

FLOW ANALYSIS-HYDRIDE GENERATION-GAS PHASE DERIVATIVE MOLECULAR ABSORPTION SPECTROPHOTOMETRIC DETERMINATION OF ANTIMONY IN ANTILEISHMANIAL DRUGS

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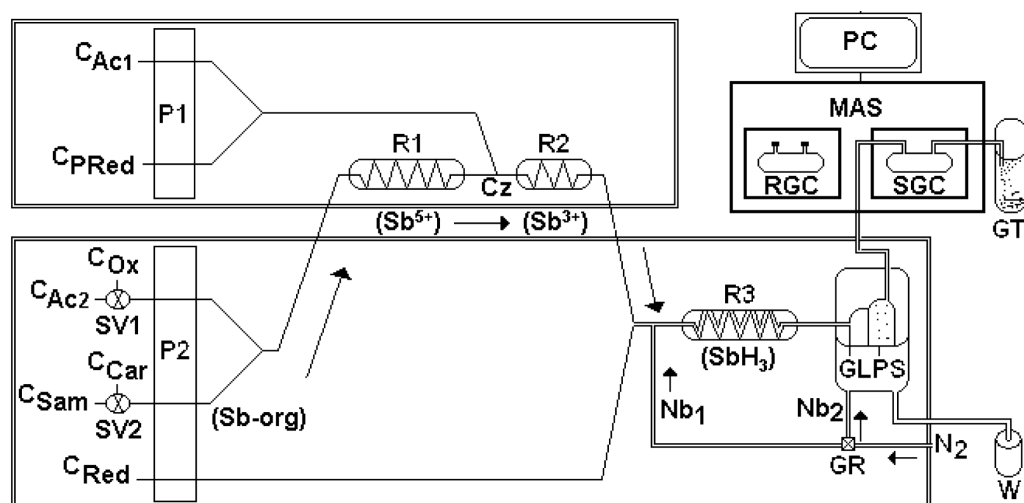


Figure 1S. Schematic diagram of the FA-HG-GPMAS coupled system proposed. C_{Ac} , acid agent; C_{PreRed} pre-reducing agent; C_{Ox} oxidizing agent; C_{Car} carrier; C_{Sam} sample/standard; C_{Red} final reducing agent; P1, independent peristaltic pump; P2, peristaltic pump from the Varian GLPS system; SV, manual selecting valve; R1, oxidation/mineralization reaction coil; R2, pre-reducing reaction coil; R3, hydride generation coil; Cz, confluence point; GLPS, gas-liquid phase separator VGA-77; N_2 , nitrogen gas entry; Nb, nitrogen branch; GR, regulated supply of nitrogen; MAS, molecular absorption spectrophotometer; RGC, reference gas cell; SGC, sample gas cell; PC, Computer; GT, gas trapper; W, waste; Sb-org, organic antimony. Further details are given in Table 1S

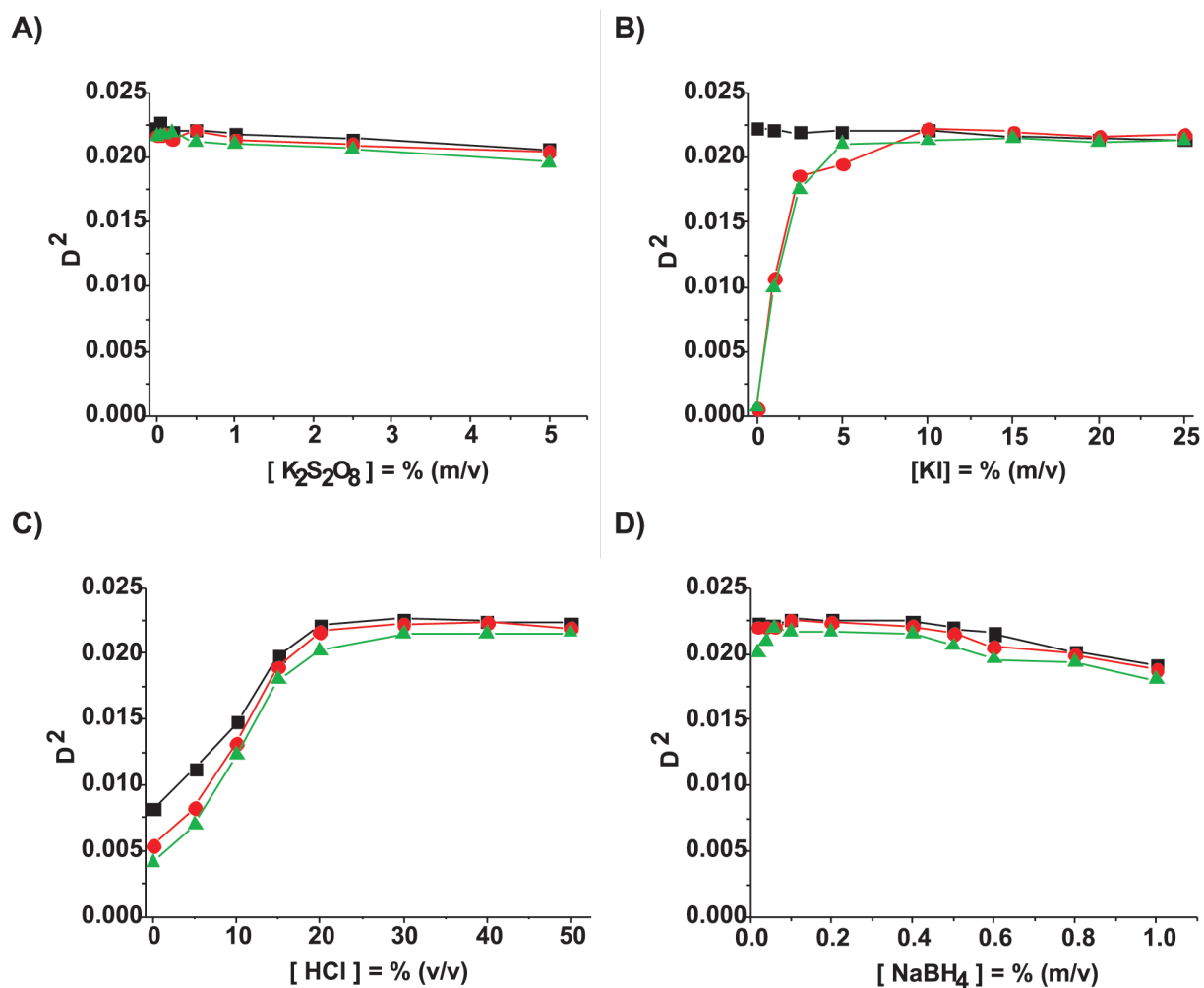


Figure 2S. Effect of chemical parameters on the analytical signal: A) oxidizing agent; B) pre-reducing agent; C) acidic medium; D) reducing agent. (■) $5.0 \mu\text{g Sb(III) mL}^{-1}$; (●) $5.0 \mu\text{g Sb(V) mL}^{-1}$; and (▲) sample solution containing an equivalent concentration to the standards ($\cong 4.86 \mu\text{g Sb mL}^{-1}$). D^2 , signal of the second derivative spectrum [peak to zero baseline $D^2_{(224 \text{ nm})}$]

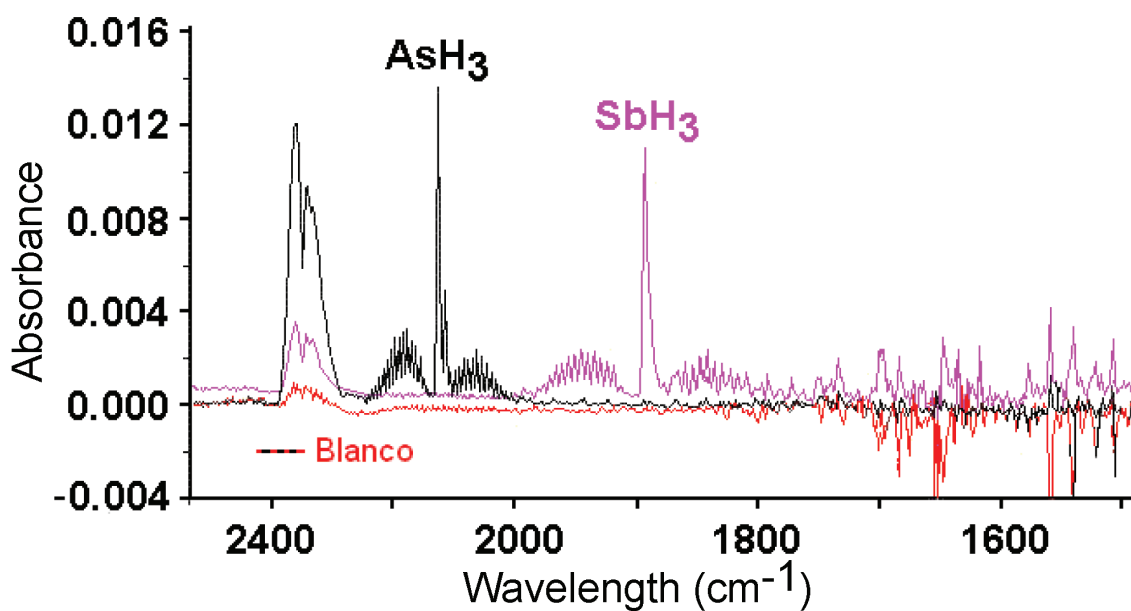


Figure 3S. Representative FTIR spectra of (a) blank, (b) sample of GCT $100 \mu\text{g Sb(V) mL}^{-1}$, and (c) sample of GCT $100 \mu\text{g Sb(V) mL}^{-1}$ enrichment with a standard of $10 \mu\text{g As(III) mL}^{-1}$. Spectra were obtained following the FA-HG-FTIR method proposed by Galignani et al.²⁵

Table 1S. Operating conditions of the FA-HG-GPMAS coupled system

Instrumental and spectroscopic parameters		Description	
UV-MAS	Spectral range	190-300 nm	
	Nominal resolution	1 nm	
	Scan speed	960 nm	
	Measurement criteria	Second order derivative of the peak to zero baseline at 224 nm	
	Gas cell type:	Manufacturer	Wilmad
		Path length	100 mm
		Internal diameter	22 mm
		Windows material	Quartz (circular shape)
		Dead volume	31.4 mL
	Operating temperature		Room (20-23 °C)
Flow analysis-Hydride generation system parameters		Description	
FA-HG	(C _{Ac1}) acid channel composition	HCl (20%, v/v)	
	(C _{Ac1}) acid channel flow rate	2 mL min ⁻¹	
	(C _{PRed}) pre-reducing agent composition	Aqueous KI (15%, m/v)	
	(C _{PRed}) pre-reducing agent flow rate	2 mL min ⁻¹	
	(C _{Ac2}) acid channel composition	HCl (50%, v/v)	
	(C _{Ac1}) acid channel flow rate	1 mL min ⁻¹	
	(C _{Car}) carrier composition	H ₂ O	
	(C _{Car}) carrier flow rate	4 mL min ⁻¹	
	(C _{Sam}) sample composition	Sb(V) from Glucantime® and Ulamina; about 5 µg mL ⁻¹	
	(C _{Sam}) standard composition	Sb(V) from K ₄ Sb ₂ O ₇ ; 0 - 10 µg mL ⁻¹	
	(C _{Sam}) diluting agent of samples/standards	H ₂ O	
	(C _{Sam}) sample/standard flow rate	4 mL min ⁻¹	
	(C _{Red}) reducing agent composition	NaBH ₄ (0.2%, m/v) in NaOH (0.1%, m/v)	
	(C _{Red}) reducing agent flow rate	1 mL min ⁻¹	
	(N _{b1}) stripping gas flow rate	45 mL min ⁻¹	
	(N _{b2}) carrier gas flow rate	45 mL min ⁻¹	
	(GR-N ₂) gas pressure	50 psi	
	(GT) gas trapping composition	AgNO ₃ (0.5%, m/v)	
	(R1) acidifying coil	PTFE (1000 x 0.5 mm)	
	(R2) pre-reducing reaction coil	PTFE (500 x 0.8 mm)	
(R3) hydride generation coil	PTFE (600 x 1.5 mm)		