

U-Pb dating of calcite veins reveals complex stress evolution and thrust sequence in the Bighorn Basin, USA.

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Supplementary Information

U-Pb Geochronology:

U-Pb geochronology via the in-situ Laser Ablation Inductively Coupled Mass Spectrometer (LA-ICP-MS) method was conducted at the Geochronology & Tracers Facility, NERC Isotope Geosciences Laboratory (Nottingham, UK). The method utilises a New Wave Research 193UC excimer laser ablation system, coupled to a Nu Instruments Attom single-collector sector-field ICP-MS. The method for calcite (Li et al., 2014; Coogan et al., 2016; Roberts & Walker, 2016; Roberts et al., 2017) is adapted from that used for zircon (see Spencer et al., 2014), with some modifications.

The laser parameters used are a 100µm static spot, fired at 10Hz, with a ~7-8 j.cm² fluence, for 30 seconds of ablation. Material is pre-ablated to clean the sample site with 150µm spots for 3 seconds. Normalisation uses standard sample bracketing to NIST 614 glass (Woodhead & Herdt, 2007) for Pb-Pb ratios, and a carbonate reference material developed in-house for ²⁰⁶Pb/²³⁸U ratios (WC-1; Roberts et al., 2017). Normalisation is based on the measured/accepted ratio derived from the session-based drift-corrected mean of the primary WC-1 reference material. No common lead correction is made; those ages that are deemed robust (based on low MSWD), are determined from lower intercepts on a Tera-Wasserburg plot. All regressions are unanchored as the spread in data permits assessment of the upper intercept accurately. Age results are based in lower intercept ages with systematic uncertainties (decay constants, reference material age uncertainty, long-term method reproducibility) propagated. Resulting Tera-Wasserburg plots (semi-total Pb plots)

are shown below (Figure S2a-e), and have uncertainties quoted as age \pm x/y, where x is without systematic uncertainties and y is with (see Horstwood et al., 2016).

The reproducibility of the primary WC-1 reference material is around 2-4% per session. An estimate of the session reproducibility is propagated (as excess variance) onto the sample data. The ages quoted have additional systematic uncertainties propagated onto the final age, these include decay constant uncertainties, the laboratory-based long-term reproducibility of the method (~2%) and the uncertainty on the reference material age (~2.75%; based on in-house isotope dilution measurements).

An additional carbonate material, Duff Brown, was measured in most sessions to provide a control on accuracy and precision. An isotope dilution age of 64.04 ± 0.67 Ma is provided by Hill et al. (2014). In each session the resulting age of Duff Brown was within uncertainty of this value (Figure S1C). The pooled data from all sessions yield an age of 63.25 ± 0.86 Ma (2s without systematic uncertainties propagated; Figure S1D).

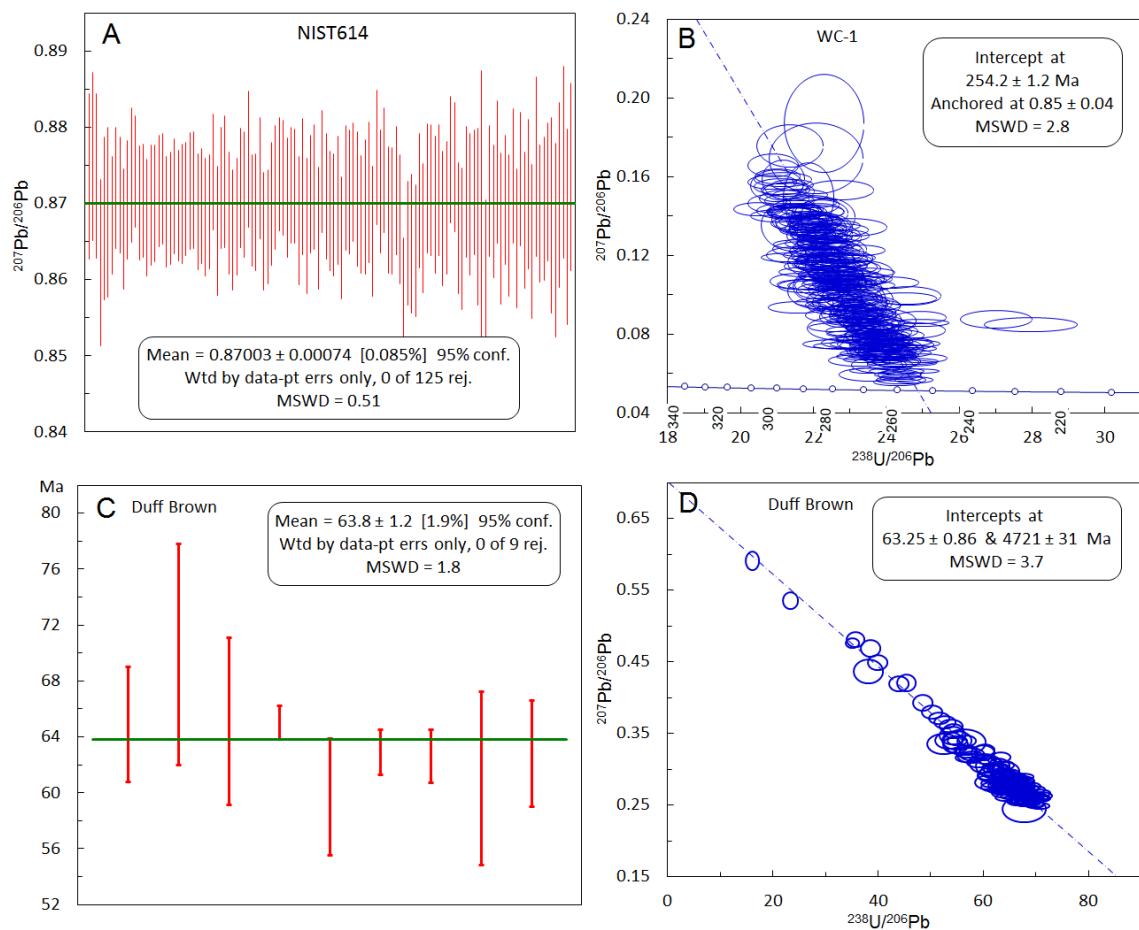


Figure S1. A) Pooled NIST614 $^{207}\text{Pb}/^{206}\text{Pb}$ measurements for all sessions. B) Pooled WC-1 measurements (Tera-Wasserburg plot) for all sessions. C) Lower intercept (Tera-Wasserburg) age for Duff Brown for nine sessions. D) Pooled Tera-Wasserburg results for Duff-Brown (the same nine sessions).

Analytical Conditions:

Laboratory & Sample Preparation	
Laboratory name	NERC Isotope Geosciences Laboratory
Sample type/mineral	Calcite
Sample preparation	In-situ in polished block
Imaging	None
Laser ablation system	
Make, Model & type	ESI/New Wave Research, 193UC
Ablation cell & volume	NWR TV2
Laser wavelength (nm)	193nm
Pulse width (ns)	3-4ns
Fluence (J.cm ⁻²)	~7-8 J.cm ⁻²
Repetition rate (Hz)	10Hz
Ablation duration (secs)	30secs
Ablation pit depth / ablation rate	~45µm pit depth, measured using an optical microscope
Spot size (µm)	100µm
Sampling mode / pattern	Static spot ablation
Carrier gas	100% He, Ar make-up gas combined ca.50% along sample line.
Cell carrier gas flow (l/min)	0.6l/min
ICP-MS Instrument	
Make, Model & type	Nu Instruments Attom SC-SF-ICP-MS
Sample introduction	Free air aspiration of desolvator
RF power (W)	1300W
Make-up gas flow (l/min)	0.8l/min Ar
Detection system	Discrete dynode MassCom ion counter
Masses measured	202, 204, 206, 207, 208, 232, 238
Integration time per peak	200µs (202, 204, 208, 232), 400µs (206), 1000µs (207, 238) 80 sweeps per integration
Total integration time per reading (secs)	0.30 seconds
Sensitivity / Efficiency (% , element)	~0.2 % for Uranium
IC Dead time (ns)	15ns
Data Processing	
Gas blank	60 second on-peak zero subtracted
Calibration strategy	NIST614 for Pb-Pb, WC-1 for Pb-U
Reference Material info	Primary: WC-1 254 +/- 6 Ma (2s) - Roberts et al., 2017 Secondary: Duff Brown 64.04 +/- 0.67 Ma (2s) - Hill et al., 2016
Data processing package used / Correction for LIEF	Nu Instruments TRA acquisition software, in-house spreadsheet data processing

Mass discrimination	$^{207}\text{Pb}/^{206}\text{Pb}$, $^{206}\text{Pb}/^{238}\text{U}$ normalised to reference materials
Common-Pb correction, composition and uncertainty	Unanchored (model 1) regressions in Tera-Wasserburg (Semi Total-Pb) plots.
Uncertainty level & propagation	Ages in the data table are quoted at 2s absolute and include systematic uncertainties, propagation is by quadratic addition.
Quality control / Validation	Duff Brown over the course of the analytical sessions gave 63.25 ± 0.86 Ma (without propagation of systematic uncertainties)
Other information	

Results:

Resulting ages and upper intercepts are quoted in addition to the plots below in Table DR1.

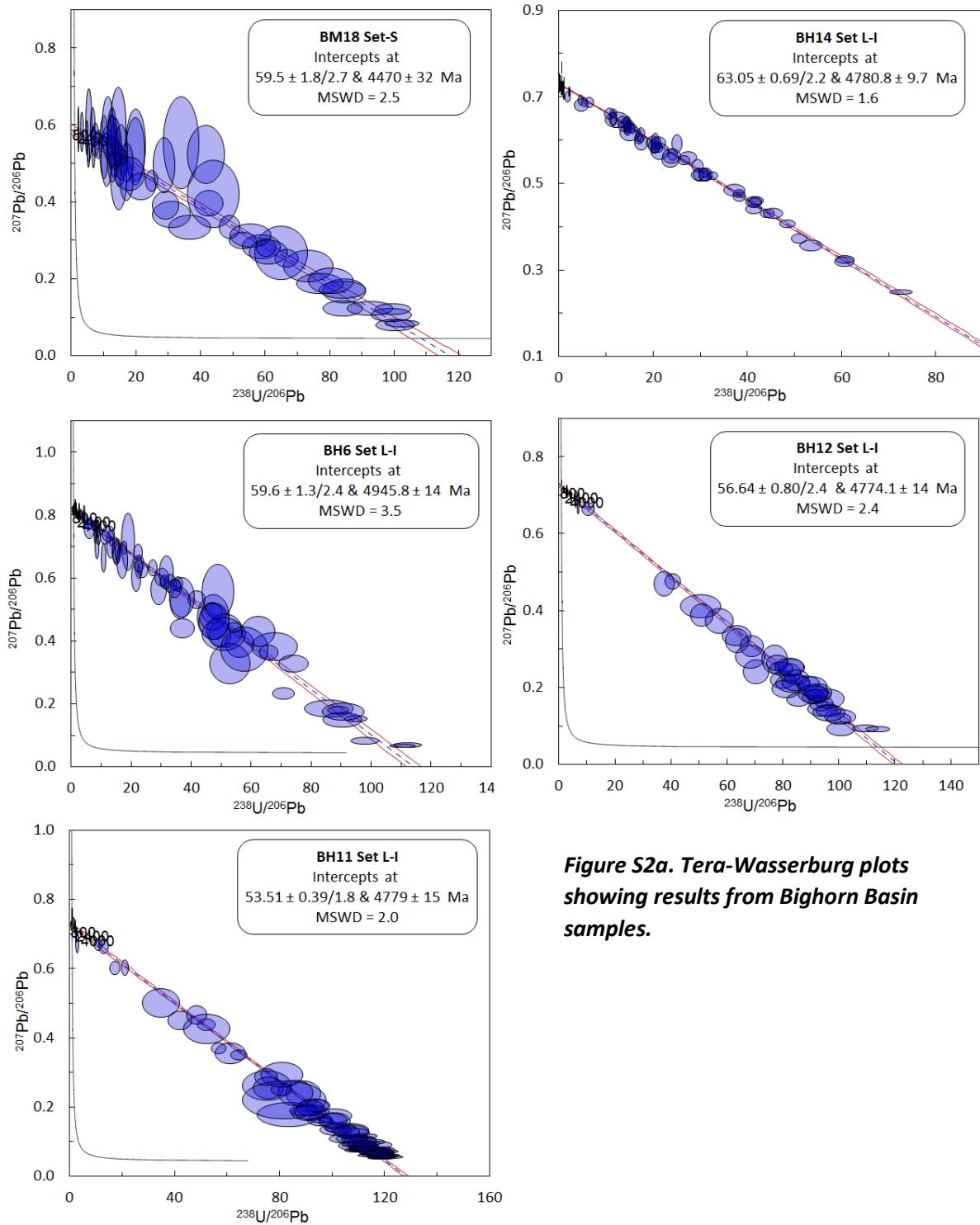


Figure S2a. Tera-Wasserburg plots showing results from Bighorn Basin samples.

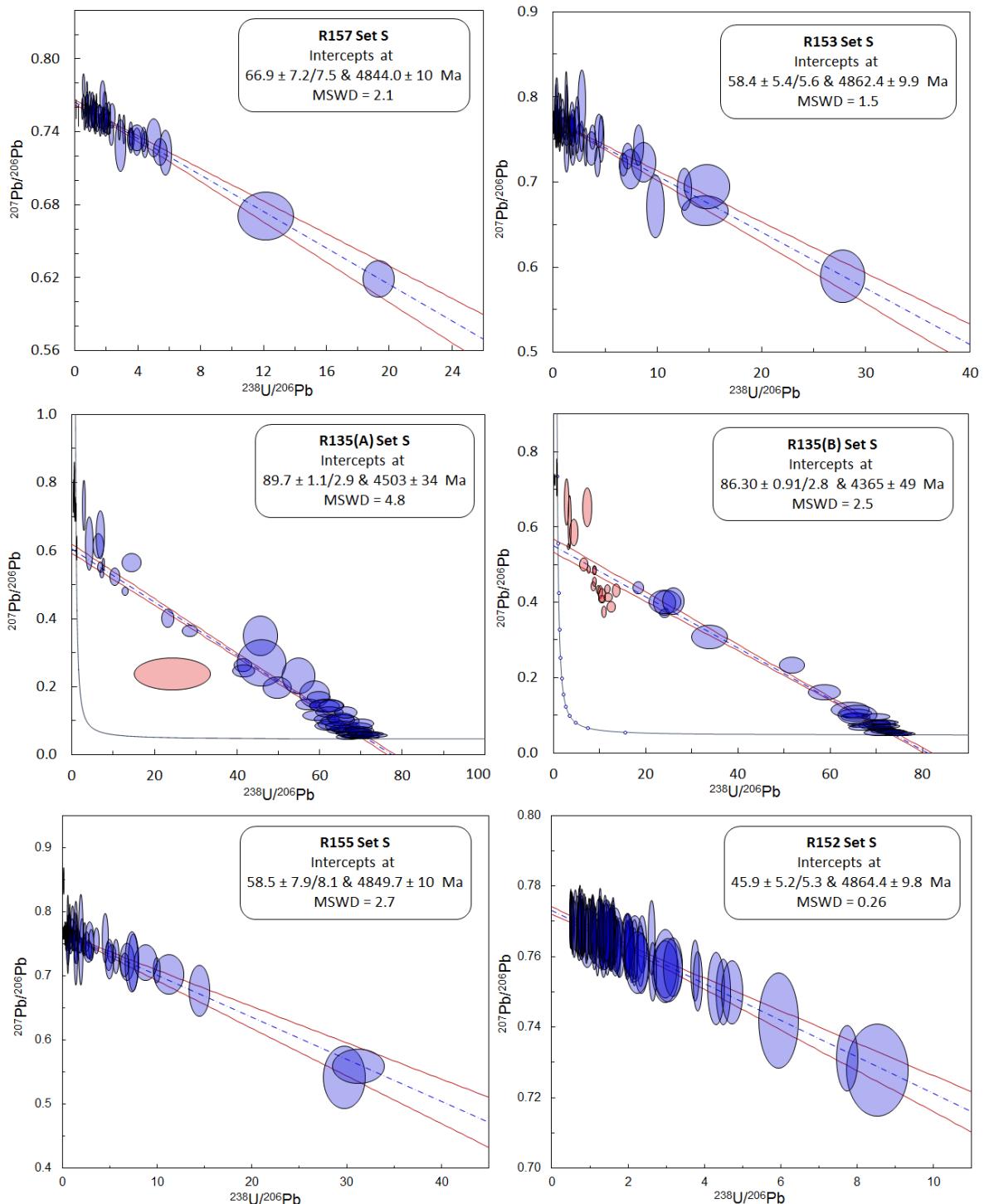


Figure S2b. Tera-Wasserburg plots showing results from Rattlesnake samples.

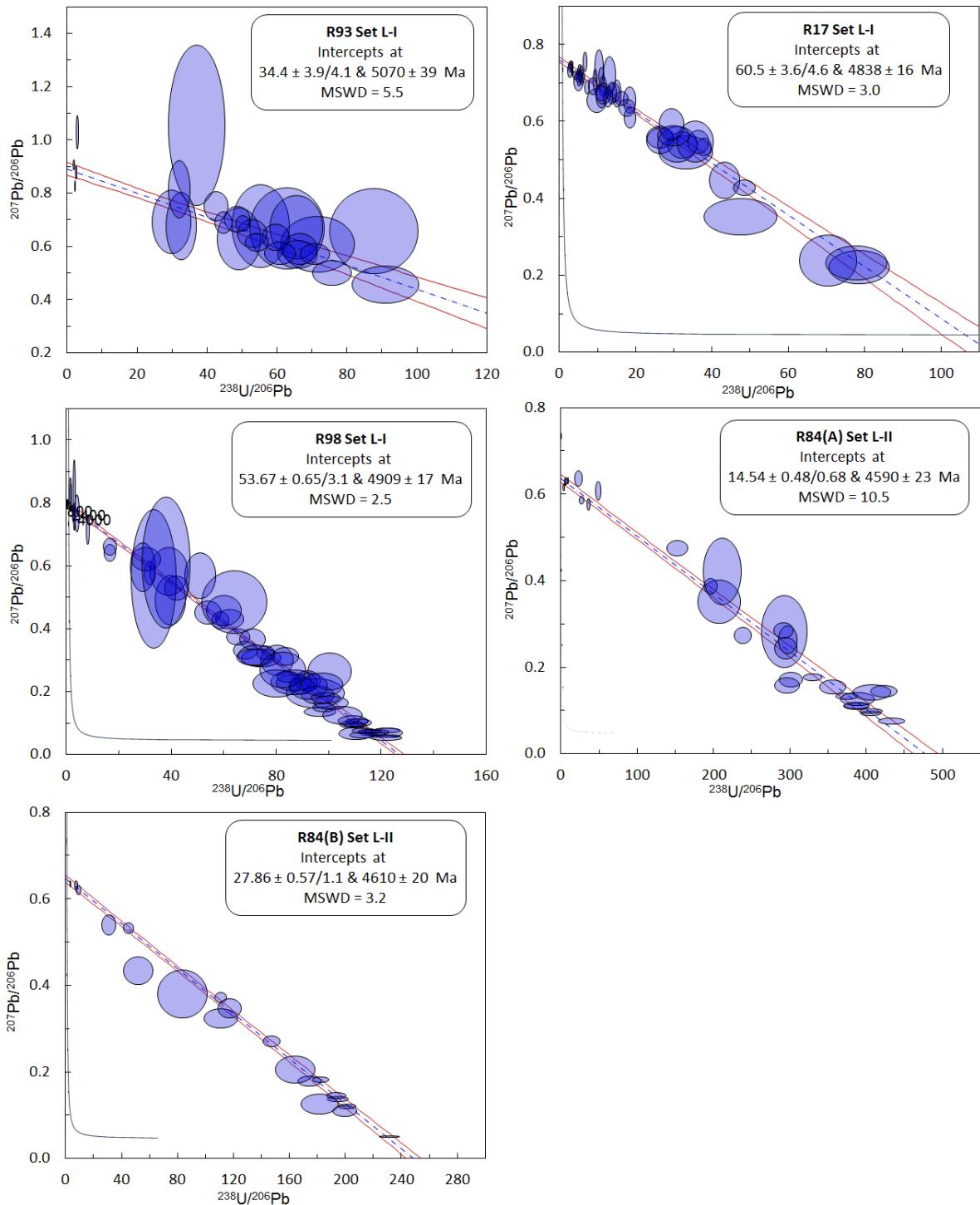


Figure S2c. Tera-Wasserburg plots showing results from Rattlesnake samples (continued).

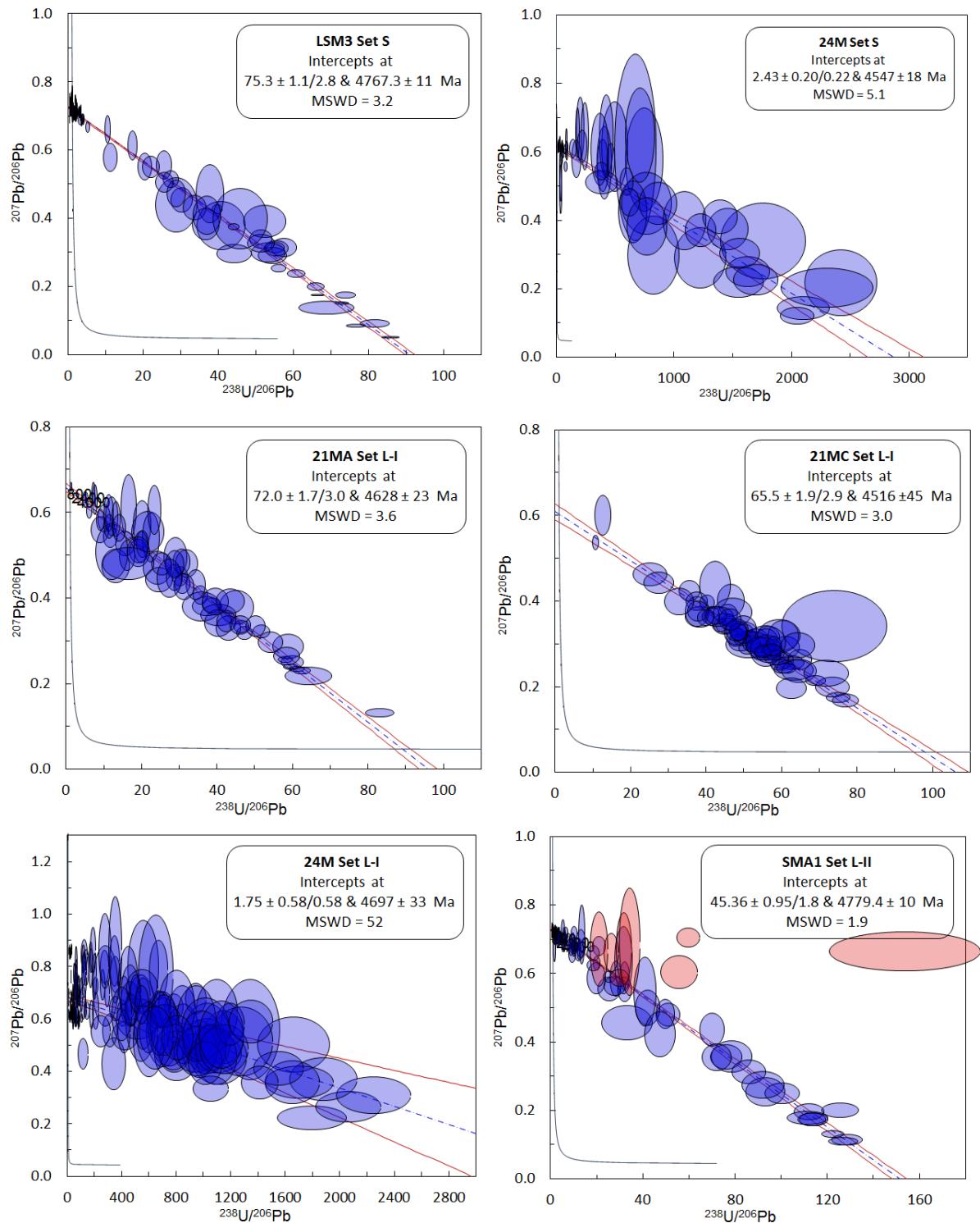


Figure S2d. Tera-Wasserburg plots showing results from Little Sheep Mountain and Sheep Mountain Anticlines.

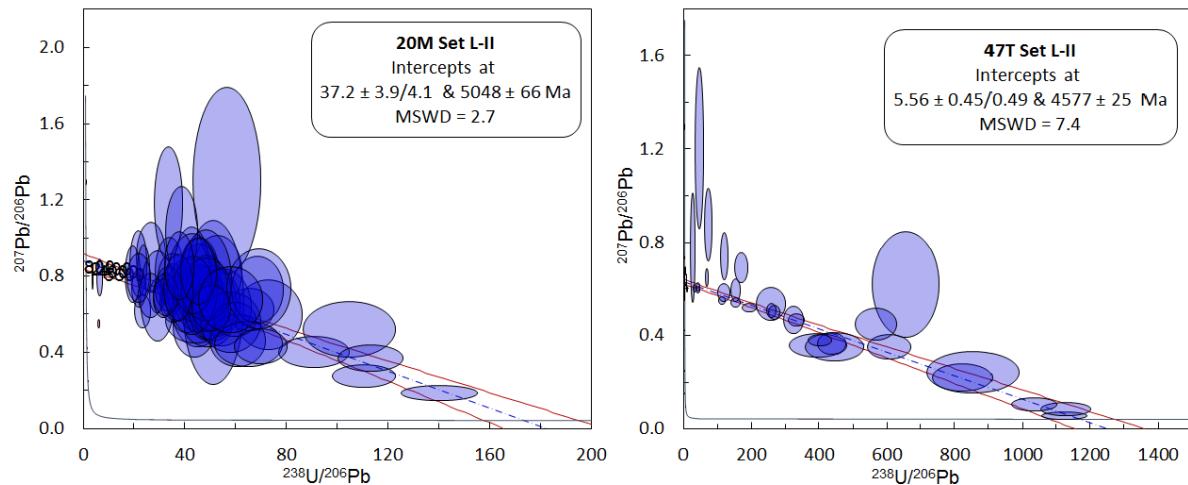


Figure S2e. Tera-Wasserburg plots showing results from Little Sheep Mountain and Sheep Mountain Anticlines (continued).

Sample Images:

Sample name	Set	Sample
Bighorn Mountain		
BM18	S	 BM18
BH14	L-I	 BH14

BH6

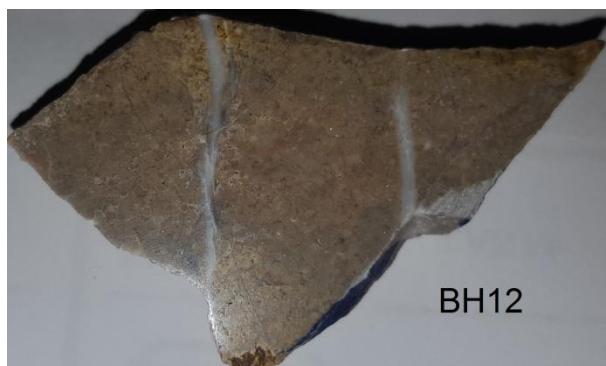
L-I



BH6

BH12

L-I



BH12

BH11

L-I



Rattlesnake Mountain

R157

S



R153

S



R135A

S



R135B

S



R155

S



R152

S



R93

L-I



R17

L-I



R98

L-I



R84(B)

L-II



R84(a)

L-II

as above

**Little Sheep Mountain and
Sheep Mountain
Anticlines**

LSM3

S



24M

S



21M A

L-I

21MA



21M C

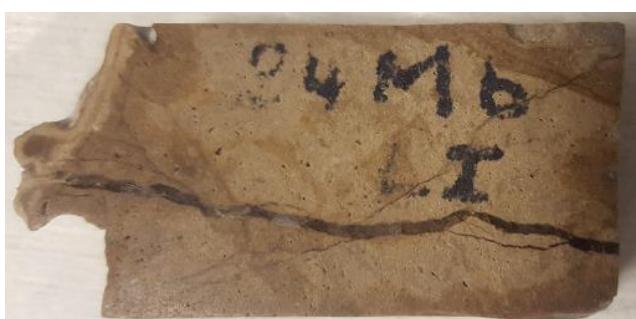
L-I



24M B

L-I

24M b
I

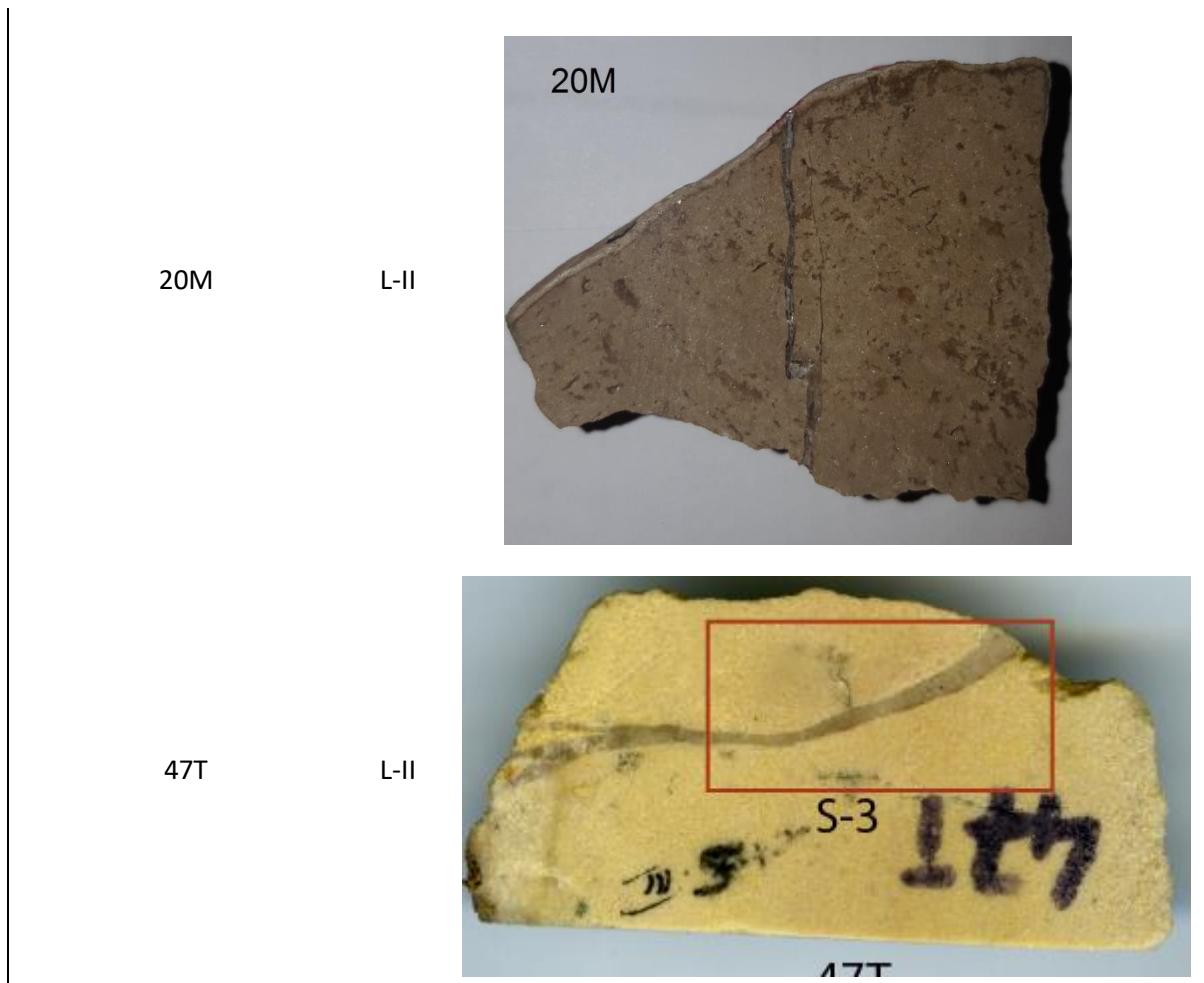


SMA1

L-II

L-II
SMA1





References:

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- Hill, C.A., Polyak, V.J., Asmerom, Y. and P. Provencio, P., 2016, Constraints on a Late Cretaceous uplift, denudation, and incision of the Grand Canyon region, southwestern Colorado Plateau, USA, from U-Pb dating of lacustrine limestone: *Tectonics*, v. 35, p. 896-906.
- Li, Q., Parrish, R.R., Horstwood, M.S.A. and McArthur, J.M., 2014, U-Pb dating of cements in Mesozoic ammonites: *Chemical Geology*, v. 376, p. 76-83.
- Roberts, N.M., Rasbury, E.T., Parrish, R.R., Smith, C.J., Horstwood, M.S. and Condon, D.J., 2017, A calcite reference material for LA-ICP-MS U-Pb geochronology: *Geochemistry, Geophysics, Geosystems*, v. 18, p. 2807-2814.

Roberts, N.M., and Walker, R., J., 2016, U-Pb geochronology of calcite mineralized faults; absolute dating of rift-related fault events on the northeast Atlantic margin: *Geology*, v. 44, p. 531-534.

Spencer, C.J., Roberts, N.M., Cawood, P.A., Hawkesworth, C.J., Prave, A.R., Antonini, A.S. and Horstwood, M.S., 2014, Intermontane basins and bimodal volcanism at the onset of the Sveconorwegian Orogeny, southern Norway: *Precambrian Research*, v. 252, p. 107-118.

Woodhead, J.D. and Herdt, J.M., 2001, Strontium, neodymium and lead isotope analyses of NIST glass certified reference materials: SRM 610, 612, 614: *Geostandards Newsletter*, v. 25, p. 261-266.

TABLE DR1 LOCATION OF SAMPLES, ORIENTATION OF VEINS AND U/Pb RESULTS

Sample name	Location			Set	Vein orientation*		Formation	U/Pb Dating				
	GPS#	Lat (°N)	Long (°W)		Strike	Slip-dir		Age (Ma)	2s prop (Ma)	MSWD	Y-Int	
<i>Eastern part of the basin</i>												
Bighorn Mountain												
BM18	746	44.79347	107.9687	S	82	81N	Madison	59.5	2.7	2.5	0.590	0.012
BH14	673	44.57419	107.7002	L-I	55	81S	Phosphoria	63.1	2.2	1.6	0.731	0.001
BH6	673	44.57419	107.7002	L-I	42	81W	Phosphoria	59.6	2.4	3.5	0.820	0.006
BH12	673	44.57419	107.7002	L-I	51	89S	Phosphoria	56.6	2.1	2.4	0.727	0.005
BH11	673	44.57419	107.7002	L-I	64	82S	Phosphoria	53.5	1.8	2.0	0.729	0.006
Little Sheep Mountain and Sheep Mountain Anticlines												
LSM3	749	44.74589	108.1911	S	125	165	Madison	75.3	2.8	3.2	0.725	0.002
24M	17	44.60951	108.1404	S	116	80S	Madison	2.43	0.22	5.1	0.617	0.007
21M A	17	44.60587	108.1410	L-I	53	86S	Madison	72.0	3.0	3.6	0.658	0.010
21M C	17	44.60587	108.1410	L-I	53	86S	Madison	65.5	2.9	3.0	0.617	0.027
24M B	17	44.60951	108.1404	L-I	46	80S	Madison	1.75	0.58	52.0	0.684	0.015
SMA1	17	44.60587	108.1410	L-II	135	85N	Madison	45.4	1.8	1.9	0.729	0.002
20M	17	44.60587	108.1410	L-II	139	72N	Madison	37.2	4.1	2.7	0.878	0.041
47T	39	44.61411	108.1401	L-II	131	72S	Tensleep	5.56	0.49	7.4	0.643	0.014
Western part of the basin												
Rattlesnake Mountain												
R157	634	44.49737	109.1752	S	97	81S	Gros Ventre	66.9	7.5	2.1	0.764	0.002
R153	634	44.49737	109.1752	S	94	90N	Gros Ventre	58.4	5.6	1.5	0.773	0.001
R135A	625	44.52832	109.2113	S	131	58N	Madison	89.7	2.9	4.8	0.606	0.013
R135B	625	44.52832	109.2113	S	131	58N	Madison	86.3	2.8	2.5	0.551	0.018
R155	634	44.49737	109.1752	S	113	73S	Gros Ventre	58.5	8.1	2.7	0.766	0.002
R152	634	44.49737	109.1752	S	113	74S	Gros Ventre	45.9	5.3	0.3	0.773	0.001
R93	205	44.50137	109.1956	L-I	52	87S	Madison	34.4	4.1	5.5	0.892	0.024
R17	126	44.50237	109.1869	L-I	78	70N	Madison	60.5	4.6	3.0	0.761	0.007
R98	205	44.50142	109.1951	L-I	48	87N	Madison	53.7	3.1	2.5	0.799	0.008
R84(a)	145	44.50707	109.1159	L-II	148	83E	Phosphoria	14.5	0.7	10.5	0.636	0.009
R84(b)	145	44.50707	109.1159	L-II	148	83E	Phosphoria	27.9	1.1	3.2	0.646	0.008

* corrected from host bedding orientation

Table DR2: Details of the LA-ICP-MS analysis