Appendix E

X-Ray Fluorescence Spectrometry Data

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The samples and artifacts were ground into powders by Brent Miller at the University of North Carolina at Chapel Hill using an aluminum-oxide shatter box and shipped to MURR in powdered form. Although about 3 grams of powder were requested for each sample to conduct the three analytical procedures at MURR, not all of the samples had this amount of material available. After aliquots totaling 500 mg were set aside for NAA and ICP-MS, the remainder (typically 2.5 grams) was used for x-ray fluorescence spectrometry (XRF). In a few samples, particularly among the artifacts, the amount left over for XRF was quite small, on the order of 1 gram.

The sample aliquots designated for XRF were used to make loss-on-ignition (LOI) measurements prior to preparation of the samples for XRF spectroscopy. The samples in glass vials were dried for 24 hours in an oven at 105°C before they were transferred into clean pre-weighed crucibles with weights recorded to the nearest 0.0001 gram. After cooling the samples for 30 minutes, the total weight of the sample and crucible were also recorded. The crucible and sample were placed in a furnace operating at 500°C for a period of four hours. The crucible and sample were removed and placed in a dessicator to fully cool. About two hours later, the crucible and ashed sample were reweighed. The percentage of LOI was then calculated.

The ashed samples were then mixed in equal parts with SpectroCertified X-Ray Mix Binding Powder Cat. No. 600 from Chemplex Industries. Mixing was performed on a Spex 8000 Mixer/Mill using a mixing time of 15 minutes. The blended mixtures of sample and binding powder were poured into 32 mm aluminum planchets with a stainless steel pellet die and placed under 25 tons of pressure. The Spectro X-Lab 2000 spectrometer produces chemical analyses of geological materials using the energy-dispersive XRF based on polarized or near monochromatic x-rays for optimal sample excitation. The X-Lab 2000 spectrometer used to perform these analyses incorporates an end-window x-ray tube that can be focused on various secondary targets to produce polarized x-rays. Using the combination of different targets, typical detection limits for the light elements (Si, Al, Mg, and Na) are in the range of 25-50 ppm. Limits of detection for the heavy elements are in the 1-5 ppm range. The Spectro X-lab 2000 spectrometer was factory calibrated using a number of international rock standards (Korotev 1996).

The recommended amount of rock sample to mix powder for proper measurement on the Spectro X-Lab 2000 is about 5.0 grams. However, several of the samples were limited to about only 0.5 grams of sample (i.e., total mass of 1.0 grams). The light mass samples made necessary development of a separate correction method after they were measured on the XRF. An experiment was conducted using a series of samples made by mixing USGS Rock Standard RGM-1 Rhyolite in equal parts with the binding powder (i.e., 0.5, 1.0, 1.5, 2.0, and 2.5 grams each of sample and binding powder). The USGS rock samples were measured under the same

conditions as the unknowns in this study. A correction to normalize the data from the USGS rock samples to a total of 100% minus LOI was found to be successful for all elements except Ba. Ba concentrations were always high, and by calculating the ratio of the normalized USGS RGM-1 rock to the certified value for USGS RGM-1, an acceptable correction factor was determined for Ba.

The XRF measurements resulted in data for 21 elements, namely Na, Mg, Al, Si, K, Ca, Ti, Mn, Fe, Cu, Zn, Ga, Rb, Sr, Y, Zr, Nb, Ba, Pb, Th, and U. The data were tabulated with EXCEL and with the major elements converted to percent oxides and the trace elements listed in parts per million. The XRF results are presented in Tables E.1-E.2.

Table E.1. Major Element Concentrations as Measured by X-Ray Fluorescence.

Sample	LOI (%)	Na_2O (%)	MgO (%)	Al_2O_3 (%)	SiO_2 (%)	K_2O (%)	CaO (%)	TiO_2 (%)	MnO (%)	Fe_2O_3 (%)
FBL001	0.083	5.88	0.000	13.26	74.23	3.484	0.8667	0.1198	0.0693	1.5846
FBL002	0.124	6.38	0.000	12.96	75.36	3.321	0.2868	0.1145	0.0350	1.1844
FBL003	0.288	4.95	0.000	13.37	74.59	3.887	0.3954	0.1192	0.0747	1.7775
FBL004	0.034	7.22	0.000	13.29	75.55	2.427	0.2586	0.1098	0.0318	1.2161
FBL005	-0.074	6.57	0.000	12.51	76.20	2.862	0.2204	0.0888	0.0181	1.2047
FBL006	-0.244	7.05	0.000	12.70	75.68	2.234	0.2810	0.1126	0.0567	1.5487
FBL007	0.133	5.05	0.000	11.30	78.18	3.946	0.1672	0.1142	0.0305	1.0263
FBL008	0.209	5.52	0.183	11.47	77.70	2.664	0.5864	0.1564	0.0356	1.7471
FBL009	0.310	5.21	0.241	11.66	76.93	3.279	0.4434	0.1578	0.0517	1.8468
FBL010	0.291	5.88	0.392	11.37	77.44	2.190	0.3062	0.1526	0.0515	1.8007
FBL011	0.332	5.81	0.171	13.06	75.04	2.985	0.4302	0.1631	0.0367	1.8685
FBL012	0.484	6.96	0.277	13.06	73.68	2.604	0.4904	0.1780	0.0370	1.9382
FBL013	0.295	7.61	0.223	11.38	76.91	1.442	0.1865	0.1474	0.0526	1.8732
FBL014	0.341	5.74	0.383	15.14	71.42	3.191	1.0300	0.2335	0.0962	2.3953
FBL015	0.508	6.62	0.097	12.43	75.82	2.673	0.3572	0.1178	0.0497	1.2768
FBL016	-0.124	5.79	0.226	13.43	74.88	3.926	0.2351	0.1007	0.0625	1.4276
FBL017	0.295	5.89	0.000	12.90	75.89	3.169	0.3699	0.1089	0.0539	1.3827
FBL018	0.269	6.05	0.000	12.73	75.90	3.176	0.4632	0.1040	0.0442	1.4605
FBL019	0.221	6.11	0.000	12.35	76.57	2.421	0.3538	0.1038	0.0617	1.5878
FBL020	0.245	3.50	0.684	14.76	73.33	2.438	1.8233	0.2925	0.0773	2.7422
FBL021	0.303	5.62	0.424	12.73	75.42	2.801	0.8004	0.2317	0.0751	1.3753
FBL022	0.222	4.31	0.268	13.35	75.12	2.615	1.3203	0.2811	0.1240	2.0429
FBL023	0.166	4.92	0.091	11.64	78.60	1.359	1.1540	0.1856	0.0798	1.7190
FBL024	0.184	5.97	0.461	12.00	75.60	0.722	1.9274	0.2168	0.0788	2.3007
FBL025	0.322	4.55	0.000	12.30	76.21	4.387	0.2199	0.1266	0.0402	1.2554
FBL026	0.305	5.42	0.000	12.73	75.87	3.836	0.5397	0.1151	0.0454	1.1734
FBL027	0.494	2.49	0.322	10.39	79.67	4.618	0.5354	0.1696	0.0215	1.0791
FBL028	0.483	0.54	0.891	13.84	71.66	8.624	0.6038	0.2890	0.0988	2.2037
FBL029	0.641	2.59	0.804	11.22	76.51	4.617	0.7217	0.2926	0.0984	2.0260
FBL030	0.402	5.81	0.853	13.13	74.00	1.438	1.6343	0.3180	0.0980	2.1754
FBL031	0.204	5.41	0.099	12.02	74.47	4.122	0.3570	0.2009	0.0439	2.6870
FBL032	0.238	6.80	0.061	12.57	73.70	3.181	0.6985	0.1681	0.0439	2.4577
FBL033	0.232	6.06	0.091	12.41	73.81	3.946	0.3122	0.1639	0.0308	2.4347
FBL034	0.210	5.70	0.117	12.44	74.02	3.808	0.5301	0.1793	0.0646	2.5356
FBL035	1.176	3.75	3.021	16.76	63.85	3.224	1.8767	0.6200	0.1205	5.4675
FBL036	0.300	8.13	0.379	14.44	70.11	3.171	0.5505	0.3241	0.0372	2.7163
FBL037	0.834	1.43	2.499	14.73	65.32	4.531	4.6353	0.6367	0.1045	5.4385
FBL038	1.281	6.28	3.334	17.59	57.36	3.277	1.6458	0.7078	0.1660	8.0967
FBL039	0.229	4.58	0.000	13.95	75.85	4.707	0.0502	0.1526	0.0070	0.5355
FBL040	1.704	4.85	5.054	15.93	52.84	0.556	5.9895	1.3581	0.2080	11.8456
FBL041	1.840	5.69	5.588	20.27	54.50	1.208	1.5661	0.7413	0.1696	8.6345
FBL042	1.444	4.48	7.591	17.59	49.32	1.338	7.3508	0.6803	0.2285	9.4170
FBL043	0.787	9.07	1.949	16.11	64.53	1.005	2.0666	0.5892	0.0913	3.8764
FBL044	0.713	7.59	0.878	12.32	75.02	0.560	0.3180	0.2846	0.0657	2.2337
FBL045	0.856	7.79	1.278	14.81	68.78	1.097	1.2377	0.5258	0.0763	3.3633
FBL046	0.654	5.20	1.451	10.08	75.67	1.038	1.1717	0.4712	0.0715	3.8333
FBL047	0.642	7.18	1.253	12.37	73.45	1.144	0.8967	0.3162	0.0903	2.3509
FBL048	0.595	9.83	1.154	15.20	68.00	0.040	1.6969	0.3737	0.0757	2.9183
FBL049	0.593	8.39	1.080	13.88	70.87	1.194	1.1670	0.3534	0.0766	2.4593
FBL050	0.523	8.40	0.682	13.91	70.49	0.710	2.5056	0.3571	0.0803	2.3322
FBL051	0.312	4.35	0.000	11.19	79.21	3.480	0.1779	0.1117	0.0290	1.1887
FBL052	0.384	2.51	0.051	6.91	86.41	2.737	0.1528	0.0857	0.0296	0.7751
FBL053	0.273	4.57	0.000	12.25	77.40	3.516	0.1980	0.1070	0.0410	1.3318
FBL054	0.361	1.78	0.000	10.93	80.98	3.687	0.1344	0.0974	0.0350	1.5066
FBL055	0.425	6.84	0.409	12.41	75.79	1.813	0.7372	0.2402	0.0578	1.4947
FBL056	0.598	0.44	0.482	10.20	80.14	6.474	0.2812	0.2818	0.0375	1.2684
FBL057	0.494	6.21	0.555	13.31	73.60	3.557	0.4470	0.1990	0.0506	1.2894

Table E.1. Major Element Concentrations as Measured by X-Ray Fluorescence (continued).

Sample	LOI (%)	Na_2O (%)	MgO (%)	Al_2O_3 (%)	SiO_2 (%)	K_2O (%)	CaO (%)	TiO_2 (%)	MnO (%)	Fe_2O_3 (%)
FBL058	0.263	5.76	0.129	12.41	74.19	4.029	0.5296	0.1779	0.0563	2.4903
FBL059	0.276	4.07	0.413	13.46	72.70	5.634	0.6688	0.1945	0.0489	2.5977
FBL060	0.273	6.12	0.096	12.83	75.43	3.418	0.6291	0.1240	0.0360	1.1665
FBL061	0.430	6.32	0.178	13.16	74.37	3.424	0.7703	0.1340	0.0368	1.3119
FBL062	0.382	6.18	0.091	13.07	74.79	3.461	0.6557	0.1236	0.0283	1.1689
FBL063	0.282	5.77	0.153	12.51	76.29	3.591	0.4604	0.0981	0.0179	1.0737
FBL064	0.630	4.64	0.188	12.87	75.35	4.032	0.6781	0.1118	0.0413	1.1773
FBL065	0.289	6.15	0.250	13.46	74.00	3.642	0.5630	0.1077	0.0285	1.2442
FBL066	0.484	8.24	0.901	14.80	70.51	0.536	1.1654	0.4154	0.0869	3.0956
FBL067	1.135	6.67	2.497	15.89	63.09	1.226	3.4280	0.6767	0.1290	4.8699
FBL068	0.451	7.96	0.621	11.02	77.36	0.299	0.7528	0.2455	0.0352	1.5539
FBL069	0.480	6.89	0.536	13.33	72.13	2.699	0.7144	0.3803	0.0561	2.4524
FBL070	1.334	2.90	8.083	18.84	47.87	0.367	8.8655	0.9673	0.2086	10.6005
FBL071	1.872	2.71	6.089	18.12	49.69	1.032	8.7490	0.9117	0.1459	10.1513
FBL072	0.428	6.98	0.000	12.42	75.54	1.757	0.5730	0.1345	0.0665	2.0896
FBL073	0.486	7.17	0.216	13.04	73.98	2.309	0.8804	0.2152	0.0690	1.5076
FBL074	0.397	6.76	0.225	12.92	73.40	3.332	1.0884	0.1615	0.0409	1.7036
FBL075	1.089	5.38	2.645	15.59	63.06	1.540	3.8232	0.8575	0.1532	5.4919
FBL076	0.546	7.32	0.394	12.58	74.26	1.973	0.5412	0.2270	0.0858	1.9630
FBL077	0.412	7.29	0.106	12.05	77.01	1.736	0.0742	0.1447	0.0382	1.3417
FBL078	0.226	5.96	0.174	12.89	74.19	2.281	1.2817	0.2884	0.0580	2.5405
FBL079	0.374	5.29	0.451	14.33	71.10	4.125	1.5660	0.2600	0.0985	2.4181
FBL080	0.226	6.72	0.143	12.21	74.16	2.531	1.5529	0.1220	0.0691	1.6950

Table E.2. Trace Element Concentrations as Measured by X-Ray Fluorescence.

Sample	Cu (ppm)	Zn (ppm)	Ga (ppm)	Rb (ppm)	Sr (ppm)	Y (ppm)	Zr (ppm)	Nb (ppm)	Ba (ppm)	Pb (ppm)	Th (ppm)	U (ppm)
FBL001	2.8	41.6	16.2	90.2	53.7	59.1	187.6	9.1	568.8	20.4	8.6	3.2
FBL002	3.1	57.4	14.8	84.3	39.0	58.2	170.1	5.6	529.5	20.3	10.3	3.3
FBL003	4.8	69.7	17.3	120.0	59.9	79.5	187.1	7.4	746.0	17.6	10.9	4.2
FBL004	1.7	12.1	16.1	70.6	31.2	54.4	186.1	8.7	476.6	11.5	12.0	4.4
FBL005	5.0	50.6	17.6	67.8	39.8	69.3	146.6	7.3	486.3	21.3	12.9	3.9
FBL006	4.2	49.3	16.1	61.1	54.6	77.4	193.2	7.4	658.8	18.2	11.2	3.6
FBL007	2.1	40.8	9.9	97.0	45.6	41.3	149.1	6.0	616.0	7.7	10.2	3.7
FBL008	5.7	61.3	14.3	80.8	66.1	69.4	228.7	4.7	388.8	14.3	9.3	2.7
FBL009	5.2	57.1	14.0	86.2	69.8	63.3	200.7	5.5	406.6	11.3	8.2	4.0
FBL010	7.9	70.3	12.0	56.0	36.4	64.3	219.1	4.7	431.2	26.8	8.9	4.7
FBL011	6.0	27.7	15.7	91.0	61.4	71.3	249.1	2.6	529.1	12.1	10.6	2.3
FBL012	5.3	27.9	17.5	70.1	55.2	71.7	216.8	6.9	402.2	15.3	12.8	3.2
FBL013	0.0	31.5	14.3	36.6	18.3	60.8	234.3	3.3	246.6	16.2	11.7	5.8
FBL014	5.2	75.8	18.9	101.0	126.4	58.3	235.2	3.4	647.9	37.5	8.1	2.3
FBL015	4.5	29.2	12.3	60.3	54.5	45.0	140.9	5.6	505.3	10.2	12.0	2.3
FBL016	4.9	42.3	16.1	128.6	37.1	60.8	144.6	5.7	579.1	15.5	10.3	3.6
FBL017	4.2	44.4	13.2	92.8	68.9	61.2	172.8	5.4	729.7	25.7	12.8	4.9
FBL018	4.8	34.5	14.5	100.8	59.9	62.6	157.5	4.5	598.4	14.4	13.2	4.8
FBL019	10.9	45.6	16.0	79.0	64.5	64.8	178.3	5.8	662.3	14.8	14.1	4.2
FBL020	15.2	57.3	12.9	69.3	203.8	37.0	161.1	1.7	564.8	21.0	9.6	3.2
FBL021	3.0	48.2	10.3	57.2	194.3	47.7	213.5	4.5	570.0	10.4	4.5	0.0
FBL022	24.4	50.2	21.9	66.5	295.9	76.8	360.5	9.7	1083.4	19.6	8.0	4.7
FBL023	2.0	20.1	14.0	31.5	128.5	42.3	180.6	4.5	707.6	8.0	3.9	3.3
FBL024	2.5	46.8	14.8	13.8	214.8	34.9	160.7	3.1	268.8	14.9	4.3	1.7
FBL025	10.0	28.9	12.9	120.1	46.5	52.1	152.2	13.3	1163.2	16.2	14.9	7.3
FBL026	7.1	41.6	11.6	107.1	54.2	46.1	133.7	9.2	917.6	25.3	11.4	5.5
FBL027	4.0	33.4	9.1	141.0	64.5	47.5	124.7	10.6	990.3	10.7	14.6	5.1
FBL028	4.2	56.9	18.0	338.4	108.5	44.3	181.0	16.2	2303.1	26.8	17.1	5.7
FBL029	10.7	53.8	14.3	165.6	145.8	44.4	162.5	9.7	738.3	21.9	12.9	3.3
FBL030	9.0	37.7	16.6	71.8	389.5	124.9	300.1	16.3	548.9	26.7	21.3	6.9
FBL031	4.1	68.2	17.0	117.6	66.9	70.5	540.3	18.3	78.0	24.3	13.6	6.3
FBL032	2.5	40.2	19.0	77.5	90.7	68.5	507.5	17.2	45.3	18.1	13.4	5.5
FBL033	3.2	103.5	19.6	103.8	47.6	71.8	538.3	17.7	39.7	21.2	13.1	2.3
FBL034	2.0	79.4	16.2	107.8	85.3	77.2	566.1	16.6	70.9	23.7	13.0	3.9
FBL035	21.0	67.0	18.3	57.1	188.6	24.6	161.0	3.7	875.1	13.2	5.6	0.0
FBL036	14.2	47.5	12.4	35.3	71.1	22.5	222.5	3.7	584.1	13.6	7.2	4.0
FBL037	59.2	75.6	18.2	107.0	399.3	28.7	167.3	2.9	985.0	16.7	7.4	0.0
FBL038	120.3	91.4	17.8	70.8	254.0	15.8	75.4	0.0	1180.9	9.2	1.1	0.0
FBL039	4.2	20.4	17.6	87.8	44.7	44.8	168.2	7.4	301.3	23.9	11.8	2.3
FBL040	15.3	117.8	19.2	8.0	378.2	23.8	60.0	0.0	198.4	5.1	0.0	0.0
FBL041	9.7	99.4	14.3	34.7	371.0	20.2	54.6	0.0	418.9	4.6	0.0	0.0
FBL042	196.2	86.4	15.5	29.0	324.2	15.5	36.4	0.0	233.2	7.3	0.0	0.0
FBL043	5.3	72.2	15.9	17.2	583.7	31.6	208.7	4.1	438.2	10.5	8.6	5.2
FBL044	5.2	66.3	14.6	14.9	164.4	43.2	195.8	4.8	105.9	16.2	3.9	2.1
FBL045	4.3	63.6	10.6	22.3	565.8	25.9	180.2	3.0	500.1	7.0	6.3	2.3
FBL046	20.3	59.2	10.2	11.8	277.7	18.6	116.2	1.8	286.2	11.0	3.2	3.9
FBL047	16.1	51.1	12.3	9.8	235.7	21.1	159.5	1.2	435.9	13.9	2.4	0.0
FBL048	15.2	60.9	13.2	1.3	383.4	28.7	209.5	2.9	44.2	17.6	2.7	1.8
FBL049	3.3	53.3	10.3	13.1	278.9	27.4	213.6	1.9	374.2	8.7	2.9	2.9
FBL050	3.9	39.8	14.3	7.8	377.9	27.4	197.3	1.7	272.4	10.8	2.6	1.4
FBL051	4.7	38.1	7.7	106.5	71.1	34.2	121.9	5.6	730.4	13.7	10.4	0.0
FBL052	11.2	39.1	6.1	83.7	48.1	32.1	107.8	3.6	618.8	18.3	8.2	6.5
FBL053	3.5	36.3	9.1	111.0	56.3	41.5	159.6	6.7	785.9	13.8	10.7	3.4
FBL054	6.6	45.7	7.6	129.9	65.3	44.4	157.7	4.5	1160.3	23.9	12.5	5.8
FBL055	15.4	39.5	8.9	35.9	92.5	29.0	115.6	2.4	414.5	17.9	3.6	2.3
FBL056	50.8	52.7	11.9	201.1	42.4	25.9	105.8	6.4	676.5	22.2	6.1	4.8
FBL057	5.8	30.4	10.1	89.6	86.7	67.8	152.3	10.9	689.5	4.2	14.6	3.0

Table E.2. Trace Element Concentrations as Measured by X-Ray Fluorescence (continued).

Sample	Cu (ppm)	Zn (ppm)	Ga (ppm)	Rb (ppm)	Sr (ppm)	Y (ppm)	Zr (ppm)	Nb (ppm)	Ba (ppm)	Pb (ppm)	Th (ppm)	U (ppm)
FBL058	5.2	96.1	20.1	104.6	75.7	75.2	579.7	15.8	58.9	21.0	14.4	3.3
FBL059	10.9	114.9	16.2	155.0	99.3	68.3	559.8	15.4	111.3	22.8	13.3	3.5
FBL060	3.0	37.2	13.4	80.8	76.7	39.9	126.9	4.8	498.8	11.8	6.3	2.2
FBL061	5.3	36.3	14.5	88.1	82.9	42.1	138.6	8.0	512.7	11.5	7.5	2.2
FBL062	4.1	25.5	13.9	80.5	81.8	38.1	122.0	5.3	521.0	9.8	7.5	3.4
FBL063	2.8	18.4	11.9	86.7	47.5	43.9	118.0	5.3	547.8	10.3	7.4	2.9
FBL064	5.2	36.0	13.5	85.2	41.4	41.5	120.1	5.3	544.6	10.2	6.4	3.0
FBL065	4.7	29.7	17.2	80.4	74.6	51.7	143.5	5.9	605.0	9.4	7.3	2.5
FBL066	34.7	71.8	16.2	7.2	220.6	34.1	278.3	4.2	163.2	8.6	3.1	2.9
FBL067	17.2	72.3	16.7	15.4	304.6	27.3	164.8	3.4	382.6	8.9	2.1	0.0
FBL068	13.3	36.9	8.1	6.9	190.0	42.4	240.8	6.1	140.4	7.6	6.2	2.1
FBL069	9.0	38.2	10.8	39.3	88.1	22.4	167.3	0.0	558.3	18.8	6.4	1.6
FBL070	30.9	84.7	20.7	8.8	633.0	6.5	39.4	0.0	102.5	17.8	0.0	0.0
FBL071	27.1	74.2	16.9	14.1	505.7	10.1	22.8	0.0	155.4	6.1	0.0	0.0
FBL072	5.4	30.6	14.9	69.8	126.8	84.7	264.1	4.0	722.4	11.0	9.6	8.0
FBL073	9.8	28.0	23.0	80.0	311.8	117.4	382.2	0.0	1214.4	18.2	12.8	5.2
FBL074	4.2	43.9	9.5	112.4	113.6	46.3	226.0	7.4	706.0	17.4	10.3	2.0
FBL075	4.7	90.4	19.1	35.1	409.9	35.3	138.5	1.4	506.4	9.8	0.0	0.0
FBL076	5.7	60.8	11.9	54.1	158.6	54.7	239.2	8.0	696.9	19.0	10.5	4.6
FBL077	2.6	43.9	11.3	33.0	65.7	40.7	181.0	4.7	459.6	9.3	7.8	4.4
FBL078	0.0	48.1	14.1	72.4	121.0	49.6	383.1	9.1	578.0	22.2	15.7	3.5
FBL079	4.5	19.3	16.8	151.3	253.1	68.4	302.2	8.0	1048.7	21.6	12.4	0.0
FBL080	3.4	34.9	20.6	66.1	175.4	69.3	227.5	3.6	707.9	12.4	11.3	3.5