



## Handheld XRF

# Type Standardization – a Simple Method for Improved Accuracy of Handheld-XRF Analysis

Application Note XRF 474

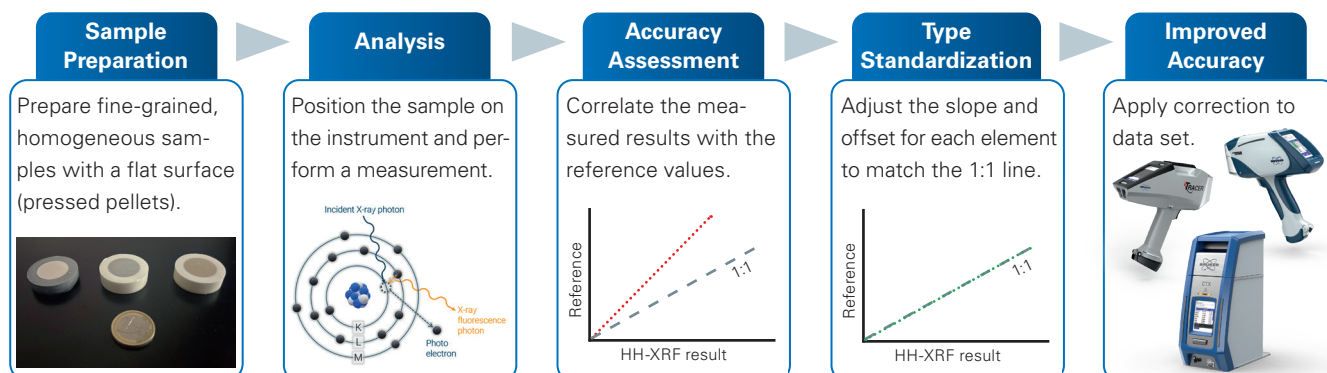
### Introduction

In X-ray fluorescence (XRF) analysis, sample presentation is critical to data quality. This includes consistent positioning of the instrument and adapting the sample type (powder or solid) to the calibration method. Misalignment or inappropriate sample presentation can lead to inaccurate and imprecise results. For example, using a Bruker handheld/portable XRF (HH-XRF/pXRF) analyzer – by default calibrated with powder samples – for solid rock may introduce systematic bias.

Handheld XRF devices are extremely flexible for use in field studies, particularly for the

analysis of core samples, because they can measure elements from Mg to U at any location. However, in order to achieve maximum accuracy, samples are typically pulverized and packed into cups, and sample preparation in the field may not be practical and time-consuming.

A simple method to improve data accuracy in the field is type standardization. Using a few reference samples, which are prepared similarly to solid unknowns, corrects biases due to differences in calibration and measurement conditions. Type standardization can be used with any of Bruker's handheld and portable XRF analyzers (S1 TITAN, TRACER 5g, CTX).



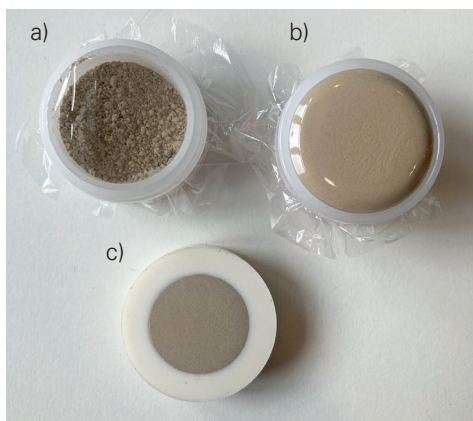
## Powders vs. solid rocks – The importance of sample preparation

XRF analysis is highly matrix-dependent. X-ray transmission and attenuation are strongly influenced by material density, requiring calibration. Other key factors are grain size (finer grains scatter less) and air gaps (which cause more scattering). In conventional XRF analysis these effects are minimized by sample preparation – pulverizing and compressing powders eliminates air gaps, improving accuracy.

Matrix effects on the XRF signal can be demonstrated using measurements of the certified reference material (CRM) SdAR-M2\* prepared in three ways:

- loose powder in a sample cup
- compacted powder in a sample cup
- pressed pellet (Figure 1).

Double-open sample cups (30.7 mm diameter, 22.9 mm height) were used and sealed with Prolene film. In the compacted samples, the powder was tamped down to reduce air gaps and prevent particle movement; the loose sample was not compacted. Pressed pellets were produced by weighing 5 g of SdAR-M2 CRM without binder into a custom-made filling tool, placing it into a 32 mm press mold and surrounding it with a mixture of 7 g of cellulose and paraffin powder as a carrier material.



**Figure 1**

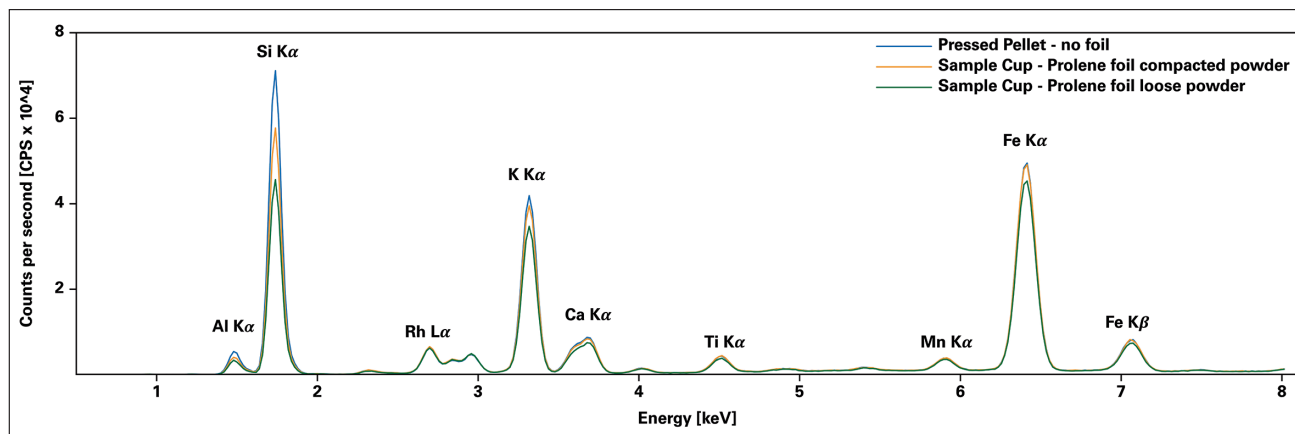
Common examples of sample preparation techniques for SdAR-M2:  
a) Loose powder in a sample cup  
b) Packed powder in a sample cup  
c) Pressed pellet from myStandards GmbH.

The press mold is then placed in an automated hydraulic press (Specac Ltd. Atlas Autotouch 40T) and pressed with a load of 40 metric tons. The programmable automated pressing process ensures exact reproducibility. Unlike powder cups, pressed pellets avoid mineral deposits and variable air gaps.

The results show that greater compaction increases signal intensity, especially for light elements such as Si and K (Figure 2). This is due to less attenuation of the fluorescence signal caused by air pockets in between loosely packed particles. These density effects, propagated through a calibration, lead to apparent differences in element concentrations between the different preparation methods of the same sample.

**Figure 2**

Comparison of element intensities in the energy range from 0.5 to 8 keV for differently prepared SdAR-M2 samples (see Figure 1). Measurements were performed with a TRACER 5g HH-XRF analyzer at 15 kV, 20  $\mu$ A, and without a filter. The measurements took 30 s. The effect of sample preparation is more pronounced for elements with lower characteristic X-ray energies.



\* Sediment Animas River, USGS/IAGeo Ltd.

## Data correction through type standardization

Bruker's pXRF/HH-XRF analyzers use empirical calibrations based on matrix-matched reference materials tailored to specific applications. For this demonstration, Bruker's factory-installed GeoExploration calibration was used to quantify rock compositions.

This calibration was developed using a large collection (~100) of certified geological reference materials covering a wide range of rock compositions, all prepared as packed powder sample cups covered with Prolene film.

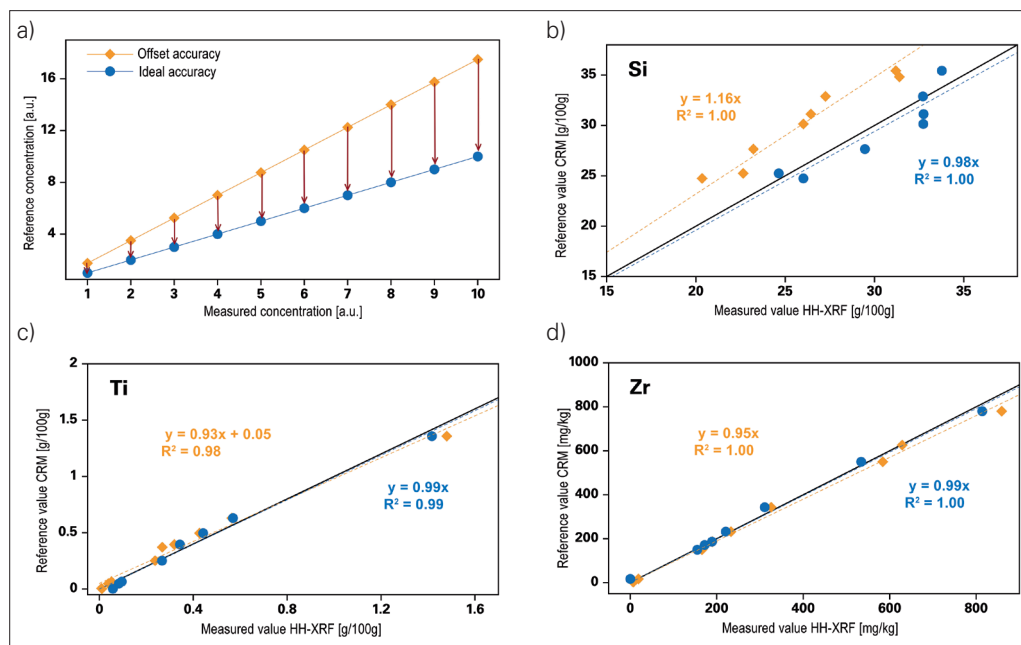
While this calibration is ideal for similarly prepared samples, accuracy for solid rock samples might be reduced. Although the Prolene film is ~4 μm thin, it absorbs low-energy X-rays to a small extent and increases scattering, which particularly affects light elements. Therefore, the use of different sample preparation techniques or uncovered rock samples can lead to systematic bias (e.g., Figure 3a).

To correct this, the type standardization method can be applied. By measuring samples that are very similar in shape and composition to the unknown target samples, bias can be identified and correction factors applied to improve accuracy. This process is demonstrated using measurements of 10 certified reference

materials pressed into pellets without Prolene cover to standardize for the analysis of solid rocks and improve accuracy for light elements. The reference materials should correspond to the range of compositions expected from the unknowns (any independently verified sample may be used).

The measurements were performed using the GeoExploration calibration with default settings (30 s per spectrum). Example results for Si, Ti, and Zr are shown in Figure 3b-d (orange diamonds), and the complete data are listed in Tables 1 and 2. The linear regressions of the measured values deviate from the 1:1 line, especially for the light element Si.

Correction factors can be calculated and applied based on the slope and offset of these regressions. For example, for Ti:  $y = 0.93x + 0.05$ , where  $x$  is the measured value, 0.93 is the slope factor and 0.05 is the offset from the origin. If the regression passes through the origin, only a slope factor is applied. Accuracy is assessed based on the agreement of the corrected data with the 1:1 line (Figure 3b-d, blue circles). Adjustments can be made to achieve the desired confidence level. Correction factors can be entered directly into the pXRF analyzer (details can be found in the S1 TITAN/TRACER 5/CTX User Manual) for real-time adjustment or applied offline to exported data.



**Figure 3**

a) Illustration of systematic bias that can be caused by sample preparation, and its correction to ideal accuracy. b-d) Two-dimensional plots for Si, Ti, and Zr showing reference values compared to measured values (using HH-XRF) for the certified reference materials used in this experiment. Yellow diamonds reflect compositions before type standardization, blue dots reflect compositions after type standardization. Ideally, the data fits a ~1:1 line (—) after type standardization.

**Table1: HH-XRF quantified values of certified reference materials after type standardization**

Element	Conc.	OKUM	ECRM 782-1	GSP-2	BCR-2	ECRM 752-1	AGV-2	ACE	OREAS-20a	GH	LOESS-1
<b>Mg</b>	%	22.27	22.18	0.64	1.74	<LOD*	1.12	<LOD	1.32	<LOD	2.27
<b>Al</b>	%	4.41	0.19	8.54	7.46	0.46	9.22	8.00	8.52	6.93	3.70
<b>Si</b>	%	28.50	<LOD	32.75	24.64	<LOD	29.46	32.71	32.73	33.77	26.01
<b>P</b>	%	0.01	0.04	0.12	0.14	0.09	0.22	<LOD	0.11	<LOD	0.08
<b>K</b>	%	0.08	0.10	4.62	1.50	0.12	2.49	3.83	3.49	4.14	1.20
<b>Ca</b>	%	4.85	22.17	1.19	4.21	39.33	3.28	0.14	2.28	0.38	13.05
<b>Ti</b>	%	0.27	0.01	0.37	1.67	0.01	0.66	0.06	0.50	0.04	0.28
<b>V</b>	ppm	540	<LOD	85	592	<LOD	87	<LOD	140	<LOD	<LOD
<b>Cr</b>	ppm	2466	<LOD	<LOD	<LOD	<LOD	28	<LOD	85	<LOD	80
<b>Mn</b>	%	0.18	0.07	0.03	0.15	0.01	0.08	0.04	0.05	0.04	0.06
<b>Fe</b>	%	9.25	0.43	3.40	9.71	0.04	4.88	1.90	3.85	1.02	1.63
<b>Ni</b>	ppm	894	14	15	<LOD	62	16	8	34	5	32
<b>Cu</b>	ppm	37	<LOD	44	12	11	48	<LOD	39	<LOD	10
<b>Zn</b>	ppm	61	74	117	139	7	87	224	63	50	30
<b>Ga</b>	ppm	9	4	20	23	5	21	41	17	24	8
<b>Rb</b>	ppm	<LOD	4	243	45	<LOD	64	151	224	380	46
<b>Sr</b>	ppm	15	20	246	355	143	670	<LOD	299	9	287
<b>Y</b>	ppm	8	<LOD	26	36	<LOD	17	182	28	78	23
<b>Zr</b>	ppm	16	<LOD	534	189	<LOD	221	814	267	155	311
<b>Nb</b>	ppm	<LOD	<LOD	22	12	<LOD	11	111	17	88	7
<b>Ba</b>	ppm	<LOD	<LOD	1306	650	72	1122	75	1063	<LOD	167
<b>Pb</b>	ppm	<LOD	33	39	<LOD	<LOD	10	39	18	65	12
<b>Th</b>	ppm	5	<LOD	111	6	<LOD	<LOD	17	20	78	<LOD
<b>U</b>	ppm	<LOD	<LOD	5	<LOD	<LOD	<LOD	3	8	17	<LOD

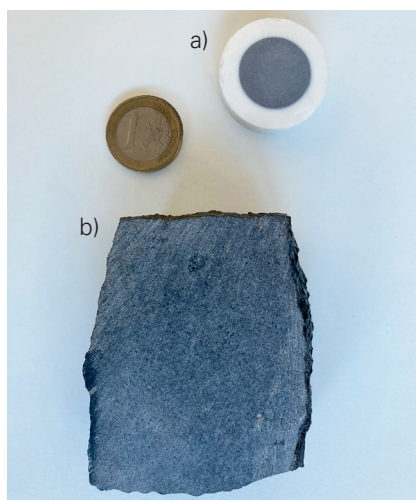
\* Limit of detection

### Practical application of type standardization

To demonstrate the usefulness of the method, the type-standardized GeoExploration calibration was applied to two basaltic samples:

- a) BHVO-2: pressed pellet made from certified Hawaiian basalt
- b) OM-11 V-09: solid basanite sample from the Oman ophiolite Batain Basin.

The basanite is fine grained, consisting of plagioclase, clinopyroxene, and olivine, low in silicon, and rich in alkali elements. It is ideal for testing due to its representative overall composition within the 8 mm spot size.



**Figure 4**

a) BHVO-2 pressed pellet, manufactured by myStandards GmbH, b) OM-11 V-09 solid piece of basanite rock, provided by Prof. Stefan Jung, University of Hamburg (Reference: Witte, M. et al. (2017) OIB signatures in basin-related lithosphere-derived alkaline basalts from the Batain basin (Oman) – constraints from  $^{40}\text{Ar}/^{39}\text{Ar}$  ages and Nd-Sr-Pb-Hf isotopes, *Lithos* (286-287), p. 109-124.

**Table 2: Values of certified reference materials used**

Element	Conc.	OKUM	ECRM 782-1	GSP-2	BCR-2	ECRM 752-1	AGV-2	ACE	OREAS- 20a	GH <sup>△</sup>	LOESS-1
<b>Mg</b>	%	22.27* ±0.06	22.18* ±0.09	0.58 ±0.02	2.17 ±0.04	0.09 ±0.01	1.09 <sup>△</sup> ±0.09	0.02 ±0.01	1.45 ±0.03	0.02 ±0.06	1.75 ±0.02
<b>Al</b>	%	4.22 ±0.02	0.06 ±0.003	7.89 ±0.11	7.13 ±0.12	0.032 ±0.01	9.01 <sup>△</sup> ±0.06	7.78 ±0.03	8.09 ±0.16	6.62 ±0.19	3.22 ±0.02
<b>Si</b>	%	20.63* ±0.07	0.12 ±0.003	31.13 ±0.37	25.24 ±0.2	0.327 ±0.02	27.65 <sup>△</sup> ±0.27	32.88 ±0.03	30.15 ±0.41	35.43 ±0.29	24.74 ±0.07
<b>P</b>	%	0.012 ±0.001	0.006 ±0.0003	0.127 ±0.009	0.157 ±0.01	0.004* <sup>△</sup>	0.211 ±0.019	0.006 ±0.002	0.101 ±0.005	0.004 ±0.004	0.057 ±0.001
<b>K</b>	%	0.04 ±0.002	0.02 ±0.001	4.47 ±0.12	1.47 ±0.02	0.008 <sup>△</sup> ±0.01	2.41 ±0.03	3.73 ±0.02	3.32 ±0.07	3.95 ±0.12	1.11 ±0.01
<b>Ca</b>	%	5.61 ±0.04	21.68 ±0.05	1.5 ±0.04	5.08 ±0.08	39.59 ±0.2	3.68 <sup>△</sup> ±0.07	0.24 ±0.01	2.58 ±0.05	0.49 ±0.09	11.81 ±0.08
<b>Ti</b>	%	0.02* ±0.002	0 ±0.003	0.4 ±0.01	1.36 ±0.02	0.005 <sup>△</sup> ±0.002	0.63 ±0.01	0.07 ±0.01	0.50 ±0.01	0.05 ±0.02	0.25 ±0.002
<b>V</b>	ppm	168* ±3	n.a.	52 ±4	418* ±5	3 <sup>△</sup> ±0.3	119 ±1	3 ±1	115 ±10	5	35 ±1
<b>Cr</b>	ppm	2460 ±31	6 ±2	20 ±6	16 ±0.4	7 <sup>△</sup> ±0.7	16 ±1	3 ±1	65 ±10	3	83 <sup>△</sup> ±3
<b>Mn</b>	%	0.14* ±0.002	0.06 ±0.002	0.03 ±0.002	0.15 ±0.003	0.008 ±0.001	0.08 ±0.002	0.04 ±0.002	0.05 ±0.001	0.04 ±0.008	0.05 ±0.001
<b>Fe</b>	%	8.26* ±0.03	0.31 ±0.001	3.43 ±0.11	9.63 ±0.19	0.016 ±0.005	4.74 <sup>△</sup> ±0.12	1.77 ±0.01	3.73 ±0.09	0.94 ±0.16	1.45 ±0.01
<b>Ni</b>	ppm	886 ±10	4 <sup>△</sup>	17 ±2	13 ±0.3	2* <sup>△</sup>	19 ±0.4	2 ±1	39 ±4	3	31 ±1
<b>Cu</b>	ppm	44 ±1	n.a.	43 ±4	20 ±1	1.5* <sup>△</sup>	52 ±1	4 ±1	45 ±4	3	10 <sup>△</sup> ±1
<b>Zn</b>	ppm	61 ±2	66 ±3	120 ±10	130 ±2	4 ±1	87 ±1	224 ±6	69 ±2	55	32 ±1
<b>Ga</b>	ppm	9 ±0.2	n.a.	22 ±2	22 ±0.2	0.2 <sup>△</sup>	20 ±0.2	39* ±4	20 ±1	23	7 ±0.2
<b>Rb</b>	ppm	1 ±0.1	n.a.	245 ±7	46 ±1	1 ±0.2	68 ±1	152 ±2	218 ±10	390	51 ±1
<b>Sr</b>	ppm	16 ±1	n.a.	240 ±10	337 ±7	161 ±20	660 ±6	3 ±1	296 ±12	10	280 ±5
<b>Y</b>	ppm	9 ±0.3	n.a.	28 ±2	36 ±0.4	2 <sup>△</sup> ±0.1	19 ±1	184 ±5	27 ±1	75	24 ±2
<b>Zr</b>	ppm	17 ±1	n.a.	550 ±30	187 ±2	2 <sup>△</sup>	232 ±2	780 ±20	89* ±23	150	343 ±6
<b>Nb</b>	ppm	0.4 ±0.1	n.a.	27 ±2	12 ±0.2	0.2 <sup>△</sup>	14 ±0.2	110 ±5	20 ±1	85	8 ±0.3
<b>Ba</b>	ppm	6 ±1	n.a.	n.a.	684 ±5	54 <sup>△</sup> ±20	1134 ±8	55 ±5	1070 ±31	n.a.	n.a.
<b>Pb</b>	ppm	0.3 ±0.02	27 ±2	42 ±3	11 ±0.2	2 ±0.4	13 ±0.2	39 ±3	22 ±2	45	11 ±1
<b>Th</b>	ppm	0.03 ±0.004	n.a.	105 ±8	6 ±0.1	0.1 <sup>△</sup>	6 ±0.1	19 ±1	22 ±1	87	8 ±0.2
<b>U</b>	ppm	0.1 ±0.1	n.a.	2 ±0.2	2 ±0.02	0.5 ±0.01	2 ±0.002	5 ±0.5	6 ±0.4	18	3 ±0.1

Reference values from Jochum et al. 2005, \* Values were not used for type-standardization, <sup>△</sup> Information values, not certified.

Figure 5a shows the deviations from ideal values for uncorrected (orange diamonds) and type-standardized (blue circles) results for 21 elements. BHVO-2 results improved significantly for light elements (Mg-Si). Type standardization was less effective for elements such as Rb, where the concentrations in the standards were close to the detection limits. This increases the uncertainty of the correction factors determined, but can be improved by better matched standards or longer measurement times.

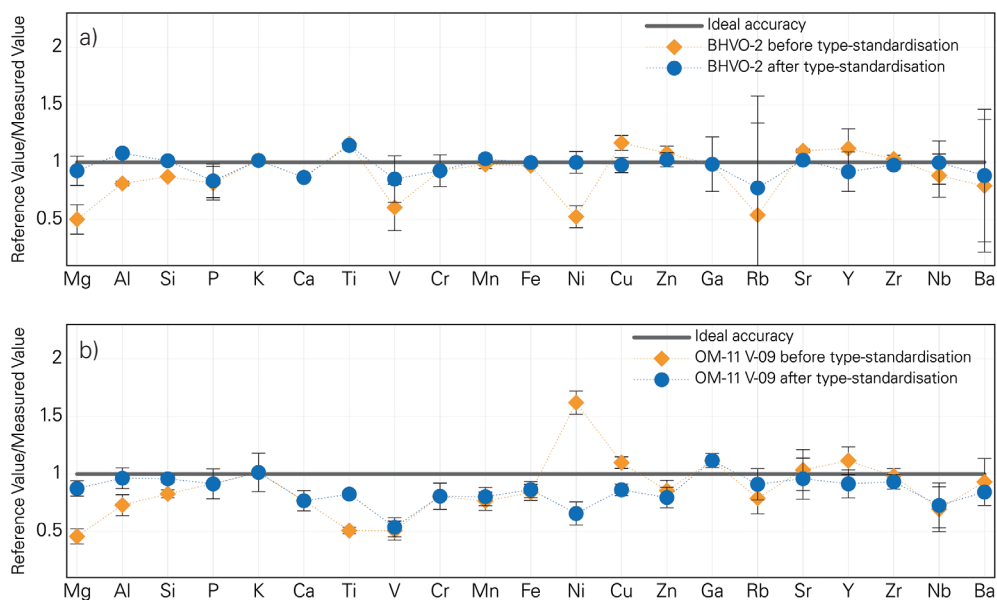
In OM-11 V-09, type standardization significantly improved accuracy of light elements, while fewer improvements were observed for heavier elements (Figure 5b). This was partly because, despite being flat and fine-grained, the sample composition was inhomogeneous across the surface, impairing data accuracy. Each data point represents four measurements, with 1-sigma relative standard deviations (RSD)

between 3% and 20%, reflecting heterogeneity or, alternatively, low concentrations. Furthermore, differences in the interaction of X-rays between porous rock and dense pellets can influence the results.

## Conclusion

Handheld and portable XRF analyzers are widely used in many industries due to their ease of use, fast data processing, and low cost. With proper handling and understanding of data collection and processing, results can be achieved that are close to laboratory quality. Type standardization is a powerful method to correct any systematic bias caused by different sample preparation or direct measurement of unprepared rocks. Note that non-systematic errors, i.e., excess scatter in a data set, cannot be corrected using this approach.

Type standardization significantly improves accuracy, but interpreting data from natural rock samples requires caution, as they can be inhomogeneous and interact differently with X-rays than powdered references. This study shows that HH-XRF analyzers deliver reliable geochemical field data as well as cost-effective, high-quality laboratory results when samples are properly prepared and presented.



**Figure 5**

a) BHVO-2 pressed pellet and b) OM-11 V-09 solid piece measured with the TRACER 5 HH-XRF analyzer, with the compositions normalized to the reference values. Yellow diamonds represent results before type standardization, blue dots represent data after type standardization. The corrections were based on measurements of micro-milled pressed pellet CRMs from myStandards GmbH.

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