

Analytical Methods SO255

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1. Sampling

Samples were collected during the R/V SONNE expedition SO255 VITIAZ along the western flank of the Kermadec Ridge, the Kermadec forearc and Kermadec trench, the Colville Ridge and within the Havre Trough back-arc basin including the Quaternary Kermadec volcanic front, between 35°S and 28°S. Sampling of various morphologic and volcanic structures was carried out using a rectangular chain bag dredge. Dredge stations were chosen based on existing datasets of predicted ocean floor morphology combined with unpublished bathymetric survey data (pers. comm. Timm) and SO255 multi-beam echo-sounding surveys. Dredge tracks were selected dependent on morphology of structures and were usually located on steep slopes, plateau edges, scarps, canyon walls, fracture zones, flanks of cones, ridges and seamounts. Dredged samples include pillow lavas and sheet flows, volcanoclastics, pumice, and sediments. Many lava flow surfaces possess volcanic glass encrustations. At sea whole rock blocks to be used for further geochemical analysis were cut from the fresh core of samples by removal of weathering rinds and encrustations using a rock saw. Glassy margins were trimmed off to obtain the raw material for further glass processing. For further details see the SO255 cruise report (Hoernle, Werner, Hauff, eds. 2017; <http://oceanrep.geomar.de/38365/>). Sampling of representative igneous rocks in each dredge for geochemistry is based on degree of groundmass alteration, degree of vesicle filling, freshness of phenocrysts and ideally presence of glassy pillow margins. The full correlated data set is found in file [SO255_ECL_ALL_DATA.xlsx](#) in [worksheet 1 Sample Data](#).

2. Sample preparation

Whole rock blocks were crushed in a steel jaw crusher, sieved, repeatedly washed in deionized water in an ultrasonic bath to remove seawater salts, fine dust and adhering fragments and afterwards dried in an oven at 50°C. To minimize secondary materials such as calcite, manganese, zeolites and clay minerals occurring in vesicles and veins, rock chips of the fraction 1 mm to 2 mm were carefully hand-picked under a binocular microscope. Finally, the whole-

rock chips were ground to powder ($<63\ \mu\text{m}$) using an agate mortar grinder followed by a planetary agate ball mill. A subset of the 0.5 mm to 1 mm fraction was picked for radiogenic isotope analysis.

Glass encrustations were present on many pillow lavas and sheet flows of the Havre Trough back-arc (BA) and the Kermadec Quaternary volcanic front (QVF) samples. Glass-rim slabs were crushed with an agate mortar and pestle. Glass-chips were then sieved, washed in deionized water in an ultrasonic bath and finally hand-picked under a binocular microscope. Suitable glass-chips (0.5 - 1 mm) were mounted with resin in epoxy mounts and polished using SiC grit paper, followed by diamond pastes to expose and polish the glass-chip surfaces.

3. Major elements

Major elements of whole rock powders were analyzed at the Institute of Mineralogy and Petrography at the University of Hamburg, Germany by X-ray fluorescence spectrometry (XRF). The measurements were performed on fused lithium-tetraborate glass pellets using a PanAnalytical MagixPro PW2540 X-ray fluorescence spectrometer. The calibration of the instrument involved 83 international rock and mineral reference materials (RM's) using the recommended values of Govindaraju (1994). RM's JGB-1, JB-2, JB-3, JA-3, JG-2 and JG-3 were run as unknowns alongside the samples. 1SD (%) reproducibility are $\text{SiO}_2: \leq 0.4\%$, $\text{TiO}_2: \leq 0.8\%$, $\text{Al}_2\text{O}_3: \leq 0.5\%$, $\text{Fe}_2\text{O}_3: \leq 0.3\%$ except JG-2 with 0.8% at c. 0.9 wt%, $\text{MgO}: \leq 0.9\%$ except JG-2 with 10.3% at 0.04 wt%, $\text{MnO}: \leq 4.7\%$, $\text{CaO}: \leq 1.2\%$ except JG-2 with 2.7% at 0.8 wt%, $\text{Na}_2\text{O}: \leq 2.1\%$, $\text{K}_2\text{O}: \leq 1.3\%$, $\text{P}_2\text{O}_5: \leq 6.2\%$. Accuracy lies within 3 % of the reference values from Govindaraju (1994) for most oxides ([worksheet 2 XRF RM's](#)). Loss on ignition (L.O.I.) was determined gravimetrically after heating the samples at 1000°C for 3 h (Lechler and Desilets, 1987).

Major element compositions of glass chips were analyzed by electron microprobe (EMPA) with a JEOL JXA 8200 wavelength dispersive microprobe at GEOMAR (Kiel, Germany) using a defocused to 5 μm electron beam, 15 kV acceleration voltage, 6 nA beam current and the conditions outlined in Portnyagin et al. (2012). For calibration and monitoring of data quality basaltic glass (USNM 113498/1 VG-A99) was used for Ti, Fe, Mg, Ca and P, rhyolitic glass (USNM 72854 VG568) for Si, Al, Na and K. All standard glasses were analyzed three times at the beginning and end of an analytical session, as well as every 50-60 analyses within a session. The reported data represents the mean compositions obtained from 4 to 6 analyses performed on 2 to 3 glass chips for each sample and were normalized to anhydrous basis. All glasses were homogeneous within the precision of analyses estimated from replicate measurements of VG-2 reference glass. ([worksheet 3 EMP RM](#)).

4. Trace elements

Trace element compositions of whole rock powders were analyzed by solution inductively coupled plasma mass spectrometry (ICPMS) at the Institute of Geosciences, Kiel University, Germany, using an Agilent 7500cs ICPMS. Sample weights of 100 mg were dissolved by table-top acid-pressure digestion in Savillex™ PFA-vials and analyzed following the method of Garbe-Schönberg (1993). Replicate analyses of reference materials BHVO-2 and AGV-2 by means of separate digests from different analytical sessions are generally better than 4% 1SD for all elements analyzed (worksheet [4 ICPMS RM's](#)). Accuracy of BHVO-2 and AGV-2 measured along with the samples lies within 4 % of the reference values of Jochum et al. (2016) for most elements. Replicate analyses of eight samples deviate within 2% for the vast majority of elements except for SO255 DR123-1 with 3-5% deviation throughout, which probably reflects sample loss during the first digestion (Dup 1). See worksheet [5 ICPMS Sample Replicates](#) for details.

Trace element composition of glass chips were determined by LA-ICPMS using a GeoLas Pro 193 nm excimer laser ablation system coupled to an Agilent 7500s quadrupole ICP-MS at the Institute of Geosciences, Kiel University, Germany. Sample mounts were loaded into a two-volume ablation cell and the ablated sample aerosol was flushed with 1 L min⁻¹ He as the carrier gas, mixed with 14 mL H₂ and Ar before entering the ablation cell. Single glass chips were analyzed with 60–80 µm crater size, 10 J cm⁻² laser fluence and 10 Hz pulse frequency. The total analysis time for every single analysis was set to 80 s (20 s background, 40 s sample ablation, and 20 s washout monitoring). The measured intensities were converted to concentrations in the Glitter software by using ⁴³Ca as internal standard and KL2-G reference glass was used for initial calibration. A drift-calibration based on KL2-G was applied to all samples using the 100 % normalization procedure. The reference glasses GOR132-G, BHVO-2G and BCR-2G were analyzed as unknowns (worksheet [6 LA-ICPMS RM's](#)). Reproducibility for multiple analyses was typically better than 5% 1SD for most elements and accuracy better than 4% relative to the values of Jochum et al. (2005, 2006). The reported trace element concentrations of samples are average concentrations, calculated from 4 to 6 analyses performed on 2 to 3 glass chips for each sample. Further details of the setup and calibration strategy can be found in Garbe-Schönberg and Müller (2014).

In addition, samples containing refractory minerals (e.g. zircon), likely not being dissolved by regular acid digestion, were analyzed by LA-ICP-MS of nano-particulate pressed powder pellets (NP). The pellets were produced from whole-rock powders that underwent additional wet milling as described by Garbe-Schönberg (2014). NP-LA-ICPMS measurements of the

powder pellets were conducted under the same conditions as for the glass-chips. The powder pellets were measured along with powder pellets of the reference materials BHVO-2P and BIR-1P (worksheet [7 NP-LA-ICPMS RM's](#)). Reproducibility is better than 5% 1SD and accuracy within 4% of Jochum et al. (2016). Exceptions are Rb, Cs and U in the depleted BIR-1P tholeiite with several times higher concentrations than the recommended values and may reflect contamination by the additional milling process. Since only alkaline rhyolites with comparatively high concentrations of these elements were measured by NP-LA-ICPMS possible contamination by the wet milling process is considered to be negligible.

5. Comparison of Ba, Sr, and Zr concentrations from XRF and ICPMS

XRF analysis of fused beads also provided selected trace element data of which Ba, Sr and Zr show reasonable (ca 20% for Ba) to good (0.3 to 4% for Sr and Zr) 1SD reproducibility for the reference materials ([worksheet 2 XRF RM's](#)). Accuracy relative to the values of Govindaraju (1994) is better than 5% at concentrations >50 ppm and deviates in excess of 10 to 30% at concentrations <50 ppm (Supplement Table1).

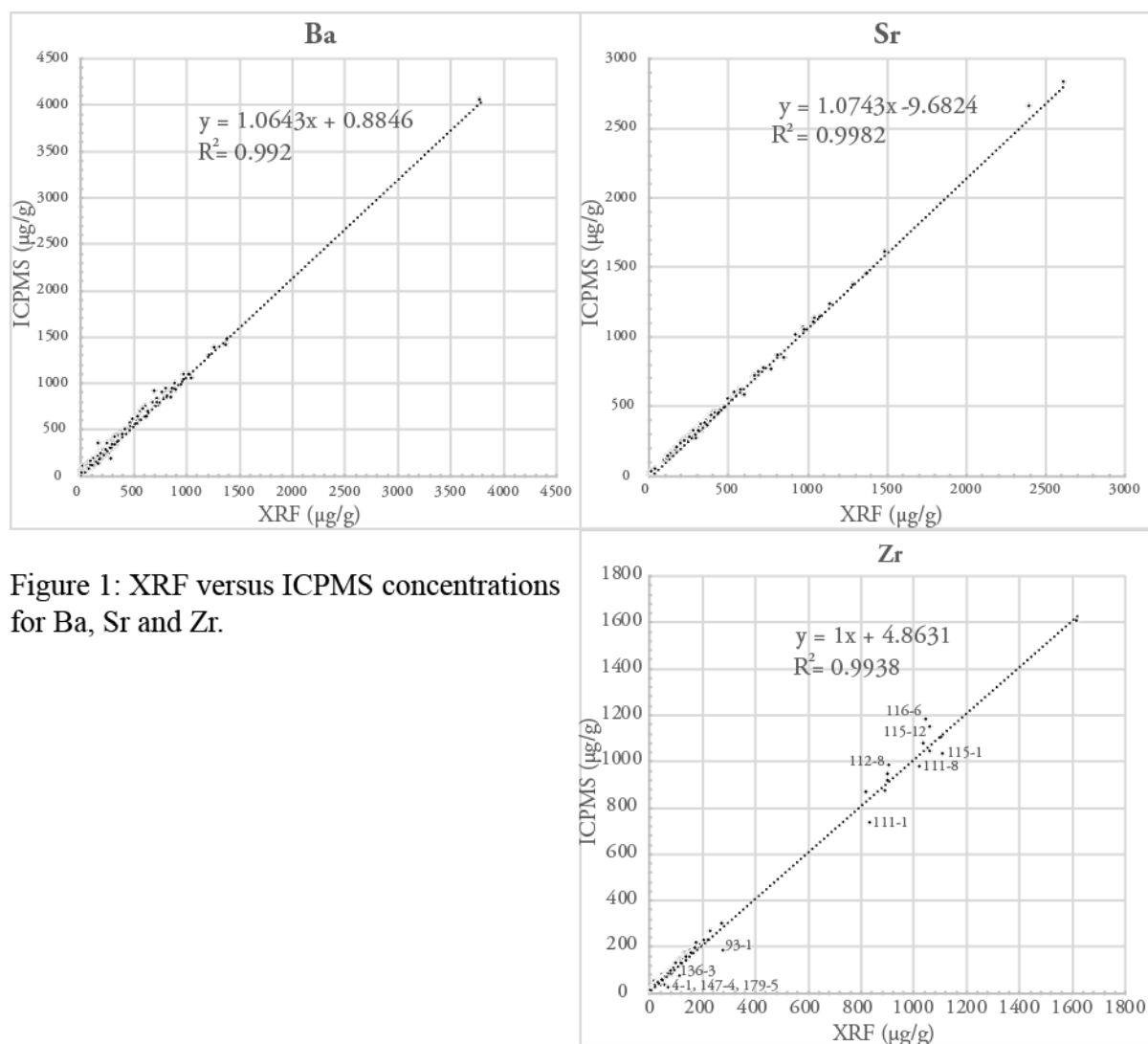


Figure 1: XRF versus ICPMS concentrations for Ba, Sr and Zr.

Ba (XRF) and Ba (ICPMS) display a good correlation ($r^2 = 0.992$) but deviate to lower Ba (XRF) for a given Ba (ICPMS). Similarly, Sr (XRF) and Sr (ICPMS) are very well correlated ($r^2 = 0.9982$). Above 500 ppm Sr, a significant offset to lower Sr (XRF) for a given Sr (ICPMS) is, however, observed. Zr also correlates very well ($R^2 = 0.9938$) with a slope of 1 and a small off-set of ca +5ppm (ICPMS axis) at the zero intercept. Still several samples fall clearly below the XRF-ICPMS correlation line. Below 300 ppm Zr these are SO255 DR4-1, 93-1, 136-3, 147-4 and 179-5 for which the ICPMS value is much lower than the XRF value. Unless replicated the Zr (XRF) value should be used for these samples and Hf ppm (ICPMS) of these samples considered to be minimum values. In particular sample 93-1 is a fairly evolved volcanic rock with 64.5 wt% SiO₂ and 0.73 wt% MgO and thus accessory zircon is likely to be present, which did not dissolve during regular digestion. Several alkaline rhyolites from the back-arc caldera complex significantly deviate above and below the XRF-ICPMS correlation line. On the one hand SO255 DR111-1, 111-8 and 115-1 have Zr ppm (ICPMS) significantly lower than XRF possibly indicating insufficient zircon disintegration and homogenization during the additional wet powder milling. On the other hand, SO255 DR112-8, 115-12 and 116-6 have significantly higher Zr concentrations (ICPMS) than corresponding XRF values. Again, this could reflect imperfect homogenization of nano-powders and regular powders used for XRF. In any case the Zr ppm from XRF should be used for the six alkali rhyolite samples mentioned above. See Supplement Figure 1 for details.

6. Radiogenic isotopes

Radiogenic isotope analyses (Sr-Nd-Pb-Hf) were conducted at the GEOMAR Helmholtz Centre for Ocean Research Kiel, Germany by thermal ionization mass spectrometry (TIMS, Sr-Nd-Pb) and MC-ICPMS (Hf) following the methods outlined in Jacques et al. (2019) and Dausmann et al. (2019) respectively. Approximately 100-200 mg hand-picked glass (250-500 μm) or whole-rock chips (500-1000 μm) were leached in 2N HCl at 70°C for 1 hour and subsequently triple-rinsed in 18.2 M Ω water to minimize alteration and handling effects. Sr-Nd-Pb ion chromatography followed established standard procedures of Hoernle et al. (2008) for Sr-Nd-Pb and Geldmacher et al. (2006) for Hf.

Sr and Nd isotope ratios were determined on a Thermo Scientific Triton *Plus* TIMS operating in static multi-collection mode and normalized within run to $^{86}\text{Sr}/^{88}\text{Sr} = 0.1194$ and $^{146}\text{Nd}/^{144}\text{Nd} = 0.7219$, respectively. Measurement errors are reported as a 2σ standard error (2SE). Reference materials were measured 4 to 5 times along with the samples on each turret and the average ratio of the standard subtracted from the preferred reference values to obtain a delta value to be added to the sample and standard data of each turret. This procedure ensures long-term

comparability of data generated at different times on different instruments by compensation of long-term machine drift caused by detector alteration. Accordingly, NBS987 gave $^{87}\text{Sr}/^{86}\text{Sr} = 0.710250 \pm 0.000008$ ($n = 101$; 2σ standard deviation (2SD) and La Jolla gave $^{143}\text{Nd}/^{144}\text{Nd} = 0.511850 \pm 0.000006$ ($n = 95$; 2SD). Possible mass interferences by ^{87}Rb and ^{144}Sm were monitored by ^{85}Rb and ^{147}Sm but none were detected. Pb isotope analyses were carried out in static multi-collection mode on the Triton *Plus* TIMS using the Pb double-spike (Pb-DS) technique described in Hoernle et al. (2011). Pb-DS corrected NBS981 values are $^{206}\text{Pb}/^{204}\text{Pb} = 16.9408 \pm 0.0018$, $^{207}\text{Pb}/^{204}\text{Pb} = 15.4975 \pm 0.0018$, $^{208}\text{Pb}/^{204}\text{Pb} = 36.7207 \pm 0.0048$, $^{208}\text{Pb}/^{206}\text{Pb} = 2.167584 \pm 0.000099$ and $^{207}\text{Pb}/^{206}\text{Pb} = 0.914803 \pm 0.000050$ ($n = 173$; 2SD since installation of the instrument in 2014). Hafnium isotopic analyses were performed statically on a Thermo Scientific Neptune *Plus* MC-ICP-MS at GEOMAR. Samples were introduced to the system in a mix of 0.5M HNO_3 –0.1M HF. Instrumental mass bias was corrected applying an exponential mass fractionation law with a natural $^{179}\text{Hf}/^{177}\text{Hf}$ ratio of 0.7325. Potentially interfering isotopes of Yb and Lu were monitored on masses ^{171}Yb , ^{173}Yb and ^{175}Lu . No interference corrections had to be made since all samples had respective Yb or Lu ion currents below 0.001×10^{-11} A, hence indistinguishable from analytical background. For comparison, corresponding Hf ion currents for 50 ppb Hf solutions were on the order of 1.25×10^{-11} A for ^{176}Hf . Standard concentrations were adjusted to sample concentrations of each measurement batch. The desolvating unit (Aridus II) was rinsed with a solution of 0.5M HNO_3 –0.05M HF between samples to ensure proper washout, and on-zero blanks were subtracted from respective ion beams. Drift corrected $^{176}\text{Hf}/^{177}\text{Hf}$ of 0.282170 ± 6 ($n = 110$) for our in-house Hf SPEX CertiPrepTM solution which corresponds to $^{176}\text{Hf}/^{177}\text{Hf} = 0.282163$ for JMC-475 Blichert-Toft et al. (1997). Total chemistry blanks were typically $<30\text{pg Pb}$, $<100\text{pg Sr}$, $<50\text{pg Nd}$ & Hf and are therefore considered negligible relative to the amounts of sample used. Sample replicates are shown in [worksheet 8 Sr-Nd-Pb-Hf Sample Replicates](#). Reference materials BCR-2 (Sr-Nd-Pb-Hf) and AGV-2 (Hf only) were repeatedly dissolved and processed under similar conditions as the samples and measured along with them. Their radiogenic Sr-Nd-Pb and Hf isotope ratios lie within the values of Fourny et al. (2016) and Todd et al (2015). See worksheet Table 6 for details. Replicate analysis of samples by means of a second digests lie within 2SD of the single element Sr-Nd-Pb-Hf standards mentioned above ([worksheet 9 Sr-Nd-Pb-Hf RM's](#)).

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Captions for worksheets in file: [SO255_ECL_ALL_DATA.xlsx](#)

1 Sample Data: Correlated data table of samples collected during cruises SO255, TAN0206, TAN1611 and B30 that were analyzed for major element, trace element and radiogenic Sr-Nd-Pb-Hf isotopic compositions.

2 XRF RM's: JGB-1, JB-1, JB-2, JA-3, JG-3 and JG-2 measured alongside the samples. Most major elements reproduce better than 1% 1SD except for oxides in the 0.1 to 0.2 wt.% range such as MnO and P₂O₅ that reproduce at several % 1SD. Trace elements reproduce better than 20% for Ba and a few % 1SD for Sr and Zr. Compared to reference values of Govindaraju (1994) most major elements deviate less than 3%.

3 EMP RM: Repeat measurements of VG-2 during two analytical sessions of SO255 glasses. SiO₂, TiO₂, Al₂O₃, FeO_t, MgO, CaO are reproduced within 2.5% 1SD whereas MnO, Na₂O, K₂O and P₂O₅ reproduce at 17%, 4.4%, 6.3% and 15.7% respectively 1SD. The larger 1SD% variation reflects low abundance of Mn, K and P in VG-2 and volatilization of Na during analysis. Reference values for VG-2 are from GeoReM (downloaded 21st Oct 2019). Deviations of VG-2 analysis of this study from GeoReM are ≤ 3% for all oxides except P₂O₅ (Δ = 14.8%)

4 ICPMS RM's: Solution ICPMS repeat analysis of reference materials BHVO-2 (n=9) and AGV-2 (n=9) by means of separate digestions. Reference values are from Jochum et al. 2016. The high Pb ppm in analysis #9 of BHVO-2 is considered an outlier.

5 ICPMS Sample Replicates: Seven replicates of SO255 samples by means of a second digestion.

6 LA-ICPMS RM's: Reference materials GOR 132G (n=84), BHVO-2G (n=79) and BCR-2G (n=14) were measured along with the samples. Reference values are from Jochum et al. 2006 and Jochum et al. 2005.

7 NP-ICPMS RM's: LA-ICPMS analysis of nano powder (NP) reference materials BHVO-2P (n=3) and BIR-1P (n=3). 1SD generally lies between 3 and 8% for most elements and thus about twice as high as for solutions ICPMS analysis. Still accuracy lies within a few % to the values of Jochum et al (2016) for most elements. Larger deviations are observed for Rb, Cs and U in the trace element depleted BIR-1 standard and may reflect contamination during the additional wet milling to obtain the NP material.

8 Sr-Nd-Pb-Hf Sample Replicates: Duplicate digests of SO255 samples. Pb ratios in italics are from low intensity measurements as testified by elevated 2SE. The replicates of these samples were at higher ion currents, overlap with the initial analysis and thus confirm validity of both analyses.

9 Sr-Nd-Pb-Hf RM's: Summary of radiogenic Sr-Nd-Pb isotope ratios for BCR-2 on eight separate dissolutions and Hf isotope ratios on BCR-2 and AGV-2 on two separate dissolutions each. Values of Fourny et al. (2016) and Todd et al. (2015) are shown for comparison. In all studies materials were leached prior to dissolution to reduce the well-known Pb contamination that was introduced during the initial preparation of these standards. Also shown are radiogenic Sr-Nd-Pb-Hf isotopic compositions of single element solutions NBS987 (Sr), La Jolla (Nd), NBS981 (Pb), and SPEX (Hf) that were run alongside the samples.