10º Encontro Nacional de Cromatografia

Bragança 2017 – 4 a 6 de dezembro

Abstracts book / Livro de resumos

Sociedade Portuguesa de Química

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Title

10th Chromatography Meeting

Título

10º Encontro de Cromatografia

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Edition / Edição

Instituto Politécnico de Bragança · 2017
5300-253 Bragança · Portugal
Tel. (+351) 273 303 200 · Fax (+351) 273 325 405
http://www.ipb.pt

Imaging services / Serviços de imagem

Atilano Suarez (Instituto Politécnico de Bragança, Portugal)

URL

http://hdl.handle.net/10198/8896

ISBN

978-972-745-234-7

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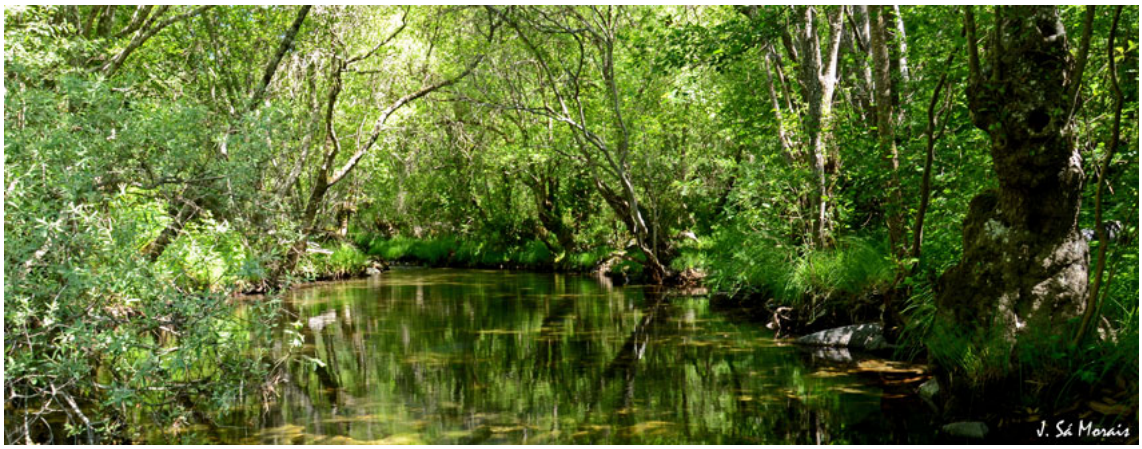

Program / Programa

Time		December 4
8:00-9:00		• Registration
9:00-10:00		• Opening session in *Auditorium Dionísio Gonçalves*
Moderator / Moderador - Auditorium Dionísio Gonçalves ***Isabel C.F.R. Ferreira*** *(Instituto Politécnico de Bragança)*		
10:00-11:00	PL-01	In-tube SPME from open tubular column (in-tube SPME-LC) to directly coupled to mass spectrometry ***Maria Eugênia Costa Queiroz*** *Universidade de São Paulo, Brasil*
11:00-11:30		• Coffee Break and panel session
Moderator / Moderador - Auditorium Dionísio Gonçalves ***Sílvia M. Rocha*** *(Universidade de Aveiro)*		
11:30-12:00	IC-01	Different Strategies Based on Micro(extraction) Followed by GC-MS/MS and LC-MS/MS for the Determination of Personal Care Products in Cosmetics and Environmental Samples ***Maria Llompart*** *University of Santiago de Compostela, Espanha*
12:00-12:30	EC-01	LCMS Technologies: Introducing the Orbitrap for Ultrahigh Resolution Exact Mass and Unequivocal ID ***Daniel Ettlin*** *Thermo Unicam Sistemas Analíticos*
12:30-14:30		• Lunch
Moderator / Moderador - Auditorium Dionísio Gonçalves ***Nuno Mateus*** *(Universidade do Porto)*		
14:30-15:00	IC-02	Back to Basics: Considerations in eco-user-friendly/cost-effective micro-extraction techniques ***José Nogueira*** *Universidade de Lisboa, Portugal*
15:00-16:30		***Oral session 1A / Sessão Oral 1A***
	OC-01	A multiresidue targeting approach for pesticide detection in olive oil: the role of dual-layer solid-phase extraction based on molecular imprinting technology ***Raquel Garcia***
	OC-02	New brush-type chiral stationary phases based on xanthone derivatives for liquid chromatography ***Carla Fernandes***
	OC-03	Chromatographic techniques to assess the profile of biomolecules in different mycorrhizal mushroom species ***Filipa Reis***
	OC-04	Multicolumn based liquid chromatography processes for the separation of nadolol racemates ***António Ribeiro***
	OC-05	An expanded bed chromatography approach for improving human mesenchymal stem cells purification ***Ricardo Silva***

OC-04

Multicolumn based liquid chromatography processes for the separation of nadolol racemates

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A few decades passed since the pharmaceutical industry realized the need to produce chiral drugs with reduced severe side-effects. To overcome this issue, the international agencies for drug safety become a real driving force, pushing more and more the industry towards the commercialization of drugs based on pure enantiomers. Traditionally, enantiomerically pure drugs are still produced in large-scale by organic asymmetric synthesis. However, it is also accepted that, in several cases, it is a too much time consuming production path. The direct resolution of racemic compounds, using multicolumn or fixed-bed liquid chromatography technologies, are nowadays, recognized at an industrial scale has a true alternative. The use of such technologies, like simulated moving bed (SMB) chromatography allows both high yields and purities of both enantiomers present in the racemic chiral compound. Also, these techniques can be applied to a wide range of racemic mixtures, since different stationary phases for enantiomer separation are now available. Nadolol is a pharmaceutical drug marketed as a mixture of four stereoisomers, used to treat cardiovascular diseases. This drug is a mixture of two pairs of racemates, therefore, its complete separation represents a challenging task. Recently, our research group reported the pseudo-binary separation of nadolol by SMB chromatography using both coated Chiralpak AD and Chiralpak IA immobilized chiral stationary phases [1,2]. In this work, it is proposed an alternative strategy, implementing a first achiral separation step, to be followed by two subsequent parallel chiral separation steps [3]. In this first achiral step, C18 columns are used to perform the separation of the two pairs of nadolol enantiomers ("racemate A" from "racemate B") under reversed-phase mode. After this preliminary achiral separation step, two parallel SMB runs must be carried out using a chiral stationary phase to achieve the complete separation of all the four nadolol stereoisomers. Extensive experimental and simulation results will be presented including solvent screening, measurement of equilibrium and kinetic data, and both fixed-bed and SMB preparative separations.

Acknowledgements:
This work is a result of project "AIProcMat@N2020 - Advanced Industrial Processes and Materials for a Sustainable Northern Region of Portugal 2020", with the reference NORTE-01-0145-FEDER-000006, supported by Norte Portugal Regional Operational Programme (NORTE 2020), under the Portugal 2020 Partnership Agreement, through the European Regional Development Fund (ERDF) and of Project POCI-01-0145-FEDER-006984 – Associate Laboratory LSRE-LCM funded by ERDF through COMPETE2020 - Programa Operacional Competitividade e Internacionalização (POCI) – and by national funds through FCT - Fundação para a Ciência e a Tecnologia.

References:
[1] A. E. Ribeiro, A. E. Rodrigues, L. S. Pais. Chirality 2013, 25, 197.
[2] R. S. Arafah, A. E. Ribeiro, A. E. Rodrigues, L. S. Pais. Chirality 2016 28, 399.
[3] A. E. Ribeiro, R. S. Arafah, A. E. Rodrigues, L. S. Pais. Proc of XXV Encontro Nacional da Sociedade Portuguesa de Quimica, Lisboa, Portugal, 2017, Flash Communication 11.