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# Posters

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# Sensitive determination of phthalates in edible oils enabled through elimination of phthalate background from HPLC–MS/MS

**Adam Vavrouš[1,2], Václav Ševčík[1,2], Karel Vrbík[1] and Alena Moulisová[1]**
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Diesters of phthalic acid (phthalates) are high production volume chemicals known for their endocrine-disrupting properties. Phthatlate control in various types of matrix may be quite complicated by omnipresent phthalate contamination. Chemicals, laboratory equipment or GC instrumentation typically introduce considerable levels of phthalates into the analytical process. Unfortunately, the role of phthalate contamination in HPLC remains unclear. For this reason, we attempted to identify and eliminate any possible blank difficulties encountered during analysis of 6 individual phthalates (DEP, DBP, DiBP, BBP, DEHP, DnOP) and 2 phthalate isomeric mixtures (DiNP and DiDP) in edible oils by HPLC–MS/MS. Several sources of phthalate contamination were identified, however, the mobile phase was the most serious. The key improvement was achieved by equipping a contamination trap, a HPLC column, generating a retention delay of mobile phase phthalates. LOQs ranging between 5.5 and 110 μg/kg reflect satisfactory blank management and good sensitivity of the employed instrumentation.

## Biography

Adam Vavrouš has graduated in 2010 from University of Chemistry and Technology, Prague. Since 2011, he has been employed as an Analytical Chemist/ Researcher at National Institute of Public Health. In 2012, he started PhD study at Charles University in Prague.

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# Performance Comparison Between Monolithic Column And Silica Based C-18 Particle Packed Column For The Determination Of Three Anti-diabetics In Pharmaceuticals

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The performance of monolithic column was compared with C-18 particle packed column for the analysis of anti-diabetic combination. Three drugs, Metformin, Glimepiride, and Pioglitazone was quantified by both monolithic and reversed phase C-18column. The mobile phase used for monolithic column was consisting of potassium dihydrogen phosphate buffer adjusted to pH 3 by o-phosphoric acid / acetonitrile (55/45 v/v) and the run time of the method was 6 minutes. While for C-18 particle packed column the mobile phase was consisting of potassium dihydrogen phosphate buffer adjusted to pH 3.5 by o-phosphoric acid / acetonitrile (40/60 v/v) and the run time of the method was 9 minutes. The flow rate was 1.5 mL/min for both methods. The HPLC methods using both columns were utilized for determination of the anti-diabetic drugs in bulk powder and marketed pharmaceutical formulation. Monolithic column showed superior results in terms of resolution, run time, peak symmetry, time and solvent saving, and finally back pressure stability.

## Biography

Dr. A. Hemdan has completed his Ph.D at the age of 31 years from Ain Shams University. He is working as an Associate professor at Faculty of Pharmacy-Ahram Canadian University. He has published more than 10 papers in reputed journals.

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# Spectrophotometric determination and recovery of Cr(VI) from waste water by using a novel calix[4] arene based on polymer inclusion membrane (PIM) modified with nanocomposite graphene oxide

**Canan Onac, Ahmet Kaya and H Korkmaz Alpoguz**
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In this study, spectrophotometric determination and recovery of Cr(VI) from chrome plating water by using a novel calix[4] arene through PIM modified with graphene oxide (GO) was investigated. New generations of carbon nanomaterials are the most important ingredients that affect the membrane performance in the production of nano-reinforced membranes. The membrane was modified with GO which improves the mechanical properties and permeability of the PIMs. Important outcomes of this work are high permeability, seamless porous structure, high selectivity and development of PIM by minimized nano-reinforced against pollution in addition of the unique physical properties of GO. The membrane performance and structure of GO/PIM optimized separately. The transport of Cr(VI) was achieved over 90% under optimized conditions from the donor to the acceptor phase through PIM by adding GO which incremented the features of membrane. The World Health Organization had also stated that Cr(VI) as one of the most toxic metal in the environment. For this purpose, the transport of Cr(VI) was carried out from Cr(VI) with a high concentration of donor to acceptor phase. The removal of Cr(VI) was achieved from the chromate plating water as a results of transport experiments performed on different parameters by determining the optimum conditions. The usage of this system is very commercial for industrial waste water applications due its high resistant. PIM/GO exhibits great stability and selectivity in the presence of other metals in chrome plating bath water for the recovery of chromium and the system can be applied on the real samples.

## Biography

Canan Onac is a PhD student at Pamukkale University, Chemistry department in Turkey. Her topics of interest are Electro Driven Membrane, Polymer Inclusion Membrane, and Wastewater Removal.

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**Notes:**

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# A link between aberrant methylation level and AA adducts: Fact or coincidence

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The main aim of this study is to estimate the effect of aristolochic acid (AA) on the genomic methylation level (5-methylcytosine, 5mc) of Sprague-Dawley rats subjected to a single oral dose of 30 mg AA/kg over three weeks-period, and to detect AA-DNA adducts in tissues of rodents. Our analytical method involves hydrolyzing the DNA extracted from different organs to 2-deoxyribose-3'-phosphate, labeling the nucleotides with a fluorescence marker, then measuring the genome wide methylation level with micellar electrokinetic chromatography using laser-induced fluorescence detector (MEKC-LIF). The results had shown different behavior patterns in the methylation level according to the tested organ. An obvious hypermethylation was observed in kidney, stomach and colon of rats dosed with AA, in comparison with their respective controls. Individual 5mc levels are (3.97±0.05, 3.90±0.04 and 4.46±0.04%) for organs, respectively. Bladder, small intestine and liver did not exhibit either hyper- or hypo- methylation. We further studied the samples for detection of AA-adducts in two complementary studies. The deoxyadenosine-AA-adducts were identified in kidney (Adduct level, 2.63 to 5.16/$10^6$ nucleotides) and colon tissues (0.28 to 1.01/106 nucleotides) among several tissues analyzed by the $^{32}$P-postlabeling method. While control samples were free from DNA adducts spots. As well, the highest AA-DNA adduct concentration was detected in kidneys of the AA-dosed rats by LC-ESI-MS method. Our study highlights the relation between the exposure to this carcinogen belonging to natural product group (AA) and the aberration of methylation level that contributes to understanding the involvement of AA-adducts in cancer development via epigenetic modification.

## Biography

Dalia Mohamed El-Zeihery received her PhD from the Bergische Universitaet Wuppertal in Germany and completed Post-doctoral studies at CRO in Germany. She is lecturer of Analytical Chemistry at Beni Suef University in Egypt. She has strong footing in teaching and research, in depth theoretical knowledge and hands-on experience in analytical techniques: like capillary electrophoresis, HPLC-MS and UV-VIS spectroscopy. She was awarded third prize of Pharmaceutical and Bio-analysis from Pfizer for her poster at the 2009 34th International Symposium on HPLC Separations and Related Techniques. His research interests are development and validation of bio-analytical methods for analysis of drugs, proteins and biomarkers.

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Danuse Tarkowska et al., J Chromatogr Sep Tech 2016, 7:5(Suppl)
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# Novel approach for targeted profiling of plant ecdysteroids by UHPLC-MS/MS vs. SFC-UV/MS

**Danuse Tarkowska and Miroslav Strnad**
Palacky University, Czech Republic

Plant ecdysteroids (PEs) are a family of about 300 polyhydroxylated triterpenoids related in structure to the major invertebrate steroid hormone 20-hydroxyecdysone (20E). ECs were originally found in animal sources and recognized as steroidal hormones controlling moulting (ecdysis) and metamorphosis in insects. Later, some of them were discovered to be present also in terrestrial plant families. Their role in plants is still not fully elucidated but at least it is clear that they don't have hormonal function like in insects and serve the most probably as the defense against insect herbivores. The pharmacological and medicinal studies in humans show that PEs act as very effective adaptogens and elicitors of anabolic effects on skeletal muscle in a non-androgenic manner. Methods based on the use of HPLC have historically been most frequently used for separating ecdysteroids isolated from different biological sources either in analytical or in preparative scale. However, in the last decade, UHPLC and SFC experienced a great boom in separation science area. Here, we present new approach for analysis of PEs that is based on UHPLC separation in combination with tandem MS detection. The selected analytes of PEs character (20E, polypodine B, ajugasterone C, stachysterone C and ponasterone A) are separated within 4.5 min using RP column and detected in MRM mode. The method provides sufficient chromatographic resolution and sensitivity with LOD ranging between 0.12 and 5.4pg. To compare the results obtained by UHPLC-(+ESI)-MS/MS technique, we developed also the method based on SFC with both UV and MS detection.

## Biography

Danuse Tarkowska has completed her PhD from Palacky University and Post-doctoral studies from Umeå Plant Science Centre. She is Senior Researcher at the Laboratory of Growth Regulators, Centre of the Region Hana for Biotechnological and Agricultural Research. His current focus is on the development of methods for extraction and purification of plant hormones, analytical methods for plant hormones and other signaling molecules. She has published more than 40 papers in reputed journals (sometimes cited without self-citations 872, h-index: 12). She is the member of Czech Chemical Society and Phytochemical Society of Europe.

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## MLC as an effective tool in the prediction of human intestinal absorption

**D S Shokry, L J Waters and G M B Parkes**
University of Huddersfield, UK

Estimation of human intestinal absorption is very important especially for orally administered pharmaceutical compounds with poor solubility. Therefore in pre-formulation studies the extent of drug absorption must be determined for new drug entities (NDE). The use of animals has been the most abundant method used in pre-formulation studies for determination of pharmacokinetics, especially, the rate and extent of intestinal absorption of pharmaceutical compounds. In this work the use of biodetergent based micellization with a form of chromatography known as micellar liquid chromatography (MLC) has been successful in the determination of human intestinal absorption. Bile salts were used as a mobile phase in the MLC chromatographic method to provide an environment more closely simulating the human intestinal environment. In this method intestinal absorption was successfully predicted by the use of a group of model compounds through measurement and calculation of the partition coefficient, $P_{mw}$. A series of model drugs were prepared in the corresponding mobile phase concentration and injected into the chromatographic system with capacity factors obtained by analysis of the retention data recorded for the model drugs used. Experimentally determined partition coefficients were used alongside other molecular descriptors in the modeling of human intestinal absorption (% HIA) using multiple linear regression. The obtained model confirmed the ability of MLC to predict human intestinal absorption (% HIA) (R2=0.86 and R2Pred.=0.75).

### Biography

D S Shokry has achieved her first degree in Pharmacy in 2009 from Ain Shams University and has completed her Master's degree in Pharmaceutical Analytical Chemistry from Cairo University in 2013. She is currently working towards her PhD as a Member of Dr. L Waters group for finding alternatives to animal testing at Huddersfield University. She has worked as a Teaching Assistant and as an Assistant Lecturer in the Analytical Chemistry department at the Future University, Egypt. She has published three papers in reputed journals and presented her work as oral/poster presentations at five conferences.

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Ebru TÜRKÖZ Acar et al., J Chromatogr Sep Tech 2016, 7:5(Suppl)
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## Simultaneous determination and validation of telmisartan and amlodipine in pharmaceutical preparations using capillary electrophoretic method

**Ebru TÜRKÖZ Acar[1], Hayati ÇELiK[1], Sevinç KURBANOGLU[2], Mehmet GÜMÜSTAS[2] and Sibel A OZKAN[2]**
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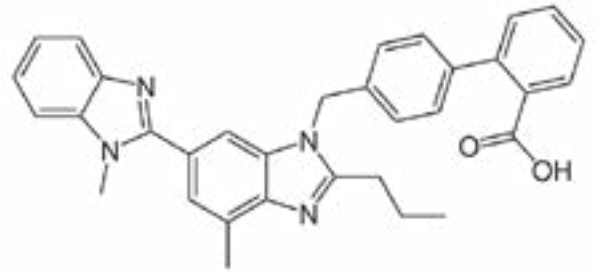
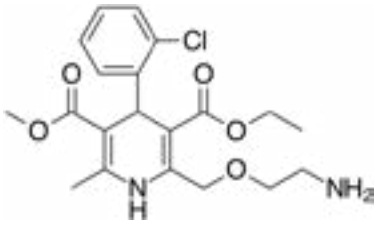
Cardiovascular diseases (CVDs) are the disorders of heart and blood vessels and primarily include coronary heart disease, hypertension, cerebrovascular disease, peripheral artery disease, rheumatic heart disease, congenital heart disease and heart failure. CVDs are the major cause of death in developed countries and also are rapidly emerging as a main cause of death in the developing World. The major risk factors involved in CVDs are high low density lipoprotein (LDL) cholesterol, raised blood pressure, increased serum homocysteine level and platelet aggregation, which are primarily caused by unhealthy diet, physical inactivity and tobacco use. There are various pharmaceutical formulations containing different active materials. One of them contains Telmisartan and Amlodipine besylate. Telmisartan is an angiotensin II receptor (type AT1) antagonist used in the management of hypertension. It is prevents the constriction (narrowing) of blood vessels.




Telmisartan




Amlodipin

Amlodipine besylate is in a class of drugs called beta-blockers. Beta-blockers affect the heart and circulatory system (arteries and veins). It is used to lower blood pressure, lower heart rate, reduce chest pain, and to reduce the risk of recurrent heart attacks. In the literature there are different studies analyzing Telmisartan, and Amlodipine besylate hence, there is no capillary electrophoresis method analyzing these drugs simultaneously. In this study a capillary electrophoretic method will be presented. The aim of study determinates the telmisartan and amlodipin besylate, simultaneously, in tablet formulation. The proposed method has been extensively validated in terms of precision, accuracy. Linear range, limit of detection and quantification values, are also calculated and discussed according to ICH Guidelines and USP criteria. The method can be used for the determination of Telmisartan and Amlodipine in their pharmaceutical preparations.

### Biography

Ebru TÜRKÖZ Acar has completed his PhD from Ondokuz Mayıs University Science Institute Analytical Chemistry Department. She is a lecturer/researcher at Yeditepe University, Faculty of Pharmacy, Analytical Chemistry department.

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# Rapid identification of anti-inflammatory active ingredients from Tumuxiang based on spectrum-effect relationship and natural products application solution with UNIFI

**Fangdi Hu and Xia Gao**
Lanzhou University, P R China

Traditional Chinese medicines (TCMs) have been playing a very important role in health protection and disease control for thousands of years. However, the low efficiency of studying material basis of TCMs is still the bottleneck of restricting Chinese medicine modernization. Nowadays, the latest Waters natural products application solution with UNIFI based on the ultra performance liquid chromatography (ACQUITY UPLC®H-Class), a quadrupole time of flight mass spectromete (Xevo®G2-S QTOf MS) and UNIFI Traditional Medicine Library can offer a turn-key solution. This method integrates data acquisition, data processing and Traditional Medicine Library in a highly automated fashion and makes the chemical composition analysis become simple, effective, sequencing and the work efficiency is greatly improved. In this work, the fingerprint chromatograms of twenty-seven Tumuxiang (TMX) samples were established by UPLC-QTOf-MS technique. At the same time, the anti-inflammatory property of TMX was evaluated by inflammatory models of dimethylbenzene-induced ear vasodilatation. Then, the spectrum-efficacy model between UPLC fingerprints and anti-inflammatory activities was investigated by principal component regression (PCR) and partial least squares (PLS). Finally, automated detection and data filtering were performed using the UNIFITM software. The results indicated that 80 peaks were identified by UNIFI, and 53 characteristic peaks had close correlation with anti-inflammatory activities. The proposed strategy showed high sensitivity, resolution and fast speed, as a brand new solution for complex component analysis of TCMs was more suitable for revealing and identifying the bioactive constituents in TMX, which provided the scientific evidence to preliminarily clarify material basis of anti-inflammatory activities of TMX.

## Biography

Fangdi Hu has completed his PhD from Lanzhou University and Post-doctoral studies from Shanghai University of Traditional Chinese Medicine. She is a Professor at the Department of Natural Medicinal Chemistry, School of Pharmacy, Lanzhou University. She is mainly engaged in the analysis of Traditional Chinese Medicine. She has published more than 25 papers in reputed journals.

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# Identification of components from Sinapis semina that act on the thoracic aorta by screening using cell membrane chromatography combined with online-high performance liquid chromatography-mass spectrum

**Fen Wei and Sicen Wang**
Xi'an Jiaotong University, China

Sinapis semina (JieZi in Chinese), which is the dried seed of *Sinapis alba* L. (Brassicaceae), has been reported for having antihypertensive efficacy. However, the active components have not been investigated. Since many antihypertensive drugs act on receptors in the vasculature, we have developed a Sprague-Dawley (SD) rat thoracic aorta cell membrane chromatography (CMC) coupled with HPLC/MS method, based on ligand-receptor interactions, to screen for active components in S. semina. Firstly, a fraction was recognized and retained by the CMC column. This retained fraction was directed onto an ODS enrichment column, and then analyzed and identified by the HPLC/MS system through switching a two-position ten-port switch valve. In this study, the activity and reproducibility of CMC column and the enrichment rate were investigated by nifedipine which was used as a positive control. The results showed that our SD rat thoracic aortas CMC column was able to recognize receptor-active compounds in a complex system. Both the reproducibility of enrichment and the enrichment rate met the experimental requirements. Then, the methanol extract of S. semina was screened using this method. Sinapine, molecular weight 310 g/mol, was identified as a potential antihypertensive compound. To confirm the effect of the active component from S. semina, tension measurements were performed *in vitro* using isolated rat mesenteric arteries at a dose of $10^{-8}$-$10^{-4}$ mol/L, with nifedipine as the positive control. *In vitro* pharmacological experiments showed that sinapine was able to relax rat mesenteric artery rings. So, sinapine may have a potential antihypertensive effect.

## Biography

Fen Wei is a PhD candidate in School of Pharmacy at Xi'an Jiaotong University. She has participated in 11 published papers in different journals and has acquired a patent as a participator. In addition, another three papers in which she is the first author are under review.

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G Abedi et al., J Chromatogr Sep Tech 2016, 7:5(Suppl)
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# Performance evaluation of different columns in hydrophilic interaction liquid chromatography for the determination of N-nitrosodiethanolamine in shampoo

**G Abedi and Z Talebpour**
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N-nitrosodiethanolamine (NDELA) as a non-volatile and carcinogen nitrosamine has been detected in a wide variety of cleaning products, cosmetic raw materials and products. Several methods have been developed to detect and determine NDELA. In this study, a novel hydrophilic interaction liquid chromatography (HILIC) method has been introduced for determination of NDELA as a polar compound in shampoos. For this purpose, different columns were utilized and their global resolutions and asymmetry factors were acquired in order to evaluate chromatographic performance of each column. Separation was achieved on CN, Si, NH2, and Zwitterionic (ZIC) columns. The flow rate and injection volume were 0.8 mL min-1 and 20 µL, respectively, with UV detection at 234 nm wavelength. In order to optimize mobile phase (MP) composition, content of acetate buffer varied from 0 to 10%. The obtained results were shown retention of NDELA was increased with decrease of water content of the MP, but at least 2% of water is needed for a sufficient hydration of the stationary phase particles. Also pH and the concentration of the buffer were changed in the range of 3.7 to 7.7 and 5 to 100 mM, respectively. Based on our results, the best separation conditions were chosen: acetonitrile/40 mM ammonium acetate, pH 4.7 (98:2, v/v) as the MP and column temperature of 35°C. With respect to the global resolution and asymmetry factor, ZIC column showed the best performance followed by an NH2 column. The proposed method can be satisfactorily applied to the inspection of shampoos.

## Biography

G Abedi is a PhD candidate in Analytical Chemistry at Alzahra University, Tehran, Iran. She has published 3 papers in ISI journals. Her current research interests are Chromatographic Methods, Mass Spectrometry, Cosmetics and Detergent Industry, Pharmaceutical Industry.

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## Analysis of endogenous aldehydes in human urine by static headspace gas chromatography–mass spectrometry

**Manuel Silva and Maria Serrano**
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Endogenous aldehydes (EAs) are intermediary or final products of the metabolism involved in a wide spectrum of biochemical and physiological processes, such as oxidative stress and cell processes. This research reports a solvent-free and automated analytical method for the determination of EAs in human urine using a static headspace generator sampler coupled with gas chromatography–mass spectrometry (HS–GC–MS). Twelve significant EAs used as markers of different biochemical and physiological processes, namely short- and medium-chain alkanals, α,β-unsaturated aldehydes and dicarbonyl aldehydes were selected as target analytes. Direct human urine samples (no dilution is required) were derivatized with O-2,3,4,5,6-pentafluorobenzyl hydroxylamine in alkaline medium (hydrogen carbonate–carbonate buffer, pH 10.3). The analytical method allows the simultaneous derivatization and extraction of EAs in human urine, completing the entire process in 20 min. The HS–GC–MS method developed renders an efficient tool for the fast, sensitive and precise determination of EAs in human urine with limits of detection from 1 to 15ng/L and relative standard deviations, (RSDs) from 6.0 to 7.9%. Average recoveries by enriching urine samples ranged between 92 and 95%. Aldehydes were readily determined at 0.005–50 μg/L levels in human urine from healthy subjects, smokers and diabetic adults. The twelve aldehydes under study were detected in the whole array of human urine samples analyzed. The quantification of aldehydes in those samples showed significantly differences in their concentrations when comparing smokers and diabetics to healthy subjects.

### Biography

Manuel Silva completed his PhD in 1978 at University of Seville. He is Full Professor and Head of the Department of Analytical Chemistry at University of Córdoba. He has published about 150 papers in reputed journals. In the last decade, his research has been focused on the detection of aldehydes as water disinfection by-products and their distribution in treated water. He has directed 15 PhD theses and has worked in several organizations at the Ministry of Education and Science.

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# Temperature controlled liquid phase microextraction, *in-situ* derivatization for determination of estrogens in water by gas chromatography-tandem mass spectrometry

**Maw-Rong Lee, Chung-Yu Chen and Yi-Yu Chen**
National Chung Hsing University, Taiwan

Estrogens, one group of endocrine disrupting chemicals (EDCs), can easily accumulate in the human body and interfere with organism's endocrine system at low concentration. The trace estrogens in environmental water system have attracted scientists' and human attention. Therefore, to develop a simple, rapid, and high sensitive analytical method for determination of the trace estrogens in environmental water is important for environmental safety. In this study, a novel temperature-controlled liquid phase microextraction (TC-LPME) by *in-situ* derivatization coupled to gas chromatography-tandem mass spectrometry (GC-MS/MS) for analyzing trace estrogens including estrone, 17α-estradiol, 17α-ethinylestradiol, mestranol, diethylstilbestrol, and steinheim in water matrix has been developed. An aliquot of 11.25 mL water sample with pH 7 and containing 5% sodium chloride reacted with 3.75 mL dansyl chloride (10 μg/mL) as derivatization agent has been prepared. After derivatization for 15 minutes at 60°C, the derivatives were extracted by liquid phase microextraction with octanol as extraction solvent and the extractant was analyzed by GC-MS/MS. The linearity of the proposed method was 0.05 to 50 ng/mL; with a coefficient of determination above 0.9940. The limits of detection (LODs) of target estrogens were between 0.3 and 1.1 ng/mL. The intra-day and inter-day precisions were between 1.2 and 10.8% and 2.9 and 17.6%, respectively. The recovery of the proposed method ranged 84.9 and 107.8%. The results demonstrated that the proposed TC-LPME-GC-MS/MS method is rapid, simple, high sensitive and high selective method for the determination of trace estrogens in environmental water matrix.

## Biography

Maw-Rong Lee has completed his MS from University of Florida. He is Dean of College of Science and Distinguished Professor of Department of Chemistry at the National Chung Hsing University, Taiwan. He has published more than 80 papers in reputed journals.

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## Origin of haloacetic acids in milk and dairy products

**Mercedes Gallego and M Jose Cardador**
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Chlorine-based disinfectants are the most common sanitizers used in the dairy industry to clean equipments and surfaces due to their effectiveness and simple use. Nevertheless, chlorine reacts with any natural organic matter present in foods and/or equipments to form disinfection by-products (DBPs); haloacetic acids (HAAs) being the major class of non-volatile DBPs. Firstly a chromatographic method was developed in order to determine the origin of HAAs in milk and dairy products. The sample treatment involves deproteination of milk and centrifugation, and then the derivatization/extraction of the HAAs in the supernatant into an automatic static headspace unit. The methylation of the HAAs was performed with tetrabutylammonium hydrogen sulfate as the ion-pairing reagent and dimethylsulfate as the methylation agent. About 20% of the samples (milk, milkshake, cream and yogurt) analyzed contained 2 HAAs at low concentrations (<2 μg/L), which can be ascribed to the contamination from sanitizers usually employed in the dairy industry. An experiment performed on the preparation of infant formula using different types of water showed that the boiling of tap water, containing HAAs, did not remove them. So, the infant formula should be prepared in mineral water free of DBPs. Another point that must be taken into account is the adulteration of milk with water. The experiments showed good correlations between the volume of water added to the milk and the total HAA concentration; therefore, the presence of HAAs in raw milk could be an indicator of adulteration with treated water.

### Biography

Mercedes Gallego completed her PhD in 1980. She is Full Professor of Analytical Chemistry at University of Córdoba. She has published about 250 papers in reputed journals. In the last decade, her research has been focused on the study of disinfection by-products related to the detection of new species and their distribution in water and food that come into contact with treated water. As a teacher, she has directed 25 PhD theses and she has had numerous foreign researchers under her guidance.

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# Introducing a versatile tandem µ-reactor-GC/MS system for rapid characterization of catalysts: Ethanol and citrus unshiu peel conversion

**Michael Soll[1], Koichi Ito[1], Norio Teramae[1], Young-Min Kim[1,2], Chuichi Watanabe[1] and Young-Kwon Park[3]**
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Due to the increased demand for the renewable energy, many researchers are focusing on the thermal and catalytic conversion of biomass and bio-ethanol. Micro scale lab reactors are universally applied to the catalyst research as the first step for developing catalytic conversion process because the downscaling of reactor size offers many advantages such as cheaper equipment, less feeding material, screening capabilities, lower utility requirements and higher accuracy. Recently, a new tandem micro-reactor system, consisted with two independent micro reactors and directly interfaced with a conventional GC/MS system, was introduced for the fast and simple test of catalyst. The tandem micro reactor system consisted of two furnaces in series and is online coupled with a conventional GC/MS. Gas, liquid or solid samples can be introduced into a 1st furnace using a micro-syringe, micro feeder, or sample cup for gas preheating, liquid vaporization, or solid pyrolysis. The vapors from the 1st furnace meet the catalyst located in the catalytic bed of 2nd reactor and converted to other chemicals over a catalyst. The final products were moved to the GC via a deactivated metal needle, separated in the column and finally detected by MS. As first application conversion of ethanol to butadiene (→styrene butadiene rubber →tires) is shown. The efficiency and selectivity of the catalytic conversion of ethanol to butadiene affects the profitability and productivity of the entire process. The second application is considering inedible C. unshiu peel. A desirable treatment method is needed to use C. unshiu waste peel for producing value added fuel or chemical source from it. The tested applications for the catalytic conversion of ethanol and citrus unshiu peel well indicated the feasibility of tandem micro reactor-GC/MS system as a simple and fast screening tool for the catalytic reaction

## Biography

Michael Soll has completed his PhD in Biology from RUB, Germany in 1993. He has been working in Business Development, Marketing and Sales of GC- and LC-MS based laboratory equipment since 20 years. In 2014, he became the Business Development Manager for the Frontier Laboratories Japan in Europe.

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# Evaluation of $Fe_3O_4.SiO_2.Al(OH)_3$ and $Fe_3O_4.Al(OH)_3$ nanostructures for extraction and pre-concentration of 1,4-dioxane from shampoo by gas chromatography

**Mina Ranjbar Zandaragh and M Soleimani**
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1,4-Dioxane presents as a by-product in many consumer products such as cleaning products, cosmetics, shampoos, and laundry detergents. Polyethoxylated raw materials are the most important ingredients of cosmetic and personal care products used as emulsifiers, foaming agents and dispersants. During the polymerizing of ethylene oxide undesirable by-product, 1,4-dioxane may be formed in range of trace to 100 and even 1000 of μg g-1. The obtained results demonstrate that 1,4-dioxane can be absorbed through skin of animals and human. It is shown carcinogenic potential for human as reported by the US Department of Health and Human Services and International Agency for Research on Cancer (IARC). Consequently, the monitoring of 1,4-dioxane in foods, drugs, cosmetic products and also in the environment is very important. Various techniques have been employed for this purpose, such as HPLC, GC, GC-MS and etc. Due to trace amount of 1,4-dioxane in complex matrices like shampoo, extraction of it is often accompanied by some problems. In this study, with regard to the nano magnetic particles (NPs) advantages, we utilized $Fe_3O_4.SiO_2.Al(OH)_3$ and $Fe_3O_4.Al(OH)_3$ in order to extract 1,4-dioxane from shampoo and minimization of matrix effect. Also, X-ray diffraction (XRD), transmission electron microscopy (TEM), Fourier transform infra-red (FT-IR) and thermal gravimetric analysis (TGA) are used for characterization of synthesized NPs. Then optimum conditions to adsorption and desorption of 1,4-dioxane are investigated. GC method has been developed for determination of 1,4-dioxane.

## Biography

Mina Ranjbar Zandaragh is pursuing her MS in Analytical Chemistry at Imam Khomeini International University, Qazvin, Iran. She is an R&D expert in Paxan Co., a leading manufacturer of detergent and hygienic products in the Middle East. Her current research interest includes “Chromatographic methods, mass spectrometry, cosmetics and detergent industry”.

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**Notes:**

Mirtes Midori Tanae et al., J Chromatogr Sep Tech 2016, 7:5(Suppl)
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# Chromatographic purification of the water extract of Virola *surinamensis* (Rol.) Warb, an Amazonian medicinal plant

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*V. surinamensis* (Myristicaceae) known as ucuúba branca is a well known Brazilian medicinal plant used to treat cramps, dyspepsia and general inflammation. The present work describes the phytochemical study of its hot water extract (tea). Seven compounds from three phenolic groups were identified by NMR and MS analyses: flavan-3-ols, dihydroflavonol and flavonols. The aqueous extract was obtained by infusion of the dried leaves powder (2.5%) with distilled water at 77°C for 30 min and stirring every 10 min. The water extract partitioned with butanol yielded the butanolic fraction which was purified by HPLC using a water/acetonitrile linear gradient in a C18 column. Aliquots of 1 mL (100 mg/mL) of the butanol fraction were injected into the column and 10 peak fractions were collected. The chemical identification was performed by nuclear magnetic resonance spectroscopy (NMR) and mass spectrometry (MS). Polyphenols were successfully isolated from the butanolic fraction of the leaves' tea. The separation time was short (30 min) at 10 mL/min flow rate. Seven compounds were identified from five fractions: procyanidin-B3, astilbin, quercitrin, neoisoastilbin, isoastilbin, engeletin and afzelin. Since our extract was prepared according to the folk use (tea), the aqueous extraction was the first choice rather than using non-polar solvents. This could explain why none of the identified compounds was previously reported in *V. surinamensis*. Therefore, this work contributes to the phytochemical study of *V. surinamensis* describing compounds present only in the water extract of the plant leaves. It may also contribute to the pharmacological evaluation of medicinal plants considering that *in vitro* preparations predominate in the majority of the published papers. On this regard, as much as the ethno-information is concerned, the present results reinforce the need for the chemical purification of medicinal plants as it is used in folk medicine, i.e. extraction with natural solvents and procedures compatible with *in vivo* administration by topic or by oral route.

## Biography

Mirtes Midori Tanae, PharmD, has special interest in chromatographic separation of natural compounds from medicinal plants. She has a Post-doctoral research position to accomplish with part of the project entitled "The pharmacological activity of Brazilian Cecropiaceae plants used in Brazilian folk medicine to treat asthma and respiratory diseases" at Federal University of São Paulo, School of Medicine (UNIFESP-EPM).

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**Notes:**

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# A validated UPLC-UV method for bisphenol A (BP-A) levels detection in imported plastic toys and drinking bottled water in Kuwait

**Naser Faisal Al-Tannak**
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Bisphenol A (BP-A) is an essential component of polyvinyl chloride, polystyrene, phthalates and polycarbonate plastics linked by ester bonds, and it can leach out of plastics at high temperature. BP-A is known to have an endocrine disrupting effect and recent studies have started to link its levels as causative factors in many diseases. Kuwait is considered as one of the hottest countries in the world, and measurements of BP-A levels due to leakage from plastics have never been reported. Therefore, this study measures the levels of BP-A in four randomly selected plastic toys and two plastic water bottles from two different companies after storage at 45°C for four days. An ultra-pressure liquid chromatography coupled with ultraviolet detector (UPLC-UV) analytical method was used to investigate BP-A levels in four of randomly chosen plastic toys (plastic tiger- plastic Lego blocks- plastic doll- small dolls) stored at 45°C for four days. The limit of detection (LOD) and the limit of quantification (LOQ) of the established analytical method were equal to 0.4 ppb and 1 ppb, respectively. BP-A levels was 239 ppb in plastic tiger, 30 ppb in plastic Lego, 4 ppb in plastic doll, 3 ppb in small dolls and 59 ppb in drinking bottled water. Surprisingly, BP-A was detected in all selected plastic toys and one out of two randomly selected drinking bottled water. Therefore, imported mineral water should be filled in a glass container rather than plastics due to high climate temperature. Moreover, toys manufacturers should use BP-A free plastics.

## Biography

Naser Faisal Al-Tannak has completed his PhD from Strathclyde University, Glasgow, United Kingdom in 2012. Currently, he is an Assistance Professor at Department of Pharmaceutical Chemistry-Faculty of Pharmacy in Kuwait University. He has published eight peer reviewed papers in reputed journals.

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**Notes:**

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# Use of gas chromatography to determine the cholesterol level in samples of meat and meat products

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Presently, consumers are increasingly attentive to the cholesterol concentration in animal foods. Hence, we used gas chromatography to determine the cholesterol level in samples of broiler meat (breast and leg) and in various chicken products (chicken frankfurters and chicken roll) and in pig meat (muscle, leg) and pig meat products (fillet). The method we used is in agreement with AOAC International 2002-AOAC 994.10 standard. The samples were processed chemically by saponification with 5% KOH in methanol, pH=2, extraction in petrol ether, concentration and dissolution in chloroform, and were thereafter analysed by gas chromatography. We used a Perkin Elmer-Clarus 500 with flame ionization detector and separation capillary HP-5, with hydrogen as carrier gas and air as burning gas. The method was validated "in house", and used as certified reference a standard chloroform solution, 10 mg/mL, SUPELCO, NIST traceable. We determined the following parameters: accuracy, fidelity, repeatability, reproducibility, sensitivity, detection limit, quantification limit and recovery, according to SR EN ISO/CEI 17025:2005, all values being within the admitted range. The following cholesterol concentrations were thus determined in chicken meat and meat products: 150.96±1.45 g/100g breast meat; 164.63±0.72 g/100g leg meat; 71.77±1.35 g/100g frankfurters and 185.44±0.34 g/100g chicken roll; and in pig meat and meat products: 151.31±4.72 g/100g muscle; 152.65±2.59 g/100g leg; 87.04±3.74 g/100g fillet.

## Biography

Ropota Mariana finished her PhD in 2000 within the Faculty of Analytical Chemistry of the Bucharest University. She is coordinating the compartment of gas chromatographic analyses within the Laboratory of Chemistry and Nutrition Physiology of the National research-Development Institute for Animal Biology and Nutrition-IBNA-Balotesti. She published more than 20 papers in national and international scientific journals, rated by ISI or by other databases.

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**Notes:**

Said A Hassan et al., J Chromatogr Sep Tech 2016, 7:5(Suppl)
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# A validated LC-MS/MS assay for the quantitative determination of hypophyllanthin and silibinin in human plasma: Application to a pharmacokinetic study in healthy volunteers

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A selective and sensitive liquid chromatography tandem mass spectrometry method (LC-MS/MS) has been developed for determination of Hypophyllanthin (HPT) and Silibinin (SBN) in human plasma. Sertraline hydrochloride was used as the internal standard (IS). Sample preparation involved liquid–liquid extraction by tert butyl metyl ether. Chromatographic separation was carried out on a C8 column (3 µm, 3.0 x 50.0 mm) with isocratic elution using a mobile phase of water:acetonitrile (10:90 v/v) at a flow rate of 0.6 mL/min. The detection was performed by tandem mass spectrometry with multiple reactions monitoring mode via electrospray ionization source in positive ionization mode. Analysis was carried out within 2.0 min over a linear concentration range of 1.00 -1000 and 1.00 – 500 ng/mL for HPT and SBN, respectively, and the LLOQ was 1 ng/ml for both compounds. The method was validated according to FDA guidelines for bioanalytical method validation and satisfactory results were obtained. This validated method was successfully applied to a pharmacokinetic study enrolling 20 male volunteers administered a single oral dose of Heptex vegetable capsules.

## Biography

Said A Hassan is a Lecturer of Analytical Chemistry and Instrumental Analysis at Faculty of Pharmacy, Cairo University. In 2015, he was awarded the PhD degree in Analytical Chemistry. He finished the MSc in Analytical Chemistry in 2012 and was awarded the best master's thesis in Faculty of Pharmacy, Cairo University. He got the BSc in Pharmaceutical Science in Faculty of Pharmacy, Cairo University in 2007 graded Excellent with honor and ranked 8th in his class. He has experience in topics such as UV-VIS spectrophotometry, chromatographic techniques, capillary electrophoresis, chemometrics & electrochemical methods of analysis.

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**Notes:**

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# Separation of seven flavonoids in *Astragali* radix using ultra-performance subcritical fluid chromatography on different stationary phases

**Shilan Feng**
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Recently, columns packed with sub-2 μm particles are widely employed in liquid chromatography (LC) but are rarely used in ultra-performance subcritical fluid chromatography (UPSFC). The purpose of this study was to compare the effects of different chromatographic column on the separation of seven different flavonoids. The analytes were including calycosin, calycosin-7-O-β-D-glucoside, medicarpin, formononetin, formononetin-7-O-β-D-glucoside, liquiritigenin and genistein. Separation of flavonoids in the analysis is still a challenge, ultra-performance subcritical fluid chromatography (UPSFC) was found to be an appropriate instrument for the rapid and efficient separation of flavonoids. Among the dedicated four different stationary phases charged hybrid modified with fluoro-phenyl moiety was found to be the most suitable providing the fast separation within 13min using gradient elution with carbon dioxide as a mobile phase and methanol as an organic modifier. Other tested stationary phases including BEH 2-EP, HSS C18 SB and BEH column. The baseline separation on these columns was achieved by mean of a change in organic modifier type, adjust temperature and pressure respectively. Quantitative performance was evaluated at optimized conditions and method validation was accomplished, the validation parameters such as linearity, sensitivity, precision, and accuracy were found to be satisfactory. Optimization techniques were successfully used in the determination of Radix *Astragali* in seven kinds of flavonoids. The sensitivity was sufficient for the analysis of real samples.

## Biography

Shilan Feng has completed his PhD in 2003 from University of Chinese Academy of Sciences. She has published more than 100 papers about Traditional Chinese medicine research and has been serving as a teacher of School of Pharmacy, Lanzhou University.

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# Preparation of tetraoxocalix [2]arence[2]triazine coated $Fe_3O_4$ @$SiO_2$ magnetic nanoparticles and its application in determination of PAHs in smokers urine

**Shusheng Zhang, Yanhao Zhang, Huifang Du and Wenfen Zhang**
Zhengzhou University, China

In the present paper, tetraazacalix[2]arene[2]triazine coated magnetic nanoparticles ($TCFe_3O_4$@$SiO_2$) was prepared and characterized. The performance using TCFe3O4@SiO2 as SPE sorbent was investigated using PAHs as probes. Under the optimized SFP condition for PAHs, the urine samples (300 mL) were directly extracted, eluted, evaporated and re-dissolved in 0.3 mL ACN, and then 20 uL was injected for HPLC separation and analysis. The recoveries were also tested and obtained for 85% for Phe, 88% for Ant, 92% for Pyr, 96% for Chr and 93% for Bap by spiking each PAH at 5pg/ml. The each PAH concentration level for the smokers was at 0.5-4.5 pg/ml, and the higher PAH concentration levels were found in the urines who smoke more cigarettes. The PAH concentration levels for the heavy smokers (40 cig/d) were doubled those for the non-smokers. The very low Bap concentration level at 0.4-0.9 pg/ml was also sensitively and accurately detected. The method showed good extraction efficiency for PAHs due to tetraoxocalix[2]arence[2]triazine having benzene rings, which interacted with PAHs based on π-π interaction. The SPE extraction is simple because of the use of the magnetic nanoparticles $TCFe_3O_4$@$SiO_2$. This SPE material can be widely used in the sample pretreatment.

## Biography

Shusheng Zhang has completed his PhD from Zhengzhou University and Post-doctoral studies from Tasmania University of Australia. He is the Director of Center of Advanced Analysis & Computational Science of Zhengzhou University. He has published more than 180 papers in reputed journals.

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**Notes:**

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# Development of a multicomponent method for the analysis of banned substances in cosmetic products by GC-MS/MS

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A new method based on solid phase extraction (SPE) followed by GC-MS has been developed for determination of 40 substances prohibited in the EU in water miscible matrices.The effect of several factors, including sorbent type, salt addition, washing step and elution solvent, on the system response was tested using Taguchi experimental design approach during optimization. Application of the statistical analysis on Taguchi's signal to noise ratios helped to find the optimal values of relevant factors for most compounds. In the optimized procedure, 60 mg of sample dissolved in water was directly extracted by a/the preconditioned SPE column, eluted with 600 µL of Ethyl Acetate, and after dilution, the extract was analyzed by GC-MS/MS without any further cleaning or concentration step. Accuracy, precision, detection limits and repeatability were evaluated during method validation. To test the method reliability, analyte recoveries were determined on spiked real samples including shampoos, shower gels, and face-wash gels. Resulting analyte recoveries varying from 40 to 110 %, repeatability (RSD) from 5 to 20 % and quantitation limits in µg/g range confirmed suitability of this method for routine testing of cosmetic products.

## Biography

Autor graduated from Charles University in 2012. He has been working as analytical chemist in National Institute of Public Health since graduated and has been studying PhD since 2013.

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**Notes:**

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# A novel toolbox for impurity pattern monitoring during inclusion body processing

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*Escherichia coli* is a well-studied recombinant host organism extensively used for recombinant protein production. Albeit E. coli is attributed with high product titres and growth on inexpensive media, overexpression of heterologous proteins often leads to accumulation of target protein in water-insoluble, misfolded aggregates called inclusion bodies (IBs). In practice, IBs are washed, solubilized and refolded to recover the target protein in its active form. Empirical complex washing and solubilization protocols are diverse and product specific and often render poor product quality with different impurities, which negatively impact subsequent refolding. Thus, fast and precise monitoring tools are needed to follow the impurity pattern along IB processing to judge the efficiency of each Unit Operation. However, such reliable monitoring tools are still scarce to date.

In this study, we developed a novel toolbox using UV chromatogram fingerprints and chemometric techniques to monitor impurity pattern in IB washing and solubilization. Furthermore, we were not only able to monitor the process but also identify the optimal time point of transfer from one Unit Operation to the next. We are convinced that this toolbox will not only facilitate DSP monitoring, but will also allow enhanced process control in the future. The different unit operations where the novel toolbox was implemented is shown in Figure 1.

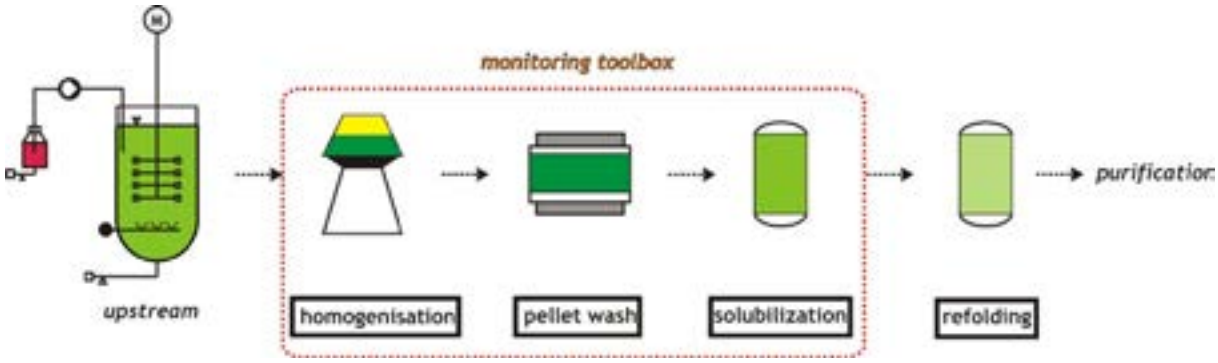


**Figure 1**. Implementation of novel toolbox for monitoring different unit operations involved in inclusion body processing

**Keywords:** process analytical technology, inclusion body processing, HPLC, process monitoring, Escherichia coli, chemometrics, chromatography

## Biography

Vignesh Rajamanickam procured his Master of Science in pharmaceutical biotechnology from Hamburg University of applied sciences, Germany and, Bachelor of Technology in Biotechnology from Anna University, India. He started his PhD on March 2014 in Biochemical engineering from Vienna University of Technology (VUT), Austria. Currently, he is working as a project assistant for developing a novel PAT tool for bioprocess monitoring and control in Christian Doppler laboratory for mechanistic and physiological methods for improved bioprocesses, VUT, Austria.

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# Feasibility of correlating separation of ternary mixtures of neutral analytes via thin layer chromatography with supercritical fluid chromatography in support of green flash separations

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Method development for flash liquid chromatography as normal phase and reversed phase traditionally employs preliminary thin layer chromatography (TLC) with conventional solvents on bare silica. Extension to green flash chromatography via correlation of TLC migration results with conventional polar/non-polar liquid mixtures and packed column supercritical fluid chromatography (SFC) retention times via gradient elution on bare silica with a suite of carbon dioxide mobile phase modifiers is reported. Feasibility of TLC/SFC correlation is individually described for eight ternary mixtures of a total of 24 neutral analytes. The experimental criteria for TLC/SFC correlation was assumed to be as follows: SFC/UV/MS retention ($t_R$) increases among each of the three resolved mixture components; while, TLC migration ($R_f$) decreases among the same resolved mixture components. Good correlations of all 24 analytes were observed via SFC on bare silica with methanol as the $CO_2$ modifier and TLC on bare silica with a methanol/dichloromethane (95/5) mixture.

## Biography

Tony Q Yan is currently working for Pfizer, Inc. (Groton, CT, USA) in the field of impurity isolation for structure elucidation in the Department of Pharmaceutical Science. He has been working in pharmaceutical research and development in the area of chiral and achiral purifications, and impurity isolation for over 20 years since he graduated from the Department of Chemistry in University of Missouri in Rolla with PhD degree in 1995.

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**Notes:**

Yanni Lv et al., J Chromatogr Sep Tech 2016, 7:5(Suppl)
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# New method of screening allergenic components from Yejuhua injection with LAD2/CMC model online UHPLC-ESI-IT-TOF-MS system

**Yanni Lv, Shengli Han and Langchong He**
Xi'an Jiaotong University, China

Yejuhua (YJH) injection is a traditional Chinese medicine (TCM) extracted from the single herb Yejuhua (*Chrysanthemi Indici Flos, dry anthotaxy of Chrysanthemum indicum L.*), which is widely used for the treatment of acute tonsillitis, upper respiratory tract infection, and throat ache in clinical practice. Linarin, a flavonoid glucoside reported to be one of the major active components of YJH injection, can cause inhibition of aldose reductase, phosphodiesterase and platelet aggregation. ADRs caused by YJH injection, majorly manifested as allergic reactions. Non-IgE mediated drug hypersensitivities, also referred to as pseudoallergic or anaphylactoid reactions have clinical manifestations that are often indistinguishable from allergic reactions. Effective and practical method for allergen screening and identification in YJH injection is in need. Cell membrane chromatography (CMC), developed by him and his colleague in 1996, is a new type of biological affinity chromatography and has been confirmed as an effective method to screen bioactive components from complex systems. In the present study, an LAD2/CMC-UHPLC-ESI-MS/MS method was established for screening, analyzing, and identifying the allergenic components from YJH injection. A retained fraction on the LAD2/CMC column was got, and identified as linarin. In order to verify whether linarin could induce LAD2 cells degranulation, histamine (HA) release assay was performed by the method of HPLC-ESI-MS/MS we established before. Results showed that linarin had an allergic effect by increasing histamine release in a dose-dependent manner from 10 to 100 μM. In conclusion, the LAD2/CMC-UHPLC-ESI-MS/MS system developed in this study can be used to screen allergenic components in other TCM injections.

## Biography

Yanni Lv is pursuing her PhD in School of Pharmacy at Xi'an Jiaotong University. She is pursuing her Doctor's Degree in Pharmaceutical Analysis and supervised by Professor Langchong He. She has completed her college studies from Xi'an Jiaotong University, a key university which is directly administered by the Chinese Education Ministry and is one of the oldest current institutions of higher education in China.

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**Notes:**

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# Screening allergenic components from Danshen injection via HMC-1/CMC online UHPLC-ESI-MS/MS system

**Yuanyuan Lin, Meng Sun, Tao Zhang, Delu Che and Langchong He**
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Danshen injection is a Traditional Chinese Medicine (TCM) injection widely used in China to treat coronary heart disease and angina. Adverse drug reactions of danshen injection, majorly manifested as allergic reactions, were among the leading causes of death from TCM injection. In the present study, an HMC-1/CMC online UHPLC-ESI-MS/MS system was established to screen and identify allergenic components in danshen injection, by which salvianolic acid A, isosalvianolic acid C, and salvianolic acid C were identified as potential allergenic components. Allergenic activities of salvianolic acid A, isosalvianolic acid C, and salvianolic acid C were investigated in HMC-1 cell intracellular $Ca^{2+}$ mobilization assay, histamine release and β-hexosaminidase release tests *in vitro*. The results showed that the changes in $Ca^{2+}$ influx in HMC-1 cell clearly increased under salvianolic acid A (100 μM), isosalvianolic acid C (12.5 μM) and salvianolic acid C (25 μM) treatment respectively. In addition, β-hexosaminidase and histamine release in HMC-1 cell were both markedly enhanced with increased concentrations of salvianolic acid A, isosalvianolic acid C, and salvianolic acid C. The HMC-1/CMC online UHPLC-ESI-MS/MS system developed in this study is an effective method for screening and identifying allergenic components from danshen injection, and it may potentially be used to screen allergenic components in other TCM injections.

## Biography

Yuanyuan Lin is pursuing her PhD from School of Medicine, Xi'an Jiaotong University. She completed her graduation from South-Central University for nationalities in 2013. Her research interests are "Development of advanced analysis methods for rapid, accurate and high throughput screening target compounds from the complex samples such as Traditional Chinese Medicine injection". She is the Co-author of three original research papers published in international journals.

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## Decolorization of the mixed dyes by immobilized white-rot fungi

**Bugra Dayi and Hatice A Akdogan**
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Wastewater from the textile industry is one of the most problematic to treat due to its color, high chemical oxygen demand (COD), biochemical oxygen demand (BOD), suspended solids, turbidity and toxic compounds. The chemical composition of the textile effluents has changed rapidly due to a shift in the consumer preferences, the most significant of these being the popularity of cotton fabrics and bright colors leading to greater usage of synthetic reactive dyes and azo dyes. By far the single class of micro-organisms most efficient in breaking down synthetic dyes is the white-rot fungi. These fungi constitute a diverse eco-physiological group comprising mostly basidiomycetous and to a lesser extent litter-decomposing fungi capable of extensive aerobic lignin depolymerization and mineralization. The mechanism of fungal decolorization mainly involves two aspects, biodegradation and biosorption. The biodegradation capability of fungi is due to their extracellular, non-specific and non-selective enzyme system. In our experience, white rot fungus *M. esculenta* was immobilized on to three different support materials (polyurethane, kaolin, cellulose). Bio-decolorization of mixed dyes was investigated and the data were compared for all immobilized cells. Polyurethane was selected as immobilization support material for the best dye removal (dye concentration: 10 $mgL^{-1}$ and 97,78%) in agitated system. At the end of the bio-decolorization, samples (10 mgL-1) were analyzed by FT-IR and UV spectrum to identify any possible metabolites. When the obtained data were examined, no metabolites were found. As a result, immobilized *M. esculanta* on to polyurethane could be used for the wastewater bioremediation.

### Biography

Bugra Dayi has completed his degree and is currently a Master's student in the Department of Chemistry, Biochemistry subdivision at Pamukkale University. He works on Environmental Biotechnology, Waste water bioremediation and Dye removal.

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## Immobilization of *Coprinus plicatilis* onto different carriers

**Hatice A Akdogan, Merve C Topuz, Abdullah Akdogan and Nazime Mercan Dogan**
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The utilization of immobilized cells has shown potential in several bioprocesses including wastewater treatment. Immobilization can be considered as the natural state for several microorganisms; for example, most fungi tend to attach firmly to natural surfaces. Therefore, it is not surprising that artificially immobilized microorganisms can produce extracellular secondary metabolites. In industrial operations, immobilized microbial cell systems could provide additional advantages over freely suspended cells such as simple reuse of the biomass, easier liquid-solid separation and minimal clogging in continuous-flow systems. The immobilization of microorganisms can be defined as any technique that limits the free migration of cells. Basically, there are two types of cell immobilization: entrapment and attachment. In the former, the organisms trapped within the interstices of fibrous or porous materials are physically restrained by a solid and porous matrix. Our research focused on the immobilization of *Coprinus plicatilis* on kaolin, Ca-alginate and gelatin. 3 or 4 age cells and different amount of cells were used for immobilization studies. To the best of our knowledge, the results showed that gelatin was chosen as a support material because it is a natural material with a higher immobilization capacity and is less expensive.

### Biography

Hatice A Akdogan has completed her PhD and works at the Pamukkale Umiversity in the Department of Chemistry, Biochemistry subdivision as an Associate Professor. She studies Environmental Biotechnology, Water and Soil Bioremediation, Chromatographic Monitoring of some organic contaminants during microbial biodegradations, microbial enzymes and their roles.

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**Notes:**

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## Formulation of telmisartan tablet, evaluation and determination by HPLC

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Telmisartan is an angiotensin II type and is used as antihypertensive drug. It poses poor solubility which leads to low bioavailability in blood stream so that; this problem guides many scientists to work in improvement of telmisartan dissolution of its solid dosage forms. The present work shows the formulations of telmisartan as tablet with high enhancement degree in their dissolutions and stability in addition to the improvement in the physical characters of the tablet dosage forms. The formulations of telmisartan were prepared in consideration of manufacturing conditions rather than laboratory productions as it is prescribed over many previous academic papers. The manufacturing procedure of the tablet according to this new formulation is simple and readily applicable in pharmaceutical industries. The method depend on turning the telmisartan to amorphous crystals by mixing with solubilizing polymer and mixed with prepared DC excipients to be compressed as tablets. The DC excipients were modified during the preparation to act as alkalizing agent which in turn enhanced the dissolution rate of the tablets. The physical properties of the powder of formulations were evaluated and it gave an excellent degree of flowability and compressibility while, the compressed tablets showed fast disintegration time and very low friability percent. In addition the dissolution profiles of the produced tablet were more than 85%. For a comprehensive evaluation of this formulation procedure of tablets, a stability indicating method of analysis was developed by using reversed phase HPLC technique to follow the expected changing that might occur on storage of the product (tablets) at accelerated conditions of storage. The HPLC method was able to detect the degradation products of telmisartan in deliberately degraded sample and the produced telmisartan tablets which were stored at accelerated conditions showed good stability.

### Biography

Kahtan J Hasoon has obtained his BSc in Pharmaceutical Sciences from College of Pharmacy, Baghdad University. He obtained his MSc in Pharmaceutical Analysis from Herriot-Watt University, UK. He has been a Member of Academic Staff of Al-Mustansiriya University. Now he is a Lecturer in the Al-Rasheed University College, and as a Technical Consultant in SAFA Pharmaceutical Industries Co., Al-Safa Group.

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## Lignocellulosic hydrolyzate characterization using anion exchange chromatography

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Hydrolyzates from lignocellulosic biomass are a complex mixture of soluble monomeric and oligomeric fragments. These fragments are degradation products from all four natural polymers cellulose, hemicellulose, pectin and lignin. As the polarity and size varies for all fragments depending on the origin, the analysis requires more than one analytical method and a broad know how. Anion exchange chromatography (AEC) has a great potential as the measurement is based on the ability of producing anions from soluble lignocellulosic biomass derived fragments. Thus, the aim of this study was to characterize hydrolyzates from different biomasses for soluble biomass derived degradation products with AEC. Therefore, the parameters column temperature (30°C to 50°C), eluent composition and chromatographic run time were investigated for separation of the biomass derived degradation products and short chromatographic run time. The final method was set to a column temperature of 40°C, an eluent flow of 1mL/min and an eluent consisting of sodium acetate and sodium hydroxide as well as ultrapure water. Using the newly developed method a run time of 70 min could be realized for degradation products from all natural polymers. Additionally, the limit of detection in the range of 0.014 mg/L for 2, 6-dimethoxyphenol and 21.9 mg/L for 4-methoxybenzyl alcohol allows for a simultaneous determination of lignin derived compounds beside the high glucose concentrations. In consequence AEC was used to characterize hydrolyzates from 17 lignocellulosic biomasses for soluble compounds derived from cellulose, hemicellulose, pectin and lignin.

### Biography

Nico Anders has been working in the field of analysis and renewables since 2009. He has obtained his PhD at the TU Braunschweig in the group of Prof. Dr. Vorlop in Technical Chemistry. Since 2013, he is working as junior research group leader in the Aachener Verfahrenstechnik at the RWTH Aachen University. The research interests of Nico Anders are analysis of lignocellulosic biomass, green analytical chemistry, conversion of lignocellulosic biomass and chromatographic separation.

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# The choice of the optimal methodology for the assessment of the molecular weight distribution of polymethylene naphthalene sulfonates

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The technical polymethylene naphthalene sulfonates (TPNS) are used in different industries. TPNS are thought to be mixture of polymethylene naphthalene sulfonates with wide molecular weight distribution (MWD) parameters and different span of the average molecular weights. MWD and average molecular weights determine the field of application of this product. For evaluation of TPNS MWD characteristics, two chromatographic methods are used gel permeation (GP) and thin layer (TL) chromatography as well as extraction-precipitation (EP) procedure. All these methods have drawbacks. For EP, the drawbacks are the duration of analysis; low reproducibility and low selectivity of fractionating and; for GP and TL chromatography, the drawbacks are the poor informativity because of the insufficient separation and complexity of the quantitative assessment of obtained data. We believe that reversed phase high performance liquid chromatography (RP-HPLC) with spectrophotometric detector is an interesting and advanced method. The systematic researches of TPNS MWD by means of this method are very rare. The objective of this research is to develop the methodology of HPLC and to compare it with known methods of the assessment of TPNS MWD. The capabilities and advantages of the methodology of HPLC are demonstrated on the TPNS samples for different industries. The high reproducibility and informativity of the method are revealed compared to exist methods. New method allows to separate, identify and quantify the full fraction makeup of TPNS. The characteristics of MWD are estimated reliably.

## Biography

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## Preparation, characterization and application of H3PO4 activated maize tassel for the remediation of eutrophic phosphorus

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Technologies for PO43- removal from contaminated waters, such as chemical precipitation with lime, are expensive. In this study, the feasibility of utilizing low-cost activated maize tassel for the adsorptive removal of phosphate was assessed. Raw maize tassel powder was impregnated with $H_3PO_4$ in the ratios 0.5:1, 1:1, 1.5:1, 2:1 and 2.5:1 and activated at 600 and 800°C under an inert atmosphere of $N^2$. The activated products were characterized by BET. Activation resulted in an increase in specific surface area and porosity. CAT4 (2:1) activated at 600°C) with SBET 803.8 $m^2$/g and pore size 2.22 nm was further characterized by SEM and used for adsorption studies. Batch experiments were performed to study the removal of phosphate from simulated samples; the optimal parameters were found to be: contact time of 90 min, pH 7 and adsorbent dosage of 1.5 g per 100 mL solution. The adsorption data were fitted to the Langmuir isotherm model ($R^2$>0.99), yielding an estimated adsorption capacity of 15.31 mg $PO_4^{3-}$ per g adsorbent. The activated product was successfully applied for the remediation of phosphate in selected samples from 3 sewage treatment plants in Northern Pretoria.

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## Two-phase olive pomace as an interesting source of biophenols

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Olive oil consumption is associated with a number of health enhancing effects such as the reduction of chronic diseases and the fight against the risk of heart disease. To produce olive oil, a new industrial process called the "two-phase centrifuge system" allows for the recovery of one hand the olive oil and on other hand a wet pomace also called alperujo composed of vegetation waters and olive pieces. The aim of this investigation was to evaluate alperujo as a potential source of phenolic compounds using a rapid, reliable and efficient analytical method. Target antioxidant compounds were followed with UHPLC-DAD/ESI-$MS^n$ in order to identify and quantify biophenols. The results obtained showed the identification of 35 phenolic compounds in 12 minutes with a tentative of identification of new molecules. Furthermore, the aglycon and glycosidic forms of hydroxytyrosol were quantified in high concentration (3 mM). These results could lead to a fast promotion of phenolic compounds in olive oil by-products in terms of a new economic source of interesting phenolic antioxidants for the health, cosmetic and food sectors. .

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## The possibilities of amino acid ionic liquids as a chiral selectors at separation of enantiomers of amino acids and β-blockers

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Chiral separation is an important subject in science as well as in technology. Various chiral selectors, such as cyclodextrins, antibiotics and crown ethers have been widely used in separation of enantiomers because of their chiral recognition abilities. However, the application of many current chiral selectors is often limited due to their low solubility, difficult synthesis, thermal instability as well as high cost. In addition, most of selectors need to be dissolved in other solvents or in a solvent system as work solution. Therefore, using chiral ionic liquids as chiral selector is promising. Amino acids ionic liquids [C4Mim][L-Pro], [C8Mim][L-Pro], [C12Mim][L-Pro] were synthesized and characterized by NMR-spectrums. In the course of the optimization of chiral separation conditions were varied: the composition background electrolyte, the structure of selectors and the concentration of ionic liquid in the modified buffer electrolytes. IL [C4Mim][L-Pro] was the most effective ionic liquid for the chiral amino acids separation under ligand-exchange capillary electrophoresis. The highest enantioselectivity factors (α=5.2) were achieved for tryptophane when complexing metal was cupper. Synergetic effect was observed at simultaneously addition 2-OH-proplyl-β-cyclodextrin and chiral IL to the running buffer as a result enantiomers of propranolol and carvedilol were separated. Analysis of drug formulation "Carvedilol zentiva" and "Anaprilin" was performed.

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## New pre-concentration techniques in capillary electrophoresis for determination of bioactive compounds in complex mixtures

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Our report is focused on the development of new analytical approaches for electrophoretic determination of biologically active substances with traditional CE and developed microfluidic chip-analyzer with different variants of on-line pre-concentration. The use of hypercrosslinked polystyrene as a sorption material in the preparation of urine and blood serum for analysis provided the decrease in detection limits for hydrophobic and hydrophilic analytes. Application of water-soluble oligosaccharide derivatives hyperbranched polyethylenimine (PEI) as a covalent coating of silica fused capillary and combination of focusing principles of different variants on-line pre-concentration for analysis of proteins resulted in a 1100-fold improvement in sensitivity. The potential of long chain ionic liquids for on-line sample concentration techniques of ionogenic and neutral analytes in biological objects by different modes of capillary electrophoresis: zone (CZE) and micellar (MEKC) modes with normal and reversed polarity were investigated. The compounds chosen were biogenic amines and steroid hormones. Imidazolium-based ionic liquids $C_{12}$MImCl, $C_{16}$MImCl were used both as modifiers of electrophoretic systems and as pseudostationary phase. Sweeping with C16MImCl micelles in BGS has provided 83-112-fold sensitivity enhancement factors for catecholamines. It was found out that using highly conductivity sample matrix in sweeping leads to a significant increase in efficiency of analytes up to $1 \cdot 10^6$ t.p. Chemometric processing of the obtained characteristic profiles of biologically active analytes of blood serum and urine samples from healthy donors and patients with endocrine diseases proved to be informative as an additional diagnostic criteria.

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## Prediction of fetal lung maturity using L/S ratio analysis with a simplified sample preparation, using a commercial microtip-column combined with mass spectrometric analysis

**Hee-Jung Chung**
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Fetal lung maturity is estimated using the lecithin/sphingomyelin ratio (L/S ratio) in amniotic fluid and it is commonly measured with thin-layer chromatography (TLC). The TLC method is time consuming and technically difficult; however, it is widely used because there is no alternative. We evaluated a novel method for measuring the L/S ratio, which involves a tip-column with a cation-exchange resin and mass spectrometry. Phospholipids in the amniotic fluid were extracted using methanol and chloroform. Choline-containing phospholipids such as lecithin and sphingomyelin were purified by passing them through the tip-column. LC-MS/MS and MALDI-TOF were used to directly analyze the purified samples. The L/S ratio by mass spectrometry was calculated from the sum peak intensity of the six lecithin, and that of sphingomyelin 34:1. In 20 samples, the L/S ratio determined with TLC was significantly correlated with that obtained by LC-MS/MS and MALDI-TOF. There was a 100% concordance between the L/S ratio by TLC and that by LC-MS/MS (kappa value=1.0). The concordance between the L/S ratio by TLC and that by MALDI-TOF was also 100% (kappa value=1.0). Our method provides a faster, simpler, and more reliable assessment of fetal lung maturity. The L/S ratio measured by LC-MS/MS and MALDI-TOF offers a compelling alternative method to traditional TLC.

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## Isolation of ulceroprotective cucurbitane type triterpenoids from *Cucumis melo* seeds

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Medicinal plants are the richest bio-resources of drugs in traditional medicinal systems, modern medicines, folk medicines, intermediate and chemicals entitled for synthetic drugs. Plants provide a source of inspiration for novel drug development as they contain a vast array of substances that treat chronic diseases. *Cucumis melo* seeds have been traditionally used for treating various health ailments. The main aim of our current study is to isolate cucurbitane-type triterpenoids from *Cucumis melo* seed extract and conduct anti-ulcerogenic activity of the isolated compound. Phytochemical investigations of methanolic seed extract of *Cucumis melo* was carried out which showed the presence of various important phytoconstituents. The main active constituents of *Cucumis melo* have shown a number of potent pharmacological activities. The isolation of Cucurbitane-type triterpenoids was carried out by column chromatography using methanolic seed extract of *Cucumis melo*. Mobile phase hexane and hexane-ethyl acetate (98:2) was used to run the column. TLC profiling was done simultaneously in an appropriate solvent system (hexane: ethyl acetate, 97:3). Various fractions were collected. The fractions with similar $R_f$ value were pooled together. Fractions giving single spot in the TLC were regarded as pure. The isolated compound showed positive result for Liebermann-Burchard test from which we can conclude that the isolated compound might be triterpenoid. The structure of the isolated compound was determined by IR, 1HNMR, 13CNMR techniques. The spectral analysis of the isolated compound showed following results: IR- it showed the peaks at 3383, 2976, 2814, 1721, 1465, 1123 cm-1 indicated the presence of alcoholic group.

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## Determination of niacinamide in cereal, vitamin supplements and cosmetics by HPLC: How the sample affects the required sample preparation

**Karyn M Usher, Daniel Keating and Henry F Rossi III, Michael J Bozym, Anna Glinko and Michelle L Owens**
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Chromatography is a technique used for the separation of mixtures. This is a great simplification of the practical application of chromatography to the separation, identification and quantification of analytes of interest in complex matrices. Sample matrices often contain interferences, thereby producing complex chromatograms in which the analyte of interest is difficult to determine. This presentation describes sample preparation involved in the analysis of niacinamide in complex matrices including beauty products, vitamin supplements and breakfast cereal. Niacinamide is often included in vitamin supplement tablets, and drinks. These samples typically require only minimal sample preparation. This section will include a discussion about common errors to avoid in sample preparation which are important even for simple methods. Niacinamide is also a common additive in personal care products. It's often used as a whitening agent in lotions to lighten skin and give more even complexion. Lotions and creams containing niacinamide are widely used around the world for both medical and aesthetic reasons. Many products list niacinamide in the ingredients but do not give the concentration used. In this study, the amount of niacinamide in several Olay products was determined using Liquid-Liquid Extraction and HPLC. Breakfast cereals are often fortified with niacinamide because it is necessary for proper body function. These foods may contain structurally/chemically similar compounds such as nicotinic acid, vitamers and precursors. This typically means that more involved sample preparation is necessary. In this study, the amount of niacinamide in a cereal was determined by solid phase extraction and HPLC.

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## Micro-extraction pre-concentration of o-phthalates in low alcohol wines coupled gas chromatographic-mass spectrometric analysis

**Valentin A Krylov, P V Mosyagin and A A Ermakov**
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Esters of phthalic acid are very dangerous for human health. Their occurrence in wines is connected with the inflow from the plasticized polymer seals, plastic piping, tanks and stoppers. In this study the high sensitive gas chromatographic-mass spectrometric determination of phthalates in low alcoholic beverages (champagne, red and white wine) coupled ultrasound-assisted emulsification-microextraction was developed. As extractants environmentally friendly hydrocarbons - octane and n-hexane are proposed. The sources of possible systematic errors were investigated: leaking of o-phthalates from chromatographic septum; contamination of phthalate in solvents; influence of macro components of wines; the hydrolysis of o-phthalates and others. For the first time it is shown that the impact of these factors can lead to an overestimation or underestimation of the actual concentration of impurities by 1-2 orders of magnitude. The methods of accounting or elimination of systematic errors are proposed. Purification of solvents by Rayleigh distillation method allows to obtain samples with impurity content lower than (1-4)x$10^{-3}$ mg L-1. Containers for sampling and storage of samples to be analyzed should be made of borosilicate glass or quartz. The content of phthalates in wines was 0.03-1 $mgL^{-1}$. The largest concentrations are characteristic for diethyl-, di-n-butyl-and di(2-ethylhexyl) phthalates. The limits of detection of esters of o-phthalic acid in low alcohol beverages achieved are at the level of $10^{-6}$-$10^{-5}$ mgL-1 and are highly competitive with the best world results. The relative expanded uncertainty of the determination of toxicants is at the level of 13-30%.

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## Gas chromatographic and gas chromatographic-mass spectrometric analysis of high-purity monoisotopic hydrides of silicon and germanium

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At present time, there is a great interest to simple substances, including isotopically enriched ones, with the ultimately low content of impurities. A suitable method for production and ultra purification of the initial substances is their use in the form of volatile hydrides. Gas chromatography-mass spectrometry is the most promising method of analysis of high-purity substances which make it possible to reliably detect the impurities with high sensitivity. It is for the first time that the method of gas chromatography-mass spectrometry was used to determine the impurity composition monoisotopic silanes $^{28}SiH_4$, $^{29}SiH_4$, $^{30}SiH_4$ and germanes $^{72}GeH_4$, $^{74}GeH_4$, $^{76}GeH_4$. Introducing of gaseous samples into gas chromatograph was carried out by automatic two-position valve "Valco EH2C6WEZPH-CER5", connected with the developed sampling vacuum system. 56 impurity components have been determined including the permanent gases, arsine, phosphine, the homologs of silane, disiloxane, sulfur hexafluoride, carbon bisulfide, hydrocarbons, chloro- and fluoroorganic substances. The positive chemical ionization method was used to identify impurities missing in the individual state and thus, not included in the library of mass spectra. It is the first found that monoisotopic hydrides contain an increased concentration of substances that have displaced isotopic composition. The quantitative analysis has been conducted by the method of absolute calibration. The determination of substances missing in the individual state was based upon the dependence of analysis sensitivity on their ionization cross section. The limits of detection for impurities are $2x10^{-6}$ – $1x10^{-9}$ mol. %, which are by 8-20 times lower than those given in literature.

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## Handling time misalignment and rank deficiency in liquid chromatography by multivariate curve resolution: Quantitation of five biogenic amines in fish

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Biogenic amines are used for identifying spoilage in food. The most common are tryptamine, 2-phenylethylamine, putrescine, cadaverine and histamine. Due to lack of chromophores, chemical derivatization with dansyl was employed to analyze these amines using HPLC-DAD. However, the derivatization reaction occurs with any primary or secondary amine, leading to co-elution of analytes and interferents with identical spectral profiles, and thus causing rank deficiency. When the spectral profile is the same and peak misalignment is present on the chromatographic runs, it is not possible to handle the data only with MCR-ALS, by augmenting on the time, or the spectral mode. To overcome both problems, this paper proposes a new analytical methodology for fast quantitation of these BAs in fish with HPLC-DAD by using the icoshift algorithm for temporal misalignment correction before MCR-ALS spectral mode augmented treatment. Limits of detection, REP and average recoveries, ranging from 0.14 to 0.50 μg mL-1, 3.5 o 8.8% and 88.08% to 99.68%, respectively. These results reaches quantification limits for the five BAs much lower than those established by FAO/WHO, and EFSA, all without any pre-concentration steps. The concentrations of BAs in fish samples ranged from 7.82 to 29.41 μg $g^{-1}$, 8.68 to 25.95 μg $g^{-1}$, 4.76 to 28.54 μg $g^{-1}$, 5.18 to 39.95 μg $g^{-1}$ and 1.45 to 52.62 μg g-1 for TRY, PHE, PUT, CAD, and HIS, respectively. In addition, the proposed method spends less than 4 minutes in an isocratic run, consuming less solvent in accordance with the principles of green analytical chemistry.

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## Setting acceptance criteria for validation of chromatographic methods of drug eluting stents: Minimum requirements for analytical variability

**Marika Kamberi**
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Chromatographic methods are commonly used for the analysis of drug eluting stents (DESs). Accuracy and reliability of the analytical results are crucial for ensuring quality, safety and efficacy of DESs. Method validation is the process used to confirm that the analytical procedure employed for a specific test is suitable for its intended use. Results from method validation can be used to judge the quality, reliability and consistency of analytical results. Validation of analytical methods includes the identification of the performance parameters relevant for the given procedure, the definition of appropriate acceptance criteria and the appropriate design of the validation studies. Achieving an appropriate consideration of the analytical variability in assay procedures and setting acceptance criteria for analytical validations is however much more difficult than usually described. Criteria which are too wide may lead to unnecessary and incorrect out-of-specification (OOS) cases, resulting in bad reject decision for products. This study concentrates on analysis, through simulation, of the relation of method variability with specification limits for the total loaded dose of the active substance on the DES. The findings of this study point what levels of precision and accuracy are needed, in other words what is the magnitude of the allowable total error from all possible effects (both systematic and random) in an assay method in order to achieve the level of performance required for the methods applied routinely for the evaluation of the total loaded dose of DES as part of lot release/stability testing.

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## Ciprofloxacin residue and their impact on biomolecules in eggs of laying hens following oral administration

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The present study was designed to evaluate ciprofloxacin residue and their impact on some biomolecules (albumin, total protein and cholesterol) in eggs of laying hens after oral administration. For that purpose, One group (A) of laying hens (n = 20) were orally administered 10 mg⁄kg ciprofloxacin for five consecutive days. The second group (n = 10) was untreated controls. Eggs were collected from day one of treatment and up to 25 days after withdrawal of treatment. Egg white and yolk from each egg were separated, and ciprofloxacin residues and biomolecules were analyzed by high-performance liquid chromatography method with fluorescence detection and humalyzer having commercial assay kits respectively. Ciprofloxacin was detectable in egg white on the first day of treatment in higher concentrations (1755 µg⁄kg) while at lower concentrations (362 µg⁄kg) in egg yolk. In both medium, concentrations increased during five days treatment period. After withdrawal of treatment, eight days and fourteen days were required to deplete the drug residue below the established LOD in albumen and yolk respectively. On the other hand, cholesterol level increased while albumin and total protein level decreased during treatment period. All these biomolecules returns to their normal level at about seventeenth or eighteenth day from the day of treatment. In all cases, the differences in drug residue concentrations and biomolecules concentrations during treatment and post treatment in egg were found significant. Based on the time needed for residue to deplete below the LOD, we can estimate that, within twenty days of treatment period, egg contents could contain harmful residue which can deplete the nutritional value of egg and thus could cause severe disease for consumer as well whereas it is safe after that period.

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## Paper chromatography experiment report

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The technique helps in analyzing, identifying, purifying and quantifying unknown separable mixtures. The mobile phase is either a liquid or gas which moves the solvent through the stationary phase during the process. The stationary phase is a liquid or solid component that is fixed in a place for the procedure. Paper chromatography works majorly on capillary attractions. The capillary attraction which depends on adhesive and cohesive forces allows the mobile phase to move up the stationary phase due to created surface tension interaction from the forces. The major types are the paper chromatography, thin layer, gas chromatography, column chromatography, high performance liquid chromatography, paper chromatography and thin layer chromatography. There are several applications of paper chromatography and other main types of chromatography techniques. This technique is applicable in pharmaceutical industries, hospitals, forensic science, environmental science and manufacturing plants. This report describes the experiment conducted using paper chromatography to identify an unknown mixture. This will be done by comparing four known amino acids with the two unknown mixtures to identify the unknown mixtures. The experiment will also help to master the technique and analyze the movements made by both unknown mixtures and the known amino acids. Materials gloves, goggles, lab coat, filter paper, toothpick, ninhydrin solution, mixtures are to be identified. The laboratory procedures entail different steps that eventually lead to identification of the unknown mixtures. This procedure is divided majorly into stationary phase preparation, mobile phase preparation and chromatograph development. For the stationary phase preparation, the required markings are made on the paper for identification and creation of baseline. The baseline marks are the 1.7 cm from the shorter left edge and 1.0 cm from the bottom of longer edge. Known amino acid symbols are mark on the paper. Spotting of the known four amino acids and two unknown mixtures are then done using separate toothpicks which will help to prevent contamination. Mobile phase preparation was done by pouring 10 ml of solvent mixture in a 400 ml of Berzelius beaker while the chromatography development was done after the filter paper is already dried.

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## Simultaneous determination of three gliptins by HPLC-UV

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Diabetes is a disorder of the metabolism mostly seen as a combination of inherited or environmental factors and resulted with over increase of blood glucose level (hyperglycemia), the prevalence is increasing day by day in Turkey and in the world. Dipeptidyl peptidase-4 inhibitors (DPP-4s), gliptins, are a new class of drugs for oral hypoglycemics and used for the treatment of type 2 diabetes. Sitagliptin, vildagliptin and saxagliptin are the members of the gliptin drugs which are available in the market in Turkey. The advantages of gliptin drugs are differ from oral hypoglycemic drugs used in the treatment of type 2 diabetes like sulphonylureas, biguanids, α-glucosidase inhibitors and meglinids by oral implementation due to its non-peptide structure, and less side effects to the gastrointestinal system since the incretin receptors are not affected directly. Practical, selective and sensitive methods are demanded for the determination of sitagliptin, vildagliptin and saxagliptin from tablets alone and in combination with metformin and not many methods are available in the literature. A fast and simultaneous HPLC method was developed for the determination of these drugs in tablets and biologial fluids. Thermo Ultimate 3000 HPLC was used for the method development. Separation was achieved on a Gemini C18 (4.6x250 mm, 5u) HPLC column with a mobile phase combination of methanol:ortho phosphoric acid, in gradient elution. Analytes were detected both on 225 and 212 nm wavelengths. The developed method will be applied to biological samples and validated.

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## A metabolomics-based strategy to screening characteristic chemical markers for quality evaluation of *Flos Chrysanthemi Indici*

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Traditional Chinese Medicine (TCM), with notable effectiveness and few side effects, is gaining greater acceptance for preventing or healing a host of ailments worldwide. However, lack of well-established criteria to quality control of TCMs has been the biggest bottleneck for the modernization and globalization of TCMs. *Flos Chrysanthemi Indici*, anthotaxy of *Chrysanthemum indicum* L. has been used widely as a heat-clearing and detoxication herb because of its anti-inflammatory and anti-bacterial activity. *Flos Chrysanthemi Indici* has more than 100 chemical components, and their relative abundances are highly variable depending on geographical origins, climate, cultivar and other factors, which make great challenge for quality control. Over the past several decades, linarin is used as the single chemical marker for quality control of the *Flos Chrysanthemi Indici* according to the Chinese Pharmacopoeia. Despite possessing easy-operation characteristics, a single chemical marker cannot provide sufficient and convincing information for herbs which contain several 100 of chemical components. Considering the synergistic effects of multiple components on the effectiveness or therapeutic function of herbs, more chemical markers or active ingredients should be considered. In the present study, an integrated strategy of global chemical profiling using ultra performance liquid chromatography coupled with tandem quadrupole time-of-flight mass spectrometry and chemometric approach was applied to screening characteristic chemical markers for quality evaluation of *Flos Chrysanthemi Indici*. The result showed that a panel of key ingredients including chlorogenic acid, 3,5-dicaffeoylquinic acid, luteolin and linarin were considered as characteristic chemical markers, which showed even better quality control ability than fingerprint analysis, to guarantee the consistency of *Flos Chrysanthemi Indici*. This metabolomics-based approach is effective to screening characteristic chemical markers for quality evaluation of TCMs.

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## Development of an UPLC-MS based method for the simultaneous quantitation of phenolic components in honey using multi-walled carbon nanotubes as solid phase adsorbents

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An UPLC-MS method has been developed for the simultaneous separation, identification and determination of 22 phenolic constituents in honey from various floral sources from Yemen. Solid-phase extraction was used for extraction of the target phenolic constituents from honey samples, while multi-walled carbon nanotubes were used as solid phase adsorbent. The chromatographic separation of all phenolic constituents was performed on a BEH C18 column using a linear gradient elution with a binary mobile phase mixture of aqueous 0.1% formic acid and methanol. The quantitation was carried out in selected ion reaction monitoring acquisition mode. The total amount of phenolic acids, flavonoids and other phenols in each analyzed honey was found in the range of 338-3312, 122-5482, and 2.4-1342 μg/100 g of honey, respectively. 4-hydroxybenzoic acid was found to be the major phenolic acid. The main detected flavonoid was chrysin, while cinnamic acid was found to be the major other phenol compound. The regeneration of solid phase adsorbent to be reused and recovery results confirm that the proposed method could be potentially used for the routine analysis of phenolic constituents in honey extract.

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## Isolation of ulceroprotective cucurbitane type triterpenoids from *Cucumis melo* seeds

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Medicinal plants are the richest bio-resources of drugs in traditional medicinal systems, modern medicines, folk medicines, intermediate and chemicals entitled for synthetic drugs. Plants provide a source of inspiration for novel drug development as they contain a vast array of substances that treat chronic diseases. *Cucumis melo* seeds have been traditionally used for treating various health ailments. The main aim of our current study is to isolate cucurbitane-type triterpenoids from *Cucumis melo* seed extract and conduct anti-ulcerogenic activity of the isolated compound. Phytochemical investigations of methanolic seed extract of *Cucumis melo* was carried out which showed the presence of various important phytoconstituents. The main active constituents of *Cucumis melo* have shown a number of potent pharmacological activities. The isolation of Cucurbitane-type triterpenoids was carried out by column chromatography using methanolic seed extract of *Cucumis melo*. Mobile phase hexane and hexane-ethyl acetate (98:2) was used to run the column. TLC profiling was done simultaneously in an appropriate solvent system (hexane: ethyl acetate, 97:3). Various fractions were collected. The fractions with similar $R_f$ value were pooled together. Fractions giving single spot in the TLC were regarded as pure. The isolated compound showed positive result for Liebermann-Burchard test from which we can conclude that the isolated compound might be triterpenoid. The structure of the isolated compound was determined by IR, $^{1}$HNMR, $^{13}$CNMR techniques. The spectral analysis of the isolated compound showed following results: IR- it showed the peaks at 3383, 2976, 2814, 1721, 1465, 1123 cm-1 indicated the presence of alcoholic group.

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## Results on the determination of fatty acids in biological samples by applying gas chromatography

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Analytical method validation is the confirmation by examination and provision of objective evidence that certain specific requirements for intentional application are achieved. So validation of analytical quality assurance represents the first step in a laboratory. The fatty acids were determined by gas chromatography which involves the transformation of the fatty acids from the sample in methyl esters and separation of the components in the chromatographic column, their identification by comparison with the standard chromatograms. The method complies with standard SR CEN ISO/TS 17764 -2: 2008, used a Perkin Elmer-Clarus 500 chromatograph with capillary injection, high polarity stationary phase (BPX70: 60 mx0.25 mm inner diameter and 0.25 μm film thickness). The method was validated "in house", and used as methylated fatty acids standard solution Mix 37 Component FAME; 10 mg/mL, (CRM) soybean oil. We determined the following parameters: accuracy=98.72%, coefficient of variation of repeatability RSD=0.414%, detection limit LoD=0.002349 μg/mL, quantification limit LoQ=0.05683 μg/mL and recovery R=98.84%, according to SR EN ISO/CEI 17025: 2005, all values being within the admitted range: RSD: 80–120%, LoQ>LoD and 80<R>120%. The following concentrations of fatty acids were determined in samples of eggs, expressed per 100 g fat extracted from the yolk. Thus, α-linolenic acid has ranged between: 0.22±0.3 g (C) and 1.19±0.13 g/100 g fat, total omega-3 has values between: 1.37±0.09 g (C) 4.84±0.32 g (E1) and total omega-6 has values between: 24.61±1.38 g (C) and 20.91±1.08 g/100 g fat (E1).

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## Simultaneous determination of six active components in astragali radix and compound preparations by HPLC-DAD-ELSD

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A quantitative method, namely high-performance liquid chromatography coupled with diode array and evaporative light scattering detectors (HPLC-DAD-ELSD), was developed for simultaneous determination of six active ingredients in astragali radix from 10 different areas and 5 compound preparations of different dosage form. The DAD wavelength at 254 nm was selected for UV detection of three isoflavonoids (1: calycosin-7-O-β-D-glycoside, 2: ononin, 3: calycosin), while the drift tube temperature at 90°C and the nebulizing gas pressure at 1.5 bar were set for ELSD detection of three astragalosides (4: astragaloside IV, 5: astragaloside III and 6: astragaloside I). The conditions of this assay were optimized and the method was fully validated with respected to linear range, precision, repeatability and recovery. The developed method was successfully applied to determination six active ingredients in 15 samples and the results showed distinctive features of the contents of isoflavonoids and astragalosides. This rapid and reliable HPLC-DAD-ELSD method is suitable for quality evaluation of astragali radix and its compound preparations from different source and manufacturing procedure.

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## Direct determination of drugs by on-line column switching chromatography

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Analysis of drugs and metabolites in biological fluids is essential for bioanalysis. An optimal and effective sample preparation method plays the most important role since the depletion of the matrix in biological fluids is the biggest issue for a trouble-free analysis. It is impossible to inject the biofluid directly to the chromatographic system with traditional methods due to possible matrix effect and clogging issues. Liquid-liquid extraction (LLE), protein precipitation and solid-phase extraction (SPE) are the most common and offline/manual sample preparation methods to deplete macromolecules (i.e., proteins) present in the biological fluid prior to liquid chromatographic analysis. To speed-up the clean-up process, fully automated on-line techniques that combine sample preparation with separation could be a remarkable alternative. This could be achieved by the hyphenation of SPE with LC via a switching valve resulting online SPE-LC. This method allows direct repetitive injection of biological sample to a single SPE column. Use of Restricted Access Materials (RAM) as SPE-column packing materials enables the depletion of high molecular weight matrix while the small analyte molecules are retaining; this fractionation is mostly based on 2D chromatography combination of size exclusion chromatography with reversed phase chromatography. Coupling SPE column with LC leads to complete automation improving the analytical quality due to enhanced reproducibility, elimination of human errors and the possibility of multiple step elutions for clean-up of complex samples, reducing the cost and analysis time required.

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**Notes:**