

出國報告 (出國類別:參加國際會議)

## 第十九屆世界質譜研討會

服務機關：國立中興大學化學系

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派赴國家：日本

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## 摘要

世界質譜研討會主要爲了增進世界質譜技術的開發與應用，所舉辦之國際性會議，每兩年舉辦一次，本次舉辦的時間爲 9 月 15 日到 9 月 21 日，地點在日本京都，參與者多爲世界各地頂尖學術機構與相關質譜應用之工業界人士，皆爲此領域之代表性人士。今年參與人數近萬人，除了六場邀請演講外，共有千餘篇研究成果分別以口頭與海報方式發表。除了最新質譜技術與相關應用之學術成果發表之外，世界知名質譜廠商也在會議中發表最新開發出的產品與其應用。藉由參與本屆世界質譜研討會，可得知目前世界上質譜技術的趨勢，與目前質譜於不同領域上的最新應用。同時也可藉由廠商的介紹，得知目前世界上商業化質譜儀的最新進展及其實際的應用範圍。本次會議本人於九月二十日以海報方式發表研究論文，論文題目爲: *Determination of Prohibited Components in Hair Dyes by Solid Phase Extraction Coupled with Liquid Chromatography-Tandem Mass Spectrometry*，主要是以固相萃取法結合液相層析串聯質譜技術，分析市售染髮劑中微量之禁用成分。

## 目次

目的·····	1
出國行程與議程·····	3
心得及建議·····	14
照片集·····	15

## 壹、目的

世界質譜研討會(International Mass Spectrometry Conference, IMSC)是由世界質譜基金會(International Mass Spectrometry Foundation, IMSF)主辦，主要目的為增進質譜技術開發與應用，所舉辦之國際性會議，為目前除了每年所舉行之美國質譜學會年會外，另一個國際間重要質譜相關會議。本研討會每兩年舉辦一次，往年皆在歐洲舉行，本屆為首次移至歐洲之外的國家舉行。本屆會議於日本京都舉行，由日本質譜學會協辦，故本次大會與本年度日本質譜學會年會合辦。本研討會與會者大多來自世界各地學術機構與質譜應用工業界人士，皆為質譜領域中最具代表性人士。大會議程中，除質譜在各項領域應用技術之開發及質譜儀發展之前瞻性論文報告及海報論文外，並有全世界質譜儀各大廠牌之廠商產品展示和 workshop，以提供目前最新型商業化質譜儀功能與其在不同領域應用之介紹。大會約有近一萬人參加，與會人士來自世界各國，除各地區學術機構的專家學者外，也包含了生化藥廠、食品業、檢測業等不同領域產業界之先進。台灣方面除了指導教授中興大學化學系李茂榮教授參加外，與會者還包含台灣大學化學系何國榮教授、中山大學化學系謝建台教授、中研院化學所陳玉如教授等六十餘位國內頂尖質譜專家參與本次大會。會議含報告研討和壁報論文方式，所發表的均為目前質譜最新進展和技術，無論是口頭報告或壁報論文，皆以特性分組，包含藥物分析(drug analysis)、蛋白質體學(proteomics)、代謝體學(metabolomics)、脂質體學(lipidomics)、儀器、食品安全、質譜影像(imaging mass spectrometry)

等各種不同領域。藉由參與世界質譜研討會，可以得知目前世界上最先進的質譜技術與研究方向，並可以與來自不同地區的質譜學家進行交流。

## 貳、出國行程與議程

本次大會除了世界質譜研討會外，同時舉行日本質譜學會年會，因本次大會前兩天主要是以日本質譜學會年會以及廠商的 workshop 為主，故筆者由大會第三天(九月十七日)開始參與。

九月十六日：搭乘日本航空公司班機由桃園機場直飛日本大阪關西機場，再從關西機場搭乘日本國鐵至本次大會舉辦地點京都。

九月十七日：早上報到後即參加大會演講，本日之大會演講者為美國印地安那大學(Indiana University)化學系 David Clemmer 教授，Clemmer 教授為目前世界知名離子移動能譜(ion mobility spectrometry)之專家。利用層析技術結合質譜技術分析複雜基質樣品中超微量成分，受限於干擾物之影響，因此仍有其缺陷，若能多增加一維的分離系統，對於複雜基質的分析有絕大的助益，目前以結合離子移動能譜與質譜技術之分析技術最受矚目。Clemmer 教授在演講中介紹離子移動能譜過去之發展及相關應用外，同時針對該技術在未來於技術之改良與在不同領域的應用，由其實針對離子移動能譜術解析度方面之提升，有詳細的描述與討論。大會演講後分為五場不同領域之口頭報告，與會者可參加同一領域之演講或僅針對個人有興趣的題目進行聆聽。筆者除了聆聽軌道阱質譜儀(Orbitrap)發明者 Dr. Alexander Makarov 針對最新軌道阱在串聯質譜技術(tandem mass spectrometry)之應用外，其餘時間皆參加質譜影像 (imaging mass spectrometry)領域之口頭報告。質譜影像為目前質譜領域中最新的發展，最主要的功能為可針

對所分析的組織中，不同物質流佈之分析。而在下午場的口頭報告中，除了影像質譜領域之聆聽外，也參加離子移動能譜-質譜技術領域之發展與應用領域之口頭報告。在口頭報告的空檔主要為觀看海報論文，並且與發表者進行內容之討論。

九月十八日：第四天大會演講者為荷蘭 Utrecht University 的 Albert Heck 教授，Heck 教授除了在 Utrecht University 執教外，同時也任職於荷蘭蛋白質體中心，而 Heck 教授主要是針對質譜技術在蛋白質體分析與結構生物學(structural biology)之應用。近年來由於質譜技術於蛋白質體學之應用，促使質譜儀每年也有相當進展，而使得蛋白質體學能獲得更進一步的結果，也使得結構生物學之研究有相當的進步。除了海報論文外，口頭報告部分，主要是參加質譜微小化(mass spectrometry miniaturized)與質譜技術於疾病診斷(mass spectrometric diagnosis)領域之報告。

九月十九日：目前除了蛋白質體學是大家所重視的領域外，代謝體學(metabolomics)、醣質體學(glycomics)與脂質體學(lipidomics)也是系統生物學(system biology)中受到大家重視的學門，主要原因為上述三種不同的分子，在生物系統的行爲中，扮演相當重要的角色。而質譜技術在此三領域的應用，無論在質譜學或生物學中，逐漸受到大家的重視。本日即針對此三領域的口頭報告，選擇自己有興趣的題目進行聆聽，其中包含大陸長春應化所劉淑瑩教授，劉教授同時也為大陸人蔘研究所所長，主要是利用質譜技術於人蔘中具有活性的皂苷類化合物與多醣體之分析與鑑定。劉教授在本次大會中主要針對利用基質輔助

雷射脫附游離法與電灑游離法質譜術於人蔘中寡醣體(oligosaccharide)與單醣體(monosaccharide)之鑑定，主要即利用質譜技術去判定人蔘寡醣中個單醣的接法，由於不同的接法會對於該寡糖活性有很大之影響，因此也可突顯分析鑑定單醣接法之重要性。

九月二十日：由於目前對於複雜基質中小分子的定量分析逐漸追求更低之偵測極限，因此層析結合質譜技術也成為目前最主要被應用的分析偵測技術。因此相較於前幾天的口頭報告主要是注重於質譜技術於蛋白質體學、代謝體學等生化應用上，今日大部分口頭報告大多為質譜技術於小分子的定性與定量分析。因此今日所聆聽的口頭報告也著重於目前質譜技術於定量分析之應用，特別是在環境分析上之應用。此外，也聆聽 UCLA 大學 Joseph Loo 教授之口頭報告。Loo 教授研究主要為開發生物質譜技術於蛋白質結構之鑑定以及生物質譜技術於蛋白質體學與疾病生物標誌(disease biomarkers)之應用。在演講中，Loo 教授介紹利用離子移動能譜技術結合不同解離方式之串聯質譜技術，於與愛滋海默症、帕金森氏症相關的蛋白質分析鑑定之應用。而筆者的論文也於今日以海報的方式發表，所發表的研究主要是開發一固相萃取-液相層析串聯質譜法分析市售染髮劑中禁用成分之分析，並於大會所規定的時間內於海報前接受與會者的問題並給予回答，藉此達到學術交流之目的。

九月二十一日：本日大會演講的演講者為美國 Vanderbilt University 的 Richard Caprioli 教授，Caprioli 教授為目前世界在質譜影像技術中最受矚目的學者，在演講中介紹以基質輔助雷射脫附游離法



(matrix-assisted laser desorption/ionization, MALDI)爲主之質譜影像術於蛋白質體、代謝體等不同領域之應用。演講內容主要介紹如何在最短時間內獲得更高解析度的質譜影像，利用 Caprioli 教授所開發之方法可在十分鐘內獲得老鼠腦部組織的質譜影像圖，解析度可高達 1-10 微米( $\mu\text{m}$ )。而本次大會最後一場邀請演講講者爲普渡大學的 Graham Cooks 教授，Cooks 教授爲目前世界上最著名的質譜學者之一，在質譜的發展中貢獻良多，Cooks 教授在本次演講中描述質譜進展之過去、現在與未來展望。演講中 Cooks 教授談及雖然目前質譜技術發展速度相當快，但是質譜儀所需的空間與相關環境如電源、氣體源等仍須要相當大的空間，若能夠將目前質譜儀微小與簡單化，即可廣泛應用於個人或家庭之分析中，此爲 Cooks 教授對於質譜技術未來展望之期待。在此兩場大會邀請演講中，仍參加不同領域的口頭報告，主要是聆聽離子捕捉技術之進展與在生化分析之應用以及大氣壓質譜法 (ambient mass spectrometry)中所開發之新技術與該技術在不同領域之應用。

九月二十二日：於京都搭乘日本關西地區之鐵路系統至關西機場，搭乘日本航空公司班機從關西回到桃園機場，完成本次參與世界質譜研討會之行程。

# 大會議程

## 第一天議程

**Saturday, 15<sup>th</sup> September**

	Main Hall	Room A	Room 103	Room C-1	Room C-2
10:00		10:00-17:00	10:00-17:00	10:00-17:00	10:00-17:00
11:00					
12:00					
13:00					
14:00	14:00-16:00	Users' Day (AB SCIEX)	Short Course Fragmentation Methods, Their Fundamentals and Application in Proteomics Roman A Zubarev	Short Course Introduction to Imaging Mass Spectrometry Mitsutoshi Setou	Short Course Fundamentals of Mass Spectrometry O David Sparkman & Jürgen Gross
15:00	Open Lectures for General Public (in Japanese)				
16:00					
17:00					
18:00					
19:00					

第二天議程

Sunday, 16<sup>th</sup> September

	Main Hall	Room A	Room C-2	Swan
10:00		10:00-15:00	10:00-15:00	
11:00				
12:00				
13:00		<b>Users' Day</b> (SHIMADZU)	<b>Short Course</b> <b>Fundamentals of</b> <b>Mass Spectrometry</b> O David Sparkman & Jürgen Gross	
14:00				
15:00				
16:00	15:30-16:15 <b>Tutorial (Plenary)</b> <b>Lecture 1</b> Nico M M Nibbering			
	16:15-17:00 <b>Tutorial (Plenary)</b> <b>Lecture 2</b> Michael L Gross			
17:00	17:00-17:30 <b>Opening Ceremony</b>			
18:00	17:30-18:15 <b>Plenary Lecture 1</b> Hiroyuki Hamada			
19:00				18:30-20:00 <b>Welcome Mixer</b>

# 第三天議程

Monday, 17<sup>th</sup> September

Life Sciences	Medical Sciences	Fundamentals	Isotope Ratio MS
Instrumentation	Ionization	Environment / Microorganism	

	Main Hall	Room A	Room B-1	Room D	Room E	Event Hall
8:00						7:30-8:00 Mounting Posters
	8:00-8:45 <b>Plenary Lecture 2</b> David E Clemmer					Poster Viewing Time
9:00	9:00-11:00 <b>Session 1</b> Developments in Tandem Mass Spectrometry - Hybrid Instrumentation "The whole is greater than the sum of its parts" (Aristotle). Chair: Morio Ishihara Keynote: Alexander A Makarov	9:00-11:00 <b>Session 2</b> Advances in Methods and MS Instrumentation for Biomolecule Characterization Chair: Vicki H Wysocki Keynote: Andrea Sinz	9:00-11:00 <b>Session 3</b> Structures and Dynamics of Atomic and Molecular Clusters Chair: Fuminori Misaizu Keynote: Knut R Asmis	9:00-11:00 <b>Session 4</b> Imaging-I Chair: Mitsutoshi Setou Keynote: Ron Heeren	9:00-11:00 <b>Session 5</b> Advances in Spray Ionization Techniques Chair: Richard B Cole Keynote: Kentaro Yamaguchi	
11:00						11:10-12:20 Poster Core Time (Odd-number)
12:00						
13:00	12:20-13:30 <b>Luncheon Seminar</b> (SHIMADZU)	12:20-13:30 <b>Luncheon Seminar</b> (AB SCIEX)	12:20-13:30 <b>Luncheon Seminar</b> (Waters)	12:20-13:30 <b>Luncheon Seminar</b> (JEOL)	12:20-13:30 <b>Luncheon Seminar</b> (Agilent Technologies)	Poster Viewing Time
14:00						13:30-14:40 Poster Core Time (Even-number)
15:00	15:00-17:00 <b>Session 6</b> Novel Approaches in Proteomics Analysis Chair: Roman Zubarev Keynote: Joshua J Coon	15:00-17:00 <b>Session 7</b> New Ionization Methods and Related Topics for the Next Generation Chair: Kenzo Hiraoka Keynote: Robert Cody	15:00-17:00 <b>Session 8</b> Collision Dynamics and Spectroscopy Using Ion Storage Rings and Traps Chair: Toshiyuki Azuma Keynote: Steen Brøndsted Nielsen	15:00-17:00 <b>Session 9</b> Imaging-II Chair: Jiro Matsuo Keynote: Nick Winograd	15:00-17:00 <b>Session 10</b> Ion Mobility Spectroscopy Based on Instrument & Theoretical Development Chair: Toshiaki Sugai Keynote: Alexandre A Shwartsburg	Poster Viewing Time
16:00						
17:00						17:00-17:30 Removing Posters
		17:15-19:15 <b>3<sup>rd</sup> Asian-Oceanic MS Conference (AOMSC3) Day-1</b>	17:15-19:15 <b>Workshop 1</b> Mass Spectrometry of Polymers and Industrial Materials Organizer: Hiroaki Sato	17:15-19:15 <b>Workshop 2</b> Hydrogen/Deuterium Exchange Mass Spectrometry Organizer: Yoshitomo Hamuro, Rachel Garlish	(Reserved for MSSJ) (Local Meeting)	
18:00						
19:00						

## 第四天議程

Tuesday, 18<sup>th</sup> September

	Life Sciences Instrumentation	Medical Sciences Ionization	Fundamentals Environment / Microorganism	Isotope Ratio MS		
	Main Hall	Room A	Room B-1	Room D	Room E	Event Hall
						7:30-8:00 Mounting Posters
8:00	8:00-8:45 <b>Plenary Lecture 3</b> Albert J R Heck					Poster Viewing Time
9:00	9:00-11:00 <b>Session 11</b> Glycomics: From Disease Markers to Therapeutic Antibody Products Chair: Hyun joo An Keynote: Carlito Lebrilla	9:00-11:00 <b>Session 12</b> On-site Mass Spectrometry -Miniaturized Instruments and Allied Technologies- Chair: Shuichi Shimma Keynote: Zoltán Takáts	9:00-11:00 <b>Session 13</b> Accelerator Mass Spectrometry Chair: Hiroyuki Matsuzaki Keynote: David Fink	9:00-11:00 <b>Session 14</b> Ion-surface Collisions: Collision-induced Dissociation and Soft Landing Chair: Jean Futrell Keynote: Julia Laskin	9:00-11:00 <b>Session 15</b> Mass Spectrometry for Nuclear Applications and Safety Chair: Jinying Li Keynote: Huanwen Chen	
10:00						
11:00						11:10-12:20 Poster Core Time (Odd-number)
12:00						Poster Viewing Time
13:00	12:20-13:30 <b>Luncheon Seminar</b> (Thermo Fisher Scientific)	12:20-13:30 <b>Luncheon Seminar</b> (AB SCIEX)	12:20-13:30 <b>Luncheon Seminar</b> (Waters)	12:20-13:30 <b>Luncheon Seminar</b> (SHIMADZU)	12:20-13:30 <b>Luncheon Seminar</b> (Advion)	
14:00						13:30-14:40 Poster Core Time (Even-number)
15:00	15:00-17:00 <b>Session 16</b> Glycoanalytical Technology for Systems Glycobiology and Functional Glycomics Chair: Jane Thomas-Oates Keynote: Pauline M Rudd	15:00-17:00 <b>Session 17</b> Non-Covalent Ion-Molecule Interactions Chair: Seung-Koo Shin Keynote: Peter B Armentrout	15:00-17:00 <b>Session 18</b> Advances in Resolution and Accuracy of Isotope Ratio Analyses Chair: Takafumi Hirata Invited: Jochen Vogl	15:00-17:00 <b>Session 19</b> Mass Spectrometric Diagnosis Chair: Toyofumi Nakanishi Keynote: Renato Zenobi	15:00-17:00 <b>Session 20</b> The Ion formation and Dissociation Mechanisms in MALDI Chair: Myung Soo Kim Keynote: Richard Knochenmuss	Poster Viewing Time
16:00						
17:00						17:00-17:30 Removing Posters
18:00		17:15-19:15 <b>3<sup>rd</sup> Asian-Oceanic MS Conference (AOMSC3) Day-2</b>	17:15-19:15 <b>Workshop 3</b> Careers in Mass Spectrometry Organizer: Tony Bristow	17:15-19:15 <b>Workshop 4</b> Mass++ and MassBank: Tools for Data Processing and Database on PC Organizer: Satoshi Tanaka, Takaaki Nishioka	17:15-19:15 <b>(Reserved for MSSJ Local Meeting)</b>	
19:00						

# 第五天議程

Wednesday, 19<sup>th</sup> September

Life Sciences	Medical Sciences	Fundamentals	Isotope Ratio MS
Instrumentation	Ionization	Environment / Microorganism	

	Main Hall	Room A	Room B-1	Room D	Room E	Event Hall
8:00						7:30-8:00 Mounting Posters
	8:00-8:30 Thomson Medal and Curt Brunnee Award Ceremony					Poster Viewing Time
	8:30-8:50 Curt Brunnee Award Lecture					
9:00	9:00-11:00 <b>Session 21</b> Platform Technology for Metabolomics Chair: Yoshiya Oda Keynote: Annie Evans	9:00-11:00 <b>Session 22</b> Instrumentation Developments in Mass Spectrometric Imaging Chair: Anastasios Giannakopoulos Keynote: Bernhard Spengler	9:00-11:00 <b>Session 23</b> Gas Phase Fragmentation Mechanisms of Biomolecular Radicals Chair: Shigeo Hayakawa Keynote: Richard A J O'Hair	9:00-11:00 <b>Session 24</b> Regulated Bioanalysis Chair: Shinobu Kudoh Keynote: Tatsuo Kurokawa	9:00-11:00 <b>Session 25</b> New Approaches to Defining the Diversity of Glycans Chair: Catherine E Costello Keynote: Jane Thomas-Oates	
10:00						Poster Core Time (Odd-number)
11:00						
12:00						Poster Viewing Time
12:20-13:30	12:20-13:30 Luncheon Seminar (Bruker)	12:20-13:30 Luncheon Seminar (AB SCIEX)	12:20-13:30 Luncheon Seminar (Waters)	12:20-13:30 Luncheon Seminar (SHIMADZU)	12:20-13:30 Luncheon Seminar (Agilent Technologies)	
13:00						Poster Core Time (Even-number)
14:00						
15:00	15:00-17:00 <b>Session 26</b> Lipidomics : Recent New Techniques and Applications Chair: Stephen Blanksby Keynote: Gavin E Reid	15:00-17:00 <b>Session 27</b> Progress in Microbiology Chair: Catherine Fenselau Keynote: Jeremy K Nicholson	15:00-17:00 <b>Session 28</b> IR Spectroscopy of Gas-phase Ions Chair: Bela Paizs Keynote: Philippe Maitre	15:00-17:00 <b>Session 29</b> The Advances in Biological Mass Spectrometry in Drug Discovery and Development: Current State of the Art and Challenges Chair: Ajai Chaudhary Keynote: Ragu Ramanathan	15:00-17:00 <b>Session 30</b> Data Processing and Informatics for SIMS Chair: DaeWon Moon Keynote: David Castner	Poster Viewing Time
16:00						
17:00						17:00-17:30 Removing Posters
			17:15-19:15 <b>Workshop 5</b> Path to Next-Generation IMS: New Concepts, Advanced Instrumentation, and Leveraging the Ion-molecule Chemistry Organizer: Toshiki Sugai, Alexandre Shvartsburg	17:15-19:15 <b>Workshop 6</b> Mass Spectrometry for Food Safety Organizer: Jen-tai Shiae	17:15-19:15 (Reserved for MSSJ Local Meeting)	
18:00						
19:00						

# 第六天議程

Thursday, 20<sup>th</sup> September

		Life Sciences Instrumentation	Medical Sciences Ionization	Fundamentals Environment / Microorganism	Isotope Ratio MS	
	Main Hall	Room A	Room B-1	Room D	Room E	Event Hall
						7:30-8:00 Mounting Posters
8:00	8:00-8:45 <b>Plenary Lecture 4</b> Hisayoshi Yurimoto					Poster Viewing Time
9:00	9:00-11:00 <b>Session 31</b> Native Mass Spectrometry and Structural Biology Chair: Satoko Akashi Keynote: Joseph A Loo	9:00-11:00 <b>Session 32</b> Formation and Dissociation of Peptide Radical Ions Chair: Dominic T W Chan Keynote: Roman A Zubarev	9:00-11:00 <b>Session 33</b> JMS Award Symposium Chair: Richard M Caprioli (Editor-in-Chief, JMS)	9:00-11:00 <b>Session 34</b> MS Informatics for Identification and Characterization Chair: Shigeki Kajihara Keynote: David Fenyo	9:00-11:00 <b>Session 35</b> Environment I Chair: Peter Haglund Keynote: Terry Bidleman	
10:00						
11:00						11:10-12:20 Poster Core Time (Odd-number)
12:00						
13:00	12:20-13:30 <b>Luncheon Seminar</b> (SHIMADZU)	12:20-13:30 <b>Luncheon Seminar</b> (AB SCIEX)	12:20-13:30 <b>Luncheon Seminar</b> (Waters)	12:20-13:30 <b>Luncheon Seminar</b> (AMR INCORPORATED)	12:20-13:30 <b>Luncheon Seminar</b> (Bruker)	Poster Viewing Time
14:00						13:30-14:40 Poster Core Time (Even-number)
15:00	15:00-17:00 <b>Session 36</b> Advances in Ion Mobility Mass Spectrometry Chair: Joseph A Loo Keynote: Michael T Bowers	15:00-17:00 <b>Session 37</b> Challenges in High Resolution and High Accuracy Mass Measurement Mass Spectrometry Chair: Evgeny Nikolaev Keynote: Alan G Marshall	15:00-17:00 <b>Session 38</b> Mass Spectrometry for Metabolic Diseases Chair: Makoto Yoshino, Seiji Yamaguchi	15:00-17:00 <b>Session 39</b> MS Informatics for Quantitation Chair: David Fenyo Keynote: Jurgen Cox	15:00-17:00 <b>Session 40</b> Environment II Chair: Takeshi Nakano Keynote: Peter Haglund	Poster Viewing Time
16:00						
17:00	17:10-18:10 <b>Thomson Medal Award Lectures</b>					17:00-17:30 Removing Posters
18:00						
19:00						Grand Prince Hotel Kyoto 18:30-21:00 Banquet

9:00-18:00 Exhibition (Event Hall & Room C-1)

# 第七天議程

Friday, 21<sup>st</sup> September

Life Sciences	Medical Sciences	Fundamentals	Isotope Ratio MS
Instrumentation	Ionization	Environment / Microorganism	

	Main Hall	Room A	Room B-1	Room D	Room E	Swan
8:00	8:00-8:45 <b>Plenary Lecture 5</b> Richard M Caprioli					
9:00	9:00-11:00 <b>Session 41</b> Chemistries of Trapped Ions and their Applications to Biological Mass Spectrometry Chair: Gavin E Reid Keynote: Scott A McLuckey	9:00-11:00 <b>Session 42</b> New Developments in Instruments and Detectors Chair: Takaya Sato Keynote: Evgeny N Nikolaev	9:00-11:00 <b>Session 43</b> Novel Proteomics Methodologies Chair: Yasushi Ishihama Keynote: Michael J MacCoss	9:00-11:00 <b>Session 44</b> Ambient Ionization Chair: Jentale Shlea Keynote: Kenzo Hiraoka	9:00-11:00 <b>Session 45</b> Cell Biology / Cellular Pathways Chair: Renato Zenobi Keynote: Tsutomu Masujima	
10:00						
11:00	11:15-11:45 <b>IMSC 2014 Geneva Presentation</b>					
12:00	11:45-12:30 <b>Plenary Lecture 6</b> R Graham Cooks					
	12:30-13:00 <b>Closing Ceremony</b>					
13:00						13:00-13:45 <b>ARIGATO &amp; SAYONARA</b>
14:00						



## 心得及建議

本次會議含報告研討和壁報論文方式，參與者能廣泛交換經驗心得，大家感到受益良多。雖然大會議程只有短短幾天，但所發表的均是目前當今質譜技術的最新進展，無論是口頭報告或壁報論文，包含儀器特性、環境應用、生化大分子、蛋白質體、代謝體、醣質體學、脂質體學等不同領域之應用，每個人可選擇與自己本身研究相關，或有研究興趣的論文進行聽講與閱讀，並與發表者直接面對面討論研究內容。自從電灑游離法與基質輔助雷射脫附游離法開發後，近幾年來論文的約有一半以上均偏向生化方面的題材，即利用質譜所能提供的資訊，協助解決系統生物學所面臨的問題，由此可見，質譜科學於生化領域之研究是不可缺少之利器。此外，近年來質譜影像技術逐漸受到大家重視，特別是應用此技術於腦中脂質之流佈，並由特定化合物的流佈，對於實際動物或植物所產生的行為與反應，能有更合理與更進一步之解釋。藉由參與世界質譜研討會等重要國際會議可讓研究人員能獲得目前世界上最新的研究進展，並可以與其他國家頂尖的專家學者們直接進行交流，對於拓展研究人員的國際觀亦有相當大助益，因此建議研究人員能多多參與國際會議，使得國內的研究能逐漸與國際同步。然而，常常會礙於經費的關係，導致許多研究人員無法參與重要國際會議，因此希望未來能多補助研究人員參與國際會議，以增進研究之水準以及與世界上相關領域專家之交流。

# 照片集



本次研討會於日本國立京都國際會館 (Kyoto International Conference Center) 舉行

## Determination of Prohibited Components in Hair Dyes by Solid Phase Extraction Coupled with Liquid Chromatography-Tandem Mass Spectrometry

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### Introduction

In the oxidative hair dyes, the mixture of p-phenylenediamine, benzotriazole, amino-phenols, sulfates, and other biologically active aromatic substrates are used as dye precursors in the process of hair coloring and widely used as primary intermediates and/or couplers in the formulation. The dye and its intermediates have been proved to have toxic, mutagenic or other carcinogenic properties in vivo. Due to the toxicity of these components, the contents of components and intermediates in the formulations of oxidative hair dyes are prohibited or restricted by the European Council Directive (75/318/EEC). This completely used for hair dye, including 2,4-diaminobenzoic acid, 2,4-diamino-diphenylamine, HC Yellow No.12, 2,3-naphthalenediol and 2,3-diaminobenzoic acid are prohibited not only by European Council Directive but also by China and Taiwan. The p-phenylenediamine (PPD), an aromatic amine, has been widely used as an ingredient of permanent hair dye formulation. Because provoking the allergic of human, the concentration of PPD used in the hair dye was restricted by European Council Directive, China, and Taiwan ranged between 2-6%. Therefore, developing of an effective analytical method for routine analysis of these components in hair dyes is necessary. In this study, a high sensitive and selective extraction method, SPE combined with LC-MS/MS, for determination of 2,4-diaminobenzoic acid, 2,4-diamino-diphenylamine, HC Yellow No.12, 2,3-naphthalenediol, 2,3-diaminobenzoic acid, and p-phenylenediamine in hair dyes was developed. The SPE conditions were systematically studied and the validation of proposed method was also evaluated. The feasibility of applying the developed method to determine prohibited and restricted components in real hair dyes obtained from local markets was examined.

### Instrument

Mass spectrometer: ThermoFinnigan TSQ Quantum Ultra ESI  
LC system: ThermoFinnigan Acela LC system  
LC column: Waters XBridge C18 (150 mm x 2.1 mm, 5 μm)  
Mobile phase: A: 1% acetic acid in water containing 0.1% formic acid and 0.05% HFBA; B: methanol (MeOH)  
Flow rate: 0.10 mL/min  
Separation gradient:

Time (min)	A (%)	B (%)
0.0	99	1
1.0	99	1
10.0	9	91
17.0	9	91
18.0	9	91
20.0	99	1

Chemical structures of six restricted and five prohibited components:

- p-phenylenediamine
- 2,4-diaminobenzoic acid
- 2,3-diaminobenzoic acid
- 2,4-diaminodiphenylamine
- HC Yellow No.12
- 2,3-naphthalenediol

### The optimal SPE conditions for extracting six analytes in hair dye samples

Steps	Condition
Sample	SPE cartridge (20 mg)
Condition	1 mL of MeOH and 1 mL of pH 2 buffer solution
Load	1 mL of hair dye sample
Washing	3.0 mL pH 8 buffer solution
Eluting	1 mL of MeOH/methanol (1/1) (20 min)

### Table 1: Tandem mass spectrometry parameters for SRM determination of prohibited and restricted compounds

Compound	Retention time (min)	Priority	Parent Ion (m/z)	Quantitative Ion (m/z)	Confirming Ion (m/z)
2,4-diaminodiphenylamine	11.82	+	206	183 (177)	198 (216)
2,4-diaminobenzoic acid	8.88	+	139	124 (14)	198 (116)
HC Yellow No.12	14.28	+	217	188 (11)	114 (23)
2,3-naphthalenediol	11.96	-	198	183 (24)	139 (20)
2,3-diaminobenzoic acid	4.45	+	139	124 (17)	187 (15)
p-phenylenediamine	4.38	+	198	82 (15)	82 (2)

### Table 2: Concentrations of prohibited and restricted compounds in hair dye samples (μg/mL)

Compound	Recovery (%)	MSD (%)				
p-phenylenediamine	180	736	2360	108	750	2360
2,4-diaminobenzoic acid	84.8	168	124	10.3	3.8	4.3
2,4-diaminodiphenylamine	192	117	111	8.3	6.7	1.9
2,3-diaminobenzoic acid	82.6	106	109	6.6	3.3	2.3
2,4-diaminodiphenylamine	86.2	108	110	13.1	8.7	4.6
2,3-naphthalenediol	83.8	164	128	8.1	5.3	2.8
HC Yellow No.12	84.1	85.1	39.8	8.4	3.6	1.5

### Table 3: Concentrations of prohibited and restricted compounds in hair dye samples (μg/g)

Compound	S1*	S2	S3	S4	S5	S6
p-phenylenediamine	38894	84428	N.D.	N.D.	N.D.	2.28
2,4-diaminobenzoic acid	2.1	N.D.	N.D.	N.D.	N.D.	N.D.
2,5-diaminobenzoic acid	190	58	N.D.	N.D.	N.D.	N.D.
2,4-diaminodiphenylamine	N.D.	N.D.	N.D.	N.D.	8.78	N.D.
2,3-naphthalenediol	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
HC Yellow No.12	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.

\* S1-S6 were purchased from local markets in Taipei city and Taichung city (N.D., not detected).

### Mass ion chromatograms of 2.5 μg/mL of six analytes in hair dye produced by SPE-LC-MS/MS:

- (a) Total ion chromatogram (TIC); (b) 2,3-naphthalenediol; (c) p-phenylenediamine; (d) 2,4-diaminobenzoic acid (RT: 4.45 min); 2,4-diaminobenzoic acid (RT: 8.88 min); (e) 2,4-diaminodiphenylamine; (f) HC Yellow No.12

### Mass ion chromatograms of real sample S1 produced by SPE-LC-MS/MS:

- (a) Total ion chromatogram (TIC); (b) 2,3-naphthalenediol; (c) p-phenylenediamine; (d) 2,4-diaminobenzoic acid (RT: 4.45 min); 2,4-diaminobenzoic acid (RT: 8.88 min); (e) 2,4-diaminodiphenylamine; (f) HC Yellow No.12

### Analytical characteristics of proposed method for prohibited and restricted compounds in hair dye

Compound	Linear range (ng/mL)	Coefficient of determination (R <sup>2</sup> )	LOD* (ng/mL)	LOQ† (ng/mL)
p-phenylenediamine	10-2000	0.9972	0	10
2,4-diaminobenzoic acid	10-2000	0.9928	3	10
2,4-diaminodiphenylamine	10-2000	0.9922	4	14
2,3-naphthalenediol	20-2000	0.9942	2	10
HC Yellow No.12	10-2000	0.9978	4	10

### Intra- and inter-day precision of the QC samples (ng/mL) of prohibited and restricted compounds in hair dye expressed by RSD (%)

Compound	Intra-day (n=3)			Inter-day (n=3)		
	100	750	2360	100	750	2360
p-phenylenediamine	2.3	2.7	3.4	8.8	5.7	4.8
2,4-diaminobenzoic acid	3.1	3.2	8.8	10.6	8.6	7.8
2,4-diaminodiphenylamine	19.2	3.6	2.4	18.3	7.8	9.2
2,4-diaminodiphenylamine	15.4	4.8	1.5	17.1	14.3	8.4
2,3-naphthalenediol	19.4	1.6	18.1	12.4	18.4	18.4
HC Yellow No.12	18.3	3.4	1.8	18.1	7.8	2.2

### Conclusions

An SPE-LC-MS/MS method was developed to determine five prohibited and one restricted components in hair dye. In SPE procedure, the highest extraction efficiency was obtained by using the SPE cartridge and a mixed solvent of 0.1% acetic acid in water (1:100) as eluting solvent. The proposed method could be offered a wide linearity over 10,000 ng/mL, and good linearity with an R.S.D. less than 10%. The LODs of developed method for all analytes were at ng/mL level. The feasibility of proposed method applying to determine of prohibited and restricted components in real hair dyes was also examined. The results showed the PPD was detected in three commercial hair dyes ranged from 2.28 to 84420 μg/g. The concentrations of three prohibited components, including 2,4-diaminobenzoic acid, 2,3-diaminobenzoic acid, and 2,4-diaminodiphenylamine were found in the range of 0.78-150 μg/g in real hair dye samples.

筆者於會議中所發表論文之海報