

## IX Edición Premios José Antonio García Domínguez

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En el marco de la XIII Reunión Científica de la Sociedad Española de Cromatografía y Técnicas Afines (SECyTA) celebrada en Tenerife del 8 al 11 de octubre de 2013 se otorgaron los premios José Antonio García Domínguez a las mejores comunicaciones orales y tipo cartel presentadas en dicha reunión. Al igual que en años anteriores, esta VIII edición de los premios ha sido patrocinada por Bruker. El jurado encargado de fallar los premios correspondientes a las mejores comunicaciones orales estaba formado por M<sup>a</sup> José González (presidenta), Elena Ibáñez, Rosa M<sup>a</sup> Marcé, Yolanda Picó, Jesús Sanz y Javier Santos, que tras debatir los méritos científicos de las presentaciones, tomó por unanimidad los siguientes acuerdos:

### 1<sup>er</sup> Premio a la mejor Comunicación Oral (800 euros)

Comunicación: YS2-04

Título: ADVANCES IN COUPLING CSEI-SWEEPING-MECK WITH DIFFERENT SAMPLE TREATMENTS FOR 5-NITROIMIDAZOLE DETERMINATION IN FOOD, ENVIRONMENTAL AND CLINICAL MATRICES

Autores: Maykel Hernández-Mesa, Diego Airado-Rodríguez, Carmen Cruces-Blanco, Ana M. García Campaña  
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### 2<sup>o</sup> Premio a la mejor Comunicación Oral (600 euros)

Comunicación: YS2-02

Título: SELECTIVE SEPARATION OF BIOACTIVE CARBOHYDRATES USING IONIC LIQUIDS

Autores: Laura Ruiz-Aceituno, Cipriano Carrero-Carralero, Lourdes Ramos, Jesús Sanz, María Luz Sanz  
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En el caso de los premios a las mejores comunicaciones tipo cartel presentadas en la XIII Reunión Científica de la SECyTA, el jurado, constituido por las personas mencionadas anteriormente, tomó por unanimidad los siguientes acuerdos:

### **1<sup>er</sup> Premio al mejor Póster (400 euros)**

Comunicación: PS1-41

Título: MODELLING OF RETENTION AND PEAK SHAPE IN COMPREHENSIVE TWO-DIMENSIONAL LIQUID CHROMATOGRAPHY

Autores: Casandra Ortiz-Bolsico<sup>1</sup>, José Ramón Torres-Lapasió<sup>1</sup>, Sandra Pous-Torres<sup>1,2</sup>, María Celia García-Álvarez-Coque<sup>1</sup>, Gabriel Vivó-Truyols<sup>2</sup>, Peter Schoenmakers<sup>2</sup>

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### **2<sup>o</sup> Premio al mejor Póster (300 euros)**

Comunicación: PS2-15

Título: SORBENT-PACKED NEEDLE MICROEXTRACTION TRAP FOR MUSK FRAGRANCES DETERMINATION IN ENVIRONMENTAL WATER SAMPLES

Autores: Laura Vallecillos<sup>1</sup>, Eva Pocurull<sup>1</sup>, Juan M. Sánchez<sup>2</sup>, Francesc Borrull<sup>1</sup>

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La entrega de los premios tuvo lugar el 11 de octubre de 2013, durante la ceremonia de clausura de la XIII Reunión Científica de la SECyTA.

Belén Gómara  
Secretaria de la SECyTA

## **1<sup>er</sup> Premio a la mejor Comunicación Oral (800 euros): comunicación YS2-04**

### **ADVANCES IN COUPLING CSEI-SWEEPING-MEKC WITH DIFFERENT SAMPLE TREATMENTS FOR 5-NITROIMIDAZOLE DETERMINATION IN FOOD, ENVIRONMENTAL AND CLINICAL MATRICES**

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Cation-selective exhaustive injection (CSEI)-sweeping is a novel methodology initially developed by Quirino and Terabe [1]. It combines two on-line concentration techniques: field-enhanced sample injection (FESI) with sweeping and it is possible to achieve concentration factors from a thousand- to almost a million-fold in relation to the normal injection in capillary electrophoresis (CE) [1]. It overcomes the lack of sensitivity that has been traditionally associated to CE-UV methods, being a very useful alternative in trace analysis. In spite of this, few methods have been reported using this new sensitive methodology, which involves the need of more contributions to check its advantages and disadvantages.

In this work, we have developed a new method based on CSEI-sweeping for the analysis of 5-nitroimidazoles (5-NDZs) in different matrices. Prior to injection, the capillary must be rinsed with a low conductivity buffer (50 mM phosphate buffer pH 2.5), followed by a plug of a higher conductivity buffer (100 mM phosphate pH 2.5, 50 mbar,  $\approx$  31.5 % total capillary volume) and a plug of water (50 mbar, 2 s). Analytes, dissolved in a solvent of lower conductivity than that of the separation medium, are electrokinetically injected at 9.8 KV for 632 s in a bare fused-silica capillary (57.2 cm, 50  $\mu$ m I.D.), and thus the desired FESI effect is obtained. 5 mM phosphoric acid with 5% of methanol was selected as injection solvent to perform FESI. Separation was carried out applying -30 KV at 20°C in 44 mM phosphate buffer pH 2.5, containing 8 % tetrahydrofurane and 123 mM SDS.

Moreover, the proposed method has been applied in different areas as food, environmental and clinical analyses. Besides, different sample pretreatments have been evaluated to be coupled with CSEI-sweeping: solid phase extraction (SPE) has been proposed for egg sample cleaning-up; dispersive liquid-liquid microextraction (DLLME) has been used prior to the analysis of water samples; and untreated human urine and serum samples has been also evaluated. Detection limits at low ng/mL have been obtained for egg and water samples, achieving the recommendations from European Community Reference Laboratories [2]. Detection limits at low  $\mu$ g/mL have been reached for urine and serum samples, which enables the detection of these drugs at their normal levels in biological fluids [3].

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## **2º Premio a la mejor Comunicación Oral (600 euros): comunicación YS2-02**

### **SELECTIVE SEPARATION OF BIOACTIVE CARBOHYDRATES USING IONIC LIQUIDS**

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Ionic liquids (ILs) are low melting point salts with unique properties such as low vapor pressure and high thermal and chemical stability [1]. These properties make them to be considered a promising recyclable alternative to traditional volatile organic solvents for a high number of applications such as those related to catalysis [1], solvents for extractions of a variety of substances [2], as stationary phases for gas chromatography (GC) [3], etc.

Interest in biological activities of carbohydrates is currently increasing in different research areas such as food, pharmaceuticals, and environmental science. Prebiotic carbohydrates can modulate the activities of the gut microbiota, whereas inositols exhibit different activities mainly connected to insulin-related diseases [4]. Most of these carbohydrates are extracted from natural products, and the presence of other co-extracted sugars which could interfere in their bioactivity makes the fractionation of the extracts mandatory. This is a challenging process considering their similar structures and concentrations. Techniques such as Ion-Exchange Chromatography [5], Activated Charcoal [6] or Pressurized Liquid Extraction [5] have been proposed for the fractionation of carbohydrates, but ILs have not been applied before to this purpose and could represent a good alternative to conventional solvents.

Before evaluating the selective fractionation of carbohydrates in ILs, new solubility data of a wide range of carbohydrates (aldoses, ketoses, inositols and linear polyalcohols) in different ILs ([EMIM][DCA], [EMIM][Ac], [MMIM][Me<sub>2</sub>PO<sub>4</sub>] and [HMIM][Cl]) at different temperatures was required. Optimization and validation of a derivatization method for their GC analysis was also a prerequisite for the quantitative studies.

Mono- and disaccharides showed higher solubilities values (at 25 °C) than inositols in [EMIM] [DCA], whereas linear polyalcohols were more soluble than the other carbohydrates in [EMIM][Ac] and [MMIM][Me<sub>2</sub>PO<sub>4</sub>]. The differences in solubilities of aldoses and ketoses in [HMIM][Cl] and [EMIM][DCA] could be also useful for their potential separation. These results indicate the efficiency of ILs in the selective fractionation of bioactive carbohydrates.

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- [3] C.F. Poole, S.K. Poole, J. Sep. Sci. 34 (2011) 888-900.
- [4] B. Macias Camero, C. Sanjuan Merino, Patent US 6699511 B2 (2004).
- [5] S. Rodríguez-Sánchez, L. Ruiz-Aceituno, M.L. Sanz, A.C. Soria, J. Agric. Food Chem. 61 (2013) 4539-4545.
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## **1<sup>er</sup> Premio al mejor Póster (400 euros): comunicación PS1-41**

### **MODELLING OF RETENTION AND PEAK SHAPE IN COMPREHENSIVE TWO-DIMENSIONAL LIQUID CHROMATOGRAPHY**

**Casandra Ortiz-Bolsico<sup>(1),\*</sup>, José Ramón Torres-Lapasí<sup>(1)</sup>, Sandra Pous-Torres<sup>(1),(2)</sup>, María Celia García-Álvarez-Coque<sup>(1)</sup>, Gabriel Vivó-Truyols<sup>(2)</sup>, Peter Schoenmakers<sup>(2)</sup>**

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The interest in developing approaches for separating highly challenging samples, such as biopharmaceuticals, peptide maps, plant extracts, food matrices, polymers, etc. is growing. In this regard, comprehensive two-dimensional liquid chromatography (LC×LC) offers the possibility of substantially increasing the peak capacity in comparison with conventional LC, by combining orthogonal separations. Contemporary instrumentation provides many new opportunities for changing the mobile phase composition in the second dimension, applying gradients, varying the initial and final compositions of the second-dimension gradient, etc. A recent example of such instrumentation is the Agilent 1290 Infinity 2D-LC solution.

However, method development in LC×LC is a bottleneck. The ability to reliably predict chromatograms after a few initial runs may help overcome the most important obstacles to the proliferation of LC×LC. Generic retention models can be generated and validated efficiently using LC×LC experiments. The resulting models may be used to formulate guidelines and strategies for method development in LC×LC.

In this work, the separation of a set of polyphenols is studied with RPLC and HILIC, using acetonitrile-water mixtures in the gradient-elution mode. These systems show “orthogonal” (complementary) behavior, while using similar eluents. Gradient times and peak profiles were modeled for both dimensions versus the gradient parameters (initial and final concentration of organic solvent and gradient slope) for HILIC×RPLC separations. The models were used to predict the retention time of all solutes at any condition, and optimize the gradients. The parameters that affect the gradient modeling (e.g. dwell time, dead volume, sample injection volume and equilibration time) were considered.

#### *Acknowledgements*

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## **2º Premio al mejor Póster (300 euros): comunicación PS2-15**

### **SORBENT-PACKED NEEDLE MICROEXTRACTION TRAP FOR MUSK FRAGRANCES DETERMINATION IN ENVIRONMENTAL WATER SAMPLES**

**Laura Vallecillos<sup>(1),\*</sup>, Eva Pocurull<sup>(1)</sup>, Juan M. Sanchez<sup>(2)</sup>, Francesc Borrull<sup>(1)</sup>**

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A sorbent-packed microextraction trap method was developed to determine a mixture of musk fragrances (polycyclic musk, nitro musk and macrocyclic musk) extensively used in personal care products from wastewater sample prior to analysis by gas chromatography ion trap mass spectrometry (GC-IT-MS).

According with previous bibliography [1, 2] the needle trap (NT) device was performed by using 22g needles with side holes and was filled with a C18 (120 µm) sorbent [3, 4]. Different parameters affecting the adsorption capacity of the sorbent, such as sampling time and temperature, headspace/sample volume, salting-out and stirring rate were optimized in both 10 µL and 100 µL syringes. Furthermore desorption and transferring of the target compounds into the GC column were studied.

Validation parameters like method detection limits, method quantification limits, linear range, intra-day repeatability and inter-day repeatability were evaluated under optimal conditions. Then, the applicability of the method was tested with wastewater samples from three urban wastewater treatment plants (WWTPs) located in the area of Tarragona with population around 140,000 inhabitants. The WWTPs receive urban sewages and some industrial discharges and use a secondary treatment based on activated sludge biological treatment. Moreover, at one of the WWTPs, and additional sample was taken from the effluent of the tertiary treatment based on reverse osmosis.

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