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Anna Gajda et al., J Anal Bioanal Tech 2016, 7:5(Suppl)
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Oral fluid LC-MS/MS analysis as ante-mortem detection of Oxytetracycline in swine

Anna Gajda, Tomasz Bladek, Małgorzata Gbylik-Sikorska and **Andrzej Posyniak**
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The misuse and unreasonable administration of antibiotics in swine may lead to residues occurrence in products of animal origin. The main material for antibacterials determination is tissue of animals. Alternative for the post-mortem residues control is oral fluid analysis, as a non-invasive, ante-mortem method for the antibiotics detection. Oxytetracycline is one of the most widely used tetracycline in swine treatment. To prove and demonstrate the utility of oral fluid for the detection of this compound, oxytetracycline was administered by intramuscular injection, swine and oral fluid samples were analysed. For the measurement of oxytetracycline in oral fluid, a liquid chromatography-tandem mass spectrometry (LC-MS/MS) method was developed. The extraction was carried out with 10% trichloracetic acid. Samples were cleaned up by filtration using PVDF filters. Chromatographic separation was achieved on a Luna C18 analytical column using mobile phase consisting of acetonitrile and 0.1% formic acid in gradient mode. During validation, a good linearity was observed ($r>0.99$). The recoveries were in the range of 90-105%. The validation results showed good accuracy with a good RSD, less than 10.0% for repeatability and less than 15% under within-laboratory reproducibility. The procedure was satisfactory sensitive with detection limit LOD=2 µg/kg and limit of quantification LOQ=5 µg/kg. The presence of oxytetracycline in oral fluid up to 21 days after IM injection indicated that this medium had the potential to be an effective way to test the antibiotic residues in live animals.

Biography

Anna Gajda has completed her PhD in 2014 at Department of Pharmacology and Toxicology, NVRI, Poland. She is responsible for antimicrobial residues determination in food of animal origin by chromatographic techniques (LC-MS/MS, HPLC-FLD/UV). She participates in realization of National Residues Control Plan in Poland. She is engaged in development a new analytical methods for the detection of veterinary drugs in food products. She has published more than 23 papers in reputed international journals.

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Comparative analysis of RP-HPLC, turbidimetric and UV methods used for the determination of Cefepime hydrochloride in pharmaceuticals

Danilo Fernando Rodrigues and **Herida Regina Nunes Salgado**
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The prospect of new analytical methods, the improvement and validation of existing methods, bring numerous benefits to the pharmaceutical industry, for the purpose to save as much in costs as the analysis time. Cefepime hydrochloride (CEF), an antimicrobial agent β-lactam belonging to the group of fourth generation cephalosporins, is a semi-synthetic product which has activity against several Gram positive and Gram negative aerobic bacteria. The objective of this study was performed a statistical analysis of average contents of CEF obtained by reversed-phase high-performance liquid chromatography (RP-HPLC), microbiological assay (turbidimetric test) and UV method, using analysis of variance (ANOVA). The RP-HPLC method was performed on a C18 column (250 mmx4.6 mm) maintained at room temperature. The mobile phase consisted of water: absolute ethanol (45:55, v/v) at a flow rate of 0.5 mL min-1, using UV detection at 258 nm. For performing the turbidimetric assay, *Staphylococcus aureus* ATCC 6538 IAL 2082 was used as the test microorganism and the culture medium chosen was the Casoy broth. The control temperature was maintained at 35ºC±2.0ºC and incubated for four hours in shaker. The readings of the results were made in spectrophotometer at 530 nm. The UV method was realized using the equipment Spectrophotometer UV Shimadzu. The absorption was obtained at a wavelength of 258 nm. The results through ANOVA showed a significant difference between the methods proposed for the 5% significance level. Thus, the methods are not equivalent and should be used in conjunction in the quality control analysis of CEF.

Biography

Danilo Fernando Rodrigues has graduated in Pharmacy from the Centro Universitario de Votuporanga (2010). He has completed his Master's degree in Biotechnology from the Universidade Estadual Paulista-UNESP (2013). He is currently a Doctoral student (PhD) in Pharmaceutical Sciences Program at the School of Pharmaceutical Sciences of UNESP funded by FAPESP.

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Direct enantioenrichment of DL-mandelic acid by *in situ* immobilization of a general resolving agent on the magnetic multi wall carbon nanotube

Ghazale Daneshvar Tarigh and **Farzaneh Shemirani**
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L-threonine (L-thr) as a general chiral selector anchored on the surface of magnetic multi wall carbon nanotube (MMWCNT) was prepared using an *in situ* electrostatic adsorption and studied as a new magnetically chiral selector for the separation of chiral DL-mandelic acid (DL-MA) as a model sample. By varying the pH, DL-MA was adsorbed on the surface of magnetic chiral selector through hydrogen bonds. It was recognized that MMWCNT with chiral ligands on its surface simultaneously possessed both magnetic property and direct chiral recognition ability. The successful immobilization of L-thr onto the surface of MMWCNT was confirmed by infrared spectra (FT-IR), X-ray diffraction patterns (XRD) and transmission electon microscopy (TEM). The FT-IR and mass spectra of supernatant and elution solutions also confirmed the immobilization of L-thr onto the surface of MMWCNT. The analysis results of specific rotation, HPLC and ultraviolet-visible spectroscopy revealed that the L-thr-MMWCNT showed stronger complexation of (+)-enantiomer than (-)-enantiomer. The functional magnetic nanotubes were easily separated from the racemic solution using an external magnetic field which demonstrated its feasibility of recycling the adsorbent. All processes including *in situ* immobilization, enantioseparation (enantioenrichment) and magnetic separation were done by single process in a short time (only 10 min).

Biography

Ghazale Daneshvar Tarigh has completed her PhD in Analytical Chemistry from University of Tehran, Iran in 2015, BSc in Pure Chemistry at the University of Zanjan in 2003 and MSc under the direction of Prof. Yadollah Yamini at TMU and Prof. Ali Jabbari at KNTU in 2009. Her field of interest is the development of new extraction technologies with an emphasis on miniaturized sample preparation methods and separation techniques.

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Dipeptidyl peptidase IV (DPP IV) inhibitors from plant extracts by TLC bioautography

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TLC bioautography is an assay that combines chromatographic separation and *in situ* determination of biological activity. It has advantages over other screening methods for its ability to screen mixtures (a wide solubility range of compounds) simultaneously. In addition, it can reduce the cost and time of screening. TLC autobiography assays have been established for screening compounds for antimicrobials, antioxidants (free-radical scavengers) and enzyme inhibitors. In this presentation, the application of TLC bioautographic techniques on enzymatic reaction will be reviewed. An example of the development of a novel TLC-bioautographic method on detection of dipeptidyl peptidase IV (DPP IV) inhibitors from plant extracts will be described. The focus of the example will be on how reaction products via derivatization can facilitate such screening technology. In this case, the enzyme (DPP IV) hydrolyses substrate (Gly-Pro-p-nitroanilide) into p-nitroaniline (pNA), which diazotizes with sodium nitrite, and then reacts with N-(1-naphthyl) ethylenediamine dihydrochloride in turn to form a rose-red azo dye which provides a rose-red background on the TLC plates. The DPP IV inhibitors showed white spots on the background as they blocked enzymolysis of the substrate to produce pNA. The method was validated with respect to selectivity, sensitivity, linearity, precision, recovery, and stability after optimizing key parameters including plate type, time and temperature of incubation, concentration of substrate, enzyme and derivatization reagents, and absorption wavelength. One natural compound harmine was isolated and identified to be a potential DPP IV inhibitor in nine medicinal herbs by this method.

Biography

Lihua Gu is currently a PhD student at Shanghai University of Traditional Chinese Medicine. Her research interest includes the research and application of HPTLC, TLC-bioautographic methods and quality control standard in TCM. She has published more than 10 academic papers.

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Utility of capillary microsampling for rat pharmacokinetic studies: Comparison of tail-vein bleeds to jugular vein cannula sampling

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Serial sampling methods have been routinely used for rat pharmacokinetic (PK) studies. It is still common to take 100-250 μL of blood at each point of time when performing a PK study in rats using serial sampling. Recently, microsampling (<50 μL) techniques have been reported as an alternative process for collecting blood samples from rats. In this report, three proprietary compounds and two marketed drugs, fluoxetine and glipizide, were dosed orally into rats. Whole blood (and plasma) and capillary microsampling (CMS) samples were collected from jugular vein cannula (JVC) and tail-vein from the same rats. For the three proprietary compounds, the blood AUC as well as the blood concentration-time profile obtained from the tail vein was different from that obtained via JVC sampling. For fluoxetine, the blood AUC was not statistically different when comparing tail-vein sampling to JVC sampling, while the blood concentration-time profile that was obtained from the tail vein was different than the one obtained from JVC sampling. For both fluoxetine and glipizide, the blood concentration profiles obtained from CMS were equivalent to the blood concentration profiles obtained from the standard whole blood sampling, regardless of the sampling site. Thus, it is recommended that a consistent blood sampling method should be used for serial micro-sampling in discovery rat PK when testing new chemical entities. If the rat tail-vein sampling method is selected for PK screening, a bridging study on the lead compound is recommended to confirm that PK from JVC sampling is comparable to the tail vein sampling.

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Differential systemic exposure to galangin after oral and intravenous administration to rats

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Galangin (3,5,7-trihydroxyflavone) is present in high concentrations in *Alpinia officinarum* Hance. It shows multifaceted *in vitro* and *in vivo* biological activities. The number and position of hydroxyl groups in this molecule play an important role in these biological activities. In order to clarify the exposure of galangin after oral and intravenous administration to rats, two LC-MS/MS methods were developed and validated and successfully applied to analyze the parent drug molecules and aglycones liberated from plasma samples via β-glucuronidase hydrolysis. Our major findings were as follows: The routes of administration showed significant influences on the systemic exposure of galangin and its metabolites; galangin was preferentially glucuronidated after p.o. dosing but sulfated after i.v. medication; kaempferol conjugates were detected demonstrating that oxidation reaction occurred; however, both glucuronidation and sulfation were more efficient and; oral bioavailability of free parent galangin was very low. The results showed that systemic exposure to galangin and its metabolites was different in rat plasma between oral and intravenous administration. Further research is needed to characterize the structures of galangin conjugates and to evaluate the biological activities of these metabolites.

Biography

Junqing Zhang has completed his MD at 2013 from Nanjing University of Chinese Medicine. She is the Dean of the College of Pharmacy, Hainan Medical University. She has published more than 25 papers in reputed journals and has been serving as an Editorial Board Member of repute.

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Kyung Mi Hwang, J Anal Bioanal Tech 2016, 7:5(Suppl)
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Validation of ICP-OES and AAS method for the determination of magnesium in infant and follow-on formula

Kyung Mi Hwang, Hyeon Suk Ham, Doo Jin Lee, Hwa Jung Lee, Soo Jung Hu, Yoon Jung Kang, Yong Woon Shin, Myung Hee Moon, Jin Hwan Hong and **Hae Seong Yoon**
National Institute of Food and Drug Safety Evaluation, Korea

This study was conducted to establish the standard method for the contents of magnesium in infant and follow-on formula. Magnesium was extracted from 0.2 g certified reference material (NIST SRM 1849a) with 70% HNO3 and 7 ml by the MARS5 microwave lab station. Digests were then analyzed with Perkin-Elmer Optima 8300 inductively coupled plasma-optical emission spectrometer (ICP-OES) and Perkin-Elmer Analyst 700 atomic absorption spectrophotometer (AAS). Performance parameters were estimated for linearity, limits of detection (LOD) and limits of quantification (LOQ), accuracy and precision. The linearity of standard solution with correlation coefficient was higher than 0.999 in range of 0.05-1.5 mg/L. LOD were 0.01 mg/L and limits of quantification were 0.03 mg/L. Recovery of CRM were determined as approximately 100%. Currently, optimized method showed high accuracy (>90%) and precision was 0.2% relative standard deviation (RSD). The method was found to be suitable for routine quality control monitoring of infant and follow-on formulas.

Biography

Kyung Mi Hwang has completed her PhD from Pusan National University in Korea and Post-doctoral studies from Konkuk University, School of Bioinformatics. She is a Scientific Officer of Nutrition and Functional Food Research Team, National Institute of Food and Drug Safety Evaluation. She has published more than 10 papers in reputed journals.

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Assessment of Feng-Liao-Chang-Wei-Kang as a potential inducer of cytochrome P450 3A4 and pregnane X receptors

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Feng-Liao-Chang-Wei-Kang (FLCWK) is a traditional Chinese patent medicine that mainly consists of *Daphniphyllum calycinum* roots and *Polygonum hydropiper*. As a complex containing several types of flavonoids, FLCWK has potential effects on the drug metabolism enzyme P450 3A4(CYP3A4) and nuclear receptors. This study aims to explore the effects of FLCWK on CYP3A1 (CYP3A4's homolog in rats) in rats and to determine whether FLCWK could participate in the processes of hPXR- and hCAR-mediated transactivation of CYP3A4. The effects of FLCWK on CYP3A1 mRNA, protein expression and catalytic activity levels in Sprague-Dawley(SD) rat liver tissues were detected using real-time PCR, western blotting and high-performance liquid chromatography (HPLC) assays. The effects of hPXR and hCAR on CYP3A4 transcriptional activity were examined using luciferase reporter gene assays. Futher study of FLCWK on the *CYP3A4* gene expression mediated by PXR pathway was investigated by transient transfection of PXR siRNA. This study found that FLCWK could significantly increase the *CYP3A1* mRNA gene and protein expression levels and CYP3A1 activity in SD rats. In PXR-CYP3A4 co-transfected cells, FLCWK could significantly induce CYP3A4 luciferase activity mediated by PXR. PXR-knockdown (transfected with siPXR construct) decreased the CYP3A4 mRNA level than in the control cells transfected with corresponding vector. Taken together, these findings suggest that FLCWK could significantly up-regulate CYP3A4 levels via the PXR-mediated pathway. This effect should be taken into consideration to predict any potential drug-drug interactions between FLCWK and other co-administered drugs.

Biography

Ling Huang has completed her PhD from School of Pharmacy of Sun Yat-sen University. She has published more than 10 papers in reputed journals and has been serving as an Editorial Board Member of repute.

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Tahisa Marcela Pedroso et al., J Anal Bioanal Tech 2016, 7:5(Suppl)
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Analytical techniques for quantifying the Ertapenem sodium with a look at green chemistry

Tahisa Marcela Pedroso[1], **Herida Regina Nunes Salgado**[1] and **Ana Claudia Dantas Medeiros**[2]
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[2]Paraiba State University, Brazil

Ertapenem sodium is the polar and ionizable compound. Thus, the most suitable for such drug is to develop a method for Hydrophilic Interaction Chromatography (HILIC). On the other hand, the RP-HPLC is considered as the analysis technique used for quantification of substances and new column are often introduced for compounds separation. Two new analytical methods have been developed. The RP-HPLC method was performed on a Waters LC system, with an Agilent™ Zorbax Bonus-RP (4.6×150 mm, 5 µm) column and the mobile phase was Water:Ethanol 80:20 v/v. While the HILIC method was performed on an ultra-high performance liquid chromatography Shimadzu with stationary phase PhenomenexTM HILIC Kinetex (4.6×100 mm, 2.6 µm) column and the mobile phase was Acetonitrile:Water 88:12 v/v. In both methods, 0.1% formic acid was added in mobile phase and was performed isocratically. The validation parameters were in accordance with ICH specifications and demonstrated accuracy, precision, selectivity, robustness and linearity. Analytical curves was constructed and was evaluated by ANOVA (y=23043×-38525, R2=0.9999 RP-HPLC and y=29928×-547879, R2=0.9994 HILIC). The robustness was evaluated by making parameters small alterations simultaneous and was evaluated by the Plackett-Burman. The results show that the method validity is maintained. The methods here described have economic advantage as well as features an eco-friendly focus. This work was developed with environmental conscience, always looking to minimize the possible generated organic waste, which is of utmost importance to the environment.

Biography

Tahisa Marcela Pedroso has completed her Pharmaceutical honors degree with merit from the Programa Universidade Para Todos (PROUNI) rated best pharmacy student at the Centro Universitário Central Paulista, São Carlos, Brazil in 2009. She has completed her Master's degree in Pharmaceutical Sciences from the Universidade Estadual Paulista, Brazil. She is currently conducting research with the antimicrobial Ertapenem sodium for the development of PhD research at the Faculdade de Ciências Farmacêuticas, Brazil and PhD exchange student at the University of Ghent-Ugent, Belgium.

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Tomasz Bladek et al., J Anal Bioanal Tech 2016, 7:5(Suppl)
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Multi-class method for the determination of antibacterials in honey by liquid chromatography-tandem mass spectrometry

Tomasz Bladek, Anna Gajda, Ewelina Nowacka-Kozak and **Andrzej Posyniak**
National Veterinary Research Institute, Poland

A liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS) method for determination and quantification of sulfonamides, tetracyclines, macrolides, fluoroquinolones, streptomycin, lincomycin and trimethoprim resideues in honey has been developed. Prior to instrumental analysis, sample preparation technique involved extraction with ammonium acetate buffer followed by solid-phase extraction cleanup using polymeric reversed phase sorbent. The chromatographic separation was performed on C18 column with gradient elution mode. Mass spectral acquisitions were performed under selective multiple reaction monitoring mode by triple quadrupole mass spectrometry detector. The method has been characterized by providing the parameters of precision, accuracy, specificity, decision limit (CCα) and detection capability (CCβ) according to Commission Decision 2002/657/EC. The calibration curves were linear in the range of 5-100 μg/kg. The recoveries ranged from 86-104%. The repeatability was below 11.3% and within-laboratory reproducibility was lower than 16.1%. Calculated CCα and CCβ values ranged from 5.8-7.6 μg/kg and from 6.9-9.9 μg/kg, respectively. The developed method fulfils the criteria for confirmatory methods.

Biography

Tomasz Bladek is a Research Assistant at the Department of Pharmacology and Toxicology, National Veterinary Research Institute in Pulawy, Poland. His major research interests include the development of new analytical methods for determination of antibiotic residues in food matrices such as muscle, milk, kidney, liver, eggs and honey. He has published several IF papers on antibiotic residues and has been serving as a Reviewer for analytical journals.

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Wei Wang, J Anal Bioanal Tech 2016, 7:5(Suppl)
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Piezoelectric positioning system technology for modulation of electrochemical imaging detection device

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The piezoelectric ceramic positioning system is considered to be the high-precision positioning device in the electrochemical imaging detector, which can achieve the nanometer scale displacement resolution. This paper describes in detail the principle of output displacement measuring for piezoelectric ceramic by the resistance strain gauge. In addition, by employing the self-built drive circuit and the displacement detecting circuit, integrated piezoelectric ceramic resistance strain gauge sensor has been investigated under both open-loop and closed-loop control, respectively. Under the closed-loop control testing, with a classic PID algorithm was applied. Test results show that, the piezoelectric ceramics in open-loop control exhibits disadvantages of serious non-linearity, hysteresis and creep resistance. However, these issues have been resolved under closed-loop control.

Biography

Wei Wang is an Associate Professor, Master of Electronic Engineering, graduated from Jilin University. He has designed and developed a variety of analytical detection equipments and obtained progress Award of Jilin Province Science and Technology in nano-structured composites and analysis instrumentation aspects in 2008.

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Yi Ding et al., J Anal Bioanal Tech 2016, 7:5(Suppl)
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Method development of carbohydrate profile for Abbott nutrional products using HPAEC/PAD

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The purpose of this research method is to determine sugar profile (mono- and disaccharides) of Abbott nutritional products in fulfillment of the "Sugar" label claim. In this method, the sugars (galactose, glucose, fructose, sucrose, lactose and maltose) were extracted from product primarily by dilution in water. The sugars were analyzed via high performance anion exchange coupled with pulsed amperometric detection (HPAEC/PAD). The HPAEC/PAD Dionex ICS5000 system was equipped with a triple pulsed electrochemical cell (ED) with a pH reference electrode (Ag/AgCl), a gold working electrode, and a borate trap in tandem with a PA1 analytical column. Quantitation was accomplished using a six level quadratic curve and peak area. Sugars were analyzed by HPAEC/PAD via a new gradient elution program to ensure adequate resolution of the analytes of interest and known interferences from AN commodities. Method specificity was successfully evaluated by comparing the retention times of a sugar standard mix vs. commonly used commodities and ingredients. Method accuracy and precision were evaluated by comparing results generated for AN research samples using this research method relative to third party laboratory results.

Biography

Yi Ding has completed his PhD in Chemistry from Division of Chemistry and Biological Chemistry, Nanyang Technological University, Singapore in 2010. He is currently a Senior Analytical Scientist of Abbott Nutrition Research and Development, Singapore.

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Young Jin Koh et al., J Anal Bioanal Tech 2016, 7:5(Suppl)
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Analysis of Raman scattering signals for chemicals by using deep UV laser

Young Jin Koh, Young-Su Jeong, Jae Hwan Lee, Juno Lee and **Yeon Chul Ha**
Agency for Defense Development, South Korea

A Raman spectroscopy is a spectroscopic technique based on in elastically scattered light and is widely used for the analysis of materials. The Raman spectrometer is very effective in detecting liquid chemicals on surfaces on the move. Due to advanced ultraviolet light source technology, the Raman spectrometer based on ultraviolet laser has been actively developed, recently. In case of using below 250 nm wavelength as a deep UV light source, it is easy to measure the Raman spectra by spectrally separating the Raman and the fluorescence area. In this research, we describe about analysis of Raman spectra for chemicals on various deep UV wavelengths and compared with Raman spectra according to laser wavelengths.

Biography

Young Jin Koh has completed her graduation from Department of Chemistry, Duksung Women's University and Master's degree from Department of Analytical Chemistry, Seoul National University in South Korea. Presently, she is a Researcher at Agency for Defense Development in South Korea.

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Measurement of Raman spectra for various real surfaces by using a 248 nm mobile Raman spectrometer

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Raman scattering occurs when light interacts with a molecule. Raman signals and their intensities are dependent on the size, shape, and bond strength of the molecule creating, thus, a distinct spectral signature for chemicals. A Raman Agent Monitoring System (RAMS) is a technique based on Raman scattering to provide stand-off detection and is made using an excimer laser operating at 248 nm. The RAMS identify toxic chemicals on the ground surfaces in moving. In this research, we measured and analyzed Raman spectra for various real surfaces in the Korean Peninsula with a RAMS. There are no significant characteristics in Raman signals of these surfaces. These results shows that RAMS can detect on ground surfaces in moving and in real time.

Biography

Young Jin Koh has completed her graduation from Department of Chemistry, Duksung Women's University and Master's degree from Department of Analytical Chemistry, Seoul National University in South Korea. Presently, she is a Researcher at Agency for Defense Development in South Korea.

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Rapid detection of aerosolized *Bacillus* spore particles by direct *in situ* analysis of MALDI-TOF mass spectrometry

Young-Su Jeong, Eugene Chong and **Jong Min Lee**
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Accurate and rapid analytical methods are essential for the detection and identification of biological warfare agents (BWA) as well as pathogens. Although various studies have investigated the uses of a matrix-assisted laser desorption/ionization time-of-flight (MALDI-TOF) mass spectrometry (MS) for bacterial classification, only a few studies have examined the applicability of method for the identification of BWAs. This study aimed to generate, collect and analyze *Bacillus* spore aerosol particles of 2-10 μm, the optimal size of a BWA. In this process, we developed an apparatus to directly deposit *B. globigii* aerosols on MALDI target plate wells for rapid MALDI-TOF analysis. *Bacillus* spore aerosol particles of 2-10 μm were rapidly analyzed using direct *in situ* MALDI-TOF MS without any pretreatment processes. The mass spectra of aerosolized Bacillus spore particles were successfully detected. For real-time detection and identification, a mass spectral database of *Bacillus* spores was constructed, and an algorithm was developed and applied. Bacillus spore particles were rapidly detected and identified by MS, which can be used for the detection and inspection of BWAs such as *B. anthracis* spore in the battle field.

Biography

Young-Su Jeong has completed his PhD from Korea Advanced Institute for Science and Technology (KAIST) in 2012. He is a Senior Researcher of Agency for Defense Development (ADD), ROK. He has published more than 18 papers in reputed journals.

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Raman spectral characteristics of toxic chemicals analyzed by a 248 nm mobile Raman spectrometer

Young-Su Jeong, Young Jin Ko, Jae Hwan Lee, Yeon Cheol Ha and **Young Il Kang**
Agency for Defense Development, South Korea

A Raman Agent Monitoring System (RAMS) is a technique which exploits Raman scattering to provide stand-off detection and identification of toxic chemicals such as chemical warfare agents and toxic industrial chemicals deposited on the ground surfaces. The RAMS collects counts on photons emitted by an agent on a surface through a telescope and displays the result on a spectrograph. Raman scattering occurs when light interacts with a molecule. The molecule vibrates and scatters wavelength shifted light. The wavelength shifts and their associated intensities are dependent on the size, shape, and bond strength of the molecule creating, thus, a distinct spectral signature for the associated chemical. In this research, we measured toxic chemicals with a RAMS, a 248 nm mobile Raman spectrometer, and analyzed Raman spectral signatures of Raman signals of that chemicals. These results can assist in selecting unique molecular markers for each chemical to allow real-time detection and identification of them in ground environments in moving.

Biography

Young-Su Jeong has completed his PhD from Korea Advanced Institute for Science and Technology (KAIST) in 2012. He is a Senior Researcher of Agency for Defense Development (ADD), ROK. He has published more than 18 papers in reputed journals.

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Characterization of sputter-deposited w-44cr-35al alloys and their corrosion behavior in aggressive media

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The synergistic effect of the simultaneous additions of chromium and aluminum in the sputter-deposited amorphous or/and nanocrystalline W-44Cr-35Al alloys is studied in 1M HCl, 0.5M NaCl and 1M NaOH solutions open to air at 25 ±1°C using immersion tests, electrochemical measurements and surface morphological analyses.

Aluminum and its alloy have been extensively used in industry because of their useful properties such as low density, good appearance and corrosion resistance. However they are susceptible to pitting corrosion when exposed to various acidic, alkaline and neutral media. The sputtered-deposited alloys, either amorphous or Nano crystalline single phase solid solutions are chemically more homogeneous and conventionally processed alloys and hence are interesting to develop the corrosion resistance materials during last few decades. It has been reported that the chemically homogeneous single phase nature of amorphous and Nano crystalline alloys are responsible for high corrosion resistance owing to the formation of uniform protective passive films that are able to separate the bulk alloy from aggressive solutions.

The aluminum alloy was prepared by sputtered deposition technique. In three component aluminum alloy consisting Al, Cr, and W the higher corrosion resistance depends on the percentage composition of Tungsten. During X-Ray Diffraction, the atoms are nanocrystalline. The Scanning Electron Microscopy/EDS shows the atoms are distributed homogeneously.

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Hygroscopic gravimetric quantitative chemical analysis

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Hygroscopic gravimetric method is based on the determination of the absorbed water vapor mass by a dry sample after holding at a specific relative humidity and temperature. Theoretical bases of the methods are considered. Mass value of the sorbed water (water content) is proportional to the concentration of a heterogeneous hygroscopic impurity. Other heterogeneous hygroscopic impurities affect proportionality while water-insoluble and homogeneous impurities have no influence on water content and proportionality. Equilibrium moisture sorption of the sample due to the total effect of all the soluble impurities allows determination of the main component content in a substance. The method can be experimentally based on, holding the sample in exsiccator, isopiestic device or in dynamic vapor sorption devices. The range of the recommended relative humidity values lies within 1-5% relative humidity below the hygroscopic point (RH0) of the main component. The method is applied to determine the amorphous impurity content in a crystalline product. Application areas of the method are outlined in the paper. Attention is paid to the fact that not only water but also organic solvents can act as sorbed vapors; the objects under analysis can be both solid and gaseous substances. Employing simple experimental technique the method provides for determination of the content of the main component (from 99 to 99.999 %) and soluble impurities (from 1 to 0.001%).

Biography

Anatoly G Tereshchenko was graduated from the Tomsk Polytechnic Institute, Chemical Engineering Department in 1969. He has obtained his PhD in 1974. Presently, he is the Head of the Information Technology Laboratory at the Institute of High Technology Physics, National Research Tomsk Polytechnic University. His scientific interests include the development of the laboratory information management system “Kimia-Analitik” (Chemistry -Analysis), quality control of the quantitative chemical analysis results within the laboratory, effect of impurities on soluble crystalline solids’ hygroscopicity, caking behavior of soluble solids and isopiestic research method. He is the author of 150 articles and 2 monographs.

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Notes:

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Determination of zoledronic acid and its related substances by high performance liquid chromatography with evaporative light scattering detection

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Zoledronic acid (ZOL), a bisphosphonic acid, is an inhibitor of osteoclastic bone resorption. It is a potent inhibitor of osteoclastic bone resorption and is clinically used for the treatment of malignant and benign bone diseases, e.g., osteoporosis. ZOL is designated chemically as (1-hydroxy-2-imidazole-1-yl-phosphonoethyl) phosphonic acid monohydrate. Due to the chemical nature of ZOL, its chromatographic separation is challenging. Since bisphosphonates contain two phosphoric acid groups, they are ionic and highly polar. In this study, a method has been developed and validated for analysis of ZOL and its related substances namely imidazole-1-yl acetic acid, phosphate, phosphite and the other possible degradation products in a single analysis by ion-pair reversed phase high performance liquid chromatography with evaporative light scattering detection (ELSD). The method validation was performed and fulfilled to ICH guidelines. The calibration plot was linear in the range 4.0 mg/mL to 6.0 mg/mL for ZOL and 6.25-100 μg/mL for related substances. The method has been demonstrated to be sensitive, with an LOQ of 1.7 μg per mL, 1.5 μg per mL, 2.5 μg per mL and 1.5 μg per mL for ZOL, imidazole 1-yl acetic acid, phosphate and phosphite, respectively. The method was rapid, linear, accurate, and reproducible. The proposed method can be used to evaluate the quality of regular samples.

Biography

Saglik Aslan Serap is a Professor in the Department of Analytical Chemistry. She is the Head of Analytical Chemistry Department, Faculty of Pharmacy in Istanbul University since 2011.

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Current trends in gas chromatography and mass spectrometry instrumentation: A hyphenated technology for analytical and bioanalytical techniques

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The advancement of hyphenated instrumentation from a variety of scientists working in the public and private sectors of research and development in the form of gas chromatography (GC) being coupled to mass spectrometry (MS) has continued to develop into a powerful multi-dimensional hyphenated instrumental technology for the use in a wide assortment of analytical and bioanalytical techniques. Examples of this development can be seen in the analysis of drugs, metabolites, pesticides, chemical warfare agents, food ingredients, medications, fuels and etc, and/or in the main category of volatile and semi-volatile organic compound analysis in fields such as forensic, toxicology, environment, defense, food and beverage, pharmaceutical, petrochemical and etc. This advancement in hyphenated GC-MS instrumentation was initially and still is driven by the need for a more comprehensive analytical and bioanalytical technique that can accurately and precisely discriminate targeted and untargeted analytes from higher complexity sample mixtures in a sensitive and selective way from within a concise window of time. With this in mind, this presentation which is based upon a recent editorial, briefly attempts to highlight some of the current trends in hyphenated GC-MS instrumentation available today and their respective contributions to the field of analytical and bioanalytical techniques.

Biography

Wes E Steiner has earned his PhD in Analytical Chemistry with focus on the development and use of analytical instrumentation to explore a variety of topics involving health, environment, agriculture and defense. Presently his research group is interested in applications that are focused on the qualitative discovery and quantitative directed analysis of bio-markers that can be correlated to a specific disease trait, state and/or rate. He has more than 30 publications to his credit.

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Electrodynamics and Electrochemistry of Inhomogeneous (Laminated, Angular) Structures

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The consistent physicomathematical model of propagation of an electromagnetic wave in a heterogeneous medium has been constructed using the generalized wave equation and the Dirichlet theorem. Twelve conditions at the interfaces of adjacent media were obtained and justified without using a surface charge and surface current in explicit form. The conditions are fulfilled automatically in each section of the heterogeneous medium and are conjugated, which made it possible to use through-counting schemes for calculations. For the first time the effect of concentration of "medium-frequency" waves with a length of the order of centimetre at the fractures and wedges of domains of size 1 - 3 μm has been established. Numerical calculations of the total electromagnetic energy on the wedges of domains were obtained. It is shown that the energy density in the region of wedges is maximum.

The results of these calculations are of special importance for medicine, in particular, when microwaves are used in the therapy of various diseases. For a small, on the average, permissible level of electromagnetic irradiation, the concentration, focus of electromagnetic energy in internal angular structures of a human body (cells, membranes, neurons, interlacements of vessels, etc) is possible.

We have constructed a consistent physicomathematical model of interaction of nonstationary electric and thermal fields in a layered medium with allowance for mass transfer. The model is based on the methods of thermodynamics and on the equations of an electromagnetic field and is formulated without explicit separation of the charge carriers and the charge of an electric double layer. We have obtained the relations for the electric-field strength and the temperature, which take into account the equality of the total currents and the energy fluxes, to describe the electric and thermal phenomena in layered media where the thickness of the electric double layer is small compared to the dimensions of the object under study with allowance for mass transfer.

We have modeled numerically the heating of an electrochemical cell with allowance for the influence of the electric double layer at the metal-electrolyte interface and mass transfer. The calculation results are in satisfactory agreement with experimental data.

Biography

Total years of work experience: researches, including investigations of transfer processes in porous media, electrolites, membranes, distributions of electromagnetic waves in inhomogeneius (laminated, angular) structures, development of mathematical and computational models. 1972-2003 – Junior and Seniour researcher at the A.V. Luikov Heat and Mass Transfer Institute, National Academy of Sciences of Belarus; 2003- at present time – Leading Researcher at A.V. Luikov Heat and Mass Transfer Institute, National Academy of Sciences of Belarus; 2005 - at present time – associate professor of Mathematical Physics Chair at Belarusian State University, Belarus. Study Course: 'Mechanics and electrodynamics of heterogeneous media'. Member of Belarusian Physics society. Member of Supreme Attestation Committee of Belarus from 2011. Academic awards and grants – Prizewinner of Koptug's Award of Siberian Branch of Russian Academy of Sciences (2013). Grinchik N. N. published more than 100 papers, has 40 inventions.

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PALMS: Parallel targeted/untargeted metabolome screening platform

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The human body contains about 10 microbes per each human cell if you do not consider red blood cells which are lacking DNA. This means microbes significantly impact the chemistry in and on our body. Despite the role of microbial chemistry on human health, we know very little about its chemistry, let alone quantitatively. One of the analytical methods that are sensitive enough to measure microbial chemistry can be mass spectroscopy (MS). Here we introduce PALMS platform that is capable of analyzing full sample profile using high accuracy Orbitrap instrument and quantify set of targeted metabolites using triple quadrupole in a single LC run. Several hundred of fecal swabs samples from the American Gut project were tested and then people were swabbed at several hundred locations for the creation of 3D quantitative maps. The system setup comprises of triple quadrupole (qqq) and Orbitrap mass spectrometers connected in parallel to a single UHPLC system. Liquid connection was split into equal ratio and directed into two MS which are equipped with identical HESI ion sources; tubing length maintained equal in order to avoid any retention time (RT) differences between two MS. The data acquisition was triggered by UHPLC system by mean of contact closure signal that is sent to both MS simultaneously. Data captured by Q Exactive recorder in untargeted profiling mode with high resolution while quantitative data on defined targets were acquired by TSQ. We first evaluated our system by screening of several hundreds of fecal swabs extracts received from American Gut project. For targeted quantification we used mix of 200 standards containing microbial primary and secondary metabolites. We evaluated a distribution of the standards in fecal samples by constructing a calibration curve with triple quadrupole data. Our results showed that even in such high background as fecal matter we can quantify metabolites with relatively low error rate. By feeding mass list to triple quadrupole we managed to achieve pictogram sensitivity at the same time acquiring a full molecular profile using Orbitrap. By analyzing untargeted data we were able to putatively identify thousands of molecules using molecular networking while quantification was performed for selected microbial metabolites. Because in molecular networking structurally similar molecules clustered together we were able to relatively quantify analogs of the standards by extrapolating their concentrations. We then used the platform for screening the microbial metabolites reside on human body. We constructed 3D chemical composition maps of human skin surface. Highly accurate, untargeted data allowed for identification of some key microbial metabolites that are distributed on the skin in the specific locations such as armpit and groin area. We then relatively quantified the metabolites as some of our standards clustered with molecules of interest, which concentration was accurately measured by triple quadrupole. Determining the quantitative information for key microbial metabolites grant us the possibility to calculate the amount of the microbial cells that are primary producers and potentially assess the impact of them on our health.

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Sub-2 µm C18 bound silica monolith particles as HPLC stationary phase of high separation efficiency

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Sub-2 µm porous silica monolith particles have been prepared successfully by sol-gel process followed by grinding and calcinations at 550 oC. The particles were derivatized with a C18 ligand followed by end-capping with a mixture of hexamethyldisilazane (HMDS) and trimethylchlorosilane (TMCS). The resultant phase was packed in glass lined stainless steel microcolumn is much better than that of commercial C-18 phase. This phase has shown some encouraging possibility for fast analysis when packed in a short column. This study offers a promising vision towards commercialization of chromatographic phases based on silica monolith particles.

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Determination of parabens in cosmetic samples using fabric phase sorptive extraction (FPSE) by high-performance liquid chromatography and ultraviolet detection

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A simple, fast and sensitive analytical method using fabric phase sorptive extraction (FPSE) followed by high performance liquid chromatography ultraviolet detection (HPLC-UV) has been developed and validated for the extraction of five parabens namely ethyl, methyl, propyl, butyl and benzyl paraben. Parabens are esters of p-hydroxybenzoic acid which are commonly used as preservatives in cosmetic products. Various factors affecting the performance of FPSE technique such as extraction time, eluting solvent, elution time and pH of the sample matrix were optimized. Determination was performed using reversed stationary phase C18 column with wavelength 254 nm. Separation was performed using mobile phase acetonitrile: water (63:37; v/v) at an isocratic flow rate of 1.0 mL/min. The calibration curves of the target analytes were prepared with good correlation coefficient values ($R2>0.992$). The limit of detection (LOD) values range from 0.192-0.225 ng/mL and the developed method was applied successfully for the analysis of parabens in various cosmetic samples such as rose water, deodorant, serum and cream with extraction recoveries found in between 88% to 114%.

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First-principles study of siloxene and germoxene: Stable conformations, electronic properties and defects

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Two-dimensional materials can be used in a variety of bioanalytical techniques. Interest in two-dimensional (2D) forms of Si and Ge has surged recently, with a focus on silicene and germanene, the Si- and Ge-based analogues of graphene, as well as their derivatives. Siloxene and germoxene are 2D materials made of honeycomb Si and Ge backbone sheets that are decorated with H atoms and OH groups. This work uses first-principles calculations to probe the properties of their various conformations. It is shown that the most stable siloxene (and germoxene) polymorph is the so-called washboard structure, and not the chair geometry assumed in previous studies. The stability of the washboard configuration relates to the formation of a network of hydrogen bonds between its hydroxyl groups. It is also found with hybrid functional calculations that siloxene and germoxene are wide band-gap semiconductors with gap values of 3.20 eV and 2.64 eV, respectively. Finally, we show that H and OH vacancies introduce spin polarization in these 2D materials and have a tendency to pair up in stable di-vacancies

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DNA binding study of Co (III) complexes of hydroxamic acid and 1, 10-phenanthroline complex

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A large number of transition metal complexes have been used as cleavage agents of DNA and also for novel potential DNA-targeted antitumor drugs. This is essential for further expected applications in many areas like biological gene regulators or cancer chemotherapeutic agents. The identification of metal complex–DNA interaction is of fundamental importance to understanding the molecular basis of therapeutic activity. Metal complexes are well known to accelerate the drug action and the efficiency of a therapeutic agent can often be enhanced upon coordination with metal ions. The pharmacological activity has also been found to be highly dependent on the nature of the metal ion and the donor sequence of the ligands, as different ligands exhibit different biological properties. In the present work a new Co (III) complexes of mixed ligands, 1, 10-phenenthroline (phen) and hydroxamic acid (L_1 = AHA (acetohydroxamic acid), and L_2 = BHA (benzohydroxamic acid) were synthesised and characterized by NMR, IR, UV-visible and elemental analysis. In the complexes, $[Co(phen)_2L_n]$ ($L_n = L_1, L_2$), the metal ion is coordinated by six atoms, two oxygen atom from hydroxamic acid and 4N atom from co-ligand 1, 10-phenenthroline to form octahedral Co(III) complexes. The interaction of these complexes with calf thymus DNA has been investigated by absorption spectroscopy and fluorescence spectroscopy.

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The effect of pH on the partitioning of polychlorinated biphenyls (PCBs) between sediment grain sizes and water

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In natural environmental media organic pollutants (OPs) undergo partitioning with particulate matter (PM) and dissolved organic carbon (DOC) in water, and with surface sites on the sediment particle material with which the water is in contact. The sorption of organic pollutants within the environmental media is one of the significant factors affecting (in most cases controlling) the distribution, transportation and fate of the pollutant as well as the remediation of polluted water within the aquatic ecosystem. The aim of this research was to investigate the effects of pH on the phase partitioning of organic pollutants such as PCB congeners between the aqueous phase and various modeled sediment grain sizes. A model sample of sediment was sieved into five grain sizes, <75 µm, 100 µm, 200 µm, 300 µm and 425 µm, respectively. The surface areas as well as pore volume distributions of the different sediment grain sizes were determined using BET-adsorption/desorption isotherms. The surface areas as shown by BET-adsorption isotherms were between 0.688-14.361 m2/g (425 µm-75 µm). The organic carbon content was determined using the Walkley Black method. The organic carbon content was found to be greater on the smaller sediment grain size of 75 µm and least in the 425 µm grain size (2.94 to 11.43 %). Laboratory batch experiments were used to evaluate the adsorption/desorption as well as the adsorption ratio of the soil/sediment (K_d) within the different sediment grain sizes. The results showed that soil grain size of <75 µm showed the most adsorption of PCB congeners, in the range of 79.90-89.97 % while a sediment grain size of 300 µm showed lowest adsorption between 32.60-64.14 %. This could be attributed to the fact that sediment grain size (75 µm,) with high surface area adsorbed more of the analytes compared to other sediment grain sizes. Also, the higher organic content associated with the 75 µm grain size further allows for more partitioning of the organic pollutant to the sediment. The effect of pH on the sorption capacity of the sediment grain sizes was also studied. A pH of 6.5 showed the highest percentage adsorption. This was due to the non-polar PCBs preferring to partition to the sediment at near neutral pH.

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Development, validation and application of RP-HPLC method: Simultaneous determination of antihistamine and preservatives with Paracetamol in liquid formulations and human serum

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In this article we described the development and validation of stability indicating, accurate, precise and simple ion-pairing RP-HPLC method for simultaneous determination of paracetamol and cetirizine HCl along with preservatives i.e., propylparaben and methylparaben in pharmaceutical dosage forms of oral solution and in serum. Acetonitrile: buffer: sulfuric Acid (45:55:0.3 v/v/v) was the mobile phase at flow rate 1.0 mL min-1 using a Hibar® Lichrosorb® C18 column and monitored at wavelength of 230 nm. The averages of the absolute and relative recoveries were found to be 99.3%, 99.5%, 99.8% and 98.7% with correlation coefficient of 0.9977, 0.9998, 0.9984, and 0.9997 for cetirizine HCl, paracetamol, methylparaben and propylparaben, respectively. The limit of quantification and limit of detection were in range of 0.3-2.7 ng mL^{-1} and 0.1-0.8 ng mL^{-1}, respectively. Under stress conditions of acidic, basic, oxidative, and thermal degradation, maximum degradation was observed in basic and oxidative stress where a significant impact was observed, while all drugs were found almost stable in the other conditions. The developed method was validated in accordance to ICH and AOAC guidelines. The proposed method was successfully applied to quantify the amount of paracetamol, cetirizine HCl and two most common microbial preservatives in bulk, dosage form and physiological fluid.

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Application of linear voltammetry in the determination of vitamin C in peppers

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The main objective of this research was to compare the content of ascorbic acid peppers (*Capsicum annuum*), in state of ripening green and red of the same variety. Pepper extract was obtained by crushing the fruit in a porcelain mortar, the liquid obtained was filtered on filter paper. For determining the content of ascorbic acid is used linear sweep voltammetry using three electrodes; as working electrode a platinum wire of high purity, glassy carbon as working electrode and reference electrode Ag/AgCl/KCl. A pattern of ascorbic acid and a solution of HNO_3 and 0.1M $NaNO_3$ as supporting electrolyte were used for the calibration curve. The results indicate that, green pepper has an average concentration of 1680 ppm of ascorbic acid, and red pepper reached a value of 2283 ppm. These results are superior to those obtained by Chaves et al., in pepper by HPLC. In this case, values of 1000 ppm to 1500 ppm for green pepper and red pepper were found respectively.

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Quantum chemistry aided FTIR spectroscopy to distinguish internal and external Brønsted acidic sites of zeolite nano-crystals

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Microporous nano-crystals of zeolites are key ingredients in more than 50% of heterogeneous catalysts, which contribute to manufacturing the majority of chemical products. Their Brønsted acidic hydroxyl groups (BA-OH) play pivotal role in many reactions. The presence of BA-OH sites on the crystallite surface bears of special interest, due to diffusion limitations inside of their micropores. Yet distinction of the internal and external BA-OH sites has been ambiguous. Here we show that clear distinction can be made by Fourier Transform Infrared (FTIR) spectroscopy when both diffuse reflectance (DR) and transmission (TR) sampling techniques are employed. Different laboratories use these techniques interchangeably for characterizing solids. To our surprise, we observed very different BA-OH spectra on some zeolites when measured by these two methods. Since physical and chemical differences do not generate such large spectral deviations, we conjectured that emphasized vibrational intensities of the surface and bulk BA-OH groups cause the differences when DR or TR technique is used, respectively. To prove our point we performed selective pyridine adsorption experiments on Chabazite (H-CHA) and H-SAPO-34 zeolites and also computed the density functional theory (DFT) based FTIR spectra of their four geometrically different BA-OH groups in the bulk and on the surface, which confirmed the conjectured vibrational differences. These zeolites are key ingredients of new catalysts for diesel-automobile exhaust control and for the methanol to olefin, MTO, technology.

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Direct-writing of paper based conductive track using silver nano-ink for eletrochemical application

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To the best of our knowledge, to date there is no report for the synthesis of silver nanoparticles capped with oleylamine (AgNPs/OLA) and then use of NPs in the preparation of paper based conductive track for electroanalytical application. The synthesized OLA capped AgNPs was characterized with TEM, UV-visible, EDX, FTIR and TGA to know the size, composition and surface modification of NPs. The best electrical conductivity of printed electronic (0.11x 10^5 S cm^{-1}) was obtained by applying a 10% AgNPs nano-ink solution on photo paper while keeping sintering temperature at 150oC for 60 min of sintering time. A conductive nano-ink was used to prepare electrodes (counter, reference and working) on photo paper and utilized in cyclic voltammetry (CV) measurement. In addition, we demonstrated the lighting of LED when conductive track was connected to a 1.5 V battery. The advantages of this paper-based flexible electrodes are user-friendly, cost effective and multiple analyses (> 50 times) in CV compared to other printed electrodes.

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Non-intrusive blood glucose sensor technology overview

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Current glucose monitoring devices are based on devices originally created in the 1960's. They have been made smaller are easier to use and can log data, their measurements basically are the same as the first laboratory sensors. The patent must prick their finger so that they can squeeze a droplet of blood on a strip coated with an enzyme (usually glucose oxidase) that reacts to form hydrogen peroxide from the available glucose and oxygen. The hydrogen peroxide generated is then measured amperometrically with an electrode. The cost, inconvenience and pain incurred in using these systems have led to heavy research to develop non-invasive glucose monitoring techniques. The major current areas of research and the sensor technologies they use will be discussed. The techniques to be covered include interstitial fluid chemical analysis, breath chemical analysis, infrared spectroscopy, optical coherence tomography, temperature modulated localized reflectance, Raman spectroscopy, polarity changes, ultrasound, fluorescence, thermal spectroscopy, ocular spectroscopy and impedance spectroscopy.

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Development of new ion source/new desorption method for mass spectrometry

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Detection and quantification of illicit compounds at trace level is very much important for public health, security and safety. Mass spectrometry (MS) has already been demonstrated its versatility for detection and quantification of a wide range of compounds at trace level because MS can provide molecular level information of the target compounds. So far, none of the ion source can ionize efficiently for a wide range of compounds. For example, electrospray ionization (ESI) and nano-ESI have been widely using for bio-molecules. Nano-ESI has shown better resolution than ESI but it has clogging problem. To overcome such a problem, several attempts have been taken to develop new ionization source, for example, MALDI, DESI, probe-ESI (PESI) etc. Recently, we have developed new atmospheric pressure chemical ionization (APCI) using alternating current (ac) instead of dc and found as a soft ionization source. Moreover, hollow cathode discharge (HCD) ionization source has been fabricated for detection of explosives at trace level. A desorption method has also been developed using an ultra-cutter to desorb highly non-volatile illicit compounds and ionized dielectric discharge (DBD) ionization source and found better limit of detection (LOD). A vacuum glow discharge ionization (vacuum-GDI) source has been fabricated for compounds those give negative ions. A further attempt has also been taken to fabricate a hybrid ionization source with DBD/ESI for polar/non-polar, volatile/non-volatile compounds.

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Layered double hydroxide materials coated carbon electrode: New challenge to future electrochemical power devices

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Layered double hydroxides (LDHs) have been widely used in the past years due to their unique physicochemical properties and promising applications in electroanalytical chemistry. The present paper focus exclusively on magnesium-aluminum and zinc-aluminum layered double hydroxides (MgAl & ZnAl LDHs) in order to investigate the property and structure of active cation sites located within the layer structure. The MgAl and ZnAl LDH nanosheets were prepared by the constant pH co-precipitation method and uniformly supported on carbon-based electrode materials to fabricate an LDH electrode. Characterization by powder X-ray diffraction, Fourier transform infrared spectroscopy, scanning electron microscopy and transmission electron microscopy revealed the LDH form and well-crystallized materials. Wetting surface properties (hydrophilicity and hydrophobicity) of both prepared LDHs were recorded by contact angle measurement showed hydrophilic character and basic property. The electrochemical performance of these hybrid materials was investigated by mainly cyclic voltammetry, electrochemical impedance spectroscopy and chronoamperometry techniques to identify the oxidation/reduction processes at the electrode/electrolyte interface and the effect of the divalent metal cations in total reactivity. The hierarchy of the modified electrode proves that the electronic conductivity of the bulk material is considerably dependent on the divalent cation and affects the limiting parameter of the overall redox process. However, MgAl LDH shows better performance than ZnAl LDH, due to the presence of magnesium cations in the layers. Following the structural, morphological and electrochemical behavior studies of both synthesized LDHs, the prepared LDH modified electrodes were tested through microbial fuel cell configuration, revealing a remarkable, potential new pathway for high-performance and cost-effective electrode use in electrochemical power devices.

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Naturally occurring exudates gums as eco-friendly inhibitors for mild steel corrosion in acidic medium

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The corrosion inhibition potentials of gum exudates from Daniella oliveri (DO) and *Commiphora africana* (CA) for the corrosion of mild steel in H_2SO_4 have been studied using weight loss and thermometric methods at 303 and 333K. Results show that the exudates gums actually reduced the rates of corrosion of mild steel. Increase in the concentrations of the exudates gums increased their percentage inhibition efficiencies. Corrosion rate was found to increase with increase in temperature in the presence and absence of the gum exudates, though the corrosion rate was slower in the presence of the exudates gums. Both DO and CA exudates gums were found to obey Temkin and Langmuir adsorption models at all concentrations and temperatures studied. Physical adsorption mechanism was proposed from the adsorption parameters. Kinetic and thermodynamic parameters revealed that the adsorption process is spontaneous, exothermic and no significant difference was found between the inhibition efficiencies of DO and CA.

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Simultaneous analysis of doping drugs in human plasma and urine using HPLC-DAD and HPLC-ESI-MS

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Two liquid chromatographic methods have been developed for determination of doping drugs in spiked plasma and urine. The first method, HPLC-DAD (diode array detector) is used for simultaneous separation and quantitation of AMI (amiloride), TOR (torasemide), FUR (furosemide) and IDP (indapamide). It is also used for simultaneous separation and quantitation of ATE (atenolol), caffeine and FUR. They are applied in spiked plasma samples. However, ATE could not be determined quantitatively due to interference with plasma. LODs were found to be 0.16, 0.15, 0.11, 0.12 and 0.25 for AMI, TOR, FUR, IDP and caffeine, respectively. LOQs were found to be 0.49, 0.45, 0.33, 0.36 and 0.75 for AMI, TOR, FUR, IDP and caffeine, respectively. The second method, HPLC-ESI-MS (electrospray ionization-mass spectrometry) has been developed for the routine detection of doping drugs in spiked urine samples. It requires only one injection per sample and is currently capable to detect 10 doping drugs, including six diuretics- FUR, AMI, TOR, hydrochlorothiazide (HCTZ), IDP and spironolactone (SPIRO), two stimulants-caffeine and phenylephrine (PHE) and two β blockers- ATE and bisoprolol in a running time of 14.5 minutes. Both positive and negative ionization modes were used depending on the structure of the separated compounds. The linearity range for most of the drugs was 10-1000 ngmL-1. All parent compounds can be detected at urinary concentrations significantly below 50 ngmL-1.The methods developed simple pretreatment procedure, protein precipitation by acetonitrile and direct dilution for spiked plasma and urine, respectively.

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Non-invasive diagnosis of breast disease by analysis of characteristic hormone ratios

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There is increasing interest in the development of noninvasive diagnostic methods for early breast cancer detection to improve the survival rate and minimize the pain of diagnosis. Common methods for diagnosis and surveillance include mammography, histopathology and blood tests. The major drawbacks of these methods involved high rate of false reports, time consuming and poor specificity. Identification of characteristic compounds by using high resolution mass spectrometer may provide a powerful approach for diagnosis of breast cancer. Here, a new noninvasive method was developed for fast screening breast disease. The hormones, which were collected by scrubbing the surfaces of armpit and nipple with alcohol swabs, were analyzed by Orbitrap mass spectrometry. The obtained mass spectra were subsequently treated statistically to identify discriminating hormones between normal vs. breast disease (breast lesions and breast cancer) patients. We found that the ratios of some hormones including progesterone to testosterone and estrogens to testosterone changed significantly among different breast diseases. The changes in some typical hormone ratios which were produced by the glands of the armpit and nipple will reflect the health status of breast and relate to the female breast disease. This method offers considerable potential as a noninvasive strategy to screen early breast cancer.

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Advanced analytical techniques for the extraction and characterization of plant-derived essential oils by gas chromatography with mass spectrometry

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In recent years, essential oils have a growing interest with positive health effects of their novel characteristics like antibacterial, antifungal and antioxidant activities. For the extraction of plant-derived essential oils, there is the need of advanced analytical techniques and innovative methodologies. An exhaustive study of hydro-distillation, supercritical fluid extraction, ultrasonic & microwave assisted extractions, solid phase micro-extraction, pressurized liquid extraction, pressurized hot water extraction, liquid-liquid extraction, liquid phase micro-extraction, matrix solid-phase dispersion and gas chromatography (one and two dimensional) hyphenated with mass spectrometry for the extraction through various plant species and analysis of essential oils have been provided. Essential oils are composed of mainly terpenes and terpenoids with lower molecular weight aromatic and aliphatic constituents that are particularly important for public health.

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Conversion of waste plastic into liquid hydrocarbon fuel

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Waste Plastic is a huge problem in USA and around the world. This is global problem and because of various inventions in the 20th century, plastics are everywhere. Society has found ample ways to use plastics. But users are less adept at managing the material when they are finished with it—often after only one use. The volume of plastics being produced, used, generated, and discarded is greater than ever before. Plastics therefore require increasing effort and ingenuity in its management. Annually, of the 120 billion pounds of plastics produced in the United States, only about 6% or 4.8 billion pounds are recycled. Inspite of all the news on plastic ban, plastic production is increasing. Waste Technologies LLC (WTL) has the solution at its disposal. This technology can produce approximately 1.3 liter of "WTL fuel" from one kilogram of plastic waste. The exact yield depends on the type of plastic, and the grade of WTL fuel desired. Typically, the process produces a residue of less than 5% of the weight of the plastic waste. This residue is rich in carbon and may be an environmentally superior substitute for coal with a higher BTU value. The WTL technology is able to cater to a wide range of diverse applications, including but not limited to fuel, gas and electrical generation. NSR's / WTL patented technology, in conjunction with WTL technology and know-how, is a simple and economically viable process to decompose the hydrocarbon polymers of waste plastic into the shorter chain hydrocarbons of liquid fuel. WTL believes that it can convert approximately one tonne of plastic into about 300 gallons of fuel at a cost of about $0.75-$1.00 per gallon and produces 4,205 ft3 (CFT) of light gas (C1-C4) byproduct when developed to commercial size. WTL's refining process is uncomplicated and promises to be very competitive with large crude oil installations. In financial projections WTL uses $30/bbl. ($0.71 per gallon) for preprocessing and refining costs. Other plastic recycling technologies generally have a very narrow band of plastics they can use. Nearly all recycling is done with plastic designations 1 or 2 while designations 3 through 7 are virtually untapped (over 70% of all plastic fall within these categories). A combination of economic and technological factors account for this situation. The advantage of WTL technology is that it can produce a profitable product from material that society generally pays to throw away. It is this low cost feedstock that is the key advantage.

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D2Dx-From diameter to diagnostics: Gold nanoparticle-enabled dynamic light scattering assay for chemical and biological target detection and analysis

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Dynamic light scattering (DLS) is an analytical technique used routinely to measure the hydrodynamic sizes of particles with diameters in the nanometer region. Gold nanoparticles are known for their exceptional light scattering properties. By combining the strong light scattering property of gold nanoparticle probes with the size measurement capability of DLS, a new technique named as D2Dx (from diameter to diagnostics) for chemical and biological target detection and analysis was developed. Gold nanoparticles can be surface-modified with various chemical ligands, antibodies or other binding molecules to form gold nanoparticle probes. The binding of chemical or biological target analytes with their specific gold nanoparticle probes can lead to nanoparticle cluster formation, and subsequently, an average particle size increase of the assay solution. Such particle size increases can be measured by DLS, and correlated to the quantity of the target analytes. D2Dx is a single-step homogeneous solution assay, easy to perform, of low cost, and has excellent sensitivity and reproducibility. So far, this technique has been applied successfully for quantitative detection and analysis of a wide range of chemical and biological targets, including proteins, DNAs, viruses, carbohydrates, small chemicals, toxic metal ions, food and environmental toxins. In this talk, I will explain the principle of D2Dx, give an overview on the application potentials of this technique in biomedical research, food safety and environmental protection, and then present several specific examples of using D2Dx for protein detection and analysis.

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Environmental estrogens: An analytical challenge

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Estrogenic compounds, particularly the synthetic estrogenic steroids-ethinyl estradiol (EE2) and mestranol (MeEE2) and natural hormone steroids-estrone (E1), estradiol (E2) and estriol (E3), have attracted a great deal of scientific and public attention during recent years due to their occurrence in surface waters and sewage treatment plant effluents and their potential adverse effects on the development and reproduction of fish, wildlife and even human beings. In this presentation, we will focus on our research on the occurrence, sources, and microbial and photochemical degradation of both synthetic and natural estrogenic steroids in fresh and marine aquatic environments and their effects on public health during the past decade. To face analytical challenges for determining trace amounts of estrogenic steroids in natural waters, GC-MS and HPLC analytical methods have been developed. The developed methods were applied to the water samples periodically collected from wastewater treatment plants, lakes, Acushnet River and Buzzards Bay. The interested compounds were detected in several of water samples in nano to micro-gram per liter concentration range, can certainly cause fish feminization and may also contribute to the observed declines in American lobster population in Buzzards Bay. Microbial and photochemical degradation of E1, E2, E3, EE2 and MeEE2 have been also investigated in seawater as well as in waste, lake and river waters as a comparison. The microbial degradation of synthetic steroid estrogens is extremely slow with a half-life of longer than 70 days in seawater. However, the photo degradation of these compounds is much faster with a half-life of 17 hours for EE2 and 19 hours for MeEE2. Humic and other dissolved organic substances significantly accelerate the sunlight-induced photo degradation of estrogenic steroids. Transition metal Fe(III), nitrate and nitrite can further catalyze the photochemical decomposition of these steroids.

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