Supplementary Information to:

A single-step purification method for the precise determination of antimony isotopic composition of environmental, geological and biological samples by HG-MC-ICP-MS

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Table S1: Intrument settings for ICP-MS and HG-ICP-MS analysis (adapted from Resongles et al. 2015[1]).

Instrument settings	ICP-MS	HG-ICP-MS
Instrument	iCAP TQ, Thermo Scientific	iCAP Q, Thermo Scientific
RF Power (W)	1550	1550
Cooling (L min^{-1})	14.0	14
Auxiliary (L min^{-1})	0.8	0.8
Sample (L min^{-1})	1.03	/
Data acquisition parameters		
Wash time (s)		120
Uptake time (s)		90
Number of blocks		3
Number of cycles per block		/
Dwell time (ms)		100
Integration time per cycle (s)	/	/
Hydride generation parameters		
System	/	ESI HydrideICP
Reducing agent	/	1% w/v NaBH4 in 0.05% w/v NaOH
Sample	/	3 M HCl, 0.5 % (w/v) KI/AA
Internal standard	/	3 M HCl + 4 µg L^{-1} Te
Gas flow (L min^{-1})		
Sample gas	/	0.2
Add gas	/	0.7
Reagent flow (ml.min^{-1})		
Reducing agent	/	0.24
Sample	/	0.48
Internal standard	/	0.12

[1]E. Resongles, R. Freydier, C. Casiot, J. Viers, J. Chmeleff and F. Elbaz-Poulichet, *Talanta*, 2015, **144**, 851–861.

Table S2: Residual Element:Sb ratios measured by ICP-MS in the purified samples after the purification process.

CRM	Residual Cd:Sb ratio	Residual Co:Sb ratio	Residual Cr:Sb ratio	Residual Cu:Sb ratio	Residual Fe:Sb ratio	Residual Ni:Sb ratio	Residual As:Sb ratio	Residual Pb:Sb ratio	Residual Sn:Sb ratio
NCS-DC-73349	0.0026	0.02	1	0.1	10.9	0.35	0.01	0.245	0.06
PACS-3	0.0002	0.03	0.3	0.2	3.4	0.09	0.004	0.03	0.02
NCS-DC-70317	0.0001	0.03	0.2	0.2	13.4	0.05	0.004	0.002	0.02
SDO-1	0.001	0.02	0.3	0.1	2.3	0.1	0.003	0.002	0.02
GSD-3	0.0005	0.01	0.2	0.1	3.1	0.08	0.004	0.01	0.02
Urine Control Clincheck II	0.0003	0.01	0.1	< 0.0004	1.1	0.03	0.003	< 0.001	0.01
Whole blood trace elements II	0.0003	0.03	0.001	2.3	3.6	0.1	0.005	0.006	0.03
BCR-176R	0.0002	0.02	0.2	< 0.0025	1.6	0.05	0.004	0.007	0.02
ERM-EC680m	0.0001	0.001	0.01	0.004	0.10	0.005	0.0002	0.007	0.004
Average BCR 482 (n=3)	0.0003	0.02 ± 0.001	0.2 ± 0.02	0.3 ± 0.2	1.6 ± 0.3	0.05 ± 0.01	0.003 ± 0.000	0.008 ± 0.001	0.02 ± 0.002
Average NIST 2711a (n=3)	0.0004 ± 0.0002	0.01 ± 0.01	0.2 ± 0.1	0.04 ± 0.02	1.8 ± 0.7	0.05 ± 0.02	0.003 ± 0.000	0.006 ± 0.002	0.02 ± 0.002
Average BCR 723 (n=3)	0.0002 ± 0.0001	0.01 ± 0.001	0.2 ± 0.1	0.17 ± 0.03	3.5 ± 3.3	0.05 ± 0.02	0.003 ± 0.000	0.012 ± 0.004	0.02 ± 0.002
Average GXR4 (n=3)	0.0012 ± 0.0002	0.02 ± 0.01	0.2 ± 0.1	3 ± 3	2.9 ± 1.0	0.07 ± 0.03	0.003 ± 0.001	0.004 ± 0.003	0.03 ± 0.02

Table S3: Comparison of the δ^{123}Sb values (‰) and their uncertainties (2 standard deviation, 2sd) obtained following the statistical treatment of isotope analysis data (outlier exclusion) using methods A and B.

	Method A		Method B		
CRM	**δ^{123}Sb (‰)**	**2sd**	**δ^{123}Sb (‰)**	**2sd**	**n**
NCS-DC-73341	-0.52	0.08	-0.52	0.06	2
PACS-3	0.16	0.07	0.16	0.02	4
NCS-DC-70317	-0.10	0.01	-0.11	0.03	3
SDO-1	0.23	0.05	0.22	0.03	3
GSD-3	0.13	0.10	0.15	0.05	4
Urines Control Clincheck II	0.19	0.12	0.17	0.04	3
Whole blood trace elements II	0.09	0.02	0.09	0.02	3
BCR-176R	-0.03	0.07	-0.03	0.03	4
ERM EC680m	0.40	0.03	0.40	0.03	3
BCR-482	0.07	0.10	0.05	0.02	9
NIST 2711a	0.06	0.03	0.06	0.02	3
BCR 723	0.03	0.05	0.03	0.05	3
GXR4	0.36	0.09	0.35	0.04	9
NIST 1643f	-0.03	0.13	-0.08	0.09	3

Table S4: Final $\delta^{123}Sb$ values (data processed using method B for the statistical treatment) of the different CRM with their uncertainties (2 standard deviation, 2sd) and the number of measurements of the purified solution (n).

CRM	$\delta^{123}Sb$ (‰)	2sd	n
NCS-DC-73349	-0.52	0.06	2
PACS-3	0.16	0.02	4
NCS-DC-70317	-0.11	0.03	3
SDO-1	0.22	0.03	3
GSD-3	0.15	0.05	4
Urine Control Clincheck II	0.17	0.04	3
Whole blood trace elements II	0.09	0.02	3
BCR - 176R	-0.03	0.03	4
ERM-EC680m	0.40	0.03	3
BCR 482	0.05	0.05	9*
NIST 2711a	0.06	0.02	3
BCR 723	0.03	0.05	3
GXR4	0.35	0.04	9*
NIST 1643f	-0.08	0.09	3

*CRM with 3 measurements for each triplicate (digestion and purification step) showing external reproducibility of the complete method

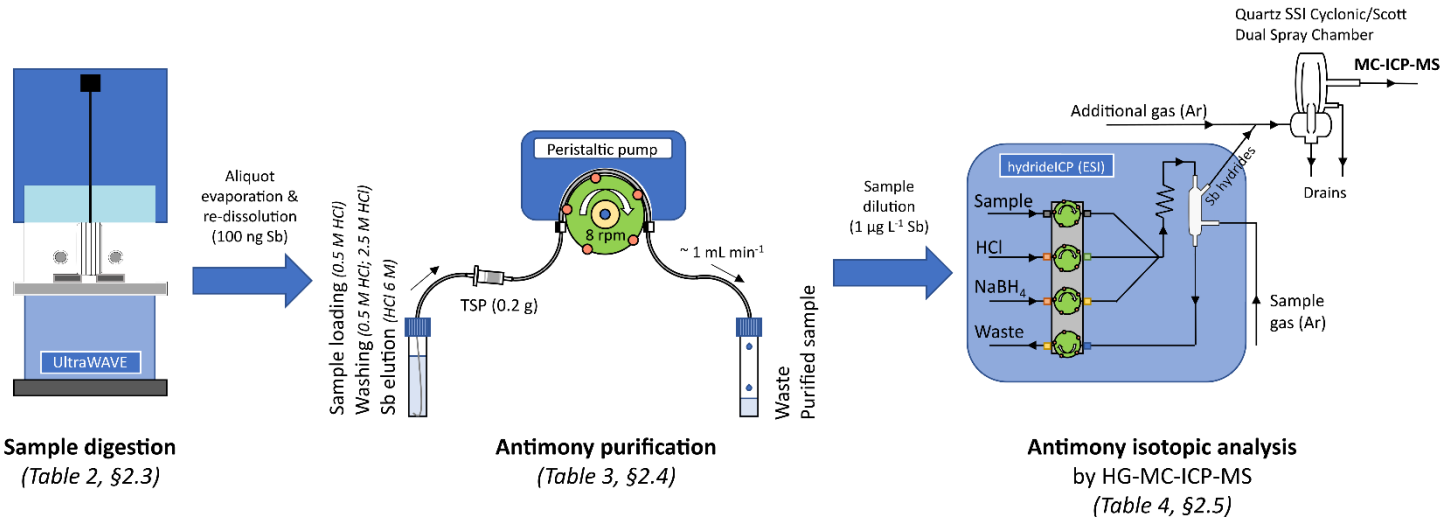

Figure S1: Scheme of the procedure for the determination of Sb isotopic composition in environmental, biological and geological solid samples including sample digestion, antimony purification and antimony isotope analysis. For the purification step, a 6-channel Gilson® peristaltic pump (at 8 rpm) with 1.02 mm i.d. Tygon® tubing allowed to run six samples simultaneously at a flow rate of ~ 1 mL min^{-1}.

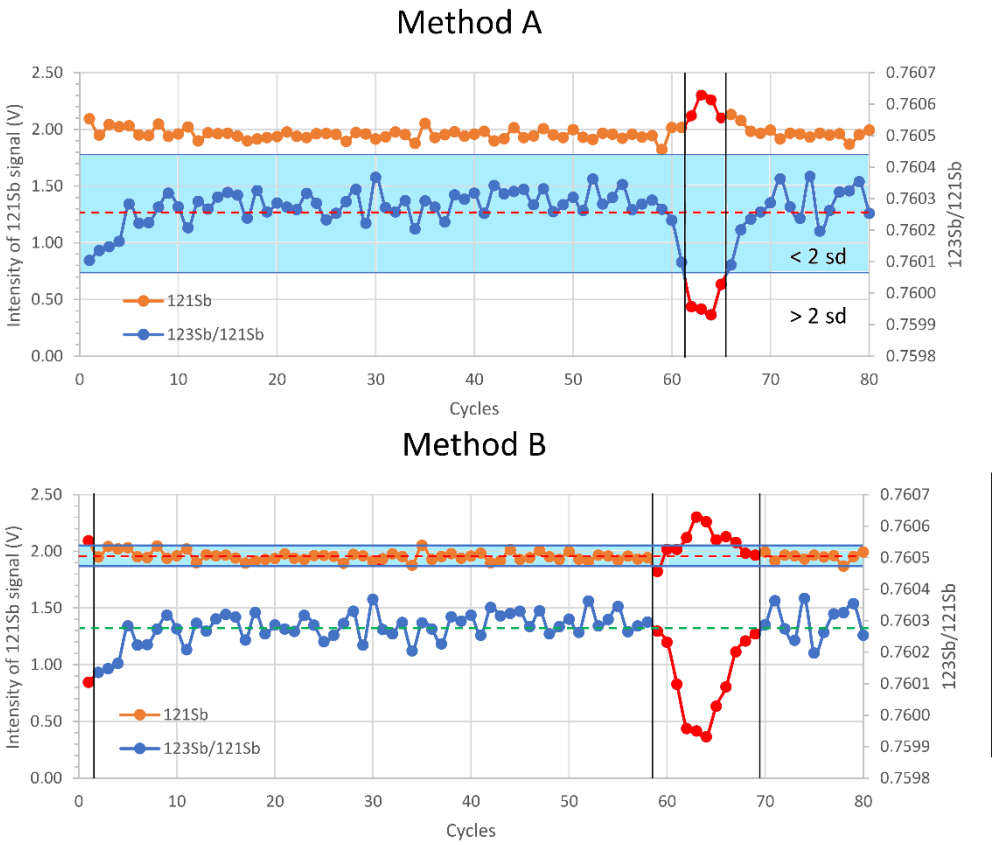

Figure S2: Comparison of methods A and B for the exclusion of outlier values of measured $^{123}Sb/^{121}Sb$ ratio. The blue area represents the 2 standard deviation area from the mean $^{123}Sb/^{121}Sb$ ratio (method A) or the mean ^{121}Sb intensity ± 5 % (method B). Dashed lines represent the mean values of signal intensity and isotope ratio.