出國報告（出國類別：開會、研究）

赴美國參加第 127 屆公定分析化學家協會(AOAC)年會

服務機關：行政院衛生福利部食品藥物管理署
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派赴國家：美國
出國期間：102 年 8 月 24 日至 102 年 8 月 30 日
報告日期：102 年 11 月 5 日

目錄

摘要

公定分析化學家協會(association of analytical chemical, AOAC)為國際知名之分析化學組織，2013 年第 127 屆年會於 8 月 25 日至 8 月 28 日於美國伊利諾州芝加哥舉行。年會主題涵蓋食品安全、檢驗方法確效、過敏原、乳酸菌等。年會之參加人員來自各國政府機構、研究機構及業界代表等，於座談會中分享其研究成果，並藉由演講後之交流時間和與會者探討研究相關問題，不論是演講者或與會者皆收穫甚多。年會中亦安排不同研究領域之壁報論文展示時間，連續三日的壁報論文展示眾多、場面盛大。透過壁報研讀及研究學者之解說，除學習新知外亦可增進國際間交流。另，大會中亦安排各家儀器廠商分享科儀新知，研究學者除精進分析技術外，亦可藉由更精密之新興分析儀器使研究成果更顯豐碩。本署出席人員包括林美智科長、施如佳專員及黃保寧技佐，並分別展出研究成果壁報各一篇，施專員亦代區管中心董靜馨科長發表一篇壁報論文。於展示期間及 AOAC 台灣分會會議時接受來自各地學者之詢問及討論，藉此展現台灣於食品檢驗分析領域中投注之心力及成果。本屆年會 AOAC 台灣分會事務會議由陳樹功理事長主持。大會主席親臨會場，而在美極具成就之周佳璜博士亦參與此會。會中報告 AOAC 台灣分會各項活動，與會者對於食品安全議題熱烈討論，亦對於會務推展及未來國際事務之參與等議題作建言，使我國未來食品分析與研究及合作事務更加深廣。

目的

公定分析化學家協會(association of analytical chemical, AOAC)成立之宗旨乃充分提供分析方法的確效及實驗室品質保證的發展，食品科學及農業化學等皆囊括於公定分析化學家協會所探討之領域。公定分析化學家協會每年度皆於美國舉辦年會，本年度年會舉辦地點為美國伊利諾州芝加哥市，參與者來自世界各國政府機關及學術單位，人數眾多且場面盛大，是為國際交流之良好場合。本屆參加此年會之目的如下：

一、代表本署參與此國際性研討會，藉由觀摩其他國家檢驗研究單位之研究成果，了解國際間最新檢驗科技之應用與發展趨勢，並增進本署之檢驗技術在國際間之能見度。

二、代表本署展出檢驗研究成果之壁報，並與其他參與壁報展出之作者探討檢驗方法及技術，加速建立國際溝通及聯絡之管道。

三、參與食品安全、檢驗方法確效等研究主題之座談會，瞭解國際間研究之趨勢並藉由研究成果學習方法及技術。

四、協助臺灣公定分析化學家協會於年會中所舉辦之 Taiwan Section Business Meeting 之進行。

過程

本署三位人員於 102 年 8 月 24 日自台北桃園機場啟程，經日本東京轉機再抵達美國芝加哥，並於 8 月 25 日開始參與為期四日之第 127 屆公定分析化學家協會年會。8 月 25 日完成報到後，開幕歡迎會於儀器展覽場地揭開序幕，會場擺滿各種應用領域之儀器設備供有興趣者詢問或索取資料。在會場中看到各國菁英，更有不少東方臉孔，包括在美之華人(台灣、大陸)、泰國、日本人等，並在會場中遇到周佳璜博士，熱心地介紹許多朋友，亦針對近期食品安全事件與檢驗相關問題交換意見。晚間 6 點的開幕歡迎會(Exhibit Hall Grand Opening Reception) 由本屆主席 Mark Coleman 揭開此次年會之序幕，藉由歡迎會認識各國專家及學者並進行意見溝通，了解該國檢驗研究現況。茶會區位於展覽會場區內，在此社交活動中，與會專家與參展之儀器商、試藥供應商、科技協會及代檢驗公司互動良好，是為吸收新知及充分溝通、詢問的良好機會。自 8 月 26 日至 8 月 28 日，大會於會場 Palmer House Hilton 各展廳舉行不同主題之座談會，並於每日上午十點至下午五點於 Red Lacquer 廳展出來自各國專家學者之壁報論文。8 月 26 日亦有 Taiwan Section Business Meeting 以及 Joint Asian Sections Meeting。由於大會安排活動甚多(Symposium, Poster presentation, Meeting, Panel, Community meeting, Workshop, Exhibition)且橫跨不同領域，以下僅就 4 天活動內容作概略性敘述。

一、Opening Session, Award Ceremony and Keynote Address

8 月 26 日上午 8:30 至 10:30 為 Opening Session，由大會主席致辭並頒獎予各領域有卓著貢獻者。有關今年得獎者略述如下：

1. Harvey W. Wiley Award 得獎者是來自 Mérieux NutriSciences Corporation 的董事長 Russell S. Flowers。Russell S. Flowers 本身是微生物專家，也以微生物方法調和化作為演講研討主題。其參與多項 AOAC 活動，並於 2010-2011 年擔任 AOAC 主席，目前則擔任 International Stakeholder Panel on Alternative Methods(ISPAM)之主席等要務。領導多項團隊如 AOAC, USDA, FDA, FLAWG(Food Laboratory Accreditation Working Group)從事實驗室認證工作，發展北美地區測試實驗室認證指引，且目前已為 ISO 採用，並由 ALACC(AOAC's Analytical Laboratory Accreditation Criteria Committee) 維持及修正。

2. Harvey W. Wiley Scholarship 由北卡羅納州 Lee-Ann Jaykus 指導之博士生 Clyde S. Manual 獲得。研究主題為將 PCR 應用於檢驗存在於甲殼類動物中之諾羅病毒(norovirus)。Clyde S. Manual 曾研究農場及牧場(Cattle pastures)中有關沙門氏菌、大腸桿菌及李斯特菌等之存在、分佈等分子流行病學。亦為國際食品保護聯盟學生會主席，並期許有一天可領導研究團隊，應用其所學找出重要之食品安全議題。
3. 會員成就獎有三人，分別是 James Bell, Yvonne M. Salfinger 及 Donna L. Zink。James Bell 有長達 30 年之 AOAC 會員資歷，專精於公定分析方法開發及儀器操作，極力支持 ISO/IEC 17025:2005 認證，目前服務於 Barrow-Agee Laboratories。Yvonne M. Salfinger 現為美國食品藥物管理局丹佛市之諮詢專家及微生物專家，曾任職於美國疾病管制局，參與食品緊急事件網路活動、AOAC 微生物專家論壇等活動，貢獻良多。Donna L. Zink 參與主持 AOAC 參考物質技術部門(Technical Division of Reference Materials, TDRM)活動，成立 TDRM 對照物質資料庫並創立 AIM Research Enterprises，提供食品及農業界研究與分析服務。
4. 多實驗室研究得獎者由 Mars Chocolate North America 之 Rebecca J. Robbins 所領導團隊獲得，參與研究實驗室包括 6 個國家，共計 13 家實驗室。研究主題為開發有關巧克力產品中 Flavanol 及 Procyanidine 之分析(Determination of Flavonol and Procyanidine (by Degree of Polymerization 1-10) Content of Chocolate, Coca Liquors, Power(s), and Coca Flavonol Extracts by Normal Phase High-Performance Liquid Chromatography)。此檢驗方法於 2012 年為 AOAC 採用，成為正式公定分析方法。
5. 本年度單一實驗方法確效得獎者由 Wyeth Nutrition 之 Ping Feng 獲得，研究主題是以胺基酸計算法定量嬰兒配方乳製品內乳清蛋白分析(Whey Protein Content in Milk-Based Infant Formula Finished Products Using Amino Acid Calculation Method)。
6. AOAC 義工獎由 Victoria Siegel 獲得，其致力於發展精準度測試(Proficiency testing)領域，服務於 AOAC 公定方法委員會(AOAC Official Methods Board)，並參與 AOAC 農產品分析委員會(AOAC Agricultural Materials Analytical Community)等單位，貢獻良多。
7. Study director 獲獎者有捷克籍 Katerina Mastovska 及 Jana Hajslova 與西班牙籍 Wendy Sorenson 三位。Mastovska 未進入業界服務前任職於美國農業部，為聯合國

農糧組織與世界衛生組織聯合會之農藥殘留專家(United Nation's Food and Agricultural Organization (FAO) panel on the Joint FAO/WHO Meeting on Pesticide Residues(JMPR)，對於國際上食品及飼料之農藥殘留(feed commodities)提供建言。活躍於 AOAC 活動，如食品分析委員會有關於化學汙染與殘留、動物用藥殘留諮詢家(Topic Advisor)會議等，另外也擔任第 50 屆北美化學殘留工作組主席(Program Chair Chemical Residue Workshop)，目前任職於 Covance Laboratories，主要負責新分析方法之開發及規劃食品及膳食補充品化學殘留與汙染之檢驗。Sorenson 於 2002 年起就參與 AOAC 方法確效委員會，協助並主導單一實驗室確效與實驗室研究工作，於 2007 年即因對於膳食補充品方法委員會之貢獻而首次得到 Study director 獎，目前為 Covance Laboratories 之 Study Coordinator，主要從事於營養化學及食品安全。

8. 技術顧問獎頒予來自 Least Cost Formulations, Ltd 具有化學背景統計方法顧問 Robert A Labudde，其獲獎無數，於 2007 年即曾獲得此獎，並於 2011 年獲得 AOAC 社區義工獎。Robert A Labudde 具動物科學家背景，服務於美國肉品協會及其他技術學會，如 ASA、AOAC、IAFP 及 APRAS。曾發表許多文章、出版兩本專書並擁有多項專利，亦對數百家製造廠提供諮詢服務，在食品安全與科學領域上是享譽國際之演說家。

9. 專家評審小組得獎者為 Joe Boison，其提出之萊克多巴胺及動物組織中 Narasin 和 Monensin 之建議檢驗方法已為公定分析方法委員會所採行。萊克多巴胺方法之確效具有良好之再現性，實驗結果亦經專家委員會審查同意。雞、豬及牛組織內 Narasin 及 Monensin 之分析則有 10 間實驗室參與，實驗結果具有良好回收率及準確度。技術與科學優越成就獎頒予 Bert Popping 及 Joe Boison 兩人所領導之團隊。

10. 參考物質成就獎由服務於美國國家衛生研究院膳食補充品部門之 Joseph M. Betz 獲得，其曾為美國 FDA CFSAN 研究化學家，亦擔任美國草藥產業聯盟(American Herbal Products Association)副主席，研究領域為植物補充品(Botanical supplements)，發表諸多天然物領域文章。

頒獎結束後，由 William C. Weldon (Vice President, Elanco Animal Health, A Division of Eli Lilly and Company Research and Development)作專題演講，主題為「Enriching People' s Lives: A

2014 Report on the Importance of Animal Source Foods」。講者以過去貧窮社會為例，所尋找食物來源可能涵蓋動物性來源為引言。糧食為維繫人類生命和健康所不可或缺的要素，而農業生產受制於天候常會導致農產價格波動且糧食供給不確定，食品安全更相形重要。糧食中之肉、蛋及奶等動物性來源富含豐富之蛋白質及維生素群，是維持腦部與肌肉發展之重要因子亦可預防疾病。對於貧窮國家維持腦部及肌肉發展之功能相形重要，然已開發國家則因面臨肥胖問題，對於動物性來源食物之需求則以維持健康需求為主。Weldon 以該公司對於開發中國家(如肯亞)之金錢支援與技術扶助為例，證明蛋確實可改善學童健康與智力，且該公司也將繼續支援或投資其他未開發國家，為人類之糧食安全與健康盡一份心意。Weldon 認為，企業除獲取利潤外，若能以己能力來回饋社會，必能為企業創造新形象。

H.W. Wiley Award 得獎者 Russell S. Flowers 之演講主題為「Microbiological Testing of Foods: Efforts to Harmonize Procedures for Validation and Verification of Methods and Laboratory Performance」。微生物試驗法為確保食品安全及品質之重要因素，常藉此建立食品成分、加工食品及加工環境的 baseline information。微生物試驗法不僅為兩國間進口及出口之貿易協定所需，亦為消費者及供給者間訂定食品規格之準則。講者首先介紹各種微生物試驗法，並說明不同的試驗法有其相符合的準確度、精密度、敏感度及專一性，且統計數據的信賴區間及不確定度亦會受實驗步驟（如取樣方法、檢測方法之靈敏度與實驗室能力等）影響，因此需針對不同方法來進行調整。

二、壁報論文展示

日期	主題	篇數
8 月 26 日	Analysis of Foodborne Contaminants and Residues Analysis of Non-Foodborne Contaminants and Residues Microbiological Methods Water and Waste Water Analysis General Pharmaceutical Analysis and Evaluation	共 102 篇
8 月 27 日	Food Nutrition and Food Allergens Emerging Issues in Food Safety and Security Performance Tested MethodsSM	共 103 篇

	General Methods, Quality Assurance and Accreditation	
8 月 28 日	Authenticity Botanicals and Dietary Supplements Detection and Measurements of Natural Toxins	共 89 篇

由於會場展示壁報眾多，茲將與業務相關之壁報內容重點及研究趨勢摘錄於下：

1. 近年來米中無機砷含量之議題廣受重視，「Speciation of Inorganic Arsenic in Rice by Solid Phase Extraction and Quantification by Hydride-Generation Atomic Fluorescence Spectrometry」是以微波消化萃取的方式，將三價砷（AsIII）氧化成為五價砷（AsV），有機砷則不變。五價砷再以強陰離子交換匣置換，並以碘化鉀氧化成三價砷。三價砷再與 NaBH4 反應產生 AsH5，最後以砷螢光分析儀檢測。
2. 「Market Basket Study of Arsenic Concentration and Speciation in Rice and Rice Products Sold in the United States」則是分析市售美國市售之 66 項米及米類製品，以感應耦合電漿質譜儀（Inductively coupled plasma mass spectrometer, ICP-MS）測總砷含量，並串聯液相層析裝置至 ICP-MS，檢測砷物種含量及種類。因目前米中砷含量對人類健康的影響情形並不明確，因此結果將作為風險評估參考。
3. 「The Use of Microflow UHPLC in Vet Residue Analysis」以 Microflow UHPLC-MS/MS 檢測食品中 Sulphonamides 及 Tetracyclines，由於一般 LC-MS/MS 設定之移動相流速大多超過 500μL，而此篇作者所使用之 Microflow UHPLC 之流速則為 40μL，除可提升待測品項之偵測極限外，亦可降低有機溶劑之使用量以減少對自然環境的破壞，並可降低實驗室執行分析之成本耗損。
4. 「Development and Validation of Screening Method for Multi Antibiotic Residue Detection Using High Resolution Mass Spectrometry」以高解析質譜儀(OrbiTrap)檢測多種食品基質包括蛋、魚及肉品中 103 種動物用藥(benzimidazoles, quinolones, nitromidazoles, macrolides, triphenylmethan dyes, sulfonamides, tetracyclines, β-lactams)之殘留，實驗以 full-scan 及 data-dependent precursor ion fragmentation 的方式偵測欲檢測品項，偵測極限可達 0.4 至 23 ppb。
5. 「Determination of Vet Drug in Meat and Fish with Solvent Extraction and LC/MS/MS Detection」係使用超高液相層析串聯質譜儀以電灑游離正離子法進行肉類及魚類基質中

quinolones、coccidiostats、ionophores、β-lactams、sulfonamides、macrolides、tranquilizers 及其他類，共計 56 種抗生素之多重殘留分析方法。首先將 5 g 檢體以萃取液(MeCN + 5% MeOH + 0.05% HCOOH)萃取，經離心過濾後，再以 Alltima C18 (250 mm x 3 mm x 5 μm) 作為層析管柱進行分析。於魚肉基質中添加 5~100 ppb 標準品之平均回收率，除 amoxicillin 及 cloxacillin，其他品項皆介於 73~114%；於牛肉基質中添加 5~100 ppb 標準品之平均回收率，除 robenidine、amoxicillin、ampicillin、cloxacillin、oxacillin、danofloxacin 及 norfloxacin，其他品項之回收率皆介於 74~116%。

6. 食品中過敏原檢測的部分，「Hazelnut Detection in Food: Effect of Thermal Treatment」作者指出，由於榛果在食品工業中大量被使用，且大多經過可能導致蛋白質構型改變的加熱過程，此一步驟亦可能導致致過敏性質的改變。實驗中將榛果以不同加熱程度進行分類：(1)未經加熱；(2)以 180℃加熱 10 分鐘；(3)以 115℃加熱 30 分鐘；(4)以 115℃加熱 20 分鐘後再以 180℃加熱 10 分鐘，再以兩種市售 ELISA kit 進行過敏原檢測。實驗結果以 kit B 針對各組別之檢驗能力無明顯差異，但以 kit A 則有明顯差異，並觀察到第(2)及(4)組的偵測敏感度低於未經加熱的第(1)組。由此可知，以不同市售 ELISA kit 檢測經高溫加工食品過敏原之結果應有差異，對於此類食品應挑選對於變性蛋白質檢測度較高之市售 ELISA kit 以避免實驗結果的誤差。
7. 本署北區管理中心以「Analysis of Heavy Metals (Cadmium, Lead and Mercury) in Rice」為題參加壁報展覽。本署監控台灣地區食米之重金屬鎘、鉛及汞含量已長達 11 年（91 年至 101 年之統計），101 年由各縣市衛生局至其轄區碾米廠採取一、二期作之食米檢體，由本署與協力之衛生局(基隆市、南投縣、台南市)依據行政院衛生福利部公告方法執行檢驗。160 件食米中鎘、汞及鉛含量檢測結果，均未超出行政院衛生福利部公告之『食米重金屬限量標準』(鎘 0.4 ppm、汞 0.05 ppm 及鉛 0.2 ppm)。壁報論文如附件一所示。
8. 本署北區管理中心董靜馨科長則發展以 HPLC-UV-ESI-MS 系統快速鑑別金針菇（Golden Needle Mushroom, Flammulina velutipes）中之 Fip-fve Protein 方法，整個實驗過程較傳統方法節省 90%以上的分析時間。壁報論文如附件二所示。
9. 本署研究檢驗組以「Multiclass analysis of 17 veterinary drugs in milk by liquid chromatography–electrospray tandem mass spectrometry」為題，本研究係使用超高液相層析串聯質譜儀(ultra performance liquid chromatograph/ tandem mass spectrometer,

UPLC/MS/MS)，以電灑游離正離子法，進行食品中巨環類(macrolides)、β-內醯胺類(β-lactam)、林可黴素類(lincosamides)及其他類，共計 17 種抗生素之快速多重殘留分析方法之開發，平均回收率分別介於 67.7~108.3%、81.1~99.4%、87.0~109.5%及 85.1~110.6%間，同日內及異日間平均變異係數小於 10%，定量極限則介於 0.5~10 ng/mL 之間。壁報論文如附件三所示。

10. 加拿大 British Columbia Institute of Technology, Natural Health and Food Products Research 的研究人員發表一篇以液相層析質譜儀分析 British Columbia 的植物和蜂蜜中的吡咯里西啶生物鹼(pyrrolizidine alkaloids, PA)。PA 具有肝毒性和致癌性，且物質會經由蜂蜜採集花粉和花蜜時造成蜂蜜內含量的蓄積，因此近年來是為學術界的研究焦點之一。天然毒素越來越受到重視，本署也曾呼籲民眾，部分植物含有天然毒素，食用這些有毒植物可能會損害健康，切勿採摘不明植物食用。

三、專題演講座談會

本年度 AOAC 年會的專題演講主題一如往年涵蓋領域相當廣泛，每個時段同時有二至三個主題於各個展廳進行演講，茲將各主題之重點整理如下。

1. Wiley Award Symposium: Evolution of Proprietary Methods in AOAC

早期食因性疾病非來自於感染或中毒，分析方法易執行但耗時。早期方法確效是與參考方法做比較，定量方法的統計評估是以 ANOVA 及 Student's t-test 作為根據比較平均值，而定性方法的統計評估則是根據 Chi square 及 McNemar's test。但隨著時代及環境的變遷，引發食因性疾病因子越來越複雜，臨床上可觀察到不僅有偶發性的病例，甚至出現急性病原菌的感染，例如病原性大腸桿菌、李斯特菌、隱孢子蟲及 Norwalk 病毒。因此更具可信度及費時短的食品檢驗方法需求增加也因此被建立。而於 1991 年，AOAC 亦發表了適用於食品及水中微生物試驗的專利方法。

2. Antibiotics in Chain

(1) Fate and Occurrence of Antimicrobials in the Environment

來自韓國食品藥品安全部 Jin-Wook Kwon 報告有關磺胺類抗生素及四環黴素於土壤及實驗室環境中衰變分解之研究。研究以固相萃取裝置進行成分分析，也對水中殘留量做研究，找出衰變時間，如 D50/D90。希藉此研究避免動物用藥殘留於環境

中，並發展正確用藥觀念以生產安全食品。

(2) Possible Up-Take of Antibiotics by Plants

奧地利 Andrew Cannavan 等人研究有關抗生素於植物中分布情形。抗生素之所以用於動物，除預防或治病外也可促進生長，惟可能吸收不完全，隨糞便或尿液排於土地，隨後由植物吸收進入食物鏈，有許多研究已顯示磺胺類抗生素、氟喹諾酮類抗生素、胺基醣苷類抗生素、Amphenicols 及林可黴素等皆可被植物吸收。此研究以相當多種類的植物進行研究，分析技術包括 Agar diffusion、LC-Iontrap MS、ELISA、TLC、HPLC-UV 及 LC-MS/MS，分別添加 14C atropine 做分析，探討被動運輸等機制。

(3) Innovative Analytical Approaches for the (Broad) Screening and Detection of Antibiotics

法國 Eric Verdon 等人研究有關動物用藥之檢驗方法的演進。以磺胺類抗生素分析為例，80 年代多以生物性方法篩選；90 年代採用 TLC 與 HPLC 法篩選；2000 年則以 LC-MS/MS 進行分析及確認；2010 年以 MRM 篩選與確認。質譜具有專一性，可做多重殘留分析。講者以 LC- Orbitrap MS 分析，添加多品項抗生素於肉及牛奶中做分析，惟尚有部分品項分析不易。講者提出對於非標的物分析可採用 Orbitrap 等高解析度質譜儀，以全掃描質譜法與建立資料庫做追蹤。

3. Emerging and New Contaminants in Food Commodities from Plant Origin: Analytical Methods and New Issues

(1) A European Survey of the Microbial Contamination of Some Fruits and Vegetables

比利時 Mieke Uyttendaele 報告過去一年在比利時、挪威或西班牙所執行有關萵苣或草莓受微生物汙染之共通性計畫(Within Veg-i-Trade, EU FP7 collaborative project)調查結果。採樣除成熟蔬果外也對土壤及灌溉用水一併研究。檢驗菌種如 O157 及沙門氏菌等，檢測方法採 PCR-VTEC。研究發現土壤中雖有沙門氏菌被檢出，但萵苣未發現有受汙染，於某些萵苣檢體有檢出 Campylobacter，也發現水污染情形有季節效應等。講者亦說明微生物有存於新鮮葉菜類及草藥的風險。

(2) Quantitative Targeted Residue and Retrospective Data Analysis of Relevant Pesticides, Antibiotics and Mycotoxins in Bakery Raw Materials and Food Commodities by LC-HRMS Executive^{TM} (Orbitrap Technique)

義大利 Michele Sumanz 以 UHPLC-HRMS 進行分析，開發農藥、黃麴毒素、Zearalenone、Trichothecene、磺胺類抗生素及氯黴素類抗生素等檢驗方法，基質為烘焙食品。實驗結果得到良好的線性、回收率、再現性及重複性。

(3) Determination of Mycotoxins in Baby Foods and Animal Feed Using Stable Isotope Dilution and Liquid Chromatography-Mass Spectrometry

美國 Kai Zhang 等人以 LC-MS 進行分析，開發 12 種黴菌毒素(包括黃麴毒素 B1、B2、G1、G2 及 M1；Deoxynivalenol；Fumonisin B1、B2 及 B3；赭麴毒素 A；T-2 Toxin；Zearalenone)之檢驗方法，並以以同位素([^{13}C]-IS)稀釋法降低基質效應。基質為牛乳、以牛乳作為基礎的嬰兒食品及動物飼料等。研究以 50%乙腈溶液進行萃取並經過濾，再以 LC-MS 進行分析。平均回收率介於 70 至 120%，且 RSD 值小於 20%。此檢驗方法具良好選擇性、敏感度、準確度及再現性。另外，講者亦以 UHPLC Orbital trap MS 進行全質譜掃描用於監測食品中黴菌毒素。

(4) Arsenic in Rice and Rice Products

由來自美國 FDA 的 Willam Mindak 報告，國際癌症研究組織已將砷歸類為 Group 1 致癌物質，砷可分為有機砷與無機砷兩大類，而無機砷毒性大於有機砷。無機砷又分為不帶價砷、三價砷及五價砷，其中以三價砷毒性最高。以 0.28M HNO3 萃取後調整 pH，以 LC-ICP-MS (EAM Method 4.11)應用於米及其產品中有關 As(III), As(V), DMA (dimethylarsinic acid), MMA (monomethylarsonic acid), AsB (Arsenobetaine)之分析。

4. Taiwan Section Business Meeting

本屆年會於 8 月 26 日晚上 6 點亦提供會議室供台灣分會舉行 Taiwan Section Business Meeting，本次會議由陳樹功理事長主持，共計約 20 位專家學者蒞會參加，多為任職於美國官方及民間機構的台灣人，此外 Dr. Albert E. Pohland (Director, Office of International Activities, AOAC INTERNATIONAL Staff)亦蒞臨致辭與勉勵。廖家鼎秘書長細心準備台灣茶及各種中式糕點提供給與會者品嘗，更增添場面的溫馨及熱鬧。會議中陳理事長以 AOAC 台灣分會之會務推展及未來國際事務之參與等議題與各專家學者進行熱烈討論，為我國分析科學交流及合作事務奠下良好基礎。

5. Advanced Strategies and Tools for the Identification of Unknowns in Food

(1) Non-Targeted Spectroscopic Screening Techniques: An ASSET to the Food and Feed Laboratory

英國 Stewart Graham 等人之研究，介紹以傅立葉轉換近紅外光光譜儀(Fourier Transform Near Infrared, FT-NIR)篩選結合化學光譜分析模式，推測可能之未知標的物之檢驗，應用於黃豆內摻雜 Melamine 及飼料中油品之偵測。此外，亦以高解析光譜檢驗貝類中 Azaspiracid 及含違法生長激素之牛肉組織。本研究以可能摻雜 biomarker 為基礎開發方法，目的是建立可廣泛被應用的檢驗方法。

(2) Non-Targeted Analysis Using Nuclear Magnetic Resonance for Ensuring Food Safety and Authenticity

英國 Adrian J Charlton 介紹以核磁共振光譜學(Nuclear Magnetic Resonance Spectroscopy , NMR)應用於未知標的物之分析，並舉例說明有關應用 NMR 及質譜於食品有效期限及蜂蜜之摻偽研究。

(3) The Use of High-Resolution Mass Spectrometry in Food and Feed Traceability

捷克 Jana Hajslova 等人以高解析質譜如飛行式質譜儀(Time-of –flight, TOF)及 Orbitrap 探討橄欖油、番紅花及大蒜之產地別，並以指紋質譜(Profiling/ Fingerprinting)應用於非標的物之分析與篩選。以番紅花之 Saffronomics 為例，分析方式以 70%乙醇超音波萃取 1 小時後，以如 SPME-GC-TOMS、DART-Orbitrap MS、UPLC-Q TOF MS 等高解析儀器進行分析。以此方法亦可檢驗食品中偶氮類酸性染劑的存在以及魚油的攙偽。

6. Quantitative and Qualitative Analysis of Emerging Substances of Concern (ESOCs) Using Liquid Chromatography - High Resolution Mass Spectrometry (LC-HRMS): Method Development, Method Verification/Validation, and Method Harmonization

(1) The Analysis of Pesticides in Fruit and Vegetables by HRMS: An Indian Perspective

由印度 Kaushik Banerjee 報告。近年來印度對於食品分析能力的顯著的提升，國內大多使用串聯四極柱及高解析質譜進行食品安全檢驗。講者並闡述印度國內的食品安全相關法規，主要控管國產食品及進口食品。Q-TOF MS 可應用於未知物的鑑定及資料庫的建立，如黴菌毒素等。

(2) Identification of New Pesticides Degradation Products in Plants and Soil by

LC/Q-TOF-MS

美國 Imma Ferrer 報告有關三種氯化農藥(imazalil、imidacloprid 及 propiconazole)分解產物之鑑定，除針對植物(洋蔥與萵苣)中農藥外，亦針對土壤進行研究。以液相層析四極柱式/飛行式串聯質譜儀(liquid chromatography coupled with quadrupole time-of-flight mass spectrometry, LC/Q-TOF-MS)進行分析，實驗中加入同位素標準品以消除基質效應。與預先建立資料庫比對，以主成分分析(Principal component analysis, PCA)模式分群以觀察代謝物分佈。研究結果亦發現 imidacloprid 之三種主要代謝物及七種新發現之代謝物。

(3) The Detection and Structural Elucidation of a Textile Dye Used as an Illegal Food Color in Spice

瑞士 Anton Kaufmann 介紹，2003 年蘇丹紅一號(Sudan I)染劑被不當添加入咖哩及辣椒中做為著色劑，起初違法使用之案件數急遽增加而其含量低，隨後件數雖漸少但濃度漸增，恐造成公共健康問題。講者以高解析質譜 Orbitrap(100,000 FWHM)結合偶極陣列偵測器(diode-array detector, DAD)進行分析，即可發現未知物，並說明初期遇到鑑定之困難是由於資料庫所載內容無法符合。

(4) Recent Development and Application of Ultra-High Performance Liquid Chromatography (UHPLC) and Micro Flow Liquid Chromatography (MFLC) Coupled with Q-Orbitrap for Analysis of Pesticides in Food

加拿大 Jiang Wang 以 QuEChERS 進行蔬果類產品之前處理，以 UHPLC/ MFLC/ Q-Orbitrap 分析蔬果中殘留之農藥約 500 個品項，MFLC 及同位素標準品添加可克服基質效應，使分析濃度可以降低 1000 倍達到 ppt 級。

(5) Monitoring of Emerging Substances of Concern Using Liquid Chromatography-High Resolution Mass Spectrometry Oral Posters from Dietary Supplements and Botanicals

加拿大 Paul Yang 以 LC-Orbitrap 搭配電灑游離法正離子模式進行分析，監控廢水中約 400 種之內分泌干擾物質、藥品及農藥等，研究結果可辨識以廢水處理之植物檢體中 117 種藥品殘留。

7. New Blood 2013: Developing Methods for Detection of Chemical Contaminants

(1) Analysis of Multiple Pharmaceuticals, Plant Toxins and Alkaloids in Botanical Dietary

Supplements by Ultra-High Performance Liquid Chromatography-Quadrupole-Orbitrap Mass Spectrometry

美國 FDA Lukas Vaclavik 等人研究，由於西藥有副作用，因此有人會改服用膳食補充品，造成該類產品之消費成長數倍。膳食補充品可能由於某種植物產量不足供應而加入其他物質，面臨摻雜及造假之風險。本篇研究以 QuEChERS 進行產品之萃取，以 u-HPLC-Q/Orbitrap MS 同時分析多種藥品、植物毒素及生物鹼等成分。

(2) 2013 Brooks Rand Labs Interlaboratory Comparison Study for Arsenic Speciation in Food and Beverages

為美國 Michelle Briscoe 等人之研究。本研究邀集 46 家實驗室參與此研究，其中 65% 來自北美地區。研究主題為共同分析白麵粉、糙米粉、海帶粉及蘋果汁等產品中所含 5 種型態之砷含量。前處理流程包含陰離子交換樹脂，再以 HPLC/ICP-MS 進行分析。各實驗室回報數據並進行統計分析後，藉以比較實驗室間能力。

(3) Determination of Inorganic Arsenic in Food and Feed-European Initiatives in Research and Standardization of Methods

丹麥之 Jens J. Sloth 及 Rie R. Rasmussen 報告，歐洲法律對於微量元素之訂定是在考量食品及飼料之安全性原則下依據總量而定，但總量與生體可利用率及毒性不見得呈現正相關性。作者以砷為主題發表目前研究成果以及歐盟之進行中計畫。另提出 EU 2006/1881/EC 規定無機砷之 Below Minimum Detectable Level (BMDL) 0.5 為 3 ug/kg BW/day 等數據。

8. SPSFAM Expert Review Panel- Heavy Metals

議程包括 AOAC 自願政策(Volunteer Policies)檢閱、專家覆審平台準則及程序概要(Expert Review Panel Guidelines and Process Overview)、標準方法執行要件概要(Overview of Standard Method Performance Requirements)、審閱重金屬檢驗方法，包含「Determination of Heavy Metals in Food by Inductively Coupled Plasma-Mass Spectrometry」、「Method Validation for the Analysis of Arsenic, Lead, and Cadmium in Juice Concentrate」等六項。小組成員對重金屬特性和檢測方法皆相當熟稔，會談的專業態度值得學習。

9. Selection of Analytical Methods for Validation of Allergen Control Plans

(1) FSMA and Allergen Control

由任職於美國 Grocery Manufacturers Association (GMA)的 George Dunaif 報告。講者一開始介紹 Food Safety Modernization Act (FSMA)為極重要之聯邦食品及藥品法案(Federal Food & Drug Act, FD&C Act)之一，其重點在於食品安全，亦為預防控制方案(Preventive Control Proposal)，而過敏原控管即為美國目前食品安全的重點控管項目之一。過敏原控管一直以來都是以 FD&C Act 下的良好作業規範(Good manufacturing practice, GMP)作為規範，而為接受控管的食品建立完整的製程紀錄為重要的控管步驟，可避免食品因交叉污染或是摻雜而含有過敏原。講者並介紹十個要點，包括風險管理、Industry flexibility、危害分析重要管制點(HACCP)、Testing/Supplier verification、確效、紀錄保存、符合 GMP、Define small business、Compliance date 及 Warehouse expansion。講者並提醒風險分析時應做好鑑別分析、評估危險因子及預防控管如流程管控、過敏原管控、消毒及產品回收等概念，並期許食品業者參照 GMP 做好自身控管。

(2) Validating a Specific Process-Method Selection

由美國 Joseph Baumert 報告，Food Safety Modernization Act(FSMA)於 2011 年公布，美國 FDA 並於 2013 年發布 Preventive Control Propose Rule，法規中並指出執行過敏原控制計畫(Allergen Control Plans, ACPs)的必要性，以降低過敏原交叉接觸後造成之危險性。FDA 建議食品業者除目視檢測外，更需開發並進行分析檢測方法的確效，分析方法如酵素免疫分析法、聚合胺連鎖反應及蛋白質測試等。講者並提到食品過敏原研究資源計畫(Food Allergy Research and Resource Program, FARRP)，並以稽核者角度表示食品安全控管無法接受目視檢測，換言之即須做到清潔與衛生確效。檢測方法則應根據產品主成分選擇適合之方法，生產線各個管制點皆應追蹤與鑑定各種過敏原，檢驗方法產生後也須確效，並做好衛生標準操作程序(Sanitation Standard Operating Procedures, SSOP)，並需經過一再地確效。

(3) Swabs to Products-Comparative Sensitivity

美國 Steve Taylor 報告，商品化之 allergen swabs 及 lateral flow strips 已成為監測食品產線上之重要產品，主要應用於評估加工設備表面之清潔度。Swabs 的偵測量約 1-10 ppm，如經萃取後，再以 ELISA 檢測而得的偵測極限將更低。講者以 Pharmaceutical Technology 於 1998 年九月發表有關 Cleaning validation 之文章為基礎，

研究花生產線中所使用的設備(split cone ribbon blender)之 swab 實驗，在 95%信賴區間下，ED 0.5 約為 3.6 mg，結果顯示 swab 的偵測極限是在安全邊緣。

(4) A Food Industry Perspective on Validation of Allergen Control Plans

美國之 Mark Domanico 報告，欲使食品中不含過敏原，應遵循衛生標準操作程序(Sanitation Standard Operating Procedures, SSOP)進行食品製程，針對高風險性與較低風險性生產線進行評估，以及不同風險等級但在同一產線之考量。為避免過敏原累積，產線清潔後亦需清楚標示。研究中是以 Swab 來進行採樣，製程中之關鍵製程、最後潤濕用水及最終產品均為採樣對象。並建立「break」生產線，在生產線的前四個小時丟棄部分產品，以隔開前段生產產品避免交叉汙染。

10. What Comes Before the MS in LC-MS Food Allergen Analysis?

(1) Importance of Sample Preparation and Digestion Conditions Prior to LC-SRM-MS of Food Allergens

瑞士 Petra Lutter 報告，應用質譜作為已知標的物之分析是趨勢，講者以牛奶及堅果為例提出過敏原蛋白質標的物選擇及前處理所面臨之挑戰。研究中每個食品基質皆挑選二至三個標的物，而食品基質對檢測結果的影響使得基質中其他成分之去除為極大挑戰。影響消化效率的因子除了胰蛋白酶的品質及量外，是否精準控制消化時間及溫度亦會影響消化效率(如微波)，而不耐酸之界面活性劑則可幫助蛋白質溶解及變性。研究以 SDS-PAGE 及 LC-SRM-MS 檢測消化效率，顯示胰蛋白酶及界面活性劑的使用可使消化效率達 93~96%。由於基質對蛋白質偵測極限的影響，除了可添加同位素胜肽標準品進行定量試驗外，基質中鹽類的去除則可使用管柱淨化裝置。

(2) Sample Prep for Food Allergen Analysis by Mass Spectrometry: Strategies and Preliminary Results

比利時 Valery Dumont 報告，相較於 ELISA，以質譜檢測食品中過敏原的偵測極限會較高，而樣品基質的前處理是否能去除基質干擾即為重要影響因素。本研究主要使用免疫親和管柱進行樣品的純化。講者以花生中過敏原分析為例，使用固相萃取(Sep-Pak Accell^{TM} Plus CM, pore size: 300 Å)裝置進行淨化，此一步驟中的 1M 氯化鈉的使用為重點，另外亦使用 ZipTip® C4 Resin (pore size: 200 Å)。研究中亦探討不

同 nominal molecular weight limit (kDa)之離心濃縮管(ultrafiltration)的過濾效果，當使用 100kDa 及 300kDa 之離心濃縮管時會導致蛋白質的流失，而使用 3kDa 及 10kDa 之離心濃縮管時雖不會導致流失，但亦不會使檢測量提升。

(3) Examples of Mass Spectrometric Food Allergen Method Development and Application at the FDA

美國 Mark Ross 之研究。質譜方法現今發展為食品中過敏原之確認定量及定性之方法，用以輔助 ELISA 及其他檢驗方法。本研究目的有二，一為檢測未知物之定性，是以 Linear Trap Quadropole (LTQ) Orbitrap 及 LC-MS/MS 進行分析；二為已知物之定量，是以 LTQ-Qtrap、QE、LC-MS/MS 及 SRM 進行分析。樣品基質來自牛乳、雞蛋及花生，且經實驗室處理為類似市面上此類食品含過敏原之狀態。研究將含過敏原之牛乳添加進入餅乾等烘焙食品進行分析，並以 casein S1 作為重點標的物，標準品則用 ^{15}N Casein S1。研究結果發現製程中的烘焙步驟會使質譜訊號降低，而 YLGYLEQLLR 肽段的穩定性則最高。另外樣品基質中麩質(Gluten)亦為一分析重點，研究之檢量線濃度為 10 至 100ppm，並以 SRM 進行分析。研究亦以花生作為基質比較蛋白質萃取效率，研究中以 PBS 或尿素進行萃取，佐以 SDS-PAGE 及 LC-MS 進行分析。針對多過敏原的測試，研究結果顯示花生中之過敏原較牛奶及雞蛋穩定，不會隨烘焙過程流失。

(4) No Hiding for Allergens: Appropriate Extraction Methodologies

來自德國的 Bert Popping 報告，自 2005 至 2013 年許多關於過敏原之研究顯示，PCR 及 ELISA 之方法對於過敏原檢測之不易。含有過敏原之食物如雞蛋、牛乳、黃豆及麵筋製品由於其雙硫鍵不易以溴化十六烷基三甲銨(CTAB)及 Tris 等常用萃取溶液打斷，因此導致過敏原之萃取困難。至今僅有以 Morinaga 公司開發的 ELISA kit 進行分析可得到較良好的回收率。講者並探討雖然以 LC-MS/MS 進行分析所得的結果可信度較高，但可能遇到的困難點即在於不同基質前處理的不易。

(5) How Does an Allergen Go from Food into an LC-MS: A Look at Protein Digestion and Sample Cleanup?

英國 Steve Lock 報告，美國約 6%的幼童及 3.7%的成人會對食物中過敏原產生過敏反應。由於蛋白質本身分子量大，若無經過消化步驟，以傳統的 LC-MS/MS 進行分

析的困難度較高，且蛋白質本身的質譜及解離性質亦使其被偵測的靈敏度較低。講者簡單介紹蛋白質分析的步驟，首先以緩衝溶液萃取含過敏原之食品以打斷三級結構，再經離心沉澱並去除沉澱物，再以二硫蘇糖醇(Dithiothreitol, DTT)將結構中的雙硫鍵打開以破壞二級結構，並使用烷基化物(如 Iodoacetamide)避免蛋白質的 refolding，最後以胰蛋白酶進行消化。講者比較四種前處理之萃取溶液的萃取效率，並以其萃取後經液相層析及質譜分析之峰型呈現作比較，如下表所示：

萃取溶液	LC	MS
Tris	差	尚可
Salt solution	差	良好
Surfactants	尚可	良好
Alcohol high %	良好	差

講者亦比較可避免 refolding 之使用試劑的穩定性及反應所需時間，如下表所示：

	穩定性	反應時間
Methyl methanethiosulfonate (MMTS)	高	20-30 分鐘
Iodoacetic acid	低	
Iodoacetamide (IAM)	低	
4-Vinylpyridine	低	90-120 分鐘

講者最後比較以胰蛋白酶等方法進行消化之所需時間，如下表所示：

	所需時間
Immobilized trypsin	2 分鐘
Microwave	20-40 分鐘
Thermal mixing	1 小時
Traditional	2 小時

(6) What Protein Markers are Best for Peanut Protein in Food Matrices

加拿大 Terry Koener 介紹，以 NIST peanut butter (defat)為例，經過萃取、消化後以 C18 管柱淨化，測定 20 個胜肽之序列，以 BLAST(Basic Local Alignment Search Tool)分析胺基酸序列以確定胜肽結構。並以花生中可溶性蛋白添加入不同基質，濃度為

100μg/mL，分析不同基質以 LC-MS/MS 所得訊號，結果顯示所得訊號大小依序為：餅乾>巧克力>冰淇淋，同時亦證明須依不同之基質選擇不同胜肽作為標的。

11. Hot Topic Symposium: Authenticity Challenges-Yesterday, Today and Tomorrow

德國 Sara Handy 介紹，最早之食品摻雜起於 1820 年，近年之重大摻偽事件則為 2007 年牛肉摻馬肉事件及 2013 年 Irish FSA tests for undeclared horsemeat。偵測細胞中粒腺體 16S rRNA 及 DNA，並以 Blast 搜尋序列，以粒腺體偵測較靈敏但專一性差。講者亦開發豬 DNA 的檢驗方法，偵測極限:0.01%；定量極限:0.05%，並提出 HALAC 認證。

12. Advancement in Methods of Sampling and Analysis for Agricultural Materials

(1) Method for Determination of Soluble Silicone in Non-Liquid Fertilizer Materials

美國 Mary Provance-Bowley 等人之報告。AAPFCO 指出，Silicon 可以改善土壤中養分之不平衡及恢復土壤生態，是植物有益物質，並認可工廠肥料之保證書表格上可以只標示總 Silicon 量。研究團隊執行肥料中 silicone 分析方法開發，發現以 sodium carbonate- ammonium nitrate 是最佳萃取液。目前方法已發表於 J AOAC，可供製造者評估品質及正確標示，並供消費者選擇合適肥料以供作物之生長。

(2) Overcoming Matrix effects in Fertilizer Sulfur Determination

美國 Sandra Hughs 等人合作之研究，改進以往 AOAC 以重量法測定硫含量之方式。並講解元素分析雖也有人採用，但因波峰形狀會改變，定量上可能提高不準確度。講者並提出新的分析方法，先克服基質效應，再以元素分析結合吸附/去吸附層析改善分析效果。

(3) Determination of Biuret by HPLC in Water-Soluble Fertilizers & Urea Solutions

美國 Michael Hojjatie 介紹，講者應用單一實驗室確效研究方法分析肥料中尿素含量。23 件分別來自不同來源之檢體 (含尿素、urea ammonium nitrate (UAN)、sulfur coated urea)，並以已知濃度添加 Aldrich 標準品進入空白檢體做為比對，以 HPLC 分析，分析管柱則使用 aminopropyl column。平均回收率約為 97%，另也和其他另外兩個實驗室做比對性試驗。

心得及建議

1. 本次 AOAC 年會期間聆聽來自不同國家的講者精采的研究成果，這樣的會議將全球分析化學家凝聚在這裡，讓大家有機會能夠直接對談、交流和分享，並且將最新的科技訊息藉此傳遞，能夠參加為期三天的國際型化學分析議題探討，與有榮焉。
2. 本次年會特別為 Authenicity 舉辦多場討論會，顯見對於之品質提升之重視，並藉由 symposia 及 poster 發表眾多論文，預期此類主題將在國際間扮演重要的角色。而有關膳食補充食品(Dietary Supplement) 之功能性成分及生理功效之議題仍相當熱絡，在未來食品工業的發展趨勢中，功能性食品與膳食補充品必然是一研討主題。除新功能食品與新膳食補充品之研發外，檢驗分析之需求，包括功效成分之定性、定量檢驗技術，以及食用安全檢驗，藉由面對面溝通討論，相互交換意見，獲益良多。此外，收集並攜回與業務相關之資訊，傳予同仁參考。有關中草藥或食品分析領域，漸漸走向高解析質譜之應用，並常搭配有關生物方法之鑑定。因此本局應積極了解此方面國際上之趨勢與走向。
3. 有關中草藥之鑑定除以 DNA 角度切入，尚有以蛋白質或糖類方面之檢測切入，今年更有不少篇壁報論文以 NMR 技術解析，或許可以提供摻偽之另一種鑑定技術。中草藥檢驗方法與規格之訂定，AOAC 目前已完成麻黃、馬兜鈴酸、銀杏類黃酮成分之公定方法確效，另外人參、蘆薈及甘草等植物性藥材亦正進行研究中。應隨時注意此方面資訊，以作為未來檢驗方法之參考。
4. 從此次年會壁報論文展中，顯示「摻雜 Sildenafil 類緣物於中草藥或 deitary supplement」之事件，應不止出現於我國。另由國外研究單位發表之報告及此次年會他國代表展出類似成果，說明 Sildenafil 類緣物似乎有氾濫之現象。為此，在未知類緣物之療效或毒性或副作用，並為保障民眾健康前提下，雖然有越來越多類緣物陸續被發現與檢出，本局仍不能鬆懈此部份之工作。更應加緊腳步，隨時蒐集各國資訊與國際接軌。
5. 年會中之專題演講或壁報論文，著墨許多有關中草藥或 deitary supplement 之毒素、微生物或重金屬污染分析方法開發或確效。目前我國尚未對上三項訂定完整限量標準，僅散諸於某些中藥材中。因此，未來對此之研發與規格之訂定，需加緊腳步，以提昇中草藥之品質。
6. 完善之中藥材的品質管制，有助於中藥品質之提昇，如何達到並建立完善相關規範，應

需政府主管機關、學界、業界共同參與努力。

7. 參與國際研討會對於研究新知、科技發展、甚至人文素養都有相當大收穫，應鼓勵同仁多參加相關研討會及儀器分析講座，即使為跨領域之研討會，亦可從中了解其他領域之研究現況與模式，有助跳脫狹隘之思緒，進而使思路清晰，可提昇研究水準並合乎持續終身學習之觀念，增長執行公務之專業能力。
8. 本局在中草藥或食品之檢驗分析累積多年多研究成果,應鼓勵同仁儘量參加學術研討會。藉參加會議，除展現本局研究成果可提升國家能見度外，更可與各領域人才接觸，透過面對面溝通，促進國際合作，藉以經營國際新關係。
9. 今年有不少食品安全討論會，除摻偽更提到過敏原之分析，未來產品自原料、製造及銷售管理與分析方法及清潔之確效上將更趨嚴謹。我國中藥雖早已實施 GMP 制度，但仍尚未能執行 cGMP，諸多產品行銷於國外，未來是否會面對更新之衝擊，應小心因應。建請主管當局，考慮是否需汲取西藥實施 cGMP 經驗，選擇適用於中藥之部份，以提昇我中藥產品品質。

活動剪影

附件

＜附件一＞

Analysis of Heavy Metals (Cadmium, Lead and Mercury) in Rice

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P-M-050

Abstract

Rice is the dominant staple food for Taiwan people. However, it may cause harm to human health because of accumulating some toxic heavy metals, such as cadmium (Cd), lead (Pb), mercury (Hg). In order to investigate the levels of heavy metals in rice, samples from the rice millers were collected from April to October, 2012. Analyses were carried out by dry ashing and graphite furnace atomic absorption spectrometry for Cd and Pb. Mercury was determined by a mercury atomic fluorescence spectrometer after the microwave digestion pretreatment. The accuracy was evaluated with 91.1%-95.0% recoveries compared with the certified reference materials-Rice Flour (NIST 1568ª) and Apples Leaves (NIST 1515). The results showed that the mean levels of Cd, Pb and Hg in rice were found 0.04 (not detected-0.17 mg/kg), 0.02 (not detected-0.10 mg/kg) and 0.003 (not detected-0.016 mg/kg), respectively. The heavy metals in rice have been monitored from 2002 to 2012 for lasting 11 years and the total average values of Cd, Pb and Hg were 0.05, 0.02 and 0.003 mg/kg, respectively. All the levels of Cd, Pb, and Hg in rice which planted in Taiwan were below the maximum tolerance levels of 0.4, 0.2, and 0.05 mg/kg set by the government of health.

Material and methods

In order to acquire a universality of the samples, this project reached out to the rice mills of 15 counties and cities to test rice specimens of the first and second phase crops, a total of 160 items. Fine-weighing five grams of milled homogeneous rice samples, we pre-treated it via dry ashing, and then detected Cd and Pb content via Zeeman Graphite Furnace Atomic Absorption Spectrometry . Furthermore, one gram of milled homogeneous rice was digested via Focused Microwave Digester, and then detected the Hg content via Mercury Fluorescence Spectrophotometer.

Results and Discussion

I. The Quality Control Analysis

1. Standard curve linear regression coefficient of determination (r^2): greater than 0.999.

2. Testing each batch (<20) of rice, we made a blank analysis, repeated analysis, and analysis of certified reference materials at least once. Results of the analysis were that blank analysis values were within the upper and lower values of control (-0.00023-0.00017 mg/kg) (Figure 1-A). Repeated analysis was below control upper value (0.56%) (Figure 1-B).

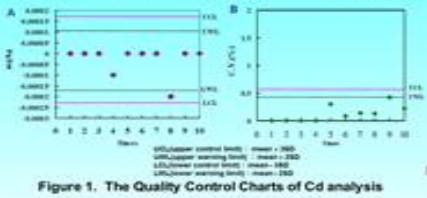


Figure 1. The Quality Control Charts of Cd analysis

3. Certified reference material (CRM) 1568a rice flour and 1515 apple leaves were used to validate the analysis of Cd, Pb, and Hg. The certified values and measured values are shown as Table 1. Cadmium, Pb, and Hg recovery rates were between 80 to 110%.

Table 1. The validation of operation procedures using CRMs

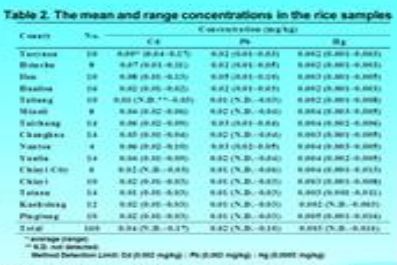
Element	Times	CRM	Certified Value (mg/kg)	Measured Value (mg/kg)	Recovery (%)
Cd	10	NIST 1568a (Rice flour)	0.022 ± 0.002*	0.020 ± 0.001	91.7 ± 3.5
Pb	10	NIST 1515 (Apple leaves)	0.470 ± 0.024	0.428 ± 0.031	91.1 ± 6.6
Hg	10	NIST 1515 (Apple leaves)	0.044 ± 0.004	0.041 ± 0.003	95.0 ± 7.7

*Average±S.D.

II. Analysis of heavy metal concentrations in rice

Among the 160 samples, the cadmium concentration overall average value was 0.04 mg/kg (not detected - 0.17 mg/kg). The total average value of lead concentration was 0.02 ppm (not detected - 0.10 mg/kg). The average value of mercury concentration was 0.003 ppm (not detected - 0.016 mg/kg). As to test results of 160 samples of rice, cadmium, lead and mercury levels did not exceed the announcement on the "limits criterion of heavy metals in rice" by the Department of Health, Executive Yuan (Cd 0.4 mg/kg, Pb 0.2 mg/kg and Hg 0.05 mg/kg).

Table 2. The mean and range concentrations in the rice samples



III. Comparison on previous results of the heavy metal concentrations in rice over the years

In order to monitor the concentrations of heavy metals in rice, testing program for rice from rice mills across Taiwan has been executed since 2002, 11 years so far, a total of 1747 samples of rice measured. After being compared with the previous results of heavy metals in rice over the years, the average concentration distribution of Cd, Pb, Hg is as Figure 2, which were 0.04-0.06 mg/kg, 0.02-0.03 mg/kg, 0.002-0.004 mg/kg respectively.

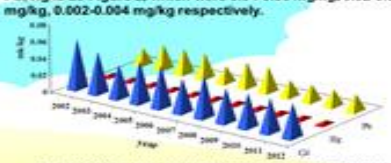


Figure 2. The average concentrations distribution of Cd, Pb and Hg in rice samples collected from 2002 to 2012.

Conclusion

The survey results show that the heavy metal contents in Taiwan produced rice are consistent with food hygiene regulations of our country, and monitoring results since the year 2002 so far up to 11 years show that harmful heavy metals content is low in Taiwan produced rice.

＜附件二＞

Identification of Fip-fve Protein in Golden Needle Mushroom (*Flammulina velutipes*) Using a On-line Desalting HPLC-UV-ESI-MS Profiling Method

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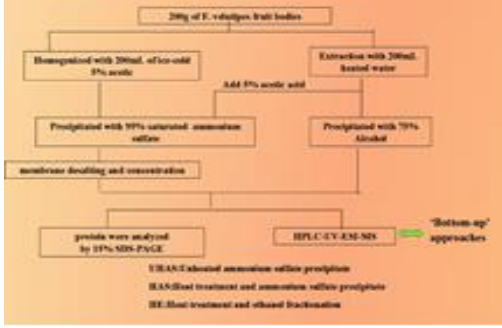
Abstract

The aim of this study is to develop a fast analytical approach to monitor the stability of FIP-fve, a fungal immunomodulatory protein from *Flammulina velutipes*, with different processing conditions. Effects of thermal treatment and ethanol fractionation on golden needle mushroom (*Flammulina velutipes*) were investigated to decide processing condition. We improved the procedures by on-line desalting HPLC-UV-ESI-MS system. On-line desalting HPLC-UV-ESI-MS system provided the desalting, qualitative and quantitative of FIP-fve analysis. UV spectral data showed accurate information for the quantity of FIP-fve. Furthermore, HPLC-UV-ESI-MS method could give valid information as confirmed by ESI-MS. In the chromatographic analysis effects of different columns, modifiers and column oven temperatures were tried for the development of the most suitable analysis conditions. The results from chromatographic analysis showed that using TFA as modifiers with C18 reversed phase columns could get efficient separation. The results showed that heating processes could cause 99% FIP-fve becoming difficult to purify from fruiting bodies. However, it was stable at 100℃ for 1 min in lower concentration of UHSA (22 mg/mL). 15 mg of Fip-fve was obtained from 100g of fresh fruit bodies. UV detection is performed at 280 nm. The calibration curve was found to be linear in the concentration range of 0.29 – 4.69 mg/mL (r^2 = 0.9999). FIP-fve could not be measured with SDS-page and mass spectrum after the procedure of ethanol fractionation. This whole experiment design comparing to the traditional analysis approach of proteins could reduce overall experiment workload by over 80%.

Experimental procedure



Results

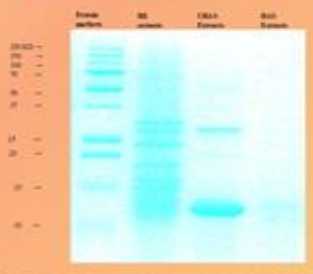


Fig. 1 SDS-PAGE analysis. Electrophoresis was carried out on 15 % SDS-PAGE. Lane 1: Molecular mass markers from bio-Rad indicate 250 kDa, 150 kDa, 100 kDa, 75 kDa, 50 kDa, 37 kDa, 25 kDa, 20 kDa, 15 kDa and 10 kDa from the top. Lane 2: HE extracts, Lane 3: UHAS extracts, Lane 4: HAS extracts

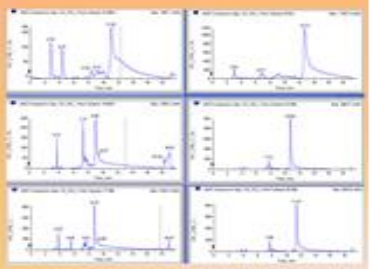


Fig. 2 Chromatograms of UHAS extracts obtained from three commercially available columns with two modified TFA, formic acid: Inertsil C-18, 5μm, 150mm × 4.6mm I.D., CTD C-18, 5μm, 150mm × 2.0mm I.D., SCE C-8, 5μm, 250mm × 4.6mm I.D. The mobile phase consisted of a combination of A (0.1% formic acid in water) and B (0.1% formic acid in acetonitrile), or a combination of A (0.1% trifluoroacetic acid in water) and B (0.1% trifluoroacetic acid in acetonitrile)

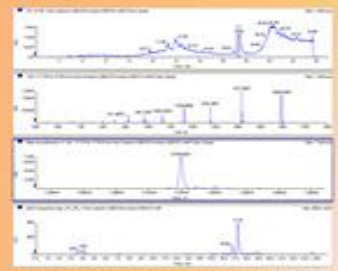


Fig. 3 LC/MS run of a UHAS extract run on the Inertsil column. A) is the TIC for the LC/MS run, B) is the MS spectra of the FIP-fve peak in the chromatogram, C) shows with reconstructed mass based on the multiple charge states, and D) is UV-240nm chromatogram. ABI Bioanalyst software was used to generate the reconstructed parent mass. Raw mass spectrum illustrating the distribution of charge states observed from m/z 700 to 1600.

Conclusion

To conclude, a method based on HPLC-UV-ESI-MS has been established for qualitative and quantitative of FIP-fve analysis. This method was found to be an excellent technique for determination small proteins in gold needle mushroom. Evidence for FIP-fve was found from the UV and mass spectra. The centrifugal filter devices and on-line desalting HPLC-UV-ESI-MS system were instead of dialysis, purification column and bottom up approach to quantitate FIP-fve. The processes could save ca 90% time comparing to traditional analysis approach of protein. UV and mass spectrum results showed that the procedures of heating and ethanol fractionation could cause FIP-fve becoming difficult to purify from fruiting body. Finally, in this study, the final identification of protein required digestion from LC separation, and the analysis of the proteolytic peptide fragments in combination with database searching.

＜附件三＞

Multiclass analysis of 17 veterinary drugs in milk by liquid chromatography–electrospray tandem mass spectrometry

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Abstract

A simple and reliable method using ultra performance liquid chromatography–electrospray tandem mass spectrometry (UPLC–MS/MS) has been developed for the determination of 17 veterinary (multiclass) drugs in milk. The analytes were extracted by acetonitrile, and LC separation was performed on a *high strength silica* based C_{18} column in gradient mode. Data acquisition under MS/MS was achieved by applying multiple reaction monitoring (MRM) of two ion transitions per compound to provide a high degree of specificity. Results showed good repeatability, and recoveries for the 10 macrolide, 2 β-lactam and 2 lincosamide antibiotics and 3 other veterinary drugs (novobiocin, orbifloxacin and virginiamycin M1) used in milk averaged 67.7–108.3%, 81.1–99.4%, 87.0–109.5% and 85.1–110.6%, respectively. The coefficients of variation (C.V.) of the recoveries were less than 10% for intraday and interday precisions. The limits of quantification (LOQs) were in the range 0.5–10 ng/ml. Overall, this method is a suitable and rapid tool to confirm the presence of 17 veterinary drug residues in milk.

Materials and Methods

Materials

Fresh milk samples were purchased from supermarkets.

Sample preparation

Two milliliters of blank fresh milk were transferred to polypropylene centrifuge tubes (50 ml) and extracted with 15 ml of acetonitrile. The mixture was then vortexed for 1 min and centrifuged at 4000 rpm for 10 min. The upper layer was removed and evaporated under nitrogen to dryness at 35 °C. The residue was reconstituted in 1 ml of 50% methanol and filtered through a 0.2 μm PVDF filter. Finally, 10 μl were injected into the UPLC–MS/MS system under the optimized conditions.

LC-MS-MS analysis

The LC separation was performed on Waters ACQUITY UPLC System with a HSS T3 column (1.8 μm, 2.1*100 mm). The mobile phase consisted of a gradient of 0.1% formic acid in water (solvent A) and 0.1% formic acid in acetonitrile (solvent B) at a flow rate of 0.3 mL/min showed in Table 1. The mass spectrometry measurement was performed on a triple quadrupole mass spectrometer XevoTM TQ from WATERS. The instrument was working with an electrospray ion source (ESI) in positive mode under multiple reaction monitoring (MRM) conditions which are shown in Table 2. The following mass spectrometer parameters were used for all substances: capillary voltage, ion source temperature, desolvation temperature, desolvation flow, con gas flow were 1.82 kV, 150℃, 600 ℃, 1200 L/hr and 26 L/hr respectively.

Method validation

Recovery was performed in triplicate by analysing blank samples, which was fortified at four concentration levels (25, 50, 100 and 200 ng/g) by using matrix-matched calibration spiking blank extracts at six different concentration levels (from 10 to 300 ng/g). Intra-day precision was studied at four concentration levels (25, 50, 100 and 200 ng/g), using triplicate per concentration level. Inter-day precision was studied spiking blank samples at the same concentration levels, and they were analysed at three different days. Limits of detection (LOD) and limit of quantification (LOQ) were determined as the minimum concentration of analyte providing a signal to noise (S/N) ratio with 3 and 10 as the minimum.

Table 1. Parameters of liquid chromatography conditions

Mobile phase	A: Water, containing 0.005% formic acid. B: Acetonitrile		
Gradient program	Time (min)	A (%)	B (%)
	0.0	100	0
	0.0→1.0	100→90	0→10
	1.0→5.0	90→60	10→40
	5.0→8.0	60→5	40→95
	8.0→10.0	5→0	95→100
	10.0→12.0	0→0	100→100
	12.0→12.1	0→100	100→0
	12.1→15.0	100→100	0→0
Flow rate	0.3 mL/min		
Injection volume	10 μL		
Analysis time	15 min		

Result and Discussion

Optimization of the extraction procedure

Sample preparation is often the most critical part of a multi-residue antibiotic method due to the different properties of the substances that have to be extracted simultaneously. Additional treatments and clean-up steps after the extraction step were investigated. Addition of n-hexane to the ACN extracts and additional clean-up with HLB were evaluated. Certain antibiotics (tylosin, spiramycin, neospiramycin, josamycin and kitasamycin) had lower recoveries when n-hexane was used, while others (i.e.,virginiamycin and tulathromycin) had lower recoveries when n-hexane and HLB were used. The highest recoveries were obtained with acetonitrile alone compared to the addition of n-hexane or HLB to the extraction (Figure 1).

Validation of method

Validation parameter including recoveries, intra-day and inter-day coefficient of variation, LODs and LOQs. Calibration was performed by use of matrix-matched calibration standards. The recoveries of macrolides were 67.7-108.3%, β-lactam antibiotics were 81.1-99.4%, lincosamides were 87.0-109.5%, miscellaneous were 85.1-110.6%, the results are summarized in Table 3. LODs and LOQs were tested by analysing blank samples, which was fortified seven concentrations (0.5, 1, 2, 5, 10 and 15 ng/g). The LOQ values of macrolides were between 1-10 ng/g, β-lactam antibiotics were between 1-5 ng/g, licosamides were 1 ng/g, miscellaneous were 1-5 ng/g, the results are summarized in Table 3. Four concentrations of mixed standard solutions of the seventeen antibiotics were used for analyzing the intra-day and inter-day repeatability. Each concentration was analyzed three times for intra-day repeatability. For inter-day repeatability, each concentration was analyzed four times for three days. The coefficients of variation of intra-day and inter-day assays were lower than 8.8% and 9.9%, respectively (Table 4).

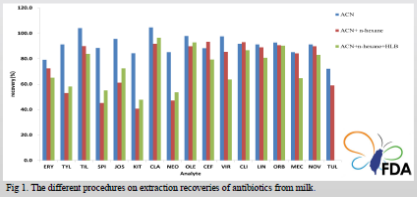

Fig 1. The different procedures on extraction recoveries of antibiotics from milk.

Table 2. The MRM transitions and parameters of 15 veterinary drugs and internal standard

Compound	Abbreviation	Retention time (min)	Parent ion (m/z)	Transition 1 (CE)	Transition 2 (CE)	Transition 3 (CE)	Cone voltage
Clarithromycin	CLA	6.5	748.7	115.9 (44)	158.0*(32)	590.5(20)	28
Erythromycin	ERY	5.7	734.6	116.0 (46)	158.1*(32)	576.5(18)	26
Josamycin	JOS	6.7	828.7	109.0*(44)	174.1 (34)	229.1(32)	46
Kitasamycin	KIT	6.4	772.6	109.0*(44)	174.1(32)	215.1(30)	46
Neospiramycin	NEO	4.0	699.6	142.1 (22)	174.1*(30)	540.4(20)	34
Oleandomycin	OLE	5.8	688.6	116.0 (42)	158.0*(28)	544.5(16)	24
Spiramycin	SPI	4.4	843.7	100.9 (40)	142.0(34)	174.0*(38)	48
Tilmicosin	TIL	5.1	869.8	115.9 (64)	132.0(50)	174.1*(46)	70
Tylosin	TYL	6.0	916.8	100.9 (50)	145.0(40)	174.0*(40)	50
Virginiamycin M1	VIR	6.8	526.4	231.0 (36)	337.1(22)	355.2*(18)	24
Cefoperazone	CEO	4.8	646.4	143.1*(40)	530.2(10)	--	18
Mecillinam	MEC	4.0	326.3	122.1 (36)	139.1(30)	167.1*(22)	32
Clindamycin	CLI	4.7	425.3	126.1*(28)	377.2(20)	389.3 (18)	30
Lincomycin	LIN	3.2	407.3	126.1*(30)	172.1(22)	359.3(18)	32
Orbifloxacin	ORB	4.1	396.3	226.1 (42)	267.1(36)	295.1*(24)	32
Novobiocin	NOV	8.1	613.4	133.0 (60)	189.1(28)	218.1(14)	22
Tulathromycin A	TUL	3.7	806.7	116.1 (50)	158.1(44)	577(24)	26

* Transitions with bold numbers were used for quantification.
** Relative standard deviation (RSD) is given in parentheses (n=18).

Conclusions

A multiresidue method was developed for rapid and simultaneous determination of 17 antibiotics in milk by LC/MS/MS. The rapid extraction and the appropriate clean-up procedure provide good validation parameters, make it suitable for the routine residue monitoring.

Table 3 The recoveries, limits of detection (LODs) and limits of quantitation (LOQs) of 17 veterinary drugs in milk

Drug / Spiked standard (μg·kg⁻¹)	Recovery (%) n = 4				LOD (μg·kg⁻¹)	LOQ (μg·kg⁻¹)
	25	50	100	200		
Macrolides						
clarithromycin	106.5	102.6	104.6	104.2	0.5	1
erythromycin	86.6	78.5	79.1	79.2	1	2
josamycin	80.1	84.9	95.6	94.9	0.5	1
kitasamycin	74.6	74.9	84.3	85.5	5	10
neospiramycin	79.5	78.2	85.1	83.1	5	10
oleandomycin	102.9	95.2	97.9	97.4	1	2
spiramycin	90.7	79.0	88.4	88.0	2	5
tilmicosin	108.3	102.8	104.1	102.8	2	5
tulathromycin A	90.6	67.7	72.1	73.8	5	10
tylosin	82.4	83.0	91.2	91.1	1	2
β-lactam						
cefoperazone	96.5	88.1	88.2	89.3	2	5
mecillinam	99.4	87.1	85.1	81.1	0.5	1
Lincosamides						
clindamycin	109.5	94.6	91.8	90.6	0.5	1
lincomycin	106.5	94.0	91.2	87.0	0.5	1
Miscellaneous						
novobiocin	108.1	95.8	93.0	92.5	2	5
virginiamycin M1	110.6	97.9	97.5	96.3	2	5
orbifloxacin	105.3	95.1	92.7	85.1	0.5	1

Table 4 Intra-day and inter-day coefficient of variation of 17 veterinary drugs in milk at various spiked levels

Drug / Spiked standard (ppb)	Intra-day / Inter-day, C.V. (%) n = 4			
	25	50	100	150
Macrolides				
clarithromycin	3.1/5.1	0.7/1.8	2.1/2.7	1.6/1.4
erythromycin	1.7/1.9	1.1/2.0	4.0/5.6	2.2/2.4
josamycin	5.5/5.4	4.5/4.7	3.1/2.1	2.4/1.5
kitasamycin	6.3/8.1	8.8/9.9	8.1/8.1	5.3/4.1
neospiramycin	6.0/6.1	7.8/6.0	3.4/3.1	1.8/1.2
oleandomycin	2.5/3.7	1.6/1.9	3.2/2.8	1.4/1.0
spiramycin	5.1/6.9	7.6/8.0	3.9/3.7	3.0/2.4
tilmicosin	0.8/4.0	1.9/3.0	1.9/3.4	0.9/1.8
tulathromycin A	5.1/6.2	6.3/7.5	5.9/5.4	3.0/3.0
tylosin	6.0/5.9	4.4/5.9	3.2/2.1	1.0/1.3
β-lactam				
cefoperazone	6.0/6.2	4.9/4.1	3.3/3.3	2.6/2.5
mecillinam	2.7/3.2	2.5/3.3	1.2/1.5	0.8/1.1
Lincosamides				
clindamycin	4.1/4.1	2.6/2.2	0.6/1.7	1.6/1.9
lincomycin	3.1/2.9	1.0/1.4	0.8/0.8	1.2/1.4
Miscellaneous				
novobiocin	3.3/3.5	1.4/2.2	2.5/3.0	1.1/1.7
virginiamycin M1	3.5/3.3	2.2/2.4	1.8/1.6	0.7/1.4
orbifloxacin	2.9/4.6	0.9/1.7	1.2/1.8	0.4/0.9

＜附件四＞

P-W-027

Isolation and Identification of a Sildenafil Analogue Adulterated in Dietary Supplements

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Taiwan Food and Drug Administration, Ministry of Health and Welfare, Taipei, Taiwan

Abstract

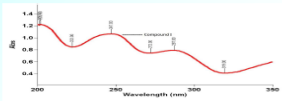
Dietary supplements have been gradually accepted by the public in the recent years as people think that they are natural and safe. Taiwan Food and Drug Administration has been testing the suspected adulterated herbal medicines for more than thirty years. The survey also found several analogues in many dietary supplements marketed for enhancing male sex ability. A new sildenafil analogue was detected from five consecutive dietary supplements in 2011. The new compound was isolated from a soft gel marketed product a by column chromatography and then determined by NMR, mass spectrum, and IR. Compared with the structure of sildenafil, 4-methyl-piperazine moiety is converted to dimethylpiperazine, sulfonyl and carbonyl groups are replaced by thiocarbonyl group. This compound has not been approved as medication but is listed a screening item in our laboratory for the public health.

Experimental

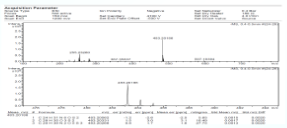
8.2 g samples + 150 mL Ethanol
↓
Shake 30 min, centrifuge 10 min at 3000 rpm, repeat 2 times
↓
Combine supernatant, concentration obtained 1.5 g extract
↓
Column chromatography (Silica gel, elution solvent: CH2Cl2-MeOH) and re-crystalization
↓
Compound I (76.3 mg)
↓
TLC/UV | IR | HREIMS | NMR

Results and Discussion

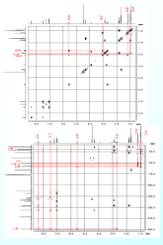
1. Yellow needle-like crystal, m.p.192-194 ℃
2. UV spectrum is similar to Dithio-desmethylsildenafil

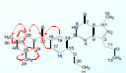


3. IR: 3260 cm⁻¹ (amine),1571 cm⁻¹ (aromatic ring), 1249 cm⁻¹ and 1245 cm⁻¹ (two thio-carbonyl)
4. HRESI: *m/z* 483 [M-H]⁻

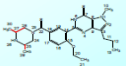


5. NMR data





The HMBC spectrum of compound I

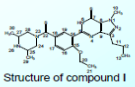


The COSY spectrum of compound I

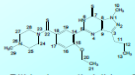
Table 1. NMR data of compound I

No.	$^1H(\delta_H)$	$^{13}C(\delta_C)$	DEPT	COSY	HMBC
3	-	146.2	0	-	
5	-	147	0	-	
7	-	171.7	0	-	
8	-	132.2	0	-	
9		134.1	0	-	
10	4.49, 3H, *s*	39.3	1	-	C-8
11	2.90, 2H, *t*, *J*= 7.4 Hz	27.5	2	H-12	C-3,C-9,C-12,C-13
12	1.82, 2H, *m*	22.2	2	H-13,12	C-3,C-12,C-13
13	0.98, 3H, *t*, *J*= 7.4 Hz	13.9	1	H-12	C-11,C-12
14	-	118.5	0	-	
15	-	156.7	0	-	
16	7.03, 1H, *d*, *J*= 8.6 Hz	112.9	3	H-17	C-14,C-15,C-5,C-18,C-19
17	7.49, 1H, *dd*, *J*= 2.3, 8.6 Hz	131.4	3	H-16	C-14,C-15,C-16,C-19,C-22
18	-	136.3	0	-	
19	8.38,1H, *d*, *J*= 2.3 Hz	128.1	3	-	C-5,C-14,C-17,C-15,C-22
20	4.32, 2H, *dq*, *J*=2.5, 7.0 Hz	65.9	2	H-21	C-15,C-21
21	1.66, 3H, *t*, *J*=6.9 Hz	14.7	1	H-20	C-20
22	-	199.2	0	-	-
24	3.91, *brd*, *J*=11.9 Hz; 2.78, *dd*, *J*=10.1, 11.9 Hz	58.7	2	H-25	C-28
25	2.92,1H, *m*	51.5	3	-	-
27	3.07, 1H, *m*	50.4	3	H-28	-
28	5.57, *brd*, *J*=12.6 Hz; 2.70, *dd*, *J*=11.6, 12.6 Hz	56.01	2		C-27,C-24,C-22,C-30
29	0.97, 3H, *d*, *J*=6.0 Hz	19.07	1	H-25	C-25,C-24
30	1.19, 3H, *d*, *J*=6.0 Hz	19.42	1	H-27	C-27,C-28

δppm in $CDCl_3$, *J* in Hz, 125 MH_z for ^{13}C, 500 MH_z for ^1H; DEPT is the number of attached protons.



Structure of compound I



Dithio-desmethylsildenafil

Conclusion

In this present, a new sildenafil analogue is founded and then isolated from a dietary supplement, and its structure is determined by UV, IR, Mass Spectrum and NMR. The chemical name is 5-(5-(3, 5-dimethylpiperazine-1-thioyl)-2-ethoxyphenyl)-1-methyl-3-propyl-1H-pyrazolo[*4, 3-d*]pyrimidine-7(6H)-thione. Until now, sildenafil, tadalafil and vardenafil are approved as erectile dysfunction medicines in Taiwan. Totally 28 analogues has been found during the pass ten years. Literature searching indicates more than 50 analogues has been identified, meaning these structure-like marketed medicines has spread widely. Analogue's structures are similar to approved medicines, detailed pharmacological and toxicological studies are still lack. Consumers taking unapproved medicines without labeling may be in a risk of health.

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2013 Annual Meeting Schedule At-A-Glance

SATURDAY, AUGUST 24, 2013

9:00 am -5:00 pm	Registration Open

SUNDAY, AUGUST 25, 2013

7:30 am -7:00 pm	Registration Open
9:00 am -11:00 am	AOAC INTERNATIONAL Board of Directors Meeting
1:00 pm -3:00 pm	SMPR/Standards Educational Session
1:00 pm -4:30 pm	TDLM Workshop: Estimating and Using Measurement Uncertainty
1:00 pm -6:00 pm	SPIFAN Working Groups: Minerals, Vitamin K, Biotin, and FOS/GOS
2:00 pm -4:00 pm	OMA Expert Review Panel (Microbiology Methods 1)
4:00 pm -6:00 pm	OMA Expert Review Panel (Chemistry Methods)
6:00 pm -8:00 pm	Exhibit Hall Grand Opening Reception
8:00 pm -10:00 pm	President's Welcome Reception

MONDAY, AUGUST 26, 2013

7:00 am -8:00 am	TDRM Executive Committee Meeting
7:30 am -5:00 pm	Registration Open
8:00 am -8:30 am	Continental Breakfast
8:30 am -10:30 am	Keynote Address and Awards Ceremony
10:00 am -5:00 pm	Exhibit Hall Open
10:00 am -5:00 pm	Poster Presentations: Analysis of Foodborne Contaminants and Residues, Analysis of Non-Foodborne Contaminants and Residues, Microbiological Methods, Water and Waste Water Analysis, and General Pharmaceutical Analysis and Evaluation
10:30 am -11:00 am	Exhibitor/Partner Presentation: Pickering Laboratories
10:30 am -11:30 am	Latin America Section Business Meeting
10:30 am -12:00 pm	Agricultural Materials Community Meeting
11:30 am -12:30 pm	Exhibitor/Partner Presentation: Agilent Technologies
11:30 am -1:00 pm	Poster Author Presentations
12:30 pm -1:00 pm	Exhibitor Presentation: bioMérieux
1:00 pm -1:30 pm	H.W. Wiley Award Address
1:00 pm -3:00 pm	SPIFAN Expert Review Panel, Whey Protein
1:00 pm -5:00 pm	OMA Expert Review Panel (Proprietary Vitamin Methods)
1:30 pm -3:00 pm	Wiley Award Symposium: Evolution of Proprietary Methods in AOAC
1:30 pm -3:00 pm	Symposium: Approaches to Improving and Demonstrating Method and Laboratory Performance
1:30 pm -3:00 pm	Symposium: Antibiotics in the Chain
3:00 pm -3:30 pm	Refreshment Break
3:00 pm -3:30 pm	Exhibitor Presentation: SPEX SamplePrep
3:30 pm -5:00 pm	Symposium: New Ideas in Statistical Methods for Method Validation: Designs and Analyses
3:30 pm -5:00 pm	Symposium: Microbiological Method Validation and Verification: Assuring Reliable Results
3:30 pm -5:00 pm	Symposium: Emerging and New Contaminants in Food Commodities from Plant Origin: Analytical Methods and New Issues
5:00 pm -5:30 pm	Exhibitor Presentation: ANKOM Technology
5:00 pm -6:30 pm	ALACC Meeting
5:00 pm -6:30 pm	New Member Welcoming Reception
5:00 pm -7:00 pm	Chemical Contaminants and Residues in Food Community Meeting
6:00 pm -7:00 pm	Japan Section Business Meeting
6:00 pm -7:00 pm	Taiwan Section Business Meeting
6:30 pm -7:30 pm	Reception for TDLM Members, Sponsored by Microbiologics
7:00 pm -8:00 pm	Joint Asian Sections Meeting

TUESDAY, AUGUST 27, 2013

7:15 am -8:15 am	Exhibitor/Partner Presentation: Waters Corporation
7:30 am -5:00 pm	Registration Open
7:45 am -8:15 am	Refreshment Break
8:00 am -12:00 pm	AAFCO Meeting
8:00 am -12:00 pm	SPSFAM Expert Review Panel - Heavy Metals
8:15 am -9:45 am	Symposium: Immunoanalytics for Water and Waste Water Analysis
8:15 am -9:45 am	Symposium: Advanced Strategies and Tools for the Identification of Unknowns in Food
8:15 am -11:45 am	TDRM/TDLM Workshop: Building the Use of Certified Reference Materials into Dispute Resolution
9:45 am -10:15 am	Exhibitor/Partner Presentation: Thermo Scientific
10:00 am -10:30 am	Refreshment Break
10:00 am -12:00 pm	Water/Waste Water Community Meeting
10:00 am -12:00 pm	Committee on Statistics Meeting
10:00 am -5:00 pm	Exhibit Hall Open
10:00 am -5:00 pm	Poster Presentations: Food Nutrition and Food Allergens, Emerging Issues in Food Safety and Security, *Performance Tested Methods*℠, and General Methods, Quality Assurance and Accreditation
10:15 am -11:45 am	Symposium: Pathogen Testing in the 21st Century: Genotyping Methods for High-Level Discrimination of Foodborne Pathogens
10:15 am -11:45 am	Symposium: New Blood 2013: Developing Methods for Detection of Chemical Contaminants

11:30 am -1:00 pm	Poster Author Presentations
12:00 pm -1:00 pm	Exhibitor Presentation: AB SCIEX
12:30 pm -2:30 pm	Committee on Sections Meeting
1:00 pm -1:30 pm	Partner Presentation: Covance Laboratories
1:00 pm -3:00 pm	AOAC Research Institute Advisory Council Meeting
1:00 pm-7:00 pm	SPIFAN Expert Review Panel, Nutrients
1:30 pm -3:00 pm	Contaminants Subgroup Meeting - Unknowns
2:00 pm -2:30 pm	Exhibitor Presentation: Neogen Corporation
2:15 pm -2:45 pm	Refreshment Break
3:00 pm -4:30 pm	Symposium: Oral Posters from Dietary Supplements and Botanicals
3:00 pm -4:30 pm	Symposium: Quantitative and Qualitative Analysis of Emerging Substances of Concern (ESOCs) Using Liquid Chromatography - High Resolution Mass Spectrometry (LC-HRMS): Method Development, Method Verification/Validation, and Method Harmonization
3:00 pm -4:30 pm	Symposium: Food and Feed Test Materials for Mycotoxin Analysis
3:00 pm -5:00 pm	OMA Expert Review Panel (Microbiology Methods 2)
4:00 pm -5:00 pm	TDLM Executive Committee Meeting
4:30 pm -5:00 pm	Exhibitor Presentation: Phenomenex
4:30 pm -6:00 pm	Membership Committee Meeting
4:30 pm -6:00 pm	Contaminants Subgroup Meeting - Veterinary Drugs
4:30 pm -7:30 pm	Mycotoxin Community Meeting
5:00 pm -6:00 pm	TDRM Members Meeting
5:00 pm -7:00 pm	Committee on Safety Meeting
5:15 pm-7:00 pm	2nd Annual bioMérieux Food Safety Signature Workshop
5:15 pm -8:15 pm	Food Allergen Community Meeting
5:30 pm -6:00 pm	Exhibitor Presentation: Bruker Optics, Inc.
6:00 pm -7:00 pm	TDRM Members Reception, Sponsored by Silliker
6:00 pm -7:30 pm	Europe Section Executive Committee Meeting
6:15 pm -7:45 pm	Contaminants Subgroup Meeting - Metals
6:30 pm -7:30 pm	China Section Business Meeting

WEDNESDAY, AUGUST 28, 2013

7:15 am -8:15 am	Exhibitor Presentation: AB SCIEX
7:30 am -5:00 pm	Registration Open
7:45 am -8:15 am	Refreshment Break
8:15 am -9:45 am	Symposium: AOAC INTERNATIONAL Stakeholder Panels Update: ISPAM, SPADA, SPIFAN, and SPSFAM
8:15 am -9:45 am	TDLM Symposium: Laboratory Accreditation and the Food Safety Modernization Act
8:15 am -9:45 am	Symposium: Selection of Analytical Methods for Validation of Allergen Control Plans
9:45 am -10:15 am	Exhibitor Presentation: Microbiologics
10:00 am -10:30 am	Refreshment Break
10:00 am -12:00 pm	AOAC Research Institute Board of Directors Meeting
10:00 am -5:00 pm	Poster Presentations: Authenticity, Botanicals and Dietary Supplements, and Detection and Measurement of Natural Toxins
10:15 am -11:45 am	Symposium: Advances in the Detection of Marine and Freshwater Toxins
10:15 am -11:45 am	Symposium: Recent Advances in Dietary Fiber and Available Carbohydrates
10:15 am -11:45 am	Symposium: What Comes Before the MS in LC-MS Food Allergen Analysis?
11:30 am -1:00 pm	Poster Author Presentations
11:45 am -1:00 pm	Technical Programming Council Meeting
12:00 pm -12:30 pm	Exhibitor Presentation: Rocky Mountain Diagnostics
1:00 pm -2:30 pm	Symposium: Will We Remain Microbiologists or become Chemists? Mass Spectrometry Platforms for the Characterization and Identification of Microbial Pathogens
1:00 pm -2:30 pm	Hot Topic Symposium: Authenticity Challenges – Yesterday, Today and Tomorrow
1:00 pm -2:30 pm	Symposium: "Chasing Gluten" What Can you Detect? How to Deal with the Results?
2:30 pm -3:00 pm	Exhibitor Presentation: Advion, Inc.
2:30 pm -3:00 pm	Refreshment Break
2:30 pm -3:30 pm	Meet Your Board of Directors
3:00 pm -4:30 pm	Symposium: International Symposium on the Challenges with the Analysis of Probiotics
3:00 pm -4:30 pm	Symposium: Advancements in Methods of Sampling and Analysis for Agricultural Materials
3:00 pm -4:30 pm	Symposium: Food and Agriculture Surveillance Screening, a Predictor of Chemicals of Concern
3:00 pm -5:00 pm	Update on Tea Collaborative Study
4:30 pm -6:00 pm	AOAC INTERNATIONAL Business Meeting
8:00 pm -11:00 pm	Annual Meeting Closing Reception

THURSDAY, AUGUST 29, 2013

7:30 am -12:00 pm	Editorial Board Meeting
8:30 am -12:00 pm	Food Industry Analytical Chemists Share Group Meeting
9:00 am -4:00 pm	Official Methods Board Meeting
1:00 pm -5:00 pm	Juice and Juice Products Community Meeting