



**INSTITUT KIMIA MALAYSIA**

*Certificate of Participation*

*This is to certify that*

**Dr. Hendrik Oktendy Lintang**

*has participated as an Oral Presenter in the*

**16th Asia Pacific International Symposium on**

**Microscale Separations and Analysis**

**APCE 2016**

7<sup>th</sup> to 10<sup>th</sup> November 2016

Johor Bahru, Johor, Malaysia

Organized By

**INSTITUT KIMIA MALAYSIA Southern Branch**

**UNIVERSITI TEKNOLOGI MALAYSIA**



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The Rector of Universitas Ma Chung hereby assigns:

Name : Dr. Hendrik Oktendy Lintang  
Employee's Number : 20160017  
Position : Principal Investigator of MRCPP

to participate in the **16<sup>th</sup> Asia-Pacific International Symposium on Microscale Separations and Analysis (APCE) 2016 as Oral Presenter** with paper title "**Phosphorescent Chemical Sensors of Alcohol Derivatives using Single Crystals of Copper(I) Pyrazolate Complexes**" organized by Institut Kimia Malaysia (IKM) and Universiti Teknologi Malaysia (UTM) which is held on 7-10 November 2016 in KSL Hotels & Resort, Johor Bahru, Johor, Malaysia,

He has to submit an official report when returns to work.

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# APCE 2016

16th Asia Pacific International Symposium on  
Microscale Separations and Analysis

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## PAPERS & AUTHORS

Abstract Code	Title of Paper & Name of Author(s)	Page No
<b>Oral Presentation (page 1-53)</b>		
PL1	<b>Development in microscale separations by nano-liquid chromatography and nano-capillary electrophoresis</b> Imran Ali	1
PL2	<b>Recent developments in enantioseparation with capillary electromigration techniques</b> Bezhan Chankvetadze	2
PL3	<b>Purpose made capillary electrophoresis instruments</b> <u>Peter C. Hauser</u> <sup>1</sup> , <u>Israel Joel Koenka</u> <sup>1</sup> , <u>Jasmine Furter</u> <sup>1</sup> , <u>Michael Liebetanz</u> <sup>1</sup> , <u>Thanh Duc Mai</u> <sup>1,2</sup> , <u>Duy Anh Bui</u> <sup>1,2</sup> , <u>Thi Anh Huong Nguyen</u> <sup>1,3</sup> , <u>Jorge Sáiz</u> <sup>1,4</sup>	3
K1	<b>Kidney like chip: from physiological model to disease model</b> Bingcheng Lin, Yong Luo, Hongli Lin	4
K2	<b>Integrating Operations in Microfluidic Chip – Capillary Electrophoresis Devices for Assay of Proteins at Trace Levels in Biofluids with High Levels of Interfering Substances</b> Y.S. Fung	5
K3	<b>Role of Structural Similarity in Quantitative Structure-Retention Relationships</b> <u>Paul R. Haddad</u> <sup>1</sup> , <u>Soo Hyun Park</u> <sup>1</sup> , <u>Maryam Taraji</u> <sup>1</sup> , <u>Yabin Wen</u> <sup>1</sup> , <u>Mohammad Talebi</u> <sup>1</sup> , <u>Ruth Amos</u> <sup>1</sup> , <u>Robert A. Shellie</u> <sup>1</sup> , <u>Roman Szucs</u> <sup>2</sup> , <u>John W. Dolan</u> <sup>3</sup> and <u>Christopher A. Pohl</u> <sup>4</sup>	6
K4	<b>Solid Extraction and Analysis of Intact Endotoxins in Surface Water by CE-LIF</b> Hua Tao Feng, Min Su, <u>Sam Fong Yau Li</u> <sup>a,b</sup>	7
K5	<b>Microscale liquid phase specific separations using fullerenes</b> Takuya Kubo, Eisuke Kanao, Toyohiro Naito, <u>Koji Otsuka</u>	8
K6	<b>Identification of protein estrogenization as an indication of biomarkers and a metabolic post-translational modification using LC-MS/MS</b> Shu-Hui Chen	9
K7	<b>Liquid Extraction Surface Analysis Coupled With Capillary Electrophoresis</b> Joon Yub Kwon, Ho Gyun Lee, In Hye Sung, and <u>Doo Soo Chung</u>	10
K8	<b>Exploiting the Capabilities of Modern Autosamplers in Microextraction Applications</b> Hian Kee Lee	11
K9	<b>Moving Reaction Boundary Electrophoresis Chip</b> L Y Fan, H Xiao, <u>C X Cao</u>	12
V1	<b>A Single Cell Sizing and Deformability Measurement on A Microfluidic Chip</b> <u>Noritada Kaji</u> <sup>1,2</sup> , <u>Takao Yasui</u> <sup>1,2</sup> , <u>Yoshinobu Baba</u> <sup>1,3</sup>	13
V2	<b>Membraneless Gas-Separation <math>\mu</math>Pad</b> <u>Piyawan Phansi</u> <sup>1,2</sup> , <u>Saichon Sumantakul</u> <sup>1,2</sup> , <u>Thinnapong Wongpakdee</u> <sup>2</sup> , <u>Nutnaree Fukana</u> <sup>2</sup> , <u>Nuanlaor Ratanawimarnwong</u> <sup>1,3</sup> , <u>Jirayu Sitanurak</u> <sup>1,2</sup> and <u>Duangjai Nacapricha</u> <sup>1,2</sup>	14
V3	<b>3D Printing Microfluidic Devices</b> <u>Niall Macdonald</u> <sup>a</sup> , <u>Feng Li</u> <sup>b</sup> , <u>Joan Marc Cabot</u> <sup>a</sup> , <u>Petr Smjekal</u> <sup>b</sup> , <u>Rosanne Guijt</u> <sup>c</sup> , <u>Brett Paull</u> <sup>a,b</sup> , and <u>Michael Breadmore</u> <sup>a,b</sup>	15

Abstract Code	Title of Paper & Name of Author(s)	Page No
V4	<b>Charge-Variant Analysis of Erythropoietin By Affinity Probe Capillary Electrophoresis</b> Kiyohito Shimura	16
V5	<b>Development and Application of Digital Electrophoresis for Bioassay</b> Kenji Sueyoshi	17
V6	<b>Molecular sieving for 1 bp separation by capillary electrophoresis</b> <u>Yoshinori Yamaguchi</u> <sup>*(1)(2)</sup> Chenchen Liu <sup>(1)</sup>	18
V7	<b>Miniaturized Sample Preparation Devices With Polymeric Extraction Media and the On-Line Coupling to Liquid Chromatography</b> <u>Yoshihiro Saito</u> <sup>1</sup> , Toshiaki Tazawa <sup>1</sup> , Tomoya Monobe <sup>1</sup> , Ikuro Ueta <sup>2</sup>	19
V8	<b>A Novel Hybrid Flow Analysis Approach For The Integration of On-Line Dynamic Fractionation And Speciation of Trace Concentrations of Inorganic Arsenic and Organic and Inorganic Mercury in Complex Environmental Solids</b> Yanlin Zhang <sup>1</sup> , Manuel Miro <sup>2</sup> , <u>Spas D. Kolev</u> <sup>1</sup>	20
V9	<b>Integration of Nobel Plasmonics Detection Techniques With Microfluidic Concentration Devices</b> Shau-Chun Wang	21
V10	<b>Ultra-sensitive CE-LIF/MS System for Single Cell Omics Research</b> <u>Takayuki Kawai</u>	22
V11	<b>Capillary Electrophoresis as a Versatile Tool to Characterize Complexes in Aqueous Solutions</b> <u>Nobuhiko Iki</u>	23
V12	<b>Development of Pillar Array Columns With Low-Dispersion Turns</b> <u>Makoto Tsunoda</u> <sup>1</sup>	24
V13	<b>Application of Magnetic Inorganic-organic Hybrid Nanomaterials in Sample Preparation</b> Sharifah Mohamad	25
V14	<b>Behaviors of Low Levels of Pseudoephedrine on Surfaces upon Deposition during Clandestine Laboratory Remediation</b> <sup>1,2</sup> Ahmad Fahmi Lim Abdullah	26
V15	<b>Differentiation and Identification of Isobaric Compounds in Drug Abuse Cases</b> Zaiton Ariffin, Hasni Harun	27
V16	<b>Graphene and Graphene Nanospheres for Biosensor Applications</b> Raja Zaidatul Akhmar Raja Jamaluddin, <u>Lee Yook Heng</u> <sup>1*</sup> , Tan Ling Ling, Chong Kwok Feng <sup>2</sup>	28
C1	<b>Polymer Inclusion Membrane as Solvent-Free Platform for Electrophoresis</b> <u>Pavisara Nanthasurasak</u> <sup>1</sup> , Hong Heng See <sup>2</sup> , Rosanne M. Guijt <sup>1,3</sup> and Michael C. Breadmore <sup>1</sup>	29
C2	<b>Rapid Mass Production Method For Fabrication of Microfluidic Paper-Based Analytical Devices Using Silk Screening Technique</b> <u>Jirayu Sitanurak</u> <sup>1,2</sup> , Yanisa Thepchuay <sup>1,2</sup> , Taweechai Amornsakchai <sup>2</sup> , Nuanlaor Ratanawimarnwong <sup>1,3</sup> , Duangjai Nacapricha <sup>1,2</sup>	30
C3	<b>Multi-Stacking From Two Sample Streams In A Standard Double T Electrophoretic Chip</b> <u>Lee Yien Thang</u> <sup>1,2</sup> , Joselito Quirino <sup>3,4</sup> , Hong Heng See <sup>1,2</sup>	31
C4	<b>Rapid Identification of Trace Impurities in API by UPLC-TOF-MS/MS</b>	32

Abstract Code	Title of Paper & Name of Author(s)	Page No
	<u>Mun Hwa Chong</u> <sup>1</sup> , <u>Pin Sheng Lee</u> <sup>1</sup> , <u>Ya Lin Tnay</u> <sup>1</sup> , <u>Eiji Nishimura</u> <sup>1</sup>	
C5	<b>Miniaturised modular capillary LC: Sensitive detection with UV-LED Z-cell</b> <u>Yan Li</u> <sup>1</sup> , <u>Pavel N. Nesterenko</u> <sup>1</sup> , <u>Roger Stanley</u> <sup>2</sup> , <u>Brett Paull</u> <sup>1</sup> , <u>Mirek Macka</u> <sup>1</sup>	33
C6	<b>LCMS Profiling And Identification Of Antioxidants Constituents For <i>Artocarpus Heterophyllus J33</i> (AhJ33) Fruit Waste From Different Extraction Methods</b> <u>Mohd Nazrul Hisham Daud</u> <sup>1,2</sup> , <u>Dian Nashiela Fatanah</u> <sup>1</sup> , <u>Noriham Abdullah</u> <sup>1,3</sup> and <u>Rohaya Ahmad</u> <sup>1</sup>	34
C7	<b>Liquid Chromatography Mass Spectrometry for the Detection And Validation of Quercetin-3-O-Rutinoside and Myricetin From Fractionated <i>L. Pumila</i> (Var <i>Alata</i>)</b> <u>Norliza Abdul Latiff</u> , <u>Chua Lee Suan</u> , <u>Mohammad Roji Sarmidi</u>	35
C8	<b>Tyrosinase Activity in Melanoma Cells by <i>Labisia Pumila</i> Fraction</b> <u>Ismail Ware</u> <sup>1</sup> , <u>Mariani Ab Hamid</u> <sup>1,2</sup> , <u>Chua Lee Suan</u> <sup>1,2</sup> , <u>Siti Hajar Mat Sarip</u> <sup>1</sup> , <u>Norliza Abdul Latiff</u> <sup>1</sup> , <u>Maizatulkamal Yahayu</u> <sup>1</sup> , <u>Nor Farahiyah Aman Nor</u> <sup>1</sup> , <u>Nur Fashya Musa</u> <sup>1</sup> , <u>Mohamad Roji Sarmidi</u> <sup>1</sup>	36
C9	<b>Multiplexing Separation and Identification of N-glycan by Capillary Electrophoresis</b> <u>Feng Hua Tao</u> <sup>a,b</sup> , <u>LI Pingjing</u> <sup>b</sup> , <u>Su Min</u> <sup>a</sup> , <u>Li Fong Yau</u> <sup>a,b</sup>	37
C10	<b>The Fabrication of Monolithic Microplatform as an Enzyme Support from Polymer-Silica Hybrid Via Sol-Gel Polymerization Method</b> <u>Sabiqah Tuan Anuar</u> <sup>1</sup> , <u>Nur Hidayah Idrohani</u> <sup>2</sup>	38
C11	<b>Optical Properties of Novel and Simple Schiff Base Complexes in Detecting DNA</b> <u>Nur-Fadhilah Mazlan</u> <sup>1</sup> , <u>Ling Ling Tan</u> <sup>1</sup> , <u>Nurul Huda Abd. Karim</u> <sup>2</sup> , <u>Lee Yok Heng</u> <sup>1,2</sup> , <u>Mohammad Imam Hasan Reza</u> <sup>1</sup>	39
C12	<b>HPLC Fingerprint Analysis For Identification and Discrimination of Botanical Raw Material For Food and Herbal Medicine From Indonesia</b> <u>Mohamad Rafi</u> <sup>1,2</sup> , <u>Aryani Sabir</u> <sup>1</sup> , <u>Fitri Handayani</u> <sup>1</sup> , <u>Latifah K. Darusman</u> <sup>1,2</sup> , <u>Eti Rohaeti</u> <sup>1</sup> , <u>Yudiwanti Wahyu</u> <sup>2</sup>	40
C13	<b>Metabolite Profiling Reveals Impact of Metabolites In <i>Wedelia Trilobata</i> Allelopathy</b> <u>Kamalrul Azlan Azizan</u> <sup>1</sup> , <u>Syammi Afiq Mustaza</u> <sup>1</sup> , <u>Nurul Haizun Abdul Ghani</u> <sup>2</sup> , <u>Mohamad Firdaus Nawawi</u> <sup>2</sup>	41
C14	<b>Development of Ionic liquid based Magnetic Nanoparticle in Magnetic Solid Phase Extraction for the Determination of toxic compounds</b> <u>Muggundha Raoov Ramachandran</u> <sup>1</sup> , * <u>Mohamad Shariff Shahrman</u> <sup>1</sup> , <u>Masrudin Md Yusoff</u> <sup>1</sup> , <u>Nur Nadhirah Mohamad Zain</u> <sup>1</sup> , <u>Noorfatimah Yahaya</u> <sup>1</sup> , <u>Sharifah Mohamad</u> <sup>2</sup>	42
C15	<b>Countercurrent Extraction for Separating Xanthorrhizol from Temu Lawak (<i>Curcuma Xanthorrhiza</i>) Essential Oil</b> <u>Rudi Heryanto</u> <sup>1,2</sup> , <u>Wulan Tri Wahyuni</u> <sup>1,2</sup> , <u>Mahwan Febrian</u> <sup>1</sup>	43
C16	<b>Ultrasound-Assisted Emulsification Microextraction Combined with Dispersive Micro-Solid Phase Extraction for the Determination of Azole Antifungals in Milk Samples by HPLC-DAD</b> <u>Nurshazliana Othman</u> <sup>1</sup> , <u>Noorfatimah Yahaya</u> <sup>1*</sup> , <u>Lim Vuanghao</u> <sup>1</sup> , <u>Mohd Marsin Sanagi</u> <sup>2</sup> , <u>Sazlinda Kamaruzaman</u> <sup>3</sup> , <u>Takahito Mitome</u> <sup>4</sup> , <u>Norikazu Nishiyama</u> <sup>4</sup>	44

Abstract Code	Title of Paper & Name of Author(s)	Page No
C17	<b>Experimental Design Approach For Micro-Solid Phase Extraction Of Acidic Drugs Using Multi-Walled Carbon Nanotubes/Alginate/Magnetic Composite Beads</b> <u>Wan Nazihah Wan Ibrahim</u> <sup>1,2</sup> , Mohd Marsin Sanagi <sup>2</sup> , Wan Aini Wan Ibrahim <sup>2</sup> , Nor Suhaila Mohammad Hanapi <sup>1,2</sup>	45
C18	<b>Multi-stacking in microchip capillary electrophoresis for the monitoring of vancomycin in biological samples</b> <u>Kah Chun Chong</u> <sup>1</sup> and Hong Heng See <sup>1,2</sup>	46
C19	<b>Miniaturised Capillary Electrophoresis Instrument: Toward Distributed Monitoring Heavy Metals In Environmental Water</b> <u>Min Zhang</u> <sup>1</sup> , Petr Smejkal <sup>1</sup> , Rosanne M Guijt <sup>2</sup> , Michael C Breadmore <sup>1</sup>	47
C20	<b>A simple and sensitive capillary electrophoresis method for simultaneous determination of five pharmaceuticals in river water</b> <u>Ambavaram Vijaya Bhaskar Reddy</u> <sup>1,2</sup> , Zulkifli Yusop <sup>1</sup> , Jafariah Jaafar <sup>2</sup> , Azmi Aris <sup>1,3</sup> , Zaiton Abdul Majid <sup>2</sup> , Nur Hidayati Jamil <sup>2</sup>	48
C21	<b>Alginate Incorporated Multi-Walled Carbon Nanotubes as Dispersive Solid Phase Extraction Sorbent for Selective and Efficient Separation of Acidic Drugs in Aqueous Matrices</b> Nur Zaimah Zaini	49
C22	<b>Fluorescence Quenching on Mesoporous Carbon Nitride by Phenol and Aniline</b> <u>Leny Yulianti</u> <sup>1,2*</sup> , Ahmad Hanami Abd Kadir <sup>3</sup> , Siew Ling Lee <sup>2,3</sup> , Hendrik O. Lintang <sup>1,2</sup>	50
C23	<b>Development of Haloalkane Dehalogenase Based Biosensor for the Determination of 1,2-Dichloroethane in Environmental Water</b> <u>Hidayah Binti Shahr</u> , Tan Ling Ling, Lee Yook Heng, Sharina Abu Hanifah, Mohammad Imam Hasan Reza	51
C24	<b>Phosphorescent Chemical Sensors of Alcohol Derivatives Using Single Crystals of Copper(I) Pyrazolate Complexes</b> <u>Hendrik O. Lintang</u> <sup>1,2</sup> , <u>Leny Yulianti</u> <sup>1,2</sup> , Nur Fatiha Ghazali <sup>3,4</sup>	52
C25	<b>In-Syringe Dispersive Micro-Solid Phase Extraction Method For the HPLC Fluorescence Determination of Aflatoxins in Peanut</b> Nor Shifa Shuib <sup>1</sup> , Bahrudin Saad <sup>2</sup>	53
<b>Poster Presentation (page 54-119)</b>		
P1	<b>Species &amp; Serotype-Specific Determination of Endotoxins Based on Oligosaccharide Fingerprints Using LC-MS</b> <u>Anna Karen Carrasco Laserna</u> <sup>1</sup> , Feng Huatao <sup>1</sup> , Sam Fong Yau Li <sup>1,2</sup>	54
P2	<b>Acid Mine Drainage Treatment Using Inorganic and Organic Materials Mixed</b> <u>Anuar Othman</u> <sup>1,2</sup> , Azli Sulaiman <sup>2</sup> , Shamsul Kamal Sulaiman <sup>1</sup>	55
P3	<b>Surface Plasmon Resonance (SPR) Sensor for Detection of Chemical Threat Agents in Water</b> <u>Baisheng Chen</u> <sup>1</sup> , Sam Fong Yau Li <sup>1,2</sup>	56
P4	<b>Flow Injection Analysis - Gas-phase Electrophoretic Mobility Molecular Analyzer (FIA-GEMMA) for Quantitation and Characterization of Urinary Proteins</b> Tongmei Ma <sup>1,2</sup> , Tsz Kwan Gigi Lui <sup>2</sup> , <u>Xiao Nam Chow</u> <sup>2</sup> , Ying Sing Fung <sup>2</sup>	57
P5	<b>Full Automation of Sampling-Extraction/Concentration-Detection Methodology using Dual-Head Autosampler</b>	58



Abstract Code	Title of Paper & Name of Author(s)	Page No
	<u>Christina Shu Min Liew</u> <sup>a,b</sup> , <u>Xiao Li</u> <sup>c</sup> , <u>Hong Zhang</u> <sup>b</sup> and <u>Hian Kee Lee</u> <sup>a,b,d,*</sup>	
P6	<b>Detection of Biological Threat Agents by a Novel Fluorescence Aptasensor</b> <u>Haiyan Li</u> <sup>1</sup> , <u>Sam Fong Yau Li</u> <sup>1,2</sup>	59
P7	<b>Drug Delivery System in Microfluidic Device: Microchannel Design and Fabrication</b> <u>Hazwan Halimoon</u>	60
P8	<b>Extraction and Characterization of Crude Oil Acids For Microemulsion Modeling</b> <u>Ivy Chai Ching Hsia</u> <sup>1</sup> , <u>Noor 'Aliaa Amira M Fauzi</u> <sup>1</sup> , <u>Voon Chang Hong</u> <sup>2</sup> , <u>Alia Khalid</u> <sup>2</sup> , <u>Hon Vai Yee</u> <sup>1</sup>	61
P9	<b>Detection of Biological Threat Agents by Molecularly Imprinted Polymers on Quartz Crystal Microbalance Sensors</b> <u>Jiixin Chen</u> <sup>1</sup> , <u>Sam Fong Yau Li</u> <sup>1,2,*</sup>	62
P10	<b>Quartz crystal microbalance surface modification by iron oxide nanostructures for mass sensitivity enhancement</b> <u>Junyi Liu</u> , <u>Sam Fong Yau Li</u>	63
P11	<b>Detection of the chemical threat agent simulant parathion methyl molecules by molecularly imprinted polyvinylidene difluoride sensor using quartz crystal microbalance</b> <u>Xuan Hao Lin</u> <sup>1</sup> , <u>Jacelyn Angkasa</u> <sup>1</sup> , <u>Kah Sing Chooi</u> <sup>1</sup> , <u>Sam Fong Yau Li</u>	64
P12	<b>Extraction of Coconut Oil From Different Coconut Species</b> <u>Nor Farahiyah Aman Nor</u> <sup>1</sup> , <u>Shakti Kaur Khalsa</u> <sup>1,2</sup> , <u>Auni Salma Shaiful Azhar</u> <sup>1</sup> , <u>Siti Nor Azlina Abd. Rashid</u> <sup>1</sup> , <u>Sulaiman Ngadiran</u> <sup>1</sup> , <u>Harisun Ya'akub</u> <sup>1,2</sup> , <u>Noor Azwani Zainol</u> <sup>1</sup> , <u>Nik Nurul Najihah Nik Mat Daud</u> <sup>1</sup> , <u>Muna Mohamed</u> <sup>1</sup> , <u>Maizatulkmal Yahayu</u> <sup>1</sup> , <u>Siti Hajar Mat Sarip</u> <sup>1</sup>	65
P13	<b>Large Scale Isolation of Xanthorrhizol from <i>Curcuma Xanthorrhiza</i> Essential Oil</b> <u>Masuri Kama Kamaruddin Shah</u> and <u>Eiji Nishimura</u>	66
P14	<b>Analysis of Sulfite in White Wine by Using Membraneless Vaporization Unit as Tool for On-Line Sample Preparation</b> <u>Nattapong Chantipmanee</u> <sup>1,2</sup> , <u>Waleed Alahmad</u> <sup>1,2</sup> , <u>Thitaporn Sonsa-ard</u> <sup>1,2</sup> , <u>Kanchana Uraisin</u> <sup>1,2</sup> , <u>Nuanlaor Ratanawimarnwong</u> <sup>1,3</sup> , <u>Thitirat Mantim</u> <sup>1,3</sup> and <u>Duangjai Nacapricha</u> <sup>1,2</sup>	67
P15	<b>Sensory Evaluation and Mineral Content of Protein Supplement Incorporated with Herbal and Vegetable Extract</b> <u>Noor Azwani Zainol</u> , <u>Nor Farahiyah Aman Nor</u> , <u>Harisun Yaakob</u> , <u>Siti NorAzlina Abd Rashid</u> and <u>Auni, Salma Shaiful Amri, Maizatulkmal Yahayu</u>	68
P16	<b>Bundled Hollow Fiber Array-Mediated Liquid Phase Microextraction of Estrogens in Aqueous Matrices</b> <u>Shalene Xue Lin Goh</u> <sup>1,2</sup> , <u>Hian Kee Lee</u> <sup>1,2</sup>	69
P17	<b>Headspace in-tube microextraction for capillary electrophoresis/mass spectrometry</b> <u>Joon Yub Kwon</u> , <u>Yun Jung Choi</u> , and <u>Doo Soo Chung</u> <sup>1</sup>	70
P18	<b>On-Column Preconcentration in High Performance Liquid Chromatography Using Valve Switching System: Application to Non-Steroidal Anti-Inflammatory Drugs (NSAIDs)</b> <u>Siti Norbayu Mohd Subari</u> <sup>1</sup> , <u>Rozita Osman</u> <sup>1</sup> , <u>Norashikin Saim</u> <sup>1</sup>	71
P19	<b>New In-pipette tip assisted graphene sol-gel based monolithic sorbent material</b>	72

Abstract Code	Title of Paper & Name of Author(s)	Page No
	<b>for the rapid analysis of UV filter in aqueous samples using HPLC-UV</b> <u>Hua Kwang Lee</u> <sup>1</sup> , <u>Chih-Kuei Yeh</u> <sup>1</sup> , <u>Vinoth Kumar Ponnusamy</u> <sup>2</sup> , <u>Jen-Fon Jen</u> <sup>1,*</sup>	
P20	<b>Bland- Altman and Measurement of Uncertainty for Determination of Common Drugs of Abuse in Dried Blood Stain (DBS) vs Whole Blood Specimen (WBS) by Liquid Chromatography-Tandem Mass Spectrometer (LCMSMS) – Application to Forensic Toxicology Cases in Malaysia</b> <u>Syaiful Izwan Ismail</u> and <u>Fathiah Ahmad Zubaidi</u>	73
P21	<b>Preparation of MIP-based QCM sensor for detection of organophosphorus compounds</b> <u>Xiamin Cheng</u> <sup>1,3</sup> , <u>Sam Fong Yau Li</u> <sup>1,2*</sup> and <u>Bin Liu</u> <sup>3,4</sup>	74
P22	<b>Automated liquid extraction surface analysis using a commercial capillary electrophoresis instrument</b> <u>Ho Gyun Lee</u> , <u>Yun Jung Choi</u> , <u>Doo Soo Chung</u>	75
P23	<b>Comparative Study of Time Vs Injection Time on the Separation of DNA Fragments Using Capillary Electrophoresis</b> <u>Nur Hafiza Md Yusop</u> , <u>Aedrianee Reeza Alwi</u> , <u>Wan Nur Zawani W.M. Samsudin</u>	76
P24	<b>Chemical Profiling of Heroin Seized in Selected Regions of Malaysia by Attenuated Total Reflectance-Fourier Transform Infra-Red Spectroscopy</b> <u>Mohd. Zawawi Yusoff</u>	77
P25	<b>Determination Of Common Drugs Of Abuse And Metabolites By Liquid Chromatography Tandem Mass Spectrometry (LCMSMS) For Forensic Cases</b> <u>Noor Hamimah Abd Hamid</u> , <u>Muhamad Akram Ali</u> , <u>Syaiful Izwan Ismail</u>	78
P26	<b>Quantitation of Fluoride in Sludge by Capillary Electrophoresis</b> <u>Low Ying Ying</u> , <u>Malarvili Ramalingam</u> , <u>Suraya Hani Ramli</u>	79
PF1	<b>New Aminopropyltrimethoxysilane Functionalized Magnetic Sporopollenin-Graphene Oxide-Based Adsorbent For The Removal of Pb(II) Ions From Industrial Waste Water</b> <u>Abdul-Aziz Mohd Hassan</u> <sup>1,2</sup> , <u>Wan Aini Wan Ibrahim</u> <sup>1,3*</sup> , <u>Mohd Bakri Bakar</u> <sup>1</sup> , <u>Mohd Marsin Sanagi</u> <sup>1,3</sup>	80
PF2	<b>Preparation and Characterization of Starch Grafted with Methacrylamide using Ammonium Persulphate Initiator</b> <u>Abdulganiyu Umar</u> <sup>a,b</sup> , <u>Mohd Marsin Sanagi</u> <sup>a,c*</sup> , <u>Ahmed Salisu</u> <sup>a</sup> , <u>Wan Aini Wan Ibrahim</u> <sup>a,c</sup> , <u>Khairil Juhanni Abd Karim</u> <sup>a</sup> , <u>Aemi Syazwani Abdul Keyon</u> <sup>a</sup>	81
PF3	<b>Improved Solid Phase Microextraction Techniques for the Analysis of Organic Contaminants in Aqueous Samples</b> <u>Nurul Nabilah Zainal Abidin</u> <sup>1</sup> , <u>Mohd Marsin Sanagi</u> <sup>1,2,*</sup> , <u>Wan Aini Wan Ibrahim</u> <sup>1,2</sup>	82
PF4	<b>Sensitivity enhancement using free liquid membrane in electrokinetic supercharging-capillary electrophoresis</b> <u>Mei Qi Chui</u> <sup>1</sup> and <u>Hong Heng See</u> <sup>1</sup>	83
PF5	<b>The improvement and application of electromembrane extraction (EME) across hollow polymer inclusion membrane for analysis of drugs and pesticides</b> <u>Nor Akma Mamat</u> <sup>1,2</sup> and <u>Hong Heng See</u> <sup>1,2</sup>	84
PF6	<b>Development of Dynamic Mixed Matrix Membrane Tip Microextraction for the Determination of Non-Steroidal Anti-Inflammatory Drugs in Effluent Water Samples</b> <u>Nurul Hazirah Mukhtar</u> <sup>1,2</sup> , <u>Hong Heng See</u> <sup>1,2</sup>	85

Abstract Code	Title of Paper & Name of Author(s)	Page No
PF7	<b>Electromembrane Extraction of Paraquat and Diquat Using a Three-Dimensional Printed Flow-Cell</b> <u>Siti Nur Ain Fatimah Abdillah</u> <sup>1</sup> and Hong Heng See <sup>1,2</sup>	86
PF8	<b>Analysis of Organic High Explosives Using Gas Chromatography–Tandem Mass Spectrometry</b> <u>Mohamad Afiq Mohamed Huri</u> <sup>1</sup> , Umi Kalthom Ahmad <sup>1</sup> , Roliana Ibrahim <sup>2</sup> , and Mustafa Omar <sup>3</sup>	87
PF9	<b>Multi-elements analysis of stingless bee honey using inductively coupled plasma optical emission spectrometry: Its potential in assigning authenticity and provenance</b> <u>Aidil Fahmi Shadan</u> <sup>1,2</sup> , Naji A. Mahat <sup>1,*</sup> , Wan Aini Wan Ibrahim <sup>1,3*</sup> , Zaiton Ariffin <sup>4</sup> & Dzulkiflee Ismail <sup>5</sup>	88
PF10	<b>Preliminary Study of Polypyrrole-Based Nanocomposites for Adsorption of Heavy Metals and Dyes in Water Sample</b> Ng Nyuk Ting <sup>1</sup> , <u>Amirah Farhan Kamaruddin</u> <sup>1</sup> and Aemi Syazwani Abdul Keyon <sup>1</sup>	89
PF11	<b>Effect of Combination of Food Grade Non-Ionic Surfactants and Stirring Method on Stability of Virgin Coconut Oil</b> Auni Salma Shaiful Amri <sup>1</sup> , Harisun Yaakob <sup>1</sup> , Siti Nor Azlina Abd Rashid <sup>1</sup> , Noor Azwani Zainol <sup>1</sup> , Nor Farahiyah Aman Nor <sup>1</sup>	90
PF12	<b>Flavonoids Distribution in Four Varieties of <i>Ficus Deltoidea</i> Using LCMS-Q-TOF: Hierarchical Cluster Analysis Technique</b> <u>Syazwani Dzinol</u> <sup>1</sup> , Rohaya Ahmad <sup>2</sup> , Mazatulikhma Mat Zain <sup>3</sup> , Mohd Ikhwan Ismail <sup>4</sup>	91
PF13	<b>Sporopollenin Modified Magnetite and Graphene Oxide as New Magnetic Solid Phase Extraction Adsorbent for Determination of Polar Organophosphorus Pesticides in Vegetable Samples</b> <u>Ayuba Markus</u> <sup>1</sup> , Wan Aini Wan Ibrahim <sup>1,2*</sup> , Abdulaziz Hassan <sup>1</sup> , Hamid Rashidi Nodeh <sup>1</sup> , Faridah Mohd Marsin <sup>1</sup> , Aemi Syazwani Abdul Keyon <sup>1</sup>	92
PF14	<b>Computational Study of Vinpocetine-Teicoplanin Aglycone Complex as a Chiral Selector</b> Hasmerya Maarof <sup>1*</sup> , <u>Nor Faradilla Roslan</u> <sup>1</sup>	93
PF15	<b>Magnetite-Polypyrrole-Reinforced Activated Carbon Sorbent for Micro-Solid Phase Extraction Combined with Gas Chromatography Micro-Electron Capture Detection for the Determination of Organochlorine Pesticides in Aqueous Matrices</b> <u>Faridah Mohd Marsin</u> <sup>1,2</sup> , Wan Aini Wan Ibrahim <sup>1,3*</sup> , Mohd Marsin Sanagi <sup>1,3</sup>	94
PF16	<b>Simultaneous Determination of Aceclofenac, Ketorolac and Sulindac in Human Urine by Solid Phase Membrane Tip Extraction and Microemulsion Electrokinetic Chromatography</b> <u>Izdiani Mohd Yatim</u> <sup>1</sup> , Wan Aini Wan Ibrahim <sup>1</sup> , Dadan Hermawan <sup>2</sup> , Aemi Syazwani Abdul Keyon <sup>1</sup>	95
PF17	<b>The Assessment of Heavy Metals in Perna Viridis Mussel and Surface Seawater in Pasir Gudang Coastal Area, Malaysia</b> <u>Aida Rasyidah Azman</u> <sup>1</sup> , Nur Kamilah Mukhtar <sup>1</sup> , Razali Ismail <sup>1</sup> , Aemi Syazwani Abdul Keyon <sup>1</sup> , Naji Arafat Mahat <sup>1</sup>	96
PF18	<b>Superhydrophobic Magnetic Nanoparticles -Free Fatty Acid Regenerated from Waste Cooking Oil for the Enrichment of Carcinogenic Polycyclic Aromatic Hydrocarbons in Sewage Sludges and Landfill Leachates</b>	97

Abstract Code	Title of Paper & Name of Author(s)	Page No
	<u>Siti Khalijah Mahmad Rozi</u> <sup>a,b</sup> , Shabnam Bakhshaei <sup>a</sup> , Ninie Suhana Abdul Manan <sup>a</sup> , Sharifah Mohamad <sup>a</sup>	
PF19	<b>Comparison of Extraction Methods Using Sol-Gel Hybrid Methyltrimethoxysilane-Cyanopropyltriethoxysilane Material for the Analysis of Non-Steroidal Anti-Inflammatory Drugs in Urine Samples</b> <u>Mashkurah Abd Rahim</u> <sup>1</sup> , Wan Aini Wan Ibrahim <sup>1,2*</sup> , Zainab Ramli <sup>1</sup> and Mohd Marsin Sanagi <sup>1,2</sup>	98
PF20	<b>Removal of Phenolic Compounds Using Benzyl Imidazolium-Based Ionic Liquids</b> <u>Nadiah Sidek</u> <sup>1,2</sup> , Ninie S. A. Manan <sup>1,2</sup> , Sharifah Mohamad <sup>1,2</sup>	99
PF21	<b>Octadecylsilyl-Silica-Impregnated Agarose-Chitosan Film for the Extraction of Phenanthrene and Pyrene from Chrysanthemum Tea</b> <u>Nyuk Ting Ng</u> <sup>1</sup> , Mohd Marsin Sanagi <sup>1,2*</sup> , Wan Nazihah Wan Ibrahim <sup>1,3</sup> , Wan Aini Wan Ibrahim <sup>1,2</sup>	100
PF22	<b>Quantification of 2, 6, 4'-Trihydroxy-4-Methoxy-Benzophenone in Various Parts of <i>Phaleria Macrocarpa</i> Fruits (Mahkota Dewa)</b> <u>Norazah Basar</u> <sup>1</sup> , Siti Nur Atiqah Md Othman <sup>1</sup> , Sharifah Maizatul Akmal Sayed Abdellah <sup>1</sup>	101
PF23	<b>Multi-Functional Ceramic Membrane for Heavy Metal Removal</b> <u>Norfazilah Muhamad</u> , Mukhlis A. Rahman, Norfazliana Abdullah, Mohd Hafiz Dzarfan Othman, Juhana Jaafar, A.F Ismail	102
PF24	<b>Effect of Churning and Thawing Temperatures in Integrated Wet Process on the Yield and Quality of Virgin Coconut Oil</b> Nor Farahiyah Aman Nor, Mohamad Roji Sarmidi*, Nur Fashya Musa, Siti Nor Azlina Abd. Rashid, Sulaiman Ngadiran, Harisun Ya'akub, Noorazwani Zainol, Norliza Abdul Latiff, Siti Hajar Mat Sarip, Muna Mohamed, Ismail Ware, Maizatulakmal Yahayu	103
PF25	<b>Molecular Design of Copper(I) Pyrazolate Complexes for High Phosphorescent Sensing Capability in Detection of Alcohol Derivatives</b> <u>Nur Fatiha Ghazalli</u> <sup>3,4</sup> , Leny Yuliaty <sup>1,2</sup> , Mustaffa Shamsuddin <sup>3</sup> , Hendrik O. Lintang <sup>1,2</sup>	104
PF26	<b>Characterization of pharmaceutical micropollutants using in-situ suspended aggregate microextraction and capillary electrophoresis</b> <u>Nur Hidayati Jamil</u> <sup>1</sup> , Jafariah Jaafar <sup>1*</sup> , Ambavaram Vijaya Bhaskar Reddy <sup>1</sup> , Zaiton Abdul Majid <sup>1</sup> , Azmi Aris <sup>2</sup> , Zulkifli Yusof <sup>2</sup>	105
PF27	<b>Electrokinetic Supercharging For on-Line Preconcentration of Endocrine-Disrupting Chemicals and Phenolic Pollutants</b> <u>Nur Syazwani Ishak</u> <sup>1</sup> , Nurul 'Izzah Abdul Karim <sup>1</sup> , Aemi Syazwani Abdul Keyon <sup>1*</sup>	106
PF28	<b>GC-MS Analysis of Organic Catalytic Oxidation Reactions</b> <u>Nurliana Roslan</u> , Nurafiqah Saadon, Zainab Ramli, Salasiah Endud, and Mohd Bakri Bakar	107
PF29	<b>Preparation And Characterization of Dithiocarbamate Funtionalized Magnetic Nanoparticle For The Magnetic Solid Phase Extraction of Heavy Metals</b> <u>Nurul Amilin Supardi</u> <sup>1</sup> , Mohd Hamzah Mohd Nasir <sup>1</sup> , Fiona How Ni Foong <sup>1</sup> , Mazidatulakmam Miskam <sup>2</sup> and Sharifah Mohammad <sup>3</sup>	108
PF30	<b>Nanocomposite silica supported onto silica gel for extraction of hippuric Acid</b> <u>Mohmad Raizul Zinalibdin</u> <sup>1,2</sup> , Jafariah Jaafar <sup>1</sup> , Zaiton Abd Majid <sup>1</sup>	109
PF31	<b>Stability of Lauric Acid Nanoemulsion Formulation by Addition of Palm Oil</b> <u>Siti Hajar Mat Sarip</u> <sup>1</sup> , Azila Abdul-Aziz <sup>2</sup> , Ismail Ware <sup>1</sup> , Norliza Abdul Latiff <sup>1</sup> ,	110

Abstract Code	Title of Paper & Name of Author(s)	Page No
	Maizatulkamal Yahayu <sup>1</sup> , Nur Fashya Musa <sup>1</sup> , Nor Farahiyah Aman Nor <sup>1</sup>	
PF32	<b>Optimization of Magnetic Sporopollenin-Cyanopropyltri-Ethoxysilane Dispersive-<math>\mu</math>-Solid Phase Extraction in the Determination of Selected Tricyclic Anti-Depressants in Water Using Box-Behnken Design</b> <u>Siti Munirah Abd Wahib</u> <sup>1</sup> , Wan Aini Wan Ibrahim <sup>1,2*</sup> , Mohd Marsin Sanagi <sup>1,2</sup> and Muhammad Afzal Kamboh <sup>3</sup>	111
PF33	<b>Physicochemical Properties of Fish Flavour Produced by Controlled Autolysis of <i>Thunnus Tonggol</i> Visceral Waste</b> Harisun Yaakob <sup>1</sup> , <u>Siti Nor Azlina Abd Rashid</u> <sup>1</sup> , Amir Husni Mohd Shariff <sup>2</sup> , Ida Idayu Muhamad <sup>3</sup>	112
PF34	<b>Theoretical Approach for Chiral Separation of Ketoconazole and Itraconazole Antifungal Drugs</b> <u>Siti Rosilah Arsad</u> <sup>1</sup> , Hasmerya Maarof <sup>1*</sup> , Wan Aini Wan Ibrahim <sup>1,2*</sup>	113
PF35	<b>Fragrance Base Note Recover in Perfume Using Gas Chromatography</b> Hasmerya Maarof <sup>1*</sup> , <u>Tengku Fazarina Tengku Ab Ghani</u> <sup>1</sup>	114
PF36	<b>Laser-Induced Breakdown Spectroscopy Coupled With Principal Component Analysis for Establishing Authenticity of Printing Inks</b> <u>Yew Wan Hui</u> <sup>1</sup> , Raja Kamarulzaman Raja Ibrahim <sup>2</sup> , Naji Arafat Mahat <sup>1</sup> and Dzulkiflee Ismail <sup>3</sup>	115
PF37	<b>Methacrylamide Functionalized Crosslinked Chitosan Beads for Microextraction of Anionic Dye from Aqueous Solution</b> <u>Zetty Azalea Sutirman</u> <sup>1</sup> , Mohd Marsin Sanagi <sup>1,2*</sup> , Khairil Juhanni Abd Karim <sup>1</sup> , Ahmedy Abu Naim <sup>1</sup> and Wan Aini Wan Ibrahim <sup>1,2</sup>	116
PF38	<b>Copper Modified Carbon Nitride as Fluorescence Sensor for Nitrate Ions</b> <u>Siti Maryam Jasman</u> <sup>1</sup> , Hendrik O. Lintang <sup>2,3</sup> , Siew Ling Lee <sup>1,2</sup> , Leny Yuliati <sup>2,3</sup>	117
PF39	<b>Assessment of Magnetite-Graphene as Magnetic Solid Phase Extraction Adsorbent For 4-Hydroxybenzoic and Protocatechuic Acids</b> <u>Marina Musa</u> <sup>1</sup> , Wan Aini Wan Ibrahim <sup>1,2*</sup>	118
PF40	<b>Micro-Solid Phase Extraction (<math>\mu</math>-SPE) Based on Alginate Multi-Walled Carbon Nanotubes Sorbent for the Determination of Bisphenol A in Canned Fruits</b> <u>Nor Suhaila Mohamad Hanapi</u> <sup>1</sup> , Nor'ashikin Saim <sup>1</sup> , Nursyamsyila Mat Hadzir <sup>1</sup> , Rossuriati Dol Hamid <sup>1</sup> , Wan Nazihah Wan Ibrahim <sup>1</sup> and Noorfatimah Yahaya <sup>2</sup>	119

# APCE 2016 Programme

<b>1040 – 1100</b>	Refreshments
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**Technical Session 6B: Forensic and others**  
**Venue: Ledang Room**  
**Chairperson: Dato' Dr Yew Chong Hooi**  
**Forensic Science Society of Malaysia (FSSM)**

<b>1100 – 1130</b>	V14 Invited Lecture Behaviour of low levels of pseudoephedrine on surfaces upon deposition during clandestine laboratory remediation Dr. Ahmad Fahmi Lim Abdullah Universiti Sains Malaysia, Malaysia
<b>1130 – 1200</b>	V15 Invited Lecture Differentiation and identification of isobaric compounds in drug abuse cases Pn. Zaiton Ariffin Department of Chemistry, Malaysia
<b>1200 – 1230</b>	V16 Invited Lecture Graphene and graphene nanospheres for biosensor applications Prof. Dr. Yook Heng Lee Universiti Kebangsaan Malaysia, Malaysia
<b>1230 – 1250</b>	C24 Contributed Lecture Phosphorescent chemical sensors of alcohol derivatives using single crystals of copper(II) pyrazolate complexes Dr. Hendrik Oktendy Lintang Universitas Ma Chung, Indonesia
<b>1250 – 1310</b>	C25 Contributed Lecture In-syringe dispersive micro-solid phase extraction method for the HPLC fluorescence determination of aflatoxins in peanut Mr. Nor Shifa bin Shuib Department of Chemistry, Malaysia
<b>1310 – 1400</b>	Lunch
<b>1400 – 1500</b>	Poster Session (Group B)

**Venue: Grand Ballroom 1**

<b>1500 – 1540</b>	Plenary Lecture 3 Chairperson: Prof. Dr. Sam Fong Yau Li National University of Singapore, Singapore Purpose made capillary electrophoresis instruments Prof. Dr. Peter C. Hauser University of Basel, Switzerland
<b>1540 – 1630</b>	Award presentations/Closing ceremony/Announcement of APCE 2017
<b>1630 – 1700</b>	Refreshments

**Thursday, November 10, 2016**

<b>0900 – 1500</b>	Social event
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**PHOSPHORESCENT CHEMICAL SENSORS OF ALCOHOL DERIVATIVES USING SINGLE CRYSTALS OF COPPER(I) PYRAZOLATE COMPLEXES**

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<sup>2</sup>Centre for Sustainable Nanomaterials, Ibnu Sina Institute for Scientific and Industrial Research, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia.

<sup>3</sup>Department of Chemistry, Faculty of Science, Universiti Teknologi Malaysia, 81030 UTM Johor Bahru, Johor, Malaysia

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Chemical sensors (chemosensors) at molecular level with changes in luminescent color and emission have been utilized using single crystals of phosphorescent metal complexes for detection of volatile organic compounds (VOCs). However, effect of molecular structures on sensing capabilities has not been addressed. Therefore, we report the systematic study on vapochromic phosphorescent sensing of alcohol derivatives using trinuclear copper(I) pyrazolate complexes (**2<sub>A-E</sub>**) synthesized from non-side chain, 3,5-dimethyl, 3,5-bis(trifluoromethyl), 3,5-diphenyl and 4-(3,5-dimethoxybenzyl)-3,5-dimethyl pyrazole ligands (**1<sub>A-E</sub>**). All complexes showed emission bands centered at 553, 584, 570 and 616 nm ( $\lambda_{\text{ext}} = 280$  nm) for complexes **2<sub>A-C,E</sub>**, respectively and 642 nm ( $\lambda_{\text{ext}} = 321$  nm) for complex **2<sub>D</sub>** with lifetime in microseconds, indicating a large Stoke shift for phosphorescent compounds with green to red emission in the dark room. Upon exposure to ethanol in 5 mins, chemosensors **2<sub>A-C</sub>** showed quenching of its intensities to 34%, 15% and 100% with color changes to less emissive, suggesting insertion of the vapors to the weak non-covalent Cu(I)-Cu(I) interactions. In particular, the best chemosensor **2<sub>C</sub>** with color OFF gave only reusability using external stimuli. Otherwise, chemosensor **2<sub>D</sub>** showed photoinduced energy transfer from 642 to 636 and 460 nm with 87% decreasing in intensity. Interestingly, chemosensor **2<sub>E</sub>** revealed blue-shifting of its emission intensity to 556 nm ( $\Delta\lambda = 60$  nm, orange to green) where its original emission band was completely and autonomously recovered in 15 mins. When alcohol derivatives were used, chemosensors **2<sub>E</sub>** only showed good sensing capability for detection of methanol ( $\Delta\lambda = 60$  nm) to propanol ( $\Delta\lambda = 22$  nm) due to a weak intermolecular hydrogen bonding interaction of alcohol (methanol to propanol) to the oxygen atoms at dimethoxybenzyl side-chains of the pyrazole ring. By increasing hydrophobicity of alcohols (butanol to hexanol), it will reduce the electronegativity of alcohols for the interaction and have lower vaporation rate.

C24

# Phosphorescent Chemical Sensors of Alcohol Derivatives using Single Crystals of Copper(I) Pyrazolate Complexes

Hendrik, O. LINTANG,<sup>1,2</sup>

Leny Yuliati,<sup>1,2</sup> and Nur Fatiha Ghazali<sup>3,4</sup>

瑪中大學

黃啓鑄 二〇〇七年七月七日

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1



## Volatile Organic Compounds (VOCs)

VOCs are commonly used as ingredients in household products or in industrial processes where they normally get vaporize at room temperature as toxic and organic vapors as well as non-organic vapor



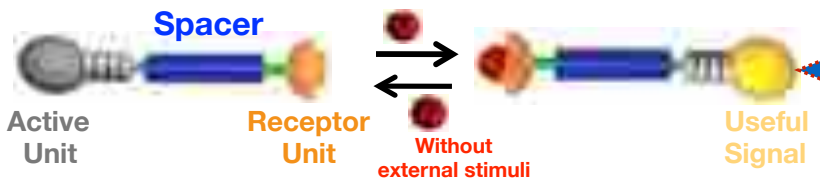
Elosua, C, et al., *Sensors* 2006, 6, 1440





# Luminescence Chemosensors

Chemical sensors (chemosensors) are a molecule, when receptor unit interact with analytes, transform specific information through spacer to the active unit to give analytical useful signals



### Changes in Properties

- Absorption
- Permittivity
- Swelling
- Mass
- Refractive index
- Thermal resistivity
- Luminescence

### Example of Luminescence Chemosensors

**Pt Complex Salt for Chemosensor of Vapors**

Grove, L.J., et al., *Journal of the American Chemical Society* 2004, 126, 1594

**Naphthalimide for Chemosensor of Metal Ions**

Xu, Z., et al., *Organic Letters* 2005, 7, 3029



# Luminescent Copper Complexes with Phosphorescent Properties

Good Phosphorescent Properties, Less Toxicity, and Cheaper Precursors



Chair-Like Coordination Geometry with Cu(I)-Cu(I) Distance 3.1-3.9 Å



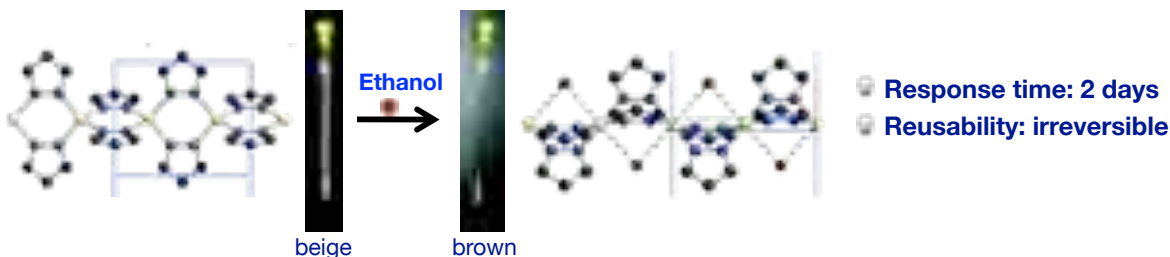
- ### Properties
- Strong Emission
  - Microsecond Lifetime
  - Large Stokes Shift
  - Thermochromics
  - Emission Dependent on Alkyl Side Chains

Dias, H.V.R., et al., *Journal of the American Chemical Society* 2005, 127, 7489



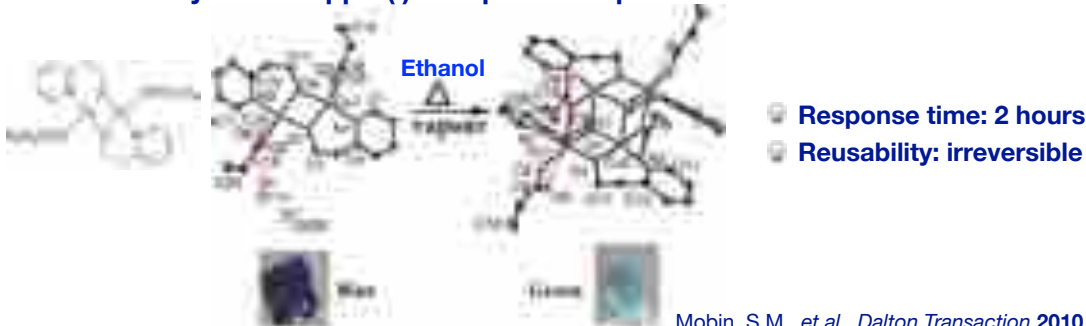
# Copper Complexes as Vapochromic Chemosensors

## 1. Dinuclear Bispyrazolate Copper(II) Complex as Vapochromic Chemosensors



Cingolani, A., et al., *Journal of the American Chemical Society* **2005**, 127, 6144

## 2. Dinuclear Pyridine Copper(I) Complex as Vapochromic Chemosensors



Mobin, S.M., et al., *Dalton Transaction* **2010**, 39, 1447

Ma Chung Research Center for Photosynthetic Bioreactors

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# Copper Complexes as Vapochromic Chemosensors

## 3. Trinuclear Pyrazole Copper(I) Complex as Vapochromic Chemosensors



### The Importance of Molecular Structure for Improving Sensing Capability



Ghazalli, N.F., et al., *Advanced Materials Research* **2014**, 970, 44

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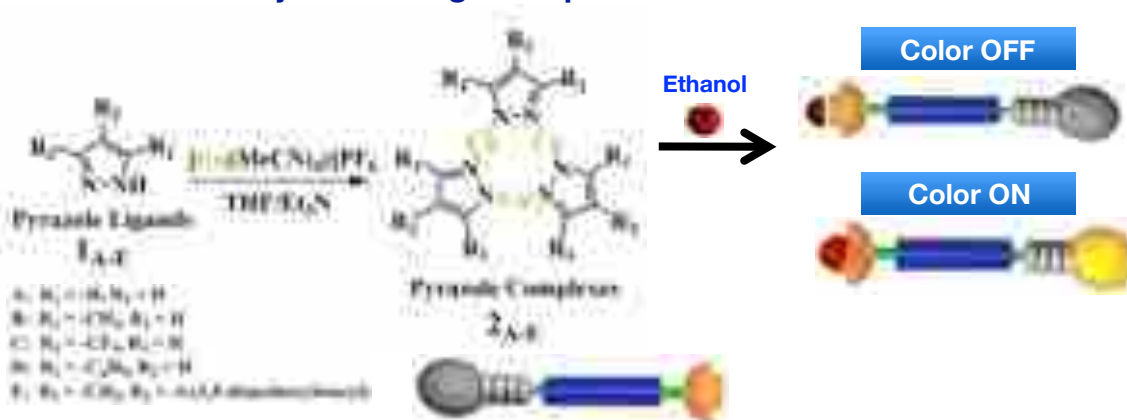
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## Research Objectives

### Effect of Molecular Structures of Copper(I) Complexes with Different Side-Chains at Pyrazole Ring on Vapochromic Chemosensors of Alcohol



- 🧠 Synthesis of Copper(I) Pyrazolate Complexes
- 🧠 Phosphorescent Properties of Copper(I) Pyrazolate Complexes
- 🧠 Sensing Capability of Copper(I) Pyrazolate Complexes



## Synthesis of Copper(I) Pyrazolate Complexes

**100 mL Schlenk (1<sup>st</sup> STAGE)**

- Vacuum
- Changed air with N<sub>2</sub> gas
- Dry THF
- pyrazole ligand (1A, 1B, 1C, 1D, 1E)
- Degassed (3 times)
- Poured  $[\text{Cu}(\text{MeCN})_4]\text{PF}_6$  1 equi.
- Mix 5 minute under N<sub>2</sub>
- Triethylamine 2 equi.
- Stirred overnight at room temperature
- Remove THF by trapping
- Dissolve with dry CH<sub>2</sub>Cl<sub>2</sub>

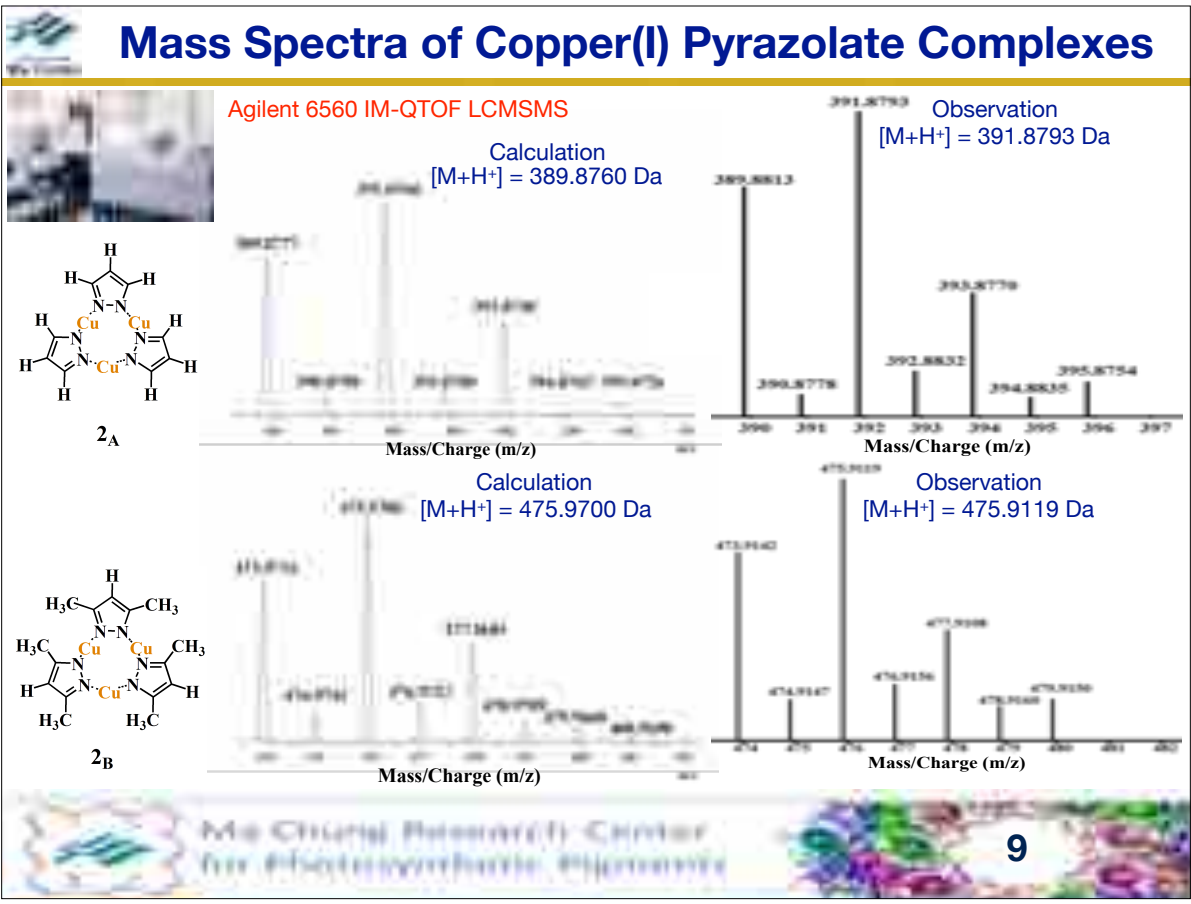
**200 mL Schlenk (2<sup>nd</sup> STAGE)**

- Vacuum
- Changed air with N<sub>2</sub> gas
- Dry methanol
- Transfer with cannula
- In-Situ recrystallization with CH<sub>2</sub>Cl<sub>2</sub> to dry methanol (except for 2<sub>C</sub> using dry hexane)
- In-Situ Filtration with cannula and filter paper
- Collected, Dry-off, and Flow N<sub>2</sub> gas

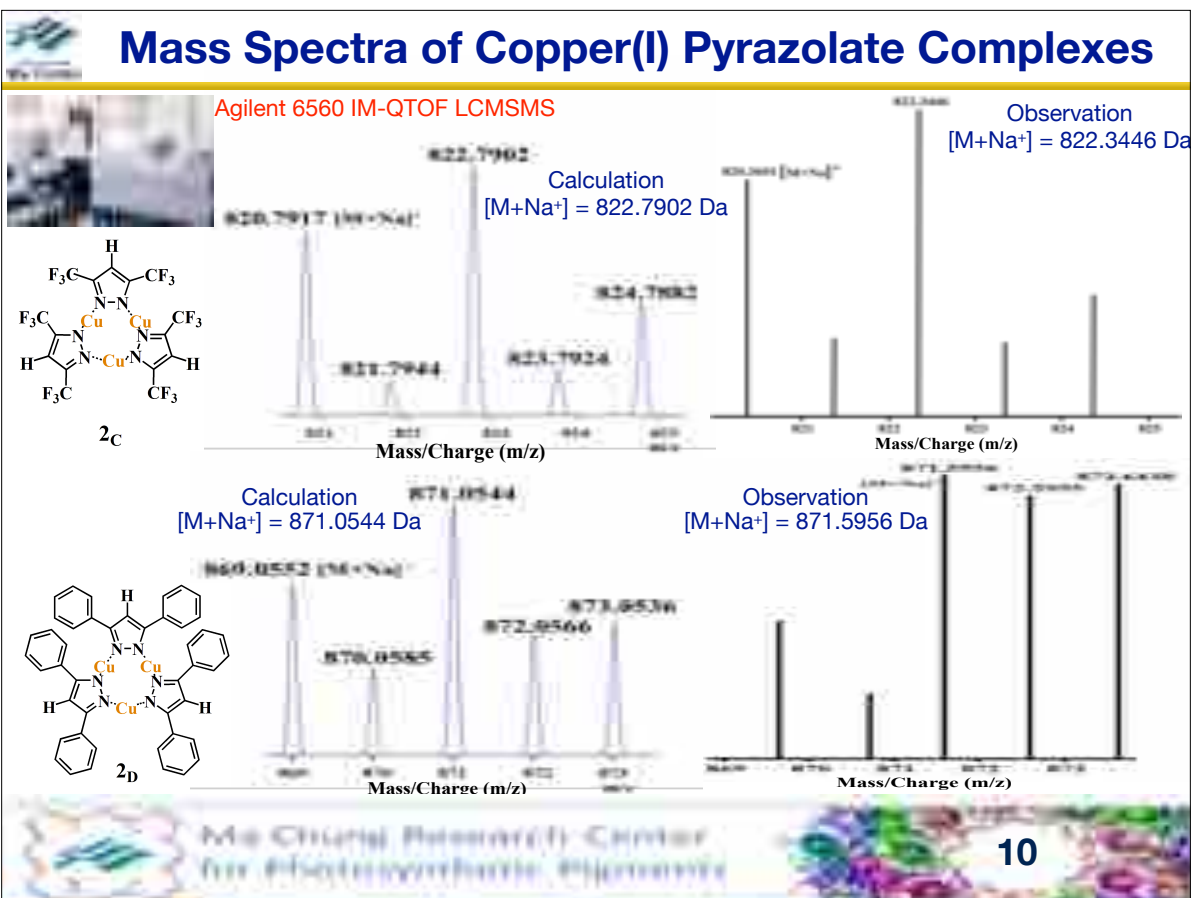
Product Copper(I) Complexes **Solid Powder**

2<sub>A</sub>: R<sub>1</sub> = -CH<sub>3</sub>, R<sub>2</sub> = H  
 2<sub>B</sub>: R<sub>1</sub> = -CH<sub>3</sub>, R<sub>2</sub> = H  
 2<sub>C</sub>: R<sub>1</sub> = -CF<sub>3</sub>, R<sub>2</sub> = H  
 2<sub>D</sub>: R<sub>1</sub> = -C<sub>6</sub>H<sub>5</sub>, R<sub>2</sub> = H  
 2<sub>E</sub>: R<sub>1</sub> = -CH<sub>3</sub>, R<sub>2</sub> = -4-(3,5-dimethoxybenzyl)

$[\text{Cu}(\text{MeCN})_4]\text{PF}_6$  = tetrakis(acetonitrile)copper(I)hexafluorophosphate



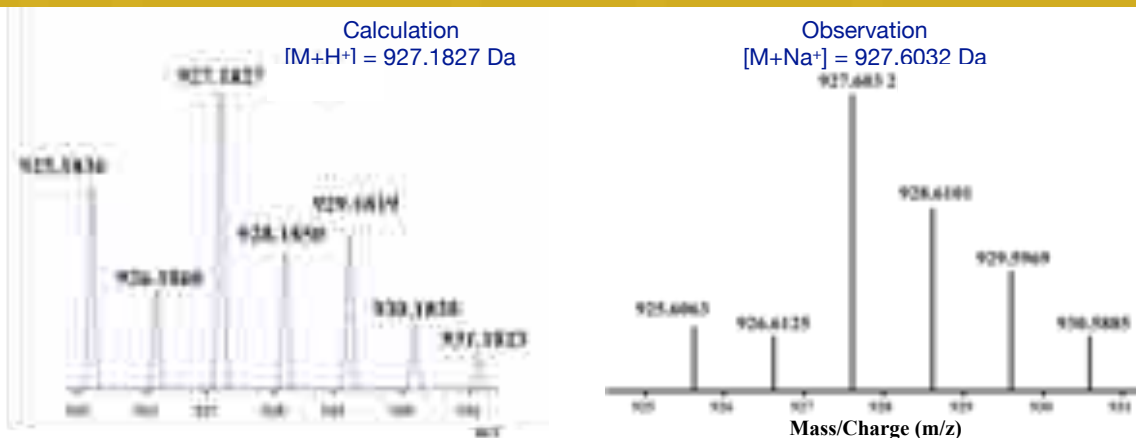
9



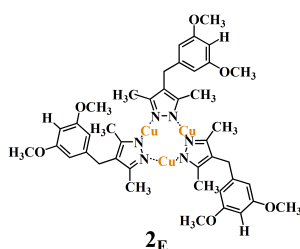
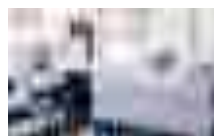
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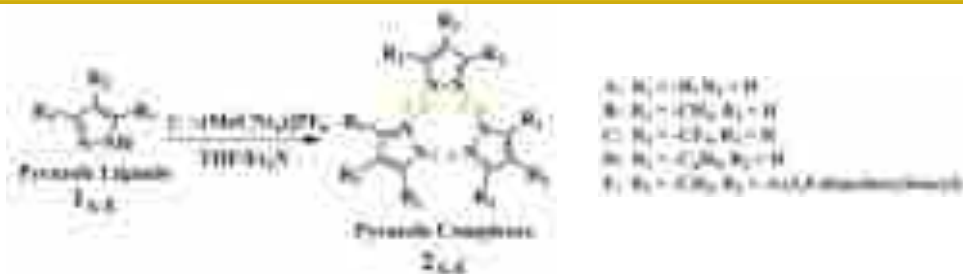
## Mass Spectra of Copper(I) Pyrazolate Complexes



Agilent 6560 IM-QTOF LCMSMS



## Mass Spectra of Copper(I) Pyrazolate Complexes

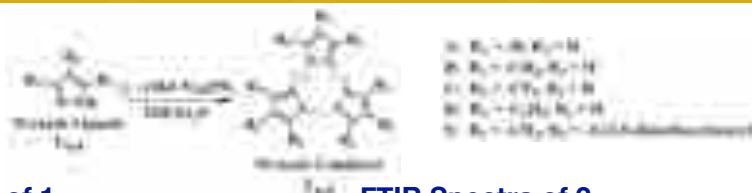


Similar monoisotope patterns and mass value (m/z) of calculated and observed copper complexes

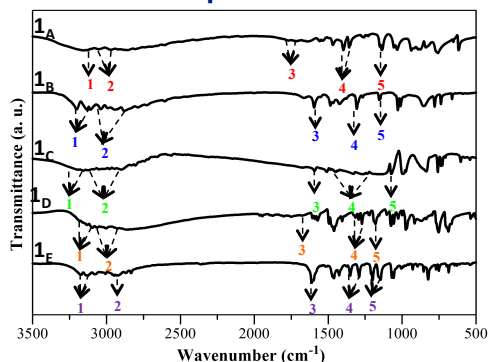
Complexes	Molecular Formula	Molecular Weight (Da.)			
		Calculated [M+H] <sup>+</sup>	Observed [M+H] <sup>+</sup>	Calculated [M+Na] <sup>+</sup>	Observed [M+Na] <sup>+</sup>
2 <sub>A</sub>	C <sub>9</sub> H <sub>9</sub> Cu <sub>3</sub> N <sub>6</sub>	389.8760	391.8793	-	-
2 <sub>B</sub>	C <sub>15</sub> H <sub>21</sub> Cu <sub>3</sub> N <sub>6</sub>	475.9700	475.9119	-	-
2 <sub>C</sub>	C <sub>15</sub> H <sub>3</sub> Cu <sub>3</sub> F <sub>18</sub> N <sub>6</sub>	-	-	822.7902	822.3466
2 <sub>D</sub>	C <sub>45</sub> H <sub>33</sub> Cu <sub>3</sub> N <sub>6</sub>	-	-	871.0544	871.5956
2 <sub>E</sub>	C <sub>42</sub> H <sub>51</sub> Cu <sub>3</sub> N <sub>6</sub> O <sub>6</sub>	927.1827	927.6032	-	-



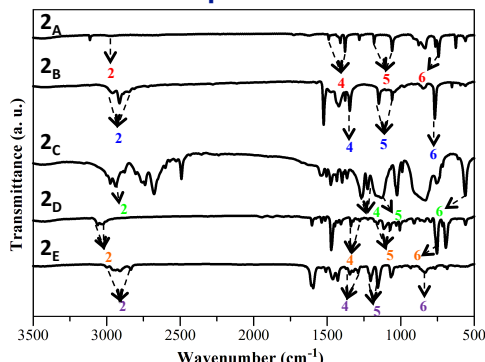
# FTIR Spectra of Copper(I) Pyrazolate Complexes



### FTIR Spectra of 1A-E



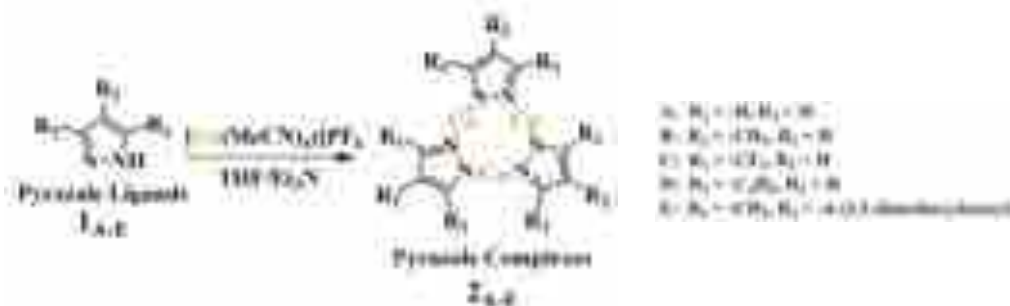
### FTIR Spectra of 2A-E



- Disappearance of vibration bands for N-H stretching and bending from 1° and 2° amine groups at 3201-3058 cm<sup>-1</sup> (1) and 1750-1595 cm<sup>-1</sup> (3) in Pyrazolate Complexes
- Formation of new vibration bands for N-Cu-N at 560-632 cm<sup>-1</sup> (6) in Pyrazolate Complexes



# Photographs of Copper(I) Pyrazolate Complexes



### Photograph Images under Daylight



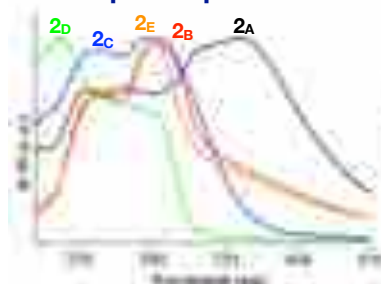
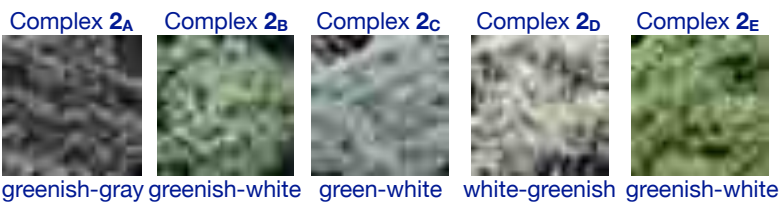


# Phosphorescent Properties of Copper(I) Complexes

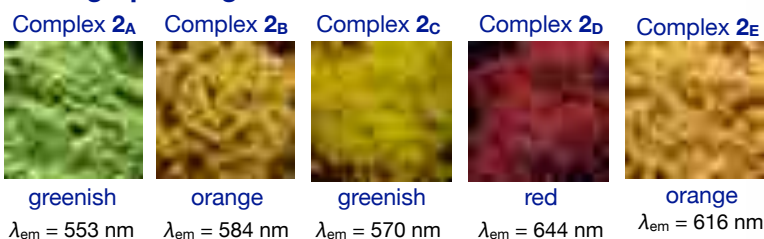
## Green to Red Emission of Trinuclear Copper(I) Pyrazolate Complexes

### Absorption Spectra of 2A-E

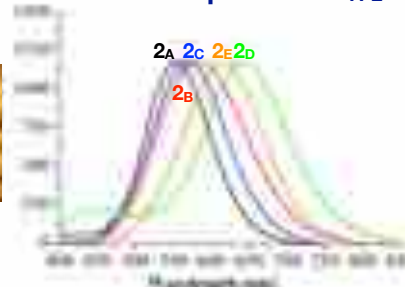
### Photograph Images under Daylight



### Photograph Images under $\lambda_{ext} = 254$ nm



### Emission Spectra of 2A-E



15

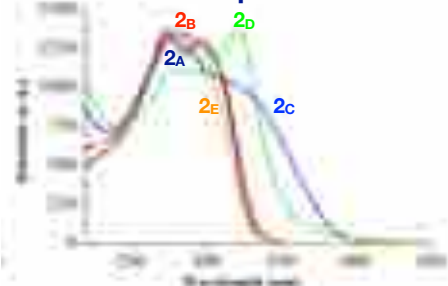
15



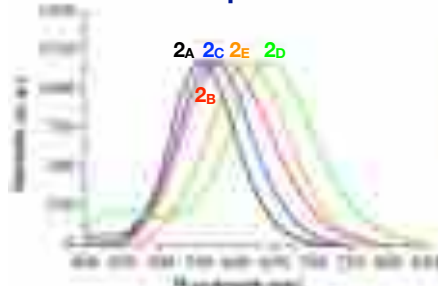
# Phosphorescent Properties of Copper(I) Complexes

## Large Stokes Shifts and Microsecond Lifetimes of Trinuclear Copper(I) Pyrazolate Complexes

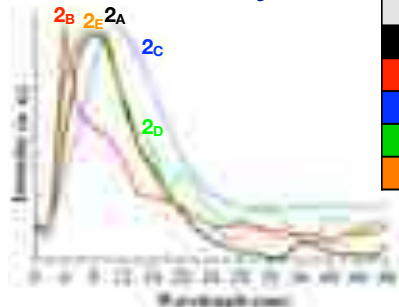
### Excitation Spectra of 2A-E



### Emission Spectra of 2A-E



### Luminescent Decay of 2A-E



Copper(I) Complexes	Luminescence Lifetime ( $\mu$ s)	$\lambda_{max}$ of Excitation (nm)	$\lambda_{max}$ of Emission (nm)	Stokes Shift (nm)
2A	$7.437 \pm 0.7$	274	553	279
2B	$7.594 \pm 1.5$	280	584	304
2C	$7.370 \pm 0.4$	280	570	290
2D	$8.685 \pm 0.3$	322	644	322
2E	$8.162 \pm 1.8$	278	616	338

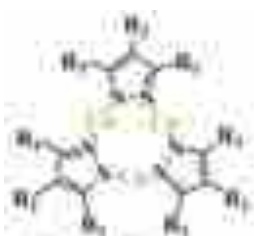


16

16



# Vapochromic Chemosensors of Ethanol



Pyrimidine Complex:

**2A**

- A)  $R_1 = H, R_2 = H$
- B)  $R_1 = -CH_3, R_2 = H$
- C)  $R_1 = -CF_3, R_2 = H$
- D)  $R_1 = -C_2H_5, R_2 = H$
- E)  $R_1 = -CH_3, R_2 = -4-(3,5-dimethoxyphenyl)$

Alcohol



1-5 mins



Cover tightly with parafilm

Ethanol solvent was injected inside after chemosensor was placed

Chemosensor was placed inside the beaker



## Sensing Capability

1. Emission Changes
2. Color Changes



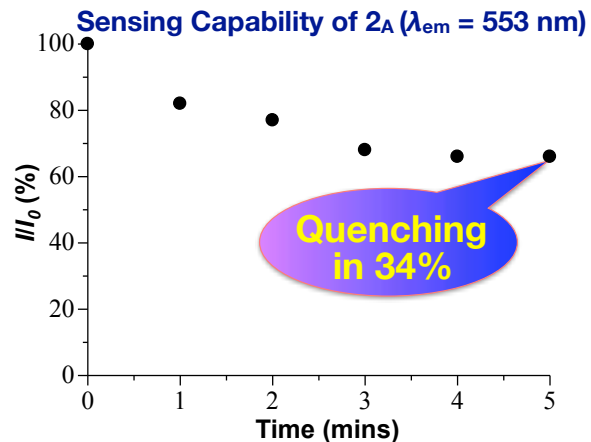
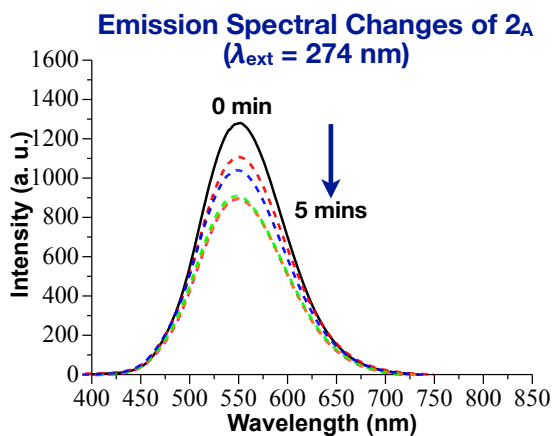
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17

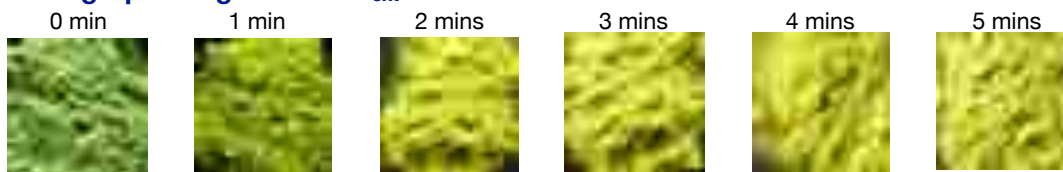
17



# Sensing Capability of Chemosensor 2A ( $R_1 = H, R_2 = H$ )



## Photograph Images under $\lambda_{ext} = 254 \text{ nm}$



Color change from green to green-yellowish in 5 minutes



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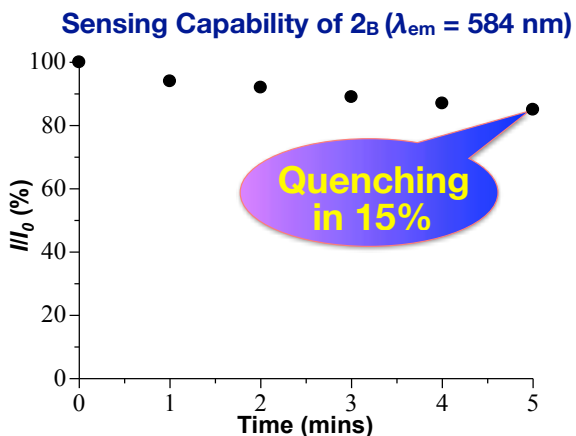
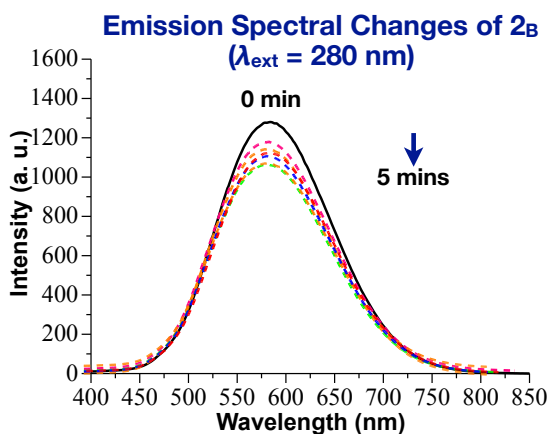
18

18

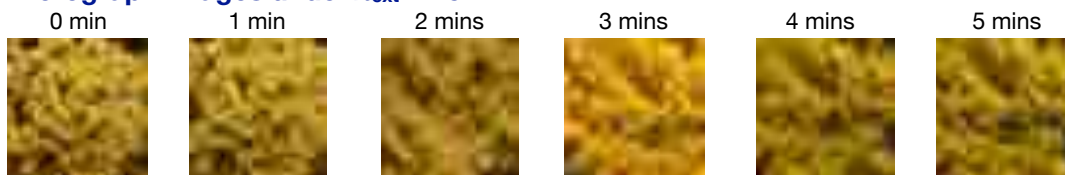




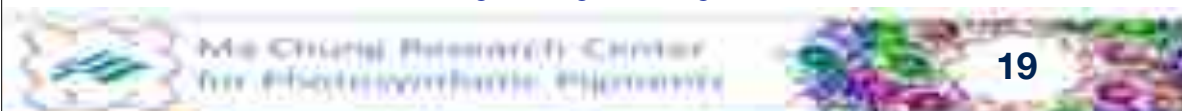
## Sensing Capability of Chemosensor 2<sub>B</sub> (R<sub>1</sub> = CH<sub>3</sub>, R<sub>2</sub> = H)



### Photograph Images under $\lambda_{\text{ext}} = 254 \text{ nm}$



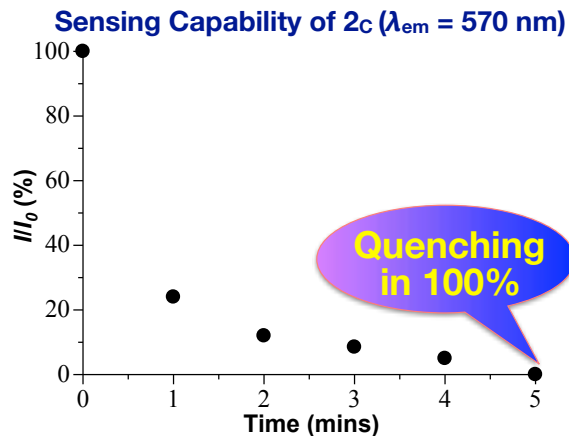
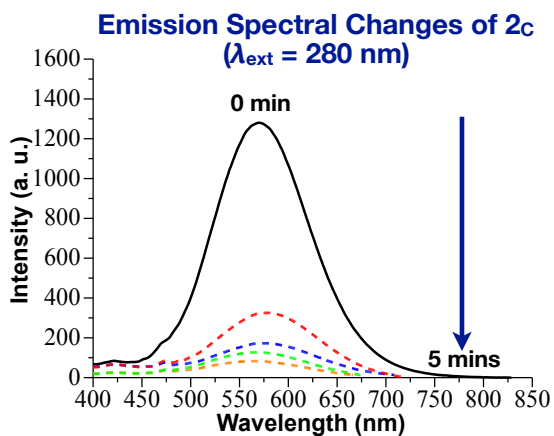
No color change of original orange in 5 minutes



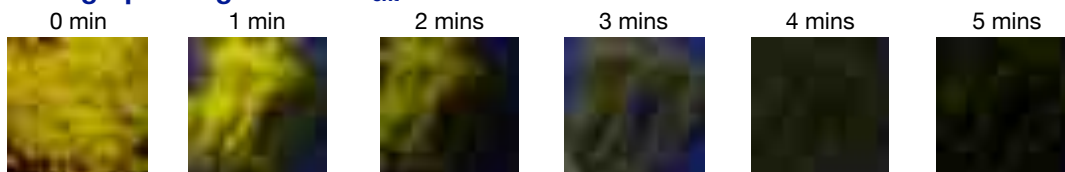
19



## Sensing Capability of Chemosensor 2<sub>C</sub> (R<sub>1</sub> = CF<sub>3</sub>, R<sub>2</sub> = H)



### Photograph Images under $\lambda_{\text{ext}} = 254 \text{ nm}$

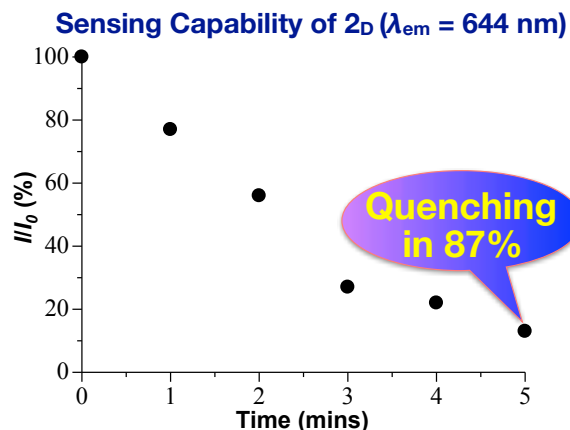
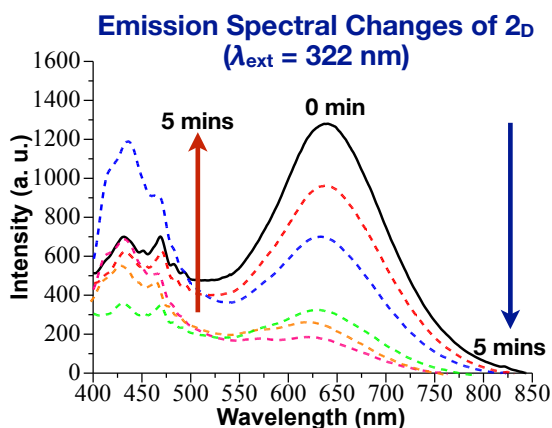


Color change from greenish to dark (less emissive) in 5 minutes

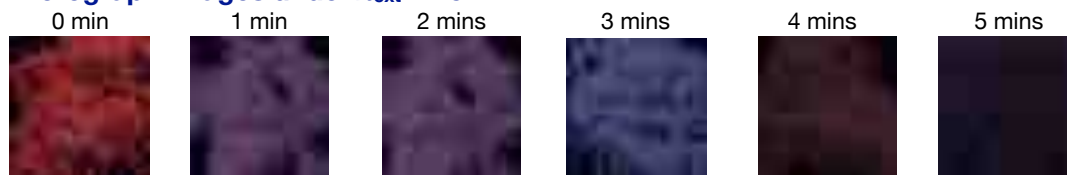


20

## Sensing Capability of Chemosensor 2<sub>D</sub> (R<sub>1</sub> = C<sub>6</sub>H<sub>5</sub>, R<sub>2</sub> = H)



### Photograph Images under $\lambda_{\text{ext}} = 254 \text{ nm}$

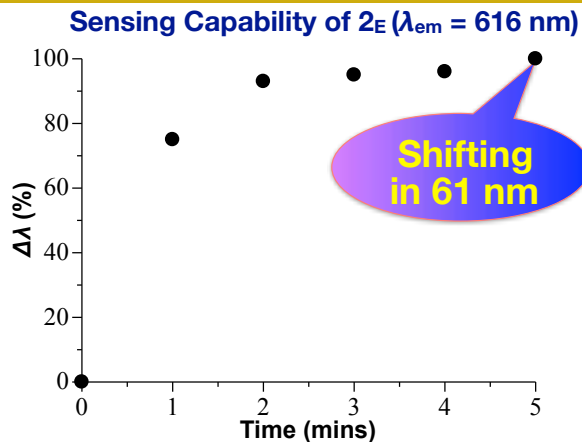
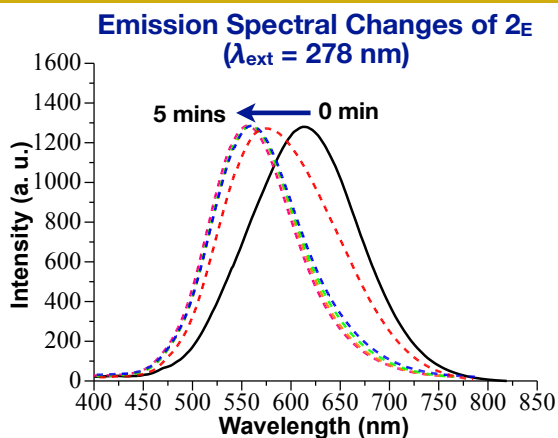


Color change from red to dark-red in 5 minutes

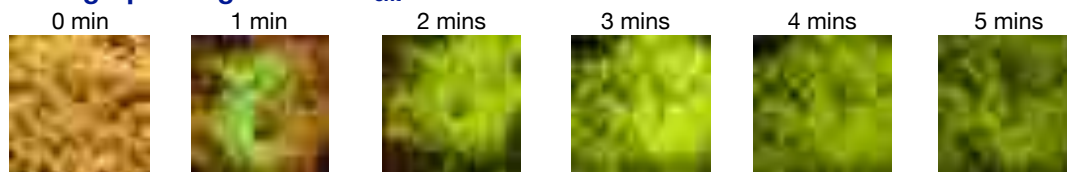


21

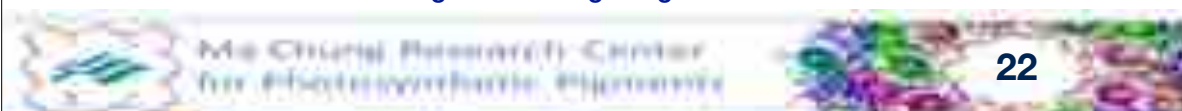
## Sensing Capability of Chemosensor 2<sub>E</sub> (R<sub>1</sub> = CH<sub>3</sub>, R<sub>2</sub> = -(OCH<sub>3</sub>)<sub>2</sub>Bz)



### Photograph Images under $\lambda_{\text{ext}} = 254 \text{ nm}$



Color change from orange to green in 5 minutes

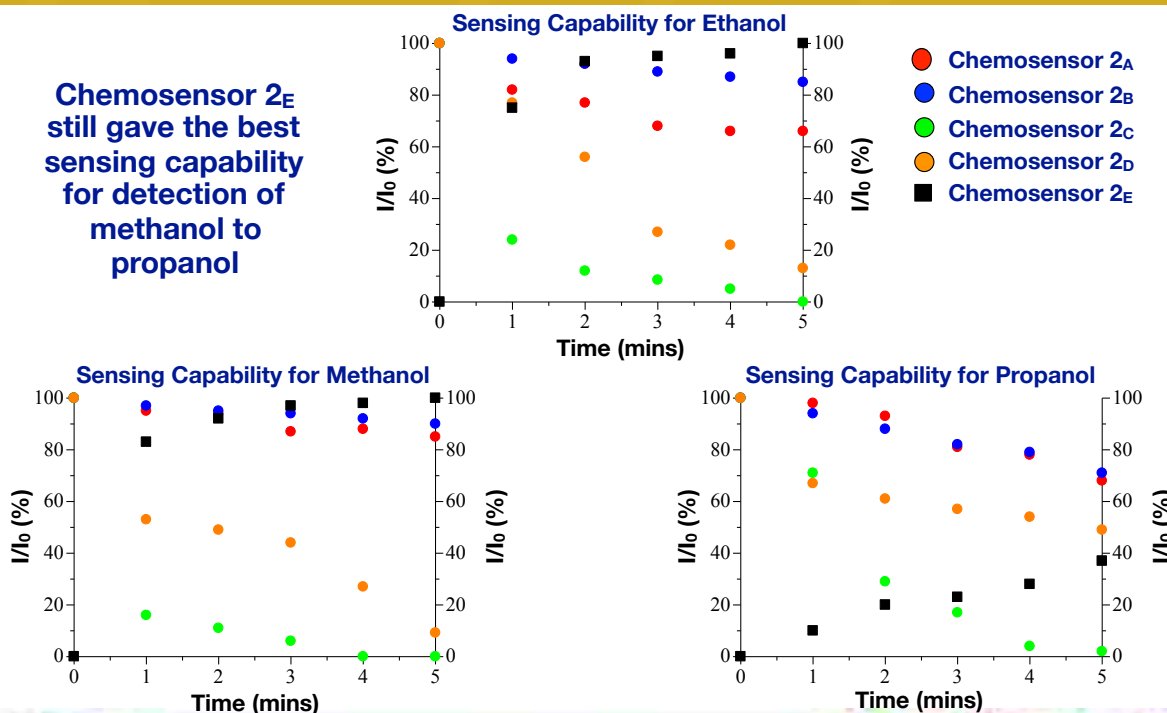


22



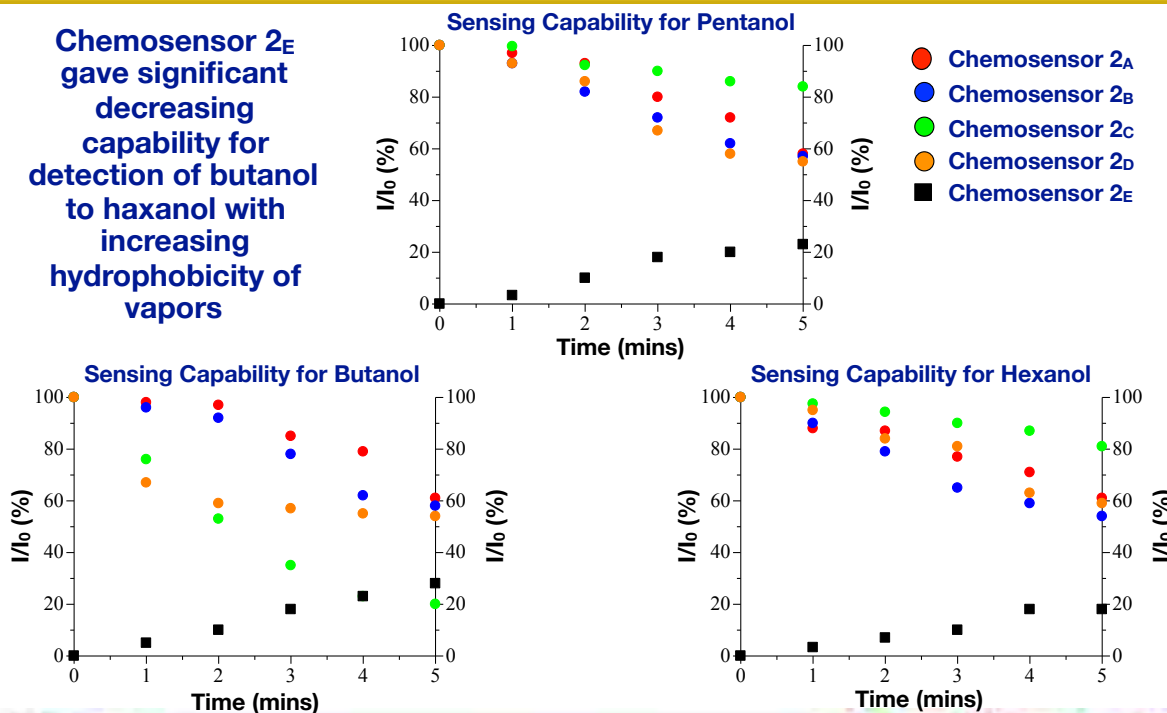
## Sensing Capability of Chemosensor 2<sub>A-E</sub>

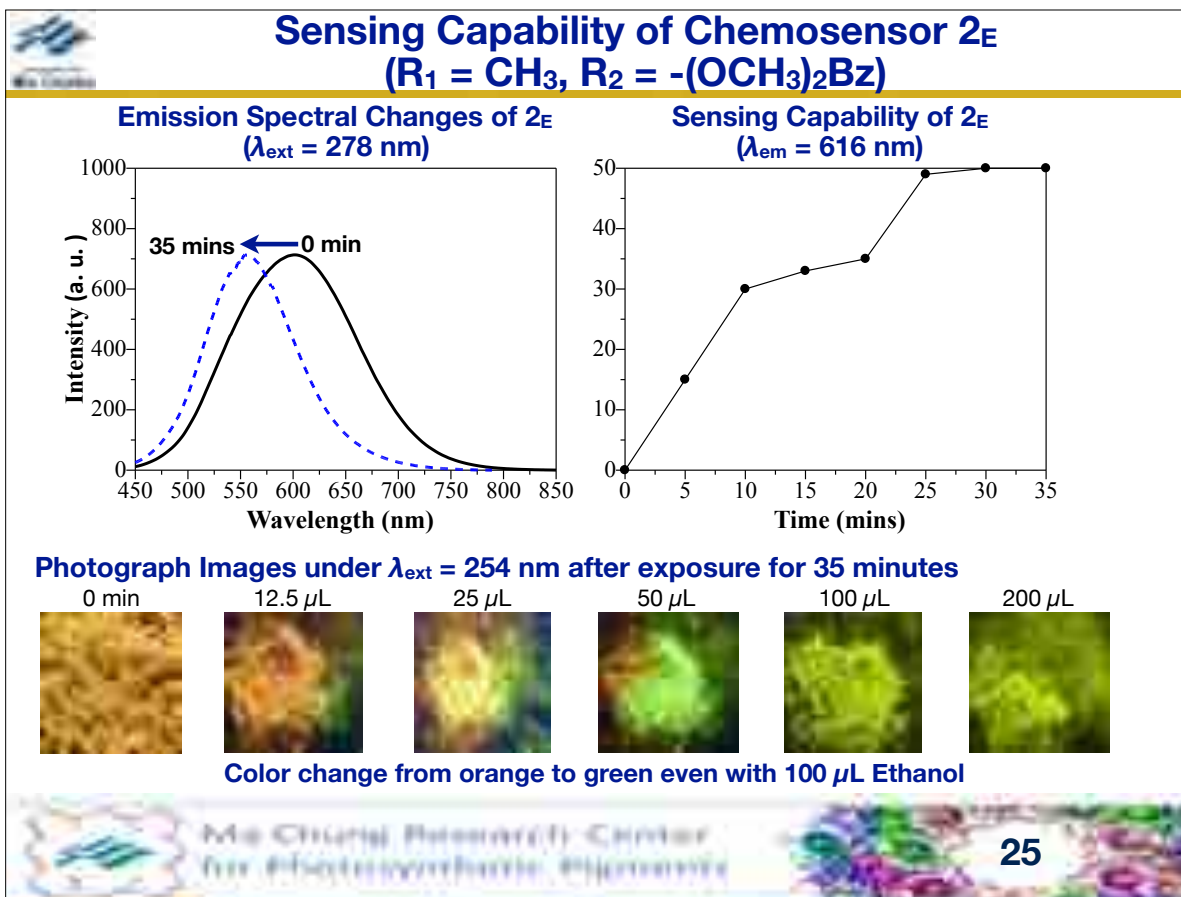
Chemosensor 2<sub>E</sub> still gave the best sensing capability for detection of methanol to propanol



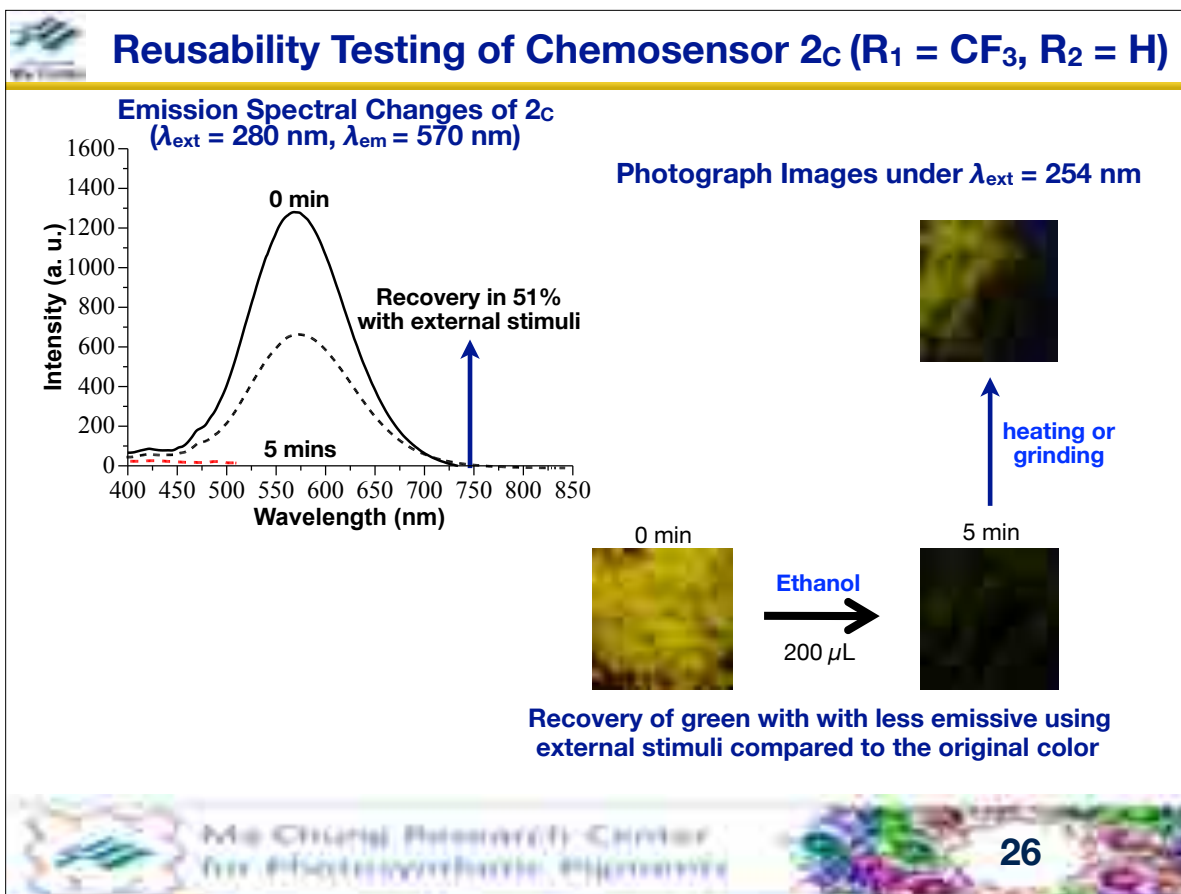
## Sensing Capability of Chemosensor 2<sub>A-E</sub>

Chemosensor 2<sub>E</sub> gave significant decreasing capability for detection of butanol to hexanol with increasing hydrophobicity of vapors





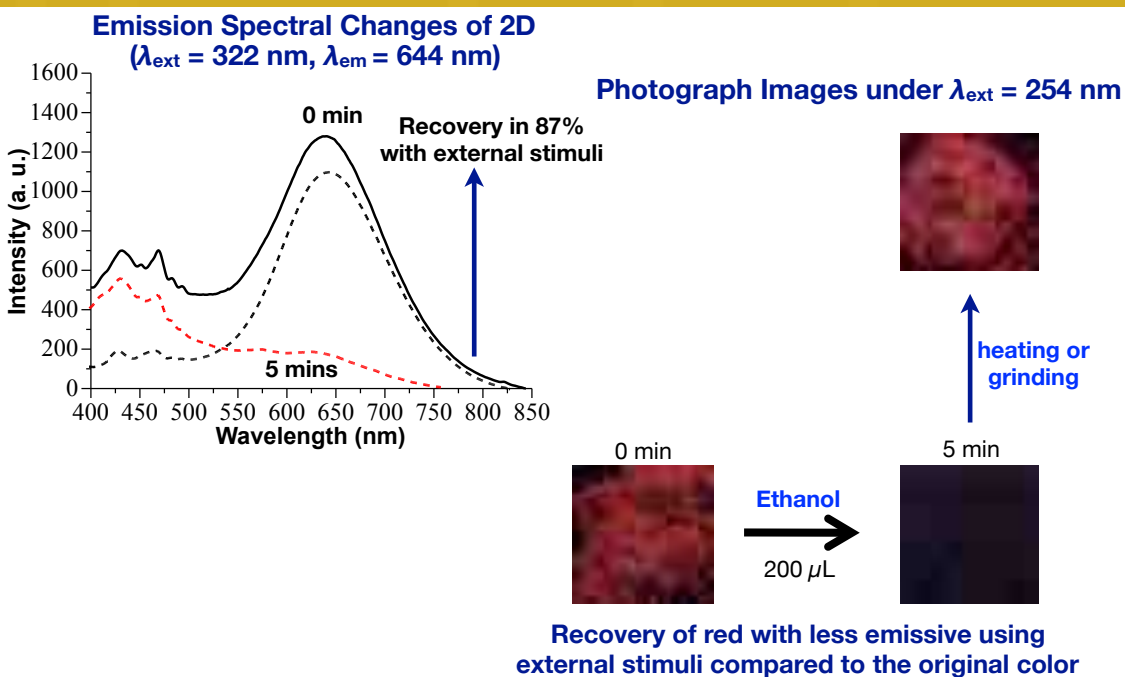
25



26



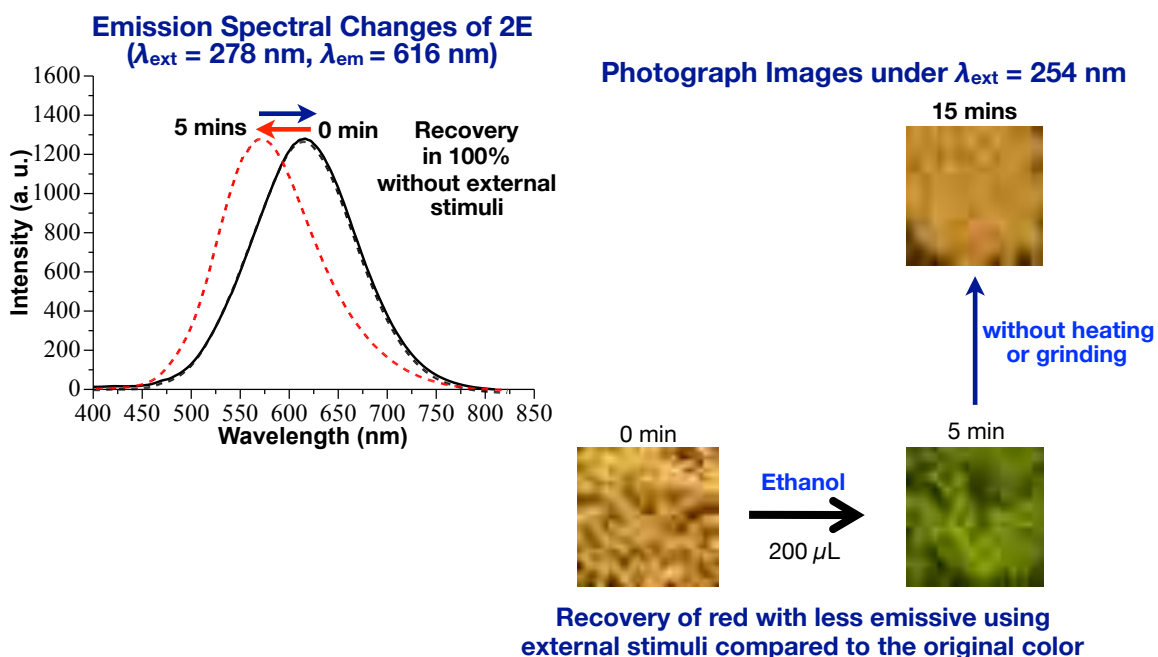
## Reusability Testing of Chemosensor 2<sub>D</sub> (R<sub>1</sub> = C<sub>6</sub>H<sub>5</sub>, R<sub>2</sub> = H)



27



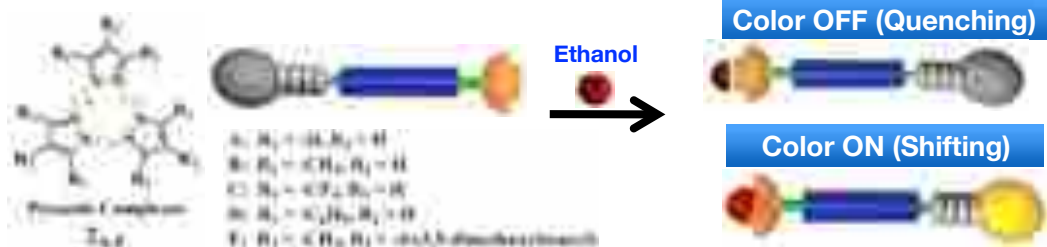
## Reusability Testing of Chemosensor 2<sub>E</sub> (R<sub>1</sub> = CH<sub>3</sub>, R<sub>2</sub> = -(OCH<sub>3</sub>)<sub>2</sub>Bz)



28



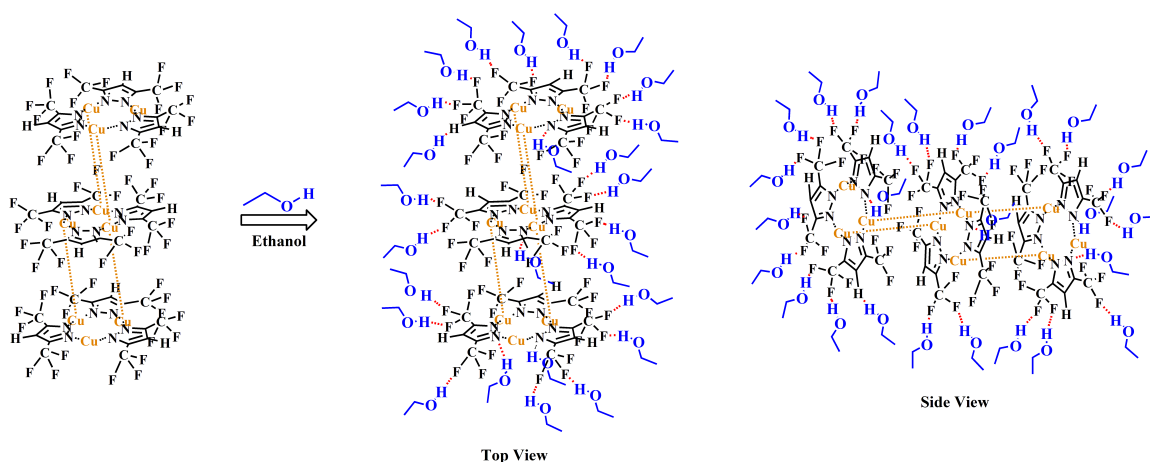
## Sensing Capability of Chemosensor 2<sub>A-E</sub>



Chemosensors	Sensitivity	Re-usability	Phenomena
2 <sub>A</sub>	Slow	No	Quenching
2 <sub>B</sub>	Slow	No	Quenching
2 <sub>C</sub>	Very Fast	51% (Heating)	Quenching
2 <sub>D</sub>	Medium	87% (Grinding)	Quenching
2 <sub>E</sub>	Very Fast	100% (Naturally)	Shifting



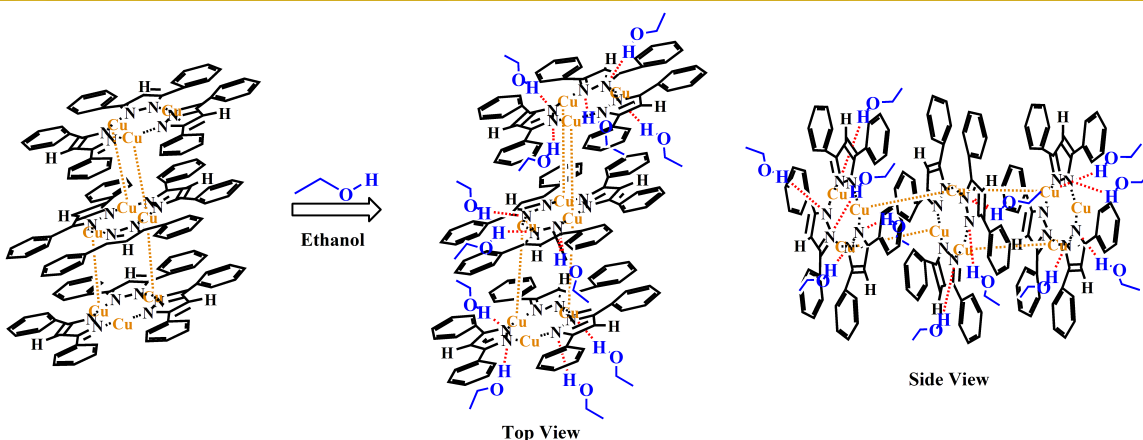
## Proposed Mechanism for Chemosensor 2<sub>C</sub>



★ Formation of intermolecular hydrogen bonding interaction between high electronegative F atom of CF<sub>3</sub> (trifluoromethyl) in *meso* position of the pyrazole ring to the OH of ethanol in the higher number (18 of F atoms)

★ Formation of intermolecular hydrogen bonding interaction between high electronegative N atom of the pyrazole ring to the OH of ethanol

## Proposed Mechanism for Chemosensor 2<sub>D</sub>



★ Formation of intermolecular hydrogen bonding interaction between high electronegative N atom of the pyrazole ring to the OH of ethanol

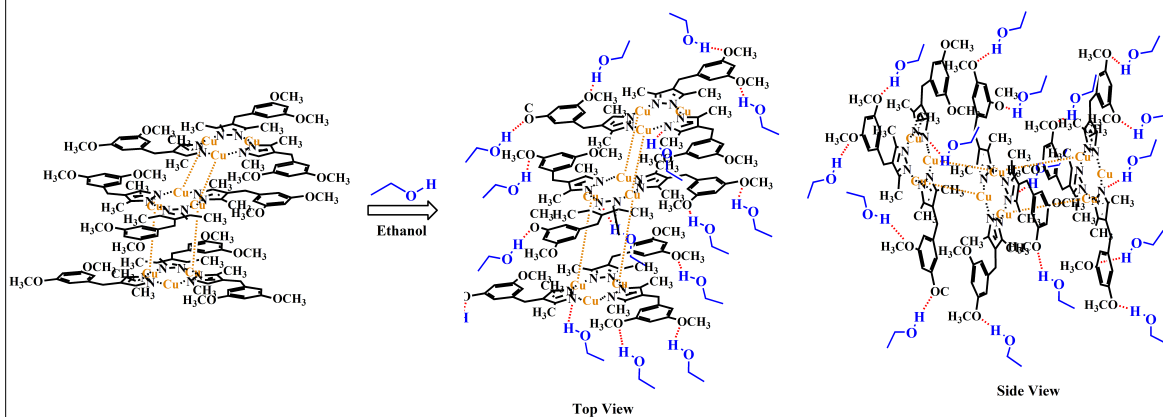
★ Formation of photoinduced energy transfer from the triplet excited state of the Cu(I)-Cu(I) interaction to the emission site of stacked phenyl ring at the *meso* position of the pyrazole ring with new emission band around 430-460 nm as a blue-green emission

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31

31

## Proposed Mechanism for Chemosensor 2<sub>E</sub>



★ Short distance of Cu(I)-Cu(I) interaction tends to encourage the formation of a weak intermolecular hydrogen bonding interaction between OH of ethanol with electronegative O atoms of the methoxy groups at the benzyl ring

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32

32



## Conclusions

1. Successful synthesis of trinuclear copper(I) pyrazolate complexes with different alkyl side-chains at the pyrazole rings as a powder with different color appearances under daylight in high yield.
2. Successful characterization of phosphorescent properties of trinuclear copper(I) pyrazolate complexes with different alkyl side-chains at the pyrazole rings as green, orange and red emission with lifetime at microsecond and a large Stokes shift.
3. Successful investigation of sensing capability of trinuclear copper(I) pyrazolate complexes with different alkyl side-chains at the pyrazole rings with chemosensors 2<sub>E</sub> as the best chemosensor for detection of methanol to propanol.



33

## Acknowledgements

Flagship Research Grant for NanoMalaysia CoE, Vol. No. R.J130000.7926.4S017, National Nanotechnology Directorate (NND), Ministry of Science, Technology and Innovation (MOSTI), Malaysia



Ma Chung Research Center for Photosynthetic Pigments (MRCPP), Universities Ma Chung, Indonesia for Financial Support as National Center of Excellence



34



## SUPRAMOLECULAR FUNCTIONAL NANOCHEMISTRY LABORATORY (RESEARCH GROUP)

# THANK YOU.....

- ☑ **Doctoral Students (7):**
  - Juan Matmin  
(Completed PhD)
  - Norsahika Mohd Basir  
(Completed PhD)
  - Nur Fatiha Ghazalli (D3)
  - Mohamad Azani (D3)
  - Nurliana Roslan (D3)
  - Abdul Hamid Umar (D3)
  - Nurul Husna Sabran (D1)
- ☑ **Master Students (1):**
  - Goh Cheow Kit (M1)

Picture with PhD Students

