<u>Supplementary Information</u> for "*Winter Wonderland Cave, Utah, USA: A Natural Laboratory for the Study of Cryogenic Cave Carbonate and Thawing Permafrost*"

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Sample Preparation and Analysis

Individual CCC samples were separated into 3 size fractions to examine whether properties of the CCCs differed with size. For each sample, wet sediment was scooped out of its plastic vial with a clean spatula and deposited on a 250-µm mesh sieve over a clean beaker. Distilled water and prodding from a spatula separated the sample components that were >250 µm, leaving the <250-µm fraction and water in the beaker. This solution was then poured through a 75-µm mesh sieve over a second clean beaker and more distilled water separated the samples into 250-75-µm and <75 µm fractions. Each fraction was placed in a 50-mL centrifuge tube, and after fine sediment had settled, excess water was carefully decanted. Samples were then frozen in a -40°C freezer for 4 hours. The uncapped centrifuge tubes were then placed in a Labconco FreeZone 6 Liter freeze dryer and allowed to dry overnight. The resulting powder was ground with a clean mortar and pestle and returned to each tube.

Scanning Electron Microscopy

A Tescan Vega 3 LMU scanning electron microscope (SEM) at Middlebury College was used to acquire images of the CCC. All samples in different grain size fractions were adhered to metal disks with double sided tape in preparation for analysis. Each metal disk was coated in gold palladium, and images were captured for the three grain size fractions for each sample at different magnifications in order to compare CCC morphologies. Energy dispersive x-ray spectroscopy (EDS) was applied to samples YS-3, YS-6, TS-2, CP, YP, and TF after carbon coating to determine if different grain morphologies observed under the SEM correspond to different elemental compositions. The semi-quantitative elemental results were converted to weight % oxides to differentiate between calcite, dolomite, and quartz. AZtecOne software was used to capture secondary electron and backscatter images and for elemental analysis. Weight % oxide data were transformed into structural formulae for common minerals.

Grain Size Distribution

The smallest size fraction (<75 μm) of each sample was analyzed using the HORIBA LA-950 to determine its grain size distribution. Samples of the <75-μm fraction were centrifuged for 2 minutes and the liquid was poured off to yield a volume of ~25 mL. Then samples were deflocculated for 30 seconds using a Vortex-T Genie 2, then sonified with an FS20D sonifying water bath for 1 minute. A clean syringe was used to transfer liquid and sediment of each sample into the HORIBA. Sample runs were duplicated to assess drift of the instrument over the course of the analysis. The HORIBA grain size distribution data were presented as volume percentages of sand (coarse, medium, and fine), silt (coarse, medium, fine, and very fine), clay (2-1 μm), and colloid (<1 μm).

X-ray Diffraction

In preparation for bulk sample x-ray diffraction (XRD) analysis, dried samples were ground using a ceramic mortar and pestle. Samples of the 250-75-µm size fraction, as well as rock samples powdered in a shatterbox, were analyzed on a Bruker D8 Advance Model X-Ray Diffractometer at Middlebury College. The mineralogy of several samples was compared across different size fractions (<75 µm and 250-75 µm) to detect any compositional differences. As no obvious differences were detected, the grain size fractions <75 µm and 250-75 µm were used interchangeably for future analyses depending on the volume of sample remaining. Nine samples of the two size fractions were analyzed using the Random Powder Long Routine (RPLR), which scans samples at 2-50° 2Θ. Samples CP, YP, and TF did not have enough material to fill a holder in the XRD, so they were transferred to a glass slide and analyzed using an Oriented Powder Long Routine, which scans samples at 2-40° 2Θ. The diffractometer operated at 40 kV and 40 mA with a solid state detector, theta-theta goniometer, and CuKα radiation. Mineral abundances were quantified using intensity ratios, and known mineral spectra peaks were identified by comparison with standard reference patterns.

X-ray Fluorescence

CCC samples were analyzed for major element composition using x-ray fluorescence (XRF) at Middlebury College. All samples were of the 75-250-µm size fraction. Samples were heated in a LECO TGA-701 thermogravimetric analyzer prior to XRF analysis to remove organics and interstitial water. Dried samples were thoroughly ground into a fine powder and combined in a

10:1 ratio of lithium borate fluxing agent to sample (0.6000 g of sample and 6.000 g fluxing agent). The combined sample and fluxing agent were melted in a Claisse LeNeo fluxer at 1050° C for 22 minutes to form a glass disk. This process was repeated for five of the collected CCC samples (YS-4, YS-6, TS-1, TS-2, and one rock sample); the other samples did not contain sufficient mass in the desired particle size range for fluxing. Glass disks were analyzed using a Thermo Scientific ARL QuantX energy dispersive (ED) XRF spectrometer to measure major elements. A glass disk made from standard 88B (dolomitic limestone) was included in each run to assess the quality of measurements. The analysis quantified the abundance of oxides, which were converted to atomic abundances of major elements (Si, Ti, Al, Fe, Mn, Mg, Ca, Na, K, P).

Inductively Coupled Plasma Mass Spectrometry

Seven samples of the size fraction <75 μm (YS-3, YS-4, YS-5, YS-6, TS-1, TS-2, and a bedrock sample), and an additional YS-4 sample of grain size 250-250 μm were prepared for analysis with inductively coupled plasma mass spectrometry (ICP-MS) to determine the abundance of trace and rare earth elements. Dried samples were combined in a 9:1 ratio of lithium metaborate fluxing agent to sample (0.2000 g sample and 1.8000 g LiBO2). A Claisse LeNeo fluxer was used to melt the sample and fluxing agent and combine the melt with 5% HNO_3. Dissolution was aided by a spinning magnetic bar. Dissolved samples were then transferred to a 100-mL volumetric flask and HNO_3 was added to make 100 mL. This solution (T1) was transferred to a 125-mL polypropylene bottle as a 100× dilution of the original sample. An additional dilution was made for trace element analysis by pipetting 5 mL of the T1 solution and 2 mL of an internal standard solution containing 1 ppm Rh, In, Re, and Bi. Nitric acid was also added to this solution to make 100 mL. This resulting T2 volume dilution was 2000×.

A Thermo Scientific iCAP Q ICP-MS was used in conjunction with Qtegra ISDS software to analyze the dissolved CCC and rock samples, along with samples of water from the cave. For each run, calibration curves were generated based on standard solutions specific to the regular or trace elements method. Quality control with internal standards to drift correct was conducted after every five samples. USGS rock standard 88B (dolomitic limestone) was analyzed at the beginning and end of every batch of CCC samples, and standard 1643f was run in conjunction with water samples. Data were synthesized with Qtegra software.

C and O Stable Isotopes

Eleven samples (all <75-µm size fraction except CP and YP 250-75 µm) were prepared for $\delta^{18}O$ and $\delta^{13}C$ stable isotope analysis in the Department of Geology at Union College. Roughly 0.09 mg of sample was weighed into glass vials and analyzed using a Thermo GasBench II connected to a Thermo Delta Advantage mass spectrometer in continuous flow mode. Analytical uncertainties were better than 0.1 ‰ (1σ) for $\delta^{18}O$ and 0.05 ‰ (1σ) for $\delta^{13}C$. All values were calibrated against international standards and reported in permil (‰) relative to Vienna Pee Dee belemnite (VPDB).

Eleven samples of bedrock, along with separate size fractions of samples YS-1 through YS-6, CP, and YP were analyzed in the Institute for Geology at the University of Innsbruck. Measurements were made on a Thermo Scientific Delta V Plus isotope ratio mass spectrometer (IRMS) connected to a GasBench II[1]. This system produces a typical precision (1σ) of ±0.06 ‰ for $\delta^{13}C$ and ±0.08 ‰ for $\delta^{18}O$[2]. All samples were run in duplicate.

$^{230}Th/^{234}U$ Disequilibrium Dating

Five samples (YS-5, YS-6, CP, YP, and TF) were analyzed using U-Th radiometric dating techniques in the Department of Earth, Atmospheric, and Planetary Sciences at the Massachusetts Institute of Technology. Two samples were divided into three replicates (YS-5 A, B, C and YS-6 A, B, C) to assess reliability of the dating for young samples with a high potential for Th contamination. One replicate for each sample did not produce a result due to a low yield in chemistry. Approximately 0.03 g of each sample was combined with a ^{229}Th-^{233}U-^{236}U tracer, then digested and purified via iron coprecipitation and ion exchange chromatography. U and Th were analyzed on separate aliquots using a Nu Plasma II-ES multi-collector ICP-MS equipped with a CETAC Aridus II desolvating nebulizer following previously published protocols[3]. U-Th ages were calculated using standard decay constants for ^{230}Th, ^{234}U, and ^{238}U[4–6]. Ages were recalculated under the assumption that the TF sample is modern, giving the sample a relative age of -61 years BP (where present is defined as AD 1950). Reported errors for ^{238}U and ^{232}Th concentrations are estimated to be ±1% due to uncertainties in spike concentration, while analytical errors are smaller. Variability of initial $^{230}Th/^{232}Th$ throughout the cave is assumed to be ±25% at 2σ.

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Table S1. CCC Samples Collected from Winter Wonderland Cave

Sample Name	Image	Location	Size Fractions	CCC type and mineralogy
YS-1		On ice surface	250-165 µm, 165-75 µm, <75 µm	CCC_{coarse} Calcite
YS-2		On ice surface	250-165 µm, 165-75 µm, <75 µm	CCC_{coarse} Calcite
YS-3		On ledge above ice surface	250-165 µm, 165-75 µm, <75 µm	CCC_{fine} Calcite, Quartz
YS-4		On ledge above ice surface	250-165 µm, 165-75 µm, <75 µm	CCC_{fine} Calcite, Quartz
YS-5		In moat at base of wall	250-165 µm, 165-75 µm, <75 µm	CCC_{coarse} Calcite

YS-6		From ice surface	250-165 µm, 165-75 µm, <75 µm	CCC_{coarse} Calcite
CP		From clear pool of water on ice surface	250-165 µm, 165-75 µm, <75 µm	CCC_{coarse} Calcite
YP		From yellow pool of water on ice surface	250-165 µm, 165-75 µm, <75 µm	CCC_{coarse} Calcite
TF		From surface of water amidst polygonal crystals	No size fractions, limited sample	CCC_{coarse} Calcite
BR		Collected *in situ* from near top of ice exposure	No size fractions, limited sample	CCC_{coarse} Calcite

Table S2. Isotope Results from Winter Wonderland Cave

Sample	Size fraction	Lab	$\delta^{18}O$	$\delta^{13}C$	Note
YS-1	Bulk	Union	-18.00	2.87	CCC_{coarse}
YS-2	Bulk	Union	-16.38	2.00	CCC_{coarse}
YS-3	Bulk	Union	-7.55	4.68	CCC_{fine}
YS-4	Bulk	Union	-7.09	7.28	CCC_{fine}
YS-5	Bulk	Union	-14.75	1.61	CCC_{coarse}
YS-6	Bulk	Union	-20.25	4.27	CCC_{coarse}
CP	Bulk	Union	-16.57	5.90	CCC_{coarse}
YP	Bulk	Union	-16.16	2.27	CCC_{coarse}
TF-1	Bulk	Union	-12.12	4.92	CCC_{coarse}
TF-2	Bulk	Union	-12.83	4.84	CCC_{coarse}
BF - 1a	Bulk	Innsbruck	-14.71	2.33	CCC_{coarse}
BF - 1b	Bulk	Innsbruck	-15.06	2.16	CCC_{coarse}
YS - 1 - 2a	165_250	Innsbruck	-12.86	6.03	More CCC_{coarse} in finer fractions
YS - 1 - 2b	165_250	Innsbruck	-13.03	6.20	
YS - 1 - 3a	75_165	Innsbruck	-14.90	4.67	
YS - 1 - 3b	75_165	Innsbruck	-14.89	4.61	
YS - 1 - 4a	<75	Innsbruck	-17.72	2.79	
YS - 1 - 4b	<75	Innsbruck	-17.63	2.80	
YS - 2 - 2a	165_250	Innsbruck	-9.20	3.65	More CCC_{coarse} in finer fractions
YS - 2 - 2b	165_250	Innsbruck	-9.39	3.59	
YS - 2 - 3a	75_165	Innsbruck	-14.43	3.05	
YS - 2 - 3b	75_165	Innsbruck	-14.52	3.05	
YS - 2 - 4a	<75	Innsbruck	-16.18	1.66	
YS - 2 - 4b	<75	Innsbruck	-16.25	1.62	
YS - 3	>250	Innsbruck	-9.48	6.76	CCC_{fine}
YS - 3 - 2a	165_250	Innsbruck	-14.75	2.17	
YS - 3 - 2b	165_250	Innsbruck	-14.60	2.15	
YS - 3 - 3a	75_165	Innsbruck	-8.83	2.69	
YS - 3 - 3b	75_165	Innsbruck	--	--	
YS - 3 - 4a	<75	Innsbruck	-7.46	4.30	
YS - 3 - 4b	<75	Innsbruck	-7.27	4.45	
YS-4	>250	Innsbruck	-5.05	5.95	CCC_{fine}
YS - 4 - 2a	165_250	Innsbruck	-7.78	5.27	
YS - 4 - 2b	165_250	Innsbruck	-7.84	5.30	
YS - 4 - 3a	75_165	Innsbruck	-8.12	5.34	
YS - 4 - 3b	75_165	Innsbruck	-8.27	5.1	

YS - 4 - 4a	<75	Innsbruck	-7.21	6.96	
YS - 4 - 4b	<75	Innsbruck	-7.17	6.95	
YS - 5 - 2a	165_250	Innsbruck	-15.61	2.01	all CCC$_{coarse}$
YS - 5 - 2b	165_250	Innsbruck	-15.59	2.06	
YS - 5 - 3a	75_165	Innsbruck	-14.57	1.82	
YS - 5 - 3b	75_165	Innsbruck	-14.53	1.80	
YS - 5 - 4a	<75	Innsbruck	-14.37	1.33	
YS - 5 - 4b	<75	Innsbruck	-14.22	1.34	
CP - 2a	165_250	Innsbruck	-16.05	5.51	all CCC$_{coarse}$
CP - 2b	165_250	Innsbruck	-16.20	5.59	
CP - 3a	75_165	Innsbruck	-15.99	5.21	
CP - 3b	75_165	Innsbruck	-16.01	5.28	
CP - 4a	<75	Innsbruck	-15.52	4.77	
CP - 4b	<75	Innsbruck	-15.66	4.72	
YP - 2a	165_250	Innsbruck	-15.90	1.62	all CCC$_{coarse}$
YP - 2b	165_250	Innsbruck	-15.65	1.62	
YP - 3a	75_165	Innsbruck	-16.09	1.91	
YP - 3b	75_165	Innsbruck	-16.17	1.79	
YP - 4a	<75	Innsbruck	-16.11	1.46	
YP - 4b	<75	Innsbruck	-16.32	1.34	
BF - 1a	Bulk	Innsbruck	-14.71	2.33	
BF - 1b	Bulk	Innsbruck	-15.06	2.16	
TF - 1a	Bulk	Innsbruck	-12.54	4.82	
TF - 1b	Bulk	Innsbruck	-12.54	4.82	
YS - 6 - 2a	165_250	Innsbruck	-18.60	4.31	all CCC$_{coarse}$
YS - 6 - 2b	165_250	Innsbruck	-19.02	4.35	
YS - 6 - 3a	75_165	Innsbruck	-19.28	4.22	
YS - 6 - 3b	75_165	Innsbruck	-19.35	4.39	
YS - 6 - 4a	<75	Innsbruck	-18.29	4.12	
YS - 6 - 4b	<75	Innsbruck	-18.39	4.14	
WW - 1a	Rock	Innsbruck	-5.34	2.64	
WW - 1b	Rock	Innsbruck	-5.19	2.73	
SR-1	Rock	Innsbruck	-4.45	3.26	
SR-2	Rock	Innsbruck	-4.09	3.28	
SR-3	Rock	Innsbruck	-4.34	3.24	
CoD-1	Rock	Innsbruck	-13.37	-3.26	
CoD-2	Rock	Innsbruck	-8.20	0.66	Dolomite
CoD-3	Rock	Innsbruck	-3.55	3.04	Dolomite
FF-1	Rock	Innsbruck	-5.83	2.35	
FF-3-1	Rock	Innsbruck	-4.45	3.06	
By-6	Rock	Innsbruck	-4.04	4.08	
Alcove	Rock	Innsbruck	-6.21	2.29	

Table S3. Major Element Abundance in Samples from Winter Wonderland Cave*

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	Fe_2O_3	Total
Bedrock	0.47	2.12	1.07	4.83	-0.09	0.08	89.42	0.06	0.02	0.52	98.51
YS-4 75-250 µm	0.05	1.84	4.88	52.19	0.11	1.11	39.12	0.22	0.14	1.76	101.41
YS-6 75-250 µm	0.45	12.16	2.03	9.55	0.85	0.86	70.70	0.07	0.02	0.56	97.25

*Presented as weight percent oxide

Table S4. Chondrite-Normalized REE Abundances for Samples from Winter Wonderland Cave

Sample	La	Ce	Nd	Sm	Eu	Gd	Dy	Er	Yb	Lu
YS-3 75 µm	69.9	67.4	28.4	15.6	6.9	10.4	9.0	9.0	10.1	10.3
YS-4 75 µm	50.6	49.8	20.8	11.6	5.5	7.8	6.4	6.3	6.9	6.7
YS-5 75 µm	12.6	8.4	4.4	1.9	0.6	1.1	0.9	1.0	1.1	1.0
YS-6 75 µm	13.4	9.1	4.6	1.9	0.5	1.0	0.7	0.9	1.0	1.1
WW rock	5.6	3.4	2.9	1.8	1.0	1.6	1.4	1.3	1.2	1.0
YS-4 75-250 µm	34.7	35.8	14.4	8.2	3.9	5.6	4.6	4.4	4.8	4.7

Table S5. U-Th Dating Results for CCC Samples from Winter Wonderland Cave

Sample ID	^{238}U (ng/g)[a]	± (2σ)	^{232}Th (pg/g)[a]	± (2σ)	$\delta^{234}U$ (per mil)[b]	± (2σ)	($^{230}Th/^{238}U$) activity	± (2σ)	$^{230}Th/^{232}Th$ ppm atomic	± (2σ)	Age (yr) (uncorrected)[c]	± (2σ)	Age (yr) (corrected)[d]	± (2σ)	$\delta^{234}U$ initial (per mil)[e]	± (2σ)	Age (yr BP) (corrected)[f]	± (2σ)
KK-5A	577	12	346497	6930	995	2.9	0.4	0.012	9.3	0.3	20780	752	5829	4129	1012	12.2	5760	4129
KK-5C	592	12	412255	8247	959	1.7	0.4	0.014	9.2	0.3	24643	924	6780	5019	977	14.0	6711	5019
KK-6A	435	9	467240	9349	407	2.0	0.5	0.021	8.0	0.3	51244	2497	8622	13716	418	16.4	8553	13716
KK-6B	448	9	470609	9414	443	1.9	0.5	0.021	7.9	0.3	47886	2312	7681	12766	453	16.6	7612	12766
KK-CP	760	15	263625	5281	1309	1.3	0.2	0.007	8.2	0.3	8744	341	1451	1929	1314	7.3	1382	1929
KK-YP	530	11	249332	4994	1516	1.7	0.3	0.009	9.0	0.3	12114	439	3016	2434	1529	10.7	2947	2434
KK-TF	164	3	42729	856	836	2.1	0.1	0.005	6.9	0.3	6889	309	8	1811	836	4.8	-61	1811

Notes:

Samples KK-5A and KK6C had a low yield in chemistry and could not be dated

[a] Reported errors for ^{238}U and ^{232}Th concentrations are estimated to be ±1% due to uncertainties in spike concentration; analytical uncertainties are smaller.

[b] $d^{234}U = ([^{234}U/^{238}U]_{activity} - 1) \times 1000$.

[c] $[^{230}Th/^{238}U]_{activity} = 1 - e^{-l_{230}T} + (d^{234}U_{measured}/1000)[l_{230}/(l_{230} - l_{234})](1 - e^{-(l_{230} - l_{234})T})$, where T is the age. "Uncorrected" indicates that no correction has been made for initial ^{230}Th.

[d] Ages are corrected for detrital ^{230}Th assuming an initial $^{230}Th/^{232}Th$ of $(6.89 \pm 1.72) \times 10^{-6}$, based on the $^{230}Th/^{232}Th$ of sample KK-TF and an assumed ±25% 2-sigma uncertainty.

[e] $d^{234}U_{initial}$ corrected was calculated based on ^{230}Th age (T), i.e., $d^{234}U_{initial} = d^{234}U_{measured} X e^{l234*T}$, and T is corrected age.

[f] B.P. stands for “Before Present” where the “Present” is defined as the January 1, 1950 C.E.

158 Decay constants for ^{230}Th and ^{234}U are from Cheng et al. (2013); decay constant for ^{238}U is 1.55125×10^{-10} yr^{-1} (Jaffey et al., 1971).

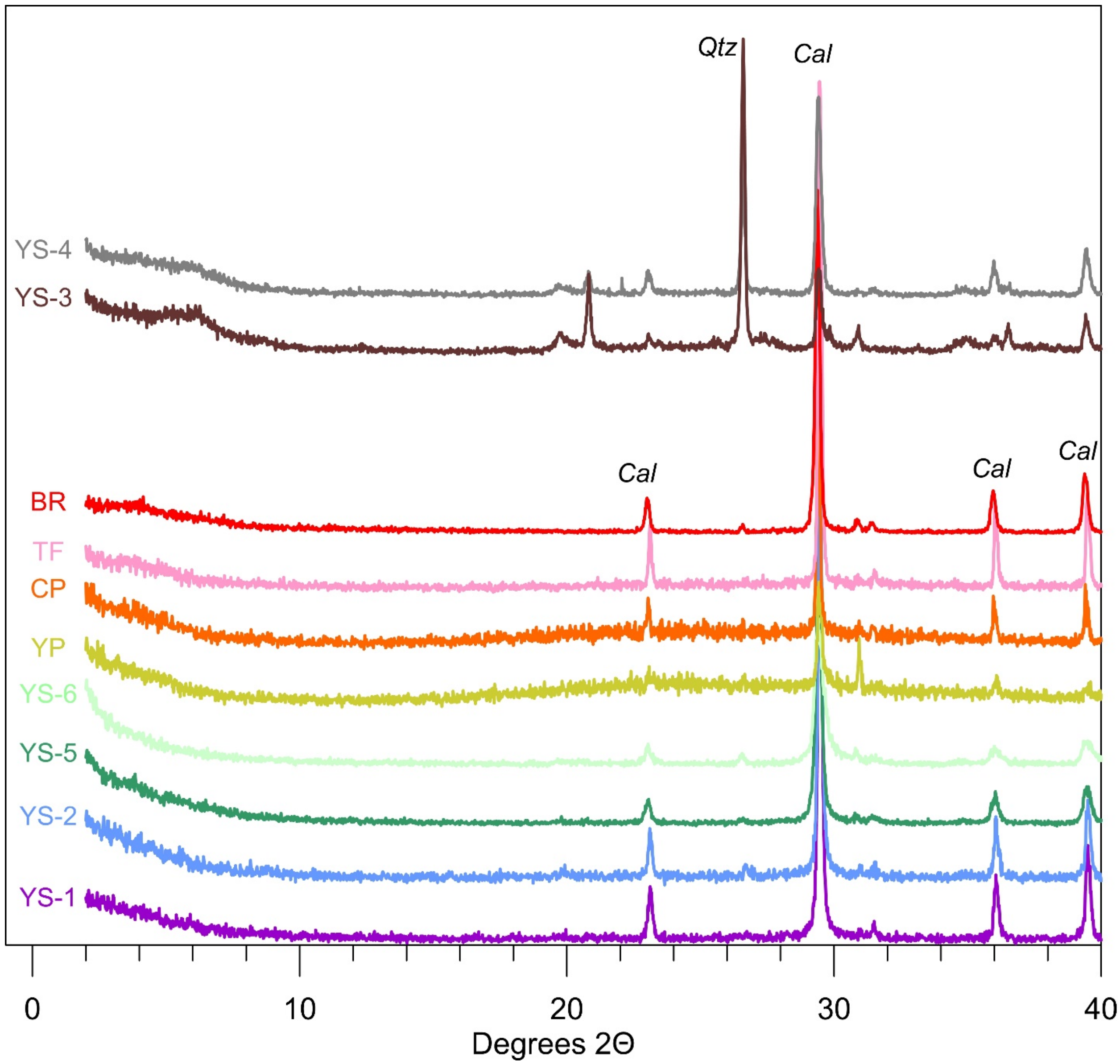

Figure S1: XRD patterns for samples from Winter Wonderland Cave. Samples YS-1 through YS-6, YP (Yellow Pool), CP (Clear Pool), and TF (Thin Film) are all CCCcoarse, dominated by calcite. The local bedrock (BR) also has a calcite-dominated mineralogy. In contrast, samples YS-3 and YS-4 are CCC_{fine}, characterized by a mixture of calcite and quartz.

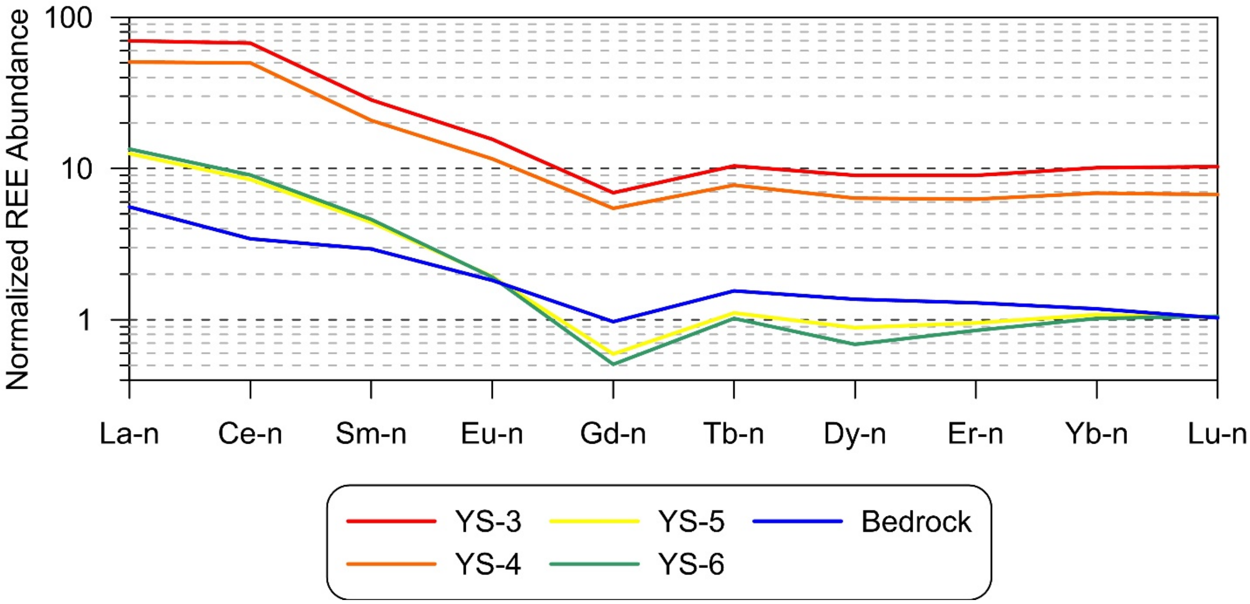

Figure S2: Rare earth element (REE) patterns for samples from Winter Wonderland Cave, normalized to a chondrite standard. REEs are notably more abundant in the two CCC_{fine} samples (YS-3 and YS-4), consistent with a greater detrital component.

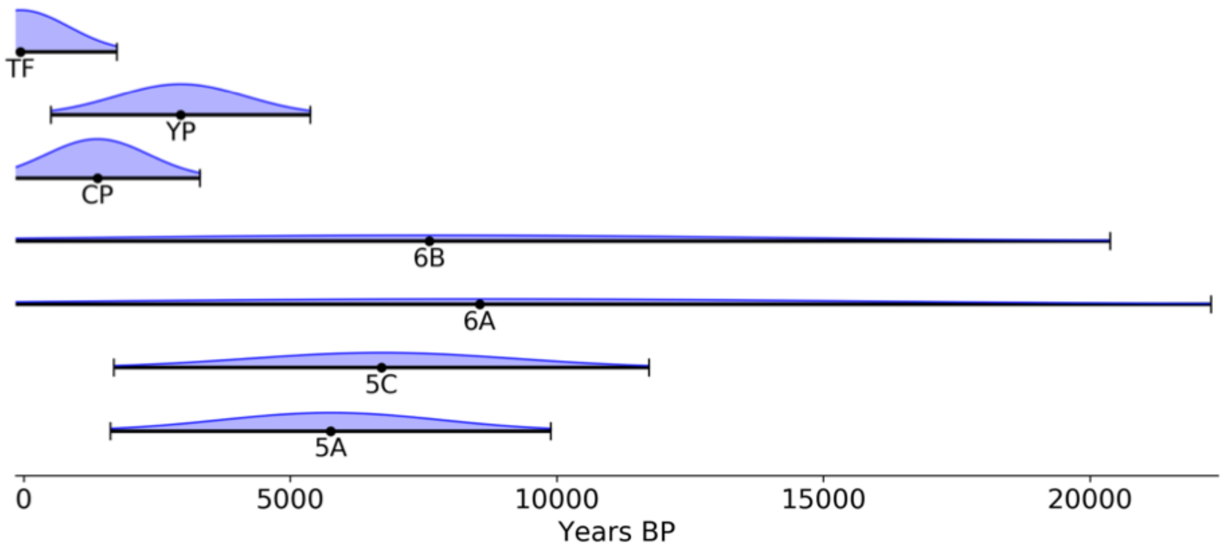

Figure S3. Results of U-Th dating of CCCs from Winter Wonderland Cave. The TF sample formed between 2016 and 2018, so the initial $^{230}Th/^{232}Th$ for this sample (6.89×10^{-6}) was used for all of the samples. 5A and 5C are replicates for sample YS-5. 6A and 6B are replicates for samples YS-6. Wide error bars are a product of a large correction for detrital ^{230}Th. Nonetheless, all of the dated samples likely formed during the Holocene, and ages for CP and YS-6 overlap with modern.

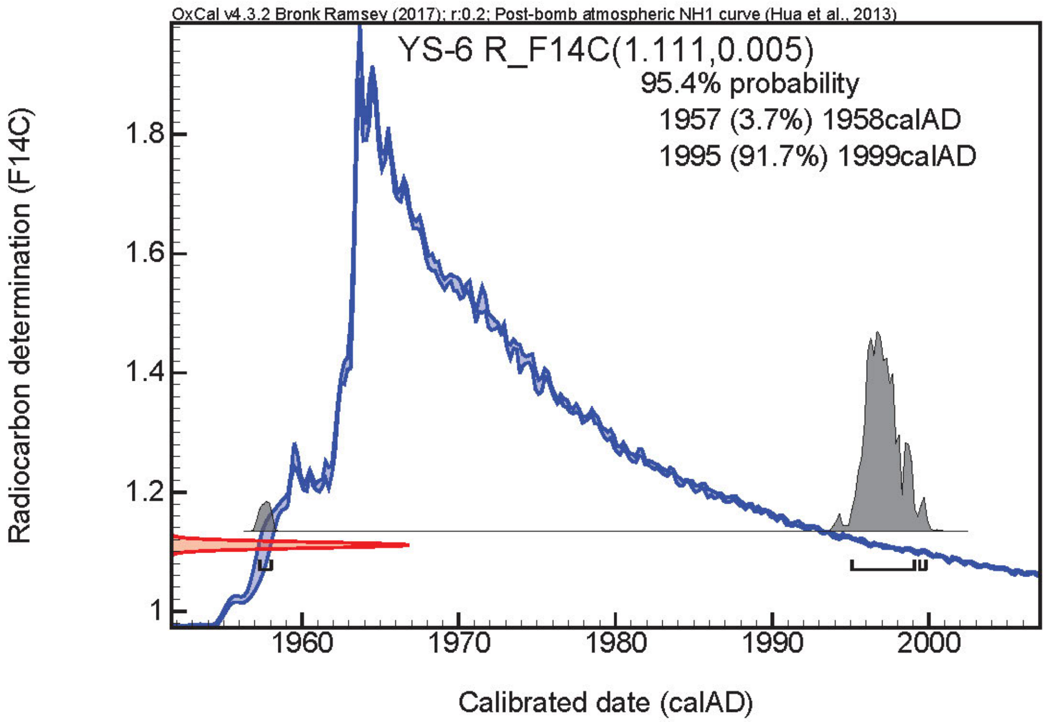

Figure S4. Results of radiocarbon dating of CCC_{coarse} sample YS-6. The sample contained excess ^{14}C indicating formation after the peak of atmospheric bomb testing. Calibrated with the NH1 bomb curve, the sample likely formed between AD 1995 and 1999, which overlaps with the distal young end of the error range on the U-Th analysis for this sample (Fig. S3).

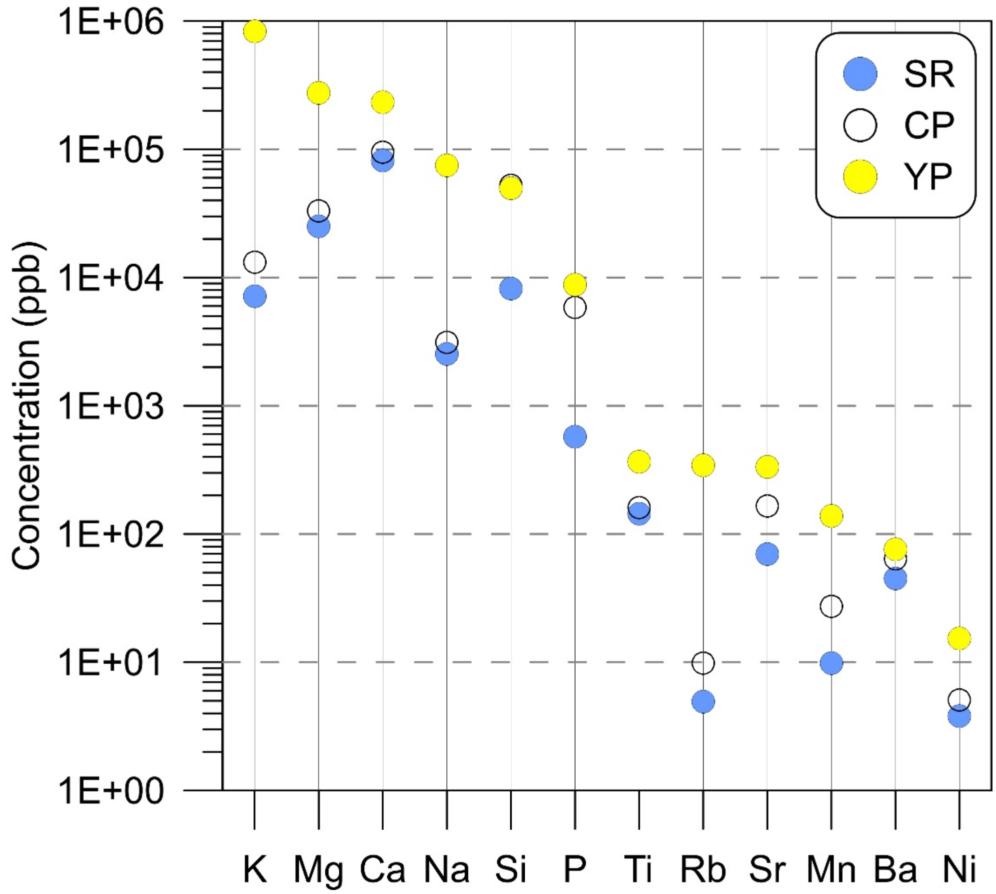

Figure S5. Abundance of consistently detectable elements in water samples from Winter Wonderland Cave. Sample SR was collected from a pool with a lid of ice in 2019 (Fig. 2b). Samples YP and CP were collected from pools on the ice surface in 2018 (Table S1). Concentration of all solutes is much higher in the YP (Yellow Pool) sample.