Supplementary Material to article:

Variability and bias in measurements of metals mass fractions in automobile shredder residue

Amund N. Løvik[a] *, Renato Figi[b], Claudia Schreiner[b], Matthias Rösslein[c], Rolf Widmer[a], Rainer Bunge[d], Thomas Pohl[d], Nathalie Korf[e], Claudia Kuntz[e], Paul Martin Mählitz[e], Vera Susanne Rotter[e], Patrick Wäger[a]

[a] Empa, Swiss Federal Laboratories for Materials Science and Technology, Technology and Society Laboratory, CH-9014 St. Gallen, Switzerland

[b] Empa, Swiss Federal Laboratories for Materials Science and Technology, Division for Advanced Analytical Technologies, CH-8600 Dübendorf, Switzerland

[c] Empa, Swiss Federal Laboratories for Materials Science and Technology, Particles-Biology Interactions Laboratory, CH-9014 St. Gallen, Switzerland

[d] University of Applied Sciences Rapperswil, Institute of Environmental and Process Engineering (Umtec), CH-8640 Rapperswil, Switzerland

[e] Technische Universität Berlin, Chair of Circular Economy and Recycling Technology, 10623 Berlin, Germany

* *Corresponding author*

Address: Lerchenfeldstrasse 5, CH-9014 St. Gallen, Switzerland

Phone: +41 58 765 7150

E-Mail: amund.loevik@empa.ch

This supplement contains:

17 pages

1 table

27 figures

Contents

1. Results of t-test S3
2. Recovery rates S4
3. Plots of all measured mass fractions S4

1. Results of t-test

The results of t-test for difference between in-house and validated observations are listed in Table S1.

Table S1 Results of t-test for difference between in-house and validated observations. Bold font indicates the element/digestion combinations that were used with the validated method. * Cd was measured in a mixture of a polymer epoxy and the metals sample rather than the pure metals samples.

	$S1_{metals}$		$S2_{metals}$		$S1_{plastics}$		$S2_{plastics}$			
	p	*δ*/%	*p*	*δ*/%	*p*	*δ*/%	*p*	*δ*/%	digestion	detection (Val/IH)
Al	4.8E-04	-38	1.9E-02	17	3.9E-01	-6.1	5.7E-03	30	HNO_3	OES/OES
Au	1.9E-01	26	2.4E-01	38	1.4E-03	480	9.1E-01	-12	HNO_3	MS/MS
Cd*	**8.7E-07**	**920**	**1.3E-06**	**3000**	**1.6E-04**	**300**	**4.1E-04**	**160**	**HNO_3**	**MS/OES**
Co	**3.7E-04**	**100**	**5.6E-01**	**8**	**3.1E-01**	**32**	**3.5E-02**	**46**	**HNO_3**	**MS/OES**
Cr	**1.5E-05**	**180**	**2.8E-08**	**-72**	**7.4E-01**	**-4.1**	**9.2E-03**	**-19**	**HNO_3**	**OES/OES**
Cu	**8.6E-03**	**26**	**2.0E-01**	**-5.6**	**1.8E-01**	**-59**	**1.0E-02**	**83**	**HNO_3**	**OES/OES**
Dy	**3.0E-07**	**-100**	**1.6E-09**	**-97**	**1.3E-05**	**-100**	**1.5E-01**	**-55**	**HNO_3**	**MS/OES**
Fe	5.0E-01	14	8.4E-01	-1.7	2.4E-01	10	1.6E-02	44	HNO_3	OES/OES
La	**5.3E-06**	**-100**	**5.2E-05**	**-100**	**1.5E-04**	**-100**	**8.5E-01**	**-16**	**HNO_3**	**MS/OES**
Nd	2.7E-05	-73	3.7E-10	-77	1.2E-02	-62	1.1E-02	-64	HNO_3	MS/OES
Ni	**6.7E-04**	**43**	**4.6E-08**	**-52**	**4.3E-02**	**-33**	**1.8E-01**	**-19**	**HNO_3**	**OES/OES**
Pb	8.3E-05	-65	1.0E-04	93	8.3E-01	1.1	7.8E-01	4.5	HNO_3	OES/OES
Pd	2.5E-04	-100	2.7E-10	2200	2.5E-04	-88	8.6E-05	-100	HNO_3	MS/OES
Pt	9.2E-05	∞	4.3E-06	∞	2.8E-01	100	1.3E-01	-58	HNO_3	MS/OES
Sb	1.4E-04	-76	6.6E-08	-89	7.6E-07	-80	4.7E-05	-88	HNO_3	MS/OES
Sn	6.6E-05	-88	2.4E-04	-78	1.8E-04	-76	4.7E-06	-64	HNO_3	MS/OES
Y	**3.0E-06**	**-41**	**5.7E-06**	**-33**	**1.6E-06**	**-50**	**8.6E-05**	**-27**	**HNO_3**	**MS/MS**
Yb	**7.7E-05**	**-25**	**1.9E-03**	**-17**	**8.1E-01**	**0.88**	**7.5E-03**	**-13**	**HNO_3**	**MS/OES**
Zn	**3.3E-04**	**-39**	**2.3E-02**	**61**	**4.5E-03**	**-15**	**5.1E-01**	**7.3**	**HNO_3**	**OES/OES**
Al	**4.8E-02**	**-19**	**5.0E-02**	**-11**	**8.7E-01**	**-1.4**	**2.0E-03**	**27**	**AR**	**OES/OES**
Au	**3.8E-03**	**-72**	**2.0E-01**	**-44**	**6.7E-01**	**11**	**4.6E-01**	**-82**	**AR**	**MS/MS**
Cd*	2.0E-09	990	7.8E-10	2900	7.2E-06	210	1.2E-03	150	AR	MS/OES
Co	1.1E-04	66	1.0E-02	44	6.6E-01	12	1.5E-02	92	AR	MS/OES
Cr	9.4E-01	1.4	5.0E-02	-19	1.2E-01	-20	5.2E-02	11	AR	OES/OES
Cu	9.8E-01	0.22	2.6E-02	13	4.4E-01	-34	1.1E-01	120	AR	OES/OES
Dy	3.5E-07	-99	3.0E-09	-89	1.3E-05	-100	3.9E-07	-96	AR	MS/OES
Fe	**8.8E-01**	**2.6**	**2.6E-01**	**3.8**	**6.3E-01**	**-2.7**	**4.1E-03**	**70**	**AR**	**OES/OES**
La	1.7E-01	-11	1.8E-01	17	2.2E-02	53	8.0E-01	-13	AR	MS/MS
Nd	**4.4E-05**	**-69**	**8.4E-09**	**-65**	**8.5E-03**	**-66**	**1.7E-02**	**-57**	**AR**	**MS/OES**
Ni	8.1E-01	2.1	5.1E-01	-2.7	8.4E-01	6.1	2.4E-01	-16	AR	OES/OES
Pb	**7.1E-03**	**-31**	**1.2E-01**	**-8.2**	**3.0E-03**	**-34**	**7.5E-03**	**-21**	**AR**	**OES/OES**
Pd	**2.5E-04**	**-100**	**4.0E-04**	**4000**	**6.2E-06**	**-100**	**8.6E-05**	**-100**	**AR**	**MS/OES**
Pt	**7.4E-04**	**∞**	**2.8E-08**	**∞**	**1.1E-01**	**670**	**4.9E-01**	**43**	**AR**	**MS/OES**
Sb	**3.4E-02**	**-40**	**5.6E-06**	**-55**	**4.3E-01**	**6.6**	**5.6E-01**	**-5.6**	**AR**	**MS/OES**
Sn	**6.5E-05**	**-31**	**6.8E-02**	**-14**	**1.1E-02**	**-9.8**	**5.8E-01**	**-4.2**	**AR**	**MS/OES**
Y	5.3E-06	-38	5.2E-01	-3.1	1.5E-08	-55	3.6E-05	-49	AR	MS/MS
Yb	4.5E-04	-23	6.0E-03	17	3.2E-01	2.1	1.1E-03	-31	AR	MS/OES
Zn	4.5E-01	5.6	4.2E-04	10	5.8E-03	-16	3.1E-02	24	AR	OES/OES

2. Recovery rates

Figure S 1 shows the calculated recovery rates of standard addition prior to chemical analysis.

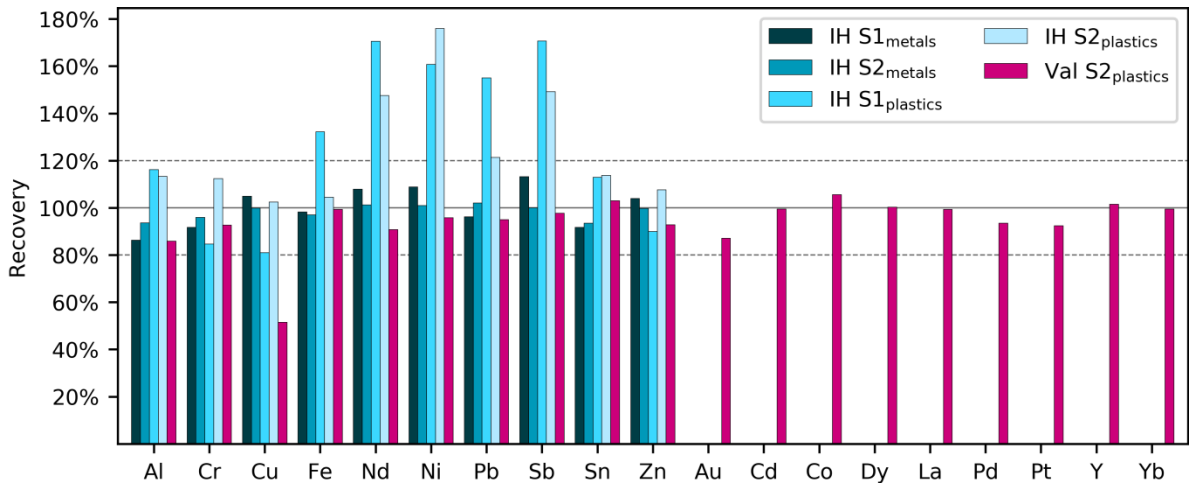

Figure S 1 Recovery rates of standard addition prior to chemical analysis. With the in-house methods, standard addition was only performed for elements Al to Zn in the figure, and only with the in-house method that used the same acid system as the validated method (aqua regia for Al, Fe, Nd, Pb, Sb and Sn; HNO_3/H_2O_2 for Cr, Cu, Ni, and Zn). Note that the low value for Cu with the validated method is due to heterogeneity of the sample.

3. Plots of all measured mass fractions

This section contains figures with all individual measurements of element mass fractions in automobile shredder light fraction (SLF) test samples. The figures are sorted by element name in alphabetical order. See the main paper for further description of samples and measurement methods.

The following abbreviations and short forms are used:

Sample names

S1p: Sample 1 plastics

S2p: Sample 2 plastics

S1m: Sample 1 metals

S2m: Sample 2 metals

Measurement method names

Val: Validated

IH: In-house

AR: aqua regia

HNO3: HNO_3/H_2O_2

OES: (inductively coupled plasma) optical emission spectrometry

MS: (inductively coupled plasma) mass spectrometry

XRF: X-ray fluorescence spectrometry

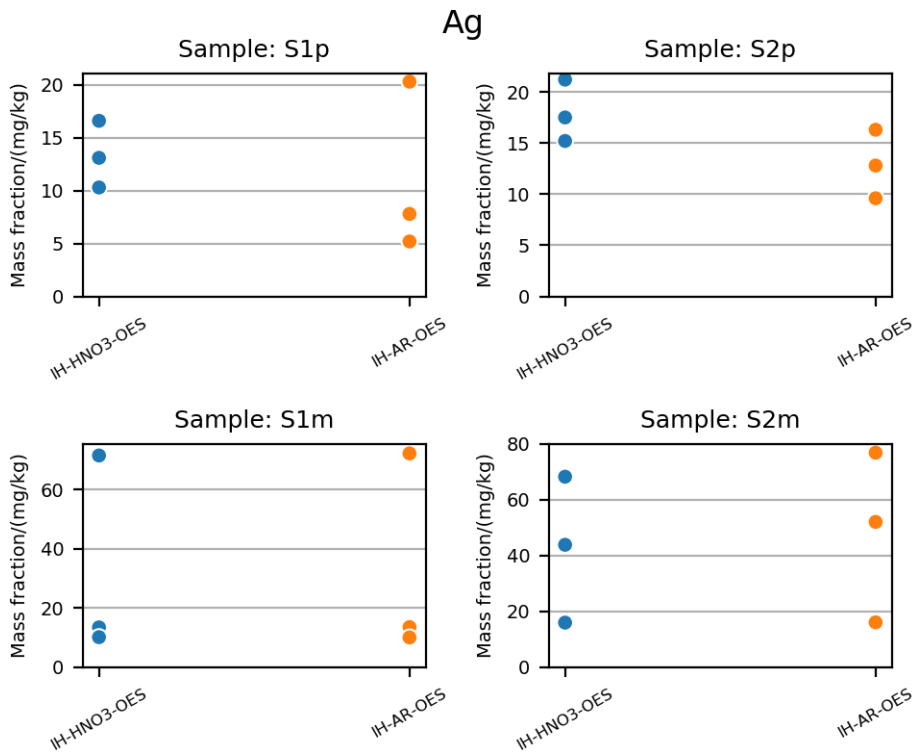

Figure S 2 Measured mass fractions of Ag in SLF samples.

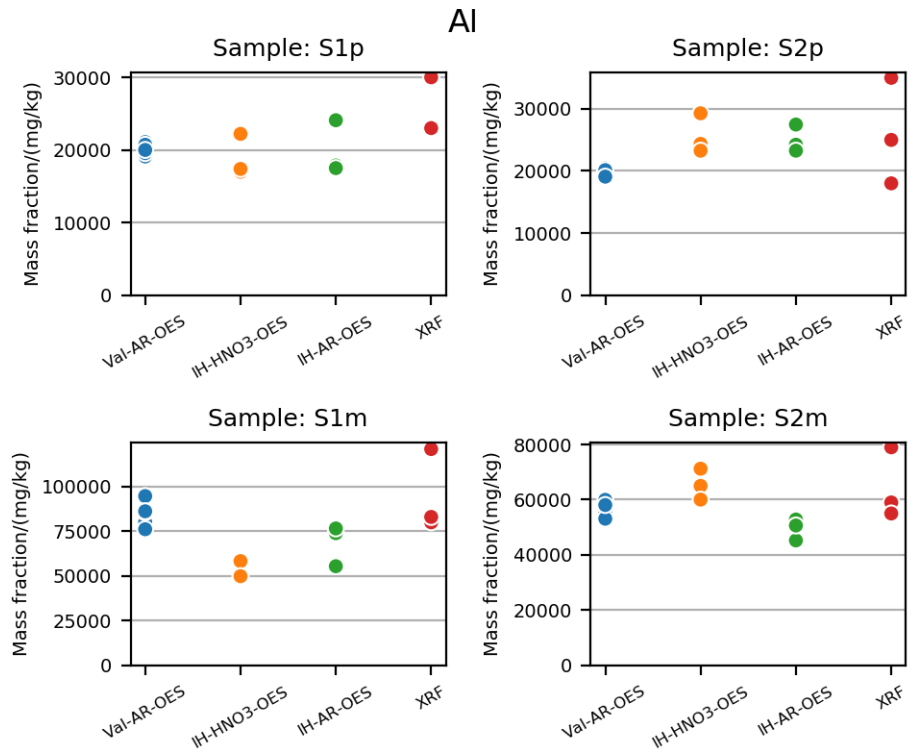

Figure S 3 Measured mass fractions of Al in SLF samples.

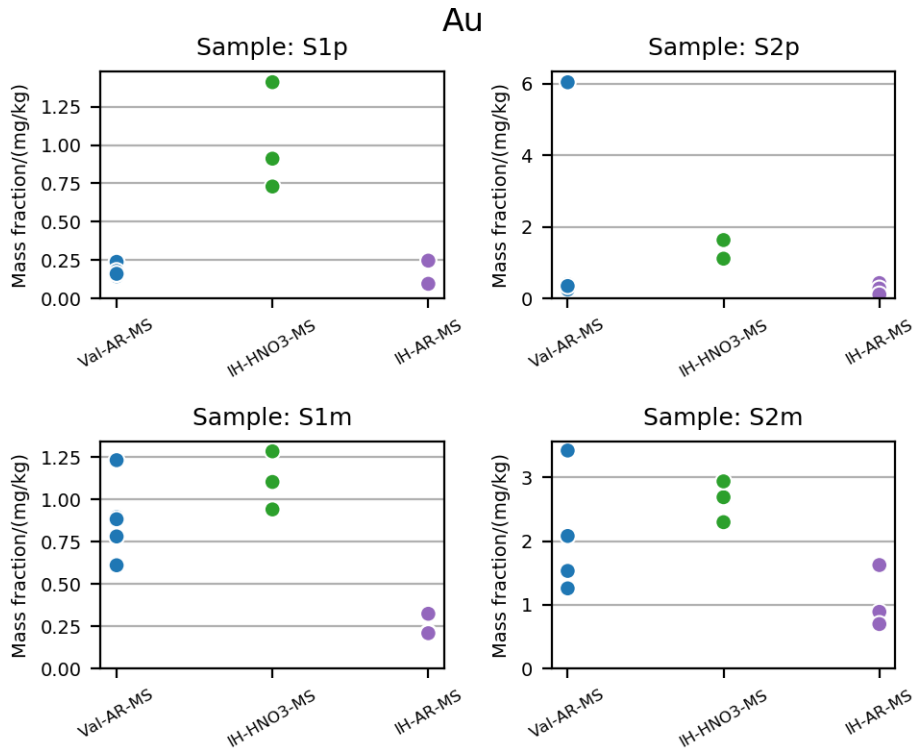

Figure S 4 Measured mass fractions of Au in SLF samples.

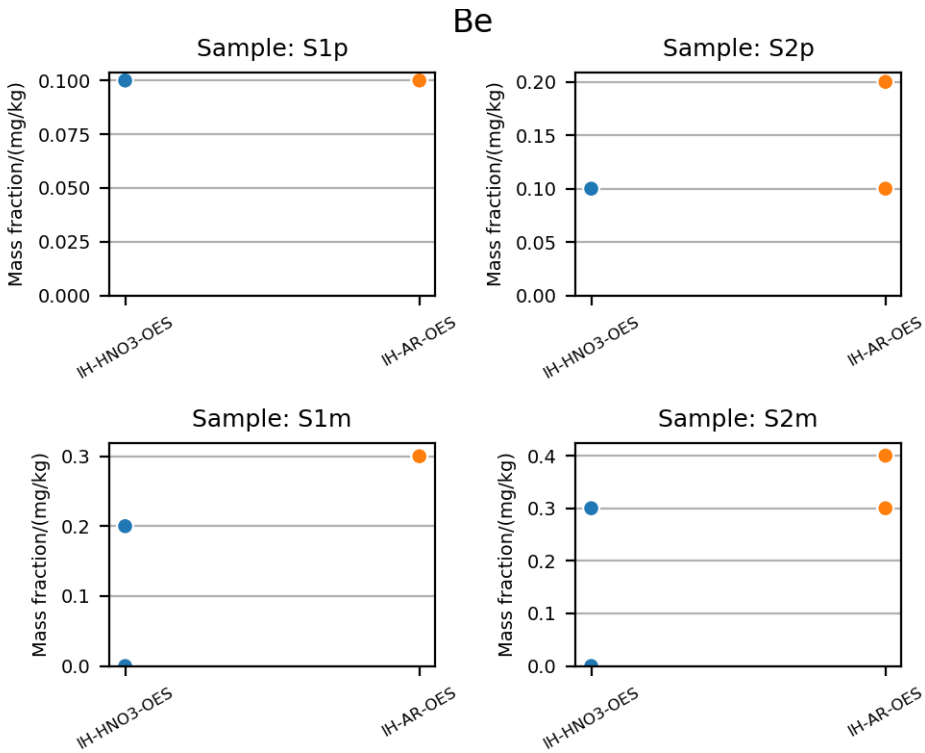

Figure S 5 Measured mass fractions of Be in SLF samples.

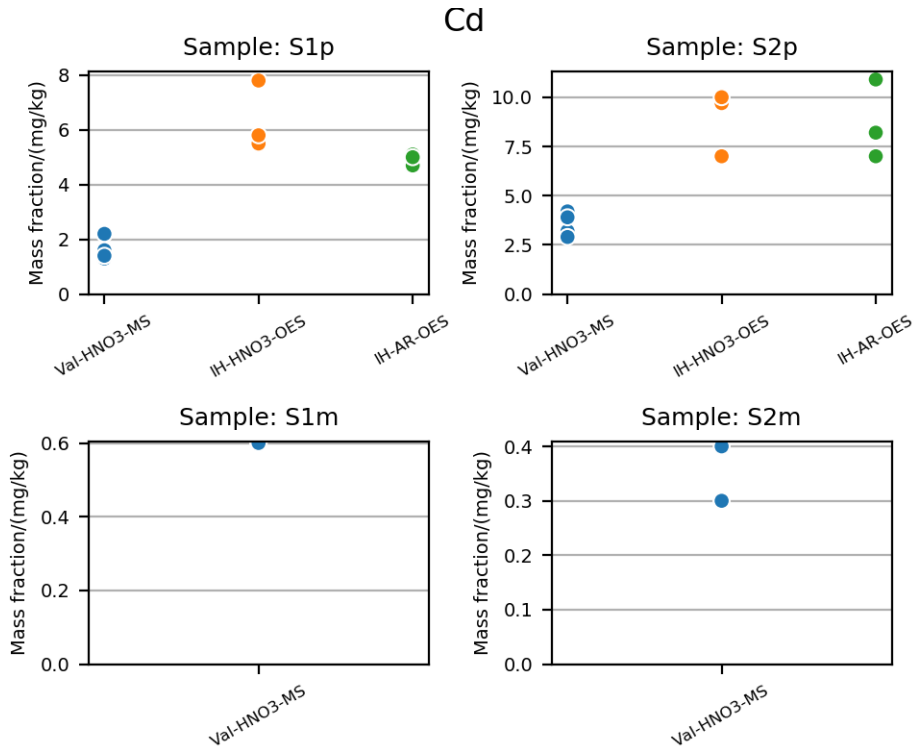

Figure S 6 Measured mass fractions of Cd in SLF samples.

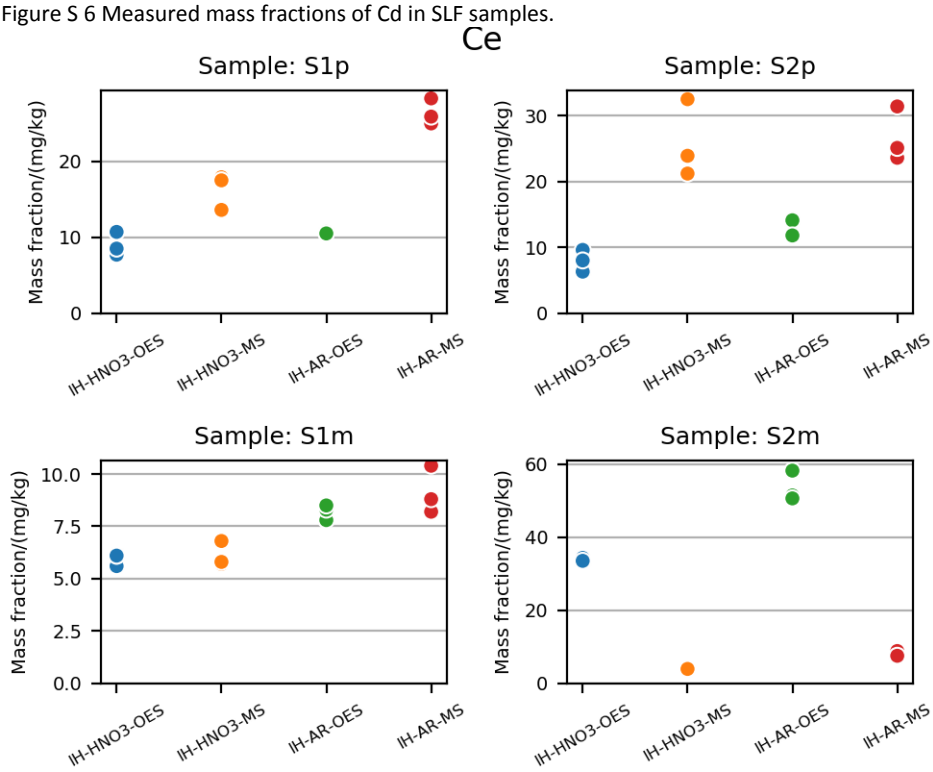

Figure S 7 Measured mass fractions of Ce in SLF samples.

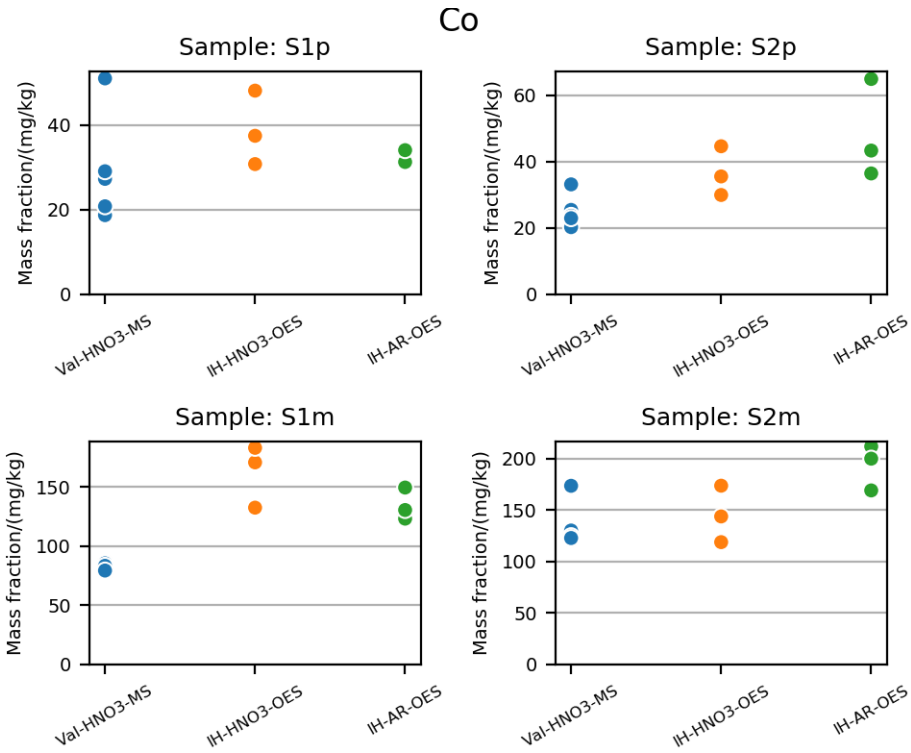

Figure S 8 Measured mass fractions of Co in SLF samples.

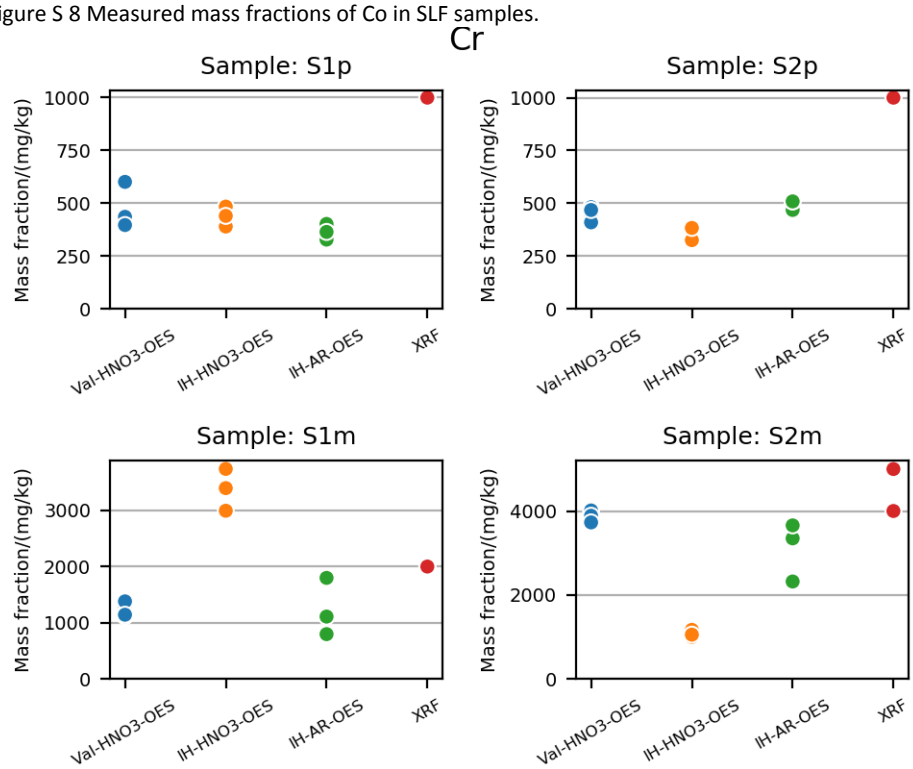

Figure S 9 Measured mass fractions of Cr in SLF samples.

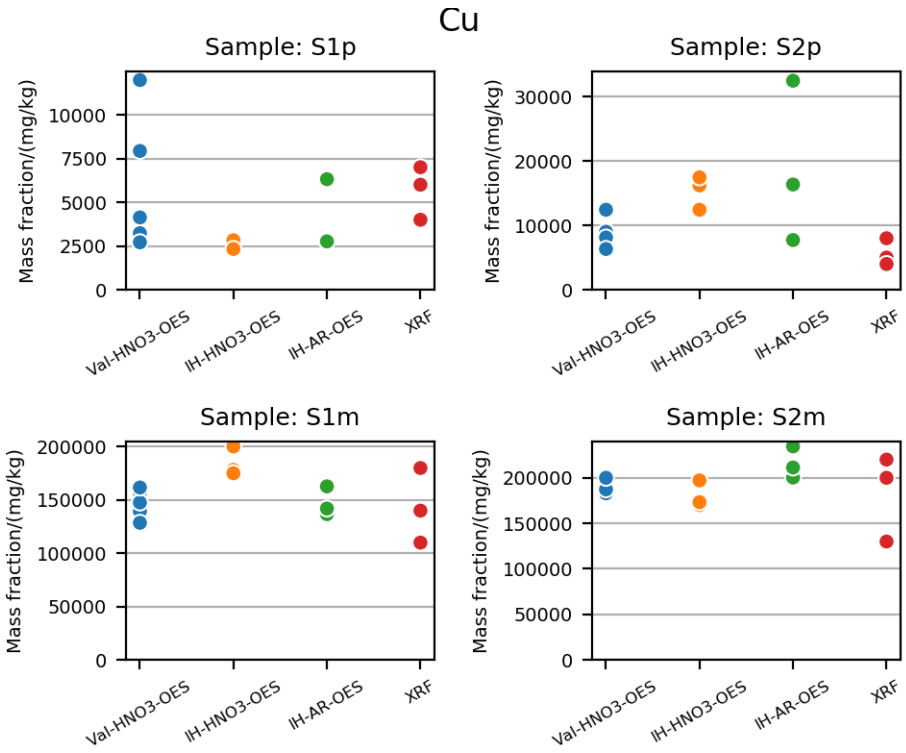

Figure S 10 Measured mass fractions of Cu in SLF samples.

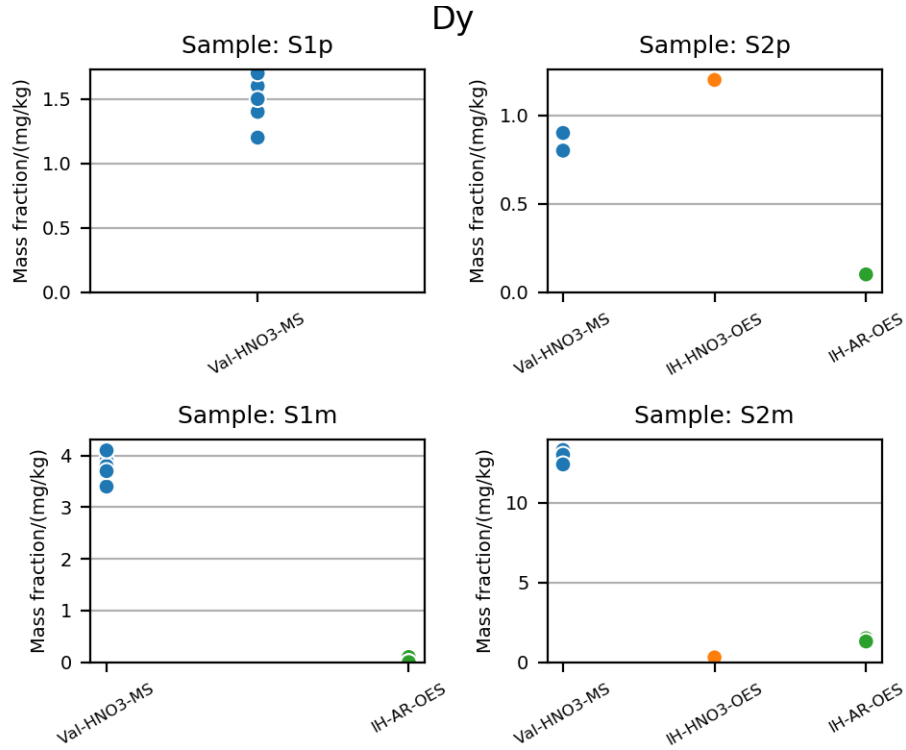

Figure S 11 Measured mass fractions of Dy in SLF samples.

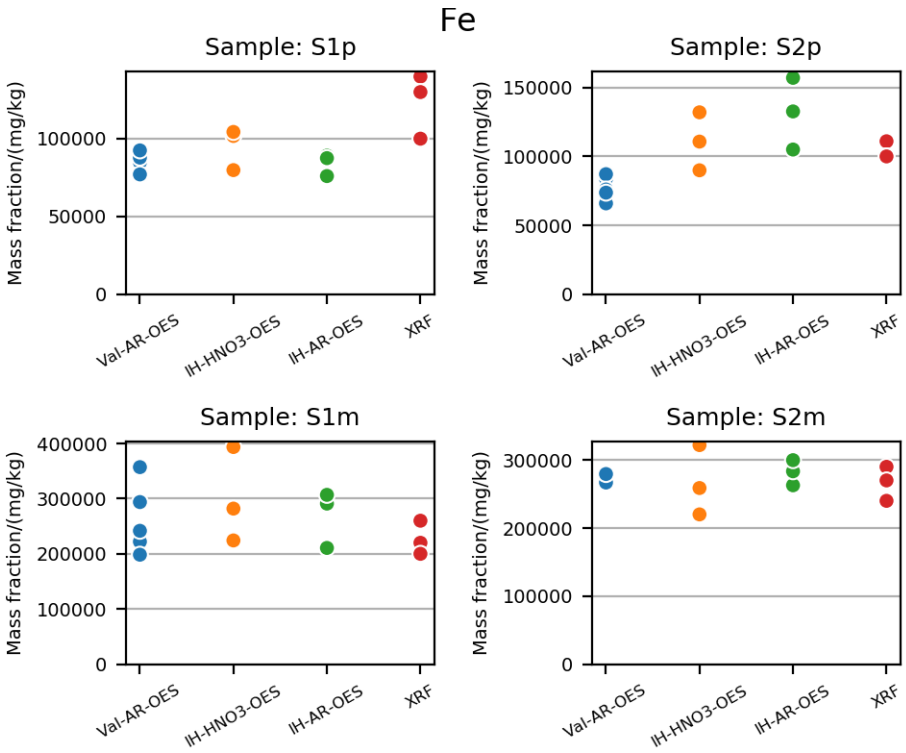

Figure S 12 Measured mass fractions of Fe in SLF samples.

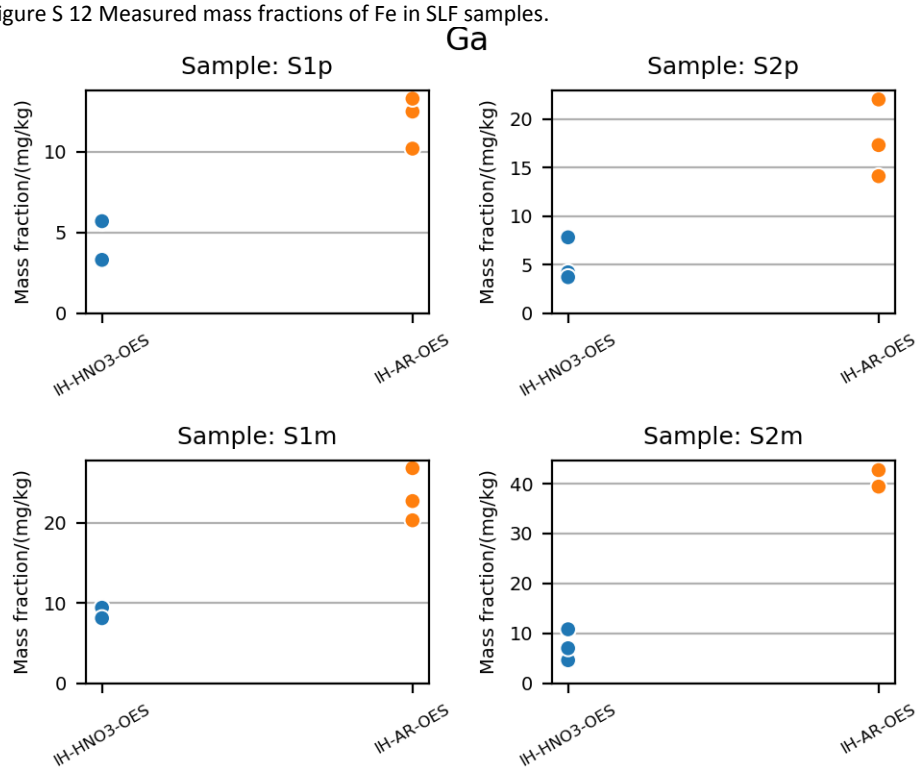

Figure S 13 Measured mass fractions of Ga in SLF samples.

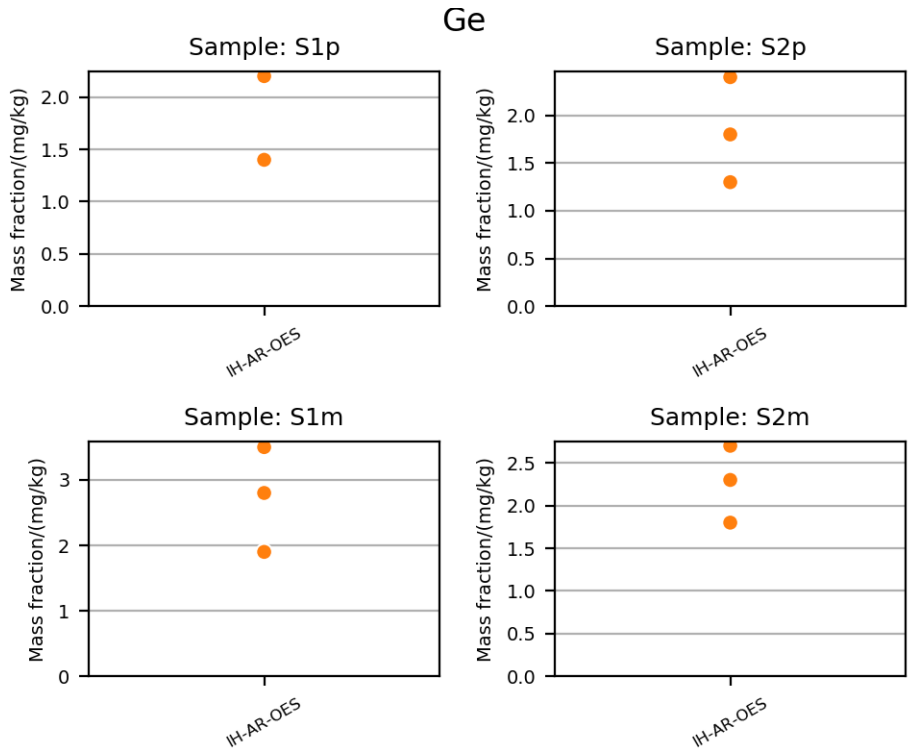

Figure S 14 Measured mass fractions of Ge in SLF samples.

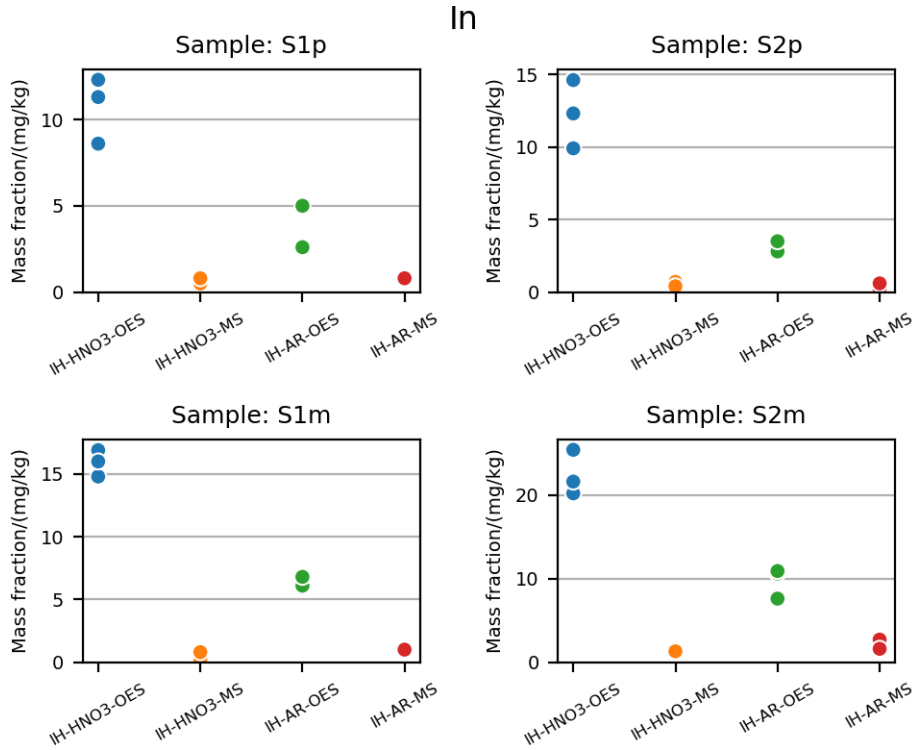

Figure S 15 Measured mass fractions of In in SLF samples.

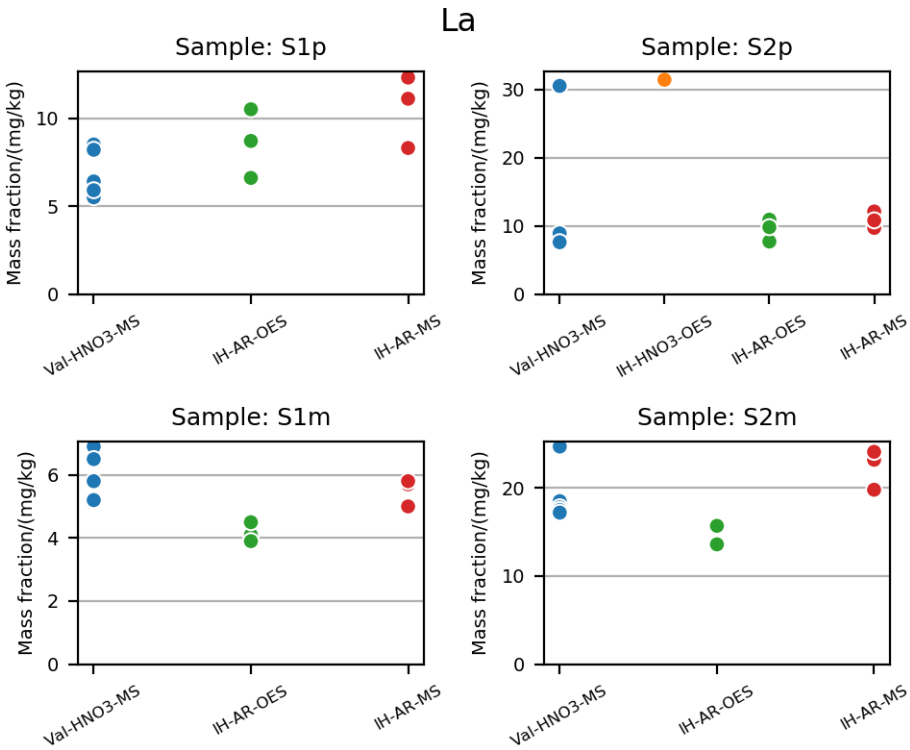

Figure S 16 Measured mass fractions of La in SLF samples.

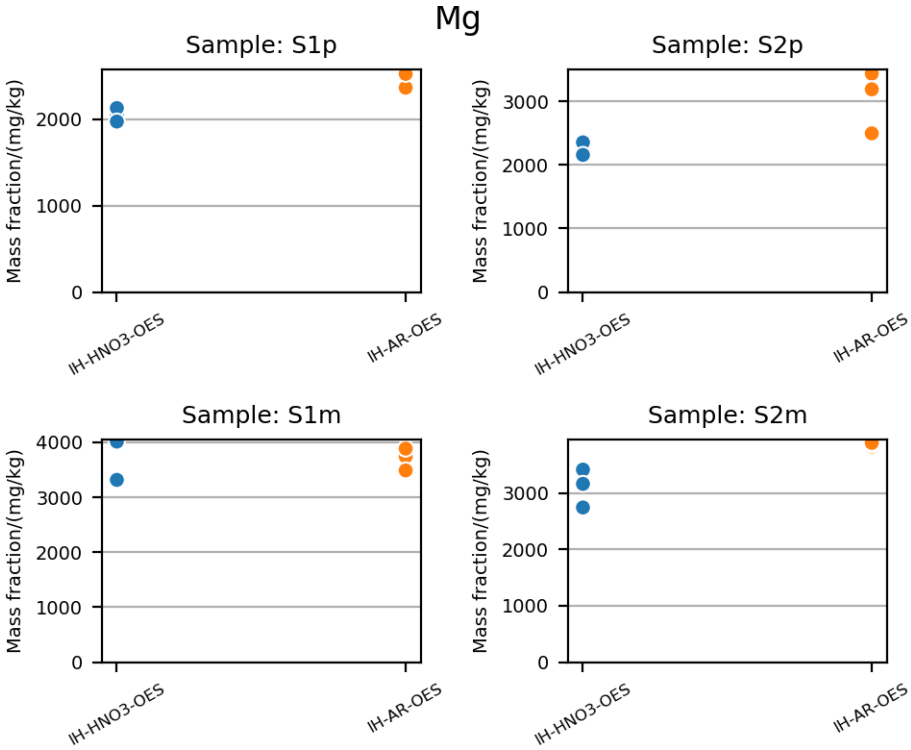

Figure S 17 Measured mass fractions of Mg in SLF samples.

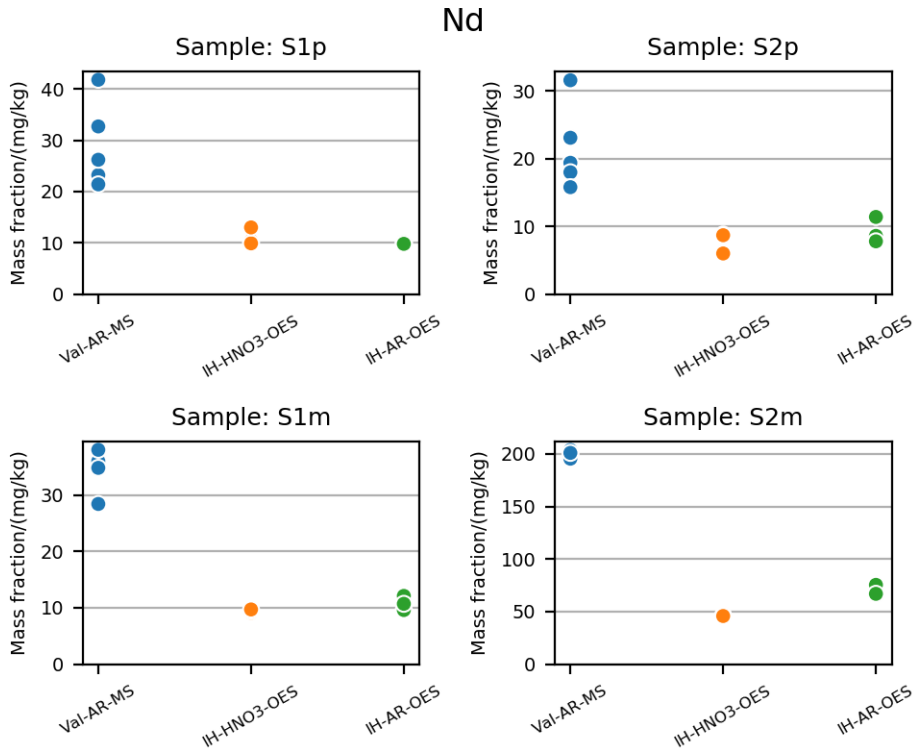

Figure S 18 Measured mass fractions of Nd in SLF samples.

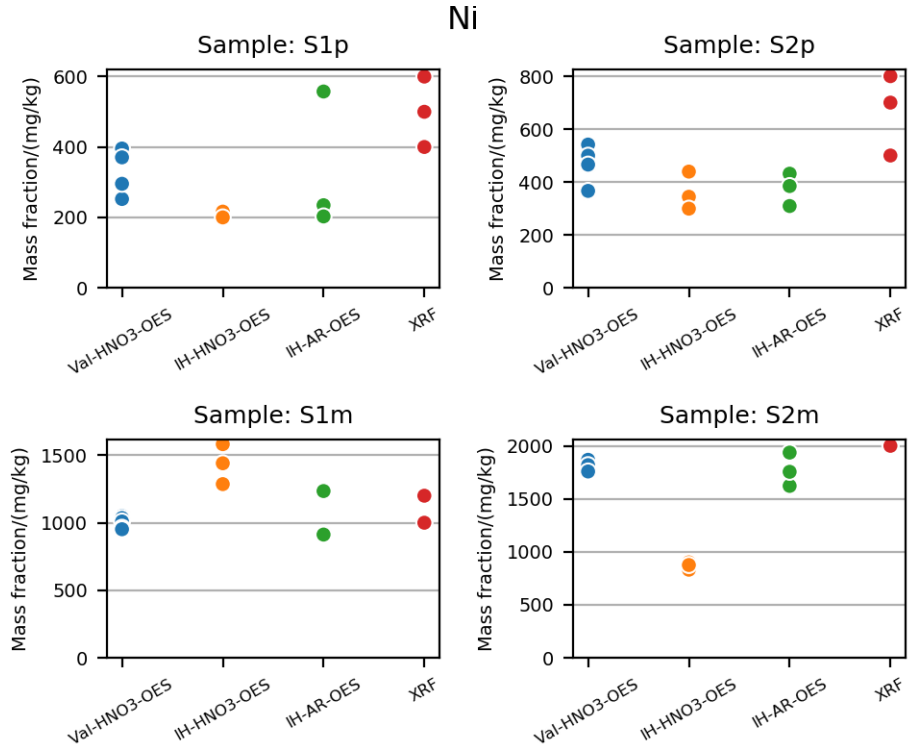

Figure S 19 Measured mass fractions of Ni in SLF samples.

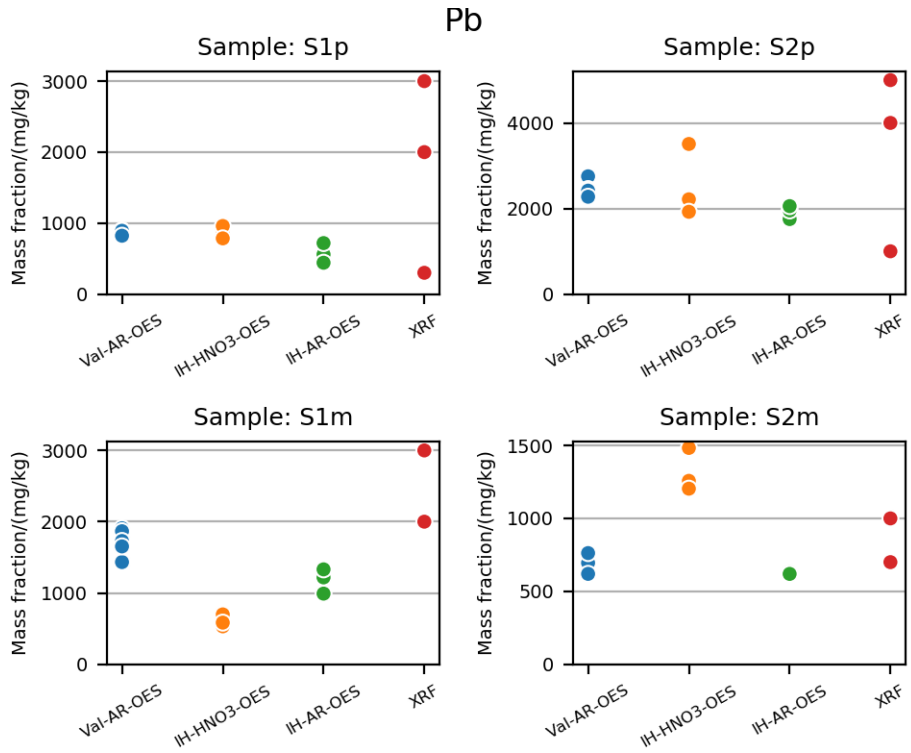

Figure S 20 Measured mass fractions of Pb in SLF samples.

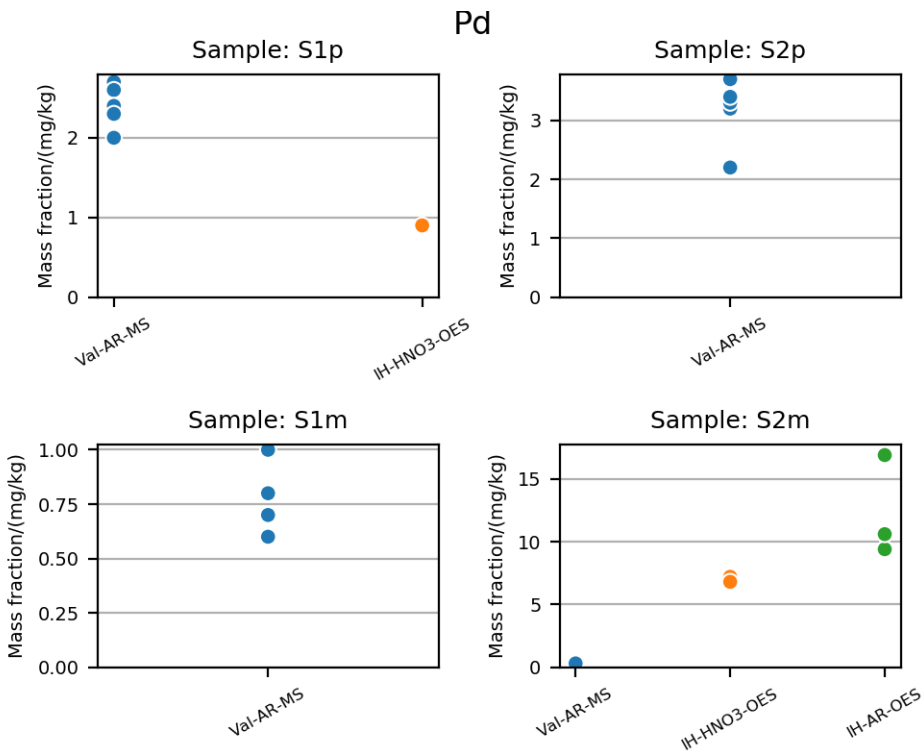

Figure S 21 Measured mass fractions of Pd in SLF samples.

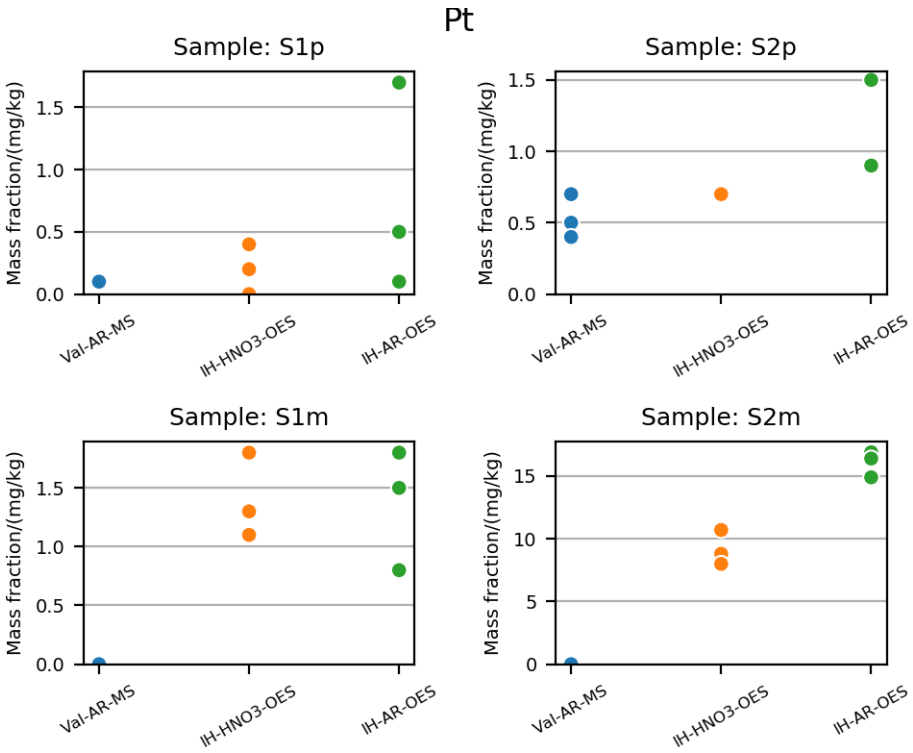

Figure S 22 Measured mass fractions of Pt in SLF samples.

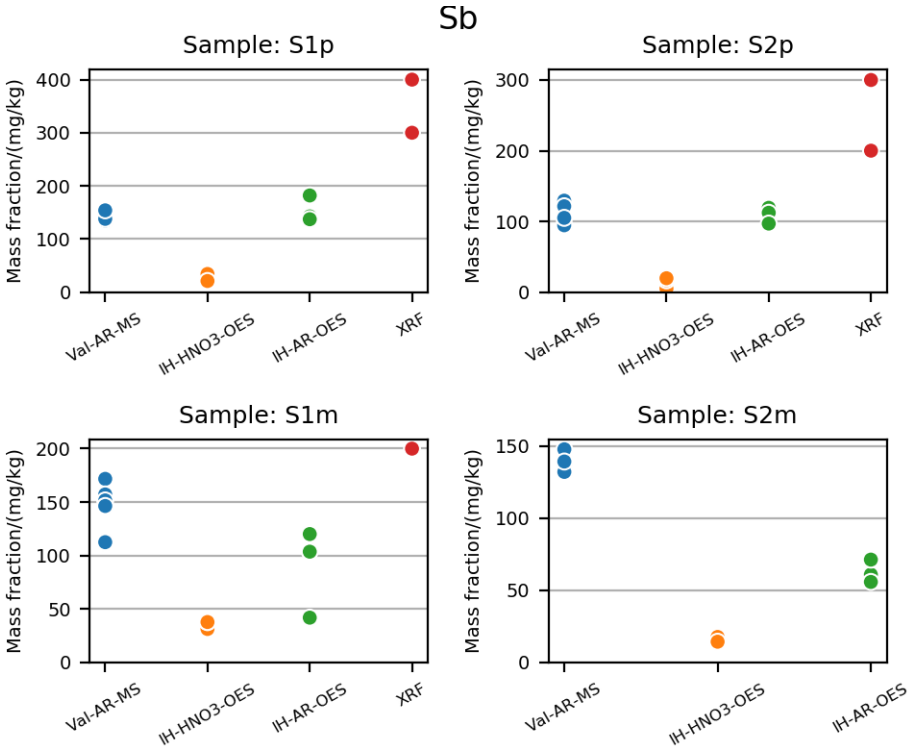

Figure S 23 Measured mass fractions of Sb in SLF samples.

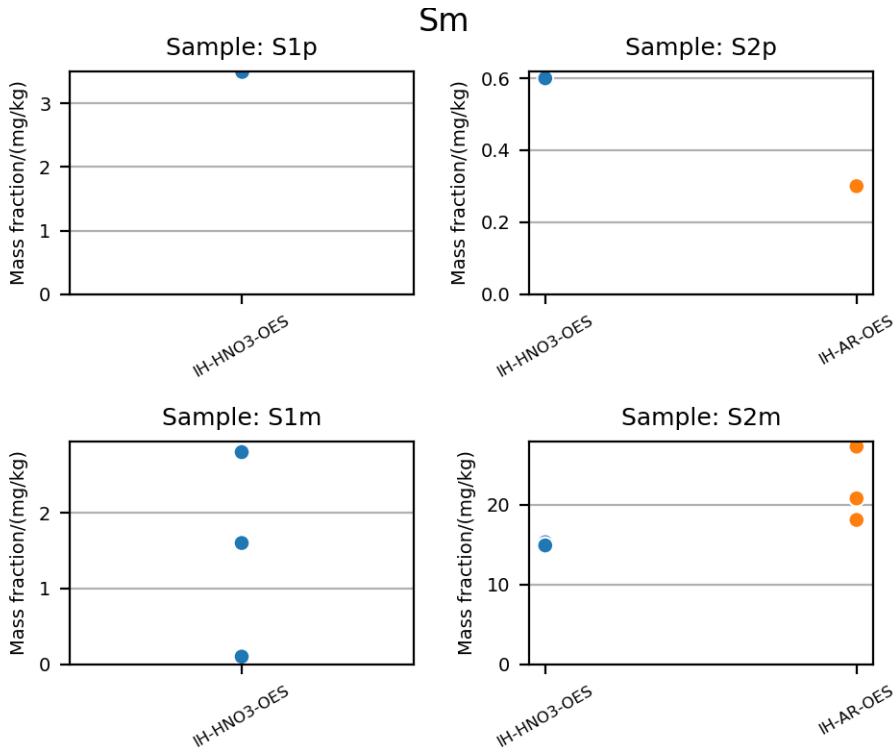

Figure S 24 Measured mass fractions of Sm in SLF samples.

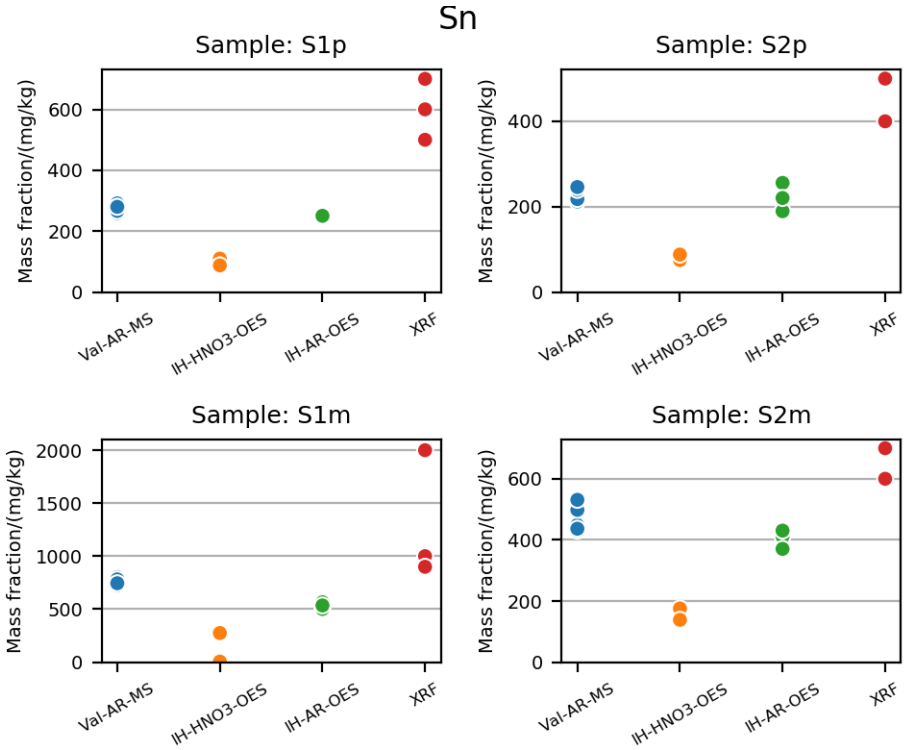

Figure S 25 Measured mass fractions of Sn in SLF samples.

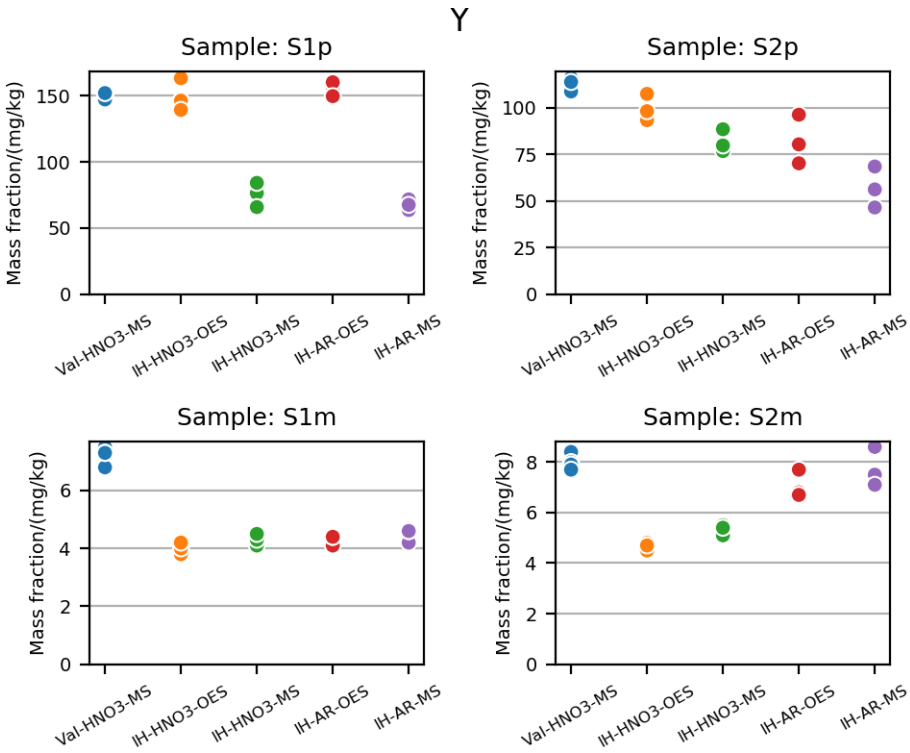

Figure S 26 Measured mass fractions of Y in SLF samples.

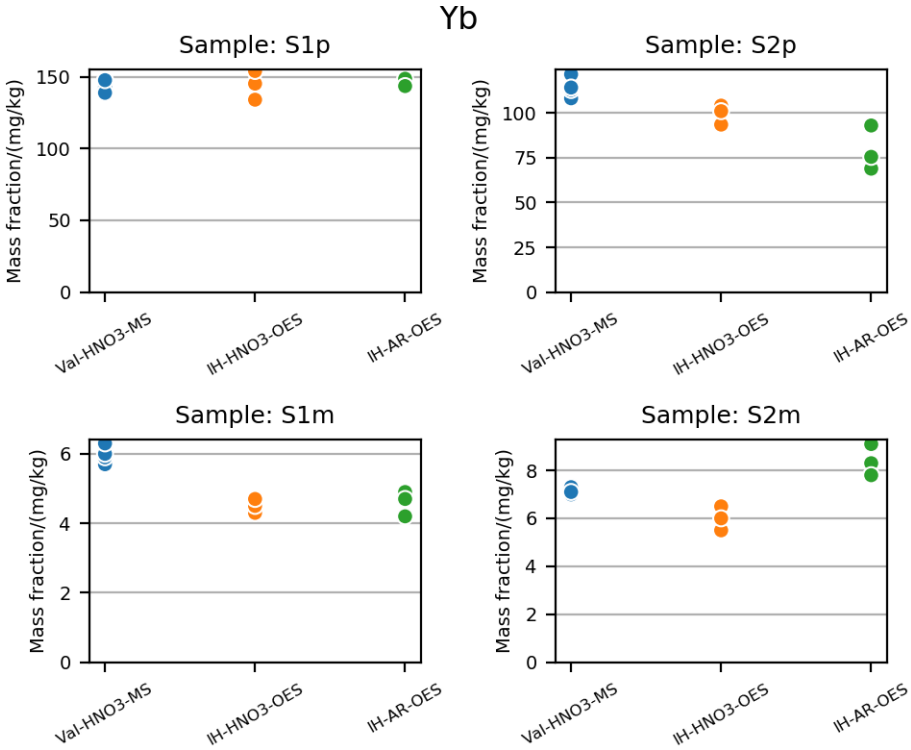

Figure S 27 Measured mass fractions of Yb in SLF samples.