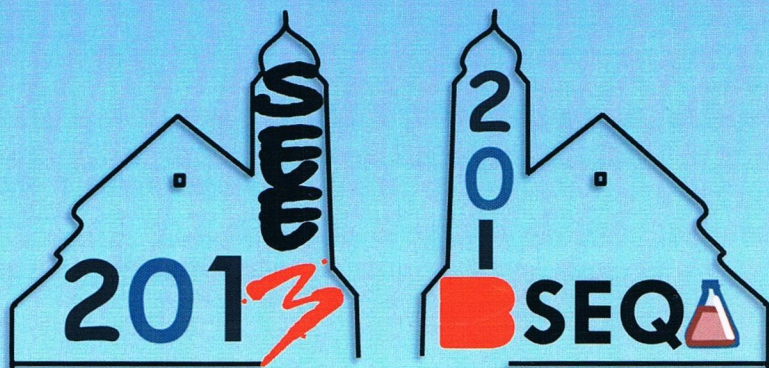


# XVIII REUNIÓN DE LA SOCIEDAD ESPAÑOLA DE QUÍMICA ANALÍTICA



## VI REUNIÓN DE LA SOCIEDAD ESPAÑOLA DE ESPECTROMETRÍA DE MASAS



Úbeda, 16–19 junio 2013



**DETERMINATION OF MERCURY BY FLOW INJECTION SOLID PHASE EXTRACTON  
COUPLED WITH ON LINE HYDRIDE GENERATION ETAAS USING AS SOLID PHASE  
EXTRACTANT A NEW FUNCTIONALIZED MESOPOROUS SILICA**

**López Guerrero, M. M., Siles Cordero, M.T., Vereda Alonso, E.I., García de Torres, A.,  
Cano Pavón, J.M.**

Dpto. Química Analítica, Universidad de Málaga, Campus de Teatinos s/n, 29071 Málaga,  
jm\_cano@uma.es

A chelating resin, [1,5 bis(di-2-pyridyl) methylene thiocarbonylhydrazide] bonded to mesoporous silica (DPTH-ms), has been used as a novel solid phase extractant. This resin has some advantages compared to most of other chelating adsorbents. Therefore, the aim is to develop a reliable method for determination of the aforementioned element from natural water samples by the on-line column preconcentration/HG-ETAAS using the resin DPTH-ms. With all experimental variables optimized, a linear calibration graph was obtained from  $0.01 \mu\text{g L}^{-1}$  to  $1.5 \mu\text{g L}^{-1}$  of Sb(III) with a regression coefficient of 0.9916, Table 1.

Table 1. Analytical performance.

Calibration equation <sup>a</sup> (n=6)	Blank signal/mV	Detection limit / $\mu\text{g L}^{-1}$	Determination limit / $\mu\text{g L}^{-1}$	Enrichment factor
$Y=0.0278x+0.0101$	$0.009\pm 0.0001$	0.002	0.035	3.39

<sup>a</sup> y, signal/mV; x, concentration/  $\mu\text{g L}^{-1}$

In order to test the accuracy and applicability of the proposed method for the analysis of real natural water samples, several certified reference materials of environmental waters were analyzed. These samples were employed for the validation of the method. These results show good agreement with the certified values, according to the t-test for a confidence level of 95% and they show sufficiently high recoveries. Because these standard reference samples have included trace elements such as transition metals, it can be said that there is no interference from these metals at  $\text{ng ml}^{-1}$  concentrations.

Table 2. Analytical applications. Mercury determination in several water samples.

Water type	Certified / $\mu\text{g L}^{-1}$	Added/ $\mu\text{g L}^{-1}$	Found/ $\mu\text{g L}^{-1}$	Recovery (%)
TMDA 54.4 <sup>a</sup> (Fortified Lake Water)		0	$12.35\pm 0.24$	
LGC6187 (River Sediment)	14.0	0	14.4	103.5
Seawater		0	$0.08\pm 0.03$	--
		0.2	$0.21\pm 0.03$	103.0
		0.4	$0.380\pm 0.005$	96.4
		0.6	$0.610\pm 0.005$	101.0

<sup>a</sup> Information value not given in the certified table of the certified reference material

The accuracy achieved for the spiked samples, demonstrates that the method is not affected by high salinity (approximately  $35\text{g l}^{-1}$ ). In conclusion, the method with the functionalized mesoporous silica has demonstrated to be rapid, easy, automatic, selective and with good sensitivity. The detection limits obtained are adequate for the analyzed natural water like seawater and river water samples, being better than others found in the bibliography for Hg.