出國報告（出國類別：研究）

參加「國際公定分析化學家協會(AOAC International)第125屆年會研習國際新穎性食品檢驗技術」報告

服務機關：行政院衛生署食品藥物管理局
姓名職稱：張美華 薦任技士
派赴國家：美國
出國期間：100 年 9 月 16 日至 100 年 9 月 24 日
報告日期：100 年 12 月 14 日

摘要

國際公定分析科學家協會(AOAC INTERNATIONAL)第125屆年會，今(2011)年於美國路易斯安那州紐奧良市之喜來登飯店(Sheraton Hotel)舉行，於9月18日起，會期共計4日，內容包括訓練課程、科學會議、壁報論文、廠商展示、專家會議及AOAC分會會議等。本次會議之學術演講內容豐富廣泛，與會專家學者來自全球各地，包括產業、政府單位、學術機構代表，共同分享交換分析檢驗之經驗、成果。

今年之專題演講主題爲「由學者、政府管理者及產業提倡者之整合觀點看食品安全」。口頭論文包括「微囊藻毒素：以LC/MS/MS建立魚類檢體快速分析方法及確效」等112篇學術論文。在壁報論文方面，共計有「以GC串聯質譜多重殘留分析375種化合物，含農藥、多環芳香族碳氫化合物及多氯聯苯」等11類主題，250篇論文發表，本局共計有3篇論文於大會中展示，內容涵蓋食品、食品容器之三聚氰胺檢驗、食因性水樣中病毒檢驗及豬肝檢體之多重動物用藥殘留檢驗等相關議題。

本人並參加「standard method performance requirements, SMPRs」訓練課程，瞭解AOAC爲使檢驗方法產出量增加、產出速度加快、方法更有彈性及信賴度，制定SMPRs指引及其相關檢驗方法確效之規範，已將所學內容攜回服務單位與同仁分享，希望提昇同仁於方法開發時多面向之思考。

檢驗方法之開發需要完善之儀器設備、技術深耕人力、檢驗資源之取得，針對方法需求之迫切性列出序位，若因人力、時間受限、技術瓶頸或突發之檢驗案等，參考AOAC徵求方法之作法，可收集國際認可之方法作爲參考檢驗方法以解決燃眉之急。

目次

壹、目的

國際公定分析化學家學會創立於 1884 年，爲非營利性全球組織，經由提供科學性之工具及步驟，使相關單位合作，建立共識，開發適合目的之方法及品質保證措施，達成其「提供全球信賴之分析結果」之願景。120 年來累積的經驗，AOAC 知道如何方法確效並推動制定、核可方法，提昇檢驗結果的信賴和信心，特別是在國際貿易頻繁，在國與國間因檢驗方法之差異衍生的爭議，產生了諸多困擾。AOAC 由代表產業、政府單位、學術機構之相關單位，經由共識，訂定需求方法的優先次序，建立國際認可之確效指引，這一過程確實帶來廣大價值。AOAC 全球共設置 15 個分會，美國、加拿大 7 個分會外，包括中國分會、歐洲分會、日本分會、拉丁美洲-加勒比海分會、低地分會(比利時、盧森堡和荷蘭)、中部加拿大分會、臺灣分會及泰國分會，各分會於年度內邀請區域內之權威或專家進行技術發表或研討，藉由參加分會活動可以瞭解最新之產業現況。每年 AOAC 舉辦年度盛會，參與者包括全球會員、分會代表、各領域之專家學者業者，臺灣分會也會派人參與，年會中展示了分析領域的研究現況、新的研究技術、方法或成果，經由參加年會，可以擴大視野、吸收經驗、會議中的訓練課程提供分析方法有關議題之完整資訊，均可轉化助益於自身檢驗專業之提昇，並且將研討會所見所聞，與同仁分享。

貳、過程

一、行程與工作紀要

日期	工作紀要
100 年 9 月 15 日～16 日	啓程
100 年 9 月 18 日	標準方法性能要求（Standard method performance requirements, SMPRs）一研習課程
100 年 9 月 19 日	參加研討會及壁報論文發表
100 年 9 月 20 日	參加研討會
100 年 9 月 21 日	參加研討會
100 年 9 月 22 日～23 日	回程

二、年會日期、地點及出席

第125屆AOAC年會及壁報展覽今(2011)年於美國路易斯安那州紐奧良市之喜來登飯店(Sheraton Hotel)舉行，各項委員會會議、工作小組會議及訓練課程自9月18日至9月21日結束，共計4天。本次會議學術研討內容豐富，與會各國專家學者眾多。本人及林澤揚技正係代表行政院衛生署食品藥物管理局與會並發表壁報論文，本局尚有研檢組曾素香科長係化學檢驗技術專業受AOAC大會邀請出席動物用藥專家審查小組（Expert review panels, ERPs）會議，廖家鼎技士目前留職停薪，於美國FDA進行博士後研究也來共襄盛舉，此外台灣分析化學家協會孫璐西理事長及其學生除發表壁報論文外，更重要任務係主持「臺灣公定分析化學家協會事務會議」。

三、年會內容

AOAC年會爲國際性大型研討會，議程（附件一）相當豐富，包括主題演講、口頭論文發表及壁報論文展示。主題演講講題爲「由學者、政府管理者及產業提倡者之整合觀點看食品安全」，由美國食物安全衛生學院(Institute for food

safety and technology, IFSH)副總裁Robert E. Brackett博士主講，近年爆發食品安全醜聞事件，突顯食品安全議題之迫切性，首先有待產業、專家學者、官方組織結合，參與專業及科學性會議、參加諮詢委員會、形成公－私合作關係，並由合作關係中改善食品安全。

口頭論文分為「追逐和追蹤食物中化合物的夥伴」、「一次廣泛篩選食物中過敏原－只是一個美夢或可行之計畫」、「植物性藥物膳食補充品產業之關注點」、「食品機構過敏原衛生SOP之驗證」、「偵測化學污染物之方法開發」、「無機物物種分析」、「替代方法及方法驗證－建立全球統一策略」、「食物過敏原之參考物質」、「化學信息學輔助質譜分析－應用、挑戰及未來發展」、「量測不確定度之用途」、「現代分析技術用於評估食品及膳食補充品中微量金屬元素」、「參考物質及實驗室認證之研討會」、「專家承擔Non-O157 STEC之偵測」、「抗氧化劑測試」、「化合物分析之方法確效及方法性能標準」、「方法統一：現行全球確效計畫」、「膳食纖維分析之惟一挑戰」、「吡咯雙烷類生物鹼，食物及飼料中不受歡迎之植物毒素」、「全球肉品產業微生物方法標準」、「海洋毒素LC及LC/MS方法之精鍊、認證及執行」、「新方法－抗菌素療效測試替代法」、「近來動物用藥研究及分析之進步」、「分析食物中小分子污染物之最好訓練」、「質譜儀分析天然毒素標的物/非標的物之新趨勢」及「貝類毒素化合物安定性及代謝」等議題，共計112篇。

壁報論文分為「食源性污染物和殘留物的分析」、「非食源性污染物和殘留物的分析」、「微生物方法」、「藥品分析真實性和安全性」、「植物性食品、寵物食品、動物飼料營養品、添加物及污染物」、「天然毒素之檢測及定量」、「食品安全性之新興議題」、「食品營養及食品過敏原」、「一般分析方法、品質保證及驗證」、「草藥及膳食補充品」及「性能測試方法」等11類，共計250篇。本局今年共有3篇壁報參展，並準備A4規格之小單張，提供對研究主題有興趣之與會者參考，第一篇題目：「Simultaneous Determination of Multiclass Veterinary Drug Residues in Porcine Liver by Liquid Chromatography-Electrospray Tendem Mass Spectrometry.」，第二篇題目：「Virusus In Water Samples from Foodborne Outbreaks in Taiwan (2010).」，第三篇題目：「Determination of Melamine in Foods and Leaching Solutions of Melamine Tableware.」（附件二）。

在廠商展示部分，年會會場超過50家國際知名廠商進駐，提供與會者直接與間接之資訊，與會者也可將自身需求傳達給廠商，達成資訊交流之目的。與會廠

商提供之服務類型非常廣汎，可歸納爲技術諮咨詢服務、實驗室試劑、標準品、標準參考物質、實驗室耗材、器具、設備、氣體、應用軟體等。

由於議程豐富而時間有限，出席之優先序位爲教育訓練，專題演講議題中環境分析物、動物用藥、農藥多重殘留檢測、未知物之分析、重金屬無機砷之物種分析等，空檔時間則觀看壁報展示，透過每一場次參與學習累積出席國際研討會之收穫厚度。

四、「臺灣公定分析化學家協會事務會議」會議內容

會期中由台灣分析化學家協會孫璐西理事長召開「臺灣公定分析化學家協會事務會議」，活動之前孫老師已備妥會議之宣傳單張，部份放置於報到處供有興趣者領取參考，部份於會議期間隨機分送，果然出席者踴躍，有孫老師學生、本局研檢組曾素香科長、林澤揚技正、廖家鼎技士、張美華技士、美國 FDA 成員周家璜博士等、臺灣留美專家、對岸之檢驗相關領域人員及儀器廠商等，清一色爲華人。孫老師於於會議中分享臺灣於 5 月發生之起雲劑添加塑化劑事件之過程，管理當局食品藥物管理局及時應變作爲及管理經驗，曾素香科長也回應與會人員有關檢驗方面之問題及建議，會議於輕鬆愉快氣氛中作交流。

五、訓練課程

本屆年會之訓練課程有別於以往報名收費方式，不收費自行參加，兩個課程爲「標準方法性能要求(standard method performance requirements, SMPRs)訓練」及「AOAC 共同體領導訓練（AOAC community leadership training）」，因爲課程時間相同，選擇與本身檢驗專業有關之「標準方法性能要求」課程，由 AOAC 首席科技官 Scott Coates 主講，目標使出席者瞭解何謂 AOAC SMPR，AOAC SMPR 目的，發展 AOAC 標準之過程，達成 Official First Action 之替代路徑，藉由參與此次課程瞭解 AOAC 方法產生之運作模式，其曾經遭遇之困境，專家會議之召開等。

AOAC 爲國際性標準發展機構，其標準訂立之過程包括透明、公開、平衡利益、正常程序、一致性、申訴處理。產出 Official first action 方法之傳統過程，係方法及確效草案提交 AOAC，AOAC 指定方法委員會之顧問，依據確效指引作決定，研究主任協調及指導實驗室比對及提交稿件，由方法委員會顧問決定

Official Action status，方法經 2 年試用，向 Official Method Board (OMB) 提建議，OMB 複審建議作成 Final action 決議。2010 年只核可 3 件方法，導致顧客失望及共同體缺乏方法來解決問題，爲解決此一困境，AOAC 於 2007 年開始發展所謂 SMPR，2009 年 SMPR 取代「接受標準」，完成水產品抗生素殘留物之 SMPR，由於 2010 年 AOAC 各計畫規格、樣式、方法性能需求及接受標準不一致，AOAC 認爲需要一個標準的過程及格式，因此發展 SMPR 指引(附件三)。2010 年 7 月 SMPR 指引草案於內分泌干擾物化合物計畫作測試，2011 年 3 月 AOAC 董事會(Board of Directors)核可以 AOAC volunteer consensus standards development process 徵求正式方法，作爲產出 Official First Action status 方法之替代方案。

AOAC 方法之替代方案(附件四)其標準發展之概述爲：諮詢小組推荐投票成員，參與 AOAC 方針，擬定主題框架及排序。由相關人士(stakeholders)募集資金成立相關小組(stakeholder panel)，相關人士成員有主席、事項專家、方法開發者、政府或管理者、方法末端使用者、學者、合同研究機構、非政府組織等，panel 受總部管理及 OMB 審查。Panel 下設工作小組(working groups)，擬定 SMRPs 草案，由 stakeholder panel 審議，公布徵求意見。當相關人士發動 SMPR，ERP 成員依據確效資料審慎評估，若通過核可，即爲 Official of First Action 方法，接著由專家成員、方法作者或 AOAC 職員寫成 AOAC 格式之草案或指派實驗室比對、ERP 報告(含科學性背景資料參考文獻等)同時作發表。方法提交後之 2 年期間，ERP 會持續監督方法之性能成果，進一步收集實驗室比對、能力試驗或其他顯示實驗室間重複性良好之測試數據，若結果未達標準即從 Official First Action 除名，反之 ERP 向 OMB 提出同意建議案，由 OMB 授予方法進入到 Final Action。

比較傳統及替代路徑產生方法模式，替代路徑方式可以產生更多的正式分析方法，解決問題的速度加快，充分運用 AOAC 專家會員之長處，方法可以立即使用，產生更多評估成果之有用數據，方法較具有彈性。

參、心得

一、 本人因係第一次參加國外之大型國際研討會,對於 AOAC 主辦單位之作爲印象深刻：

(一)上一屆年會結束即宣布下一屆年會舉辦地點、時間、邀稿時間及議題，有意願參加人士可及早作準備。

(二)年會相關訊息，如報名、投稿、交通資訊、會議議程、邀稿內容、參展廠商資訊等，均很明確，可於網頁上蒐尋。

(三)確定報名及投稿程序後，相關事項之傳達透過電子郵件快速回覆，並提供查詢帳號及電話。

(四)會議前參展廠商透過主辦單位，以電子郵件邀請參加其相關展示訊息。

(五)會議結束後針對年會各項服務內容,以電子郵件邀請填寫網路意見調查表。並將調查結果回應，作爲以後舉辦活動之參考。

(六)開放權限給參與者，可上網取得相關演講議題之簡報檔。

二、 經由年會議程可瞭解今年與會者其研究重點爲：環境荷爾蒙如雙酚 A、多環芳香族碳氫化合物之分析、農藥多重、動物用藥、重金屬污染物、砷物種分析、海洋毒素、黴菌毒素、未知物分析等，本局歷年來研究重點均已涵括，與國際接軌良好。

三、 經由參加此次研討會訓練課程瞭解 AOAC 爲國際性專業分析協會,創會超過百年，其制定方法之嚴謹，猶面臨方法產出不敷使用之困境，故 2011 年起制定完整方法評估之標準作業及邀請專家參與評估審查，以徵求方法之方式使方法產出增加、快速、彈性，更具可信度。

四、 已將參加「國際公定分析化學家協會」第 125 屆年會心得於 11 月 11 日與同仁分享(附件五)。

肆、建議事項

一、 對照 AOAC 檢驗方法之開發猶須徵求方法，由國際間專家成立 ERP panel 進行方法審查，目前本局爲提供食品衛生檢驗方法之相關機關，更是受限於人力資源，以協助檢驗開發之助理而言，係不定期人力，因工作條件欠缺穩定性，助理流動性增加，不斷重新訓練人力勢必影響計畫之執行及檢驗品質。建議人力資源力求穩定，平時亟須與產、官、學界建立互動，瞭解檢驗方法之需求現況，評估方法需求之急迫性，尋求研擬檢驗方法之夥伴，經由實驗室比對縮短檢驗方法研擬時程。

二、 AOAC 方法嚴謹，對檢驗技術提昇極具參考價值，並提供 online method 及 online journal 查詢下載，建議局裡可申辦團體會員，以利資源之取得。此次研討會對岸華人參與者人數眾多，相較之下臺灣出席人數蓼蓼可數，因 AOAC 爲與檢驗技術相關之國際性研討會，臺灣亦爲分會成員，宜增加出席人數提昇能見度。

三、 會議中主題演講係食品安全之議題，特別是未知物的分析，將面臨挑戰。必須強化高階之儀器設備(高解析度之質譜儀)添置及人員檢驗技術提昇。目前同仁開會、文書等行政作業之時間付出太多，壓縮檢驗本業之時間、精神投入。鼓勵同仁多參加相關研討會、教育訓練課程，累積經驗技術。不定期邀請儀器廠商作儀器性能介紹、應用及發展現況或展望等。

附件一

2011 Annual Meeting Schedule At A Glance

Saturday, September 17, 2011

7:30 am – 12:00 pm	Rhythms 1	Editorial Board Meeting
9:00 am – 5:00 pm	Napoleon Foyer	Registration Open
5:00 pm – 6:00 pm	Evergreen	Journal Section Editors Meeting

Sunday, September 18, 2011

7:30 am – 7:00 pm	Napoleon Foyer	Registration Open
8:00 am – 9:00 am	Rhythms 1	Finance Committee Meeting
9:00 am – 11:00 am	Rhythms 1	AOAC INTERNATIONAL Board of Directors Meeting
12:00 pm – 4:00 pm	Nottoway	Methods Committee on Antimicrobial Efficacy Testing Meeting, Part 1
12:00 pm – 4:00 pm	Rhythms 3	Dietary Supplements Task Force and Community Meeting
1:00 pm – 4:00 pm	Oak Alley	Community Leadership Training
2:00 pm – 4:00 pm	Napoleon A	Standard Methods Performance Requirements Education
4:00 pm – 6:00 pm	Waterbury	Methods Committee on Microbiology Meeting
6:00 pm – 8:00 pm	Napoleon	Exhibit Hall Grand Opening Reception
8:00 pm – 10:00 pm	Rhythms	President's Welcome Reception

Monday, September 19, 2011

7:00 am – 8:00 am	Bayside B	TDRM Executive Committee Meeting
7:30 am – 8:00 am	Armstrong Foyer	Continental Breakfast
7:30 am – 5:00 pm	Napoleon Foyer	Registration Open
8:00 am – 10:30 am	Armstrong	Keynote Address and Awards Ceremony
10:00 am – 5:00 pm	Napoleon	Exhibit Hall Open
10:00 am – 5:00 pm	Napoleon	POSTER PRESENTATIONS: Analysis of Foodborne Contaminants and Residues, Analysis of Non-Foodborne Contaminants and Residues, Microbiological Methods, Pharmaceutical Analysis, Authenticity and Safety, and Plant Food, Pet Food and Animal Feed Nutritives, Additives, and Contaminants
10:30 am – 1:00 pm	Borgne	Latin America Section Business Meeting
10:45 am – 11:15 am	Rhythms 3	EXHIBITOR/PARTNER PRESENTATION: Roka Bioscience
11:30 am – 1:00 pm	Napoleon	Poster Author Presentations
11:45 am – 12:15 pm	Rhythms 3	EXHIBITOR/PARTNER PRESENTATION: Biotage AB
1:00 pm – 1:30 pm	Rhythms 1/2	H.W. Wiley Award Address
1:00 pm – 5:15 pm	Bayside C	AOAC Expert Review Panel
1:30 pm – 3:00 pm	Rhythms 1/2	Wiley Award SYMPOSIUM: Partners in Chasing and Tracing Chemicals in Food
1:30 pm – 3:00 pm	Napoleon A	ROUNDTABLE: Comprehensive Screening for Food Allergens in One Shot – Just a Nice Dream or a Feasible Project?
1:30 pm – 5:00 pm	Waterbury	ROUNDTABLE: Hot Areas of Interest in Botanicals for Dietary Supplement Industry
3:00 pm – 3:30 pm	Rhythms 3	EXHIBITOR/PARTNER PRESENTATION: Dionex – Part of Thermo Fisher Scientific
3:00 pm – 3:30 pm	Napoleon	Refreshment Break
3:30 pm – 5:00 pm	Rhythms 1/2	SYMPOSIUM: New Blood 2011 - Developing Methods for the Detection of Chemical Contaminants
3:30 pm – 5:00 pm	Napoleon A	ROUNDTABLE: What Do You Mean You Can't Clean It? Validation of Allergen Sanitation SOP in Food Establishments
4:30 pm – 5:30 pm	Bayside B	Laboratory Proficiency Testing Program Advisory Committee Meeting
4:30 pm – 6:30 pm	Southdown	Methods Committee on Antimicrobial Efficacy Testing Meeting, Part 2
5:00 pm – 6:00 pm	Rhythms 3	Media Reporting on Science: Implications for the Analytical Community, Supported by The Coca-Cola Company
5:00 pm – 6:30 pm	Gallery	New Member Welcoming Reception, Sponsored by MATHESON
5:00 pm – 7:00 pm	Oak Alley	Chemical Contaminants and Residues in Food Community Meeting
5:00 pm – 7:30 pm	Grand Chenier	Marine and Freshwater Toxins Community Meeting
5:15 pm – 8:15 pm	Maurepas	Food Allergen Community Meeting
6:00 pm – 7:00 pm	Bayside C	Taiwan Section Business Meeting
6:00 pm – 7:00 pm	Bayside A	Japan Section Business Meeting
6:00 pm – 8:00 pm	Gallier	Agricultural Materials Community Meeting
6:30 pm – 7:30 pm	Lagniappe	Reception for TDLM Members, Co-Sponsored by Microbiologics®, Inc.
7:00 pm – 8:00 pm	Rhythms 3	Joint Asian Sections Meeting

Tuesday, September 20, 2011

7:15 am – 8:15 am	Rhythms 3	EXHIBITOR/PARTNER PRESENTATION: Waters Corporation
7:15 am – 8:15 am	Edgewood	Nominating Committee Meeting
7:30 am – 5:00 pm	Napoleon Foyer	Registration Open
7:45 am – 8:15 am	Napoleon Foyer	Refreshment Break
8:00 am – 12:00 pm	Bayside A	AAFCO Meeting
8:00 am – 12:00 pm	Maurepas	AOAC Expert Review Panel
8:15 am – 9:45 am	Napoleon A	SYMPOSIUM: Inorganic Speciation Topics
8:15 am – 9:45 am	Waterbury	SYMPOSIUM: Reference Materials for Food Allergens... Heaven Must Wait?
8:15 am – 9:45 am	Rhythms 1/2	SYMPOSIUM: Alternative Methodology and Method Validation - Building an Internationally Harmonized Approach
9:00 am – 11:00 am	Southdown	Water/Waste Water Community Meeting
9:45 am – 10:15 am	Rhythms 3	EXHIBITOR/PARTNER PRESENTATION: Thermo Scientific
10:00 am – 10:30 am	Napoleon	Refreshment Break
10:00 am – 12:00 pm	Bayside C	Committee on Statistics Meeting
10:00 am – 5:00 pm	Napoleon	Exhibit Hall Open
10:00 am – 5:00 pm	Napoleon	POSTER PRESENTATIONS: Detection and Measurement of Natural Toxins, Emerging Issues in Food Safety and Security, and Food Nutrition and Food Allergens

10:15 am – 11:45 am	Napoleon A	SYMPOSIUM: Cosmetics at AOAC
10:15 am – 11:45 am	Rhythms 1/2	SYMPOSIUM: Chemoinformatic Aided Compound Identification in Mass Spectrometry - Applications, Challenges, and the Future Development
10:15 am – 11:45 am	Waterbury	TDRM/TDLM SYMPOSIUM: The Many Uses of Measurement Uncertainty
11:30 am – 1:00 pm	Napoleon	Poster Author Presentations
11:45 am – 1:15 pm	Bayside B	Contaminants Subgroup Meeting - Pesticides
12:00 pm – 1:00 pm	Rhythms 3	EXHIBITOR/PARTNER PRESENTATION: Agilent Technologies
12:30 pm – 2:30 pm	Southdown	Committee on Sections and Membership Meeting
1:00 pm – 3:00 pm	Edgewood	Methods Committee on Pesticides and Disinfectant Formulations Meeting
1:00 pm – 5:00 pm	Maurepas	AOAC Expert Review Panel
1:30 pm – 2:00 pm	Rhythms 3	EXHIBITOR/PARTNER PRESENTATION: Microbiologics®, Inc.
1:30 pm – 3:00 pm	Bayside B	Contaminants Subgroup Meeting - Unknowns
2:30 pm – 3:00 pm	Rhythms 3	EXHIBITOR/PARTNER PRESENTATION: Pickering Laboratories
2:30 pm – 3:00 pm	Napoleon	Refreshment Break
2:30 pm – 4:30 pm	Cornet	Veterinary Drug Residues Expert Review Panel
3:00 pm–4:30 pm	Napoleon A	SYMPOSIUM: Modern Analytical Techniques Used in Evaluating the Concentration of Trace Metals in Foods and Dietary Supplements
3:00 pm – 4:30 pm	Waterbury	TDRM/TDLM WORKSHOP: Reference Materials and Laboratory Accreditation
3:00 pm–4:30 pm	Rhythms 1/2	SYMPOSIUM: Experts Take on Detection of Non-O157:H7 STEC
4:30 pm – 5:00 pm	Rhythms 3	EXHIBITOR/PARTNER PRESENTATION: LECO Corporation
4:30 pm – 6:00 pm	Bayside B	Contaminants Subgroup Meeting - Veterinary Drugs
4:30 pm – 7:30 pm	Oak Alley	Mycotoxin Community Meeting
5:00 pm – 6:00 pm	Bayside C	TDRM Members Meeting
5:00 pm – 7:00 pm	Bayside A	Committee on Safety Meeting
5:30 pm – 6:00 pm	Rhythms 3	EXHIBITOR/PARTNER PRESENTATION: Advanced Chemistry Development, Inc. (ACD/Labs)
6:00 pm – 7:00 pm	Lagniappe	TDRM Members Reception, Co-Sponsored by Silliker
6:00 pm – 7:30 pm	Ellendale	Europe Section Executive Committee Meeting
6:15 pm – 7:45 pm	Bayside B	Contaminants Subgroup Meeting - Metals
7:00 pm – 8:00 pm	Southdown	China Section Business Meeting

Wednesday, September 21, 2011

7:15 am – 8:15 am	Rhythms 3	EXHIBITOR/PARTNER PRESENTATION: Phenomenex
7:30 am – 5:00 pm	Napoleon Foyer	Registration Open
7:45 am – 8:15 am	Napoleon Foyer	Refreshment Break
8:15 am – 9:45 am	Rhythms 1/2	Hot Topic SYMPOSIUM: Antioxidant Testing - "Fit for Purpose" Goes Beyond Statistics
8:15 am – 9:45 am	Waterbury	SYMPOSIUM: Method Validation and Method Performance Criteria of Chemical Analysis
8:15 am – 9:45 am	Napoleon A	SYMPOSIUM: Methods Harmonization - CURRENT Global Validation Schemes
9:45 am – 10:15 am	Rhythms 3	EXHIBITOR/PARTNER PRESENTATION: Covance Laboratories
9:45 am – 10:15 am	Napoleon	Refreshment Break
10:00 am – 12:00 pm	Nottoway	AOAC Research Institute Board of Directors Meeting
10:00 am – 12:00 pm	Gallery	Collaborative Study for Pesticide Multiresidues in Tea – Progress Report
10:00 am – 5:00 pm	Napoleon	POSTER PRESENTATIONS: General Methods, Quality Assurance and Accreditation, Botanicals and Dietary Supplements, and *Performance Tested Methods*℠
10:15 am – 11:45 am	Rhythms 1/2	SYMPOSIUM: Unique Challenges of Dietary Fiber Assays
10:15 am – 11:45 am	Waterbury	SYMPOSIUM: Pyrrolizidine Alkaloids, Undesirable Plant Toxins in Food and Feed
10:15 am – 11:45 am	Napoleon A	SYMPOSIUM: Microbial Method Criteria Used in the Global Meat Industry
11:30 am – 1:00 pm	Napoleon	Poster Author Presentations
11:45 am – 1:00 pm	Borgne	Technical Programming Council Meeting
12:00 pm – 1:00 pm	Rhythms 3	EXHIBITOR/PARTNER PRESENTATION: AB SCIEX
1:00 pm – 2:00 pm	Bayside B	TDLM Executive Committee Meeting
1:00 pm – 2:30 pm	Rhythms 1/2	SYMPOSIUM: Refining, Validating and Implementing LC and LC-MS Methods for Marine Toxins
1:00 pm – 2:30 pm	Napoleon A	SYMPOSIUM: New Methods - New Surrogates for Efficacy Testing of Antimicrobials
1:00 pm – 2:30 pm	Waterbury	SYMPOSIUM: Recent Advances in Veterinary Drug Research and Analysis
2:30 pm – 3:00 pm	Napoleon	Refreshment Break
2:30 pm – 3:00 pm	Rhythms 3	EXHIBITOR/PARTNER PRESENTATION: bioMérieux
2:30 pm – 3:30 pm	Jefferson Suite #4904	Meet Your Board of Directors
2:30 pm – 4:30 pm	Nottoway	AOAC Research Institute Advisory Council Meeting
3:00 pm – 4:30 pm	Waterbury	SYMPOSIUM: Best Practices for the Determination of Small Molecule Contaminants in Foods
3:00 pm – 4:30 pm	Napoleon A	SYMPOSIUM: New Trends in Natural Toxins for Targeted/Non-Targeted Analysis by Using Mass Spectrometers
3:00 pm – 4:30 pm	Rhythms 1/2	SYMPOSIUM: Chemical Stability and Metabolism of Shellfish Toxins
4:30 pm – 6:00 pm	Maurepas	AOAC INTERNATIONAL Business Meeting
7:00 pm – 10:00 pm	Armstrong	Annual Meeting Closing Reception

Thursday, September 22, 2011

8:30 am – 1:00 pm	Bayside A	Grocery Manufacturers Association Meeting
9:00 am – 4:00 pm	Bayside C	Official Methods Board Meeting
1:00 pm – 5:00 pm	Southdown	Juice and Juice Products Community Meeting

附件二

Determination of Melamine in Foods and Leaching Solutions of Melamine Tableware

Mei-Hua Chang, Wei-Chih Cheng, Wei-Liang Yan, Ya-Min Kao and Daniel Yang-Chih Shih

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ABSTRACT

In this study, methods were developed for the quantitation and confirmation of melamine in foods and leaching solutions of melamine tableware. Fish muscle and milk powder samples fortified with the melamine isotope internal standards, extracted with acetonitrile/water (6：4, v/v) and leaching solutions from tableware fortified with isotope internal standards were cleaned up by MCX solid phase extraction cartridge, eluted with 5% ammonia water in acetonitrile and analyzed by liquid chromatograph with tandem mass spectrometry (LC/MS/MS). The chromatographic separation was accomplished by elution of acetonitrile/20 mM ammonium acetate (95：5, v/v) on a BEH HILIC column. Data acquisition under MS/MS were achieved by applying multiple reaction monitoring (MRM) of two mass transitions. Melamine spiking levels were 0.05~0.2 μg/g in foods and 0.1~0.5 μg/mL in leaching solutions. Average recoveries were 101.1~106.5% in foods and 97.1~108.4% in leaching solutions, and the coefficients of variation were less than 4 %. The dectection limits were below 0.025 ppm. All results show the satisfactory recoveries, repeatbility and sensitivity.

Determination of Melamine in Foods and Leaching Solutions of Melamine Tableware

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Introduction

Melamine is an industrial chemical. Ingestion of melamine may lead to reproductive damage, bladder or kidney stones, which can lead to bladder cancer. Melamine combined with formaldehyde in the production of melamine-formaldehyde resin is approved for use as food contact articles. They are used extensively by children and eating-out persons owing to their characters of bright colors, cheapness, reusability and durability. However, melamine residual monomer may migrate into the foodstuffs if the polymerization is not complete.

Melamine-tainted milk products incident happened in Taiwan in 2008. This event caused Taiwan health authorities to concern about the amount of melamine in foods and leaching solutions of melamine tableware. This study developed melamine analysis method using isotope internal standard and liquid chromatograph/tandem mass spectrometry. Participation in the proficiency test conducted by European Commission Joint Research Centre Institute for Reference Materials and Measurements (JRC-IRMM) resulted in a satisfactory result in 2009.

Mateials and Methods

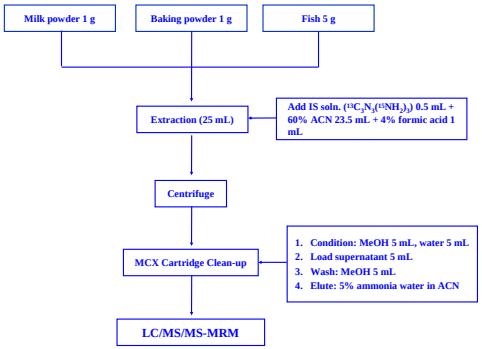

Figure 1. Analysis process for food sample

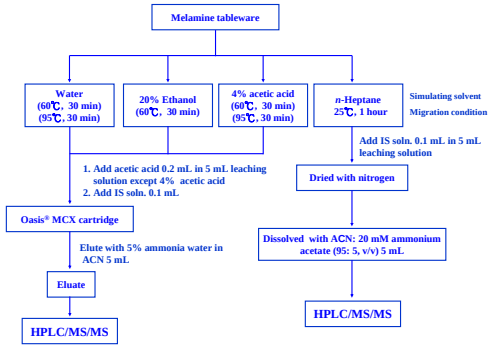

Figure 2. Analysis process for melamine tableware samples

UPLC condition

Liquid Chromatograph: Waters Acquity™ UPLC system。
Column: ACQITY UPLC BEH HILIC，100 × 2.1 mm i.d. ， 1.7 μm。
Mobile phase: Acetonitrile: 20 mM ammonium acetate (95 : 5, v/v)。
Flow rate: 0.4 mL/min。
Injection volume: 5 μL。

MS/MS condition

Mass spectrometer: Waters Micromass® Quattro Premier XE System。
Ionization mode: ESI⁺。
Capillary voltage: 3.2 KV。
Desolvation gas: Nitrogen 900 L/hr。
Desolvation temp: 400℃。
Source temp: 130℃。
Cone gas: Nitrogen 50 L/hr。
Acquisition: Multiple Reaction Monitoring (MRM)。
Collision Gas: Argon, 3.5×10^{-3} mBar。

Table 1. Transitions and instrument parameters for melamine and IS

Compound	Precursor ion (*m/z*)	Product ion (*m/z*)	Cone Voltage (V)	Collision energy (eV)
Melamine	127	85	40	17
	127	68	40	22
$^{13}C_3N_3(^{15}NH_2)_3$	133	89	40	17

Results

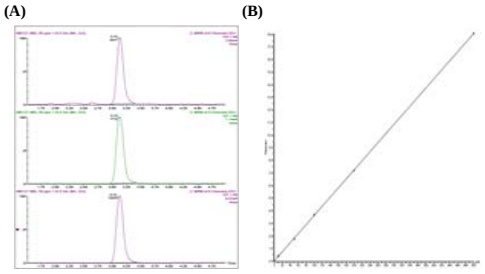

Figure 3. (A) LC/MS/MS chromatograms of melamine, (B) standard curve of melamine.

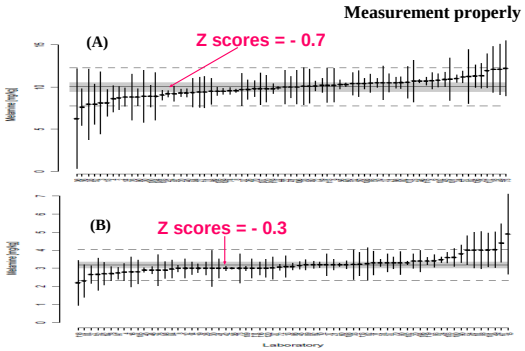

Figure 4. Measurement uncertainty (k=2) for (A) milk powder, (B) baking mix of Pre-TFDA lab (lab No. 42).

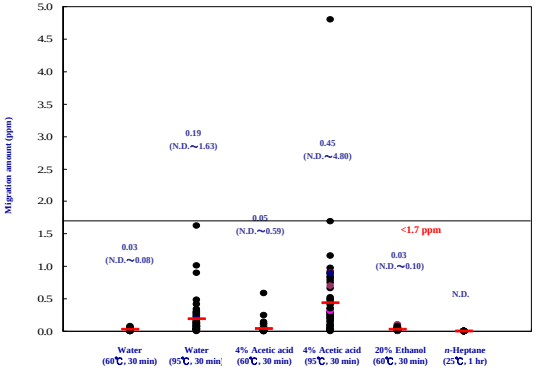

Figure 5. Migration of melamine from melamine tableware.

Table 2. Recoveries of melamine spiked into food

Sample blank	MEL added (μg/kg)	MEL measured (μg/kg)	Recovery (%)	CV (%)
Infant formula (ND)	200	211, 209, 214, 201, 204, 199	103	3
Yellow fish (ND)	50	52, 56, 53, 52	107	4
	200	208, 200, 200, 201	101	2

MEL: melamine.
ND＜10 μg/kg.

Table 3. Recoveries of melamine spiked into leaching solution of melamine tableware

Simulant solution	Recovery (%)[a]		
	0.1 ppm[b]	0.25 ppm	0.5 ppm
Water	102.2 (0.5)[c]	99.5 (0.7)	100.2 (0.5)
4% Acetic acid	108.4 (2.1)	100.2 (0.4)	99.9 (0.4)
20% Ethanol	102.5 (2.0)	100.4 (0.2)	99.5 (0.9)
n-Heptane	97.1 (2.6)	99.2 (0.7)	98.4 (2.1)

[a]Average of triplicate.
[b]Spiked level.
[c]Number in parentheses represents coefficient of variation (%).

Conclusion

Melamine spiking levels were 0.05~0.2 μg/g in foods and 0.1~0.5 μg/mL in leaching solutions. Average recoveries were 101.1~106.5% in foods and 97.1~108.4% in leaching solutions, and the coefficients of variation were less than 4%. The detection limits were below 0.025 ppm.

The improved LC/MS/MS method had been validated for melamine determination in foods and leaching solutions of melamine tableware.

In a survey of 52 melamine tableware samples the levels of melamine migrated from 51 samples in water (95℃, 30 mins), 4% acetic acid solution (95℃, 30 mins) and 20% ethanol solution (60℃, 30 mins) excluding one sample in which melamine was undetected in all six kinds of conditions. There was melamine migrating from 3 samples at the levels of 1 ~ 5 ppm. Melamine in *n*-heptane solution was not detected and this may be related to the undissolvable property of melamine in *n*-heptane.

Viruses in Water Samples from Foodborne Outbreaks in Taiwan (2010)

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ABSTRACT

Enteric viruses cannot multiply in the environment, but they may survive longer in water than most intestinal bacteria and are more infectious than most other microorganisms. In many respects, PCR is more effective than conventional cell culture and has proven to be a rapid, sensitive, specific and inexpensive method for detecting viruses. We developed methods to concentrate and detect viruses in water. Water sample 100 mL was concentrated by Amicon ultra-15 centrifugal filter units. For 1000 mL water, it was filtered through negatively charged membrane first, then the eluate was further concentration by using Amicon ultra-15 centrifugal filter units. Viral RNA was detected by reverse transcription PCR (RT-PCR) after RNA extraction. While HAV (strain HM175) were inoculated into 15 and 1000 mL distilled water, the detection limits were 50 and 100 genome equivalents, respectively. Thirty-two water samples, from foodborne outbreaks in Taiwan throughout 2010, were examined. The results showed, among norovirus GI, norovirus GII, HAV and astrovirus, the detected ratios were 3.1%, 9.4%, 12.5%, and 25%, and sapovirus, rotavirus (A~C), and HEV were non-detected at all.

INTRODUCTION

Enteric viruses and enterically transmitted hepatitis viruses have been associated with many outbreaks of nonbacterial gastroenteritis or hepatitis in different countries every year. These viruses are transmitted by the human fecal-oral route either via contaminated food and water or person to person spread through body contact. Enteric viruses are high stable in the environment, maintaining their infectivity even after exposure to treatment processes. Viral contamination of wastewater, recreational water, drinking water, irrigation water, ground or subsurface water has been frequently reported. Considering the low infectious dose of these viruses, only a small amount, present in the contaminated water, is usually sufficient to infect a human host. Thus, it is important to develop sensitive and efficient methods to detect viruses in water. Several concentration methods for viruses, in water samples, have been described. Among them, the filtration technique seems promising, since it enables the filtration of a large amount of water, while eliminating simultaneously potential inhibitors present in the sample. Food-borne viruses are the second most important cause of food-borne outbreaks in the European Union (EU) after Salmonella. In 2009, they were responsible for 19% of all outbreaks in the EU causing over 1000 outbreaks and affecting more than 8700 citizens. The total number of outbreaks caused by viruses has been increasing since 2007. In the United States, approximately 21 million illnesses attributable to norovirus are estimated to occur annually. According to data from the Real-time Outbreak and Disease Surveillance System operated by the Taiwan Centers for Disease Control (Taiwan CDC), there were around 50% reported diarrhea clusters tested positive for gastroenteritis virus infection, and caused by a variety of viruses, among them rotavirus and norovirus were the two most common agents. We had established a rapid method and applied to detect viruses in water samples from foodborne outbreaks in Taiwan.

MATERIALS AND METHODS

Viral strain. HAV RNA working reagent containing approximately 2000 genome equivalents of HAV strain HM175 (purchased from NIBSC) was used to spike into 15 and 1000 mL distilled water.

Concentration methods. For 100 mL samples, inoculated water was concentrated using Amicon ultra-15 centrifugal filter units. For 1000 mL samples, inoculated water was filtered through negatively charged membrane and the eluate was further concentrated using Amicon ultra-15 centrifugal filter units. The procedures were shown in Figure 1.

RT-PCR assay. One-step RT-PCR was performed in a reaction mixture (25 μL) contained 5 μL of RNA sample, 1.5 μL of each primer (10 μM), 5 μL of QIAGEN OneStep RT-PCR Buffer, 1 μL of dNTP Mix, 1 μL of Enzyme Mix and 10 μL of RNase-free water. The RT-PCR program was as following: reverse-transcription at 50℃ for 30 min, followed by 40 cycles of 95℃ for 30 s, 50℃ for 30 s, 72℃ for 1 min and final extension at 72℃ for 10 min. The primers used in this study was shown in Table 1.

Electrophoresis and sequencing. PCR products were analyzed on 2% agarose gel and electrophoresis was carried out. The PCR amplicons were directly sequenced by ABI 3730 XL DNA Analyzer. The resulting sequences were compared with other nucleotide sequences in GenBank.

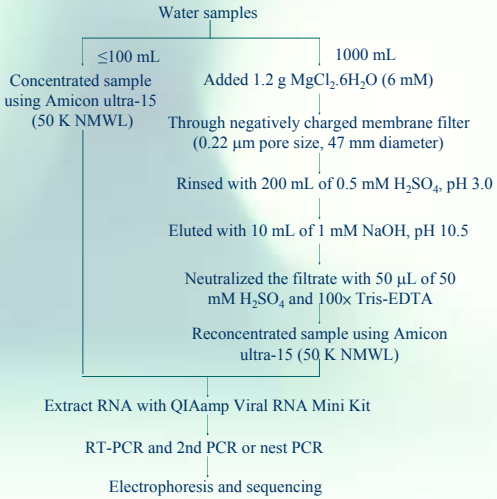

FIGURE 1. Steps to concentrate and detect viruses from water samples.

TABLE 1. Primer pairs used in this study

Target virus	Primer set	Sequence 5' to 3'	Product size (bp)
Norovirus GI	COG1F/ G1SKR	5'-CGYTGGATGCGNTTYCATGA-3' 5'-CCAACCCARCCATTRTACA-3'	383
Norovirus GII	QNIF2D/ G2SKR	5'-ATGTTCAGRTGGATGAGRTTCTCWGA-3' 5'-CCRCCNGCATRHCCRTTRTACAT-3'	391
Astrovirus	MON340/348	5'-CGTCATTATTTGTTGTCATACT-3' 5'-ACATGTGCTGCTGTTACTATG-3'	289
Enterovirus	EV05/EV06	5'-CACGGACACCCAAAGTA-3' 5'-CAAGCACTTCTGTTTCCCCGG-3'	400
HAV	VP1-4/5	5'-CGTTGCTTCCCATGTCAGAG-3' 5'-GACCTTCCCATAAACTTGTAG -3'	369
HEV	HEV-F/R	5'-CCTTGGGCCTAGAGTGTGCT-3' 5'-ACCGGCGAAGCGCACGACA-3'	406
Sapovirus	SLV5317/ SLV5749	5'-CTCGCCACCTACRAWGCBTGGTT-3' 5'-CGGRCYTCA AAVSTACCBCCCCA-3'	434
Rotovirus A	Beg9/VP7-1	5'-GGCTTTAAAAGAGAGAATTTCCGTCTGG-3' 5'-ACTGATCCTGTTGGCCATCCTTT-3'	395
Rotovirus B	ADG9-1F/1R	5'-GGCAATAAAATGGCTTCATTGC-3' 5'-GGGTTTTTACAGCTTCGGCT-3'	814
Rotovirus C	G8NS1/G8NA2	5'-ATTATGCTCAGACTATCGCCAC-3' 5'-GTTTCTGTACTAGCTGGTGAA-3'	351

Detection limit. 50 and 100 genome equivalents of HAV were inoculated into 15 mL and 1000 mL distilled water and obtained final concentrations of 3.3 genome equivalents/mL for 15 mL samples and 10^{-1} genome equivalents/mL for 1000 mL. The preparation was then handled by methods described above.

Determination of occurrence of viruses in water samples from foodborne outbreaks. The occurrence of nororivus GI, norovirus GII, astrovirus, enterovirus, HAV and HEV in 32 suspicious water samples of foodborne outbreaks was determined by using the methods we developed.

RESULTS AND DISCUSSION

Concentration methods. When using negatively charged membrane to concentrate virus, addition of cation to a freshwater sample was necessary in the virus adsorption to a membrane. The concentration of the cation also played an important role. The optimized concentration of cation in our study was about 6 mM. Higher and lower concentration both decreased absorption.

RT-PCR assay. The lowest detection limit of the RT-PCR assay was approximately 4 genome equivalents/reaction, as shown in Figure 2. The size of PCR amplicons was 396 bp.

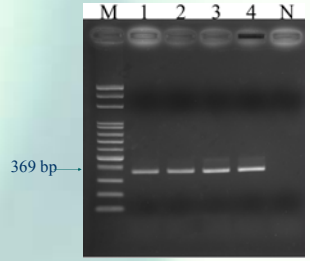

FIGURE 2. Detection limit of RT-PCR. Lane M: 100 bp marker; Lane 1-4: 4,8,12,16 genome equivalents of HAV/reaction; Lane N: no template control, NTC.

Detection limit. Since the levels of human enteric virus in water are normally low, detection methods with a high sensitivity are needed. The detection limits of the developed methods were 50 genome for 15 mL samples and 100 genome for 1000 mL samples, (Figure 3 and 4).

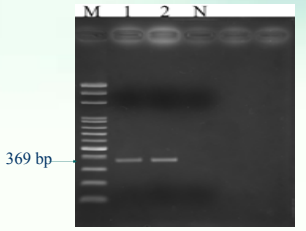

FIGURE 3. Detection limit for 15 mL samples. Lane M: 100 bp marker; lane 1-2: 50, 100 genome equivalents of HAV/15mL; lane N: NTC.

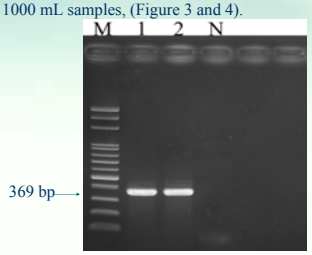

FIGURE 4. Detection limit for 1000 mL samples. Lane M: 100 bp marker; lane 1-2: 100 genome equivalents of HAV/L; lane N：NTC.

TABLE 2. Occurrence of virus in water samples

	Percentage of positive sample	
Year	2009	2010
Norovirus GI	6.8%	3.1%
Norovirus GII	25%	9.4%
Astrovirus	0%	25%
Enterovirus	34%	0%
HAV	2.3%	12.5%
HEV	0%	0%
Sapovirus	0%	0%
Rotavirus(A,B,C)	0%	0%
Sample number	44	32

Occurrence of viruses in water samples. By means of the developed methods, GI norovirus, GII norovirus, astrovirus, enterovirus and HAV were detected in real water samples. The results were showed in Table 2.

CONCLUSION

In summary, we developed sensitive RT-PCR methods for detecting virus in water samples. The developed methods were applied to the detection of viruses in 32 suspicious water samples from 2010 foodborne outbreaks in Taiwan and effectively detected GI norovirus, GII Norovirus, astrovirus and HAV in these samples. Hopefully, it could help to increase the detecting rate of etiology of foodborne outbreaks.

teamwork

Simultaneous Determination of Multiclass Veterinary Drug Residues in Porcine Liver by Liquid Chromatography-Electrospray Tandem Mass Spectrometry

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Abstract

The aim of this study was to develop a rapid method by using liquid chromatography tandem mass spectrometry (LC/MS/MS) combined with positive electrospray ionization to identify different classes of 15 antibiotics in porcine liver (macrolides, β-lactam antibiotics, lincosamides and miscellaneous antibiotics). Sample preparation was included liquid/liquid extraction followed by methanol, Na_2EDTA and acetonitrile. The extracts were cleaned up by hyflo supercel, and were evaporated to dryness under a stream of nitrogen. The chromatography was carried out on a Waters Acquity UPLC HSS T3 column, mobile phase component A was water with 0.005% formic acid, while component B was acetonitrile. The average recoveries were 50.2% to 115.8 %, and coefficients of variation were from 2.8% to 15.1 %. Estimated limits of quantification were 0.25- 10 ppb.

Introduction

Veterinary drugs are widely used for the treatment and prevention of disease in livestock . Main veterinary drugs used today include β-lactams, sulfonamides, tetracyclines aminoglycosides, chloramphenicol, macrolides and quinolones. Despite the positive effects of these drugs, inadequate use of antibiotics poses a potential health risk to consumers. Multiclass, multi residue methods are gaining importance for residue control in food products. Microbiological, immunological assays and liquid chromatography (LC) with ultraviolet (UV) or fluorimetric detecter are the traditional screening techniques, but they are often lengthy and not sufficiently specific for analytical purposes. Liquid chromatographic–tandem mass spectrometric (LC–MS/MS) affords a highly specific and rapid method for simultaneous determination of a number of residual veterinary drugs in foods.

In this report, a rapid and simple method for simultaneous determinaion of 15 antibiotics in porcine liver, from different classes of antibiotics namely, macrolides, β-lactam antibiotics, lincosamides and miscellaneous antibiotics, is described.

Materials and Methods

Materials

Porcine liver was purchased from supermarkets.

Sample preparation

Weight 1g of homogenized sample and mix with with 10 mL methanol and 0.5 mL 0.1M Na_2EDTA. The mixture was centrifuged at 3,200 *x g* for 10 min, and then the supernatant was decanted to the new centrifuge tube. The pellet was homogenize with 15 mL acetonitrile, and the homogenate was centrifuge for 10 min at 3,200 *x g*. The supernatant was combined with the first extraction portion, and the solid remnant was discarded. 2 g Hyflo Super-Cel was added to the extract and shake vigorously for 5 min The mixture was centrifuged at 3,200 *x g* for 10 min, and then the supernatant was decanted to the new centrifuge tube. The pellet was washed with 10 mL acetonitrile, centrifuge for 10 min at 3,200 *x g*. The extract was combined and dryness with a stream of N_2 at 35℃ water bath. The residue was dissolved with 1 mL 50% acetonitrile and was filtered through a 0.2 μm PVDF syringe filter for LC-MS/MS analysis.

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 0.005% formic acid solution (solvent A) and 100% 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 2.5 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 minium.

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→1.5	**100→100**	**0→0**
	1.5→4.0	**100→50**	**0→50**
	4.0→7.0	**50→20**	**50→80**
	7.0→9.0	**20→20**	**80→80**
	9.0→13.0	**20→5**	**80→95**
	13.0→14.0	**5→5**	**95→95**
	14.0→14.1	**5→100**	**95→0**
	14.1→20.0	**100→100**	**0→0**
Flow rate	**0.3 mL/min**		
Injection volume	**10 μL**		
Analysis time	**20 min**		

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

Compound	Abbre-viation	Retention time (min)	Parent ion (*m/z*)	Transition 1 (CE)	Transition 2 (CE)	Transition 3 (CE)	Cone voltage	Ion ratio (%)**
clarithromycin	**CLA**	4.7	748.7	115.9(44)	**158.0*(32)**	590.5(20)	28	26.2 (0.7)
erythromycin	**ERY**	4.4	734.6	116.0(46)	**158.1*(32)**	576.5(18)	26	38.4 (1.3)
natamycin	**NAT**	4.4	666.5	463.3(32)	485.3(14)	**503.3*(12)**	54,18,18	55.9 (4.1)
oleandomycin	**OLE**	4.3	688.6	116.0(42)	**158.0*(28)**	544.5(16)	24	46.4 (1.8)
tilmicosin	**TIL**	4.1	869.8	115.9(64)	132.0(50)	**174.1*(46)**	70	28.5 (3.2)
troleandomycin	**TRO**	5.0	814.7	**200.1*(26)**	158.0(46)	116.0(46)	34	15.3 (0.5)
virginiamycin M1	**VIR**	5.4	526.4	231.0(36)	337.1(22)	**355.2*(18)**	24	82.7 (2.0)
cefoperazone	**CEO**	4.3	646.4	**143.1*(40)**	526.2(10)	530.2(18)	18,18,28	63.9 (4.4)
cloxacillin	**CLO**	5.5	436.2	114.0(34)	160.0(12)	**277.1*(14)**	16	100.7 (4.7)
mecillinam	**MEC**	3.8	326.3	122.1(36)	139.1(30)	**167.1*(22)**	32	13.0 (0.3)
oxacillin	**OXA**	5.3	402.3	114.1(32)	160.0(12)	**243.1*(12)**	16	41.9 (4.0)
clindamycin	**CLI**	4.1	425.3	**126.1*(28)**	377.2(20)	389.3 (18)	30	5.9 (0.3)
lincomycin	**LIN**	3.5	407.3	**126.1*(30)**	172.1(22)	359.3(18)	32	7.9 (0.3)
morantel	**MOR**	4.0	221.1	**122.9*(34)**	111.0(26)	164.0(28)	42	97.7 (3.0)
orbifloxacin	**ORB**	3.9	396.3	226.1(42)	267.1(36)	**295.1*(24)**	32	16.7 (0.4)
roxithromycin (I.S.)	**ROX**	4.7	837.8	**158.1*(36)**	679.5(22)	522.4(26)	32	63.7 (3.6)

* Transitions with bold numbers were used for quantification.

** Relative standard deviation (RSD) is given in parentheses (n=18).

Result and Discussion

Extraction solvent comparison

Initial experiments were aimed at finding the best solvent in term of recovery of the analytes. Methanol , acetonitrile, and 0.1M Na_2EDTA, methanol, acetonitrile were selected for this study. A porcine liver samples were spiked with solution of standard mixture then extracted with different extraction solvent, and results are shown in Fig. 1. The results showed that, compared to methanol, acetonitrile afforded much higher analyte recovery to most of macrolides but the result of β-lactam antibiotics are opposite. Therefore, the suitable solvent for the subsequent experiments were performed by using methanol, 0.1M Na_2EDTA and acetonitrile.

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 average recoveries of macrolides were 50.2-104.8%, β-lactam antibiotics were 77.4-104.1%, lincosamides were 100.9-115.8%, orbifloxacin was 105.9% and morantel was 114.5%, the results are summarized in Table 3. LODs and LOQs were tested by analysing blank samples, which was fortified seven concentrations (0.25, 0.5, 1, 2.5, 5, 10 and 20 ng/g). The LOD values of macrolides were between 1-10 ng/g, β-lactam antibiotics were between 2.5-10 ng/g, licosamides were between 0.5-2.5 ng/g, morantel was 5 ng/g and orbifloxacin was 2.5 ng/g, the results are summarized in Table 4. Four concentrations of mixed standard solutions of the fifteen 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 three times for three days. The coefficients of variation of intra-day and inter-day assays were lower than 15.8% and 18.6%, respectively (Table 5).

Conclusions

A multiresidue method was developed for rapid and simultaneous determination of 15 antibiotics in porcine liver 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.

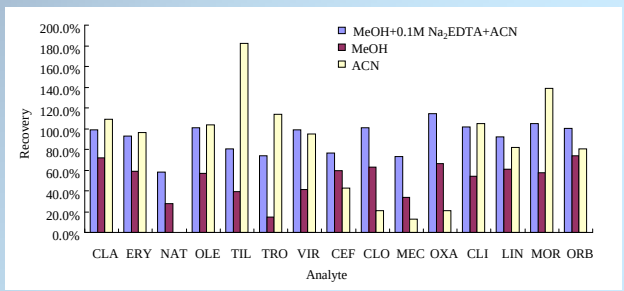


Fig 1. The recoveries of 15 veterinary drugs in porcine liver with different extraction buffers.

Table 3 The recoveries of 15 veterinary drugs in porcine liver sample

Drug / Spiked standard (ppb)	Recovery (%) n = 3				
	25	50	100	200	average
Macrolides					
clarithromycin	100.8	102.8	102.2	103.7	102.4
erythromycin	109.1	107.0	101.1	101.9	104.8
natamycin	53.7	51.0	45.3	50.6	50.2
oleandomycin	99.5	103.2	96.3	100.2	99.8
troleandomycin	62.7	65.4	60.4	59.6	62.0
tilmicosin	75.7	91.5	88.0	84.2	84.9
virginiamycin M1	74.3	77.2	72.0	73.2	74.2
β-lactam					
cefoperazone	75.6	88.2	74.2	71.7	77.4
cloxacillin	84.9	109.6	109.1	112.6	104.1
mecillinam	76.7	81.3	76.5	76.1	77.7
oxacillin	82.6	109.6	110.1	106.4	102.2
Licosamides					
clindamycin	116.6	121.9	113.2	111.5	115.8
lincomycin	106.4	101.6	97.4	98.3	100.9
Miscellaneous					
Orbifloxacin	111.5	104.8	100.4	106.9	105.9
morantel	117.9	118.8	110.4	110.7	114.5

Table 4 Limits of detection (LODs) and limits of quantitation (LOQs) of 15 veterinary drugs in porcine liver

Drug	LOD (μg kg^{-1})	LOQ (μg kg^{-1})
Macrolides		
clarithromycin	0.5	1
erythromycin	1	2.5
natamycin	5	10
oleandomycin	0.5	1
troleandomycin	1	2.5
tilmicosin	2.5	5
virginiamycin M1	5	10
β-lactam		
cefoperazone	5	10
cloxacillin	2.5	5
mecillinam	1	2.5
oxacillin	5	10
Licosamides		
clindamycin	1	2.5
lincomycin	0.25	0.5
Miscellaneous		
morantel	2.5	5
orbifloxacin	1	2.5

Table 5 Intra-day and inter-day coefficient of variation of 15 veterinary drugs in porcine liver at various spiked levels

Drug / Spiked standard (ppb)	Intra-day / Inter-day, C.V. (%) n = 3				
	25	50	100	200	average
Macrolides					
clarithromycin	5.8/17.0	9.3/11.2	8.8/11.6	5.8/10.9	7.4/12.7
erythromycin	3.7/10.8	7.9/9.3	6.8/7.2	7.7/8.4	6.6/8.9
natamycin	10.2/13.0	14.4/12.7	9.5/8.0	9.6/6.3	10.9/10.0
oleandomycin	15.8/11.2	11.9/10.6	12.0/10.4	13.1/7.5	13.2/9.9
troleandomycin	7.9/18.6	6.4/13.0	13.5/15.1	9.5/13.7	9.3/15.1
tilmicosin	7.6/12.1	2.3/4.8	0.5/4.0	0.9/3.6	2.8/6.1
virginiamycin M1	10.6/4.0	14.7/10.0	1.0/5.8	3.7/7.3	7.5/6.8
β-lactam					
cefoperazone	14.8/13.3	11.5/12.0	13.3/10.1	4.6/11.9	11.0/11.8
cloxacillin	7.4/9.4	10.6/5.8	7.6/11.8	8.0/12.8	8.4/10.0
mecillinam	11.7/9.2	8.9/9.4	9.6/7.8	9.9/5.4	10.0/8.0
oxacillin	6.2/14.3	12.2/5.2	12.5/8.7	9.8/13.4	10.2/10.4
Licosamides					
clindamycin	6.1/6.8	6.5/9.6	6.8/8.5	7.3/7.4	6.7/8.1
lincomycin	8.8/9.1	5.8/5.0	8.7/7.8	8.2/8.3	7.9/7.6
Miscellaneous					
orbifloxacin	5.5/10.3	7.3/9.4	9.9/7.8	8.4/5.4	7.8/8.2
morantel	6.8/7.4	6.4/6.9	6.8/5.1	7.9/4.2	7.0/5.9

附件三

AOAC SMPR 文件之標準格式及指引

一、方法名稱：必須含分析物、基質及分析技術。

二、核可單位：相關小組或專家審查小組名稱。

(一)目的用途：有關方法及使用條件之更多資訊。

(二)適用性：列出更多基質(當大於 1 種)、提供分析物之 IUPAC 命名及 CAS Number，明確說明基質性質如生的、熟的、錠劑或粉末等。

(三)分析技術：提供分析技術之詳細說明或符合 SMPR 引用之方法。

(四)定義：表列及定義措詞。

(五)方法性能要求：表列方法/分析物/基質性能參數及接受標準。

(六)系統適合性測試或分析品質管制：描述系統最少管制及 QC 措施。

(七)參考物質：確定適當參考物質，或缺乏適當參考物質而使用自力設計之參考物質。

(八)確效指引：因爲方法分類，所須性能參數如下：

1. 定量方法(主要成份)

單一實驗室確效：參考方法比較、應用範圍、偏差、精密度、回收率。

獨立實驗室：由議題專家決定。

實驗室比對：重複性。

2. 定量方法(微量或污染物)

單一實驗室確效：參考方法比較、應用範圍、偏差、精密度、回收率、檢出限量(LOD)、定量限量(LOQ)。

獨立實驗室：由議題專家決定。

實驗室比對：重複性。

3. 定性方法(主要成分)

單一實驗室確效：參考方法比較、選擇性、專一性、環境干擾、實驗室變異、偏差、於關鍵值檢出率。

獨立實驗室：由議題專家決定。

實驗室比對：於分析物濃度爲 0 下之檢出率(POD(0))，於濃度 C 下之檢出率(POD(C))，最低檢出濃度之檢出率。

4. 定性方法(微量或污染物)

單一實驗室確效：參考方法比較、選擇性、專一性、環境干擾、實驗室變異、偏差、最低檢出濃度之檢出率。

獨立實驗室：最低檢出濃度之檢出率。

實驗室比對：POD(0)，POD(C)，最低檢出濃度之檢出率。

5. 鑑別方法

單一實驗室確效：參考方法比較、選擇性、專一性、精密度、環境干擾、偏差。

獨立實驗室：偏差。

實驗室比對：POD(0)，POD(C)，最低檢出濃度之檢出率。

(九)測試所需最長時間。

確效試驗之草案(對所有 SMPR 是必須的)

簡介：提供方法類別適用之確效概要，及 SMPR 方法確效層級之一般資訊。

層級 1：方法開發者之確效協定：

層級 2：獨立實驗室之確效協定：

層級 3：實驗室比對之確效協定：

評估建議：

1. 準確度
2. 偏差(若有參考物質可用)
3. 環境干擾物質
4. 專一性
5. 選擇性
6. 偵測極限(Limit of Detection)
7. 定量極限(Limit of Quantitation)
8. POD
9. 重複性

Table 1. Expected precision (repeatability) as a function of analyte concentration

Analyte %	Analyte ratio	Unit	RSD%
100	1	100%	1.3
10	10^{-1}	10%	1.9

1	10^{-2}	1%	2.7
0.1	10^{-3}	0.1%	3.7
0.01	10^{-4}	100 ppm	5.3
0.001	10^{-5}	10 ppm	7.3
0.0001	10^{-6}	1 ppm	11
0.00001	10^{-7}	100 ppb	15
0.000001	10^{-8}	10 ppb	21
0.0000001	10^{-9}	1 ppb	30

10.回收率

Table 2. Expected recovery as a function of analyte concentration

Analyte %	Analyte ratio	Unit	Mean Recovery (%)
100	1	100%	98-102
10	10^{-1}	10%	98-102
1	10^{-2}	1%	97-103
0.1	10^{-3}	0.1%	95-105
0.01	10^{-4}	100 ppm	90-107
0.001	10^{-5}	10 ppm	80-110
0.0001	10^{-6}	1 ppm	80-110
0.00001	10^{-7}	100 ppb	80-110
0.000001	10^{-8}	10 ppb	60-115
0.0000001	10^{-9}	1 ppb	40-120

11.相對標準偏差

12.重複性(實驗室比對)

13.標準偏差

瞭解 POD 模式。

Table 3. Terminology

Traditional terminology	Concept	POD equivalent
False positive	The probability of the	POD(0) POD at

	method giving a (+) response when the sample is truly without analyte	conc = 0
Specificity	The probability of the method giving a (-) response when the sample is truly without analyte	1-POD(0)
False negative (at a given concentration)	The probability of a (-) response at a given concentration	1-POD(c)
Sensitivity (at a given concentration)	The probability of a (+) response at a given concentration	POD(c)
True negative	A sample that contains no analyte	C = 0
True positive	A sample that contains analyte at some positive concentration	C ＞ 0

由實驗室內部數據(intra-laboratory)定義及計算 HorRat 值。

名詞定義

RSD(r)或 RSD_r：實驗室內標準差。

RSD(R)或 RSD_R：實驗室間標準差。

Predicted relative standard deviation ＝ PRSD(R)或 $PRSD_R$。

Table 4. Predicted relative standard deviation of reproducibility ($PRSD_R$)

Concentration, C	Mass fraction, C	PRSD (R) (%)	PRSD (r) (%)
100%	1.0	2	1
1%	0.01	4	2
0.01%	0.0001	8	4
1 ppm	0.000001	16	8
10 ppb	0.00000001	32	16
1 ppb	0.000000001	45	22

Horrat value(R)= RSD_R/PRSD(R)

Horrat value(r)= RSD_r/PRSD(r)

Acceptable Horrat value：Among laboratory：0.5-2.0；Within laboratory：0.3-1.3。

P17 #⑩

AOAC INTERNATIONAL

ALTERNATIVE PATHWAY to OFFICIAL FIRST ACTION METHOD STATUS REQUIREMENTS

Expert Review Panels

-Must be supported by relevant stakeholders.

-Constituted solely for the ERP purpose, not for Standard Method Performance Requirements (SMPR) purposes or as an extension of an SMPR.

-Consist of a minimum of seven members representing balance of key stakeholders.

-ERP constituency must be approved by the Official Methods Board (OMB).

-Holds transparent public meetings only.

-Remains in force as long as method in First Action Status.

Official First Action Method Status decision

-Must be made by an ERP constituted or reinstated post 2011-03-28 for Official First Action Status Method Approval (OFASMA).

-Must be made by an ERP vetted for OFASMA purposes by OMB post 2011-03-28.

-Method adopted by ERP must perform adequately against the SMPR set forth by the stakeholders.

-Method must be adopted by unanimous decision of ERP on first ballot, If not unanimous, negative votes must delineate scientific reasons.

-Negative voter(s) can be overridden by 2/3 of voting ERP members after due consideration

-Method becomes Official First Action on date when ERP decision is made.

-Methods to be drafted into AOAC format by a knowledgeable AOAC staff member or designee in collaboration with the ERP and method author.

-Report of OFAMS decision complete with ERP report regarding decision including scientific background (references etc) to be published concurrently with method in traditional AOAC publication venues.

Method in First Action Status and Transitioning to Final Action Status

-Further data indicative of adequate method reproducibility (between laboratory) performance to be collected. Data may be collected via a collaborative study or by proficiency or other testing data of similar magnitude.

-Two years maximum transition time (additional year(s) if ERP determines a relevant collaborative study or proficiency or other data collection is in progress).

-Method removed from Official First Action and OMA if no evidence of method use available at the end of the transition time.

-Method removed from Official First Action and OMA if no data indicative of adequate method reproducibility is forthcoming as outlined above at the end of the transition time.

-ERP to recommend Method to Official Final Action Status to the OMB.

-OMB decision on First to Final Action Status

Board of Directors Adopted May 25, 2011
Revised June 27, 2011

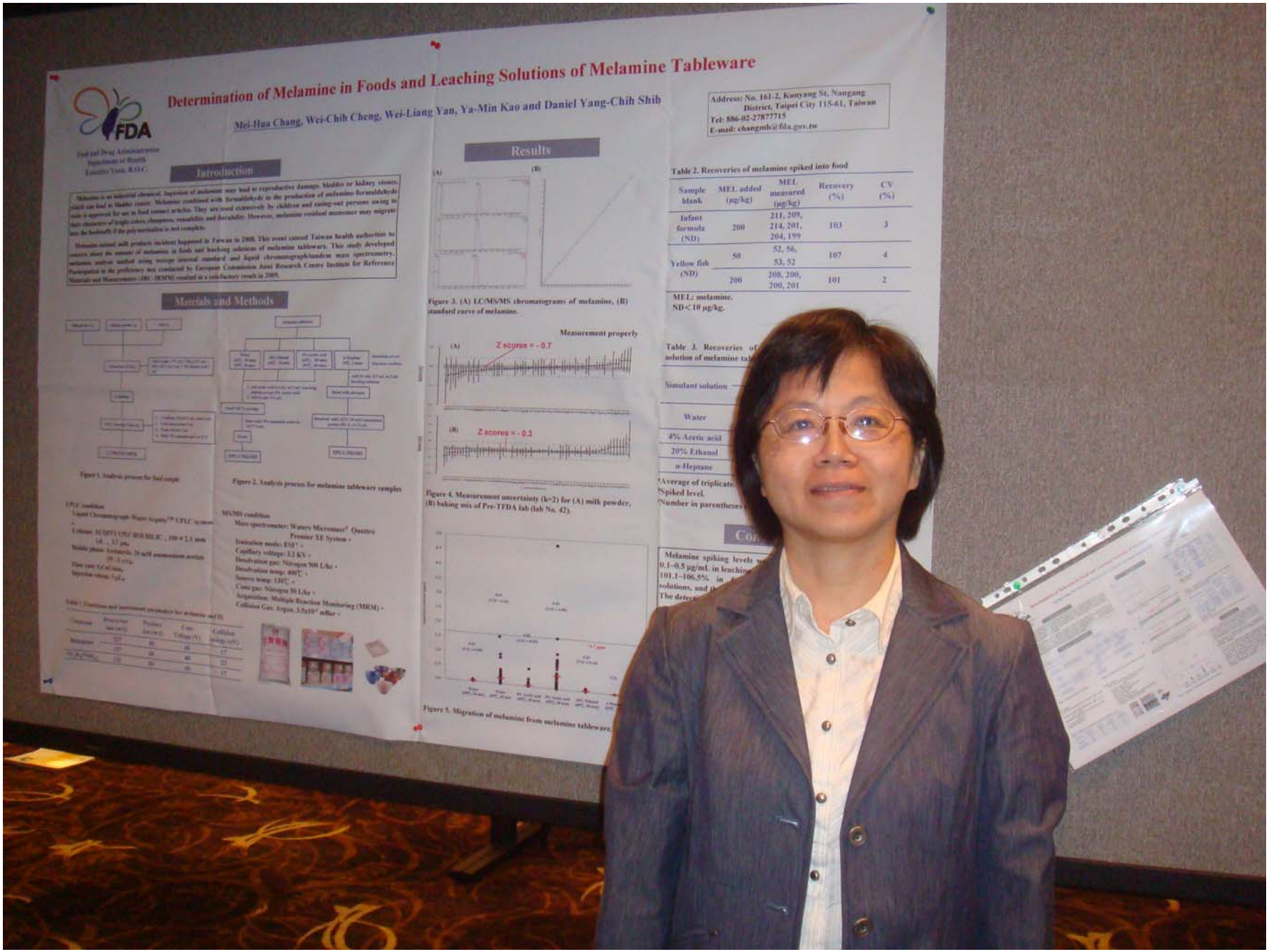
附件五

參加「國際公定分析化學家協會(AOAC International)研習國際新穎性食品檢驗技術 」心得分享

- 報告人：張美華
- 年會期間：100 年9 月18 日至100 年9月21日
- 報告日期：100 年11 月11 日

年會議程內容

- Scientific sessions
- Poster presentation
- Education sessions
- Exhibitor/partner presentation


Determination of Melamine in Foods and Leaching Solutions of Melamine Tableware
Mei-Hua Chang, Wei-Chih Cheng, Wei-Liang Yan, Ya-Min Kao and Daniel Yang-Chih Shih
Address: No. 161-2, Kunyang St, Nangang District, Taipei City 115-61, Taiwan
Tel: 886-02-27877715
E-mail: changmh@fda.gov.tw
Introduction
Materials and Methods
Results
Table 2. Recoveries of melamine spiked into food
Sample blank
MEL added (μg/kg)
MEL measured (μg/kg)
Recovery (%)
CV (%)
Infant formula (ND)
200
211, 209, 214, 201, 204, 199
103
3
Yellow fish (ND)
50
52, 56, 53, 52
107
4
200
208, 200, 200, 201
101
2
MEL: melamine.
ND < 10 μg/kg.
Measurement properly
Z scores = - 0.7
Z scores = - 0.3

國際研究趨勢重點-1

- Bisphenol A ：molecularly imprinted polymer, Oasis HLB cartridge baby food, bisphenol A in total diet, Drinking water. (5篇)
- Phthalate ：Combination of Quechers extraction method with GC-triple quard detection, Separation of 21 phthalates using both LC/MS/MS and GC/MS (Phenomenex). (2篇)
- Multiple veterinary residues： Quechers, Q-Exactive bench top Orbitrap, UPLC-Q-TOF,

國際研究趨勢重點-2

- Mutiple pesticide：Quechers, Program temperature vaporization, matrix-match calibration curve, Direct sampling analysis (DSA) TOF mass spectrometry
- Heavy metal：Arsenic speciation, ICP-TOF mass
- Unknown analysis：high resolution mass

Education session

- Standard methods peformance requirements education (SMPR)

Traditional path to official first action

AOAC Managed

Method & Validation protocol submitted to AOAC

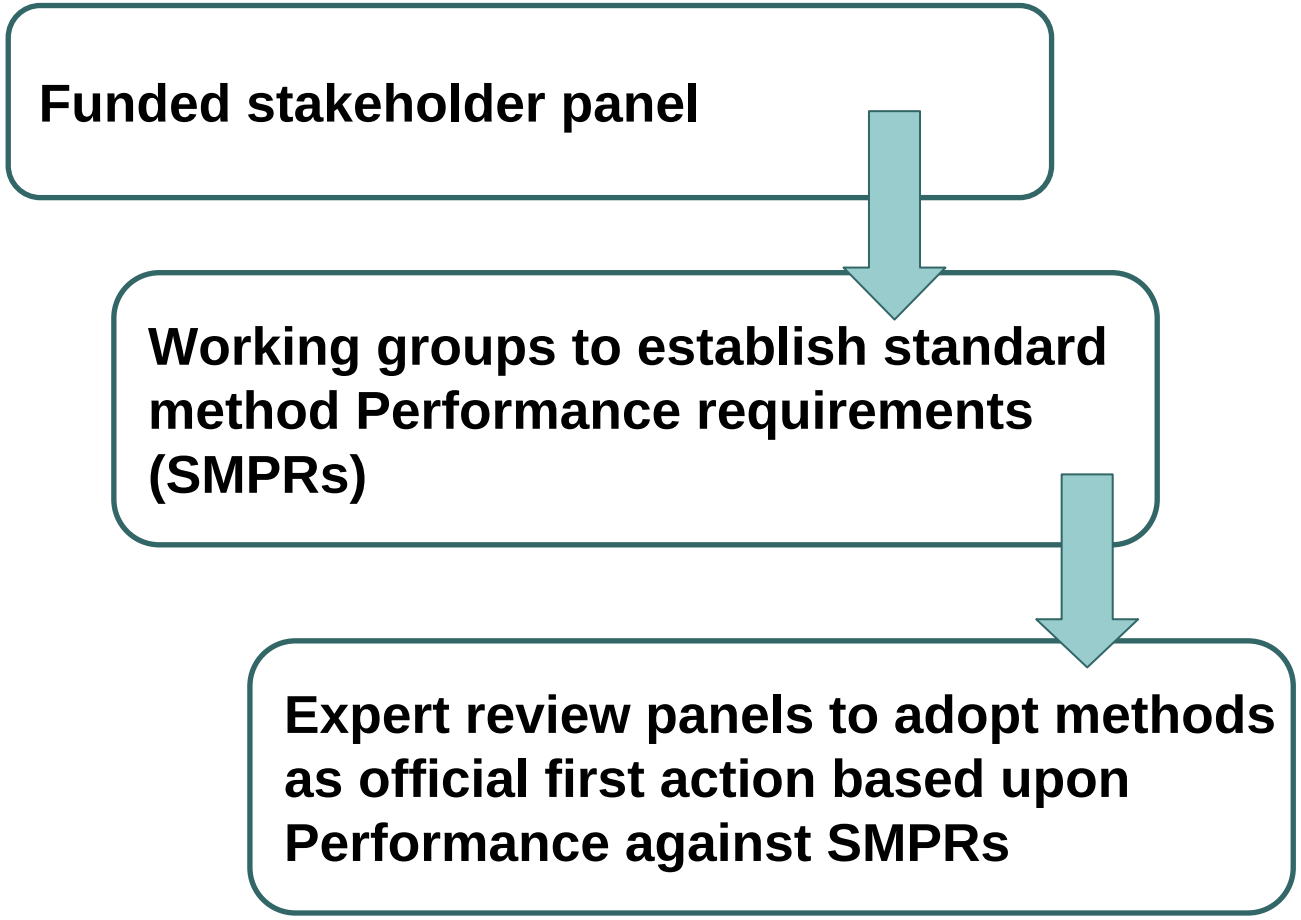
Study director coordinates and conducts collaborative study and submits manuscript

Method in use for 2 years and a recommendation is made to OMB regarding final action status after the 2 year period

Official methods board reviews recommendation and makes decisions on final action status

Alternative path to achieve an official method

Approved on March, 2011



Expected benefits

- More official methods of analysis generated
- Can provide solutions faster and take full advantage of collective expertise of AOAC members
- Methods can be put into regular use right away
- OMA can be more flexible
- More cofidence is given to final action methods

Standard method performance requirement

- **Standard format and guideance**
  - **Intended use**
  - **Applicability**
  - **Analytical technique**
  - **Definitions**
  - **Method performance requirement**
  - **System suitability tests and/or analytical quality control**
  - **Reference material**
  - **Validation guidance**
  - **Maximum time-to-determination**

Recommendations for evaluation

- Accuracy
- Bias
- Limit of detection (LOD)
- Limit of Quantiation (LOQ)
- Repeatability (precision)
- Recovery
- Relative standard deviation (RSD)
- Reprodubility (Collaborative study)

Expected precision and revovery as a function of analyte concentration

Unit	RSD%	Recovery (%)
100%	1.3	98-102
10%	1.9	98-102
1%	2.7	97-103
0.1%	3.7	95-105
100 ppm	5.3	90-107
10 ppm	7.3	80-110
1 ppm	11	80-110
100 ppb	15	80-110
10 ppb	21	60-115
1 ppb	30	40-120

Prdicted relative standard deviation of reproducibility ($PRSD_R$)

Concentration, C	Mass fraction, C	PRSD (R) (%)	PRSD (r) (%)
100%	1.0	2	1
1%	0.01	4	2
0.01%	0.0001	8	4
1 ppm	0.000001	16	8
10 ppb	0.00000001	32	16
1 ppb	0.000000001	45	22

PRSD (R) $= 2C^{-0.15}$ **C is expressed as a mass fraction**

Horrat values

Horrat **(R)** = **RSD (R) / PRSD (R)**

Horrat **(r)** = **RSD (r) / PRSD (r)**

Acceptable Horrat value

Among laboratory：0.5-2.0

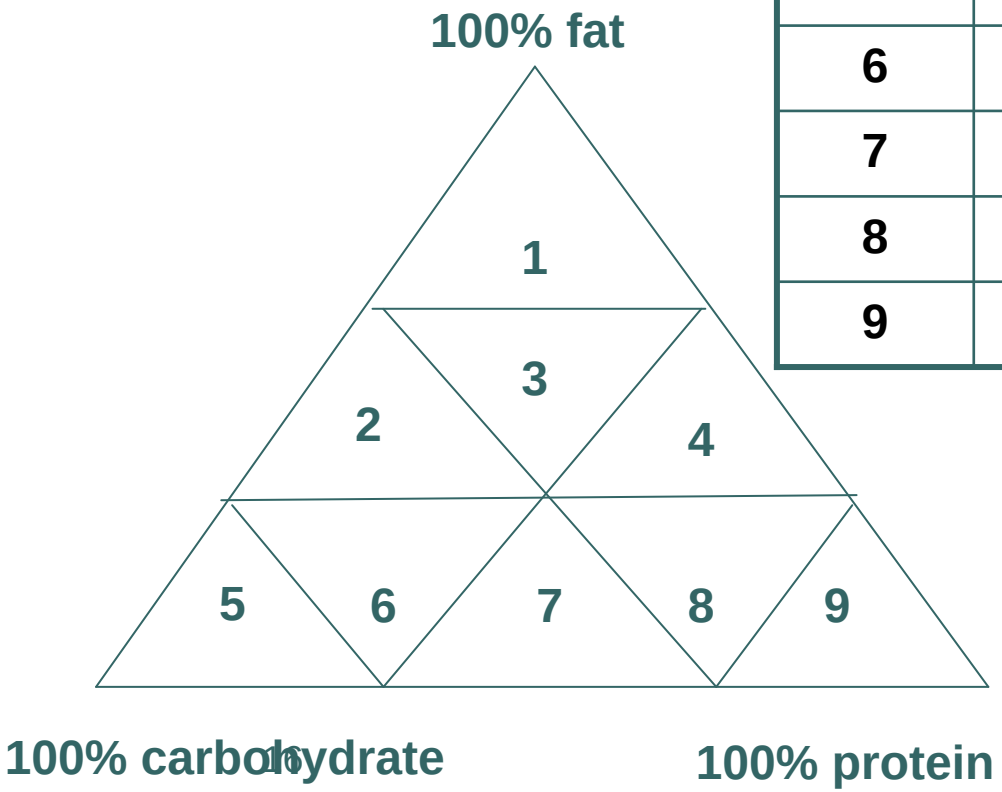
Within laboratory：0.3-1.3

Reference material

- CRM：provide with a certified value as well as the statistical data for the analyte, can be used to optimize method
- Not currently available
- Most CRMs are certified for a limited number of analytes

AOAC international's task force on methods for nutrition labelling

Sector	RM No.	Matrix
1	NIST 3274	Botanical oil
2	NIST 2384	Baking chocolate
3	NIST 2387	Peanut butter
4	LGC 7150	Proceseed meat
5	BCR-382	Wheat flour
6	NIST 1849	Nutritional formula
7	NIST 1566b	Oyster tissue
8	NIST 1946	Lake trout
9	NIST 1974a	Mussel tissue



Thanks for your attendence