POSTER SESSION I

WEDNESDAY, MAY 30 th, 2012

CHAIRPERSONS: Ł. Komsta and J. Jampilek

1.

DETERMINATION OF THE CRITICAL FACTORS IN CHIRAL SUPERCRITICAL FLUID CHROMATOGRAPHY BY USE OF A PLACKETT-BURMAN DESIGN.

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As the number of chiral drugs launched onto the market is yearly increasing, the need for fast and performant enantioseparation methods with minimum costs and preferably with low environmental impact is becoming more compelling. In this context, supercritical fluid chromatography (SFC) has great potential, since high flow rates are possible, while it permits to increase the throughput capacity, without compromising the efficiency. “Green chemistry” properties are assigned to this technique, which is applicable, both at an analytical and a preparative scale. These properties in addition to the recent instrumental improvements renewed the interest in SFC.

To enable more efficient chiral method development, screening strategies are defined. These strategies start with a screening step in which the enantioselectivity of several complementary systems is evaluated. In this way a broad enantioselective range is covered. Results from previous work enabled to define a chiral screening step for SFC.

After execution of this screening step and selection of the most appropriate system, an optimization is required to achieve the desired separation. In order to define efficient optimization steps, critical influencing factors have to be identified.

In this work, a Plackett-Burman based, three-level screening design is used to determine the critical factors in chiral SFC separations. These results can then be used to complete a generic separation strategy.

2.

FUSED-CORE STATIONARY PHASES FOR FINGERPRINT DEVELOPMENT OF PHYLLANTHUS AND MALLOTUS SPECIES

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Chromatographic fingerprint development is an accepted methodology for the identification and quality control of herbal medicinal products, which are popular to prevent or to treat diseases in many countries. The composition of herbs and their extracts is generally very complex and depends on several factors. Identification and quality control is therefore needed to avoid adverse effects as a result of adulteration or (accidental) exchange of plants. The World Health Organization (WHO) accepts chromatographic fingerprint analysis as a tool for the identification and quality control of herbal medicines because it reflects the composition of the total herbal sample or -extract. Fingerprints can also be used to model and predict given activities, e.g. antioxidant or cytotoxic activities, as a function of the fingerprints. These models can also be used to indicate in the fingerprints the peaks with potentially interesting activities. However, fingerprint development and analysis are time consuming (often 60 min runs) and the search for new approaches to speed up these processes is still ongoing. Fused-core stationary phases may present an alternative to reduce the analysis time of fingerprints without losing information. These recently introduced stationary phases consist of fused-core particles that are composed of a solid core surrounded by a porous layer. In combination with Ultra Fast Liquid Chromatography (UFLC), high resolutions, short analysis times and narrow peaks can be obtained compared to conventional stationary phases.
Fused-core in UFLC will be used to analyze different Mallotus and Phyllanthus species in order to model the antioxidant activity and to indicate interesting compounds. In a first part of this study, fingerprints will be developed for 51 Vietnamese samples. A relatively simple gradient program with different analysis times (60 min, 35 min, 22,5 min and shorter) will be tested, occasionally followed by a further optimization. Then the antioxidant activity of the samples will be determined with the Trolox Equivalent Antioxidant Capacity (TEAC) assay.
Data-analysis on the developed fingerprints will be performed in a second part of this study. In a first step, principal component analysis (PCA) will be performed to explore and reveal the data structure and to visualize occasional outliers. The antioxidant activity will be modeled using Partial Least Squares (PLS) and Orthogonal Projections to Latent Structures (O-PLS) regression methods. The obtained models for each dataset allow predicting the antioxidant activity for unknown samples and indicating the peaks in the chromatogram which potentially are responsible for the activity. All models will be compared to conclude whether the shortest fingerprints (22,5 min or shorter) still contain the same important information as the longer fingerprints (35 min and 60 min).

3.

Pharmacophore-Based Database Mining for Probing Fragmental Drug-Likeness of Diketo Acid Analogues

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The crucial objective of the computer-based techniques for the rational identification of the prospective drug candidates is the efficient and comprehensive mapping of the compound topology and/or geometry into the chemical property space. Despite the attention directed towards the development of the computational methods there is still a deficiency of the robust procedures for mining chemical space and designing molecular properties, especially in a fully user-defined mode. Furthermore, the understanding of the mutual relationships between a chemical structure, typically defined by an ensemble of the calculated descriptors, and the corresponding pharmacological activity (SAR) is still a fundamental issue in medicinal chemistry and molecular design.

While the amount of the accessible structural data has been increasing steadily it still exemplifies only a tiny fraction of all molecules in the 'infinite' chemical space (CS), representing approximately $10^{100} \div 10^{200}$ compounds. This makes a need for a proper design, filtration and/or enumeration of virtual structures, a problem often referred as 'molecular diversity'. The *in silico* transformation of the large number of compounds into corporate collections storing multidimensional data seemed to be an obvious solution. Hence, we developed the MoStBioDat environment for managing and analysing molecular and structural database information.

In our work we report the practical application of the system for the mapping of the fragmental drug-likeness topology (FDT) and the intramolecular hydrogen bonded (IHB) motifs in the diketo acid (DKA) related compounds. The DKA arrangement is a commonly observed structural feature found to be of crucial importance for HIV-1 integrase (IN) inhibitors.

A number of the structurally diverse chemical compounds with the functional DKA subunit(s) have been revealed by the combined *on line* and MoStBiodat 3D pharmacophore-guided ZINC and PubChem database screening. We used the structural data available from such screening to analyze the similarities of the compounds containing the DKA fragment. Generally, the analysis by PCA and SOM reveals four families of the compounds complying with the chemical constitution (aromatic, aliphatic) of the compounds. From practical point of view similar studies can reveal potential bioisosters of the known drugs, e.g., raltegravir/elvitegravir. In this context, it seems that *mono* halogenated aryl substructures with *para* group show the closest similarity to these compounds in contrast to the structures where aromatic ring is halogenated in both *orto*- and *para*-locations.

4.

Thin layer chromatography data in QSAR study of compounds with affinity for serotonin receptors

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A quantitative structure-activity relationship (QSAR) study has been made on 20 compounds with serotonin (5-HT) receptor affinity. A set of physicochemical parameters calculated by HyperChem 7.0 and ACDLabs 8.0 programs and chromatographic data were applied in the analysis. RP2 TLC 60F254 plates (silanized) impregnated with solutions of propionic acid, ethylbenzene, 4-ethylphenol, and propionamide (used as analogues of the key receptor amino acids) and their mixtures (denoted as S1–S7 biochromatographic models) were used in two developing phases as models of drug-5-HT receptor interaction. Correlation and multiple linear regression analysis were used to search for the best QSAR equations. The correlations obtained for the compounds studied represent their interactions with the proposed biochromatographic models. The good multivariate relationships (R^2=0.78–0.84) and leave-one/many-out cross validation procedures applied on final regression equations, demonstrated that these models have significant predictive ability (Q^2=0.57-0.70) and can be used for predicting the quantitative effect of biological activity of different compounds with 5-HT receptor affinity.

5.

HPLC analysis of aripiprazole and its impurities

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Abstract

Aripiprazole is a novel atypical antipsychotic drug used in the treatment of schizophrenia. This paper describes characterization of chromatographic behavior of aripiprazole and its nine related substances, which significantly differ in polarity. For this purpose, sensitive and reproducible method was developed and validated. The separation was performed on Phenomenex Luna® C18 column (5.0 µm particle size, 250 × 4.6 mm id) using a gradient with mobile phase A [phosphate buffer pH 3.0] and mobile phase B [acetonitrile] at the working temperature of 25°C. The buffer was 1.11g KH_2PO_4 with 1.2g sodium pentanesulfonate /L of the solution, adjusted to pH 3.0 with orthophosphoric acid. The flow rate was 1.0 mL/min. The detection was carried out at 215 nm using a diode array detector.

The proposed method is convenient and reliable for the purity control in both, raw materials and dosage forms.

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6.

TERPENES AS BIOLOGICALLY ACTIVE CONSTITUENTS OF MEDICINAL PLANTS

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The spread of bacterial resistance has become a major public health problem which is treated by many international organizations as a priority. Another expanding problem that affects society are free radicals which give negative influence on the processes occurring in the human body. Therefore, the increasing concern is given to an alternative medicine based on natural compounds with antimicrobial and antioxidant properties. New biologically active substances of plant origin should be less toxic and more effective than conventional ones. Plants such as: *Salvia officinalis, Thymus vulgaris* and *Menthae piperitae* are widely considered to be therapeutic. The most commonly isolated fraction from the plant are volatile terpenes [1].

Biological properties of terpenes contained in essential oils and tinctures of the above mentioned plants have been examined. In particular, the aim of our research was to establish the composition of the analyzed extracts by TLC and HPLC as well as to determine biological activity of their main components by TLC-DB (thin layer chromatography-direct bioautography) and DPPH methods [2]. In TLC-DB bacteria grow directly on a chromatogram containing separated substances. After staining with MTT dye solution, antibacterial activity is observed as white inhibition zones on purple background. The antimicrobial activity of the plant extracts against two strains of bacteria: *Escherichia coli* and *Bacillus subtilis* was investigated. The antioxidant activity of the essential oils and plant tinctures was assessed by their ability to scavenging 2,2-diphenyl-1-picrylhydrazyl stable radicals (DPPH). The experiments pointed to both antibacterial and antioxidant activity of many components of tested extracts.

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7.

A GC/MS and TLC study of the volatile fraction contained in creeping thyme (*Thymus serpyllum* L.) and common thyme (*Thymus vulgaris* L.)

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This study is a consecutive step in our research project devoted to fingerprinting of the volatile fraction contained in the selected sage (Salvia) species from the mint (Lamiaceae) family and presented in a series of papers (e.g., in [1-5]). In papers [1,2], we compared the performance of different techniques serving the purpose of deriving the volatile fraction prior to proper analysis by means of gas chromatography with mass spectrometric detection (GC/MS). The techniques compared were the "headspace" derivation and the two hydrodistillations carried out in the Deryng apparatus and the Clevenger apparatus. Moreover, we have performed "fingerprinting" of the volatile fraction contained in the different sage species by means of the low-temperature thin-layer chromatography (TLC) with densitometric and mass spectrometric detection, and the combined 2D technique, TLC – LC – MS [3-5].

In this study, an analogous comparison was performed for the volatile fractions derived from two other oily herbs belonging to the mint family, i.e., from creeping thyme (*Thymus serpyllum* L.) and common thyme (*Thymus vulgaris* L.). Herbal specimens originated from several different sources, i.e., from the Garden of Medicinal Plants, Faculty of Pharmacy, Medical University of Lublin and from the local market of culinary herbs (several different manufacturers). The following comparisons were performed: (i) of the volatile fraction yields depending on the different derivation techniques, prior to the "fingerprinting" by means of GC/MS, and (ii) of the "fingerprints" of the volatile fractions derived by means of different methods. Moreover, partial identification was performed of the volatile fraction components with aid of the virtual library of the mass spectra. Finally, "fingerprinting" of the volatile fraction was performed by means of the low-temperature thin-layer chromatography implemented with different detection modes. The obtained results were presented in the form of figures and tables, and upon this outcome, relevant conclusions were drawn.

References:

1. J. Rzepa, Ł. Wojtal, D. Staszek, G. Grygierczyk, K. Labe, M. Hajnos, T. Kowalska, M. Waksmundzka-Hajnos, J. Chromatogr. Sci. 47 (2009) 575.
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8.

A GC/MS and TLC study of the volatile fraction contained in rosemary (*Rosmarinus officinalis* L.), narrow-leaved lavender (*Lavandula angustifolia*), anise (*Pimpinella anisum* L.), and the fruit of clove tree (*Eugenia caryophyllata* Thunb.)

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This study is a consecutive step in our research project devoted to fingerprinting of the volatile fraction contained in the selected sage (Salvia) species from the mint (Lamiaceae) family and presented in a series of papers (e.g., in [1-5]). In papers [1,2], we compared the performance of different techniques serving the purpose of deriving the volatile fraction prior to proper analysis by means of gas chromatography with mass spectrometric detection (GC/MS). The techniques compared were the "headspace" derivation and the two hydrodistillations carried out in the Deryng apparatus and the Clevenger apparatus. Moreover, we have performed "fingerprinting" of the volatile fraction contained in the different sage species by means of the low-temperature thin-layer chromatography (TLC) with densitometric and mass spectrometric detection, and the combined 2D technique, TLC – LC – MS [3-5].

In this study, an analogous comparison was performed for the volatile fractions derived from two other oily herbs belonging to the mint family, i.e., for rosemary (*Rosmarinus officinalis* L.) and narrow-leaved lavender (*Lavandula angustifolia*), and besides, for anise (*Pimpinella anisum* L.) belonging to the Apiaceae family and for the fruit of clove tree (*Eugenia caryophyllata* Thunb.). Herbal specimens originated from several different sources, i.e., from the Garden of Medicinal Plants, Faculty of Pharmacy, Medical University of Lublin and from the local market of culinary herbs (several different manufacturers). The following comparisons were performed: (i) of the volatile fraction yields depending on the different derivation techniques, prior to the "fingerprinting" by means of GC/MS, and (ii) of the "fingerprints" of the volatile fractions derived by means of different methods. Moreover, partial identification was performed of the volatile fraction components with aid of the virtual library of the mass spectra. Finally, "fingerprinting" of the volatile fraction was performed by means of the low-temperature thin-layer chromatography implemented with different detection modes. The obtained results were presented in the form of figures and tables, and upon this outcome, relevant conclusions were drawn.

References:

1. J. Rzepa, Ł. Wojtal, D. Staszek, G. Grygierczyk, K. Labe, M. Hajnos, T. Kowalska, M. Waksmundzka-Hajnos, J. Chromatogr. Sci. 47 (2009) 575.
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9.

A TLC, HPLC/DAD, and HPLC/ELSD study of the phenolics contained in two sage species (*Salvia triloba* and *Salvia staminea*), and in two thyme species (*Thymus serpyllum* L. and *Thymus vulgaris* L.)

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This study is a consecutive step in our research project devoted to fingerprinting of the selectively extracted fractions of phenolic acids and flavonoids contained in the selected sage (Salvia) species from the mint (Lamiaceae) family and presented in a series of papers (e.g., in [1-7]). Respective analyses were carried out by means of liquid chromatography (basically, TLC, HPLC/DAD, and HPLC/ELSD). Due to the fact that the phenolics contained in the discussed herbs possess the well recognized anti-atherosclerotic activity as the free-radical scavengers, composition of the phenolics fraction and also quantification thereof in herbal extracts is of considerable importance for pharmacognosy. Herbs with particularly high levels of the phenolics can obtain the status of medicinal plants and an official entry in pharmacopoeia. So far, the only sage species recognized by Polish and European Pharmacopoeia is common sage (*Salvia officinalis* L.).

In this study, we compare chromatographic fingerprints for the selectively extracted fractions of phenolic acids and flavonoids derived from the four herbs belonging to the mint (Lamiaceae) family, i.e., Greek sage (*Salvia triloba*), *Salvia staminea*, creeping thyme (*Thymus serpyllum* L.), and common thyme (*Thymus vulgaris* L.). All herbal specimens originated from the Garden of Medicinal Plants, Faculty of Pharmacy, Medical University of Lublin and they are all widely recognized as culinary and decorative plants, and moreover, as medicinal herbs in various different geographical and cultural regions. Fingerprinting was performed with use of TLC, HPLC/DAD, and HPLC/ELSD. In this study, a comparison was made of (i) fingerprints valid for the phenolic acid and flavonoid fractions derived from the sage and the thyme species, and (ii) the informative value of fingerprints originating from the planar and column chromatographic techniques. On the basis of the collected experimental evidence, the relevant conclusions were drawn.

References:

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10.

A TLC, HPLC/DAD, and HPLC/ELSD study of the phenolics contained in two dragon's head (*Dracocephalum moldavica* L.) varieties

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This study is a consecutive step in our research project devoted to fingerprinting of the selectively extracted fractions of phenolic acids and flavonoids contained in the selected sage (Salvia) species from the mint (Lamiaceae) family and presented in a series of papers (e.g., in [1-7]). Respective analyses were carried out by means of liquid chromatography (basically, TLC, HPLC/DAD, and HPLC/ELSD). Due to the fact that the phenolics contained in the discussed herbs possess the well recognized anti-atherosclerotic activity as the free-radical scavengers, composition of the phenolics fraction and also quantification thereof in herbal extracts is of considerable importance for pharmacognosy. Herbs with particularly high levels of the phenolics can obtain the status of medicinal plants and an official entry in pharmacopoeia. So far, the only sage species recognized by Polish and European Pharmacopoeia is common sage (*Salvia officinalis* L.).

In this study, we compare chromatographic fingerprints for the selectively extracted fractions of phenolic acids and flavonoids derived from the herb belonging to the mint (Lamiaceae) family, i.e., dragon's head (*Dracocephalum moldavica* L.), appearing in two varieties, with the white and blue flowers. The investigated herbal specimens originated from the Garden of Medicinal Plants, Faculty of Pharmacy, Medical University of Lublin. Dragon's head is mostly recognized as a medicinal and decorative plant, particularly rich in essential oil (composed mostly of geranyl acetate, geranial, and neral [8]). Fingerprinting was performed with use of TLC, HPLC/DAD, and HPLC/ELSD. In this study, a comparison was made of (i) fingerprints valid for the phenolic acid and flavonoid fractions derived from the two dragon head's varieties, and (ii) the informative value of fingerprints originating from the planar and column chromatographic techniques. On the basis of the collected experimental evidence, the relevant conclusions were drawn.

References:

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11.

Simultaneous Multiple Development HPTLC Quantification of Water- and Oil Soluble Sunscreens

Anna W. Sobańska*, Jarosław Pyzowski

Abstract

A complex mixture of sunscreens of different lipophilicities was quantified for the first time by Thin Layer Chromatography followed by densitometric scanning in absorption mode. Multiple Development Normal Phase TLC was performed on silica gel 60 as stationary phase. Two mobile phases were used: A - cyclohexane-diethyl ether 5:1 (v/v) and B - ethyl acetate-ethanol-water 70:35:30 (v/v/v). After development with mobile phase A two oil soluble sunscreens: avobenzone (AVO) and octyl salicylate (OS) were analyzed at 360 and 300 nm, respectively. Subsequent development of the same plates with mobile phase B made it possible to quantify a water soluble sunscreen - phenylbenzimidazol sulfonic acid (PBS) at 300 nm. Calibration curves were non-linear. Limits of detection and quantification were: LOD (OS) 0.02 μg spot^{-1}, LOQ (OS) 0.06 μg spot^{-1}, LOD (AVO) 0.03 μg spot^{-1}, LOQ (AVO) 0.08 μg spot^{-1}, LOD (PBS) 0.02 μg spot^{-1}, LOQ (PBS) 0.06 μg spot^{-1}. The method was validated and applied to the analysis of a commercially available cosmetic product.

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12.

Quantification of sunscreen 2-phenylbenzimidazole-5-sulfonic acid in bathing water samples by TLC/densitometry with fluorescent detection

Anna W. Sobańska*, Karolina Derecka, Jarosław Pyzowski

Abstract

The water-soluble sunscreen 2-phenylbenzimidazole-5-sulfonic acid (PBS) was quantified in bathing water samples by Thin Layer Chromatography followed by densitometric scanning in fluorescent mode (cut-off filter 370 nm, analytical wavelength – 300 nm). Normal Phase TLC was performed on silica gel 60 as stationary phase. Mobile phase used was ethyl acetate-ethanol-water 70:35:30 (v/v/v). Limit of detection (LOD) was 0.0004 μg $spot^{-1}$ and limit of quantification (LOQ) – 0.001 μg $spot^{-1}$ without any sample pre-concentration. The method was validated.

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13.

Quantification of sunscreen benzophenone-4 in shampoo samples by Normal-Phase Thin Layer Chromatography/densitometry

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Abstract

The water-soluble sunscreen benzophenone-4 (BZ-4) was quantified in shampoo samples by Thin Layer Chromatography followed by densitometric scanning in absorption mode (analytical wavelength – 285 nm). Normal Phase TLC was performed on silica gel 60 as stationary phase. Mobile phase used was ethyl acetate-ethanol-water-pH 6 phosphate buffer 72:35:30:5 (v/v/v/v). Limit of detection (LOD) was 0.03 μg $spot^{-1}$ and limit of quantification (LOQ) – 0.1 μg $spot^{-1}$. The calibration plot was non-linear. The method was applied to model shampoo samples prepared in the lab as well as to the samples of commercial products. The method was validated.

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Application of gas chromatography in comparative study of steam co-gasification of hard coal and various energy crops focused on hydrogen-rich gas production

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About 87% of the renewable energy balance and over 49% of renewable electricity production in Poland in 2008 was based on solid biomass. Poland has also one of the largest in Europe area of land suitable for poplar, willow and Miscanthus cultivation (excluding forest land and land highly suitable for cereals). An increasing trend in the renewable resources share in the final energy use (9.13% in 2010 and 15.48% in 2020) as well as support given to the development of distributed energy systems and highly efficient technologies, like gasification is expected. Co-gasification of coal and energy crops is claimed to offer several advantages when compared to coal or energy crops gasification. Co-gasification gives the benefits of reliable supplies of abundant solid fuel – coal and credits resulting from utilization of renewable, zero-emission energy resource – biomass. It makes feasible energy crops gasification in larger scale and thereby with higher efficiency and with lower specific operating costs than in conventional biomass gasification plants of usually <50 MW_e.

The results of experimental comparative study on steam co-gasification of hard coal and energy crops, such as *Salix Viminalis*, *Spartina pectinata*, *Helianthus tuberosus L.*, *Sida hermaphrodita R.* and *Miscanthus X Giganteus* in a fixed bed reactor under atmospheric pressure and at the temperatures of 700, 800 and 900^{o}C are presented in the paper. The ability of coal and energy crops to undergo thermochemical transformations was determined based on their chars' reactivities. Moreover the wider view on the energy crops and hard coal chars reactivities in the process of steam co-gasification and their physical and chemical properties with a use of chemometrics methods such as the hierarchical clustering analysis and the principal component analysis is presented. The chemometric methods allow to extract the information on similarities/dissimilarities between tested samples and to identify the optimal one in terms of the highest hydrogen yield in the process of steam co-gasification with coal. Moreover, the synergy effect in the process of co-gasification consisting in an increase in the volume of hydrogen produced, when compared to the tests of coal and energy crops gasification, was investigated.