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# Data report: major and trace element geochemistry of upper oceanic crust at IODP Site C0012<sup>1</sup>

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## Abstract

This data report presents the bulk rock major and trace element compositions analyzed by inductively coupled plasma–atomic emission spectroscopy (ICP–AES) and inductively coupled plasma–mass spectrometry (ICP–MS) of the upper oceanic crustal rocks recovered during Integrated Ocean Drilling Program Expedition 333.

## Introduction

Integrated Ocean Drilling (IODP) Site C0012 in Shikoku Basin (Holes C0012E–C0012G) penetrated almost 100 m into igneous basement and recovered the sediment/basalt interface intact around 525 meters below seafloor (mbsf; Expedition 333 Scientists, 2011). The Shikoku Basin oceanic crust was formed by back-arc spreading during the time period of 15–25 Ma (Expedition 333 Scientists, 2011; Okino et al., 1994). The recovered igneous rocks are pillow lavas basalts and sheet flow basalts that were all moderately to heavily altered (Expedition 333 Scientists, 2011). The drilling cores provide the opportunity to conduct a systematic investigation of the in situ depth profile of the subducting uppermost igneous basement rocks before they reach the deformation front. This data report presents the bulk rock major and trace element compositions of the basaltic rocks and gabbros recovered during IODP Expedition 333.

## Methods and materials

A total of 19 representative rock samples were selected to constrain the Shikoku Basin basalts drilled during Expedition 333. Special care was taken to remove surface contamination by saw marks and altered rinds resulting from drilling by grinding off the outer surfaces on a diamond-impregnated disk. The cleaned rock blocks were then ultrasonicated in trace-metal grade methanol, deionized water, and Milli-Q water (18.2 M $\Omega$ ) to remove contamination during drilling and cutting. The cleaning procedures were done repeatedly until the silver nitrate solution test was negative to ensure the complete removal of seawater. The cleaned rocks were then dried for 10–12 h at 110°C. These dry and clean samples were fragmented to small chips by crushing them between two disks of Delrin plastic in a hydraulic press. The rock chips

<sup>1</sup>Gao, Y., and Casey, J.F., 2014. Data report: major and trace element geochemistry of upper oceanic crust at IODP Site C0012. *In* Henry, P., Kanamatsu, T., Moe, K., and the Expedition 333 Scientists, *Proc. IODP, 333*: Tokyo (Integrated Ocean Drilling Program Management International, Inc.). doi:10.2204/iodp.proc.333.204.2014

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were then ground to a fine powder in an aluminum ceramic mill.

Analysis for major and minor element concentrations (Si, Al, Mg, Fe, Mn, Ti, Ca, Na, K, and P) were conducted on a Thermo-Jarrell Ash sequential inductively coupled plasma–atomic emission spectrometer (ICP-AES) at the University of Houston (TX, USA) using methods described in Lytwyn and Casey (1993) and Smith (1994). Loss on ignition (LOI) was determined from the total weight change of the sample powder by heating in a Lindberg Model 51440 furnace at 1000°C for 30 min. After determination of LOI, 0.2000 ± 0.0002 g of the ashed (“ignited”) sample powder was mixed with 1.0000 ± 0.0002 g of lithium metaborate (Aldrich, 99.9% trace metal grade) in a high purity graphite fusion crucible (SCP Science) for the fusion process. The fusion process was conducted in a Lindberg Model 51440 furnace at 1125°C for 15 min. After 15 min, the molten bead was immediately poured into 100 mL of 1.5 N HNO<sub>3</sub> (made from double-distilled HNO<sub>3</sub>) for dissolution in a 150 mL Teflon beaker on a hot plate with magnetic stirring. After the molten bead completely dissolved, the solution was passed through Whatman No. 40 ashless filter paper to filter out any carbon residue. The filtered solutions were then further diluted with 1.5 N HNO<sub>3</sub> (made from double-distilled HNO<sub>3</sub>) to a dilution factor of 1:5000 for major element analysis with ICP-AES. The measured elemental abundances were calibrated against five international reference standards (AGV-1, BHVO-2, JGb-1, BIR-1, and W-2) and a LiBO<sub>2</sub> flux blank. Relative errors (precision and accuracy) monitored by repeated analysis of international reference standard BCR-2 are generally <1% to 5% for analyzed elements (Table T1).

For trace element (Li, Be, B, Sc, Ti, V, Cr, Co, Ni, Cu, Zn, Ga, Rb, Sr, Y, Zr, Nb, Cs, Ba, La, Ce, Pr, Nd, Sm, Eu, Tb, Gd, Dy, Ho, Er, Yb, Lu, Hf, Ta, Pb, Th, and U) analysis, powdered samples (without “ignition”) were digested with mixed acids in a clean room based on a slightly modified method described by Gao et al. (2009). All acids used (HNO<sub>3</sub>, HCl, and HF) were double-distilled, and ultrapure 18.2 MΩ Milli-Q water was used. Precisely weighed samples (100 mg) were placed into Savillex PFA beakers, and then 1 mL of 16 N HNO<sub>3</sub> and 2 mL of HF were added. The samples were then dried on a hot plate at about 150°C to evaporate the SiF<sub>4</sub>. Then 3 mL of 12 N HCl, 1 mL of 16 N HNO<sub>3</sub>, and 4 mL of 24 N HF were added to the beakers. The capped beakers were heated on a hot plate at about 180°C for at least 24 h. The samples were then dried to incipient dryness and refluxed with 4 mL of 8 N HCl. The samples were repeatedly

fluxed twice with 2 mL of 16 N HNO<sub>3</sub> to get rid of HF and HCl. After adding 4 mL of 8 N HNO<sub>3</sub>, the capped beakers were placed on a hot plate at temperatures about 100°C for 5–12 h so that the samples redissolved. After transferring the sample solutions into acid-cleaned low-density polyethylene bottles, a known amount of internal standard solution was added and then diluted with Milli-Q H<sub>2</sub>O to a dilution factor of 1:1000. The resulting solutions contain 2% HNO<sub>3</sub> and a nominal internal standard concentration of 10 ppb. The internal standards used were Rh, In, Tm, Re, Bi, and enriched isotopes <sup>6</sup>Li, <sup>61</sup>Ni, <sup>84</sup>Sr, and <sup>145</sup>Nd, the mass of which spaces through the entire mass spectrum of all the analytes. This multiple internal standards technique provides the ability to monitor and correct the complex mass-dependent fractionations encountered in inductively coupled plasma–mass spectrometry (ICP-MS) multi-element analysis (Gao et al., 2009, Eggins et al., 1997). The samples were then analyzed with a Varian 810 ICP-MS at the University of Houston.

For ICP-MS analysis, the data reduction was performed offline, for which both drift and oxide interference were corrected by external and internal standards (Gao et al., 2009). Analytical precision and accuracy monitored by repeated analysis of international reference standard JGb-1, which was processed along with the samples, are typically better than 5% (Table T1).

## Results

Results for major and trace element compositions are presented in Table T2.

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**Table T1.** Major and trace element compositions of BCR-2 and JGB-1 analyzed by ICP-AES and ICP-MS along with Expedition 333 samples.

Trace (mg/g)	JGB-1 (Ref)	JGB-1 (measured) (N = 6)	RSD (%) (N = 6)	Major (wt%)	BCR-2 (Ref)	BCR-2 (measured) (N = 4)	RSD (%) (N = 4)
Li	4.4	4.6	3.7	SiO <sub>2</sub>	54.1	53.9	0.1
Be	0.34	0.32	6.45	Al <sub>2</sub> O <sub>3</sub>	13.5	13.5	1.0
B	7.0	6.3	6.9	Fe <sub>2</sub> O <sub>3(t)</sub>	13.8	13.7	0.4
Sc	36.4	39.7	5.5	MgO	3.59	3.66	4.1
Ti	9513	9967	4	MnO	0.20	0.19	0.6
V	668	710	3	TiO <sub>2</sub>	2.26	2.25	1.5
Cr	64	58	2	CaO	7.12	7.10	1.1
Co	63.4	61.0	1.0	Na <sub>2</sub> O	3.16	3.17	1.8
Ni	25.3	26.4	0.5	K <sub>2</sub> O	1.79	1.76	2.4
Cu	79.6	83.3	2.0	P <sub>2</sub> O <sub>5</sub>	0.35	0.39	4.5
Zn	101.7	105.8	0.6	LOI	0.69	0.58	2.1
Ga	20.0	20.1	1.8				
Rb	5.9	5.7	1.3				
Sr	327	319	1				
Y	9.7	8.8	1.9				
Zr	28.5	25.0	0.7				
Nb	2.35	2.04	0.61				
Cs	0.20	0.22	2.92				
Ba	62.9	64.1	2.4				
La	3.49	3.53	1.46				
Ce	8.06	8.77	0.91				
Pr	1.11	1.22	2.17				
Nd	4.96	5.56	1.99				
Sm	1.44	1.50	0.94				
Eu	0.61	0.65	1.16				
Gd	1.63	1.77	0.89				
Tb	0.27	0.28	1.36				
Dy	1.69	1.81	1.19				
Ho	0.35	0.38	1.23				
Er	1.01	1.08	1.05				
Yb	0.95	1.07	1.32				
Lu	0.14	0.14	1.45				
Hf	0.81	0.81	1.22				
Ta	0.15	0.15	3.06				
Pb	1.59	1.65	1.05				
Th	0.42	0.43	6.59				
U	0.10	0.10	1.14				

Reference values for JGB-1 and BCR-2 are taken from GeoREM ([georem.mpch-mainz.gwdg.de](http://georem.mpch-mainz.gwdg.de))

Table T2. Major and trace element compositions of crust rocks, Site C0012. (Continued on next page.)

Hole:	C0012G	C0012G	C0012G	C0012G	C0012F	C0012G	C0012G	C0012G	C0012G	C0012E	C0012G	C0012G
Core, section, interval (cm):	15R-1, 81-84	13R-1, 58.5-62.5	2R-2, 119-123	9R-1, 35-39	1R-1, 58-62	11R-CC, 6-12	7R-1, 20-25	12R-3, 12-18	12R-1, 90-95	12-3X-8, 68-72	14R-3, 42-46	3R-CC, 17-21
Depth (mbsf):	626.34	616.13	526.12	591.39	520.62	601.12	572.25	613.50	611.45	526.82	623.72	534.82
Major elements (wt%):												
SiO <sub>2</sub>	50.9	49.1	53.1	49.7	49.9	52.4	50.7	48.3	50.9	51.3	50.5	48.4
Al <sub>2</sub> O <sub>3</sub>	16.2	14.8	14.8	15.1	15.7	13.5	14.9	14.4	15.0	16.1	14.4	18.5
Fe <sub>2</sub> O <sub>3</sub>	8.72	12.4	9.70	12.2	10.5	12.0	11.6	13.3	10.8	9.40	12.3	9.63
FeO <sub>t</sub>	7.84	11.13	8.73	11.02	9.49	10.82	10.45	11.95	9.74	8.45	11.09	8.67
MgO	8.82	7.61	7.45	6.40	7.95	7.20	6.38	6.69	6.06	6.40	6.88	6.52
MnO	0.15	0.28	0.26	0.37	0.55	0.21	0.24	0.32	0.29	0.34	0.26	0.24
TiO <sub>2</sub>	0.88	1.56	1.35	2.12	1.55	1.55	1.64	2.03	1.76	1.49	1.60	1.38
CaO	10.18	8.33	8.12	8.00	9.86	6.78	6.90	8.62	9.23	10.52	9.04	10.21
Na <sub>2</sub> O	3.71	2.02	2.57	3.23	3.25	2.75	7.11	2.82	3.40	3.63	3.15	4.45
K <sub>2</sub> O	0.37	3.74	2.43	2.66	0.53	3.35	0.39	3.30	2.38	0.67	1.71	0.51
P <sub>2</sub> O <sub>5</sub>	0.06	0.20	0.17	0.20	0.15	0.19	0.18	0.18	0.17	0.16	0.18	0.17
LOI	4.86	3.04	1.69	2.37	1.82	2.36	3.82	3.74	1.88	1.65	1.64	5.47
Mg#	67	55	60	51	60	54	52	50	53	57	53	57
Trace elements (ppm):												
Li	21.8	12.5	11.1	21.2	11.6	12.3	20.2	19.0	21.0	9.4	19.1	14.7
Be	0.36	0.35	0.36	0.48	0.36	0.53	0.36	0.47	0.47	0.33	0.39	0.35
B	14.7	22.1	19.5	16.8	16.5	22.7	18.2	12.9	9.9	10.6	11.3	20.7
Sc	70.5	46.8	59.5	55.4	64.0	47.1	63.6	57.5	51.6	57.4	49.6	56.9
Ti	5271	9378	8071	12716	9308	9320	9842	12158	10553	8913	9569	8281
V	269	371	339	448	365	343	368	432	356	346	357	331
Cr	391	75	182	78	171	74	105	83	80	141	80	163
Co	39.7	42.8	40.4	44.3	44.3	38.5	44.5	47.2	45.7	37.2	42.5	40.1
Ni	94.8	53.6	61.9	41.0	74.7	38.6	49.2	42.4	58.7	49.5	41.2	53.0
Cu	90.9	58.6	66.9	32.6	71.3	65.4	32.7	56.1	17.0	59.4	56.4	67.6
Zn	65.8	87.0	73.8	98.2	85.9	75.6	81.0	94.7	74.8	75.8	63.1	91.7
Ga	16.1	16.0	19.9	19.4	20.0	15.6	14.6	22.6	20.7	18.6	19.0	24.3
Rb	10.4	42.9	38.6	35.5	15.9	36.6	16.3	44.3	39.1	8.5	31.8	13.8
Sr	399	520	138	531	160	155	104	732	456	165	277	467
Y	22.5	38.3	32.4	43.4	31.7	36.7	36.4	42.4	33.9	30.4	34.8	31.7
Zr	48.8	93.9	74.8	123	80.9	89.9	96.1	118	98.2	82.8	94.0	77.5
Nb	0.95	1.79	1.43	2.27	1.65	1.70	1.81	2.11	1.81	1.61	1.76	1.53
Cs	3.15	0.46	0.57	0.28	0.37	0.25	3.91	0.31	0.61	0.21	0.22	5.40
Ba	65.1	1228	892	256	70.7	1081	67.7	259	59.6	128	46.2	94.4
La	2.32	3.98	3.72	4.72	4.08	4.15	3.90	4.15	3.88	3.49	4.13	3.28
Ce	6.85	12.8	11.1	15.2	12.1	12.8	12.9	13.8	12.8	11.2	12.9	10.4
Pr	1.22	2.26	1.94	2.76	2.08	2.20	2.22	2.46	2.24	1.98	2.25	1.86
Nd	6.35	12.2	10.3	13.9	10.9	11.6	12.0	13.5	11.7	10.5	12.0	9.8
Sm	2.19	4.03	3.51	4.79	3.62	3.86	4.09	4.43	3.92	3.55	3.91	3.33
Eu	0.89	1.51	1.32	1.72	1.35	1.39	1.44	1.63	1.46	1.29	1.41	1.25
Gd	3.08	5.53	4.76	6.26	4.79	5.17	5.30	6.10	5.33	4.68	5.27	4.56
Tb	0.54	0.94	0.81	1.10	0.83	0.88	0.93	1.01	0.88	0.79	0.88	0.77
Dy	3.67	6.28	5.58	7.27	5.39	6.15	6.25	7.11	5.96	5.35	5.95	5.22
Ho	0.81	1.38	1.22	1.55	1.17	1.34	1.40	1.52	1.27	1.16	1.27	1.16
Er	2.36	4.04	3.64	4.51	3.29	3.84	4.05	4.50	3.66	3.37	3.65	3.35
Yb	2.24	3.59	3.24	4.09	2.83	3.52	3.67	4.08	3.29	3.05	3.34	3.06
Lu	0.33	0.55	0.48	0.61	0.41	0.53	0.55	0.59	0.48	0.44	0.50	0.44
Hf	1.44	2.65	2.14	3.19	2.37	2.53	2.64	3.04	2.59	2.31	2.58	2.16
Ta	0.08	0.13	0.11	0.16	0.12	0.13	0.13	0.15	0.13	0.12	0.12	0.11
Pb	0.38	0.77	0.88	0.85	2.70	1.80	0.87	1.11	2.64	3.94	1.47	1.21
Th	0.18	0.26	0.22	0.30	0.26	0.23	0.27	0.28	0.24	0.24	0.22	0.22
U	0.04	0.16	0.04	0.11	0.10	0.06	0.11	0.10	0.29	0.06	0.08	0.04

LOI = loss on ignition (wt%), Mg# = 100 × Mg/(Mg + Fe).

Table T2 (continued).

Hole:	C0012F	C0012G	C0012G	C0012G	C0012G	C0012G	C0012G
Core, section, interval (cm):	2R-1, 60.5–65	4R-CC, 25–32	10R-2, 41–46	14R-1, 37–41	6R-1, 23–28	8R-1, 84.5–89.5	5R-1, 26–32
Depth (mbsf):	523.15	544.37	596.75	620.91	562.78	582.40	553.32
Major element (wt%):							
SiO <sub>2</sub>	50.7	51.2	50.3	50.3	51.7	50.4	47.3
Al <sub>2</sub> O <sub>3</sub>	15.1	13.6	14.6	14.6	14.1	15.7	19.5
Fe <sub>2</sub> O <sub>3</sub>	10.6	12.1	11.8	12.1	11.5	10.4	9.40
FeO <sub>t</sub>	9.50	10.90	10.61	10.93	10.37	9.32	8.46
MgO	8.00	7.40	5.92	6.06	6.20	6.31	5.37
MnO	0.36	0.27	0.33	0.30	0.31	0.28	0.22
TiO <sub>2</sub>	1.51	1.93	1.90	1.87	2.10	1.91	1.41
CaO	9.30	6.61	9.65	9.72	7.67	9.55	11.92
Na <sub>2</sub> O	3.58	3.71	3.34	3.50	4.76	5.03	4.31
K <sub>2</sub> O	0.69	2.91	1.96	1.32	1.40	0.31	0.41
P <sub>2</sub> O <sub>5</sub>	0.17	0.24	0.17	0.19	0.22	0.20	0.17
LOI	2.05	2.31	1.38	1.34	2.23	3.44	6.18
Mg#	60	55	50	50	52	55	53
Trace element (ppm):							
Li	17.1	22.2	22.8	20.2	21.2	23.4	11.0
Be	0.47	0.78	0.55	0.57	0.65	0.51	0.35
B	14.8	15.5	14.8	20.4	26.3	16.8	8.30
Sc	64.7	58.3	59.9	52.9	70.2	66.7	44.1
Ti	9075	11554	11393	11205	12608	11472	8441
V	360	436	395	396	428	420	309
Cr	193	110	81	81	91	114	68
Co	48.6	47.0	42.0	40.9	41.2	51.6	30.9
Ni	69.7	63.3	50.6	42.5	44.3	56.3	29.5
Cu	77.8	71.8	53.5	10.8	47.3	68.0	49.7
Zn	65.6	71.2	80.7	86.2	96.0	95.1	74.4
Ga	20.8	15.6	22.0	22.0	18.3	22.6	24.9
Rb	14.3	33.8	43.6	23.1	24.7	17.4	5.8
Sr	171	278	394	263	295	265	595
Y	32.3	43.7	38.9	41.1	46.8	38.4	30.2
Zr	83.4	113	111	108	123	103	82.0
Nb	1.65	2.06	2.03	1.99	2.25	1.96	1.56
Cs	0.47	0.39	0.59	0.26	0.71	1.93	0.11
Ba	506	776	148	39.8	180	58.3	56.6
La	3.81	5.11	4.50	4.47	5.79	4.16	3.11
Ce	11.4	15.8	14.7	14.0	17.0	13.5	10.4
Pr	2.13	2.76	2.58	2.47	2.95	2.40	1.86
Nd	11.1	14.1	13.4	13.3	15.5	12.9	10.3
Sm	3.61	4.68	4.45	4.31	4.97	4.34	3.39
Eu	1.36	1.67	1.59	1.55	1.80	1.57	1.21
Gd	4.79	6.07	5.79	5.82	6.61	5.57	4.60
Tb	0.83	1.04	0.99	0.98	1.10	0.97	0.76
Dy	5.64	6.93	6.85	6.67	7.54	6.66	5.10
Ho	1.22	1.53	1.46	1.47	1.65	1.44	1.12
Er	3.58	4.31	4.34	4.22	4.82	4.11	3.19
Yb	3.34	4.24	4.06	3.99	4.44	3.90	2.92
Lu	0.51	0.65	0.61	0.60	0.67	0.57	0.42
Hf	2.38	2.89	3.02	2.86	3.17	2.83	2.21
Ta	0.12	0.15	0.15	0.14	0.16	0.14	0.11
Pb	1.31	4.18	1.93	0.54	1.37	0.59	1.33
Th	0.24	0.28	0.27	0.26	0.31	0.28	0.22
U	0.06	0.08	0.31	0.11	0.12	0.12	0.05