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Short-wavelength infrared spectroscopy: A new petrological tool in low- to very low-grade pelites

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In the following we provide: i) further detail on the analytical procedure for XRD and SWIR measurements; ii) intra-sample variance data relevant for the reproducibility of various parameters used in the study; iii) a sample description (see also Table DR3); iv) a figure with the sample names and localities (Figure DR1) used in Figure 2; and v) data tables with the results of KI and ISM measurements (Table DR1), K-white mica *b* cell dimension and 2200wvl measurements (Table DR2), and the rocktype and mineralogy of the samples (Table DR3).

i) Methods

Sample preparation and XRD measurements were carried out according to the recommendations given by Kisch (1991)

Kübler index

Kübler index (KI) and K-white mica *b* cell dimension for the samples from the Montagne Noire was measured on oriented, air-dried aggregates (<2µm fraction), using a D501 Bruker-AXS (Siemens) diffractometer at the University of Giessen (CuK_{α} radiation at 40 kV and 30 mA; automatic divergence slits, primary and secondary V20, with a secondary graphite monochromator). KI was calculated using the software MacDiff 4.2.5 (written by Dr. R. Petschick, Goethe-University, Frankfurt/Main; see Doublier et al. 2010 for details). KI values were calibrated using a correlation with the standard samples of Warr and Rice (1994), and the recalibration of the standard after Kisch et al. (2004).

KWM *b* cell dimension

The measurements for the determination of the K-white mica *b* cell dimension using powder XRD have been performed on randomly oriented specimen of the <2 µm fraction. The position of the (060) peak has been measured with the software DIFFRACPlus (Bruker AXS®, see Doublier et al. 2010 for details). The samples from New Caledonia were measured using a D5000 Bruker-AXS (Siemens) diffractometer at the University of Basel, with the same settings described above.

Short-wavelength infrared spectroscopy

The SWIR measurements have been carried out on the oriented XRD discs using a CSIRO-developed a drill core/chip logging system called HyChips™ which integrates an Analytical Spectral Devices (ASD) FieldSpec-3 spectrometer with a spectral range of 350–2500 nm and a spectral resolution of ~8 nm. The spectra measured were processed with CSIRO-developed software — The Spectral Geologist (TSG™).

ii) Reproducibility of SWIR measures

To estimate the methodological errors for the spectral parameters presented here, the intra-sample variance (Robinson et al., 1990) has been determined by repetitive measurements of various specimens (‘variance specimen’) from one sample. The intra-sample variance includes various effects that may cause errors for XRD-based KI (e.g. Blenkinsop, 1988; Robinson et al., 1990), such as sample preparation, thickness of specimen, evaluation procedure, and machine fluctuation. Also, the intra-sample variation integrates factors that may influence the spectral measurements like spectral bandwidth, detector sensitivity and light intensity (e.g., Clark, 1999). For the KI, Doublier (2007) estimated the intra-sample variance of the Montagne Noire samples by calculating the mean values of nine variance specimen, each measured twice, at 1.29% (one standard deviation, 1σ).

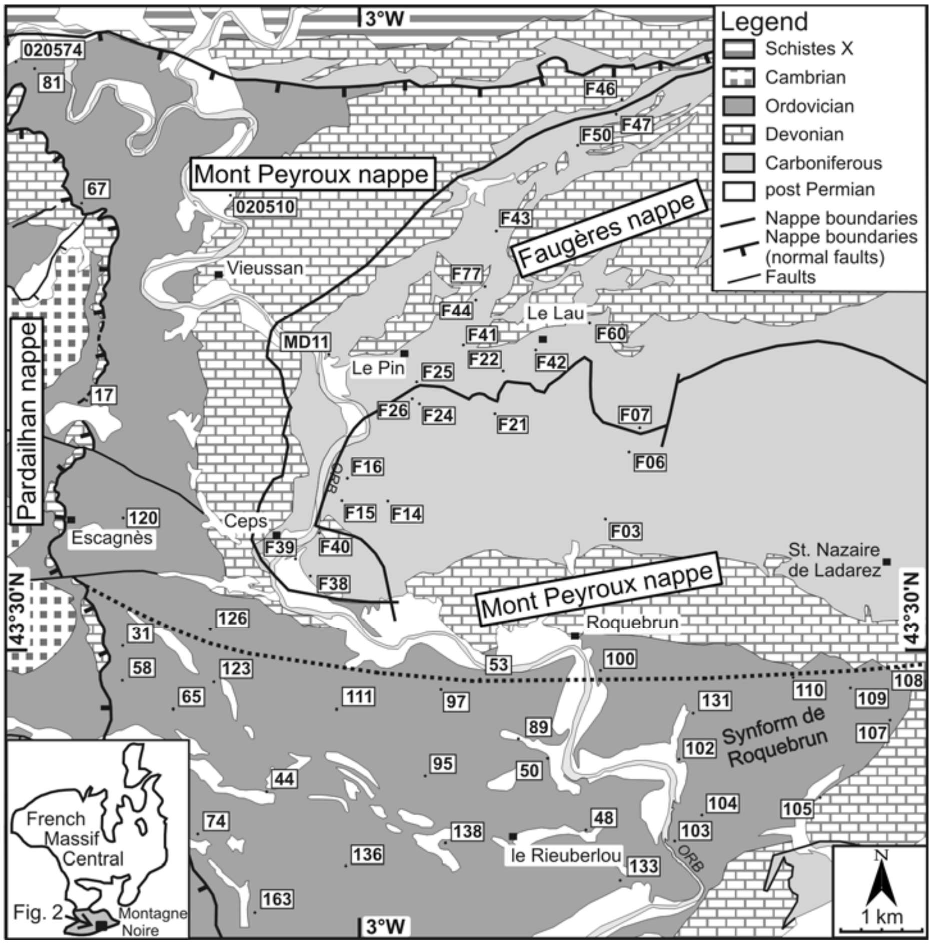
For the ISM parameters used in this study, a ‘relative’ standard deviation has been calculated. In a first step, a standard deviation has been calculated from the mean values of ten variance specimens, with each mean calculated from five measurements. The ‘relative’ standard deviation was calculated as the ratio: standard deviation (variance samples)/range of observed values. Because the dataset does not cover the whole range of values realized in very low-grade rocks, this estimate is conservative. The results are comparable to those observed for KI with 1.71% for ISM(A/D) and 2.91% for ISM(H_2O). For the ISM(as), no intra-sample variance could be determined, since all variance samples yielded exactly the same value. The lack of variance in ISM(as) is due to a high degree of ‘channel granularity’ factored into peak asymmetry calculations that results in a limited number of values (see Doublier et al. 2010 for detail).
The lowest relative standard deviation of all SWIR measures was the 2200wvl with 0.17%.

iii) Sample description

The samples used in this study are shales and slates, which cover a wide range of conditions observed in very low-grade terrains in terms of metamorphic grade (KI range: 0.21–1.08 Δº2Θ) and KWM *b* cell dimension (range: 8.977– 9.059 Å). They come from two different areas, the Montagne Noire in the southernmost part of the French Massif Central and the Diahot terrane of New Caledonia. While the samples from the Montagne Noire show medium-P (eastern Monts de Lacaune area and Monts de St. Gervais; Doublier et al., 2006) and medium to low-P (Mont Peyroux and Faugères nappes; Doublier, 2007) conditions, the Diahot samples have been metamorphosed under high-P conditions (Fitzherbert et al., 2003; Potel et al., 2006).
XRF analyses of 32 samples indicated similar whole-rock chemistry throughout the dataset and plot in the logarithmic scheme from Herron (1988) in the shale field (Doublier et al., 2010). The mineralogy of the samples as determined by XRD is shown in Table DR3: all samples contain quartz and KWM, and chlorite and albite are present in most. Other minerals identified in some samples include paragonite, kaolinite, pyrophyllite, calcite and goethite. In some samples mixed layer minerals illite/smectite and chlorite/smectite have been detected. The smectite content has been estimated after the method of Moore and Reynolds (1989) at < 10% in most illite/smectites and < 20% in the chlorite/smectites.

The high-P samples from New Caledonia have a distinct mineralogy and usually contain stilpnomelane and/or lawsonite. Glaucophane has been detected in one sample.

iv) Figure DR1: samples localities of the samples shown in Figure 2



v) Data Tables

Table DR1: Sample names, tectonic units, and results of ISM and KI measurements

Not all samples recorded in Table DR1 are plotted in Figure 1 for the following reasons: the paragonite bearing samples have been excluded, because paragonite may have an influence on the 2200 absorption feature which may disturb the definition of the ‘unassignable value range’. The other samples -all part of the regional example (Fig. 2)- do not contain paragonite and were included to test whether the method can be applied to samples that haven’t been used to define the ‘unassignable value range’.

TABLE DR1. RESULTS OF ISM AND KI MEASUREMENTS

Sample name	Tectonic unit	Kübler Index (Δ°2Θ)	ISM(as)	ISM(H_2O)	ISM(A/D)
2*	MP	0,221	1,029	0,925	1,507
11*	MP	0,361	0,829	0,709	1,957
17	MP	0,428	0,834	0,690	4,932
23*	P	0,485	0,545	0,971	4,729
31	MP	0,477	0,545	1,196	6,429
36*	P	0,302	0,818	1,454	2,982
40*	P	0,808	0,364	0,715	5,242
44	MP	0,522	0,343	0,744	6,360
48*	MP	0,709	0,636	0,638	6,728
50*	MP	0,653	0,545	0,546	5,960
53*	MP	0,378	0,727	1,256	2,337
58*	MP	0,519	0,581	1,013	4,358
65	MP	0,626	0,391	0,463	6,254
67	MP	0,309	0,584	0,694	4,457
72*	MP	0,900	0,364	0,401	6,587
74*	MP	0,749	0,364	0,541	6,063
78*	P	0,333	1,000	1,657	3,281
81	MP	0,292	1,289	0,664	3,248
89*	MP	0,647	0,364	0,664	5,628
95	MP	0,684	0,364	0,475	6,415
97	MP	0,588	0,470	0,680	5,656
100	MP	0,479	0,545	0,801	3,664
102*	MP	0,586	0,473	0,908	5,105
103	MP	0,749	0,545	0,444	6,613
104	MP	0,649	0,545	0,482	6,722
105*	MP	0,895	0,727	0,436	6,840
107*	MP	0,852	0,455	0,328	6,823
108*	MP	1,075	0,455	0,336	6,856
109*	MP	0,668	0,545	0,570	6,435
110*	MP	0,632	0,545	0,793	5,928
111*	MP	0,465	0,636	1,199	4,376
120	MP	0,396	0,545	1,018	2,172
123	MP	0,398	0,439	1,156	6,333
126*	MP	0,347	0,545	1,936	4,148
131*	MP	0,473	0,545	1,295	5,514
133*	MP	0,730	0,545	0,775	5,889
136*	MP	0,893	0,364	0,302	6,280
138*	MP	0,816	0,364	0,377	6,386
139*	MP	0,852	0,545	0,538	6,541
140*	P	0,989	0,364	0,593	6,416
142*	P	0,669	0,364	0,714	6,188
143*	P	0,675	0,364	0,552	6,262
144*	P	0,955	0,273	0,622	6,761
146*	P	0,419	0,545	1,526	4,738
148*	P	0,479	0,455	0,883	5,132

TABLE DR1 CONTINUED

Sample name	Tectonic unit	Kübler Index (Δ°2Θ)	ISM(as)	ISM(H_2O)	ISM(A/D)
152*	P	0,726	0,364	0,735	5,973
161*	P	0,416	0,545	1,164	5,752
163*	MP	0,817	0,364	0,442	6,589
164*	MP	0,613	0,364	0,678	6,181
167*	MP	0,815	0,364	0,561	6,418
170*	P	0,785	0,364	0,601	6,244
174*	P	0,572	0,545	0,693	4,375
178*	P	0,357	0,909	1,607	3,358
190*	P	0,296	0,909	1,533	3,805
195*	P	0,306	0,727	1,479	3,691
197*	P	0,289	0,909	2,031	3,528
203*	P	0,278	0,545	1,266	4,481
212*	P	0,376	0,727	1,776	4,334
216*	P	0,235	0,727	2,376	2,860
228*	P	0,439	0,545	1,168	3,955
232*	P	0,304	0,909	1,288	4,409
234*	P	0,279	0,727	1,649	4,085
242*	M	0,345	0,545	1,101	2,962
245*	M	0,260	1,200	1,510	3,967
246*	M	0,266	1,091	1,710	3,215
247*	M	0,319	1,455	0,842	4,621
249*	M	0,245	1,018	1,585	3,461
250*	M	0,234	1,091	1,433	3,567
252*	M	0,281	1,273	1,060	4,754
254*	M	0,233	1,091	1,048	4,030
261*	P	0,269	1,273	1,122	4,640
264*	P	0,332	1,091	0,582	3,921
265*	P	0,287	1,018	1,421	3,394
266*	M	0,273	1,091	1,632	3,784
267*	P	0,251	1,091	1,649	3,336
268*	P	0,280	1,325	1,080	4,190
275*	P	0,330	1,273	1,068	4,641
276*	P	0,266	0,545	1,939	4,684
277*	P	0,265	1,273	0,967	4,360
278*	P	0,244	1,055	2,126	3,285
020510*	MP	0,289	0,545	1,223	2,462
020536*	MP	0,397	0,654	1,191	5,020
020574	MP	0,307	1,095	0,609	1,429
F03	MP	0,312	0,926	2,175	4,022
F06	MP	0,325	0,820	1,837	2,494
F07*	F	0,259	0,909	1,113	1,799
F14*	MP	0,318	0,818	1,546	2,413
F15*	MP	0,289	0,727	1,400	2,197
F16*	MP	0,294	0,727	1,469	2,458
F21	MP	0,301	0,788	2,031	4,802
F22*	F	0,306	1,091	1,321	1,968
F24*	MP	0,315	0,727	1,716	2,641

TABLE DR1 CONTINUED

Sample name	Tectonic unit	Kübler Index (Δ°2Θ)	ISM(as)	ISM(H_2O)	ISM(A/D)
F25*	F	0,257	0,909	1,246	2,061
F26*	MP	0,308	0,614	1,180	1,749
F30*	MP	0,489	0,727	0,658	4,651
F31*	MP	0,563	0,727	0,763	4,536
F32*	MP	0,517	0,727	0,487	4,459
F33*	MP	0,531	0,727	0,453	4,401
F36*	MP	0,419	0,727	1,052	4,760
F37*	MP	0,451	0,727	0,749	4,544
F38	F	0,365	0,545	1,102	3,037
F39	F	0,379	0,545	1,076	3,288
F40	F	0,333	0,545	1,133	3,636
F41*	F	0,256	1,255	0,875	1,829
F42*	F	0,256	0,715	0,778	1,628
F43*	F	0,260	1,634	0,685	1,611
F44*	F	0,264	1,026	0,695	1,711
F46	F	0,269	1,114	2,092	5,044
F47	F	0,275	1,131	1,111	5,030
F50	F	0,286	0,641	1,870	4,758
F60*	F	0,289	0,909	1,660	2,934
F77*	F	0,300	0,909	1,558	2,776
IB02*	MdSG	0,264	1,273	1,172	4,476
IB03*	MdSG	0,234	1,091	1,324	4,236
IB04*	MdSG	0,216	1,091	1,225	3,660
IB20*	MdL	0,281	1,455	1,301	5,031
IB29*	MdL	0,214	1,091	1,553	3,980
IB30*	MdL	0,218	1,091	0,958	3,002
IB31*	MdL	0,220	1,182	1,326	3,583
IB32*	MdL	0,244	1,364	1,139	3,939
IB33*	MdL	0,259	1,182	1,110	3,674
IB44*	MdL	0,304	1,091	0,918	3,833
IB49*	MdL	0,223	1,182	0,807	3,532
IB53*	MdL	0,270	1,091	1,241	4,109
IB54*	MdL	0,229	1,091	1,115	3,344
IB56*	MdL	0,226	1,273	1,180	3,845
MD11*	F	0,293	1,467	1,010	1,762
N09*	MdL	0,320	0,909	0,736	3,705

*samples used in Figure 1

MP - Mont Peyroux nappe (southern Montagne Noire, France)

P - Pardailhan nappe (southern Montagne Noire, France)

M - Minervois nappe (southern Montagne Noire, France)

F- Faugères nappe (southern Montagne Noire, France)

MdSG - Monts de St. Gervais (northeastern Montagn Noire, France)

MdL - Monts de Lacaune (northeastern Montagne Noire, France)

Table DR2: Results of K-white mica *b* cell dimension and 2200wvl measurements

TABLE DR2. RESULTS KWM *b* CELL DIMENSION AND 2200wvl

Sample name	Tectonic unit	KWM *b* cell dimension (Å)	2200wvl (nm)
020510	MP	8,991	2201,20
020536	MP	8,978	2202,49
2	MP	8,986	2202,66
36	P	8,996	2204,80
53	MP	8,993	2203,20
78	P	9,012	2209,87
126	MP	8,994	2201,67
146	P	8,991	2200,20
161	P	8,996	2200,98
178	P	9,008	2208,43
190	P	9,014	2208,81
195	P	9,002	2203,29
197	P	9,010	2207,69
203	P	8,997	2201,11
212	P	9,007	2202,30
216	P	9,010	2206,20
232	P	9,015	2206,73
234	P	9,012	2204,85
242	M	8,988	2200,51
245	M	9,029	2215,09
246	M	9,017	2212,66
247	M	9,037	2218,52
249	M	9,021	2210,01
250	M	9,027	2211,36
252	M	9,038	2215,29
254	M	9,040	2211,70
261	P	9,042	2215,11
264	P	9,031	2211,32
265	P	9,020	2209,79
266	M	9,023	2214,34
267	P	9,018	2211,37
268	P	9,024	2215,09
275	P	9,031	2215,16
276	P	8,996	2199,46
277	P	9,035	2216,43
278	P	9,024	2210,38
F07	F	9,006	2207,87
F14	MP	8,998	2205,89
F15	MP	8,998	2205,40
F16	MP	9,001	2205,50
F22	F	9,007	2208,82
F24	MP	8,994	2204,30
F25	F	9,003	2206,94
F26	MP	9,005	2206,76
F36	MP	8,995	2203,55

TABLE DR2 CONTINUED

Sample name	Tectonic unit	KWM *b* cell dimension (Å)	2200wvl (nm)
F41	F	9,011	2207,21
F42	F	9,005	2206,22
F43	F	9,003	2208,23
F44	F	9,002	2207,67
F60	F	9,006	2207,03
F77	F	9,004	2206,70
IB02	MdSG	9,025	2216,46
IB03	MdSG	9,026	2214,66
IB04	MdSG	9,029	2212,19
IB20	MdL	9,029	2217,83
IB29	MdL	9,031	2213,06
IB30	MdL	9,024	2213,24
IB31	MdL	9,025	2214,36
IB32	MdL	9,030	2216,97
IB33	MdL	9,026	2212,78
IB44	MdL	9,027	2213,36
IB49	MdL	9,027	2214,20
IB53	MdL	9,030	2214,04
IB54	MdL	9,012	2212,75
IB56	MdL	9,033	2215,02
MD11	F	9,005	2207,50
MF3027	Diahot	9,040	2223,58
MF3028	Diahot	9,050	2220,75
MF3031	Diahot	9,040	2223,42
MF3140	Diahot	9,056	2224,20
MF3141	Diahot	9,059	2229,56
MF3142	Diahot	9,056	2230,38
MF3143	Diahot	9,052	2226,47
MF3144	Diahot	9,050	2227,26
N09	MdL	9,005	2206,60
PS11	Diahot	9,048	2228,11
PS12	Diahot	9,048	2228,76
PS13	Diahot	9,050	2230,37
PS18	Diahot	9,052	2229,72
PS20	Diahot	9,040	2226,19
PS21	Diahot	9,058	2214,26
PS22	Diahot	9,050	2231,65
PS24	Diahot	9,058	2229,21

MP - Mont Peyroux nappe (southern Montagne Noire, France)
P - Pardailhan nappe (southern Montagne Noire, France)
M - Minervois nappe (southern Montagne Noire, France)
F- Faugères nappe (southern Montagne Noire, France)
MdSG - Monts de St. Gervais (northeastern Montagn Noire, France)
MdL - Monts de Lacaune (northeastern Montagne Noire, France)
Diahot - Diahot Terrane (New Caledonia)

Table DR3: Rocktypes and mineralogy of the samples (as determined by XRD analysis)

TABLE DR3. ROCKTYPE AND MINERALOGY

Sample name	rocktype	Qtz	Ab	KWM	Chl	ML	others
2*	slate	x	x	x	x		
11*	slate	x		x	x		Prl
17	slate	x		x	x		Prl
23*	slate	x	x	x	x		
31	slate	x	x	x	x		
36*	slate	x	x	x	x		
40*	slate	x	x	x	x		
44	silty slate	x	x	x	x		Pg
48*	slate	x	x	x	x	I/S, C/S	
50*	silty slate	x	x	x	x	I/S, C/S	
53*	slate	x	x	x	x		
58*	slate	x	x	x	x	I/S	
65	slate	x		x	x	I/S	
67	slate	x		x	x		Pg
72*	slate	x	x	x	x	I/S	
74*	slate	x	x	x	x		
78*	slate	x	x	x	x		
81	slate	x		x	x		Pg
89*	slate	x		x	x		
95	slate	x	x	x	x		
97	slate	x	x	x	x		
100	slate	x	x	x	x		Pg
102*	silty slate	x	x	x	x		
103	silty slate	x	x	x	x		
104	silty slate	x	x	x	x		
105*	slate	x	x	x	x	I/S	
107*	silty slate	x	x	x	x	I/S	
108*	slate	x	x	x	x	I/S	
109*	slate	x	x	x			
110*	slate	x	x	x			
111*	slate	x	x	x	x		
120	silty slate	x		x	x		Pg
123	silty slate	x		x	x		Pg
126*	silty slate	x	x	x	x		
131*	silty slate	x	x	x	x		
133*	silty slate	x	x	x	x		Kln
136*	slate	x		x	x	I/S	
138*	slate	x		x	x	I/S	
139*	slate	x	x	x		I/S	
140*	slate	x		x	x	I/S	
142*	silty slate	x		x	x		
143*	silty slate	x		x		C/S	
144*	slate	x		x	x	I/S	Kln
146*	silty slate	x	x	x	x		
148*	silty slate	x		x	x		
152*	slate	x		x	x		

TABLE DR3 CONTINUED

Sample name	rocktype	Qtz	Ab	KWM	Chl	ML	others
161*	slate	x		x	x		
163*	slate	x		x	x	I/S	
164*	silty slate	x		x	x		
167*	slate	x		x	x	I/S	
170*	silty slate	x		x	x	I/S	
174*	silty slate	x	x	x	x		
178*	silty slate	x	x	x	x		
190*	slate	x	x	x	x		
195*	slate	x	x	x	x		
197*	silty slate	x	x	x	x		
203*	silty slate	x	x	x	x		
212*	silty slate	x	x	x	x		
216*	slate	x	x	x	x		
228*	slate	x	x	x	x		
232*	silty slate	x	x	x	x		
234*	silty slate	x	x	x	x		
242*	slate	x	x	x	x		
245*	slate	x	x	x	x		
246*	slate	x	x	x	x		
247*	silty slate	x	x	x	x	C/S	
249*	slate	x	x	x	x		
250*	slate	x	x	x	x		
252*	slate	x	x	x			
254*	silty slate	x	x	x	x		
261*	silty slate	x	x	x	x		
264*	slate	x	x	x	x	C/S	
265*	slate	x	x	x	x		
266*	slate	x	x	x	x	C/S	
267*	slate	x	x	x	x		
268*	slate	x	x	x	x		
275*	slate	x	x	x	x		
276*	slate	x	x	x			
277*	silty slate	x	x	x	x		
278*	slate	x	x	x	x		
020510*	silty slate	x		x	x		
020536*	siltstone	x		x	x		
020574	slate	x	x	x	x		Pg
F03	slate	x	x	x	x		
F06	slate	x	x	x	x		
F07*	slate	x	x	x	x		
F14*	slate	x	x	x	x		
F15*	slate	x	x	x	x		
F16*	slate	x	x	x	x		

TABLE DR3 CONTINUED

Sample name	rocktype	Qtz	Ab	KWM	Chl	ML	others
F21	slate	x	x	x	x		
F22*	slate	x	x	x	x		
F24*	slate	x	x	x	x		Cal
F25*	slate	x	x	x	x		
F26*	slate	x	x	x	x		
F30*	silty slate	x	x	x	x	C/S	Cal
F31*	silty slate	x	x	x	x	C/S	Cal
F32*	slate	x	x	x	x	I/S, C/S	Cal
F33*	slate	x	x	x	x	I/S, C/S	Cal
F36*	slate	x	x	x	x	C/S	Cal
F37*	slate	x	x	x	x	C/S	
F38	silty slate	x	x	x	x		Pg
F39	slate	x	x	x	x		Pg
F40	silty slate	x	x	x	x		Cal, Pg
F41*	slate	x	x	x	x		
F42*	slate	x	x	x	x		
F43*	slate	x	x	x	x		
F44*	slate	x	x	x	x		
F46	slate	x	x	x	x	C/S	
F47	slate	x	x	x	x	C/S	
F50	slate	x	x	x	x	C/S	
F60*	slate	x	x	x	x		
F77*	slate	x	x	x	x		
IB02*	siltstone	x	x	x	x		
IB03*	siltstone	x	x	x	x		
IB04*	slaty siltstone	x	x	x	x		
IB20*	slate	x		x	x		Cal
IB29*	slaty siltstone	x	x	x	x		
IB30*	silty slate	x	x	x	x		Cal
IB31*	silty slate	x	x	x	x		Cal
IB32*	slate	x	x	x	x		
IB33*	silty slate	x	x	x	x	I/S	
IB44*	slaty siltstone	x		x	x		
IB49*	silty slate	x	x	x	x		
IB53*	slaty siltstone	x		x	x	C/S	
IB54*	silty slate	x	x	x	x		
IB56*	silty slate	x	x	x	x		
MD11*	slate	x		x	x		Cal
MF3027	slate	x	x	x	x		Lws
MF3028	slate	x	x	x		K/S	Lws
MF3031	slate	x	x	x	x		Lws, Gln
MF3140	slate	x	x	x	x	C/S	Lws
MF3141	slate	x	x	x	x	C/S	Stp
MF3142	slate	x	x	x	x	C/S	Lws, Stp
MF3143	slate	x	x	x	x		Lws
MF3144	slate	x	x	x	x		Lws, Stp

TABLE DR3 CONTINUED

Sample name	rocktype	Qtz	Ab	KWM	Chl	ML	others
N09*	silty slate	x	x	x	x		Gt
PS11	slate	x	x	x	x		
PS12	slate	x	x	x	x		Lws
PS13	slate	x	x	x	x		Stp
PS18	slate	x	x	x	x		Stp
PS20	slate	x	x	x	x		Lws, Stp
PS21	slate	x	x	x	x		
PS22	slate	x	x	x	x		Stp
PS24	slate	x	x	x	x		Lws

*samples used in Figure 1
Mineral abbreviations are from Kretz (1983) except: C/S - chlorite-smectite; I/S - illite-smectite; K/S - kaolinite-smectite; KWM - K-white mica; ML - mixed-layer mineral;

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