POSTER SESSION I

WEDNESDAY, JUNE 5 th, 2013

CHAIRPERSONS:

Agnes Móricz and Maja Natić

1. Probing an artificial polypeptide receptor library using a series of novel histamine H3 receptor ligands

Andrzej Bak[1] , Michal Daszykowski[1], Violetta Kozik[1], Krystyna Jarzembek[1], Zbigniew Kaminski[2], Katarzyna Kiec-Kononowicz[3], Kamil Kuder[3], Justyna Fraczyk[2], Beata Kolesinska[2], Patrycja Ciosek[4] and Jaroslaw Polanski[1]

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Artificial receptors that are capable of selectively binding small chemical molecules under controlled conditions are not only interesting for modeling molecular recognition effects. This method can be also used for the development of flexible sensors and diagnostic platforms or as molecular probes enable to interfe with an actual biological event. Combinatorial libraries can provide us with an important tool not only for the design and discovery of novel chemical molecules but also for the exploration of interactions that are too complex for qualitative and quantitative rationalization. A systematic exploration of the provided data is of the great importance to design and synthesize a library of receptors which can emulate the interactions between drugs and living organisms is made possible. Therefore, the question of how to efficiently design a library to probe a certain receptor type and/or biological effect is as important as the problem of the analysis of the data that is acquired. Unfortunately, the nature of the interactions that govern the binding process of any ligand to its biological target, which is understood in terms of inter/intramolecular forces, is a complicated phenomenon.

Hence, the application of the potential that is represented by libraries of artificial targets binding a given guest molecule in physiological conditions as a model system for the emulation of ligand-receptor interactions has recently been reported. The ensemble of artificial 'biosensors' formed by the self-organization of *N*-lipidated peptides immobilized on cellulose arranged as a molecular probe matrix proved to be an efficient tool to recognize the shape, size and polarity of ligands.

The aim of this study was to probe a library of artificial receptors using a series of novel histamine H_3 receptor inhibitors. An artificial polypeptide receptor (APR) library was created by using the self-organization of *N*-lipidated peptides attached to cellulose via m-aminophenylamino-1,3,5-triazine. The response of the library was probed using a series of novel H_3 receptor ligands. Since no guidelines on how to design an APRs selective vs. certain receptor types exist, a diverse set of amino acids (Ala, Trp, Pro, Glu, His, Lys and Ser) were used and coupled with one of three gating fatty acids (palimitic, ricinoleic or capric). A competitive adsorption-desorption of an appropriate reporter dye was used for the indirect visualization of the interactions of guests with particular receptors. The resulted library response to individual inhibitors was then arranged in a matrix, preprocessed and analyzed using the principal component analysis (PCA) and partial least squares (PLS) method. The most important conclusion obtained from the PCA analysis is that the library differentiates the probed compounds according to the lipophilicity of the gating unit. The PC3 with a dominant absolute contribution of the receptors containing Glu allowed for the best separation of the ligands with respect to their activity. This conclusion is in agreement with the fact that Glu 206 is a genuine ligand counterpart in the natural histamine receptor.

2. UPLC analysis for diosgenin detection infenugreek extracts obtained with different methods

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Fenugreek (*Trigonella foenum-graecum*) is one of the several plant sources that produce diosgenin. Diosgenin, a steroidal sapogenin belonging to the group of triterpenes, is a very important compound in the pharmaceutical industry. It is used primarily as a precursor for the synthesis of steroidal drugs, oral contraceptives and sex hormones. Diosgenin plays an important role in the control of cholesterol metabolism and has anticancer activity, antagonistic effect on rheumatoid arthritis, cardiovascular -, and antimalarial action. The aim of this work was comparison of different extraction methods of diosgenin and identification this compound with the use of UPLC method and two way peaks detection by DAD and CAD detectors.

We tested efficiency of five different methods of diosgenin extraction. Method (1) proposed by Savikin-Fodulovic used hydrolysis of plant material with sulfuric acid in isopropanol and then extraction with hexane. Next, in the three liquid phase (TLPS) procedure (2), diosgenin was extracted with ethanol, ammonium sulphate and petroleum ether. In a protocol described by Li (3), the lyophilized material was subjected to ultrasonication and then hydrolyzed under reflux with sulfuric acid and extracted with petroleum ether. Another method (4)used hydrolysis under reflux with hydrochloric acid and then extraction with chloroform. The last (5) method is simple extraction with only two solvents: hexane and methanol.

To investigate of the extraction efficiency, the chromatographic separation of the plant extracts was performed in Waters Acquity UPLC^{TM} system (Waters Corp., Milford, MA, USA) with DAD and CAD detector. An Acquity UPLC^{TM} BEH RP18 column (50 mm×2.1 mm I.D., 1.7 μm) also from Waters was used. The column and sample temperature were maintained at 30°C. The mobile phase consists acetonitrile and water (9:1 V/V). The injection volume was 50 μL. The detection wavelength (DAD detector) was 203, 236 and 248 nm. The flow rate was set at 0.4 mL/min.

3. Piperidine and piperine: extraction and content assessment in black pepper

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Piperidine is a cyclic secondary amine, which can be considered as a parent molecular structure for many alkaloids. The mature black pepper fruit contains a specific substance – piperine – which chemically is an amide and a source of pungent taste, highly valued with this culinary spice. Piperine is a piperidine derivative as a condensation product with an aromatic acid derived from 3,4-dimethoxycinnamic acid. Nowadays, it is widely used in various herbal cough syrups, and it is also employed in the anti-inflammatory, anti-malarial, and anti-leukemia treatment [1]. Piperine can contribute to an increased bioavailability of many substances according to a number of mechanisms. It inhibits several enzymes responsible for metabolizing nutritional substances and it stimulates amino acid transporters in the intestinal lining. Moreover, recent studies demonstrate that piperine can reduce the fat level in the bloodstream by inhibiting the fat cell differentiation [2].

In our studies, the classical solvent extraction and the Accelerated Solvent Extraction (ASE) of alkaloids from the ground black pepper fruit was performed. Based on the literature [1], we selected the following system for running the thin-layer chromatographic analysis of piperine: stationary phase, silica gel; mobile phase, acetone + *n*-hexane, 3:2 (*v*/*v*). In our efforts to determine piperine in the commercially traded spices, we decided for TLC/densitometry as a well suited and very promising analytical technique. As main advantages of this technique, we consider its simplicity, rapidity, and cost-friendliness, and the quantitative results obtained with its aid are often comparable with those originating from HPLC. The results obtained in this study confirm an excellent performance of TLC/densitometry in the analysis of piperine contained in botanical material.

We also quantified piperine and piperidine by means of high-performance liquid chromatography (HPLC). The analysis was carried out with use of Pursuit 5 C18 chromatographic column and pure methanol as mobile phase. For piperidine, the ELSD detector was employed, and piperine was quantified with DAD detector at a selectively operating wavelength λ = 343 nm. The obtained quantitative results were tabulated and a comparison was made with respect of the employed measuring technique.

References

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4. Analysis of perindopril and quinapril, the ACE inhibitors by GC-FID

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Perindopril and quinapril are angiotensin-converting enzyme (ACE) inhibitors, commonly used in therapy of hypertension and other circulatory system diseases. The compounds have related chemical structures and similar physicochemical properties. The aim of presented study was to analyze these compounds together, using capillary gas chromatography with flame-ionization detector (GC-FID). Despite FID is the most common detector in gas chromatography, there were no elaborations in this area yet. Simultaneous analysis of both compounds in one run was successfully executed. Each compound served as internal standard for another. Direct analysis without derivatization step was performed. Analysis was possible despite compounds high boiling points, because of their sufficient volatilization in applied conditions. The retention times (t_R) equaled about 3,8 min. and 7,9 min. for perindopril and quinapril respectively. Gas chromatography was performed with a Trace GC (Thermo Finnigan), Compounds were separated on a 15 m × 0.25 mm i.d. WCOT column (Hewlett–Packard) coated with polydimethylsiloxane. Helium served as carrier gas. The temperature program was set as followed: 200˚C to 300˚C with temperature rise 10˚C/min. The total run time was 10 minutes. The inlet temperature was set at 300˚C, whereas injection volume was 4 μL and split 30. The detector, with base temperature of 260˚C was supplied. In the next step, in order to prove method suitability, the analysis in pharmaceutical products was done. No interferences were found, though other peaks were eluted.

5.

HPLC/DAD, HPLC/ELSD, and LC-MS investigation of spontaneous oscillatory reactions of L-phenylalanine and L-hydroxyproline in 70% aqueous acetonitrile solutions

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In previous studies, it has been shown that profen drugs, amino acids and hydroxy acids in aqueous or non-aqueous solutions can undergo spontaneous oscillatory condensation. For example in the studies with L-lactic acid dissolved in pure acetonitrile and 70% ethanol, it was found out that this hydroxy acid can undergo oscillatory oligopeptidization, and also a theoretical model was proposed to describe this phenomenon.

To check that another pair of amino acids (L-hydroxyproline–L-phenylalanine) can undergo spontaneous oscillatory oligomeryzation, we used the achiral high performance liquid chromatography with spectrophotometric detection (HPLC/DAD), the light scattering detection (HPLC/ELSD) and the mass spectroscopic detection (LC-MS). The choice of these amino acids was due to their important functions in human body. L-Hydroxyproline is major component of the protein known as collagen and it is responsible for its stability, and hence, for the tissue architecture and strength. L-Phenylalanine is a precursor for tyrosine, dopamine, noradrenaline, adrenaline, and the skin pigment melanin, and therefore it is an amino acid necessary for proper functioning of human body.

The obtained results demonstrate the oscillatory instability of the pair of amino acids (L-Phe–L-Hyp). In this spontaneous oscillatory reaction homo- and heterooligopeptides are obtained and most probably the additional reaction products (like esters) are also generated.

6.

Gas chromatography in determination of fuel char reactivity and gasification process efficiency - experimental study on steam gasification of various fuels in fixed-bed reactor

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Increasing energy demand, unstable market of fossil fuels and environmental concerns reflected in relevant regulations enforce development of technologies of sustainable energy production. Much attention is given to the development of integrated gasification combined cycle based on gasification process, with a use of which power and hydrogen, as an environment friendly energy carrier, are produced. Utilization of renewable energy resources, including biomass, is also supported. Since waste biomass is estimated to satisfy only approximately 7.5% of the world energy demand, cultivation of energy crops is considered on wastelands and post-industrial areas. Improvement in energy systems efficiency, mitigation of CO_2 emission and more effective utilization of biomass are key aspects of the development of Polish energy sector, based in approximately 90% on coal. Gasification technologies make feasible more efficient utilization of fuels when compared to combustion systems, with a use of variety of gaseous, liquid and solid fuels and production of synthesis gas of wide application potential in heat and power generation, chemical synthesis and liquid fuel production. These technologies have been successfully implemented in over 140 plants of the total syngas capacity of 71,000 MW_{th}. Nevertheless, there are still some technical and technological aspects requiring further optimization, especially when waste and biomass utilization is considered in gasification systems, including optimization of feeding system, fuel pretreatment (drying, grinding, torrefaction, pelletization), selection of operating parameters and type of reactors suitable for a particular fuel and required product gas composition as well as development of effective measures of prevention of low melting point ash and tars accumulation.

Gas chromatography (GC) is one of the key techniques applied in the above mentioned research works making feasible assessment of fuel char reactivity in the gasification process and process efficiency in terms of gas composition, gas heating value, carbon conversion and energy efficiency. Here, the results of application of the GC technique in the study on steam gasification of various fuels in a laboratory scale fixed bed reactor installation are presented.

7.

Specific mobility of selected phytochemicals in thin-layer chromatographic systems and its possible relevance to pharmacokinetics

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On video images of the chromatograms present in numerous publications on the TLC analysis of medicinal plant extracts, striking skewness of the separated chromatographic bands with many phytochemicals can be seen, although no attention has ever been paid to this odd mass transfer effect. Lateral relocation (i.e., sidewise deviation of the analytes' migration route from linearity) has been reported in our earlier studies on the thin layer chromatographic enantioseparation of chiral low molecular weight carboxylic acids belonging to the groups of profen drugs, amino acids, and hydroxy acids. Then we found out that lateral relocation was observed with these analytes only, which structurally resembled molecular rotors. In this study, we investigate the role played by the thin-layer chromatographic stationary phases in lateral relocation of botanically relevant molecular rotors. Thus, three carboxylic acids were selected as the test analytes, all of them resembling molecular rotors and abundantly present in the medicinal plant extracts. We selected two of the most popular thin-layer chromatographic stationary phases: silica gel (characterizing with microcrystalline chirality) and alumina (achiral). These results show that the mobility of tested compounds on the chiral stationary phase surface characterizes with lateral relocation. A conclusion was drawn that the chiral stationary phase makes a complementary contribution to lateral relocation (along with the propeller-like molecular structure of the analytes). In our view, specially devised thin-layer chromatographic systems can prove a convenient nano-platform for future investigation of the drug transport patterns, advantageous in the pharmacokinetic studies.

8.

Chromatographic methods of investigating spontaneous oscillatory reactions of L-phenylalanine and L-proline in aqueous solutions

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In our earlier studies, it has been shown that the low molecular weight chiral compounds from the groups of profen drugs, amino acids, and hydroxy acids in the solution can undergo spontaneous oscillatory condensation. These reactions are characteristic of single compounds or the mixtures of compounds in the aqueous or non aqueous solvents. In the studies with a mixture of L-proline and L-hydroxyproline dissolved in 70% methanol, it was found out that these compounds not only undergo oscillatory peptidization, but a theoretical model was also developed which describes this phenomenon.

In this study, another pair of amino acids (L-proline–L-phenylalanine) was investigated. To this effect, we used the achiral high performance liquid chromatography with spectrophotometric detection (HPLC/DAD), the light scattering detection (HPLC/ELSD), and the mass spectroscopy detection (LC-MS). The choice of these amino acids was due to their important functions in human body. L-Proline is major components of the protein known as collagen and it is responsible for its stability, and hence, for the tissue architecture and strength. L-Phenylalanine is an exogenous amino acid, one of twenty common amino acids which build proteins. It is a precursor for tyrosine, dopamine, noradrenaline, adrenaline, and the skin pigment melanin, and therefore it is an amino acid necessary for proper functioning of human body.

The obtained results demonstrate the oscillatory instability of the investigated amino acids, which consists in the spontaneous oscillatory oligopeptidization, i.e., in the sequential formation and decomposition of homo- and heterooligopeptides as the products of the spontaneous peptidization process.

9.

TEREPHTHALAMIDES OBTAINED FROM METHYL ESTERS OF CHOSEN AMINOACIDS

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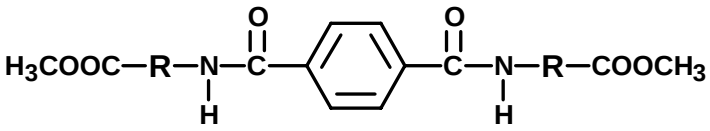
This study was aimed at synthesizing terephtalic acid diamides from methyl esters of chosen aminoacids**,** examining physicochemical properties of the new compounds and determining their crystallographic structure.



Przykłady:

R =

(Gly-OMe) —CH_2— , (βAla-OMe) —CH_2—CH_2— , (Ala-OMe) —CH(—CH_3)— , (Asp-OMe) —CH(—CH_2—$COOCH_3$)—

(Val-OMe) —CH(—$HC(CH_3)_2$)— , (Phe-OMe) —CH(—CH_2—C_6H_5)— , (Ile-OMe) —CH(—$HC(CH_3)$—CH_2—CH_3)— , (Met-OMe) —CH(—CH_2—CH_2—S—CH_3)—

Chemical structure of intermediate products and diamides was confirmed by 1H NMR and 13C NMR. Additionally, FTIR and ESI-MS spectra were recorded. Solubility and melting point temperature were determined for new diamides. Crystallization was attempted in order to obtain monocrystals.

As the result of our investigations seventeen (17) aminoacid methyl ester hydrochlorides were obtained, as well as seventeen (17) terephtalic acid diamides, so far unreported in the literature.
The structure of all the obtained compounds was corroborated by spectroscopic methods.

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10.

Polyphenolic profiles of Serbian polyfloral honeys and discrimination of the geographical origin

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Honey is a natural sweet product produced by honeybees. It is essentially an aqueous solution of saccharides, primarily glucose and fructose, and other substances, such as organic acids, amino acids, proteins, polyphenolic compounds, minerals and other chemicals. In this paper, a total of 58 polyfloral honey samples from different regions in Serbia were studied to determine their polyphenolic profile, total phenolic content and antioxidant capacity, as well as the relationships between them. Total phenolic content (TPC) was determined by the modified Folin-Ciocalteu method, and radical scavenging activity (RSA) by the DPPH (1,1-diphenyl-2-picrylhydrazyl) method. A good correlation (R = 0.865) was observed between TPC and RSA. Total phenolic content ranged from 0.03 to 1.39 mg/g and radical scavenging activity ranged from 1.31 to 25.61%. UHPLC–Orbitrap–MS made possible identification of a total of 38 different compounds, and quantification was done using 14 available standards. Data on polyphenols allowed the discrimination and classification of honeys in accordance to their geographical origin using pattern recognition technique, Linear Discriminant Analysis (LDA). LDA showed significant separation between honey extracts originating from different regions in Serbia: Zlatibor, South, East, Vojvodina, and Central.

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11.

A comparison of the plant extraction methods upon an example of common thyme (*Thymus vulgaris*)

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Pharmacognosy is an area of science involved in the investigation of botanical materials and of chemical composition thereof. It is based mostly on plant anatomy and morphology, but also on chemical analysis of plant constituents and their biological properties [1]. Until quite recently, basic knowledge about herbal preparations used for the curative purposes originated from the experience and tradition of folk medicine. With time and owing to the growing sophistication of analytical and pharmacodynamic methods of evaluating curative preparations, increased information has been cumulated on the properties and medical efficacy of plants and the plant-derived medicaments [2].

One of more important plants which are grown on a commercial scale in Poland is common thyme (*Thymus vulgaris*). Phenolic acids contained in this plant show an indisputable pharmacological activity, e.g., as antibacterial and anti-inflammatory agents [1,3].

At the preliminary stage of this study, phenolic acids were extracted from the plant tissue by means of two techniques. One technique was classical extraction in Soxhlet apparatus with use of pure methanol as an extraction agent, and the second one was the Accelerated Solvent Extraction (ASE), with use of the aqueous methanol mixtures in different volume proportions. Central composite design was adopted to optimize the solvent concentration and temperature of the ASE. At the second stage, all the obtained extracts were analyzed with use of 1D isoctratic thin-layer chromatography, in order to compare the respective extraction yields.

The obtained extracts were point-wise spotted on to the chromatographic plates in the 10-μL aliquots with use of an automatic applicator. Then the chromatograms were developed to the distance of 15 cm in the horizontal sandwich-type chromatographic chambers. Silica gel was used as stationary phase and ethyl acetate + acetic acid + formic acid + water in volume proportion 100:11:11:13 as mobile phase. For visualization of the chromatograms, two visualizing agents were used, i.e., the 1% methanol solution of (2-aminoethyl) diphenyl borate and the 10% methanol solution of sulphuric acid. In that way, two fingerprint-type chromatograms were obtained for each plant extract, which were then twice photographed (before and after visualization) upon illumination with UV light at two different wavelengths (λ=254 and 366 nm). For the assessment of the chromatograms, scanning densitometry was also used. The densitograms were recorded before and after visualization at the two different wavelengths (λ=340 and 380 nm). All this information in a digitalized form was then utilized for chemometric comparison of the extraction performance with the two employed extraction methods.

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12.

Chromatographic fingerprints and chemometrics as a tool for prediction of the total antioxidant capacity of rooibos infusions

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Over the last years antioxidant compounds have gained popularity due to their ability to scavenge free radicals – reactive species causing damage in living organisms. Food (e.g., fruits, vegetables, and herbs) is a natural source of such compounds. Food consists of complex mixtures of many antioxidant compounds, complicating their analysis. The total antioxidant capacity (TAC) of samples can be determined using chemical methods that exploit the reaction between artificially generated free radicals and all the antioxidants contained in the tested sample. The reaction is monitored with a suitable instrumental technique (e.g., spectrophotometry or fluorometry). Examples of these methods are FRAP, ORAC or DPPH assays [1]. Despite the simplicity of chemical methods (just to monitor the reaction) they are time and reagent consuming and expensive. Determining the TAC of samples using chromatographic techniques is challenging, as well as time and reagent consuming. On the other hand, chromatographic fingerprints could be used to characterize the antioxidant compounds in foods.

Our goal is to propose an approach to predict TAC of complex natural samples (rooibos infusions) directly from their chromatographic fingerprints (HPLC-DAD chromatograms) without the need to quantify potential antioxidants. The proposed model describes the TAC parameter determined with standard chemical method (DPPH assay) on the basis of a set of fingerprints. The partial least squares regression, PLS [2] is used as a multivariate calibration method. The proposed approach offers satisfactory prediction properties, eliminating the need for additional TAC tests, and considerable reduction of time and chemical reagents.

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13.

Chemical characterization of sour cherry wine produced in Serbia

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Wines contain a number of polyphenolics which contribute to beneficial effects on human health and because of that have attracted much attention in recent years. In this study, wine of sour cherry cultivar Oblačinska and five red wines (Vranac Potkranjski, Cabernet Sauvignon (Radovanović), Cabernet Sauvignon (Rogljevo), Cabernet Sauvignon (Bailo) and Frankovka Banoštor) were purchased directly from different wineries in Serbia during 2011. The aim of this study was the determination and comparison of the total phenolic (TPC), total anthocyanin content (TAC), and radical scavenging activity (RSA) in cherry wine and five red wines. Polyphenolic compounds possess antimicrobial, anti-inflammatory, antimutagenic, antitumor and antioxidative activity. Liquid chromatography coupled with a hybrid mass spectrometer (UHPLC- MS/MS Orbitrap) was used for a study of the phenolic components of wines. RSA was determined with DPPH reagent using slightly modified standard method. The content of total phenolics and anthocyanins was compared with RSA. TPC ranged from 1.19 to 2.50 g GAE/l wine. The highest content of total anthocyanins was found in Frankovka Banoštor wine (0.219 g cyn-3-glu/l wine). Correlation analysis was used to explore relationships among RSA, TPC and TAC. RSA is a relatively high correlated with TPC (r = 0.990).

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14.

Development and validation of a TLC-densitometric method for the quantitative determination of amygdalin

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Amygdalin became popular like a potential anti-cancer drug. Although there is no scientific evidence to support claims that it may treat cancer, recently, interest in amygdalin is gradually increasing and its use as secondary cancer therapy has been encouraged. Amygdalin can also be used for the treatment of asthma, bronchitis, diabetes, for preventing and treating migraine, hypertension, chronic inflammation etc. In view of its pharmacological effects, an efficient and simple method for amygdalin analysis from natural sources is highly desirable.

Soxhlet extraction is the most commonly used technique for amygdalin extraction while for its determination different HPLC methods were used. In this study, we have explored accelerated solvent extraction (ASE) as an alternative to Soxhlet extraction. Instead of HPLC the TLC is chosen as cheap, simple and sensitive liquid chromatographic method.

The reversed-phase TLC system was consisted of RP-18 silica as the stationary phase and ACN-water, 50:50 (v/v) as mobile phase. Densitometric evaluation was performed at 210 nm.

Validation of the proposed method was carried out with respect to the following parameters: linearity and range, precision, limit of detection and limit of quantitation. The linear regression data for the calibration plots showed good linear relationship with $R^2 = 0.998$ in the concentration range 2.5-50.0 μg per spot. The average recovery was found to be 97.34%. The limits of detection and quantitation were 1.28 μg and 4.28 μg per spot, respectively.

Statistical analysis proves that the developed TLC method is repeatable, selective, and accurate and can be applied for the identification and quantitative determination of amygdalin.

15.

Application of gas chromatography in the study of the polycyclic aromatic hydrocarbons concentrations in gas sampled from the burning mine waste dump located in Ruda Śląska, Upper Silesia, Poland

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Polycyclic aromatic hydrocarbons (PAHs) is a group of environmental contaminants, which constitute an extremely large and diverse class of organic molecules [1], includes over a hundred of various compounds, formed typically during incomplete combustion of organic matter at high temperatures produced primarily from anthropogenic sources [2]. Mine waste dumps are highly heterogeneous, i.e. they are composed of various materials, often flammable, posing a potential risk of autoxidation and self-ignition. Burning mine waste dumps cause a threat to the environment increasing with time, due to the potential fire propagation, and pollution of the atmosphere with dust and noxious gases [3]. The autoxidation and self-heating processes result in intensive release of various gases, including PAHs. Therefore, mine waste dumps may be considered to be a serious source of organic contaminants, including PAHs, demonstrating persistent, bio-accumulative properties [4]. The main objective of the study presented was to determine the PAHs concentrations in gases released from the burning mine waste dump in Ruda Śląska (Upper Silesia, Poland). All samples were extracted using Accelerated Solvent Extraction ASE 200 equipment (DIONEX) and hexane as a solvent. Liquid chromatography method (HPLC 1200 Series Agilent Technologies) with a column ZORBAX Eclipse PAH (4.6 mm × 150 mm, 3.5 μm) was applied in the analyses.

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16.

Chromatographic vs. calculated lipophilicities of selected cosmetic raw materials

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The chromatographic behavior of 22 selected cosmetic raw materials (sunscreens, preservatives and vitamins) was investigated using thin-layer chromatography on RP-18 stationary phase using water-organic modifier (acetone, methanol, dioxane, acetonitrile, dimethylformamide or tetrahydrofurane) binary mixtures as mobile phases. Good linear correlations (R^2>0.98) were found between R_m values and volume concentrations of the organic modifiers. Obtained by extrapolation to zero concentrations of six organic modifiers R_m^0 values were correlated with lipophilicities calculated *via* different methods (ALOGPs, AClogP, ABlogP, milogP, ALOGP, MLOGP, KOWWIN, XLOGP2, XLOGP3, ACDLab). Comparison of correlations between R_m^0 and calculated logP values revealed that methanol, acetone and dioxane are better modifiers in chromatographic lipophilicity measurements than dimethylformamide, acetonitrile or tetrahydrofurane. This conclusion was further supported by analysis of relationships between R_m^0 and experimental $logP_{o/w}$ found for 9 out of 22 investigated compounds.

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17.

ANALYSIS OF NEW STATIONARY PHASE FOR AMINES DETECTION BY UHPLC UTILIZING MULTIPLE DETECTION METHODS

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Keywords: liquid chromatography, gas chromatography, molecularly imprinted polymers, quality control.

Molecularly imprinted polymers (MIPs) are synthetic polymeric materials with specific recognition sites complementary in shape, size and functional groups to the template molecule, involving an interaction mechanism based on molecular recognition. These recognition sites mimic the binding sites of biological entities such as antibodies and enzymes. Their stability, ease of preparation and low cost for most of the target analytes make them attractive for numerous applications. The use of MIPS as stationary phases for HPLC is one of the best studied application of imprinted polymers, largely because it provides a convenient method for quantitative assessment of the quality of imprints produced by a particular strategy. A wide range of chemical compounds have been imprinted successfully, ranging from small molecules, such as drugs, to large proteins and cells. The best results have been obtained for molecules with molecular weights in the range of 200–1200 Da. The resulting polymers are robust, inexpensive and, in many cases, possess affinity and specificity that is suitable for industrial applications.

In this announcement we present an investigation of new polymeric stationery phase for amines detection using the Nexera UHPLC equipped with a Pinnacle DB PAH 1.9 μm, 50 x 2.1 mm column and the multiple DAD and FLD detectors enabling detection of trace-level components. The described method allows for selective absorption of amines molecules.

18.

Analysis of volatile fraction from selected thyme (*Thymus* L) species by means of GC-MS and HS-GC-MS

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The influence of aromas on physical and psychical well-being of humans has long been recognized in psychology and philosophy. Therefore in aromatherapy, the curative potential of essential oils (plant hormones) is utilized. Essential oils are among the main constituents of plants which are used in plant pharmacy and homeopathy, although too rarely in classical pharmaceuticals. Along with a recognized curative potential, essential oils can also exert toxicity. The high quality pure essential oils are available on the Polish market in the drugstores, selling spots specialized in trading medicinal plants, and in the centers for aromatherapy [1,2].

Essential oils are liquids characterizing with relatively high boiling point (ca. 105^0C) and considerable volatility. From the chemical point of view, essential oils are mixtures of hydrocarbons, alcohols, aldehydes, ketones, esters, and ethers, which are mainly mono- and sesquiterpenes, and also the phenylpropane derivatives. Essential oils exert diverse biological activity which depends on pharmacological activity of their predominant constituents (this activity can be, e.g., antibacterial, decongestant, sedative, anti-inflammatory, etc.). Essential oils can by derived from plants, e.g., by means of hydrodistillation or pressing [3,4].

Common thyme (*Thymus vulgaris* L.) is the plant originating from Mediterranean zone. It is a semi-shrub with characteristic, pleasant aroma of thymol, which contains ca. 3.5% (and sometimes even up to 5.4%) essential oils, and up to 10% tannins, polyphenolic acids, and flavonoids. It exerts a recognized decongestant, antispasmodic, antifungal, and antibacterial activity [5,6].

Plant material utilized in this study originated from Botanical Garden of Maria Curie-Skłodowska University in Lublin. Three different thyme species were investigated, i.e., *Thymus pulegioides*, *Thymus vulgaris*, and *Thymus kosteleckyanus*. We compared the volatile fraction contained in each investigated species by using two different methods of extraction and analysis the volatile compounds. In first approach, essential oils were separated through hydrodistillation in the Deryng apparatus, and then analyzed by means of GC-MS. In second approach, the analysis was carried out by means of headspace-GC-MS.

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19.

Application of chromatographic data to build an analytical model of ligand-G protein-coupled receptors interaction

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The purpose of this study was to examine the structure-activity relationships of agents acting on G protein-coupled receptors. Selected set of GPCR agonists and antagonists was identified from the literature. The chromatographic data and calculated molecular descriptors obtained for representative set of compounds were used in SAR analysis.

Normal phase thin layer chromatography system was used for determination of the chromatographic data. The analysis was carried out in two variants of the mobile phase: acetonitrile-methanol-ammonium acetate buffer (pH 7.4; 0.02 M) (40:40:20, v/v/v) and acetonitrile-methanol-methylene chloride- ammonium acetate buffer (60:10:10:20, v/v/v/v). Glass TLC silica gel 60 F_{254} plates (20×20 cm, Merck, Darmstadt, Germany) were used as the stationary phase. The stationary phase was modified by impregnation with 0.03 M L-aspartic acid in automatic TLC spray chamber (ChromaJet DS20, Desaga, Germany). Such modified stationary phase was used as analytical model of ligand-receptor interaction.The plates were scanned densitometrically at 254 nm by means of a Desaga CD 60 densitometer with Windows-compatible ProQuant software (Desaga, Germany).

Principal component analysis and stepwise discriminant analysis were employed to obtain the relationship between the descriptors and receptor binding affinities, expressed as pK_i values. The set of ligands was classified into two groups according to their degree of biological activity.

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20.

Separation aminoacid methyl ester hydrochlorides sample

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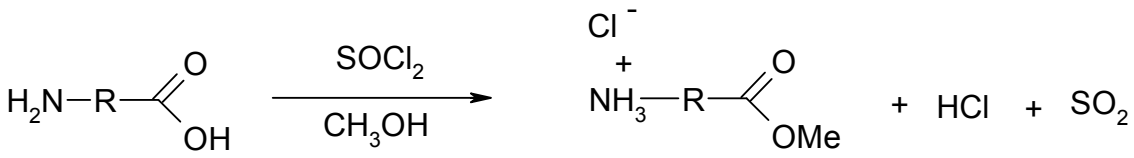
This study was aimed at synthesizing hydrochloride methyl esters of chosen aminoacids**,** examining physicochemical properties of the compounds and separation sample enantiomers.



Chemical structure of intermediate products was confirmed by 1H NMR and 13C NMR. Additionally, FTIR and ESI-MS spectra were recorded. Solubility and melting point temperature were determined for compouds. Crystallization was attempted in order to obtain crystals.

As the result of our investigations a few aminoacid methyl ester hydrochlorides were obtained. The structure of all the obtained compounds was corroborated by spectroscopic methods.

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