Title
ENERGY-DISPERSIVE X-RAY FLOURESCENCE (EDXRF) ANALYSIS OF MAJOR, MINOR AND TRACE ELEMENTS FOR VOLCANIC ROCKS FROM GRAND CANYON-PARASHANT NATIONAL MONUMENT, UTAH

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ENERGY-DISPERSIVE X-RAY FLOURESCENCE (EDXRF) ANALYSIS OF MAJOR, MINOR AND TRACE ELEMENTS FOR VOLCANIC ROCKS FROM GRAND CANYON-PARASHANT NATIONAL MONUMENT, UTAH

by

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Report Prepared for

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INTRODUCTION

The analysis here of 62 rock samples indicates a large diversity of rock types, based on the TAS plot and bivariate plots of four trace elements (Tables 1 and 2, Figures 1 and 2). While basalt is a common rock type, it is not the dominant volcanic in the assemblage based on this analysis (see Discussion).

LABORATORY SAMPLING, ANALYSIS AND INSTRUMENTATION

All archaeological samples are analyzed whole. Each analyzed sample was selected based on size and a flat presentation to the instrument (Shackley 2011a). Each sample was subjected to high pressure air to remove any dust matrix from the rock, particularly with vesicular samples.

The results presented here are quantitative in that they are derived from "filtered" intensity values ratioed to the appropriate x-ray continuum regions through a least squares fitting formula rather than plotting the proportions of the net intensities in a ternary system (McCarthy and Schamber 1981; Schamber 1977). Or more essentially, these data through the analysis of international rock standards, allow for inter-instrument comparison with a predictable degree of certainty (Hampel 1984; Shackley 2011a). The

All analyses for this study were conducted on a ThermoScientific *Quant'X* EDXRF spectrometer, located at the Geoarchaeological XRF Laboratory, Albuquerque, New Mexico. The spectrometer is equipped with a thermoelectrically Peltier cooled solid-state Si(Li) X-ray detector, with a 50 kV, 50 W, ultra-high-flux end window bremsstrahlung, Rh target X-ray tube and a 76 µm (3 mil) beryllium (Be) window (air cooled), that runs on a power supply operating from 4-50 kV/0.02-1.0 mA at 0.02 increments. The spectrometer is equipped with a 200 l min^{-1} Edwards vacuum pump, allowing for the analysis of lower-atomic-weight elements between sodium (Na) and titanium (Ti). Data acquisition is accomplished with a pulse processor and an analogue-to-digital converter. Elemental composition is identified with digital filter background removal, least squares empirical peak deconvolution, gross peak intensities and net peak intensities above background.

The data from the WinTrace™ software were translated directly into Excel for Windows software for manipulation and on into JMP 12.0.1 for Windows or IGPET ver. 2013 for plotting. In order to evaluate these quantitative determinations, machine data were compared to measurements of known standards during each run. BCR-2 a USGS basalt (basaltic andesite) standard was analyzed during each run of ≤20 samples. The USGS recommended values are included in Table 1. Trace element data exhibited in Table 1 is reported in weight percent and Table 2 in parts per million (ppm), a quantitative measure by weight.

Trace Element Analysis

In the analysis of mid Zb condition elements Ti-Nb, and high Zb Pb, Th, the x-ray tube is operated at 30 kV, using a 0.05 mm (medium) Pd primary beam filter in an air path at 100 seconds livetime to generate x-ray intensity $K\alpha_1$-line data for elements manganese (Mn), cobalt (Co), nickel (Ni), copper, (Cu), zinc, (Zn), gallium (Ga), rubidium (Rb), strontium (Sr), yttrium (Y), zirconium (Zr), niobium (Nb), and $L\alpha_1$ data for lead (Pb), and thorium (Th). Trace element intensities were converted to concentration estimates by employing a linear calibration line ratioed to the Compton scatter established for each element from the analysis of international rock standards certified by the National Institute of Standards and Technology (NIST), the US. Geological Survey (USGS), Canadian Centre for Mineral and Energy Technology, and the Centre de Recherches Pétrographiques et Géochimiques in France (Govindaraju 1994). Line fitting is linear (XML) for all elements. When barium (Ba) is analyzed in the High Zb condition, the Rh tube is operated at 50 kV and up to 1.0 mA, ratioed to the bremsstrahlung region (see Davis 2011; Shackley 2011a). Further details concerning the petrological choice of these elements in Southwest obsidians is available in Shackley (1988, 1995, 2005; also Mahood and Stimac 1991;

and Hughes and Smith 1993). Nineteen specific pressed powder standards are used for the best fit regression calibration for elements Ti-Nb, Pb, Th, and Ba, and include G-2 (basalt), AGV-2 (andesite), GSP-2 (granodiorite), SY-2 (syenite), BHVO-2 (hawaiite), STM-1 (syenite), QLO-1 (quartz latite), RGM-1 (obsidian), W-2 (diabase), BIR-1 (basalt), SDC-1 (mica schist), TLM-1 (tonalite), SCO-1 (shale), NOD-A-1 and NOD-P-1 (manganese) all US Geological Survey standards, NIST-278 (obsidian), U.S. National Institute of Standards and Technology, BE-N (basalt) from the Centre de Recherches Pétrographiques et Géochimiques in France, and JR-1 and JR-2 (obsidian) from the Geological Survey of Japan (Govindaraju 1994).

Major and Minor Oxide Analysis

Analysis of the major oxides of Na, Mg, Al, Si, P, K, Ca, Ti, V, and Fe is performed under the multiple conditions elucidated below. This fundamental parameter analysis (theoretical with standards), while not as accurate as destructive analyses (pressed powder and fusion disks) is usually within a few percent of actual, based on the analysis of the USGS BCR-2 basaltic-andesite standard, the analysis is relatively accurate in the elements of interest (Table 1). The fundamental parameters (theoretical) method is run under conditions commensurate with the elements of interest and calibrated with 11 USGS standards (RGM-1, rhyolite; AGV-2, andesite; BHVO-1, hawaiite; BIR-1, basalt; G-2, granite; GSP-2, granodiorite; BCR-2, basalt; W-2, diabase; QLO-1, quartz latite; STM-1, syenite), and one Japanese Geological Survey rhyolite standard (JR-1). The USGS BCR-2 basaltic andesite standard used in this analysis is a relatively new variety from the Bridal Veil quarry approximately 29 miles east of Portland, Oregon in the Cascade Range, collected in 1996, and recently released. It is a more recent version of the original BCR-1 that is now expended (see https://crustal.usgs.gov/geochemical_reference_standards/basaltbcr2.html)

Conditions Of Fundamental Parameter Analysis[1]:

Low Za (Na, Mg, Al, Si, P)

Voltage	6 kV	Current	Auto[2]
Livetime	100 seconds	Counts Limit	0
Filter	No Filter	Atmosphere	Vacuum
Maximum Energy	10 keV	Count Rate	Low

Mid Zb (K, Ca, Ti, V, Cr, Mn, Fe)

Voltage	32 kV	Current	Auto
Livetime	100 seconds	Counts Limit	0
Filter	Pd (0.06 mm)	Atmosphere	Vacuum
Maximum Energy	40 keV	Count Rate	Medium

High Zb (Sn, Sb, Ba, Ag, Cd)

Voltage	50 kV	Current	Auto
Livetime	100 seconds	Counts Limit	0
Filter	Cu (0.559 mm)	Atmosphere	Vacuum
Maximum Energy	40 keV	Count Rate	High

Low Zb (S, Cl, K, Ca)

Voltage	8 kV	Current	Auto
Livetime	100 seconds	Counts Limit	0
Filter	Cellulose (0.06 mm)	Atmosphere	Vacuum
Maximum Energy	10 keV	Count Rate	Low

[1] Multiple conditions designed to ameliorate peak overlap identified with digital filter background removal, least squares empirical peak deconvolution, gross peak intensities and net peak intensities above background.

[2] Current is set automatically based on the mass absorption coefficient.

Discussion

While the number of basalt samples is relatively small compared to the assemblage overall, a remarkable character of this assemblage of rocks is the similarity in the proportion of the alkalis, likely a result of similar magma sampling in the region overall (Table 1 and Figure 1). Isotopic analyses could be useful in this regard (see Shackley et al. 2018). The variability in the trace elements may be more revealing (see Shackley 2011b; Table 1 and Figure 2 here).

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Table 1. Major, and minor elemental concentrations for the rock samles, and USGS BCR-2 . All measurements in weight percent (%) as noted.

Sample	**Na2O**	**MgO**	**Al2O3**	**SiO2**	**P2O5**	**K2O**	**CaO**	**TiO2**	**V2O5**	**MnO**	**Fe2O3**
	%	%	%	%	%	%	%	%	%	%	%
DELL-140520-01	3.15	1.37	11.18	39.33	1.01	1.81	16.03	2.66	0.16	0.37	22.34
140513-01	3.96	2.04	9.15	30.06	1.11	1.74	13.38	5.52	0.22	0.40	30.90
140513-02	0.56	28.01	9.01	51.37	0.00	0.12	1.32	0.00	0.01	0.14	9.18
140513-03	3.31	6.82	15.66	39.73	0.54	1.47	11.45	3.61	0.11	0.22	16.56
140513-04	0.44	40.68	4.20	40.56	0.00	0.18	3.43	0.05	0.02	0.17	9.87
140513-05	2.50	2.51	8.33	30.29	0.98	2.50	16.01	5.19	0.21	0.43	29.99
140513-06	2.25	1.73	9.23	33.38	1.11	1.49	17.63	3.94	0.18	0.47	27.72
140513-07	3.32	3.56	19.04	46.55	0.45	1.17	10.67	2.23	0.11	0.20	12.28
140514-01	2.92	6.04	17.48	46.81	0.34	1.06	10.03	2.08	0.10	0.21	12.57
140514-02	3.18	4.55	18.63	48.60	0.24	1.01	10.09	1.71	0.12	0.20	11.39
140514-03	3.43	3.35	7.72	33.74	1.13	1.27	15.16	3.57	0.21	0.42	29.09
140514-04	2.82	1.07	9.02	28.90	0.95	1.64	13.60	4.27	0.15	0.55	35.65
140514-05	3.41	4.11	19.57	46.82	0.35	1.13	10.39	1.90	0.08	0.19	11.60
140514-06	2.49	1.53	10.68	37.64	0.89	1.71	17.16	3.28	0.18	0.33	23.16
140514-07	3.14	4.98	17.09	48.91	0.43	1.15	11.38	1.75	0.11	0.17	10.56
140514-08	2.29	2.55	8.45	32.82	1.02	1.55	14.88	4.07	0.18	0.46	30.76
140515-01	3.70	1.94	9.66	31.48	1.39	1.80	13.34	4.27	0.20	0.47	30.67
170920-01	2.48	0.88	10.94	39.95	0.84	1.98	16.78	2.64	0.22	0.37	22.37
170920-02	2.19	1.77	8.69	32.96	0.96	2.67	13.69	5.36	0.18	0.37	30.55
170920-03	3.07	0.72	6.79	29.46	1.11	2.13	12.93	6.59	0.20	0.43	35.76
170921-01	2.56	1.92	8.84	32.91	0.94	2.37	15.57	4.67	0.11	0.40	28.84
170921-02	2.69	2.48	9.90	37.04	1.19	2.26	18.16	3.68	0.14	0.32	21.42
170922-01	2.64	7.30	17.95	46.76	0.57	1.10	10.66	1.56	0.05	0.18	10.92
170922-02	4.00	0.59	10.62	40.05	1.22	2.21	13.86	4.07	0.17	0.30	22.28
170922-03	2.66	2.27	9.49	31.92	1.20	2.34	17.52	4.05	0.25	0.39	26.49
170922-04	2.37	1.13	10.63	36.32	0.78	2.83	15.12	4.54	0.22	0.34	24.93
170922-05	2.75	1.27	9.19	34.93	0.76	2.68	15.17	4.33	0.21	0.38	27.66
170923-01	2.76	2.36	8.70	33.86	1.12	1.79	17.83	3.50	0.13	0.42	26.57
170923-02	2.64	1.02	10.64	35.31	1.31	1.80	17.46	3.20	0.11	0.40	25.05
170923-03	2.87	2.19	9.55	35.67	1.17	1.71	16.47	3.27	0.13	0.36	25.64
170923-04	2.68	2.17	9.64	36.08	1.23	1.85	17.33	3.03	0.17	0.35	24.63
170923-05	2.89	2.01	9.99	37.04	1.01	2.46	16.47	3.27	0.13	0.36	23.65
170923-06	3.23	8.42	16.32	47.17	0.40	1.30	9.02	1.88	0.09	0.19	11.64
170923-07	2.43	10.38	17.26	41.85	0.38	0.80	10.44	1.89	0.10	0.22	13.92
170925-01	3.21	2.20	9.29	33.15	1.11	2.18	17.18	4.50	0.19	0.33	25.91
170925-02	2.75	3.40	7.88	36.81	0.94	1.62	17.31	3.09	0.21	0.28	24.81
170925-03	3.61	4.53	10.10	37.93	1.26	3.16	14.52	3.18	0.14	0.46	20.27

Sample	Na2O	MgO	Al2O3	SiO2	P2O5	K2O	CaO	TiO2	V2O5	MnO	Fe2O3
170925-04	3.35	8.08	16.52	44.13	0.54	1.61	9.57	2.59	0.16	0.20	12.83
170925-05	3.06	8.18	17.00	43.41	0.60	1.55	10.95	2.57	0.14	0.18	11.98
170926-01	2.92	1.65	9.66	36.73	0.97	1.60	15.60	3.25	0.24	0.39	26.34
170926-02	2.71	2.80	8.36	34.26	1.22	1.51	15.71	3.18	0.19	0.44	28.48
170926-03	2.31	2.21	9.28	33.94	1.06	1.91	15.70	3.76	0.14	0.46	28.22
170926-04	2.98	2.21	9.15	33.53	1.02	1.30	16.05	3.52	0.11	0.42	28.68
170928-01	3.03	1.77	9.46	38.93	0.94	1.55	15.10	2.64	0.11	0.37	25.64
170928-02	2.82	6.18	17.56	48.84	0.23	0.91	8.48	1.51	0.08	0.20	12.99
170928-03	2.36	1.32	9.95	37.72	0.83	1.38	16.95	2.97	0.17	0.39	25.45
170929-01	3.90	2.59	10.98	40.89	1.30	1.30	15.46	2.73	0.19	0.32	19.61
170929-02	2.99	0.97	11.11	41.89	1.13	1.65	16.88	2.27	0.15	0.30	19.88
170929-03	2.59	2.52	8.93	31.57	1.30	1.46	19.76	3.38	0.20	0.42	26.73
170929-04	3.08	8.30	16.21	46.70	0.31	1.06	9.53	1.93	0.08	0.18	12.37
170930-01	2.83	2.67	8.74	31.04	1.44	2.10	21.11	3.16	0.19	0.47	24.83
171002-01	2.73	2.54	8.04	32.85	1.25	2.48	18.74	3.92	0.19	0.37	25.98
171002-02	2.71	2.64	8.33	30.22	1.16	2.16	17.78	4.96	0.15	0.43	28.22
171002-03	2.44	2.85	7.74	32.55	1.08	2.87	16.84	4.40	0.29	0.40	27.56
171002-04	3.31	1.41	9.36	34.94	0.98	2.68	14.61	3.63	0.14	0.40	27.83
171002-05	2.40	2.00	8.48	33.12	1.06	2.08	16.51	3.88	0.21	0.39	29.26
171003-01	2.87	0.91	10.66	38.23	0.89	3.21	16.51	3.29	0.23	0.33	22.25
171003-02	2.90	1.71	8.62	32.93	1.31	1.92	19.46	3.68	0.13	0.41	25.70
171003-03	3.38	3.76	18.30	48.81	0.35	1.49	8.82	2.00	0.11	0.18	12.56
171004-01	2.72	2.62	8.43	32.12	1.06	2.26	17.23	4.30	0.20	0.40	27.58
171005-01	3.19	1.37	9.87	37.26	1.01	2.49	16.02	3.37	0.14	0.36	24.25
171005-02	3.18	1.87	9.68	38.21	1.15	2.62	16.99	2.96	0.18	0.35	22.15
BCR-2	3.22	2.71	14.24	53.81	0.09	1.90	7.11	2.47	0.16	0.20	13.91
BCR-2	3.30	2.70	14.11	53.82	0.11	1.90	7.11	2.41	0.19	0.21	13.97
BCR-2	3.28	2.84	14.33	53.63	0.10	1.89	7.10	2.46	0.14	0.19	13.86
BCR-2	3.13	2.54	14.24	53.91	0.03	1.92	7.15	2.47	0.14	0.21	14.09
BCR-2 recommended	3.16	3.59	13.50	54.10	0.35	1.79	7.12	2.26	nr[1]	nr	13.80

[1] nr = not reported

Table 2. Trace element concentrations for the rock samples and USGS BCR-2 basaltic andesite standard.

Sample	Cl	Mn	Co	Ni	Cu	Zn	Ga	Rb	Sr	Y	Zr	Nb	Ba	Pb	Th
	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
DELL-140520-01	0	1367	12	23	87	105	20	18	508	29	154	26	1053	3	4
140513-01	424	1263	14	43	52	138	21	32	1114	27	224	64	1770	3	8
140513-02	62	1373	37	657	18	51	8	0	30	7	22	1	37	0	4
140513-03	675	1334	21	55	71	119	23	26	887	23	226	60	1250	8	7
140513-04	0	1230	36	534	0	57	11	0	34	7	19	5	36	0	4
140513-05	911	1417	31	55	54	133	21	19	868	18	223	72	1187	0	12
140513-06	0	1218	21	44	128	104	19	18	610	17	150	39	1200	7	7
140513-07	0	1366	26	26	112	99	23	16	891	33	179	50	1750	4	14
140514-01	0	1325	18	38	85	102	19	16	684	25	157	36	1419	3	4
140514-02	0	1399	21	40	83	98	19	11	642	25	141	42	1220	3	4
140514-03	0	1315	37	87	97	127	20	13	548	20	145	39	1070	2	4
140514-04	68	1184	10	33	61	101	18	21	556	30	146	34	1191	8	10
140514-05	0	1273	9	22	118	93	23	17	790	33	158	44	1885	7	6
140514-06	285	1125	10	28	72	85	21	13	739	20	152	39	1541	6	6
140514-07	0	1189	10	25	101	88	17	14	773	31	153	42	1671	7	15
140514-08	0	1282	22	54	67	113	18	20	568	24	150	25	1057	9	4
140515-01	317	1413	28	56	77	144	20	19	609	25	151	37	1054	3	4
170920-01	0	1020	18	27	60	91	19	14	411	22	125	24	742	4	4
170920-02	0	1283	25	53	53	155	20	22	667	20	205	45	532	3	4
170920-03	0	1143	47	48	71	142	19	19	647	22	192	47	525	0	4
170921-01	0	1280	31	65	47	130	20	20	746	25	197	62	1062	7	7
170921-02	527	1407	27	65	78	180	22	19	861	28	179	53	1501	8	4
170922-01	0	1212	28	62	78	89	20	12	690	21	151	41	1063	1	4
170922-02	0	1111	14	21	72	169	24	14	610	24	181	43	887	5	6
170922-03	190	1305	31	62	58	102	20	13	1053	26	203	57	2188	2	9
170922-04	0	1138	37	32	58	94	21	22	685	24	199	39	1124	5	4
170922-05	0	1158	28	28	34	111	24	20	579	31	174	36	749	6	5
170923-01	0	1189	40	79	88	90	19	9	670	25	144	43	1093	3	11
170923-02	0	1167	26	54	78	91	21	14	657	26	158	46	1441	4	4
170923-03	0	1154	24	78	98	92	19	10	698	24	146	54	1171	2	12
170923-04	0	1165	38	80	71	105	21	14	719	24	156	49	1112	2	4
170923-05	0	1163	30	47	68	109	25	22	693	29	178	40	888	0	4
170923-06	0	1426	42	72	58	112	20	22	795	29	185	54	1314	0	4
170923-07	0	1489	36	88	74	93	17	8	603	22	123	37	1127	5	4
170925-01	0	1273	30	59	63	106	17	17	676	19	178	52	1068	5	4
170925-02	270	1018	29	110	50	128	17	27	774	26	237	56	871	7	4
170925-03	326	1274	11	80	74	78	17	7	731	25	222	49	796	11	4

Sample	Cl	Mn	Co	Ni	Cu	Zn	Ga	Rb	Sr	Y	Zr	Nb	Ba	Pb	Th
170925-04	153	1405	33	83	73	121	19	22	1001	29	253	76	1442	4	18
170925-05	0	1288	35	67	62	92	23	26	865	30	237	67	1310	2	9
170926-01	0	1272	34	42	75	101	24	17	441	21	138	25	713	0	4
170926-02	0	1273	35	85	152	122	16	13	671	17	139	38	1240	9	4
170926-03	287	1395	24	77	56	102	21	13	791	24	163	52	1315	12	15
170926-04	0	1348	23	63	74	106	18	5	740	24	143	44	1291	7	4
170928-01	0	1115	34	45	56	97	20	22	294	24	116	13	499	2	4
170928-02	0	1431	30	58	56	100	20	18	304	23	115	12	494	5	4
170928-03	426	1108	42	35	69	100	20	17	318	23	115	14	467	6	5
170929-01	0	1117	33	51	56	124	21	10	743	18	150	45	1368	7	6
170929-02	0	981	13	27	76	87	17	16	458	26	122	24	1377	7	6
170929-03	280	1454	25	64	144	91	20	11	816	25	162	57	1628	5	4
170929-04	135	1454	34	84	76	100	18	17	509	24	153	30	759	1	4
170930-01	594	1392	32	63	60	91	19	26	1072	24	217	79	1992	10	8
171002-01	0	1345	39	72	61	113	17	20	780	30	231	67	1167	4	5
171002-02	650	1522	22	67	61	116	24	23	1135	25	310	90	1651	9	4
171002-03	392	1425	51	90	98	136	20	39	727	28	221	52	1163	2	6
171002-04	466	1158	28	40	46	105	19	30	480	24	172	27	545	2	4
171002-05	0	1169	51	79	66	125	19	26	512	21	182	33	517	0	4
171003-01	0	1009	22	22	52	92	21	43	608	25	188	36	894	10	20
171003-02	366	1197	20	46	76	94	17	19	817	27	187	55	1541	5	18
171003-03	0	1265	33	34	47	105	20	15	565	20	179	33	861	6	9
171004-01	0	1370	31	73	69	107	19	15	881	22	200	57	1453	5	7
171005-01	0	1014	24	29	62	85	21	16	527	19	158	30	687	6	5
171005-02	0	1027	13	41	96	90	19	17	521	27	163	27	660	5	7
BCR-2	0	1487	29	20	9	125	22	52	345	36	170	13	812	5	4
BCR-2	0	1549	17	21	27	118	23	53	338	36	169	13	850	5	8
BCR-2	0	1463	18	21	24	119	21	46	335	37	163	21	854	16	4
BCR-2	0	1508	35	23	21	121	22	50	337	39	170	16	790	5	4
BCR recommended[1]	nr	1520	37	nr	19	127	23	48	346	37	188	nr	683	11	6.2

[1] Barium values have been consistently high for newer "preliminary" rock standards at USGS (Shackley 2011).

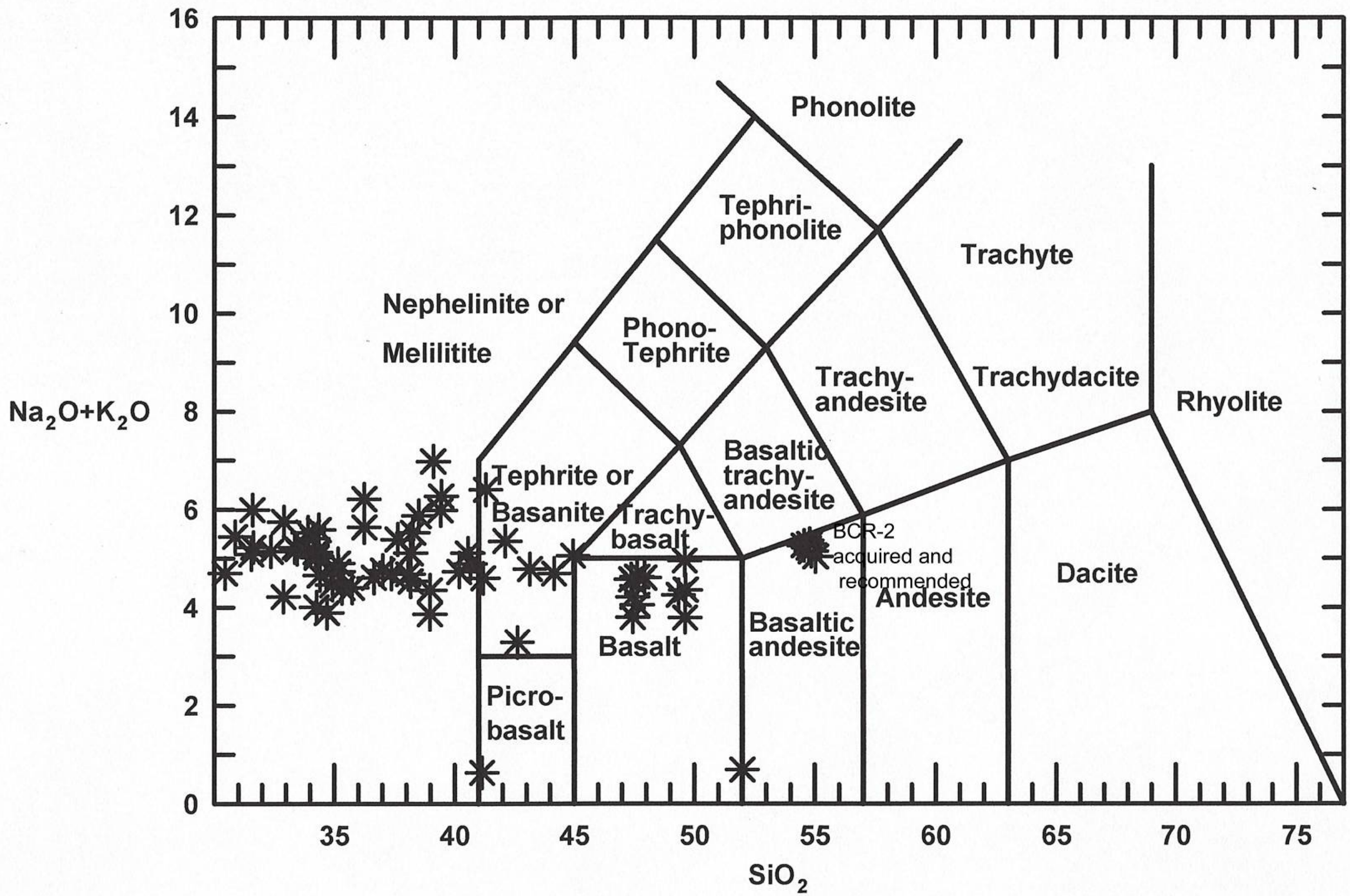

Figure 1. TAS plot of the rock samples and USGS BCR-2 and recommended values for BCR-2 (Le Maitre et al. 1989).