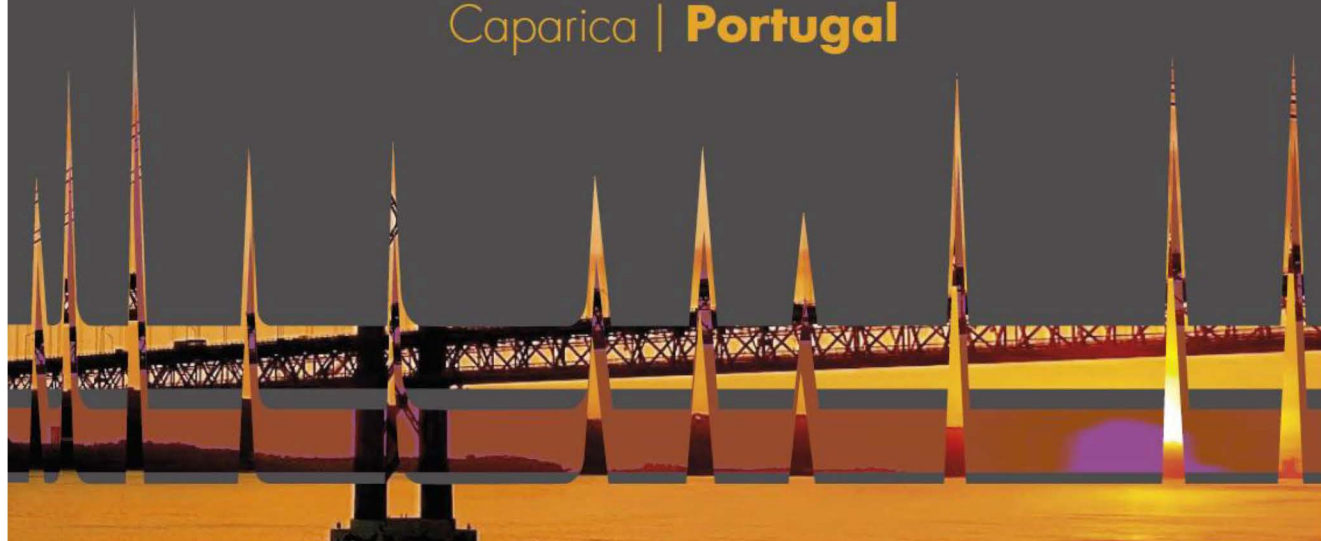


11^o CONGRESSO
NACIONAL
DE CROMATOGRÁFIA

20 anos
CROMATOGRÁFIA

11th NATIONAL MEETING ON CHROMATOGRAPHY

9 | 11 Dezembro 2019
Caparica | Portugal



Faculdade de Ciências e Tecnologia,
Universidade NOVA de Lisboa



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15:40 O10 *The Use of Ion Mobility-MS to Resolve and Discover Sample Complexity In Small Molecule Analysis*

Alberto Méndez¹

¹ Waters Corporation

16:05 O11 *Analysis of skin volatiles using a membrane-SPME/GC-MS approach to unveil putative biomarkers for neurodegenerative diseases*

Beatriz Andrade¹, Jorge Pereira¹, José Câmara^{1,2}

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16.25 Coffee Break & Posters Session

AFTERNOON SESSION

Session 4 Chair: Cristina Dias - Universidade de Évora

17:00 O12 *Determination of the phenolic composition of vine-canessubcritical water extracts and its utilization for production of a topical formulation*

Manuela M. Moreira¹, Francisca Rodrigues¹, Olena Dorosh^{1,2}, Diana Pinto,¹ Andreia F. Peixoto,³ Paulo Costa⁴, Simone Morais¹, Cristina Freire³, Cristina Delerue-Matos¹

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17:20 O13 *HPLC and UHPLC Selectivity – Finding a Selectivity Starting Point*

Zeshan Aqeel¹, Felipe Silva², PhD. Jason Anspach¹, and Ryan Splitstone¹

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² Phenomenex C/ Valgrande 8, planta 2.1.B., 28108 Alcobendas, Madrid, Spain

17:45 O14 *Separation of Nadolol Racemates by High pH Reversed-Phase Fixed-Bed and Simulated Moving Bed Chromatography*

R. Arafah^{1,2}, A. Ribeiro^{1,2}, A. Rodrigues², L. Pais^{1,2}

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18:05 O15 *Pharmaceutical drugs as emerging pollutants in aqueous media of Northeast Portugal*

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O14 Separation of Nadolol Racemates by High pH Reversed-Phase Fixed-Bed and Simulated Moving Bed Chromatography

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Nadolol is a pharmaceutical chiral drug worldwide prescribed to the relief of some diseases mainly related with the cardiovascular system. Although some studies refer that the therapeutic effect of this drug is related with only one enantiomer, nadolol is still being marketed as a mixture of four stereoisomers, in a form of a racemic mixture of two racemates. The separation of all the four stereoisomers, despite being a very challenging task, will be very helpful to provide the pharmaceutical industry of any amounts of pure compounds to perform individual pharmaceutical and pharmacologic studies.

Recently, our research group reported the pseudo-binary separation of RSR-nadolol stereoisomer by simulated moving bed (SMB) technology using both coated Chiralpak AD and immobilized Chiralpak IA chiral stationary phases, with an eluent normal-phase mode. In this work, we present an alternative strategy, implementing a first achiral separation step, by using C18 columns to perform the separation of the two nadolol racemates under reversed-phase mode. This introduces much more deep and new challenges involving selection of the packing to be used, optimization of the solvent composition, and the strategy design for defining the different separation steps and its sequences. Different separation strategies can be designed and optimized, enlarging the packing materials possibilities, from fully chiral (Chiralpak) to achiral (C18) – chiral (Chiralpak) separation combinations and, so, the use of both normal and reversed-phase chromatography. For each step, the optimization of the solvent composition will be carried out, using pure alcohol, alcohol-hydrocarbon and alcohol-water mixtures, all with a basic modifier, considering the strong basic nature of the nadolol stereoisomers. The separation technology to be used will also be tested, including fixed-bed and SMB liquid chromatography. The different alternatives will be evaluated in terms of the real capacity to achieve complete separation of all the four nadolol stereoisomers and in terms of system productivity and solvent consumption.

Considering the previous tasks, both modelling-simulation and experimental tools will be fully used, namely in what concerns the knowledge of the equilibrium adsorption isotherms, kinetic data (axial dispersion and resistance to mass transfer), and the prediction of preparative fixed-bed and SMB performances. This chemical engineering approach will allow the deep knowledge of all the separation processes and its optimization at preparative scale. Extensive experimental and simulation results will be presented, including solvent screening, measurement of equilibrium adsorption isotherms, breakthrough measurements, preparative HPLC (Azura pilot unit) and SMB (FlexSMB-LSRE unit) experimental separations of nadolol racemates using C18 columns. At the end is expected the clear definition of the best separation strategy for the complete separation of nadolol stereoisomers and the experimental availability of all the four pure stereoisomers [1-4].

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