

ANALYTICAL METHODS

Over 300 metasandstones, plus 78 pelites and 31 intrusive rocks were collected for this study. Calcite + quartz and wollastonite-bearing rocks were collected both close (<1 m) and far (>2000 m) from exposed pluton-wall rock contacts, and at various elevations. Detailed, multi-sample traverses of the wollastonite isograd were made at the meter to centimeter scale. Data from samples selected for detailed study are presented in Tables 2–5 and sample location maps are included with the Data Repository Item (Figs. DR1–6). Lackey (2000) reports latitude and longitude of samples.

Separating and concentrating minerals for stable isotope analysis required a variety of crushing, acid, and density separation techniques. Quartz grains were separated from sandstones by dissolving calcite from 5–20 grams of sandstone with dilute hydrochloric acid at room temperature. The remaining material was sieved, to isolate the coarsest material, from which quartz grains were handpicked under a binocular microscope. Each $\delta^{18}\text{O}(\text{Qt})$ analysis (1.2 to 1.8 mg) comprised three to six, crack-free, spherical quartz grains (500–710 μm diameter).

Quartz grains were liberated from wollastonite-bearing sandstones by dissolution of wollastonite in full strength fluoroboric acid (HBF_4) at room temperature for 5–10 days. No significant quartz dissolution occurs using this technique, and intact quartz grains, free of impinging wollastonite, were hand picked after dissolution. Wollastonite was separated from sandstones as follows: (1) crushing and sieving (150–300 μm); (2) cleaning the sieved material with dilute acetic acid; (3) separation of quartz from wollastonite with tetrabromoacetylene adjusted to $\rho \approx 2.8 \text{ g/cm}^3$, and (4) handpicking of wollastonite under a binocular microscope. Calcite isotopic analyses were performed on representative whole-rock powders ground in corundum and steel mortars. Powders were obtained from 3–5 mm diameter plugs drilled in the fresh face of a cut slab, in an area judged to be representative of the whole rock, or by hand picking pieces from a fresh sample surface. Veins and other small-scale features were avoided when drilling for whole rock powders. The amount of CO_2 given off during gas extraction for isotope analyses was used to determine the calcite content of the samples in weight %. There is < 5 wt.% variation in calcite content from powders obtained by drilling multiple spots on the same hand sample, and within a single outcrop (e.g., samples 99LV789 to 795, Table 2), and thus the calcite contents determined by this sampling method are representative. Carbonaceous matter, analyzed for $\delta^{13}\text{C}$, was isolated from whole rock carbonate and pelitic samples using dilute hydrochloric acid (~25°C, overnight), to remove calcite and oxides, and full strength nitric acid (~50°C, 1–2 hours) for sulfides.

Stable isotope ratios from silicate and carbonate minerals were measured at the stable isotope laboratory at the Department of Geology and Geophysics, University of Wisconsin–Madison. Isotope ratios for oxygen and carbon were measured on a Finnigan MAT 251 mass spectrometer. Data are reported using the standard per mil (‰) notation, relative to VSMOW (oxygen) or PDB (carbon). Unless otherwise noted, analytical precision about the mean is reported to one standard deviation (1SD).

Oxygen was liberated from silicates with BrF_5 using a 30W CO_2 laser, then purified cryogenically, passed through hot Hg (to remove F_2), and finally converted to CO_2 with a hot carbon rod (Valley et al., 1995). Silicate analyses were standardized daily by four or more analyses of UWG-2, Gore Mountain garnet standard, and sample $\delta^{18}\text{O}$ was corrected to the accepted value of 5.80‰ for UWG-2. The average $\delta^{18}\text{O}$ of UWG-2 for twenty-four days of analyses ($n=148$) is $5.72 \pm 0.12\text{‰}$ (day-to-day average = $5.72 \pm 0.09\text{‰}$; standard error ($1\sigma=1\text{SD}/(n^{0.5})$) is $\pm 0.01\text{‰}$). Daily corrections average 0.08‰. Average $\delta^{18}\text{O}$ of NBS-28 is $9.52 \pm 0.09\text{‰}$, $n=8$, and $\Delta(\text{NBS-28-UWG-2})$ averaged $3.74 \pm 0.04\text{‰}$, in good agreement with the UW lab long-term $\Delta(\text{NBS-28-UWG-2})$ of $3.70 \pm 0.06\text{‰}$ (Valley et al., 1995). Replicate analyses are better than $\pm 0.05\text{‰}$ for quartz, by the rapid heating technique of Spicuzza et al. (1998), and average better than $\pm 0.13\text{‰}$ for wollastonite.

Carbonate extractions were performed following the phosphoric acid reaction technique (McCrea, 1950; Sharma and Clayton, 1965). Whole-rock sample powders were reacted with concentrated phosphoric acid ($\rho \geq 1.92$). Initial extractions in the study ($n \approx 30$) were done overnight at 25°C ($\alpha\text{CO}_2\text{-Cc} = 1.010249$) with an average CO_2 yields for pure calcite standards of 89%; the remaining analyses ($n \approx 200$) were done at 50°C ($\alpha\text{CO}_2\text{-Cc} = 1.009225$) for four hours or longer, and produced better yields (97%). No differences in measured isotope ratios are seen in replicated standards. Evolved CO_2 was purified cryogenically and yields were measured by mercury manometer. At least one aliquot of the UW lab calcite standard, 93GV10, was used to monitor each batch of carbonate measurements. Twenty-five analyses of 93GV10 had an average $\delta^{13}\text{C}$ of $2.86 \pm 0.02\text{‰}$, and $\delta^{18}\text{O}$ of $24.36 \pm 0.08\text{‰}$. Three analyses of NBS-19 (accepted $\delta^{13}\text{C} = 1.93\text{‰}$; $\delta^{18}\text{O} = 28.67\text{‰}$; Coplen et al., 1983) had an average

$\delta^{13}\text{C}$ of $1.88 \pm 0.01\text{‰}$, $\delta^{18}\text{O} = 28.63 \pm 0.07\text{‰}$; three analyses of NBS-18 (accepted $\delta^{13}\text{C} = -5.08\text{‰}$; $\delta^{18}\text{O} = 7.35\text{‰}$; Coplen et al., 1983) had an average $\delta^{13}\text{C} -5.12 \pm 0.04\text{‰}$ and $\delta^{18}\text{O}$ of $7.09\text{‰} \pm 0.10\text{‰}$.

Carbonaceous matter was analyzed by oxidizing to CO_2 in the presence of excess CuO in sealed, quartz tubes at 1050°C for 12 hours; CO_2 was cryogenically purified before mass spectrometry. Quartz tubes were heated to 1050°C in air for 12 hours and cooled before loading samples to remove possible carbon contamination. The $\delta^{13}\text{C}$ of three analyses of a lab standard (UW-graphite) averaged $\delta^{13}\text{C} = -26.84\text{‰} \pm 0.01\text{‰}$ (accepted UW graphite $\delta^{13}\text{C} = -26.85$ Dunn and Valley, 1992). Two analyses of NBS-21 yielded an average $\delta^{13}\text{C}$ of -28.32 ± 0.05 (accepted NBS-21 = -28.15‰ , Hut, 1986).

We compare the $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ values of mineral pairs throughout this study to equilibrium fractionation factors, to test if minerals have exchanged oxygen or carbon, and the degree to which they approach isotopic equilibrium. Isotopic fractionation factors are expressed as:

$$\Delta_{i-j} = \delta^{18}\text{O}(\text{mineral } i) - \delta^{18}\text{O}(\text{mineral } j) \approx 1000 \ln \alpha(i-j)$$

The same expression is used for carbon isotope fractionation factors. Equilibrium fractionation factors are calculated for a particular temperature using the expression:

$$\Delta(\text{mineral } i - \text{mineral } j) \approx A_{i-j} \times 10^6/T^2 + B_{i-j} \times 10^3/T + C_{i-j},$$

In the expression T is temperature (K) and A, B, and C are experimentally or empirically determined coefficients; if no coefficient is given for A, B, or C, then its value is 0.0. The following factors are used for oxygen isotopes: quartz-calcite A = 0.87 (Sharp and Kirschner, 1994); quartz-wollastonite A = 2.66 (Matthews, 1994); quartz- H_2O A = 2.51, C = -1.46 (Clayton et al., 1972) and quartz-zircon A = 2.64 (Valley et al., 2003). Factors used for carbon isotopes are: calcite-graphite A = 5.81, C = -2.61, (Dunn and Valley, 1992, but see Valley, 2001). The CO_2 -calcite carbon isotope fractionation of Chacko et al. (1991) is used, and the reader is directed to their study for the details of the seventh order polynomial expression of this fractionation.

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EVALUATING QUARTZ $\delta^{18}\text{O}$ ZONING AT LAUREL MOUNTAIN

Grain Size Dependence of $\delta^{18}\text{O}$

Grains of different diameter were analyzed in non-crushed bulk separates. Grains were separated from calcite + quartz rocks by dissolution of calcite in dilute hydrochloric acid (room temperature, overnight) and from wollastonite-bearing rocks by dissolution of wollastonite with full strength fluoroboric acid (room temperature, 5-10 days). Smaller grains ($75\mu\text{m}$ - $150\mu\text{m}$) tend to be slightly higher in $\delta^{18}\text{O}$ than large diameter grains (500 – $710\mu\text{m}$) (average $\Delta_{\text{Fine Qt-Coarse Qt}} = 0.19\text{‰} \pm 0.06\text{‰}$, $n=10$), in rocks with and without wollastonite (Table DR1-1).

Air Abrasion Experiments

Air abrasion of coarse grains ($>500\mu\text{m}$ diameter) removed material from rims. Abraded grains show similar average difference in $\delta^{18}\text{O}$ between cores and rims. Rims of grains are heavier: $\Delta_{\text{Unabraded-Abraded}} = 0.22\text{‰} \pm 0.02\text{‰}$, Table DR1-2). Air abrasion of grains from coarse recrystallized quartzite next to the pluton, a sample likely to be isotopically homogeneous, revealed no significant difference in $\delta^{18}\text{O}$ ($\Delta_{\text{Unabraded-Abraded}} = 0.01\text{‰}$, Table DR1-2) of abraded versus non-abraded grains. Abrasion of more than ~ 30 wt.% did not result in significant differences between abraded and non-abraded quartz, indicating that the outermost rims of quartz grains are isotopically zoned.

Crushing Separation Experiments

A technique for removal of detrital quartz overgrowths for isotopic analysis involved crushing coarse quartz grains (liberated from wollastonite-bearing rocks by fluoroboric acid dissolution) in a steel mortar, and then sieving the material to recover the $<75\mu\text{m}$ and $>300\mu\text{m}$ diameter particles for analysis. This assumes that overgrowths and exterior material are preferentially concentrated in the fine fraction (Lee and Savin, 1985; Graham et al., 1996). The two experiments with this method showed a large variation in $\delta^{18}\text{O}$ for quartz grains: ($\Delta_{\text{Fine-Coarse}} = -0.62\text{‰}$ $\Delta_{\text{Fine-Coarse}} = 0.67\text{‰}$, Table DR1-3).

HF Leaching Experiments

Large diameter quartz grains were leached in hydrofluoric acid at room temperature for 45 to 90 minutes. The results show that etched grains have a lower average $\delta^{18}\text{O}$ ($\Delta_{\text{Unetched-Etched}} = 0.28\text{‰} \pm 0.17\text{‰}$, $n=7$, TABLE DR1-4). A recrystallized quartzite yielded a $\Delta_{\text{Unetched-Etched}}$ of -0.01‰ . The results of grain-scale tests of oxygen isotope zonation indicate that smaller diameter grains and material from the exterior of large grains tends to be heavier in $\delta^{18}\text{O}$ by about 0.2 – 0.6‰ . This material does not appear to be volumetrically significant and is consistent with findings that quartz overgrowths in sedimentary rocks have high $\delta^{18}\text{O}$ values ($\Delta^{18}\text{O}_{(\text{Overgrowth-Core})} = 25\text{‰}$, Graham et al., 1996), relative to detrital grains (e.g., Lee and Savin, 1985). These findings are also consistent with minor amounts of diffusion at the grain rims during cooling of the aureole.

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TABLE DR1-1. Comparison of $\delta^{18}\text{O}$ versus Quartz Grain Diameter

Sample	500 μm	300 μm	150 μm	75 μm	$\Delta_{\text{Smallest-Largest}}$
94LV145	16.82	17.10			0.28
94LV147	17.70	17.93			0.23
98LV416	16.50	16.62		16.24	-0.25
98LV420	16.13			16.37	0.24
98LV422	14.62	14.74		14.84	0.22
98LV426	15.60		15.71	15.33	-0.27
98LV428	13.35			13.57	0.22
98LV432*	16.58	16.68			0.10
98LV444	13.89		13.90		0.01
98LV445	14.20		14.34		0.14
99LV771	15.33	15.42			0.09

*Calcite cemented sandstone. All others are wollastonite-bearing sandstones. All grain size splits were analyzed in duplicate and average reproducibility is $\pm 0.05\text{‰}$.

TABLE DR1-2 Results of Quartz Grains Abrasion Tests

Sample	Initial $\delta^{18}\text{O}$ of grains	Wt.% Removed	$\delta^{18}\text{O}$ of abraded grains	
95LV206†	13.64	50%	13.63	
98LV416	16.50	50%	16.30	
98LV445	14.43	90%	14.23	Dust ζ
99LV766	15.58	13%/41%	15.48/15.34	16.35

*Abrasion is in weight % of quartz removed from the grains.

†Sample of recrystallized quartzite believed to be homogeneous.

ζ Abrasion dust was collected for a single analysis.

TABLE DR1-3 Crushing Separation of Material for Oxygen Isotope Analysis.

Sample	$\delta^{18}\text{O}$ of Isolated Material	
	>300* μm	<75* μm
99LV729	15.92	16.54
99LV769	15.58	14.91

Both samples are from wollastonite-bearing rocks. Crushed grains were hand-picked from quartz liberated from the rocks by dissolution in fluoroboric acid. *>300=coarse crushing residue; <75=fine residue

Table DR1-4. Variations in Quartz Grain $\delta^{18}\text{O}$ due to Acid Etching.

Sample	Initial Quartz	Acid Dissolved Quartz*	$\Delta_{\text{Initial-Acid Leached}}$	1 S.D.
94LV145	16.96	16.97	-0.01	0.05
95LV208†	13.35	13.36	-0.01	0.06
95LV291	14.34	14.20	0.14	0.05
95LV347	15.87	15.52	0.35	0.05
98LV416	16.50	16.17	0.33	0.1
98LV419	16.38	15.91	0.47	0.05
98LV420	16.13	15.86	0.34	0.05
98LV444	13.89	14.02	-0.13	0.02
98LV445	14.42	14.32	0.1	0.1

*Acid Leaching was commonly 45 minutes but was 90' in the case of 291.

†Sample of recrystallized quartzite believed to be isotopically homogeneous.

Table DR2. XRF-determined Whole Rock Composition of Calcite + Quartz Sandstones and Marbles of the Mount Morrison Sandstone

Sample	99LV726-A	99LV732	99LV736	99LV738	99LV789	99LV790	99LV793	99LV799	99LV821	99LV822	99LV824	99LV826	99LV829	99LV832
Oxides														
SiO ₂	67.1	57.70	68.80	74.60	68.10	64.90	62.60	66.80	17.90	17.50	47.90	47.00	15.40	46.80
Al ₂ O ₃	0.6	0.73	0.86	0.99	0.81	0.78	0.76	0.86	0.53	0.86	1.09	1.01	0.58	0.59
CaO	17.3	23.20	16.80	13.00	17.10	19.10	20.10	17.60	45.30	45.60	28.00	28.90	46.20	29.10
MgO	0.47	0.39	0.41	0.32	0.43	0.42	0.41	0.33	0.58	0.66	0.64	0.60	0.54	0.42
Na ₂ O	0.04	0.02	0.01	0.03	0.05	0.04	0.06	0.02	0.05	0.04	0.15	0.08	0.05	0.03
K ₂ O	0.46	0.59	0.74	0.77	0.61	0.60	0.61	0.67	0.28	0.52	0.54	0.52	0.31	0.41
Fe ₂ O ₃	0.55	0.39	0.37	0.45	0.44	0.45	0.42	0.30	0.31	0.33	0.36	0.34	0.32	0.38
MnO	—	—	—	—	—	—	—	—	—	—	—	—	—	—
TiO ₂	0.044	0.04	0.04	0.03	0.04	0.04	0.04	0.04	0.04	0.05	0.05	0.05	0.04	0.04
P ₂ O ₅	0.1	0.12	0.05	0.18	0.09	0.13	0.09	0.08	1.45	0.38	0.11	0.13	0.59	0.26
Cr ₂ O ₃	0.06	0.02	0.03	0.05	0.03	0.05	0.03	0.02	—	—	0.01	0.01	—	0.02
LOI	12.5	16.20	12.10	9.70	12.40	13.90	14.70	13.20	33.30	34.40	21.40	21.60	34.80	22.20
Sum	99.3	99.5	100.2	100.1	100.2	100.3	99.9	100.0	99.8	100.3	100.2	100.2	98.9	100.2
Trace Elements														
Rb		29			35			34			30			
Sr		157			104			125			264			
Y		—			—			—			—			
Nb		2			4			3			3			
Ba		166			146			143			265			

XRF-determined Whole Rock Composition of Wollastonite + Quartz Sandstones and Marbles of the Mount Morrison Sandstone

Sample	99LV726-B	99LV733	99LV737	99LV791	99LV792	99LV800	99LV820	99LV823	99LV825	99LV828	99LV831
Oxides											
SiO ₂	72.9	62	75	71.6	63.9	70.3	27.6	57.7	57.3	22.1	56.3
Al ₂ O ₃	1.1	0.73	1.22	0.83	0.7	0.99	1.38	1.3	1.14	0.24	0.75
CaO	21.8	31.8	20.5	23.1	27.5	18.8	45.3	32.7	34	46	31.8
MgO	0.57	0.36	0.41	0.38	0.42	0.36	0.96	0.76	0.92	1.28	0.55
Na ₂ O	0.12	-0.01	-0.01	-0.01	0.02	0.04	0.08	0.08	0.09	0.03	0.03
K ₂ O	0.56	0.5	0.43	0.61	0.46	0.72	0.35	0.55	0.51	0.03	0.37
Fe ₂ O ₃	0.58	0.44	0.44	0.5	0.44	0.36	0.68	0.35	0.45	0.42	0.44
MnO	0.02	0.07	0.04	0.03	0.03	—	0.02	0.01	0.01	0.02	0.02
TiO ₂	0.054	0.034	0.035	0.045	0.041	0.041	0.077	0.051	0.054	0.04	0.041
P ₂ O ₅	0.13	0.11	0.05	0.09	0.09	0.09	0.49	0.16	0.13	0.95	0.3
Cr ₂ O ₃	0.03	0.02	0.03	0.03	0.02	0.02	—	—	0.02	—	0.02
LOI	2.05	1.35	2.15	1.85	5.15	8.4	23.3	4.85	2.95	29.2	8.35
Sum	100	97.4	100.4	99.2	98.8	100.2	100.2	98.5	97.7	100.3	99
Trace Elements											
Rb		28		39		30			19		
Sr		92		92		116			352		
Y		7		4		3			—		
Nb		—		—		3			—		
Ba		79		207		158			238		

Sample pairs are as follows: 726A-726B, 732-733, 736-737, 790-79, 792-793 (on strike), 799-800, 820-(821&822) (823 & 825)-(824 & 826), 828-829, 831-832. XRF detection limits are 0.01 wt.% for major oxides; 2 ppm for Rb, Sr, Y, Nd, 20 ppm Ba.

Table DR3: Laurel Mountain Tremolite Analyses

	99LV581	99LV578	99LV824
# Grains	n=8	n=6	n=7
SiO₂	57.29	56.91	57.30
TiO₂	0.02	0.02	0.05
Al₂O₃	1.31	1.58	1.33
FeO	0.91	0.77	1.12
MnO	0.03	0.02	0.02
MgO	23.26	23.36	22.88
CaO	13.32	13.31	13.30
Na₂O	0.01	0.01	0.01
K₂O	0.20	0.18	0.09
F	0.96	1.04	0.48
OH	1.65	1.84	1.91
Total	98.35	98.16	98.09
X[OH]	0.633	0.640	0.800
Si	7.897	7.848	7.920
Al (iv)	0.103	0.152	0.080
Tetrahedral	8.000	8.000	8.000
Al (vi)	0.110	0.104	0.137
Ti	0.002	0.002	0.005
Cr	0.000	0.000	0.000
Fe(iii)	0.017	0.077	0.000
Fe(ii)	0.087	0.012	0.129
Mn	0.003	0.002	0.002
Mg	4.780	4.803	4.715
C Site	5.000	5.000	4.988
Ca	1.967	1.967	1.969
Na	0.002	0.002	0.002
B Site	1.969	1.968	1.971
K	0.035	0.031	0.016
A Site	0.035	0.031	0.016
Fe/(Fe+Mg)	0.02	0.02	0.03
α Tremolite	0.32	0.33	0.48

OH calculated as 100 minus the total of oxides + F.

$$\alpha \text{ Tremolite} = (\text{XCa}_{[\text{M2 site}]}) (\text{XMg}_{[\text{M1,2,3 site}]})^5 (\text{XOH})^2$$

Figure DR-1

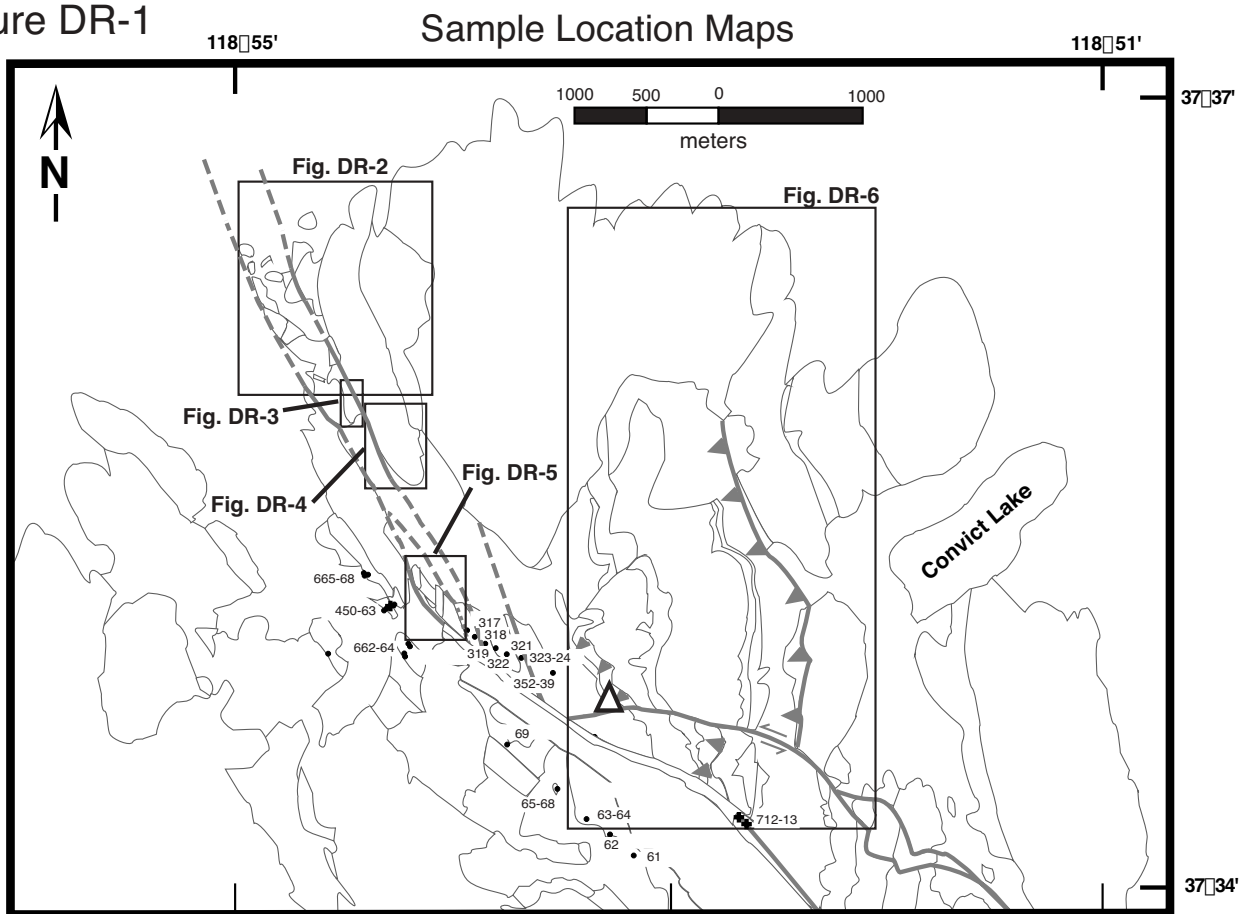


Figure DR-2

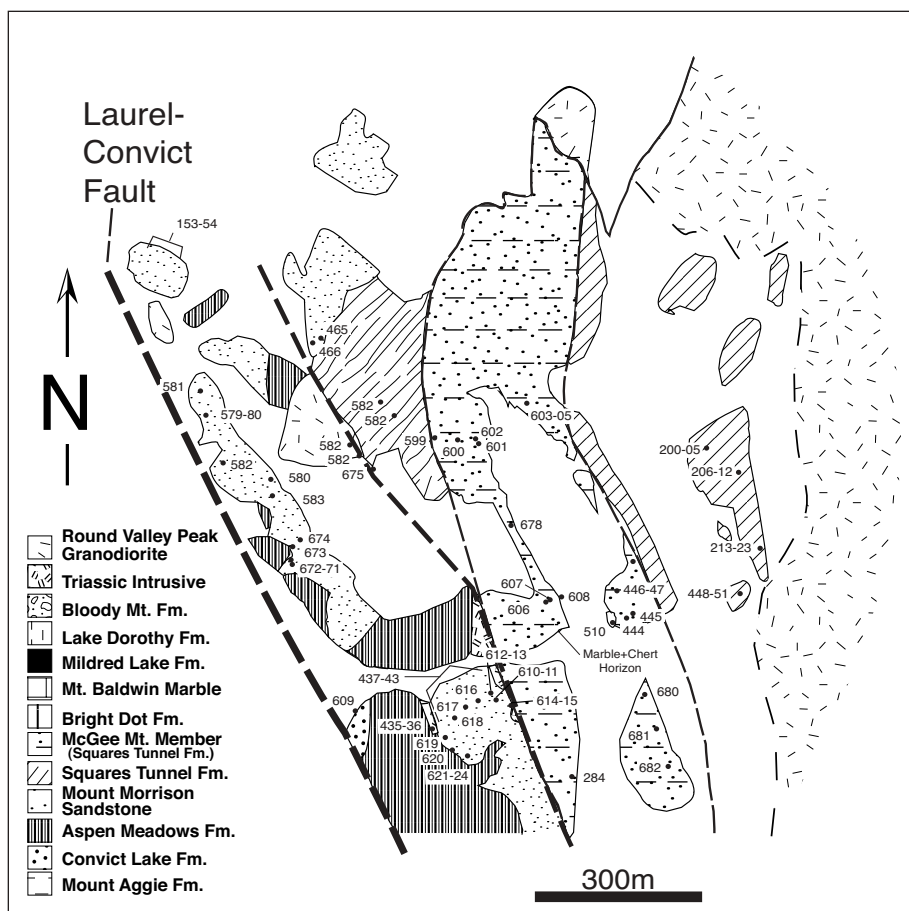


Figure DR-3

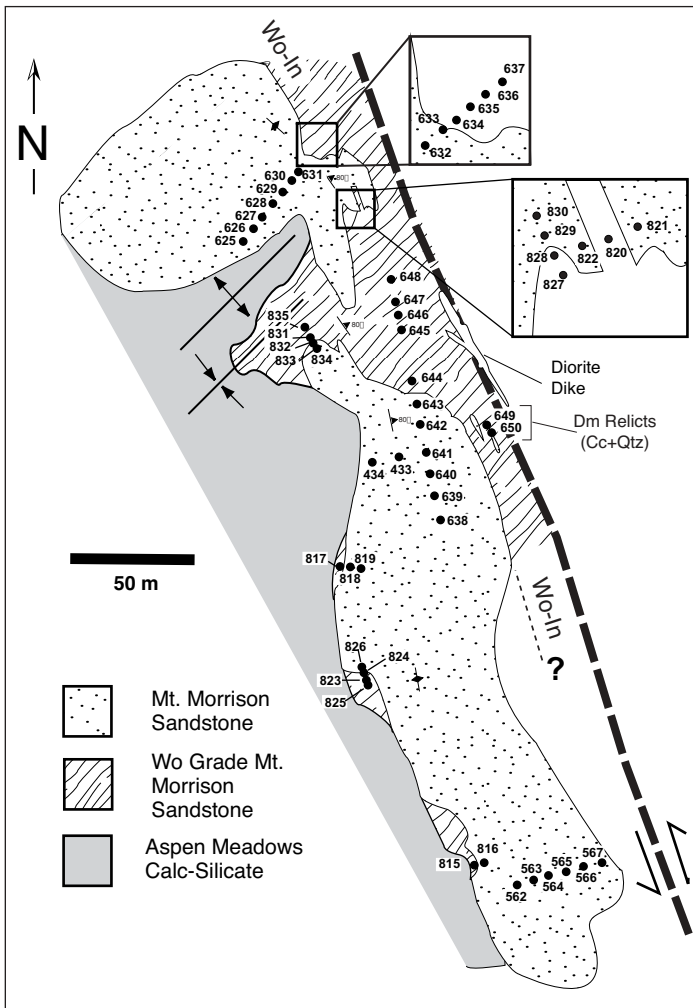


Figure DR-4

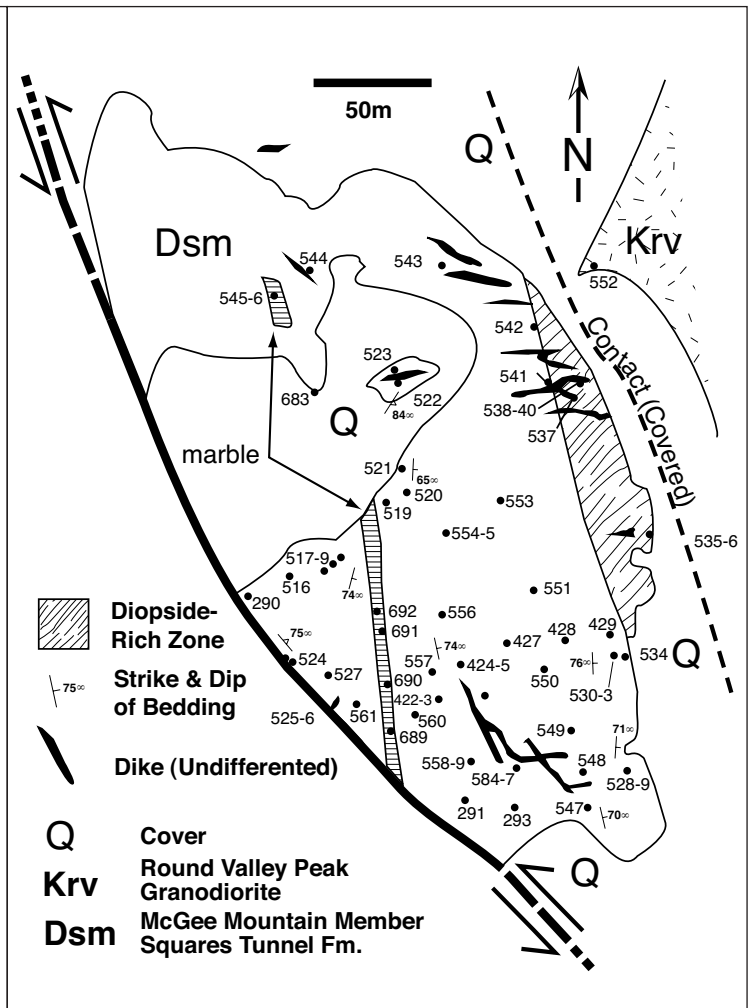


Figure DR-5

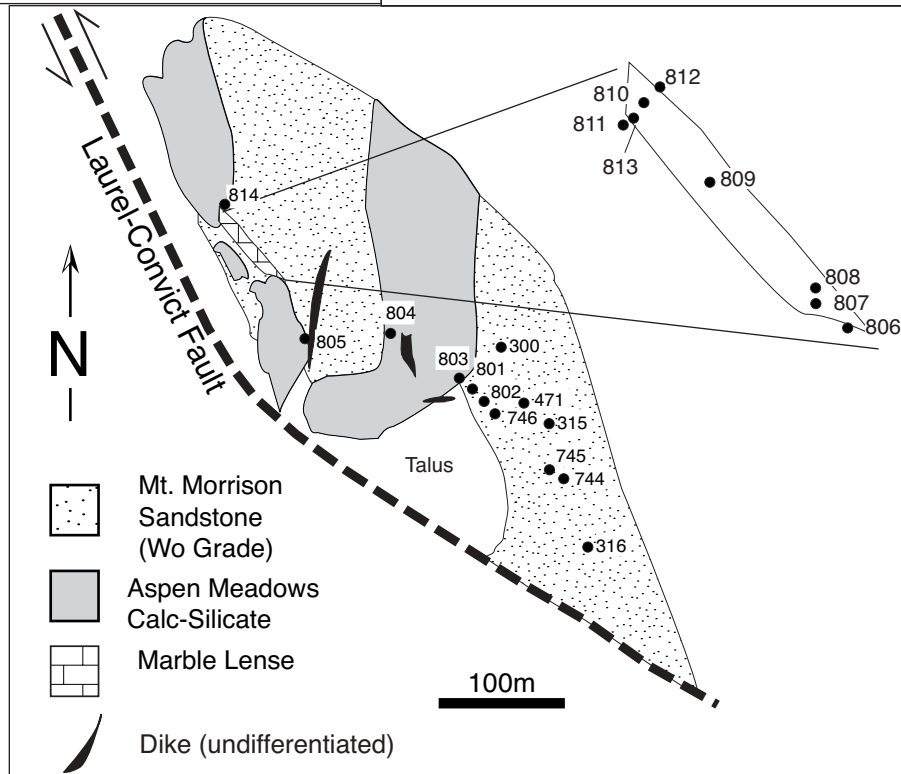


Figure DR-6

