolomite-ankerite	WI-STD-80 UWC-3 SW gr. WI-STD-80 UWC-3 SM gr.1 WI-STD-80 UWC-3 gr.4 HV no change WI-STD-80 UWC-3 gr.3 average and 2SD bracket average and 2SD	35-38: analyses of other non-dolomite-ankerit	WI-STD-80 UWO-1 gr.1 WI-STD-80 UWO-1 gr.2 WI-STD-80 UWO-1 gr.3 WI-STD-80 UWO-1 gr.4 average and 2SD		MI-STD-80 UWC-3 SW gr. WI-STD-80 UWC-3 gr.4 WI-STD-80 UWC-3 gr.4 EM HV adjust not cf. WI-STD-80 UWC-3 gr.3	WH-STD-80 UWC-3 gr.4 average and 2SD bracket average and 2SD
20140224@399.asc 20140224@400.asc	20140224@401.asc 20140224@402.asc 20140224@403.asc 20140224@404.asc 20140224@404.asc	20140224@405.asc 20140224@406.asc 20140224@406.asc 20140224@408.asc 20140224@409.asc 20140224@410.asc 20140224@411.asc 20140224@41.asc 20140224@413.asc 20140224@413.asc	20140224@414.asc 20140224@415.asc 20140224@415.asc 20140224@418.asc 20140224@418.asc 20140224@418.asc	SIMS session S	Calibration standard	20140722@7.asc 20140722@8.asc 20140722@9.asc 20140722@10.asc	20140722@11.asc 20140722@12.asc 20140722@13.asc 20140722@14.asc				20140722@15.asc 20140722@16.asc 20140722@17.asc 20140722@18.asc		20140722@19.asc 20140722@20.asc 20140722@21.asc 20140722@22.asc		Data lines 20140722@	20140722@31.asc 20140722@33.asc 20140722@33.asc 20140722@34.asc 20140722@34.asc	Data lines 20140722@	20140722@39.asc 20140722@40.asc 20140722@41.asc 20140722@42.asc		20140722@43.asc 20140722@44.asc 20140722@45.asc 20140722@46.asc	20140/ <i>22</i> @4/.asc

Regular Regular Regular Regular Regular Regular Regular Regular Regular

Crack nearby

Calibration standard mount: 2014\_MGS\_Carb\_02

											0.504 0.504 0.648 0.648 0.648 0.508 0.508 0.504		N/A N/A 0.237 0.237 0.216 0.016		0.226 0.235 0.055 0.037 0.037 0.037 0.036 0.036	
0.002536708 0.002763304 0.002368479 0.003162217	0.002783131 0.002872387 0.002888767 0.003057995		0.003058871 0.002961962 0.003347447 0.003396709							0.006512637 0.006225072 0.006693113 0.006485464	0.006975568 0.006857608 0.006820973 0.007481611 0.0077307875 0.007188016 0.00753002	0.006890548 0.007093519 0.007222319 0.006872909	0.001575008 0.001772033 0.001968557 0.007013892 0.00549105 0.006649105 0.008667856 0.04469159	0.006473298 0.006619624 0.006864361 0.006423098	0.005872952 0.006538249 0.0055915915 0.005915915 0.0050811996 0.0060811078 0.00587741 0.00587741 0.005847798	0.005814877 0.005992556 0.006029309 0.005769292
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681 672 662 654	-992 -1721 -2383 -2718		627 619 611 603							61 50 27	-1406 -1406 -1406 -1406 -1406 -1400 -1400 -1400	64 52 45	-1315 -1320 -1320 -1319 -1319 -1310 -1320 -1182 -1182	68 61 54 47	-1288 -1288 -1293 -1272 -1273 -1279 -1249 -1249 -1243	69 55 48
-114 -115 -116	332 1028 827 326		- 119 - 119							-116 -117 -118	-1025 -1018 -1014 -1004 -997 -1011	-128 -128 -128 -128	-1037 -1037 -1034 -1040 -1040 -1028 -881 -881	-138 -138 -138 -138	-1061 -1065 -1065 -1042 -1050 -1055 -1055 -1055	-149 -149 -149
7/22/2014 23:31 7/22/2014 23:39 7/22/2014 23:49 7/22/2014 23:57	7/23/2014 0:05 7/23/2014 0:12 7/23/2014 0:20 7/23/2014 0:27		7/23/2014 0:35 7/23/2014 0:42 7/23/2014 0:51 7/23/2014 1:00							7/23/2014 20:39 7/23/2014 20:48 7/23/2014 20:58 7/23/2014 21:05	7/23/2014 21:17 7/23/2014 21:26 7/23/2014 21:34 7/23/2014 21:51 7/23/2014 21:51 7/23/2014 22:08 7/23/2014 22:08	7/23/2014 22:25 7/23/2014 22:32 7/23/2014 22:43 7/23/2014 22:41	7/23/2014 23:00 7/23/2014 23:16 7/23/2014 23:16 7/23/2014 23:22 7/23/2014 23:32 7/23/2014 23:32 7/23/2014 23:52	7/24/2014 0:09 7/24/2014 0:16 7/24/2014 0:27 7/24/2014 0:34	7/24/2014 0:44 7/24/2014 0:52 7/24/2014 1:00 7/24/2014 1:07 7/24/2014 1:24 7/24/2014 1:24 7/24/2014 1:32 7/24/2014 1:32 7/24/2014 1:31	7/24/2014 1:56 7/24/2014 2:04 7/24/2014 2:13 7/24/2014 2:20
34.714 34.936 35.187 34.873	39.318 39.111 39.408 39.530		34.297 33.892 34.458 35.167							45.645 48.228 48.689 48.242	59.576 60.839 61.827 58.678 58.678 52.816 63.694 53.694 59.670 59.670	45.025 45.023 47.991 48.319	37.651 36.071 35.766 56.833 56.633 42.272 47.612 45.909	46.079 45.627 48.383 48.228	58.483 56.737 56.827 51.057 49.829 49.829 49.829 49.816 51.726 51.726 50.989	46.541 46.633 48.849 49.208
0.55 0.70 0.50 0.72 <b>1.28</b>	0.55 0.59 0.47 0.48 <b>0.62</b>		0.84 0.49 0.67 <b>0.90</b>							0.56 0.45 0.41 0.54	0.55 0.52 0.56 0.56 0.51 0.45	0.49 0.53 0.55 0.47 0.47	0.61 0.55 0.55 0.45 0.45 0.90 1.83	0.58 0.59 0.61 0.18 0.18	0.47 0.53 0.47 0.63 0.63 0.48 0.48 0.48	0.61 0.59 0.59 0.59 0.71
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	-15.91	8.27 0.64	-18.35	olution series						-23.21	-13.97 -13.97 -14.01 -14.01 -15.77 -14.01 -13.65 -13.65 -13.97	-23.48	-14.01 -14.01 -14.01 -15.89 -15.89 -15.76 -22.73 -22.73	-24.12 -23.80	-16.50 -16.26 -16.40 -20.77 -21.79 -21.86 -21.86 -22.65 -22.65	-24.45 -24.29
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change	9.220	bias(STD-UW6220) Prop. error (2SE):	12.49	ing the dolomite-anker	bias*(STD-UW6220 9.00 9.31 8.27					io change 22.60	18.07 19.88 18.91 20.185 23.49 23.49 19.27 19.27	nge; Cs res> 86 22.60 22.60	24.67 24.35 24.56 22.31 22.31 23.30 23.30	no change, Cs res> E 22.60 22.60	21.35 21.73 21.85 21.65 21.67 21.67 23.94 23.93	no change; Cs res> E 22.60 22.60
2014. MGS. Carb. 02 UWC-3 2014. MGS. Carb. 02 UWC-3 2014. MGS. Carb. 02 UWC-3 2014. MGS. Carb. 02 UWC-3 2014. MGS. Carb. 02 UWC-3 average and 2SD	2014 MGS Carb D2 Ank93418-08 gr 1 2014 MGS Carb D2 Ank93418-08 gr 2 2014 MGS Carb D2 Ank93418-08 gr 3 2014 MGS Carb D2 Ank93418-08 gr 8 2014 MGS Carb D2 Ank93418-08 gr 7 verage and 25D		2014. MGS. Carb. 02 UWC-3 2014. MGS. Carb. 20 UWC-3 2014. MGS. Carb. 02 UWC-3 2014. MGS. Carb. 02 UWC-3 average and 2SD bracket average and 2SD	ction model for sample compositions ald	Fe# = [ Fe/(Mg+Fe) ] 0.004 0.179 0.179	$0) = \frac{(bias *_{max})x^n}{k^n + x^n}$ ants:	n n 12 k 0.10	molar ratio] of analyzed sample spot	<b>36 1217.3</b> '	C129996 1217.3' UW6220 C129996 1217.3' UW6220 C129996 1217.3' UW6220 C129996 1217.3' UW6220 E1217.3' UW6220 average and 2SD	C12996 1277.3 A sport doi-ank C12996 1277.3 A sport doi-ank C12996 1277.3 A sport doi-ank C12996 1277.3 A sport doi-ank C129966 1277.3 A sport doi-ank C129966 1277.3 A sport doi-ank C129966 1277.3 A sport doi-ank C12996 1277.3 A sport doi-ank	C129996 1217.3' UW6220 dol C12996 1217.3' UW6220 dol C12996 1217.3' UW6220 dol C12996 1217.3' UW6220 C12996 1217.3' UW6220 bracket average and 2SD bracket average and 2SD	C12969 1217.3" A1 spot 9 QQ C12969 1217.3" A1 spot 10 QQ C12959 1217.3" A1 spot 11 QQ C12959 1217.3" A1 spot 12 004-ank C12959 1217.3" A1 spot 13 004-ank C12959 1217.3" A1 spot 14 dok-ank C12959 1217.3" A1 spot 16 dok-ank C12959 1217.3" A1 spot 16 dok-ank	C12896 1217.3' UW6220 dol C12896 1217.3' UW6220 dol C12996 1217.3' UW6220 dol C12996 1217.3' UW6220 dol C12996 1217.3' UW6220 dol bracket average and 2SD bracket average and 2SD	CT2866 1217.3 A1 spot 17 dol-ank CT2866 1217.3 A1 spot 18 dol-ank CT2866 1217.3 A1 spot 18 dol-ank CT2866 1217.3 A1 spot 18 dol-ank CT2866 1217.3 A1 spot 20 dol-ank	C12996 1217.3' UW6220 dol C12996 1217.3' UW6220 dol C12996 1217.3' UW6220 dol C12996 1217.3' UW6220 dol EM HV adj C12996 1217.3' UW6220 dol Everage and 25D bracket average and 25D
20140722@48.asc 20140722@49.asc 20140722@50.asc 20140722@51.asc	20140722@52.asc 20140722@53.asc 20140722@55.asc 20140722@55.asc IMF adjusted		20140722@56.asc 20140722@57.asc 20140722@58.asc 20140722@59.asc	SIMS 5 <sup>18</sup> O bias corre	Standard UW6220 dolomite UWAnk1 UWAnk4	bias «(STD-UW622 Session-specific consta	bias* <sub>n</sub>	x = Fe# [= Fe/(Mg+Fe)	Sample mount: C1295	20140722@144.asc 20140722@145.asc 20140722@146.asc 20140722@147.asc	20140722@148.asc 20140722@149.asc 20140722@150.asc 20140722@151.asc 20140722@152.asc 20140722@152.asc 20140722@155.asc 20140722@155.asc	20140722@156.asc 20140722@157.asc 20140722@158.asc 20140722@159.asc	20140722@160.asc 20140722@161.asc 20140722@162.asc 20140722@153.asc 20140722@164.asc 20140722@165.asc 20140722@165.asc 20140722@165.asc 20140722@167.asc	20140722@168.asc 20140722@169.asc 20140722@170.asc 20140722@171.asc	20140722@172.asc 20140722@173.asc 20140722@175.asc 20140722@175.asc 20140722@176.asc 20140722@176.asc 20140722@178.asc 20140722@178.asc 20140722@178.asc 20140722@180.asc	20140722@181.asc 20140722@182.asc 20140722@183.asc 20140722@184.asc
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0.71 0.41 0.56 0.53 0.61 0.48 0.48	0.45 0.55 0.53 0.61 0.61	0.54 0.71 0.49 <b>0.22</b>	0.47 0.52 0.32 0.49 0.45 0.45 0.45	0.62 0.49 0.59 0.56 0.71	0.66 0.54 0.54 0.54 0.58 0.58 0.56 0.56 0.56	0.65 0.54 0.52 0.59 1.11			0.72 0.61 0.54 0.54	0.63 0.54 0.60 0.60 0.65 0.36 0.36	0.67 0.51 0.67 0.48 0.80	0.56 0.55 0.64 0.66 0.43 0.43 0.43 0.38	0.58 0.44 0.60 0.67 0.74 0.65	0.61 0.54 0.57 0.62 0.52 0.52 1.65
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C131467 277,9A1 UW6220 dol C131467 277,9A1 UW6220 dol C131467 277,9A1 UW6220 dol C5 res -> 96, EM HV no change average and 250 dol C5 res -> 95, EM HV no change Dareket average and 250 22.60 Dareket average and 250 22.60	3857'	C4006 3657 UW6220 dol C4008 3657 UW6220 dol EM HV adj> 2411 V C4008 3657 UW6220 dol EM HV adj> 2411 V C4008 3657 UW6220 dol	C4006 3957 A2 R2 Spot 1 16.65 C4006 3957 A2 R2 Spot 2 ank 20.05 C4006 3957 A2 R2 Spot 2 ank 19.87 C4006 3957 A2 R2 Spot 3 ank 19.87 C4006 3957 A2 R2 Spot 4 ank 16.03 C4006 3957 A2 R2 Spot 6 ank 15.10 C4006 3957 A2 R2 Spot 6 ank 15.11 C4006 3957 A2 R2 Spot 9 ank 15.13 C4006 3957 A2 R2 Spot 9 ank 15.13 C4007 A2 R2	C4006 3857 UW6220 dol C4006 3857 UW6220 dol EM HV adj> 2443V, Cs res>97 C4006 3857 UW6220 dol EM HV adj> 2443V, Cs res>97 C4006 3857 UW6220 dol EM HV adj> 2260 bracket average and 25D bracket average and 25D	C4008 3857 A9 R6 Spot 1 Ank 13.63 C4008 3857 A9 R6 Spot 3 Ank 13.64 C4008 3857 A9 R6 Spot 3 Ank 16.34 C4008 3857 A9 R6 Spot 4 Ank 16.34 C4008 3857 A9 R6 Spot 6 Ank 16.29 C4008 3857 A9 R6 Spot 6 Ank 15.67 C4008 3857 A9 R6 Spot 6 Ank 17.25 C4008 3857 A9 R6 Spot 8 Ank 17.25	C4006 3857 UW6220 dol C4006 3857 UW6220 dol C4008 3857 UW6220 dol C4008 4857 UW6220 dol
20140722@350.asc C 20140722@355.asc C 20140722@352.asc C 20140722@353.asc P <b>P</b>	Sample mount: C4006 38!	20140722@354.asc C 20140722@355.asc C 20140722@356.asc C 20140722@357.asc C	20140722@358.astc 20140722@358.astc 20140722@359.astc 20140722@351.astc 20140722@351.astc 20140722@351.astc 20140722@356.astc 20140772@356.astc 20140772@356.astc 20140772@356.astc 20140772@356.astc 20140772@356.astc	20140722@367.asc C 20140722@368.asc C 20140722@369.asc C 20140722@370.asc C 20140722@370.asc P b	20140172@371.asc C 2014072@373.asc C 2014072@373.asc C 20140722@374.asc C 20140722@376.asc C 20140722@376.asc C 20140722@376.asc C 20140722@376.asc C	20140722@379.asc C 20140722@380.asc C 20140722@381.asc C 201407729@487.asc C
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