

## 12. DATA REPORT: INTERLABORATORY ANALYSES OF FRESH AND ALTERED DACITES FROM OCEAN DRILLING PROGRAM LEG 193<sup>1</sup>

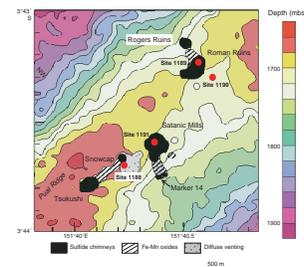
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### INTRODUCTION

Two interlaboratory reference samples were prepared from material collected during Ocean Drilling Program (ODP) Leg 193. These samples are composites of dacite representing fresh lava from Site 1191 (Sample 1191A) and altered lava from Site 1188 (Sample 1188B). The sample from Site 1188 was prepared from two fist-sized pieces of altered dacite recovered in the advanced diamond core barrel while attempting to deepen the hole. These pieces were not curated since we could not know where in the 72 m of penetration the pieces originated. The sample from Site 1191 was prepared as a composite by picking small fragments of fresh dacite from the working halves of the three cores recovered from Hole 1191A. No curated depth is assigned to either sample. These samples were collected and prepared only for comparison of analytical results from various laboratories.

Site 1188 is at the Snowcap hydrothermal site (Fig. F1), an area with low-temperature diffuse venting. The interlaboratory reference sample prepared from this site is pervasively altered, bleached from white to light gray, and sparsely vesicular. Rare vesicles are lined by anhydrite, with minor pyrite. Site 1191 is situated in the Satanic Mills hydrothermal site between the Snowcap (Site 1188) and Roman Ruins (Site 1189) hydrothermal sites (Fig. F1). The material in Sample 1191 is virtually aphyric. Rare plagioclase, clinopyroxene, and magnetite microphenocrysts are present but are <0.5% of the volume of the rock. The most

F1. Hydrothermal deposits, p. 6.



<sup>1</sup>Miller, D.J., Binns, R.A., and Paulick, H., 2006. Data report: Interlaboratory analyses of fresh and altered dacites from Ocean Drilling Program Leg 193. In Barriga, F.J.A.S., Binns, R.A., Miller, D.J., and Herzig, P.M. (Eds.), *Proc. ODP, Sci. Results*, 193, 1–9 [Online].

Available from World Wide Web: <[http://www-odp.tamu.edu/publications/193\\_SR/VOLUME/CHAPTERS/207.PDF](http://www-odp.tamu.edu/publications/193_SR/VOLUME/CHAPTERS/207.PDF)>. [Cited YYYY-MM-DD]

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abundant microphenocryst phase is plagioclase, and the largest laths are <1 mm long.

After Leg 193, the samples were trimmed with a rock saw to remove weathered surfaces, cleaned in deionized water, dried overnight at 100°C, crushed, and then milled in an alumina-ceramic shatterbox vessel at the ODP shore-based laboratory. Aliquots of the powders were distributed to requesting shipboard scientists for analysis. Analyses reported in this paper were performed at the Commonwealth Scientific and Industrial Research Organisation (CSIRO; Sydney, Australia), Technische Universität Bergakademie Freiberg, Universität Bonn (Germany; listed in Table T1 as “TU Freiberg” analyses), and Acme Analytical Laboratories, Vancouver, BC (Canada; listed in Table T1 as “TAMU analyses”).

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T1. Results comparison, p. 7.

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## **ANALYTICAL METHODS**

### **Commonwealth Scientific and Industrial Research Organisation**

#### **Inductively Coupled Plasma–Atomic Emission Spectrometer**

The CSIRO analyses were performed on an inductively coupled plasma–atomic emission spectrometer (ICP-AES) calibrated using aqueous elemental standard solutions with a calibration range to suit samples compositions. Samples were decomposed in multiacid solutions and analyzed along with at least two blanks and international reference materials. For silicon, a separate digestion was prepared in a sealed teflon container. Major element oxides reported in weight percent are SiO<sub>2</sub>, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>(t), MnO, MgO, CaO, Na<sub>2</sub>O, K<sub>2</sub>O, and P<sub>2</sub>O<sub>5</sub>. Trace elements reported in parts per million are Li, Be, S, Sc, V, Cr, Co, Ni, Cu, Zn, As, Sr, Y, Zr, Mo, Ag, Cd, Sb, Ba, La, Yb, and Pb.

#### **Inductively Coupled Plasma–Mass Spectrometer**

CSIRO uses a quadruple design inductively coupled plasma–mass spectrometer (ICP-MS) as the detection device, and the entire mass range from 1 to 269 atomic mass units may be scanned in milliseconds. Samples were prepared as for ICP-AES. Trace elements reported in parts per million are Sc, V, Cr, Co, Ni, Zn, As, Rb, Y, Mo, Cd, In, La, Ce, Pr, Nd, Sm, Eu, Tb, Dy, Ho, Er, Tm, Yb, Lu, Hf, and Pb. Trace elements reported in parts per billion are Sb, Te, Cs, Tl, Bi, Th, and U.

#### **X-Ray Fluorescence**

X-ray fluorescence (XRF) was used to measure major and trace elements on fused beads. Beads were prepared with LiBO<sub>2</sub> flux on an automated fusion machine casting into 40 mm beads. Oxidizing conditions were used to prevent loss of sulfur and reduction of metals from the sample melt. Chlorine is lost from the glass and is only indicative of the concentration in the sample. The calibrations were derived from processing a large set of international and in-house reference and pure chemical samples; analyses are routinely checked with in-batch reference samples and minimum 10% repeat samples. Loss on ignition analysis was done on a separate portion of the sample at 1050°C. Major element oxides reported in weight percent are SiO<sub>2</sub>, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>,

Fe<sub>2</sub>O<sub>3</sub>(t), MnO, MgO, CaO, Na<sub>2</sub>O, K<sub>2</sub>O, and P<sub>2</sub>O<sub>5</sub>. Trace elements reported in parts per million are S, Cl, V, Cr, Co, Ni, Cu, Zn, Ga, Rb, Sr, Y, Zr, Nb, Ba, La, Ce, and Pb.

### **Neutron Activation Analyses**

Neutron activation analyses (NAA) were performed at Becquerel Laboratories, Lucas Heights, NSW Australia. Fe<sub>2</sub>O<sub>3</sub>, Na<sub>2</sub>O, and K<sub>2</sub>O are reported as weight percent. Trace elements reported in parts per million are Sc, Cr, Co, Zn, Ga, Ge, As, Se, Br, Rb, Zr, Mo, Ag, Cd, Te, Cs, Ba, La, Ce, Sm, Eu, Yb, Lu, Hf, Ta, and Hg. Trace elements reported in parts per billion are Sb, Ir, Au, Th, and U.

## **Technische Universität Bergakademie Freiberg**

### **X-Ray Fluorescence**

Major element oxide and trace elements were measured by XRF on fused beads. The major elements SiO<sub>2</sub>, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>(t), MnO, MgO, CaO, Na<sub>2</sub>O, K<sub>2</sub>O, and P<sub>2</sub>O<sub>5</sub> are reported as weight percent oxides and Ba, Rb, Sr, Y and Zr are reported in parts per million.

### **Inductively Coupled Plasma–Mass Spectrometer**

TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, MnO (reported in weight percent) and trace elements (Sc, V, Co, Ni, Cu, Zn, Ga, As, Rb, Sr, Y, Zr, Nb, Mo, Ag, Cd, Sb, Ba, La, Ce, Pr, Nd, Sm, Eu, Tb, Dy, Ho, Er, Tm, Yb, Lu, Hf, Tl, Pb, Bi, Th, and U, reported in parts per million) were analyzed by ICP-MS. Solutions were prepared using hydrofluoric and nitric acids under atmospheric conditions.

## **Acme Analytical Laboratories**

### **Inductively Coupled Plasma–Atomic Emission Spectrometer**

Samples were prepared by fusing with LiBO<sub>2</sub> flux and dissolving the molten sample in acid. Standard reference materials (U.S. Geological Survey [USGS] BIR-1, BHVO-1, and W-2) and blanks were prepared using similar techniques. The major element oxides SiO<sub>2</sub>, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>(t), MnO, MgO, CaO, Na<sub>2</sub>O, K<sub>2</sub>O, and P<sub>2</sub>O<sub>5</sub> are reported in weight percent. Sc and Ba are reported in parts per million.

### **Inductively Coupled Plasma–Mass Spectrometer**

Samples for trace element analysis were prepared in the same manner as those for ICP-AES analyses. V, Cr, Co, Ni, Cu, Zn, Ga, Rb, Sr, Y, Zr, Nb, Cs, La, Ce, Pr, Nd, Sm, Eu, Tb, Dy, Ho, Er, Tm, Yb, Lu, Hf, and Pb are reported in parts per million. Au is reported in parts per billion.

## **RESULTS**

Major element oxide data from the three laboratories compare favorably. Totals for the altered sample (Sample 1188B) are low, probably because of the presence of anhydrite and pyrite. Only a few trace elements show some discrepancy, and possible reasons are indicated in Table T1.

Rare earth elements show disparity for Gd, Ho, Er, and Yb. USGS standard reference materials analyzed as unknowns with the Acme Analytical Laboratories samples are shown in Table T2. This compilation suggests that the samples analyzed were homogeneous and that published bulk rock analyses from these three laboratories on samples collected during Leg 209 are congruous.

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T2. Standard reference values, p. 9.

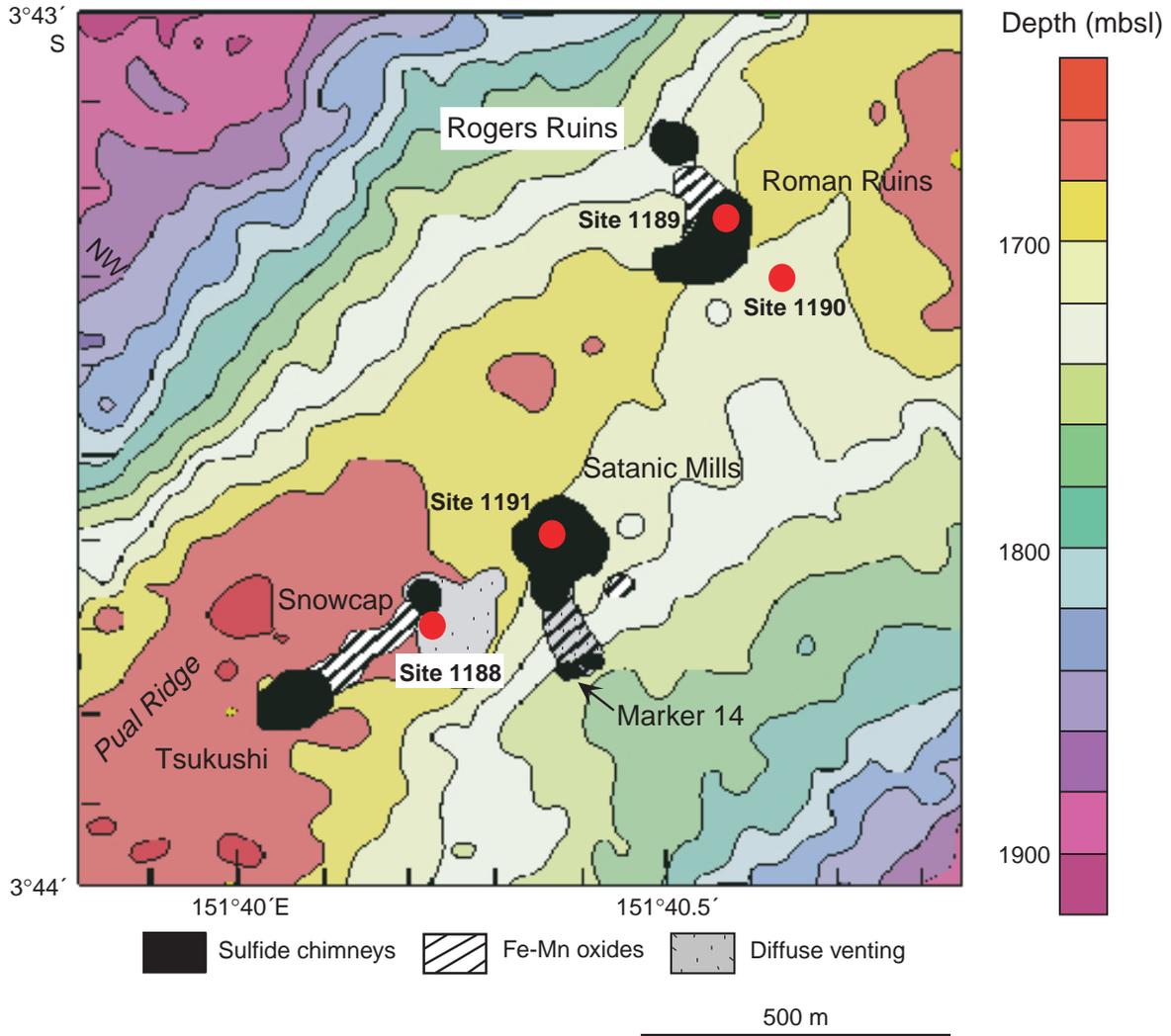
## **ACKNOWLEDGMENTS**

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## **REFERENCE**

Binns, R.A., Barriga, F.J.A.S., Miller, D.J., et al., 2002. *Proc. ODP, Init. Repts.*, 193 [CD-ROM]. Available from: Ocean Drilling Program, Texas A&M University, College Station TX 77845-9547, USA. [[HTML](#)]

**Figure F1.** Distribution of hydrothermal deposits within the PACMANUS field along the crest of Pual Ridge. Names are assigned to active sites containing massive sulfide chimneys. Figure is based on bottom-tow photography and submersible dive observations during the PACMANUS (*Franklin*; 1991, 1993, 1996, 1997), EDISON-I (*Sonne*; 1994), ManusFlux (*Yokosuka*; 1995), BIOACCESS (*Natsushima*; 1996, 1998), and KODOS'99 (*Onnuri*; 1999) cruises (Binns, Barriga, Miller, et al., 2002).



**Table T1.** Comparison of analytical results for interlaboratory samples prepared from Leg 193 core material. (See table notes. Continued on next page.)

Sample	Laboratory	Method	Major element oxide (wt%)											Trace element (ppm)								
			SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	MnO	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	LOI	Total	S total*	Li	Be	Cl*	Ge	Se	Br	In
1188B	CSIRO	ICP-AES	64.86	0.55	13.14	4.15	3.93	1.25	0.06	2.22	0.39	0.15	4.76	95.46	24,960	<5	<2					
		XRF	64.85	0.55	13.32	4.4	3.92	1.28	0.06	2.35	0.37	0.14	4.76	96	20,190			520				
		ICP-MS																	1.9			130
	TU Freiberg	NAA				4.7				2.4	<0.24									<5	2.7	
		XRF	67.11	0.6	13.66	4.99	3.99	1.31	0.06	2.17	0.4	0.14	3.7	98.13	25,400							
	TAMU	ICP-MS		0.63		5.7			0.05													
ICP-AES		65.7	0.59	13.3	4.6	3.91	1.33	0.06	2.29	0.38	0.16	3.9	96.22									
1191A	CSIRO	ICP-AES	66.76	0.59	13.81	4.29	0.83	2.89	0.12	4.83	1.73	0.14	1.46	97.45	274	13	<2					
		XRF	67.57	0.6	13.93	4.57	0.87	2.92	0.13	5.09	1.71	0.13	1.46	98.98	120			5,870				
		ICP-MS																	2.5			70
	TU Freiberg	NAA								5.2	1.3									<5	13.5	
		XRF	68.22	0.63	14.1	4.72	0.83	2.96	0.13	4.83	1.74	0.13	1.44	99.73	400							
	TAMU	ICP-MS		0.7		5.9			0.11													
ICP-AES		68.08	0.63	13.91	4.73	0.89	3.02	0.12	4.88	1.76	0.13	1.5	99.65									
		ICP-MS																				

Sample	Laboratory	Method	Trace element (ppm)																		
			Te	Hg	Cs	Ba	Cu	Pb	Zn	Ag	As	Bi	Cd	Cr	Co	Ga	Hf	Mo	Nb	Ni	Rb
1188B	CSIRO	ICP-AES				139	111	<50	143	<5	<25		<5	15	6			<5		<10	
		XRF				107	115	37	136					<10	<10	17			<4	<10	<5
		ICP-MS	0.33		0.31			21.2			14.1	0.026	0.28	15	4.9	16.1		2.1		6.2	3.8
	TU Freiberg	NAA	<1	<5		135			139	<5	16.2		<20	17.1	5.3		3.5	<5			<20
		XRF				134			150												7
	TAMU	ICP-MS			0.3	231	108	26	393	<0.5	17.7	<5	<0.5	29.3	4.4	16.4	2.3	0.89	1.4	9	4
ICP-AES					140									4.5	14.7	3.5		1.5	6.2	3.3	
1191A	CSIRO	ICP-AES				348	18	<50	81	<5	<25		<5	13	5			<5		<10	
		XRF				285	18	15	74					<10	<10	17			<4	<10	20
		ICP-MS	<0.17		0.69			5.1			13.9	0.043	0.28	13.8	4.9	16.9		2.1		3.2	27.2
	TU Freiberg	NAA	<1	<5	0.7	352			<100	<5	3.2		<20	12.4	4.9		3.6	<5			29
		XRF				376			83												28
	TAMU	ICP-MS				411	33	6	300	<0.5	4.5	<5	<0.5	13.5	5.1	18.8	3.7	1.27	1.62	6	31
ICP-AES					353																
		ICP-MS				14	5.6	79						5.2	16.7	3.7			1.7	2.7	28.5

Table T1 (continued).

Sample	Laboratory	Method	Trace element (ppm)																				
			Sb	Sc	Sr	Ta	Th	Tl	U	V	Y	Zr	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	
1188B	CSIRO	ICP-AES	<50	6	142						28	13	57	11									
		XRF			160						23	31	111	<10	25								
		ICP-MS	0.77	6.3			0.65	0.14	0.43	31.4	12.9			9.6	22.7	3.3	13.8	3.3	0.93	2.9	0.47	2.4	0.51
	TU Freiberg	NAA	0.61	12		<1	1.19						<500	9.9	22.4			3.9	1.16				
		XRF			153						33	102											
		ICP-MS	0.6	9.4	160		1.22	0.09	0.55	26	21	67											
TAMU	ICP-AES		11.5																				
	ICP-MS			161	0.1				27.5	30.8	102	10.3	22.7	3.2	13.9	4.4	1.13	4.4	0.66	2.89	1.04		
1191A	CSIRO	ICP-AES		10	246						22	31	130	13									
		XRF			264						29	33	114	<10	31								
		ICP-MS	0.15	10.9			1.18	0.21	0.66	25.4	32.3			11.1	24.8	3.6	16.5	4.3	1.27	2.9	0.47	2.4	0.51
	TU Freiberg	NAA	<0.2	11.6		<1	1.14						<500	10.8	25.3			4.3	1.25				
		XRF			255						35	110											
		ICP-MS	<0.5	16.1	308		1.74	0.21	0.62	21	38	124	11.1	22.9	3.4	14.8	4.1	1.13	4.55	0.73	4.96	1.08	
TAMU	ICP-AES		11.5																				
	ICP-MS			304	0.1				24	37.4	119	11.3	24.8	3.5	17.4	4.4	1.32	4.67	0.89	5.36	1.24		

Sample	Laboratory	Method	Trace element (ppm)				Trace element (ppb)	
			Er	Tm	Yb	Lu	Ir	Au
1188B	CSIRO	ICP-AES			<2			
		XRF						
		ICP-MS	1.3	0.19	1.2	0.19		
	TU Freiberg	NAA			3.3	0.47	<20	5.6
		XRF						
TAMU	ICP-MS	2.14	0.28	2.44	0.38		5.8	
1191A	CSIRO	ICP-AES						
		XRF			4			
		ICP-MS	3.5	0.53	3.6	0.58		
	TU Freiberg	NAA			3.6	0.52	<20	<5
		XRF						
		ICP-MS	3.3	0.5	3.5	0.54		
TAMU	ICP-AES							
	ICP-MS	3.66	0.53	3.7	0.64			

Notes: Sample 1188B is pervasively hydrothermally altered dacite. Sample 1191A is fresh, aphyric rhyodacite. CSIRO = Commonwealth Scientific and Industrial Research Organisation, TU Freiberg = Technische Universität Bergakademie Freiberg, TAMU = Acme Analytical Laboratories. ICP-AES = inductively coupled plasma-atomic emission spectrometer, XRF = X-ray fluorescence, ICP-MS = inductively coupled plasma-magnetic spectrometer, NAA = neutron activation analysis. \* = possible loss in fusion. † = uncertain value.

**Table T2.** U.S. Geologic Survey standard reference material reported and analyzed values.

Sample	Method	Major element oxide (wt%)											Trace element (ppm)								
		SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	MnO	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	LOI	S total	Li	Be	Cl	Se	Cs	Ba	Cu	Pb
BIR-1	Reported value	47.96	0.96	15.5	11.3	9.7	13.3	0.175	1.82	0.03	0.021			3.6	0.58*	0.26*			7*	125	3*
	ICP-AES	47.62	0.97	15.5	11.2	9.55	13.37	0.16	1.78	0.02	0.03	0.1									
BHVO-1	Reported value	49.94	2.71	13.8	12.23	7.23	11.4	0.17	2.26	0.52	0.273		102*	4.6		92*	0.07*	0.13	139	136*	2.6
	ICP-AES	50.1	2.82	13.6	11.96	6.95	11.5	0.16	2.29	0.52	0.28										
	ICP-MS																	0.1			

Sample	Method	Trace element (ppm)																				
		Zn	As	Bi	Cr	Co	Ga	Hf	Nb	Ni	Rb	Sb	Sc	Sr	Ta	Th	U	V	Y	Zr	La	Ce
BIR-1	Reported value	70	0.44*		370	52		0.6	0.6*	170		0.58*	44	110				310	16	18	0.63	1.9
	ICP-AES																					
BHVO-1	Reported value	105	0.4*	0.02*		52	16.3	0.6	0.5				110				320	16	16	0.68		
	ICP-AES					45	21	4.4	19*		11		31.8	403	1.2*	1.1		317	28	179	16	39
	ICP-MS					46	22.2	4.5	18.6		10.7			410	1.3	1.2	0.5	319	28.3	169	17	40

Sample	Method	Trace element (ppm)											
		Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
BIR-1	Reported value		2.5	1.1*	0.55	1.8		4				1.7	0.26*
	ICP-AES												
BHVO-1	Reported value	0.38	2.4	1	0.54	1.7	0.37	3.4	0.52	1.8	0.25	1.72	0.25
	ICP-AES		25	6.2	2.06	6.4*	0.96*	5.2	0.99*	2.42*	0.33*	2	0.29*
	ICP-MS	5.6	25	6	2.1	6.6	0.97	5.4	1	2.54	0.4	2.1	0.3

Notes: All analyses were performed at the Acme Analytical Laboratories. ICP-AES = inductively coupled plasma-atomic emission spectrometer, ICP-MS = inductively coupled plasma-magnetic spectrometer. \* = informational values.