## A hydrofluoric acid-free method to dissolve and quantify silica nanoparticles in aqueous and solid matrices

David Bossert,<sup>1</sup> Dominic A. Urban,<sup>1</sup> Mattia Maceroni,<sup>1</sup> Liliane Ackermann-Hirschi,<sup>1</sup> Laetitia Haeni,<sup>1</sup> Phattadon Yajan,<sup>1</sup> Miguel Spuch-Calvar,<sup>1</sup> Barbara Rothen-Rutishauser,<sup>1</sup> Laura Rodriguez-Lorenzo,<sup>1</sup> Alke Petri-Fink, \*<sup>1,2</sup> Fabienne Schwab\*<sup>1</sup>

## Affiliations:

<sup>1</sup>Adolphe Merkle Institute, University of Fribourg, Chemin des Verdiers 4, 1700 Fribourg, Switzerland

<sup>2</sup>Chemistry Department, University of Fribourg, Chemin du Musée 9, 1700 Fribourg, Switzerland

\*Correspondence: alke.fink@unifr.ch +41 26 300 9501, fabienne.schwab@unifr.ch +41 26 300 9510

## SUPPLEMENTAL INFORMATION

This supplemental information contains more details on the nanomaterial characterization (**Supplementary Figure S1 + Table S1**), the temperature and pressure profile of the microwave digestion (**Supplementary Figure S2**), and data on the calibration parameters (**Supplementary Table S2**).

## **Supporting Information**



**Supplementary Figure S1:** Transmission electron microscopy (TEM) characterization of in house synthesized amorphous colloidal silica nanoparticles (SiO<sub>2</sub>-NPs) and commercially available amorphous fumed Aerosil<sup>®</sup> 200 SiO<sub>2</sub>-NPs (Evonik, former Degussa).

**Supplementary Table S1**: Overview of particle parameters as determined by transmission electron microscopy (TEM) and dynamic light scattering (DLS) of the amorphous silica nanomaterials. PDI: Polydispersity index. Zeta: Zeta potential, which represents the surface charge of the particles.

	d <sub>тем</sub> (nm)	d <sub>DLS</sub> (nm)	PDI	Zeta (mV)
Colloidal SiO <sub>2</sub> -NPs	397 ± 22	412	0.005	-82.7 ± 7.7
Fumed SiO <sub>2</sub> -NPs	13 ± 5ª	267	0.22	-42.4 ± 2.8

<sup>a</sup>diameter of primary particles.



**Supplementary Figure S2:** Temperature and power measured in the microwave PTFE digestion vessels. The temperature in each vessel was monitored by a built-in infrared sensor. The graph shows the temperature in black and the applied power in red for the 24 PTFE microwave vessels in one run. The dotted line represents the programmed route.

	Si				Υ	
	Sensitivity (a.u. µg <sup>-1</sup> L)	Intercept (a.u.)	R² (-)	Blanks (μg L <sup>-1</sup> )	Sensitivity (a.u. μg-1 L)	Intercept R <sup>2</sup> (-)
ICP-OES	mean ± s.d.	mean ± s.d.		BEC ± s.d. LOD LOQ	mean ± s.d.	mean ± s.d.
matrix-matched + H <sub>2</sub> SO <sub>4</sub> + digested	652 ± 9.5	8731 ± 5342	0.9987	23.2 ± 1.3 27 35.8	17279 ± 368	-414464 ± 208303 0.9977
matrix-matched+H <sub>2</sub> SO <sub>4</sub>	619 ± 4.7	10267 ± 2633	0.9997	18.4 ± 0.8 20.7 26.2	16467 ± 211	-52758 ± 119408 0.9992
background solution	602 ± 3.4	14898 ± 1898	0.9998	26.1 ± 0.2 26.6 27.8	18204 ± 321	-115106 ± 181764 0.9984
water+H <sub>2</sub> SO <sub>4</sub>	624 ± 3.6	13501 ± 2031	0.9998	15.9 ± 0.1 16.3 17.4	17692 ± 170	18118 ± 95968 0.9995
	Sensitivity (a.u. mg <sup>-1</sup> L)	Intercept (a.u.)				
Colorimetry	mean ± s.d.	mean ± s.d.				
RT+KOH0.1	0.722 ± 0.03	-0.0394 ± 0.076	0.9960	53.6 ± 7.8 69.2 123		
RT+KOH1.0	$0.715 \pm 0.02$	-0.0459 ± 0.037	0.9980	56 ± 9 82 143		
au · arbitrary units sd·s	tandard deviation	BEC background	equivalent c	oncentration I OD: instrument lir	nit of detection IO	O: instrument limit of

Supplementary Table S 2: Fitting parameters for the different types of calibrations measured by inductively coupled plasma – optical emission spectrometry (ICP-OES) and colorimetry.

a.u.: arbitrary units. s.d.: standard deviation. BEC: background equivalent concentration. LOD: instrument limit of detection. LOQ: instrument limit of quantification. RT: room temperature.