## **Supplementary Material**

Simultaneous speciation analysis of chromium and antimony by novel carboxyl-functionalized hybrid monolithic column solid phase microextraction coupled with ICP-MS

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Parameter	Settings	
Rf power	1100 W	
Nebulizer gas flow rate	0.92 L min <sup>-1</sup>	
Auxiliary gas flow rate	1.2 L min <sup>-1</sup>	
Plasma gas flow rate	15 L min <sup>-1</sup>	
Sampling cone	Ni/1.1 mm	
Skimmer cone	Ni/0.9 mm	
Acquisition mode	Time-resolved data acquisition	
Reaction mode	KED mode	
Scanning mode	Peak-hopping	
Dwell mode	250 ms	
Integration mode	Peak area	
Isotope monitored	<sup>52</sup> Cr, <sup>121</sup> Sb	

 Table S1. Operating parameters of ICP-MS.

Element	Percentage of weight (%)	
С	14.50±0.36	
Н	3.82±0.11	
Ν		
S		

**Table S2.** Elemental analysis of the TMOS-*co*-CES column.

Co-existing	Concentration	Recovery (%)	
ion	(mg L <sup>-1</sup> )	Sb(III)	Cr(III)
Na <sup>+</sup>	5	97.94±0.63	98.10±0.04
$K^+$	5	98.10±0.99	98.37±0.03
Ca <sup>2+</sup>	5	98.30±0.35	97.80±1.06
$Mg^{2+}$	1	98.84±0.10	98.66±0.01
$Zn^{2+}$	1	97.83±1.33	98.72±0.13
$Al^{3+}$	1	98.75±0.25	98.69±0.03
Fe <sup>2+</sup>	1	95.35±1.29	98.73±0.04
Fe <sup>3+</sup>	1	95.41±0.66	98.86±0.12
Ni <sup>2+</sup>	1	96.00±3.56	98.47±0.16
Ba <sup>2+</sup>	1	98.73±0.82	98.12±0.03
Cu <sup>2+</sup>	0.5	98.68±0.12	98.60±0.07
$Pb^{2+}$	0.5	96.01±4.04	98.76±0.03
NO <sub>3</sub> -	13.5	97.94±0.63	98.10±0.04
PO <sub>4</sub> <sup>3-</sup>	5	98.97±0.06	98.55±0.14
SO <sub>4</sub> <sup>2-</sup>	4	98.84±0.10	98.66±0.01
Cl-	2	97.83±1.33	98.72±0.13

Table S3. The interference effects of diverse ions.



Fig. S1. Pressure of the TMOS-co-CES column with different elution flow rates.



Fig. S2. Energy dispersive X-ray of the TMOS-*co*-CES column.



Fig. S3. Element mapping images of the TMOS-*co*-CES column.



Fig. S4. Adsorption rates of inorganic Sb on the thiol-functionalized hybrid monolithic column under different pH. Sample volume: 1 mL. Concentration of each species: 20  $\mu$ g L<sup>-1</sup>. Flow rate: 20  $\mu$ L min<sup>-1</sup>.



**Fig. S5.** Effect of flow rate in the extraction step. Sample pH: 4.5. Other conditions are the same with Fig. S4.



**Fig. S6.** Effect of flow rate in the elution step. Eluent: 150  $\mu$ L 10% HNO<sub>3</sub> (v/v). The flow rate in the extraction step: 200  $\mu$ L min<sup>-1</sup>. Other conditions are the same with Fig. S5.



**Fig. S7.** Effect of sample volume on the adsorption rates of Cr(III) and Sb(III) on the TMOS-*co*-CES monolithic column measured by using various volume solutions (1.0, 2.0, 4.0, 5.0 and 10 mL, respectively) (pH 4.5) containing 50 ng Cr(III) and Sb(III) as sample solutions. Flow rate: 200  $\mu$ L min<sup>-1</sup>.



**Fig. S8.** The breakthrough curve obtained with 5 cm TMOS-*co*-CES column for Sb(III) and Cr(III). Concentration of Cr(III)/Sb(III): 2.0 mg L<sup>-1</sup>.



Fig. S9. The adsorption isotherms of Sb(III) and Cr(III) on the TMOS-co-CES column.



Fig. S10. The reusability of the TMOS-co-CES column under the optimized conditions.