

Sample preparation

Lava samples were cut with a diamond saw, and the sawn samples cleaned with water and dried. Approximately 100 g of each sample was crushed in a manganese steel jaw crusher and then powdered in a tungsten carbide Gyromill to produce a fine, visually homogeneous powder.

Tephra samples were sieved to remove size fractions smaller than medium ash (<1 mm), washed with water to remove fine particles of soil and organic matter, and dried at 90°C. Hand-selected clasts were then crushed and powdered in preparation for XRF analysis. Glassy fragments in the medium to coarse ash size fraction were hand-picked and mounted in epoxy resin for analysis by electron microprobe.

X-ray fluorescence (XRF) analysis

X-ray fluorescence (XRF) analyses were carried out in the Grant Institute of Earth Sciences at the University of Edinburgh. The techniques used are similar to those described by *Fitton et al.* (1998) with modifications given by *Fitton and Godard* (2004).

Major element concentrations were determined after fusion with a lithium tetraborate flux containing La_2O_3 as a heavy absorber. Sample powders were dried overnight at 110°C. A nominal but precisely weighed 1 g aliquot of sample powder was weighed into a Pt5%Au crucible and ignited in a muffle furnace at 1100°C for 20 minutes to determine the mass loss on ignition (LOI). The residue was then mixed with Johnson Matthey SpectrofluxTM105 in a sample:rock ratio of 1:5 based on the unignited sample mass, and fused at 1100°C for a further 20 minutes. After initial fusion, the crucible was reweighed and any flux weight loss was made up with additional flux. After a second fusion over a Meker burner, the sample was swirled several times to ensure a homogeneous mixture. The molten sample was then cast onto a graphite mould and flattened into a thin disc with an aluminium plunger. The mould and plunger were maintained at 220°C on a hotplate.

Trace element concentrations were determined on pressed powder pellets. Eight grams of sample powder were mixed thoroughly with 8 drops of a 2% aqueous solution of polyvinyl alcohol. The mixture was loaded into a 40 mm diameter aluminium cup in a stainless steel die, and compressed against a polished tungsten carbide disc in a hydraulic press at 0.6 tons/cm².

The fused discs and pressed powder samples were analysed on a Philips PW2404 automatic X-ray spectrometer with a Rh-anode end-window X-ray tube, using the analytical conditions given by *Fitton et al.* (1998) and *Fitton and Godard* (2004). Long count times were used at both peak and background positions. Trace element background positions were placed as close as possible to peaks. For most trace element determinations, background count rates were measured on either side of the peak, with the count time divided evenly between the two positions.

	BHVO-1				BIR-1			
	R	E	1σ	%P	R	E	1σ	%P
SiO ₂ (wt.TiO ₂)	2.71	2.73	0.01	0.19	0.96	0.94	0.00	0.18
Al ₂ O ₃	13.80	13.68	0.08	0.57	15.50	15.53	0.05	0.30
Fe ₂ O ₃	12.23	12.36	0.02	0.15	11.30	11.37	0.01	0.05
MnO	0.17	0.17	0.00	1.13	0.18	0.17	0.00	0.34
MgO	7.23	7.17	0.04	0.54	9.70	9.51	0.02	0.17
CaO	11.40	11.46	0.03	0.26	13.30	13.21	0.02	0.15
Na ₂ O	-	-	-	-	1.82	1.80	0.01	0.72
K ₂ O	0.52	0.53	0.00	0.42	-	-	-	-
P ₂ O ₅	0.27	0.29	0.01	2.59	-	-	-	-
<i>n</i>		14				4		

Table 1: Precision estimates for major element concentrations in international standards measured by XRF spectrometry at the University of Edinburgh. R is the recommended value from *Govindaraju* (1994). E is the mean, 1σ the standard deviation and %P the percentage precision of *n* measurements carried out at the University of Edinburgh.

Corrections for matrix effects on the intensities of major element lines were made using theoretical alpha coefficients calculated using the Phillips software. The coefficients were calculated to allow for the additional flux replacing volatile components in the sample, such that the analytical totals should be 100% less the measured LOI. Matrix corrections were applied to the intensities of longer-wavelength trace element lines (La, Ce, Nd, Cu, Ni, Co, Cr, V, Ba, Sc) using alpha coefficients based on major element concentrations measured at the same time on the pressed powder samples. The intensities of the remaining trace elements were corrected for matrix effects by using the count rate from the Rh-K α Compton scatter line as an internal standard (*Reynolds*, 1963). Line overlap corrections were applied using synthetic standards.

The spectrometer was calibrated against USGS and CRPG standards using the values given by *Govindaraju* (1994) for all elements, with the exception of Nb and Zr in BCR-1 and BHVO-1, where the values of *Jochum et al.* (1990) were used. These standards produced excellent calibration lines. The precision and accuracy are comparable to the values reported by *Fitton et al.* (1998) and *Fitton and Godard* (2004). Four USGS standards were analysed alongside the Iceland samples, and the data from tables 1 and 2) can be used to assess accuracy and precision. Further discussion of the precision and accuracy of XRF data from the University of Edinburgh can be found in *MacLennan et al.* (2003) and *Passmore et al.* (2012).

	BHVO-1				BCR-1				BIR-1			
	R	E	1 σ	%P	R	E	1 σ	%P	R	E	1 σ	%P
La (ppm)	15.8	14.7	2.10	14.29	24.9	24.9	0.70	2.81	0.62	0.9	1.10	122.2
Ce	39	39.2	0.60	1.53	53.7	53.7	1.30	2.42	1.95	1	1.80	180.0
Rb	11	9.1	0.27	2.97	47.2	47.1	1.20	2.55	0.25	0.2	0.13	65.0
Ba	139	135.2	3.22	2.38	681	681.7	2.88	0.42	7	8.2	0.70	8.54
Th	1.08	1.4	0.50	35.71	5.98	6.1	0.40	6.56	0.03	nd		
Pb	2.6	2.5	0.50	20.00	13.6	14	0.40	2.86	3	nd		
Nb	19.8	19.8	0.05	0.25	13.6	13.0	0.07	0.54	0.6	0.6	0.14	23.3
Sr	403	396.4	0.94	0.24	330	336.2	1.72	0.51	108	107.5	1.16	1.08
Zr	182	175.0	0.10	0.06	186	192.3	0.60	0.31	15.5	16.2	0.19	1.17
Y	27.6	27.3	0.09	0.33	38	38.0	0.43	1.13	16	16.3	0.15	0.92
Zn	105	105.3	0.42	0.40	129.5	130.4	0.31	0.24	71	66.7	0.60	0.90
Cu	136	132.0	0.54	0.41	19	19.7	0.35	1.78	126	130.0	0.40	0.31
Sc	31.8	33.0	0.88	2.67	32.6	33.5	0.39	1.16	44	42.3	0.51	1.21
V	317	311.4	1.48	0.48	407	403.0	2.20	0.55	313	323.3	2.37	0.73
Cr	289	292.5	2.58	0.88	16	9.7	6.54	67.42	382	385.7	5.19	1.35
Ni	121	124.5	0.96	0.77	13	15.8	4.25	26.90	166	160.3	2.76	1.72
Nd	25.2	26.4	0.80	3.03	28.8	28.6	1.00	3.50	2.5	2.0	1.00	50.0
<i>n</i>	<i>6</i>				<i>4</i>				<i>10</i>			

Table 2: Precision estimates for trace element concentrations in international standards measured by XRF at the University of Edinburgh, compared with recommended values. R is the recommended value from *Govindaraju* (1994), except Nb and Zr in BHVO-1 and BCR-1, which are from *Jochum et al.* (1990). E is the mean, 1 σ the standard deviation and %P the percentage precision of *n* measurements carried out at the University of Edinburgh.

Electron Microprobe (EPMA)

Major and selected minor element compositions of mineral phases and quenched glass were determined by WDS Cameca SX100 electron microprobe at the University of Edinburgh. A review of electron microprobe methods is given by *Reed* (2005). The samples were polished and carbon-coated. Concentrations of major and selected minor elements were determined in WDS mode with a peak count time of between 20 and 60 seconds depending on the abundance of the element within the mineral or glass under analysis. The background count time was half the peak count time, or equal to the peak count time for minor elements such as phosphorous. Quantitative WDS analysis uses a least-squares profile-fitting technique. Elementary peak profiles from calibration standards were determined and stored at the beginning of every session. Internal data reduction was performed using the inbuilt Cameca X-Phi PeakSight software for glass analyses, or PAP corrections for mineral analyses. Details of the analytical conditions used are given in *Hayward* (2012).

To maintain data quality, analyses with totals outwith the range 98-101 wt.% total oxides were rejected. Repeat analyses of international and internal standards were made at the beginning and end of every session to monitor instrumental drift and to assess the instrumental precision and accuracy (Table 3).

	BHVO-2G				UoE-Lipari*				St John's Island Olivine*				Orthoclase*			
	R	E	1 σ	%P	R	E	1 σ	%P	R	E	1 σ	%P	R	E	1 σ	%P
SiO ₂ (wt.%)	49.90	49.76	0.40	0.80	74.03	73.43	0.38	0.51	40.82	40.84	0.13	0.32	64.46	64.99	0.17	0.27
TiO ₂	2.73	2.79	0.02	0.59	0.08	0.08	0.01	7.93	-	-	-	-	-	-	-	-
Al ₂ O ₃	13.50	13.62	0.17	1.28	12.72	13.18	0.23	1.74	-	-	-	-	18.22	17.87	0.23	1.28
FeO	11.07	11.04	0.17	1.50	1.75	1.14	0.08	6.87	9.55	9.67	0.12	1.26	-	0.53	0.01	1.31
MnO	0.17	0.17	0.01	4.11	0.08	0.07	0.00	3.45	0.13	0.13	0.01	4.02	-	-	-	-
MgO	7.23	7.21	0.09	1.21	0.00	0.04	0.00	7.72	49.19	48.78	0.17	0.36	-	-	-	-
CaO	11.40	11.26	0.15	1.33	0.72	1.07	0.11	9.94	0.05	0.04	0.00	6.26	-	-	-	-
Na ₂ O	2.22	2.29	0.07	3.15	4.06	4.07	0.06	1.50	-	-	-	-	2.21	2.00	0.12	6.00
K ₂ O	0.52	0.51	0.02	3.93	5.18	4.96	0.03	0.55	-	-	-	-	15.11	15.09	0.07	0.44
P ₂ O ₅	0.27	0.24	0.01	4.52	-	-	-	-	-	-	-	-	-	-	-	-
SrO	-	-	-	-	-	-	-	-	-	-	-	-	-	0.11	0.01	7.73
BaO	-	-	-	-	-	-	-	-	-	-	-	-	-	0.02	0.00	0.00
NiO	-	-	-	-	-	-	-	-	0.43	0.35	0.01	2.26	-	-	-	-
<i>n</i>		<i>77</i>				<i>5</i>				<i>9</i>				<i>10</i>		

Table 3: Precision estimates for electron microprobe analyses. R is the recommended value for each standard. E is the mean, 1 σ the standard deviation and %P the percentage precision of *n* measurements carried out at the University of Edinburgh during this study. * denotes internal standard.

Inductively coupled plasma mass spectrometry (ICP-MS)

Selected samples were prepared for ICP-MS analysis at Scottish Universities Environmental Research Centre (SUERC), East Kilbride, using a HF/HNO₃-HNO₃-HCl tri-acid digestion procedure similar to that of *Olive et al.* (2001). A nominal but precisely weighed 0.1 g of each sample powder was weighed into a clean 15 ml PFA Teflon screw-top beaker, and 2 ml of Analar concentrated HF and approximately 0.2 ml of Primar concentrated (50%) HNO₃ was added to each beaker. The beakers were covered and the samples refluxed overnight at a temperature of 90°C, after which time the powder had been digested. The sample solutions were left to evaporate to near dryness. Then 2 ml of Primar concentrated HNO₃ was added to the residue, and the samples were covered, left to reflux overnight at 90°C and evaporated to near dryness. Next, 0.5 ml of Primar concentrated (50%) HCl was added to the residue. Samples were covered and allowed to reflux for 2 hours, then evaporated to near dryness. The samples were then picked up with 5 ml of 5% HNO₃ and transferred to 100 ml volumetric flasks. The flasks were completed with 5% HNO₃ and allowed to stand overnight, before decanting into polythene bottles which were sealed for storage. At this stage the samples were at 1:1000 dilution. Procedural blank solutions and reference standard solutions of BCR-1 and BCR-2 were prepared alongside the sample batches using the same method.

The sample solutions were analysed at SUERC using a VG Elemental PQ2 Plus quadrupole-based ICP-MS fitted with a Meinhard nebuliser and a water-cooled glass Scott double-pass spray chamber. The acquisition parameters are given in Table 4. For REE analysis, the instrument was calibrated using an internal standard synthetic ‘ α ’ solution containing the fourteen REEs and Ba (*Olive et al.*, 2001). Prior to each run, the α solution and dilutions of x2, x5, x10 and x20 were used to construct a calibration curve, fitted using the linear regression option in the instrument software (PQ Vision 4.1.2, Fisons Instruments). Calibrations were accepted for correlation coefficients (r) >0.99. Additional elements U, Th, Ta, Hf and Pb were analysed in a separate run using a different internal standard calibration solution.

Basaltic samples were diluted by a further factor of 10 to 1:10,000 to prevent any drop in sensitivity due to saturation in the plasma. At this stage all calibration, standard and sample solutions were spiked with 0.01 ml of ¹¹⁵In (200 ppb) and ¹⁰²Ru (200 ppb), plus 0.01 ml of ¹⁸⁵Re (500 ppb) for REEs or 0.01 ml of ²⁰⁹Bi (200 ppb) for U, Th, Ta, Hf and Pb. The spikes enabled any variations in sensitivity to be monitored throughout the analytical run. The ICP-MS was washed with dilute HNO₃ between each analysis.

Every fourth sample analysed consisted of the standard solution BCR-1, and BCR-2 comprised every twelfth sample. A correction was applied to all the analyses such that each analysis of BCR-1 was normalised to agree with the accepted standard composition of BCR-1. This correction was also applied to BCR-2, and thus analyses of BCR-2 analyses can be used as an estimate of instrumental precision and accuracy. Data from repeat analyses of BCR-2 (10 repeats) and unknown sample M5 (3 repeats) are presented in Table 5.

Sample uptake rate	0.8 ml min ⁻¹
Washout time	180 s
Uptake time	90 s
Acquisition time	90 s
Internal standards	¹⁰² Ru; ¹¹⁵ In; ¹⁸⁵ Re or ²⁰⁹ Bi
Data acquisition mode	Peak jumping
Dwell time	10.24 ms
Points per peak	3
No. of replicates	3

Table 4: ICP-MS acquisition parameters.

	BCR-1				M5		
	R	E	1 σ	%P	E	1 σ	%P
La (ppm)	24.9	23.42	0.24	1.01	6.64	0.19	2.89
Ce	53.7	50.34	0.55	1.08	16.43	0.30	1.85
Pr	6.8	6.38	0.08	1.27	2.34	0.05	2.14
Nd	28.8	27.05	0.30	1.12	11.09	0.19	1.75
Sm	6.59	6.18	0.09	1.49	3.14	0.04	1.42
Eu	1.95	1.87	0.02	0.83	1.14	0.01	0.63
Gd	6.68	6.29	0.11	1.74	3.59	0.03	0.90
Tb	1.05	0.99	0.01	1.08	0.63	0.00	0.19
Dy	6.34	5.98	0.09	1.44	3.99	0.01	0.18
Ho	1.26	1.19	0.02	1.33	0.82	0.01	0.94
Er	3.63	3.43	0.05	1.58	2.34	0.04	1.51
Tm	0.56	0.53	0.00	0.88	0.36	0.00	0.48
Yb	3.38	3.18	0.03	1.03	2.17	0.00	0.10
Lu	0.51	0.48	0.01	1.15	0.33	0.01	2.66
Hf	4.950	4.850	0.086	1.78	2.433	0.489	20.1
Ta	0.810	0.793	0.058	7.36	1.509	0.504	33.4
Pb	13.600	9.955	1.137	11.42	0.068	0.371	546.4
Th	5.980	5.884	0.169	2.88	0.601	0.147	24.4
U	1.750	1.714	0.057	3.33	0.204	0.039	19.3
<i>n</i>			10			3	

Table 5: Precision estimates for ICP-MS analyses. R shows the recommended values for BCR-2. Three repeats of Askja sample M5 were also analysed to assess precision. E is the mean, 1 σ the standard deviation and %P the percentage precision of *n* measurements carried out at the Scottish Universities Environmental Research Centre during this study.

Secondary ion mass spectrometry (SIMS)

Rare earth and selected trace element concentrations in matrix glasses and olivine- and plagioclase-hosted melt inclusions were determined by secondary ion mass spectrometry (SIMS) on the Cameca ims-4f ion microprobe at the University of Edinburgh. The polished samples were gold-coated and then bombarded with a 15 kV primary beam of O^- ions. Positive secondary ions were accelerated to 4500 V, with an offset of 75 ± 5 V to suppress molecular ion interference. The beam current was maintained at 5 ± 1 nA and rastered over a 20 ± 5 μm area. Only inclusions >25 μm in diameter were analysed.

The following isotopes were analysed in each cycle of a 10-cycle run, with counting time in seconds given in parentheses: ^{26}Mg (2), ^{30}Si (2), ^{39}K (2), ^{49}Ti (2), ^{85}Rb (3), ^{88}Sr (3), ^{89}Y (5), ^{90}Zr (3), ^{93}Nb (5), ^{138}Ba (5), ^{139}La (5), ^{140}Ce (3), ^{141}Pr (5), ^{143}Nd (5), ^{149}Sm (8), ^{151}Eu (5), ^{154}Gd (3), ^{156}Gd (3), ^{157}Gd (5), ^{159}Tb (5), ^{161}Dy (5), ^{165}Ho (5), ^{167}Er (5), ^{169}Tm (5), ^{171}Yb (5), ^{175}Lu (5). Peak positions were verified before each analysis. Mass 130.5 was measured as background for 5 s in each cycle and was always zero. Counts were normalised to ^{30}Si . The Si content of each melt inclusion was previously determined by electron microprobe, and these values were used in the calculation of absolute elemental concentrations in the melt inclusions. Oxide interference was monitored by measurement of $^{154}\text{BaO}/\text{Ba}$ and $^{156}\text{CeO}/\text{Ce}$. Neither ratio varied systematically with time, but both exhibited minor dependence on the MgO content of the melt inclusions. Average values for the melt inclusions were $^{154}\text{BaO}/\text{Ba} = 0.088 \pm 0.018$ (1σ) and $^{156}\text{CeO}/\text{Ce} = 0.8 \pm 0.1$. The value of $^{156}\text{CeO}/\text{Ce}$ was used to estimate an effective offset voltage for each analysis, the value of which was used in the calculation of interference corrections. The average offset voltage was 65 ± 5 V.

Ion yields were assessed by repeat analyses of synthetic standard glass NIST-SRM610. Ion yields from the first morning of analytical work were used to calibrate all the melt inclusion and glass data. Repeat analyses were performed each morning and on at least one further occasion each day. These repeats were used to correct subsequent data for any systematic drift, which reached 3-5% for the light REE and up to 10% for the heavy REE.

Precision and accuracy were monitored by repeat analyses of USGS standards BCR-2G and BIR-1G, whose compositions bracketed the observed range of unknown compositions. For BCR-2G, precision was better than 5% for Mg, Ti, Sr and Y, 5-15% for Zr and the majority of the REE, and 10-15% for K, Rb, Nb, Ba, Gd, Tb, Tm and Yb. Precision estimates for BIR-1G were better than 3% for Mg, K, Ti, Sr, Y, Zr, Ba, Pr and Er, 5-7% for Ce, Sm, Nd, Eu and Tb, 10-15% for La, Gd, Ho, Tm and Lu, and 20-30% for Nb, Dy and Yb (Table 6). The Rb precision is very poor for BIR-1G. The elemental concentrations obtained were consistently between 10 and 20% lower than the accepted standard concentrations for both BCR-2G and BIR-1G, and therefore a systematic correction was applied to the concentrations of the unknown samples by dividing by average analysed concentrations in BCR-2G and normalising to the accepted concentrations of BCR-2G. When this correction is applied to the BIR-1G analyses the accuracy appears to be similar to the precision.

NIST-SRM610				BCR-2G				BIR-1G				
	R	E	1σ	%P	R	E	1σ	%P	R	E	1σ	%P
Si (ppm)	327210	327210	0.0	0.0	252880	252880	0.0	0.0	222030	222030	0.00	0.0
Mg	465	463	5.6	1.2	21649	21305	451.4	2.1	58374	55682	348.23	0.6
K	486	502	32.4	6.5	14810	11964	1568.3	13.1	183	150	4.32	2.9
Ti	434	432	10.1	2.3	13500	11783	439.7	3.7	5400	4960	74.71	1.5
Rb	425.7	428	38.7	9.0	47	35	5.3	15.1	0.197	0	0.41	11824
Ba	435	415	31.2	7.5	683	513	54.78	10.7	6.5	6.52	0.10	1.5
Nb	419	414	27.3	6.6	12.5	10	1.18	11.6	0.52	0.51	0.12	22.9
Sr	515.5	485	21.6	4.5	342	270	13.0	4.8	109	109	2.47	2.3
Zr	440	435	18.1	4.2	184	171	10.3	6.0	14	14.48	0.60	4.2
Y	450	440	16.0	3.6	35	30	1.4	4.5	14.3	16.54	0.57	3.5
La	457	447	25.7	5.7	24.7	21.2	1.45	6.8	0.609	0.68	0.10	14.4
Ce	448	439	27.5	6.3	53.3	42.8	2.77	6.5	1.89	1.95	0.12	6.0
Pr	430	420	25.9	6.2	6.7	5.3	0.46	8.7	0.37	0.43	0.00	0.4
Nd	431	437	28.8	6.6	28.9	24.5	1.92	7.8	2.37	2.81	0.19	6.7
Sm	451	445	28.9	6.5	6.59	5.4	0.39	7.1	1.09	1.13	0.07	5.8
Eu	461	467	31.6	6.8	1.97	2.5	0.21	8.2	0.517	0.37	0.02	6.7
Gd	444	434	29.6	6.8	6.71	5.3	0.64	12.0	1.85	1.59	0.20	12.3
Tb	443	434	31.6	7.3	1.02	0.9	0.13	14.2	0.35	0.33	0.02	6.3
Dy	427	421	29.4	7.0	6.44	5.7	0.46	8.1	2.55	2.56	0.70	27.2
Ho	449	439	32.0	7.3	1.27	1.2	0.11	8.6	0.56	0.51	0.05	10.2
Er	426	419	30.4	7.3	3.7	3.0	0.29	9.6	1.7	1.83	0.03	1.7
Tm	420	413	31.4	7.6	0.51	0.4	0.06	15.0	0.24	0.28	0.03	11.2
Yb	445	451	33.5	7.4	3.39	3.2	0.36	11.3	1.64	1.59	0.52	32.8
Lu	435	424	35.5	8.4	0.503	0.5	0.04	9.2	0.248	0.24	0.03	12.6
<i>n</i>		17				12				2		

Table 6: Precision estimates for trace and rare earth elements analysed by secondary ion mass spectrometry. R is the recommended value for each standard. E is the mean, 1 σ the standard deviation and %P the percentage precision of n measurements carried out at the University of Edinburgh during this study. The mean, standard deviation and precision values for BCR-2G and BIR-2G are for data that has been corrected for systematic analytical drift and normalised to the accepted concentrations of BCR-2G, as described in the text.

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