出國報告（出國類別：開會）

參加歐洲農藥殘留研討會報告

服務機關：行政院衛生署食品藥物管理局
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派赴國家：奧地利
出國期間：101 年 6 月 23 日至 101 年 7 月 6 日
(6 月 29 至 7 月 5 日爲自費停留，7 月 6 日搭機返台)
報告日期：101 年 9 月 17 日

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摘要

第 9 屆歐洲農藥殘留研討會 (European Pesticide Residue Workshop，EPRW) 於 6 月 25 日至 6 月 28 日在奧地利 (Austria) 舉行。專題演講討論的主題包含：對於檢驗和研究所該抱持的態度、現今農藥檢驗所面臨的挑戰、新穎儀器的應用、各式研究開發、殘留容許量和方法確效等內容。另主辦單位有鑑於日益複雜的殘留定義 (Residue Definitions)，選了一天專門就殘留定義相關議題來進行演講和討論。參展壁報分 5 大類別：PV (廠商發表)、PA (分析方法之開發及應用)、PM (監測及取食評估)、PR (法規及風險評估) 和 PO (其他主題)，由內容可知分析方法的開發和應用仍爲主要焦點。藉由專題演講和壁報的研究內容可以發現：一般蔬果多重殘留分析已逐漸成熟，大家慢慢把研究觸角朝向「複雜基質」，如蜂蜜、紅酒和茶葉等以及「不適合多重分析的農藥品項」，如極性農藥等；而廠商發表的新儀器所具備的功能仍是關注的焦點之一；另外大家開始關注有關農藥代謝物和殘留定義的問題。這些趨勢皆可作爲本局日後擬訂研究目標的參考方向。爲期 4 天的會議不論在農藥領域上各式議題的訊息吸收或是異國生活的體驗都讓我受益良多，此次會議參與人數超過 500 人，來自 50 個以上的國家，足以顯現農藥領域廣大的研究能量。本局應持續鼓勵同仁多參與此類國際型會議和持續關心國際間發表的學術期刊或文件來提昇研究水平及視野。

壹、目的

現今科技發展快速、人民生活水準提高，伴隨著全球貨物的互通和大眾對食品安全的重視，使食品中農藥殘留成爲重要議題。EPRW 是歐洲大型會議之一，主題爲介紹和討論食品及飲品中農藥殘留的最新理念及發展。來自世界各地農藥相關領域的公共機構、監管機構、商業和政府實驗室等專家與學者於會議期間藉由口頭簡報、張貼研究成果壁報等方式互相流和分享資訊。首屆會議於 1996 年由荷蘭 (Netherlands) 的阿爾克馬爾 (Alkmaar) 拉開序幕，之後每 2 年舉辦 1 次。

食品中殘留農藥檢驗技術開發及公告方法研擬爲本局重要工作之一。爲能與國際接軌，前藥檢局於 2008 年開始積極參與此項國際盛會。很榮幸能受派參加第 9 屆的 EPRW 並張貼壁報發表本局研究成果，希望藉由參與此次會議來了解現階段農藥殘留領域上各國所關注的問題、新穎的研究和儀器發展等資訊，期能幫助提升本局殘留農藥檢驗技術及加快公告方法之研擬，並建立與國際農藥檢驗專家之聯絡溝通管道。

貳、過程

一、EPRW 2012 科學組織委員會成員及主辦單位背景

第 9 屆的 EPRW 於奧地利的維也納 (Vienna) 舉行，由奧地利衛生暨食品安全署 (Austrian Agency for Health and Food Safety，AGES) 負責主辦。AGES 於 2002 年成立，將原本分散的食品控制、公共健康、動物用藥和農藥等 18 部門合併爲單一組織。使 AGES 成爲跨部門的管理者，執行有關公共衛生及食品安全的相關業務，且遵循奧地利和歐盟的法律規定進行研究、分析和調查，主要負責領域爲預防及控制植物、動物和人類相關疾病；此外亦有責任審核藥物、藥用儀器、種子或農藥等相關事務。

此次會議科學委員會成員由下列 8 個組織所組成：

1. 奧地利衛生暨食品安全署 (Austrian Agency for Health and Food Safety，AGES)。
2. 德國風險評估聯邦局（Federal Institute for Risk Assessment，BfR）。
3. 瑞典國家食品局（National Food Agency，NFA）。
4. 荷蘭食品及消費產品安全局（Food and Consumer Product Safety Authority，VWA）。
5. 法國聯合服務實驗室 (Laboratories，SCL)。
6. 義大利國家衛生研究院 (National Institute of Health，ISS)。

7. 英國食品及衛生研究署 (Food and Environment Research Agency，FERA)。
8. 西班牙阿梅里亞大學 (University of Almeria，UAL)。

二、關於 EPRW 2012

此次研討會爲期 4 天 (6/25 ~ 6/28)，根據主辦單位的統計，參加者來自約 52 個國家共 514 人。爲了解各國對農藥領域及此會議的關注程度，歸納台灣及鄰近國家參與人數如下：台灣 2 人 (另 1 位爲農委會藥物毒物試驗所的涂青宇副研究員)、大陸 1 人、日本 9 人和南韓 8 人；值得注意的是來自德國的參加者有 139 人，比主辦國家還多，可能原因包含：鄰近的地理位置、儀器廠商代表者多等，但也反映出德國對此議題重視。514 個來自不同國家的人爲農藥此一議題聚集於此，足以顯現農藥領域廣大的研究能量、各國的重視度高和 EPRW 的權威性。

研討會期間兩大主要交流和討論的橋樑爲「專題演講」及「壁報發表」。專題演講計 29 篇，演講主題相當廣泛，包含對於檢驗和研究所該抱持的態度、現今農藥檢驗所面臨的挑戰、新穎儀器的應用、各式研究開發、殘留容許量和檢驗方法確效等。專題演講有 3 場爲主題演講 (Keynote Lecture)，另科學委員會將 6/27 訂爲主題日，專門就殘留定義進行相關議題的演講；參展壁報計 214 篇，分 5 大類別：PV (廠商發表) 計 38 篇、PA (分析方法之開發及應用) 計 101 篇、PM (監測及取食評估) 計 36 篇、PR (法規及風險評估) 計 9 篇和 PO (其他主題) 計 30 篇。本局亦發表 1 篇研究成果屬於分析方法之開發及應用類別，主題爲「Analysis of Pesticide Residues in Agricultural Products by Modified QuEChERS Method for LC/MS/MS and GC/MS/MS Determination」，相關內容詳見附錄四。

另研討會期間每人都擁有 1 張投票權，可投給自己最喜歡和印象深刻的參展壁報，主辦單位亦有安排審查員進行評分。於會議的最後 1 天將邀請得票最高的 3 位作者上和大家分享研究並接受表揚。會議結束前會公佈下屆開會的地點，也就是 EPRW2014。

1. 主題演講 (6/25)：

主題演講共有 3 場，茲介紹與業務相關性較高的其中 2 場。

Steven J. Lehotay 博士爲 QuEChERS 共同發表作者之一，在農藥界是位享有名聲的專家，其演講主題是「MMM means multiclass, multiresidue method, which accommodates “more more more,” but when is “more” enough?」。演講內容除分享他的研究成果，也藉由呈現事實來提出疑問，如同以問句作爲演講題目一樣。簡要摘要如下：隨著科技和生活的進步，我們可以吃到來自各國

的食物、人們也願意花更多的錢來飽餐一頓，緊接著我們願意監測更多的食品類別和農藥品項來確保安全，因爲有市場需求，儀器愈來愈強大，當然價格也愈來愈昂貴；但進行檢驗分析的依據應是環保、健康和食品安全，所面臨的挑戰也應是提高利益/金錢的比例，不應花精力和時間去從事不符合實際需求的事，所以何時 more 才算足夠？演講過程中並沒有給出確切的答案，但提供不同思考面向。最後他提到，事實上我們隨著時間流逝而逐漸成長，如果我們愈來愈聰明，那我們應該展現出我們應該做的而不是我們能做的，期勉隨著時間的推移，一切可以更美好！Lehotay 博士爲 EPRW 2012 進行開場，發表了 1 場激勵人心的演說。

Jana Hàjslovà 博士演講的主題爲「Current challenges in the analysis of pesticide residues.」，簡要摘錄如下：「準確分析困難基質」、「整合食品中不同群體的汙染物或毒物成爲單一檢驗方法」、「針對具獨特理化性的分析物量身訂做檢驗方法」以及「評估新興技術和分析策略的潛力」等皆爲當前所面臨的挑戰項目。對於較難分析的複雜基質如茶葉和香料等，可尋求合適的前處理流程，例如補足檢體水分幫助萃取或選擇適合的萃取溶劑等方式來去除基質干擾；無法分離的分析物和干擾物可考慮利用高解析度質譜儀 (HRMS) 來取代低解析度質譜儀 (LRMS)。以 Fenhexamid 此品項爲例，分析物和基質干擾物質量差距僅有 0.07 Da，使用 HRMS 可避免干擾；接著也介紹關於較難分析農藥的單一分析方法：利用 DART-MS 篩選水果中的 Dithiocarbamates，其內容爲比較 DART-TOF/MS 和 DART-orbitrap/MS 於偵測梨子中 Thiram 和 Ziram (Ziram 在結構上比 Thiram 多了個鋅) 品項之間的差異。結果顯示，雖然 DART-TOF-MS 感度較好，但分析物無法與干擾物完全分開，相關成果已發表於期刊。綜合上述，Hàjslovà 博士利用不同研究例子來說明如何運用前處理技術的改變和選擇適合的儀器來解決檢驗分析所遇到的困難。

2. 主題日 (6/27)：

現今殘留量的定義變的愈來愈複雜，當殘留定義愈複雜則愈難被執行，主因爲缺少合適的分析方法和適合的標準物質，而不能強制執行的 MRL 將無法充分保護消費者。爲了加強對消費者的保護，科學委員會決定強調「殘留量定義」之重要性，故將 6 月 27 日訂爲主題日 (共計 7 場演講) 來廣泛討論此項議題，相關內容摘要如下：

農藥存在於動物或植物體中可能藉由氧化、還原或水解作用產生代謝和鍵

結 (conjugation) 等情形而轉變成不同結構但仍具毒性或甚至毒性更高的化合物。殘留量可能依不同考量和目的而有不同的定義，例如「膳食風險評估 (dietary risk assessment)」和「MRL 的執行 (MRL enforcement purpose)」等。講者也舉一些農藥品項做例子：Fipronil 於植物中代謝後主要存在 parent compound、Fipronil sulfone 和 Fipronil amide 3 種化合物，然而所建議的殘留定義只以 Fipronil 和 Fipronil sulfone 的總量視爲 Fipronil。因爲 Fipronil amide 的毒性很低且殘留量太接近正確指標化合物所訂定的定量極限 (LOQ)，故將其排除；Bifenazate 於植物產品代謝後主要存在 parent compound (柑橘類佔 80%、蘋果類佔 40%、葡萄類佔 60%) 和 Bifenazate-diazene (柑橘和蘋果類佔 5%、葡萄類佔 40%) 2 種化合物，所建議的殘留定義也是以 Bifenazate 和 Bifenazate-diazene 的總量視爲 Bifenazate。因爲兩者化合物於溫和環境中即會互相轉換且轉換比例並不固定，故無法分開定義；Melamine 爲 Cyromazine 的代謝物，但兩者具不同毒性機制且來源可能不同，故 Cyromazine 和 Melamine 殘留定義分開計量。另外，理想上我們都希望多重殘留分析方法可適用殘留定義中的所有化合物，但因某些農藥具獨特理化性只能適用單一分析方法所以這很難被實現。

最後講者也提到未來的願景：「檢驗分析和殘留評估可以互相妥協」、「使用少量的標記化合物 (Marker compound) 即可辨別目標分析物是否有殘留，而不需偵測所有分析物」，期待有朝一日能夠實現。

3. 壁報展覽：

現今檢驗方法開發以追求簡單、快速、環保、便宜和穩定爲目標，加上儀器分析能力愈來愈強大，故自 QuEChERS (Quick、Easy、Cheap、Effective、Rugged 和 Safe 的簡稱) 前處理技術於 EPRW 2002 問世後，其成爲一股風潮且十年後仍於檢驗方法上佔有一席之地。來自各地的壁報發表人依據自家實驗室或國家所關心的議題進行研究，與 QuEChERS 前處理技術相關的檢體類別包羅萬象，包括蔬菜、水果、米、黃豆、茶葉、中草藥、大蒜、葡萄酒和蜂蜜等，分析方式則多以多重分析爲主，分析品項從數十至數百項不等。由參展的壁報可以發現，大部分把焦點放在困難基質分析方法改良和方法的確效測試。參展壁報分爲 5 大類別，茲就業務相關性較高的主題進行介紹，涵蓋的內容包含食品及動物的分析方法之開發及應用和極性農藥等獨特農藥的單一方法開發。此外還會介紹一些特別或有趣的研究。

(1) 食品、動物

檢驗分析流程不外乎萃取、淨化、儀器分析和數據處理這些部分，依基質的特性不同搭配不同的處理方式。萃取部份舉例來說，水含量少的檢體於萃取前添加水份可增加萃取效率 (茶葉、蜂蜜等)；有些檢體分析前要先經過處理：Lippold R 等人萃取蜂蜜中的農藥殘留前先將蜂蜜置於 40℃環境中平衡一整晚，Philippe Gros 等人則是在進行葡萄酒分析前先加入 5 M 的 NaOH 100 μL 來調整 pH 值。淨化部份也會依不同檢體特性 (高脂肪、高色素或高醣含量等) 進行設計並搭配不同的淨化粉劑，舉例來說，Ana Lozano 等人在進行淨化時以 $CaCl_2$ 取代原流程中的 $MgSO_4$，因其可移除較多的基質干擾且移除水分比例和 $MgSO_4$ 相似。另外依檢驗目的和需求的不同也會搭配不同分析儀器，利用 LC-MS-QTOF 可一次篩檢很多品項，不像 LC-MS/MS 受限於檢測數目。有時則是想保留較多的檢體訊息而使用「Dilute and Shoot」的前處理方式搭配較高性能的儀器進行分析。

動物檢體部分，目前大部分發表者仍把分析品項設定在有機氯 (Organochlorine) 類別的農藥，主要是考量有機氯的穩定性和非極性農藥易殘留和累積於動物檢體脂肪中等因素。由於動物基質較複雜和非極性農藥本身的特性，所以有些作者會減少取樣克數、改變萃取溶劑、添加內標或增加淨化步驟來改善分析從而得到較穩定的結果。

(2) 極性農藥

極性農藥也算是此會議的焦點之一，其獨特的理化特性使其需要獨特的方法來進行檢驗分析。相關主題壁報超過 16 篇以上，主要分析品項幾乎都含有嘉磷塞 (Glyphosate) 主要代謝物爲 AMPA。Glyphosate 是一種除草劑，利用抑制酶參與合成芳香族氨基酸的機制來抑制生長快速的植物成長，因現今抗除草劑的基因改良作物廣泛的被栽種，故 Glyphosate 被廣泛使用，但就像其它農藥一樣有藉由葉子或其他部分的吸收而污染最終產品的疑慮，所以爲各國關心的議題之一。去年 (2011) 歐盟參考實驗室 (European Union Reference Laboratories，EURL) 於所屬網站上公佈一篇名爲 QuPPe (Quick Method for the Analysis of Residues of Highly Polar Pesticides in Foods of Plant Origin Involving Simultaneous Extraction with Methanol and LC-MS/MS Determination) 的方法，此方法可用來分析不適合 QuEChERS 方法的高極性農藥，它是以甲醇進行萃取且無額外的淨化步驟，偵測儀器則爲 LC-MS/MS。此次會議所發

表的相關主題有些依循 QuPPe、有些將 QuPPe 進行改良或測試驗證，由此可見各國對新知接收的高度積極性。舉例來說，Julia Helbling 等人發表了 1 篇高極性農藥的多重檢驗方法。作者認爲 QuPPe 方法雖可用來分析高極性農藥，但不同群體的極性農藥需不同層析系統和不同的管柱，且方法中無額外淨化處理，容易因共萃物 (co-extractives) 的存在而干擾分析，故 Julia Helbling 等人發展出另 1 方法可同時偵測 Ioxynil、Dicamba、AMPA-FMOC、Glyphosate-FMOC、2,4-D、Paraquat 和 Diquat 等 7 種化合物且流程中增加 ZIC-HILIC SPE cartridge 淨化步驟來減少共萃物的含量。

(3) 特別和有趣的研究

Uwe Bohn 和 Wolfgang Schwack 介紹了 1 台 QuEChERS 前處理時可使用的自動化機器。其可自動進行的流程包含：萃取液和內標的添加、均質和萃取、清洗均質裝置、吸取上清液至淨化離心管和吸取檢液至自動進樣樣品瓶。作者比較手動和機械操做之結果差異，發現機械操作有較好的一致性。但我認爲以長期來說，仍需更多的實驗和不同檢體類別來進行印證。

Don Shelly 等人利用 QuEChERS 前處理技術進行紅酒中農藥殘留分析，但於淨化步驟時以自行設計的迷你淨化管柱 (mini-cartridge，由 $MgSO_4$ 和 PSA 組成) 來取代原方法中的分散固相萃取 (dispersive solid phase extraction，dSPE)。此 mini-cartridge 可承載約 1 mL 的萃取液，上接注射針筒來盛裝萃取液，利用針筒推桿壓力使液體通過淨化管柱收集於樣品瓶中，類似針頭式過濾器 (Syringe Filter) 的概念，利用此裝置使淨化更簡單快速 (< 1 min) 且結果數據也令人滿意！作者認爲此裝置可運用於高檢驗量的監測實驗室來加快檢體前處理的速度。

Magnus Jezussek 等人做了個有趣的研究，選擇橘子、柳橙和柚子作爲測試樣品，想了解水果皮的厚度與農藥殘留量和殘留濃度的相關性。結果顯示，削皮過程中果肉在沒有受到汙染的情況下，當皮的厚度愈高其農藥殘留量愈低且殘留濃度也愈低。

Sadat Nawaz 和 Helen Barker 統整了近 10 年的監測結果 (分析檢體約 16,000 件，共產生 2,500,000 組結果)，想藉此了解英國農藥殘留的趨勢及變化。作者觀察到以下趨勢：

分析方法的改進 (萃取技術和分析儀器)：大家朝向多重分析 (增加分析品項) 並逐漸降低 LOQ 數值，使農藥殘留分析更加快速、有效率；法規的改變：

有效減少某些農藥的殘留率，例如梨子中的 Tolylfluanid、Carbendazim、Azinphos methyl 和 Chlormequat 殘留量減少；新活性成分 (品項) 的產生：減少舊品項的使用率，以殺真菌劑爲例，Carbendazim、Imazalil 和 Thiabendazole 使用頻率降低，而 Boscalid、Fenhexamid 和 Strobiluins 使用頻率增加：地理的差異：使每個樣品農藥殘留平均數目增加，於非歐盟區域此現象較明顯。

4. 得獎作品：

3 篇得獎作品分別爲：Jasak Julia 等人發表的「The analysis of Triazole-Metabolites in Plant Materials Using DMS-MS/MS」、Julia Hepperle 等人發表的「Analysis of Amitraz (sum) from QuEChERS Extracts - Comparison of the Method involving Analysis of Individual MRM-Amenable Metabolites (DMF, DMPF and DMA) with a Method Involving Cleavage to DMA」和 Paula Medina 等人發表的「Conversion Factors e-learning」。接下來將簡單介紹此 3 篇研究內容：

(1) The analysis of Triazole-Metabolites in Plant Materials Using DMS-MS/MS

1,2,4-Triazole (TRZ) 和主要的結合物 (conjugates)：Triazole acetic acid (TAA)、Triazole lactic acid (TLA) 和 Triazole alanine (TAL)，此 4 樣化合物被視作 Triazole 衍生代謝產物 (Triazole derivative metabolites，TDM_S)，爲來自 Triadimefon 或 Tebuconazole 的降解產物。TDM_S 之高極性和低分子量的特性使很難於基質存在下進行定性和定量分析，即便使用 LC-MS/MS 亦很難進行偵測。爲了降低分析時共萃物的干擾並使 LOQ 值 ≤ 0.01 mg/kg，應於進入 MS/MS 系統前加裝增強選擇性的過濾裝置。作者選擇使用 DMS (differential mobility spectrometer) 一種類似化學預濾器 (chemical pre-filter) 的作用，接在 MS/MS 前端，即成爲 DMS-MS/MS 的裝置，想藉此達到分離上述 4 化合物之目的。

DMS 的機制原理爲：在大氣壓力下利用離子於高和低電場之不同遷移率進行離子的分離。DMS 置於 Q1 前端，由分離電壓 (separation voltage，SV) 和補償電壓 (the compensation voltage，CoV) 2 組電極組成。SV 所扮演的角色爲利用不同週期的電場分量帶領離子以不同速率朝某方向移動，最後與電極壁碰撞而轉成中性使其無法被偵測；DC 則是扮演施加正確的電壓將所選擇的離子導入正確軌跡使能順利被偵測的角色。實驗結果顯示，LC-DMS-MS/MS 爲一強大分析工具，可應用於植物基質中 TDM_S 的定性及定

量鑑別。作者所使用的儀器為 AB SCIEX SelexION™搭配 AB SCIEX QTRAP® 5500。

(2) Analysis of Amitraz (sum) from QuEChERS Extracts - Comparison of the Method involving Analysis of Individual MRM-Amenable Metabolites (DMF, DMPF and DMA) with a Method Involving Cleavage to DMA

此篇研究是關於 Amitraz 和其代謝產物 DMF、DMPF 和 DMA 之間轉換比率的確認。一般認為 Amitraz 會以 1：2 的形式代謝成 DMA。然而 EURL 於 2007 年公佈 1 篇以 QuEChERS 為基礎的多重方法：可使用 LC-MS/MS 同時偵測 Amitraz、DMF、DMPF 和 DMA 等化合物，其文件認為 Amitraz 會以 1：1 的形式代謝成 DMPF。作者為確認 Amitraz 和代謝產物間的關係設計了 1 個方法：以梨子 (pear) 作為基質將 QuEChERS 處理完之萃取液 (未經淨化) 加入 NaOH 使其完全水解為 DMA，所得的含量與原 EURL 公佈之方法結果相比較。

結果顯示，使用 EURL 公佈方法僅測得 DMPF 化合物，且其含量與水解處理後之 DMA 含量相近。故可知於梨子基質中，Amitraz 會以 1：1 比例轉成 DMPF；而鹼處理後之 DMPF 與 DMA 轉換比例亦為 1：1。因此如使用 Amitraz 以 1：2 方式代謝成 DMA 之公式進行反推，將會低估 Amitraz 的殘留量。

(3) Conversion Factors e-learning

此篇壁報主要是介紹有關 EURL 所提供的實用工具，可利用此工具進行殘留農藥定義中以加總方式計算殘留量及轉換係數的演算。進入此網頁 (http://www.eupt.es/e-learning/index.php) 可利用字母搜尋、鍵入名子或鍵入 CAS 代號來尋找所需要的品項，對於每個搜尋的到的農藥，系統皆提供結構、分子式及分子量供使用者參考。目前資料庫僅包含現階段歐盟於蔬菜及水果殘留農藥監測中屬多重殘留化合物加總的品項，不過此網頁將持續更新、擴大。

5. EPRW 2014：

待得獎者在結束研究分享、接受頒獎和表揚後，此次會議的主席 Sonja Masselter 博士上台發表致詞：感謝參與者和辛苦的工作夥伴並接受科學委員會的獻花，場面溫馨且夾雜一些些的感傷。最後 Masselter 博士將 EPRW 的代表令旗交棒給下屆主席，接著由他來介紹他的國家並邀請大家一同參加下次

會議。

下屆會議將於比利時 (Belgium) 的首都布魯塞爾 (Brussels) 舉行。比利時是個奇特的地方，它分成 3 個語言區塊：荷蘭語、法語和德語。隨著下屆主席演說結束，Masselter 博士再次上台和我們道再見，此次會議圓滿落幕。

參、心得

很慶幸此次會議可以與涂副研究員一路同行，較不孤單。維也納這裡環境優美、大部分的人們都很友善、氣候宜人、治安良好，最重要的交通便利。我很喜歡這裡的環境，讓從外地來的我可以心情愉悅的面對每 1 天！但不可否認，此地物價較高。

爲期 4 天的議程滿滿，老實說維持專注力有點辛苦，想要全盤吸收所有的訊息不太可能，尤其到最後幾天那種累積的疲累讓我有點吃不消！但會議期間每場專題演講和參展壁報上各式的研究主題等訊息都讓我獲益良多。畢竟台灣是個島國，即便現在網路無國界，但地理的限制多多少少會造成阻礙，藉著參加此種國際型的研討會能幫助我們更直接、更深入的了解別人都在做什麼？做到了什麼程度？此次會議大部分他們所關心的議題，在工作時多少都有接收到類似資訊，可見台灣並沒有跟其他國家有太大的脫節。

藉由此次壁報得獎者的主題可以發現，大家開始關注有關農藥代謝物和殘留定義的問題，主辦單位甚至爲殘留定義設了 1 天主題日來專門討論此議題；EURL 也特別建置 e-learning 網站供大家學習和使用，在在都顯示代謝物議題逐漸成爲趨勢，這提醒我們也該留意此類問題。另外也可從演講主題和壁報內容發現，一般蔬果多重殘留分析技術已逐漸成熟，大家慢慢把研究觸角朝向難以分析的基質和不適合多重分析的農藥品項，對於我們來說，這也是遲早要面對的問題。新的儀器功能愈來愈強大，雖然我們必須考慮所增加的功能是否有其必要性，但不可否認當功能符合趨勢所需，它將會成爲趨勢 (LC-MS/MS)，故我們仍應隨時注意新儀器的資訊。舉例來說，得獎研究之一：AB SCIEX SelexION™搭配 AB SCIEX QTRAP® 5500 儀器即受到不少的關注。

肆、建議

此次 EPRW 之行不論是農藥領域上的充實或不同生活的體驗都讓我獲益良多，再次感謝食品藥物管理局能給我機會參加。以下是本人所提出的一些建議：

一、持續鼓勵同仁多參與此類國際型會議：

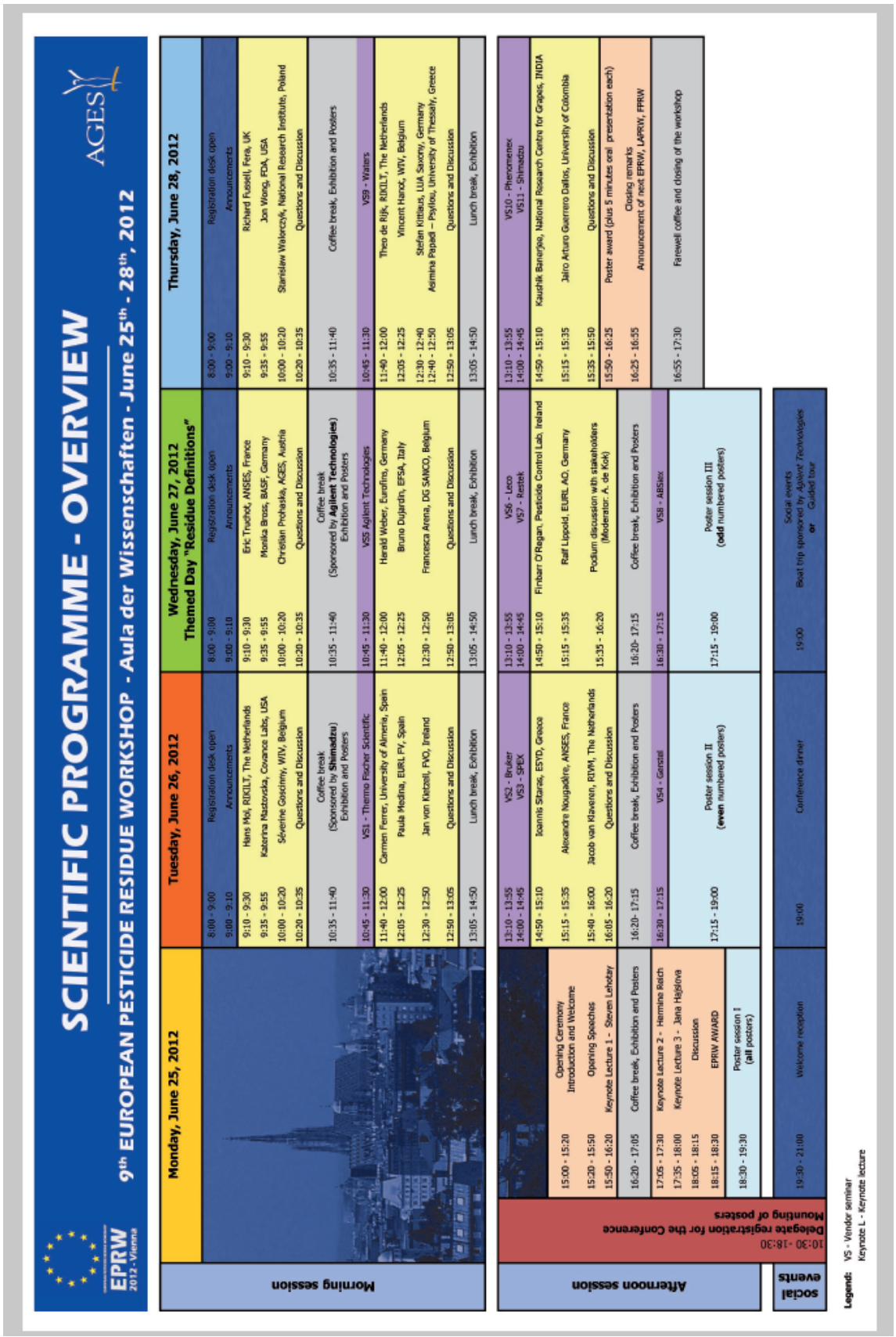
檢驗方法開發是本組的核心業務，但我們所面臨的一個事實：無法全心全意花

費時間和精力只做研究，我們勢必要縮短研究及開發方法所花費的時間，最快的方式當然就是「了解世界的趨勢去鎖定研究方向」和「參考別人的研究成果來進行改良」。故建議持續鼓勵同仁積極爭取參與國際型會議的機會。

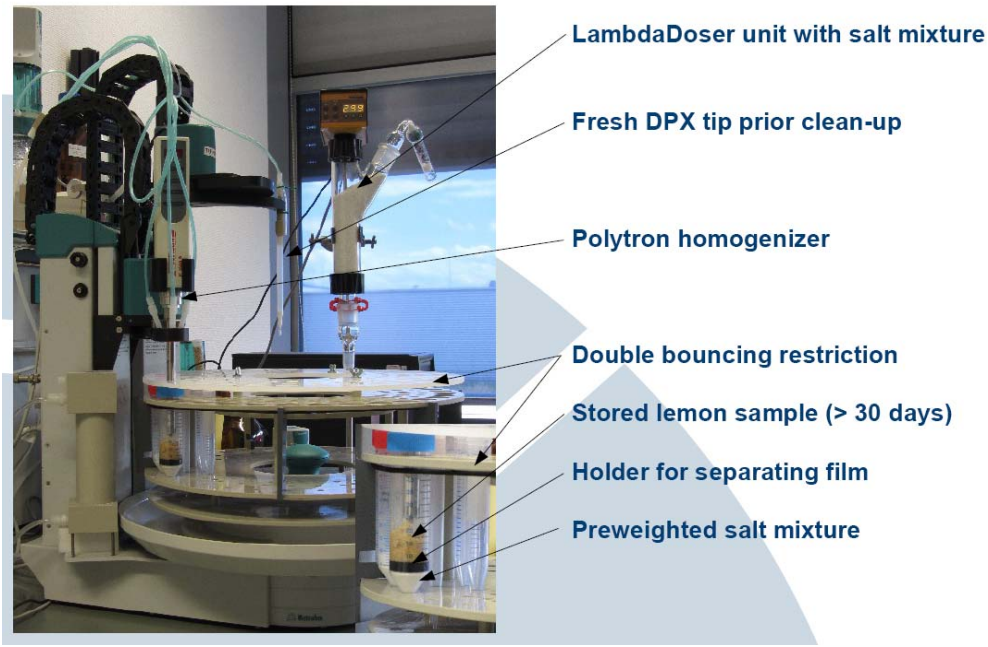
二、持續關心國際間發表的學術期刊或文件：

除了藉由參加國際會議來了解檢驗開發的趨勢外，也應同步更新品質規範、確效或分析技術等相關文件等的觀念或知識，舉例來說，EURL 於今年更新了方法確效和品質的文件：「SANCO/12495/2011」、也會不定期公佈新的檢驗方法：QuPPe 等。如能快速掌握此類訊息將有助於提升我們的提昇研究水平。

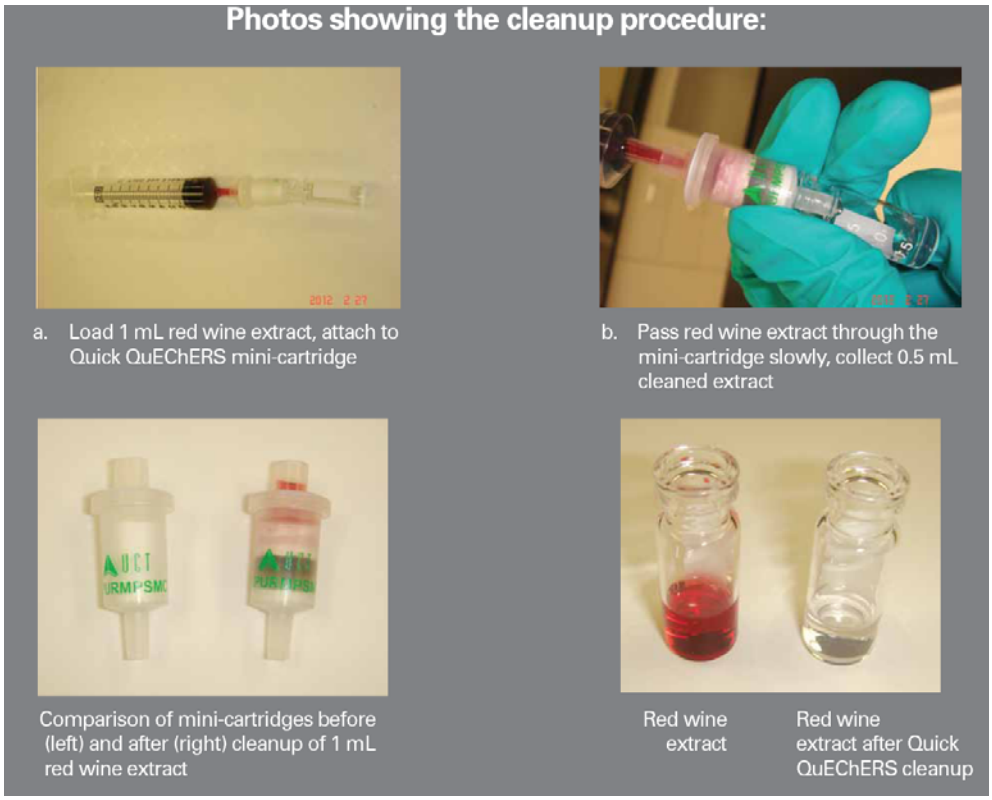
附錄一：議程


SCIENTIFIC PROGRAMME - OVERVIEW
9th EUROPEAN PESTICIDE RESIDUE WORKSHOP - Aula der Wissenschaften - June 25th - 28th, 2012
EPRW 2012 - Vienna
AGES
Monday, June 25, 2012
Tuesday, June 26, 2012
Wednesday, June 27, 2012
Themed Day "Residue Definitions"
Thursday, June 28, 2012
Morning session
Afternoon session
social events
Delegate registration for the Conference
Mounting of posters
Opening Ceremony
Introduction and Welcome
Opening Speeches
Keynote Lecture 1 - Steven Lehotay
Coffee break, Exhibition and Posters
Keynote Lecture 2 - Hermine Reich
Keynote Lecture 3 - Jana Hajslova
Discussion
EPRW AWARD
Poster session I
(all posters)
Welcome reception
Registration desk open
Announcements
Hans Mol, RIKILT, The Netherlands
Katerina Mastovska, Covance Labs, USA
Séverine Goscinny, WIV, Belgium
Questions and Discussion
Coffee break
(Sponsored by Shimadzu)
Exhibition and Posters
VS1 - Thermo Fischer Scientific
Carmen Ferrer, University of Almeria, Spain
Paula Medina, EURL FV, Spain
Jan van Klaveren, FVO, Ireland
Questions and Discussion
Lunch break, Exhibition
VS2 - Bruker
VS3 - SPEX
Ioannis Stanos, ESYD, Greece
Alexandre Nougadère, ANSES, France
Jacob van Klaveren, RIVM, The Netherlands
Questions and Discussion
Coffee break, Exhibition and Posters
VS4 - Gerstel
Poster session II
(even numbered posters)
Conference dinner
Eric Trachot, ANSES, France
Monika Bross, BASF, Germany
Christian Prchalska, AGES, Austria
Questions and Discussion
Coffee break
(Sponsored by Agilent Technologies)
Exhibition and Posters
VS5 Agilent Technologies
Herald Weber, Eurofins, Germany
Bruno Dujardin, EFSA, Italy
Francesca Arena, DG SANCO, Belgium
Questions and Discussion
Lunch break, Exhibition
VS6 - Leco
VS7 - Restek
Finbarr O'Regan, Pesticide Control Lab, Ireland
Ralf Lippold, EURL AO, Germany
Podium discussion with stakeholders
(Moderator: A. de Kok)
Coffee break, Exhibition and Posters
VS8 - ABSciex
Poster session III
(odd numbered posters)
Social events
Boat trip sponsored by Agilent Technologies
or
Guided tour
Registration desk open
Announcements
Richard Fussell, Fera, UK
Jon Wong, FDA, USA
Stanislaw Walorczyk, National Research Institute, Poland
Questions and Discussion
Coffee break, Exhibition and Posters
Theo de Rijk, RIKILT, The Netherlands
Vincent Hanot, WIV, Belgium
Stefan Kittlaus, LUA Saxony, Germany
Asimina Papadi – Psyllou, University of Thessaly, Greece
Questions and Discussion
Lunch break, Exhibition
VS10 - Phenomenex
VS11 - Shimadzu
Kaushik Banerjee, National Research Centre for Grapes, INDIA
Jairo Arturo Guerrero Dallos, University of Colombia
Questions and Discussion
Poster award (plus 5 minutes oral presentation each)
Closing remarks
Announcement of next EPRW, LAPRW, FPRW
Farewell coffee and closing of the workshop
Legend: VS - Vendor seminar
Keynote L - Keynote lecture

附錄二：參展壁報相關資訊



圖一、Uwe Bohn 和 Wolfgang Schwack 設計的 QuEChERS 自動機械裝置 (EPRW 2012-PA006)。

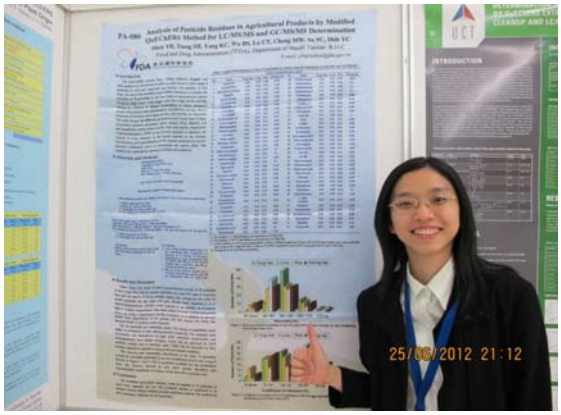


圖二、Xiaoyan Wang 等人設計的 mini-cartridges 裝置 (EPRW 2012-PA079)。

附錄三：生活點滴

圖三、與會場大門合影。

圖四、專題演講場地。



圖五、與發表壁報合影。

圖六、壁報展覽場地之一角。

圖七、博物館晚餐聚會。

圖八、與涂青宇副研究員、Polly Grundy 博士和 Sabine Henning 博士 (右) 於博物館合影。

圖九、城市導覽。

圖十、維也納摩天輪 (Wiener Riesenrad)。

圖十一、熊布倫宮 (Schloss Schonbrunn) 一景。

圖十二、百水公寓 (Hundertwasserhaus)。

圖十三、與 Jon Wong 博士合影。

圖十四、Sonja Masselter 主席接受獻花。

圖十五、Masselter 主席交接 EPRW 代表令旗給 EPRW2014 主席。

圖十六、會議結束主辦單位於大門前立的祝福牌 (可惜沒有中文)。

圖十七、EPRW 2012 會場大門留影。

PA 080

Analysis of Pesticide Residues in Agricultural Products by Modified QuEChERS Method for LC/MS/MS and GC/MS/MS Determination

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Due to the number of newly authorized pesticides grow quickly and trade between countries frequently, multi-pesticide residue methods become a necessary trend. In this study, the used of the modified QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) techniques to evaluate the suitability of 78 pesticides in the four kinds of representative matrix (tong hao, lime, rice and dry oolong tea) by liquid gas chromatography-ESI-tandem mass spectrometry (LC/MS/MS) and gas chromatography-EI-tandem mass spectrometry (GC/MS/MS) for detection. Under this study, it devised the different procedures and different clean-up powders (primary secondary amine sorbent (PSA), MgSO4, C18 and graphitized carbon black (GCB)) for different matrix respectively.

Preparation of sample is divided into extraction and purification procedure. Extraction and purification of fruit and vegetable samples were the same as the buffered AOAC 2007.01 method. Considered the matrix properties, the vegetable, fruit, rice and dry tea was weighed 15 g, 15 g, 5 g and 2 g respectively, then it added the 10 mL water into the rice and dry tea respectively to make sure the effective extraction of pesticides from low water content sample (water content<80%). After extraction, the adjustment of lime pH value to 5-5.5 to increase the stability of the acid-sensitive pesticides. About clean-up powders, additional C18 sorbent was added during rice purification procedure to help removing the grease; more amount of PSA sorbent and additional GCB sorbent were added during dry tea purification procedure to reduce water-soluble co-extractions, such as theophylline, polyphenol and chlorophyll. Triphenylphosphate (TPP) as an internal standard to eliminate the sources of error inherent in the liquid transfers in the method. Identification and quantification of the selected pesticides by matrix-matched calibration curve to compensate the matrix effect.

The result showed some pesticides got unsatisfied result due to their physicochemical properties. For example, both cyromazine and dinotefuran are high polar pesticides; pymetrozine and chinomethionat have planar structure which will be adsorbed by GCB sorbent; oxolinic acid is quinolone class which will be adsorbed by PSA sorbent; captafol is unstable in acetonitrile and alkaline condition. However, the other 72 pesticides available for this method, the recovery between 60 and 120% (mostly 80-100%) and reproducibility (coefficient of variation of less than 25%) performed well, the limit of quantification between 0.01 and 0.25 ppm, The result indicated these modified QuEChERS methods could be applied to 72 pesticides in most fruit, vegetable and tea.

QuEChERS, LC/MS/MS, GC/MS/MS, agricultural products

S.J. Lehotay. 2007. Determination of pesticide residues in foods by acetonitrile extration and partitioning with magnesium sulfate: Collaborative Study. J. AOAC Int. 90(2): 485-520.

PA-080 Analysis of Pesticide Residues in Agricultural Products by Modified QuEChERS Method for LC/MS/MS and GC/MS/MS Determination

Shen YR, Tseng SH, Yang KC, Wu BS, Lu CT, Cheng MW, Su SC, Shih YC

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Introduction

The QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) method was introduced in 2003; it could extract a wide range of pesticides in fruit and vegetable has become very popular. In this study, the used of the modified QuEChERS techniques to evaluate the suitability of 78 pesticides in the four kinds of representative matrix [Tong hao (high water), Lime (high acid), Rice (high starch) and Dry Oolong tea (difficult or unique commodity)] by liquid chromatography-ESI-tandem mass spectrometry (LC/MS/MS) and gas chromatography-EI-tandem mass spectrometry (GC/MS/MS) for detection. The study devised the different procedures and various type of clean-up powders [primary secondary amine sorbent (PSA), MgSO4, C18 and graphitized carbon black (GCB)] with each matrix, respectively. Triphenylphosphate (TPP) as an internal standard to eliminate the sources of error inherent in the liquid transfers in the method. Identification and quantification of the selected pesticides by matrix-matched calibration curve to compensate the matrix effect. The method was validated by recovery, precision and sensitivity.

Materials and Methods

Homogenized sample:
Tong Hao (Crown daisy) 15 g;
Lime 15 g;
Rice 5 g + 10 mL cold water;
Dry Oolong tea 2 g + 10 mL cold water

↓

Add 15 mL 1% acetic acid in acetonitrile

↓

Add internal standard (Triphenylphosphate)

↓

Add extraction powders (6 g $MgSO_4$ anhydrous + 1.5 g $CH_3CHOONa$ anhydrous)

1. Shake vigorously for 1min.
2. Shake for 1min at 1500 rpm.
3. Centrifuge for 1min at 3500 rpm.
4. The adjustment of lime pH value to 5-5.5.

↓

8 mL of the upper layer + clean-up powders:
Tong Hao and Lime: 400 mg PSA + 1200 mg $MgSO_4$;
Rice: 400 mg PSA + 1200 mg MgSO4 + 400 mg end-capped C18;
Dry Oolong tea: 600 mg PSA + 1200 mg $MgSO_4$ + 400 mg end-capped C18 + 64 mg GCB

1. Shake vigorously for 30 sec.
2. Centrifuge for 2 min at 4000 rpm.

↓

LC/MS/MS:
500 µL of the extracts transfer into a vial, mix with 300 µL methanol and 200 µL water.

GC/MS/MS:
1. 4 mL of the extracts transfer into a 15 mL centrifuge tube, evaporation to dryness by a stream of nitrogen, redissolved in 1 mL acetonitrile with 1% acetic acid 500 µL of the extracts transfer into a vial, mix with 300 µL methanol and 200 µL water.
2. 500 µL of the extracts transfer into a vial, mix with 200 µL acetone and 300 µL acetonitrile with 1% acetic acid.

Table 1 Limits of quantification (LOQ) of 72 pesticides in various crops are detected by LC/MS/MS[a] and GC/MS/MS[b]

		Limits of quantification (ppm)[c]								
No.	Name	Tong Hao	Lime	Rice	Oolong tea[d]	No.	Name	Tong Hao	Lime	Rice	Oolong tea
1	Abamectin	0.01	0.05	0.05	0.05	38	Tetraconazole	0.01	0.01	0.01	0.05
2	Azoxystrobin	0.01	0.01	0.01	0.05	39	Tetramethrin	0.01	0.01	0.01	0.1
3	Bensulfuron-methyl	0.01	0.01	0.01	0.05	40	Thiabendazole	0.01	0.01	0.05	0.1
4	Boscalid	0.01	0.01	0.05	0.05	41	Thiamethoxam	0.01	0.01	0.01	0.1
5	Butocarboxim	0.01	0.01	0.01	0.05	42	Thiobencarb	0.01	0.01	0.01	0.05
6	Carbendazim	0.01	0.01	0.01	0.05	43	Tolfenpyrad	0.01	0.01	0.01	0.05
7	Cinosulfuron	0.01	0.01	0.01	0.05	44	Triadimenol	0.01	0.01	0.01	0.05
8	Clomazone	0.01	0.01	0.01	0.05	45	Trifloxystrobin	0.01	0.01	0.01	0.05
9	Cyazofamid	0.01	0.01	0.01	0.05	46	Triflumizole	0.01	0.01	0.01	0.05
10	Cyclosulfamuron	0.01	0.01	0.01	0.05	47	Triforine	0.01	0.05	0.05	0.1
11	Cyproconazole	0.01	0.01	0.01	0.05	48	XMC	0.01	0.01	0.01	0.05
12	Dimethenamid	0.01	0.01	0.01	0.05	49	α-BHC	0.01	0.01	0.01	0.05
13	Dimethomorph	0.01	0.01	0.01	0.05	50	α-chlordane	0.01	0.01	0.01	0.05
14	Etrimfos	0.01	0.01	0.01	0.05	51	β-chlordane	0.01	0.01	0.01	0.05
15	Famoxadone	0.05	0.01	0.01	0.05	52	Bromophos-ethyl	0.01	0.01	0.01	0.05
16	Fluazifop-P-butyl	0.01	0.01	0.01	0.05	53	Chloropropylate	0.01	0.01	0.01	0.05
17	Halfenprox	0.01	0.01	0.01	0.05	54	Chlozolinate	0.01	0.01	0.01	0.05
18	Haloxyfop-methyl	0.01	0.01	0.05	0.1	55	Cypermethrin	0.01	0.05	0.01	0.05
19	Hexaflumuron	0.05	0.01	0.01	0.1	56	Dicofol	0.05	0.05	0.01	0.25
20	Imibenconazole	0.05	0.05	0.01	0.05	56	DCBP	0.01	0.01	0.01	0.05
21	Imidacloprid	0.05	0.01	0.01	0.1	57	Epoxiconazole	0.01	0.01	0.01	0.05
22	Indoxacarb	0.01	0.01	0.05	0.05	58	Fenbuconazole	0.01	0.01	0.01	0.05
23	Isazofos	0.01	0.01	0.01	0.05	59	Fenpropimorph	0.01	0.01	0.01	0.05
24	Kresoxim-methyl	0.01	0.01	0.01	0.05	60	Fenvalerate	0.01	0.01	0.01	0.05
25	Metconazole	0.01	0.01	0.01	0.05	61	Fthalide	0.01	0.05	0.01	0.05
26	Molinate	0.01	0.05	0.05	0.1	62	Heptachlor	0.05	0.01	0.01	0.05
27	Nuarimol	0.01	0.01	0.01	0.25	63	Hexazinone	0.05	0.05	0.01	0.05
28	Oxycarboxine	0.01	0.01	0.01	0.05	64	Iprodione	0.01	0.05	0.25	0.1
29	Pirimicarb	0.01	0.01	0.01	0.05	65	Isoprothiolane	0.01	0.25	0.01	0.05
30	Propamocarb hydrochloride	0.01	0.01	0.01	0.05	66	γ-BHC Lindane	0.01	0.01	0.01	0.05
31	Propaphos	0.01	0.01	0.01	0.05	67	Oxadixyl	0.01	0.01	0.01	0.05
32	Propargite	0.01	0.01	0.01	0.05	68	Oxyfluorfen	0.01	0.01	0.01	0.05
33	Pyraclostrobin	0.01	0.01	0.01	0.05	69	Parathion-ethyl	0.01	0.25	0.01	0.05
34	Pyriproxyfen	0.01	0.01	0.01	0.05	70	Quinalphos	0.01	0.01	0.01	0.05
35	Quinoxyfen	0.05	0.05	0.05	0.05	71	Salithion	0.01	0.01	0.01	0.05
36	Quizalofop-ethyl	0.01	0.01	0.01	0.1	72	Vinclozolin	0.01	0.01	0.01	0.05
37	Tebuconazole	0.01	0.01	0.05	0.05						

a: The number of 1-48 pesticides are detected by LC/MS/MS.
b: The number of 49-72 pesticides are detected by GC/MS/MS.
c: LOQ is defined as the lowest acceptability criteria validated spike level from 0.01 to 0.25 ppm (mean recoveries (n=5) for each crops in the range 60-120%, with an CV≤25%; qualitative ion's S/N>3 and quantitative ion's S/N>10).
d: Oolong tea is the leaf after processing

Results and Discussion

Table 1 shows the result of limit of quantification (LOQ) of 78 pesticides in four crops. The LOQ for mostly pesticides can meet 0.01 ppm in tong hao, lime and rice matrix. Even in complex matrix like oolong tea, the LOQ for mostly pesticides can also meet 0.05 ppm. Dicofol easily degrades to *4, 4'*-dichlorobenzophenone (DCBP) when exposed to an alkaline environment, light or a higher temperature. This study added 1% acetic acid in acetonitrile which can reduce degradation during extraction. It is difficult to prevent dicofol from degradation in GC system. For this reason, the study also detected DCBP to confirm result of dicofol.

The six pesticides got unsatisfied result. The reason of unsatisfied result might be attributed to their physicochemical properties. For example, both cyromazine and dinotefuran are high polar pesticides; pymetrozine and chinomethionat have planar structure which will be adsorbed by GCB sorbent; oxolinic acid is quinolone class which will be adsorbed by PSA sorbent; captafol is unstable in acetonitrile and alkaline condition.

The recovery and repeatability distribution of the other 72 pesticides (except six unsuitable pesticides) at the low fortification level in four crops is showed in Figure 1 and 2. The 72 pesticides available for this fortification level, the recovery between 60 and 120% (mostly 80-100%) and reproducibility (coefficient of variation of less than 25%) perform well.

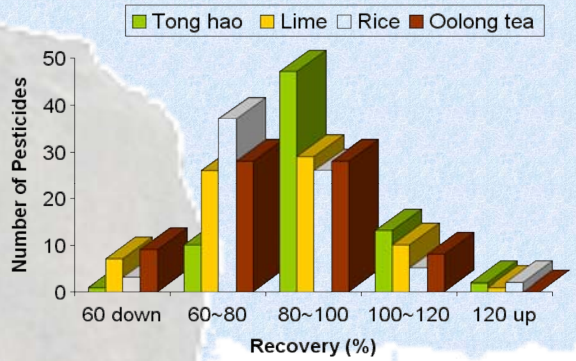


Figure 1. Recovery (%) of 72 pesticides at the 0.01 ppm and 0.05 ppm (Oolong tea only) fortification level in four crops (n=5).

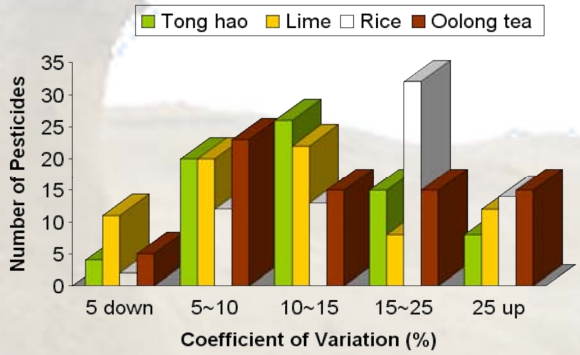


Figure 2. Coefficient of variation (%) of 72 pesticides at the 0.01 ppm and 0.05 ppm (Oolong tea only) fortification level in four crops (n=5).

Conclusion

The modified QuEChERS methods could be applied to 72 pesticides in most fruit, vegetable and tea. This proposed method is considered to be another Taiwan official multiple pesticide analytical method. The method has now undergone validation for 213 pesticides.

附錄五：心得分享簡報內容

參加歐洲農藥殘留研討會心得報告

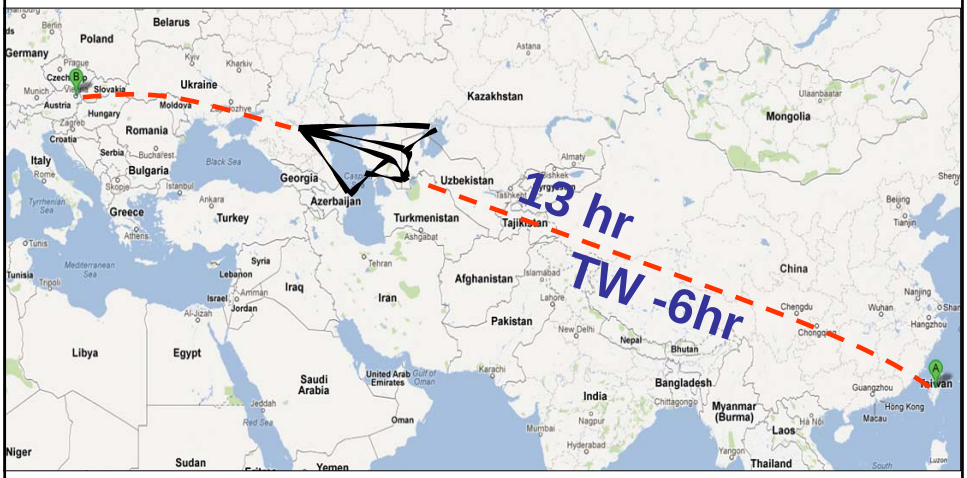
9th EPRW2012 6/25-6/28

沈盈如

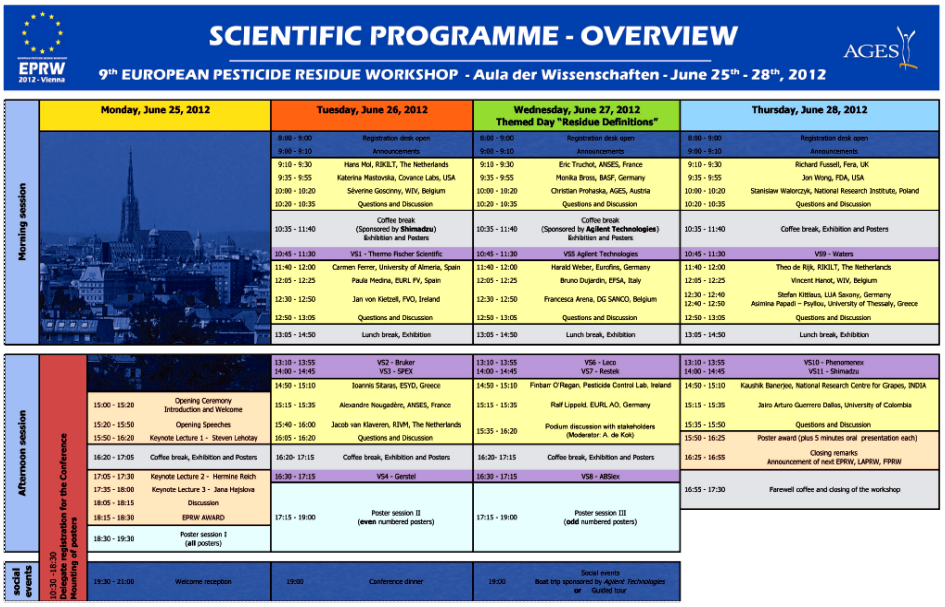
What is EPRW?

- European Pesticide Residue Workshop
- Participants, Vendors, Oral/Poster presentations…
- 1996~
- 2012EPRW_9th in Vienna
  - AGES, Austrian Agency for Health and Food Safety

4465 Miles ≒ 7186 Kilometer



Google Map



SCIENTIFIC PROGRAMME - OVERVIEW

9th EUROPEAN PESTICIDE RESIDUE WORKSHOP - Aula der Wissenschaften - June 25th - 28th, 2012

EPRW 2012 - Vienna

AGES

	Monday, June 25, 2012	Tuesday, June 26, 2012	Wednesday, June 27, 2012 Themed Day "Residue Definitions"	Thursday, June 28, 2012
Morning session		8:00 - 9:00 Registration desk open 9:00 - 9:10 Announcements 9:10 - 9:30 Hans Mol, RIKILT, The Netherlands 9:35 - 9:55 Katerina Mastovska, Covance Labs, USA 10:00 - 10:20 Séverine Goscinny, WIV, Belgium 10:20 - 10:35 Questions and Discussion 10:35 - 11:40 Coffee break (Sponsored by **Shimadzu**) Exhibition and Posters 10:45 - 11:30 VS1 - Thermo Fischer Scientific 11:40 - 12:00 Carmen Ferrer, University of Almeria, Spain 12:05 - 12:25 Paula Medina, EURL FV, Spain 12:30 - 12:50 Jan von Kietzell, FVO, Ireland 12:50 - 13:05 Questions and Discussion 13:05 - 14:50 Lunch break, Exhibition	8:00 - 9:00 Registration desk open 9:00 - 9:10 Announcements 9:10 - 9:30 Eric Truchot, ANSES, France 9:35 - 9:55 Monika Bross, BASF, Germany 10:00 - 10:20 Christian Prohaska, AGES, Austria 10:20 - 10:35 Questions and Discussion 10:35 - 11:40 Coffee break (Sponsored by **Agilent Technologies**) Exhibition and Posters 10:45 - 11:30 VS5 Agilent Technologies 11:40 - 12:00 Harald Weber, Eurofins, Germany 12:05 - 12:25 Bruno Dujardin, EFSA, Italy 12:30 - 12:50 Francesca Arena, DG SANCO, Belgium 12:50 - 13:05 Questions and Discussion 13:05 - 14:50 Lunch break, Exhibition	8:00 - 9:00 Registration desk open 9:00 - 9:10 Announcements 9:10 - 9:30 Richard Fussell, Fera, UK 9:35 - 9:55 Jon Wong, FDA, USA 10:00 - 10:20 Stanislaw Walorczyk, National Research Institute, Poland 10:20 - 10:35 Questions and Discussion 10:35 - 11:40 Coffee break, Exhibition and Posters 10:45 - 11:30 VS9 - Waters 11:40 - 12:00 Theo de Rijk, RIKILT, The Netherlands 12:05 - 12:25 Vincent Hanot, WIV, Belgium 12:30 - 12:40 Stefan Kittlaus, LUA Saxony, Germany 12:40 - 12:50 Asimina Papadi – Psyllou, University of Thessaly, Greece 12:50 - 13:05 Questions and Discussion 13:05 - 14:50 Lunch break, Exhibition
Afternoon session	10:30 -18:30 Delegate registration for the Conference Mounting of posters 15:00 - 15:20 Opening Ceremony Introduction and Welcome 15:20 - 15:50 Opening Speeches 15:50 - 16:20 Keynote Lecture 1 - Steven Lehotay 16:20 - 17:05 Coffee break, Exhibition and Posters 17:05 - 17:30 Keynote Lecture 2 - Hermine Reich 17:35 - 18:00 Keynote Lecture 3 - Jana Hajslova 18:05 - 18:15 Discussion 18:15 - 18:30 EPRW AWARD 18:30 - 19:30 Poster session I (**all** posters)	13:10 - 13:55 VS2 - Bruker 14:00 - 14:45 VS3 - SPEX 14:50 - 15:10 Ioannis Sitaras, ESYD, Greece 15:15 - 15:35 Alexandre Nougadère, ANSES, France 15:40 - 16:00 Jacob van Klaveren, RIVM, The Netherlands 16:05 - 16:20 Questions and Discussion 16:20- 17:15 Coffee break, Exhibition and Posters 16:30 - 17:15 VS4 - Gerstel 17:15 - 19:00 Poster session II (**even** numbered posters)	13:10 - 13:55 VS6 - Leco 14:00 - 14:45 VS7 - Restek 14:50 - 15:10 Finbarr O'Regan, Pesticide Control Lab, Ireland 15:15 - 15:35 Ralf Lippold, EURL AO, Germany 15:35 - 16:20 Podium discussion with stakeholders (Moderator: A. de Kok) 16:20- 17:15 Coffee break, Exhibition and Posters 16:30 - 17:15 VS8 - ABSiex 17:15 - 19:00 Poster session III (**odd** numbered posters)	13:10 - 13:55 VS10 - Phenomenex 14:00 - 14:45 VS11 - Shimadzu 14:50 - 15:10 Kaushik Banerjee, National Research Centre for Grapes, INDIA 15:15 - 15:35 Jairo Arturo Guerrero Dallos, University of Colombia 15:35 - 15:50 Questions and Discussion 15:50 - 16:25 Poster award (plus 5 minutes oral presentation each) 16:25 - 16:55 Closing remarks Announcement of next EPRW, LAPRW, FPRW 16:55 - 17:30 Farewell coffee and closing of the workshop
social events	19:30 - 21:00 Welcome reception	19:00 Conference dinner	19:00 Social events Boat trip sponsored by *Agilent Technologies* or Guided tour	

- Participants: 514
  - Country: 52 (from 5 continents)
    - Taiwan: 2
    - South Korea: 8
    - Japan: 9
    - China: 1
    - Germany: 139
- Oral: 29
- Poster: 214
  - PV(38), PA(101), PM(36), PR(9) and PO(30)
- Winners: 3
- End……Next…10th EPRW 2014

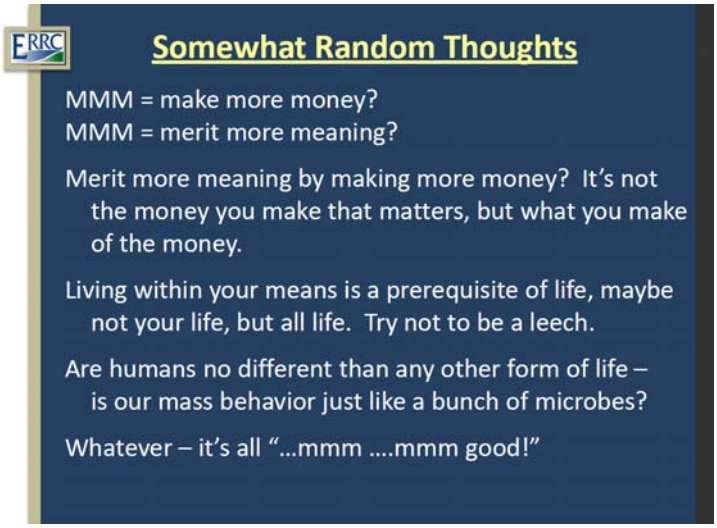
- ORAL PRESENTATIONS
- POSTER PRESENTATIONS
- WINNER'S POSTER
- 心得
- End……Next…10th EPRW 2014

ORAL PRESENTATIONS

Keynote Lecture:

1. MMM means multiclass, multiresidue method, which accommodateds “more more more”, but when is “more” enough? –Steven J. Lehotay
2. Current challenges in the analysis of pesticide residues –Jana Hajslova

MMM means multiclass, multiresidue method, which accommodates “more more more”, but when is “more” enough? –Steven J. Lehotay



Steven J. Lehotay

MMM means multiclass, multiresidue method, which accommodateds “more more more”, but when is “more” enough? –Steven J. Lehotay

My Participation in Previous EPRWs

1996 – SFE talk + 3 posters
1998 – PLE & CE talk + 3 posters
2000 – DSI-GC/ITD talk + 2 posters
2002 – QuEChERS talk + 3 posters
2004 – MS Identification talk + 4 posters
2006
2008
2010
2012 – MMM talk + 0 posters

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QuEChERS/d-SPE Applications

Application	Publications
Pesticides	≈ 330
Veterinary drugs	≈ 50
Environmental (*e.g.* PAHs)	≈ 25
Mycotoxins	≈ 25
Natural Products (*e.g.* alkaloids)	≈ 20
Reviews	≈ 10
Other	≈ 15

(*e.g.* BPA, acrylamide, PFOS/PFOA, nerve agents, phthalates, dyes/ink, sildenafil, melamine, seafood toxins, Cu)

Matrices

Fruits/Vegetables, Grains, Processed Foods (*e.g.* baby food), Honey, Nuts, Animal Tissues (meat, liver, kidney), Fish/Seafood, Milk, (Vegetable) Oils, Soil, Dried Fruits, Feeds, Eggs, Juices (*e.g.* sugar cane), Tea, Wine/Beer, Plants, Mushrooms, Blood, Water, Cactus, Compost, Roots, Tobacco, Dietary Supplements, Chinese Medicines

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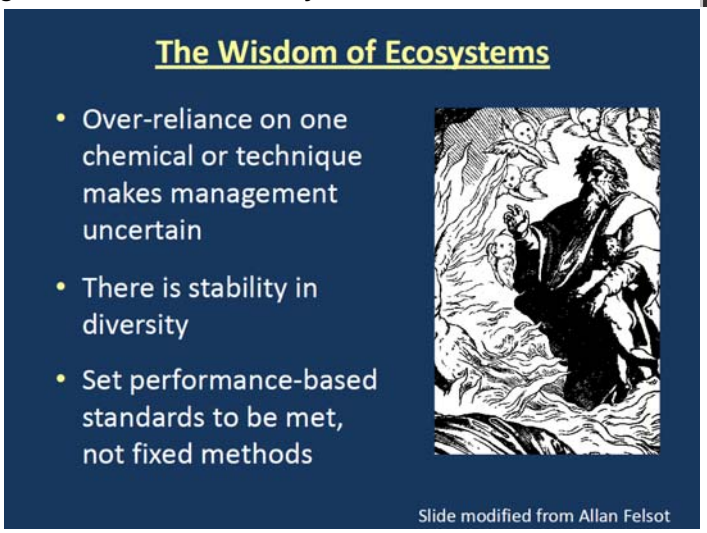

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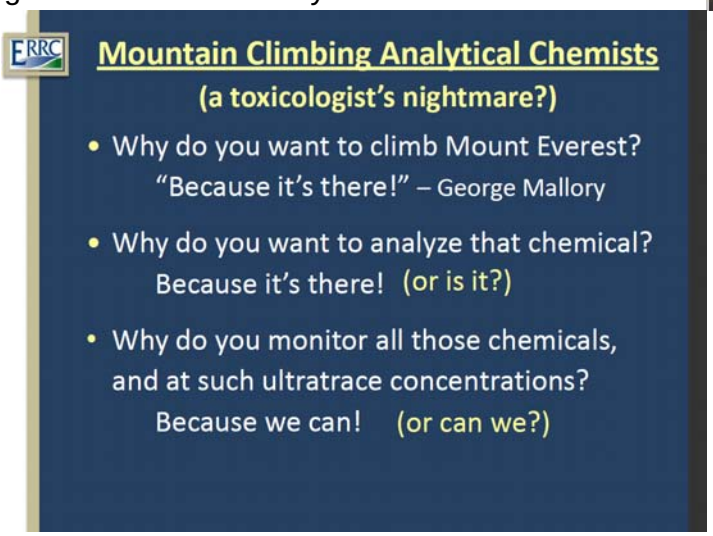

Steven J. Lehotay

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The Situation with Chemical Residues

- The issue of pesticides and other residues in food is not a pressing health priority, but it must be done to enforce laws, conduct trade, provide data, and protect consumers and the environment.
- If you are going to do the analyses, then do it well enough to meet those needs, but costs should be kept as low as possible.
- All is a waste of money and effort if the results don’t meet real needs.

Steven J. Lehotay

MMM means multiclass, multiresidue method, which accommodateds “more more more”, but when is “more” enough? –Steven J. Lehotay

What is the point of residue analysis?

The point is not to create steady jobs for chemists or income for laboratories and instrument companies.

The point is Environmental Protection, Health, and Food Safety/Security

Chemical residue analysis costs money, and adds value if done to suit real needs, but the cost should not exceed this value.

As Always, Today’s Analytical Challenge is to Increase the Benefit/Cost Ratio

Steven J. Lehotay

MMM means multiclass, multiresidue method, which accommodateds “more more more”, but when is “more” enough? –Steven J. Lehotay

Mega-Monitoring Methods (MMMs)

- Mol *et al, Anal. Chem.* (2008) showed that many pesticides, veterinary drugs, mycotoxins, and other chemicals can be monitored in foods/feed by the same method.

MMM =

Mini-Mol Method for pesticide residues and

Mega-Mol Method

for chemicals of concern in foods

Steven J. Lehotay

MMM means multiclass, multiresidue method, which accommodateds “more more more”, but when is “more” enough? –Steven J. Lehotay

Being More Efficient

If mega-methods save more time, money, and effort, then they have more value. But they are inherently designed to cost less, thus make less profit!

Greater efficiency is considered a good thing to which we have to adapt.

We’ll be out of a job if we do “more more more” about “less less less.”

Don’t turn business into busyness – we all try to do something worthwhile, but we all have to eat, too.

Steven J. Lehotay

MMM means multiclass, multiresidue method, which accommodateds “more more more”, but when is “more” enough? –Steven J. Lehotay

Conclusions

- Conclusions aren’t valid unless there is enough experimental and/or observational statistical confidence (>99.9%?) to support them.
- Sometimes more is too much.
  Sometimes more is not enough.
  Sometimes less is more.
- Simplify, not complify. (“Bushism”)

Steven J. Lehotay

MMM means multiclass, multiresidue method, which accommodateds “more more more”, but when is “more” enough? –Steven J. Lehotay

Conclusions?

Growth is not always economic! Progress can be made without MORE MORE MORE, and in fact, most of us are growing as we are slowly dying.

If we’re growing wiser, let’s show it by not doing what we can do, but let’s do what we should do.

I have hope for the next generation – with values like, “Don’t be evil” - things are getting better over time. Try to do your part as you pass your time.

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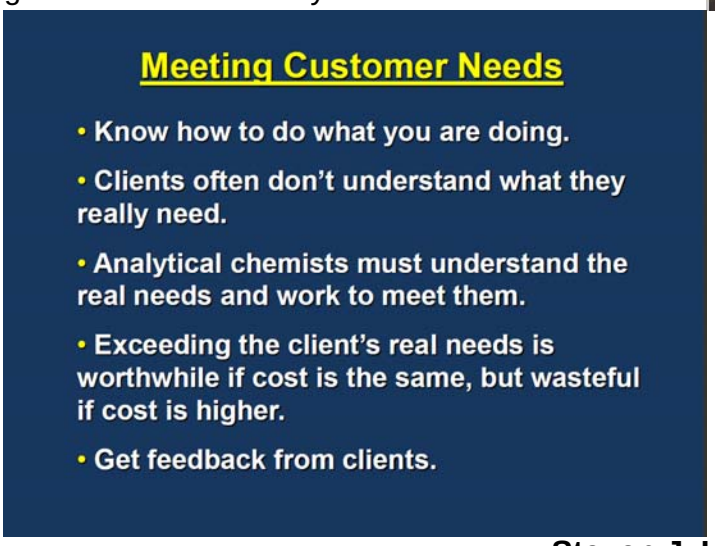
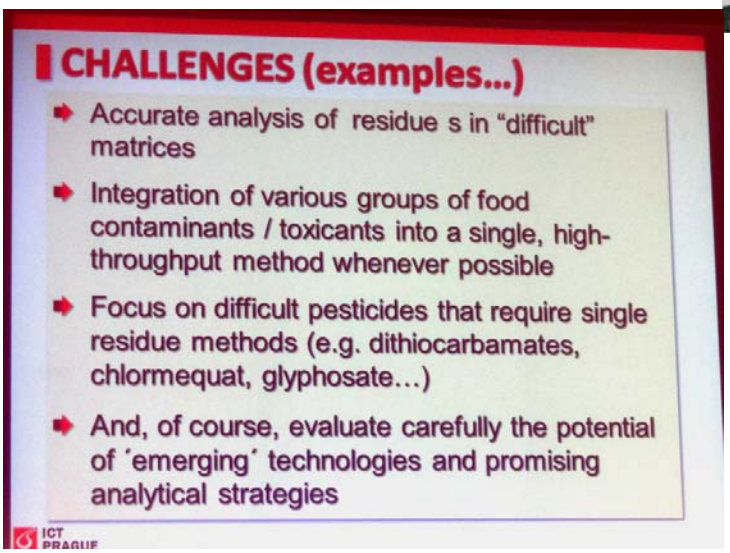


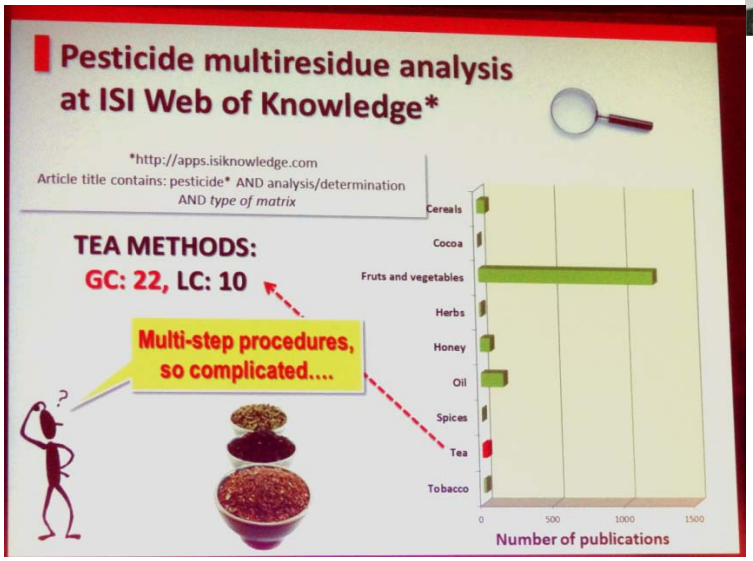
Steven J. Lehotay

Current challenges in the analysis of pesticide residues



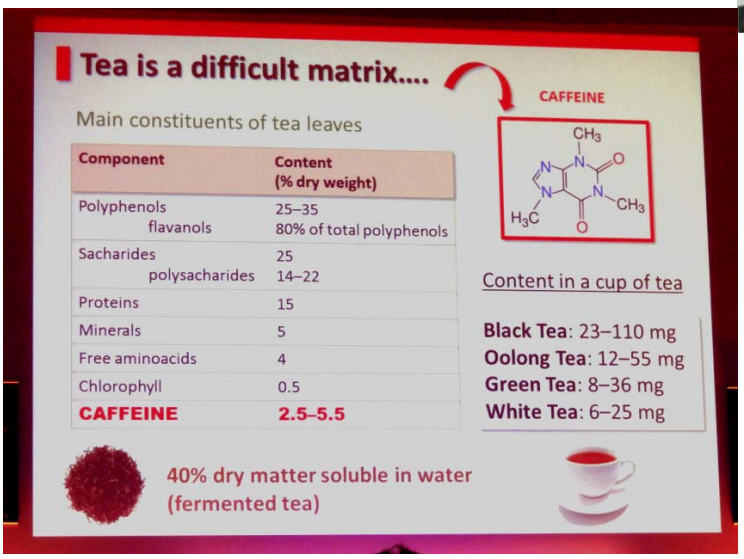
Jana Hajslova

Current challenges in the analysis of pesticide residues



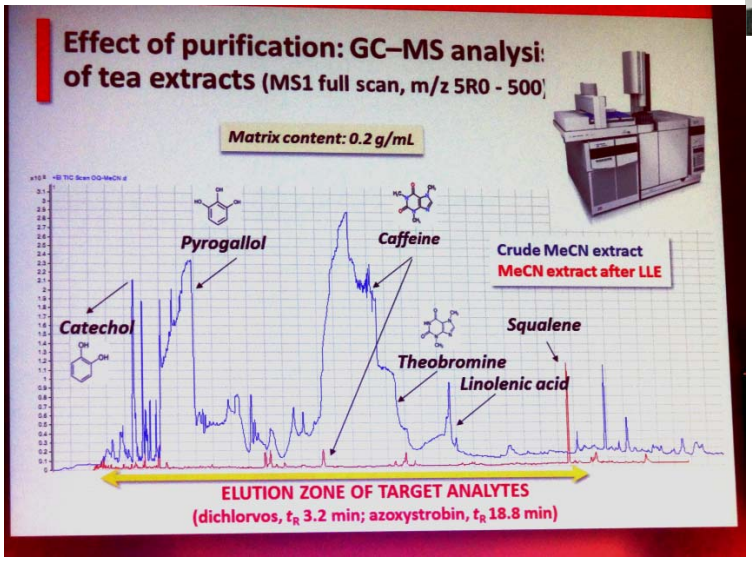
Jana Hajslova

Current challenges in the analysis of pesticide residues



Jana Hajslova

Current challenges in the analysis of pesticide residues



Jana Hajslova

Current challenges in the analysis of pesticide residues

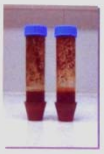
SANCO/10684/2009 strictly advises to use water for swelling the matrix before the extraction

- However, recently, studies dealing with the extraction of pesticide residues from tea using pure MeCN without previous addition of water for matrix hydratation reported

G.F. Pang, *et al.*, *J. AOAC Int.* **94** (2011) 1253–1296

X.M. Xu, *et al.*, *J. Sep. Sci.* **34** (2011) 210–216

Z. Huang *et al.*, *J. Sep. Sci.* **32** (2009) 1294–1301



METHOD A (WITH WATER)	Method B (without water)
2 g sample + 10 mL distilled water	5 g sample + 15 mL MeCN
matrix swelling for 30 min	Turrax for 1 min
10 mL MeCN	centrifuge 5 min at 10,000 rpm
shake for 1 min	MeCN into 25 mL vol. flask
4 g $MgSO_4$, 1 g NaCl	repeat with 15 mL MeCN
shake for 1 min	combine MeCN extracts (25 mL)
centrifuge 5 min at 10,000 rpm	

1 mL of MeCN per 1 mL hexane and 5 mL 20% NaCl solution,
shake for 1 min, centrifuge 1 min at 10,000 rpm
hexane layer into a vial for GC–MS/MS analysis

Jana Hajslova

Current challenges in the analysis of pesticide residues

Which of the existing extraction strategies provides the best performance parameters?

- *Five matrices (different content of moisture, starch, fat, essential oils...)*
- *Three sample preparation procedures*
- *≈ 300 pesticides & ≈ 50 mycotoxins*

- Apples
- Wheat (cereals)
- Sunflower seeds
- Black pepper
- Paprika

„QuEChERS" – Method A [1]
H_2O:MeCN (1:1, v/v), shaking, solvent partition

„Dilute & shoot" – Method B [2]
H_2O:MeCN (1:3, v/v), shaking and sonication

Spice method – Method [3]
Pure MeCN, sonication

UHPLC–ESI(+)–MS/MS

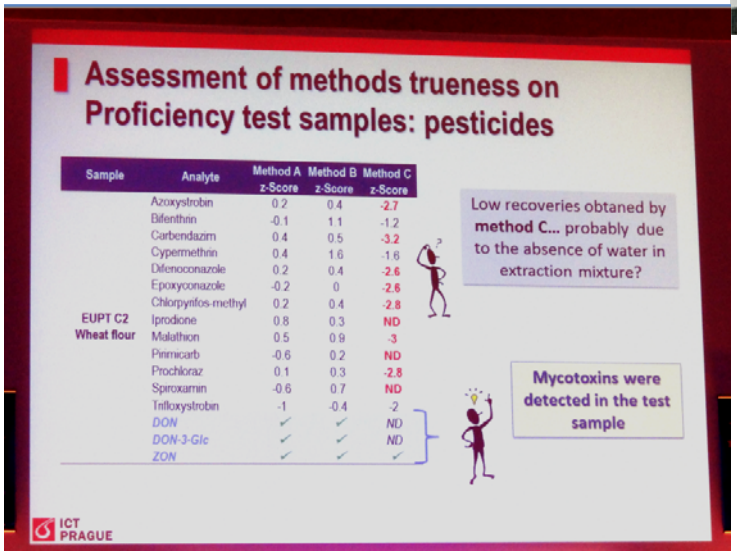
UHPLC–ESI(-)–MS/MS

(1) M. Anastassiades *et al.*, J. AOAC Int. 86 (2003) 412
(2) H.G.J. Mol *et al.*, Anal. Chem. 80 (24) (2008) 9450
(3) C.F. Amate *et al.*, Anal. Bioanal. Chem. 397 (2010) 93

ICT PRAGUE

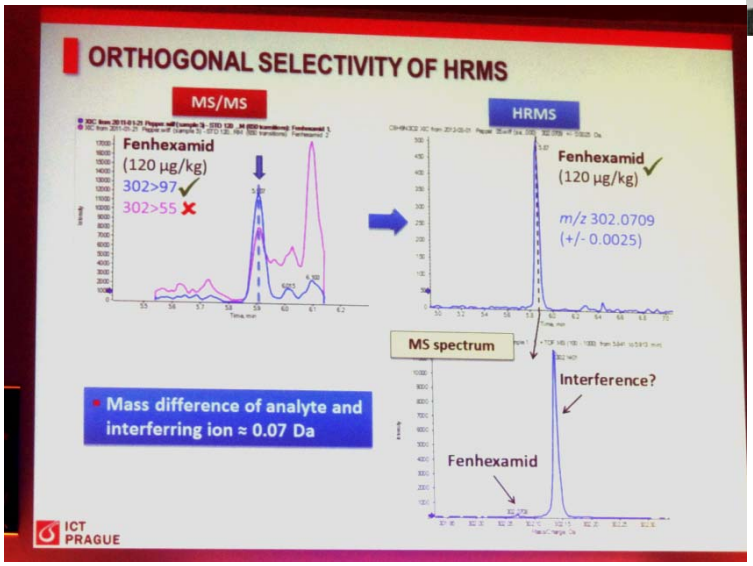
Jana Hajslova

Current challenges in the analysis of pesticide residues



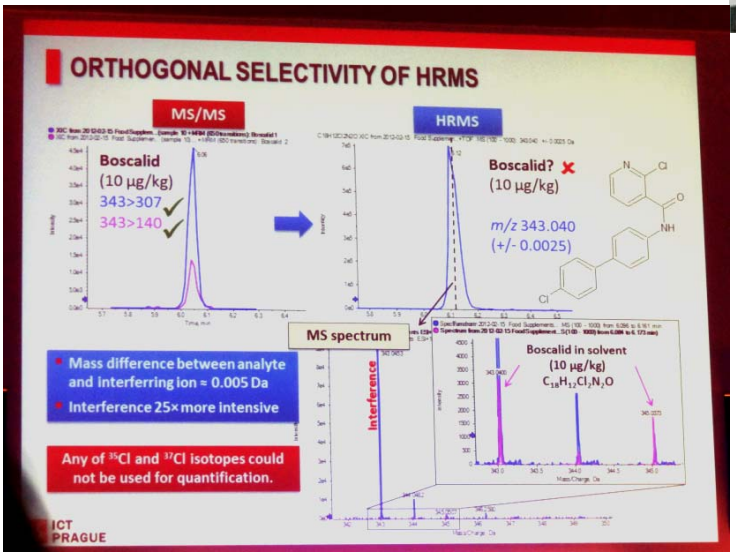
Jana Hajslova

Current challenges in the analysis of pesticide residues



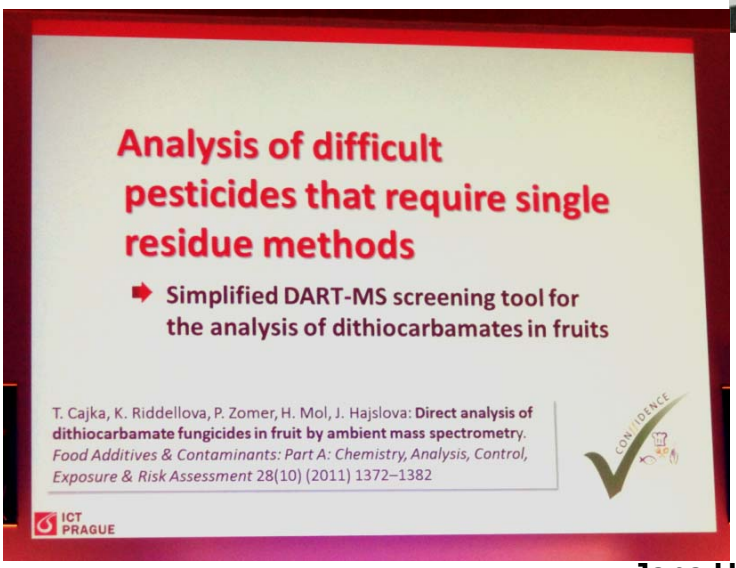
Jana Hajslova

Current challenges in the analysis of pesticide residues



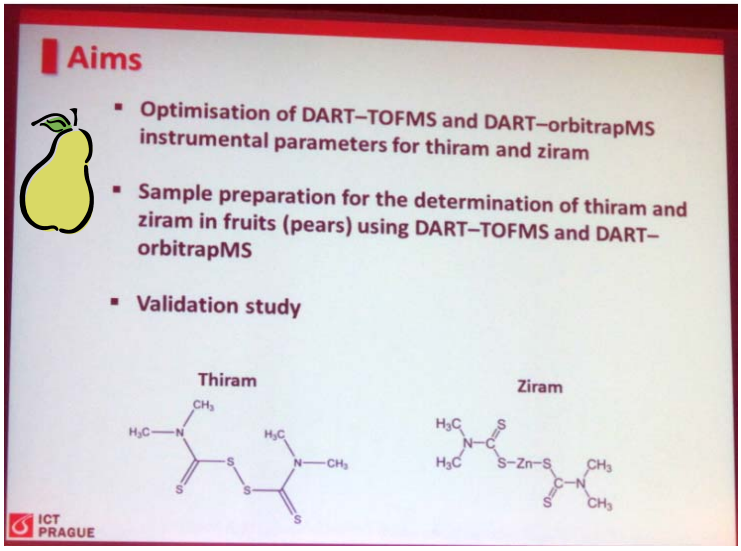
Jana Hajslova

Current challenges in the analysis of pesticide residues



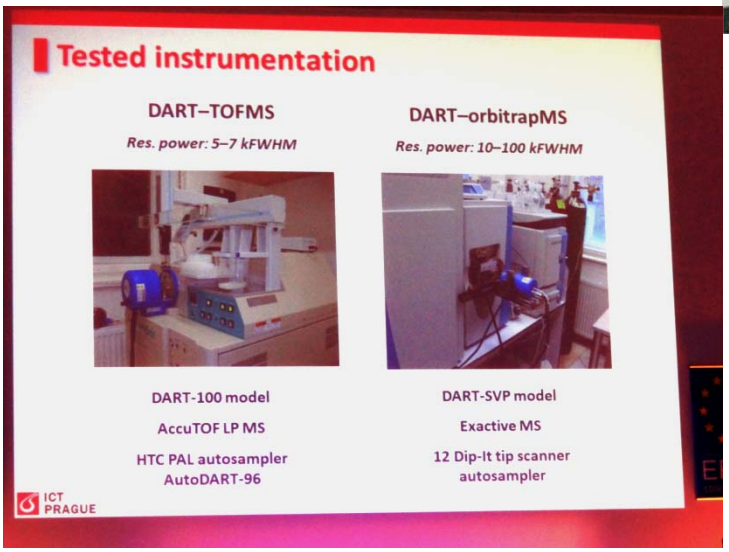
Jana Hajslova

Current challenges in the analysis of pesticide residues



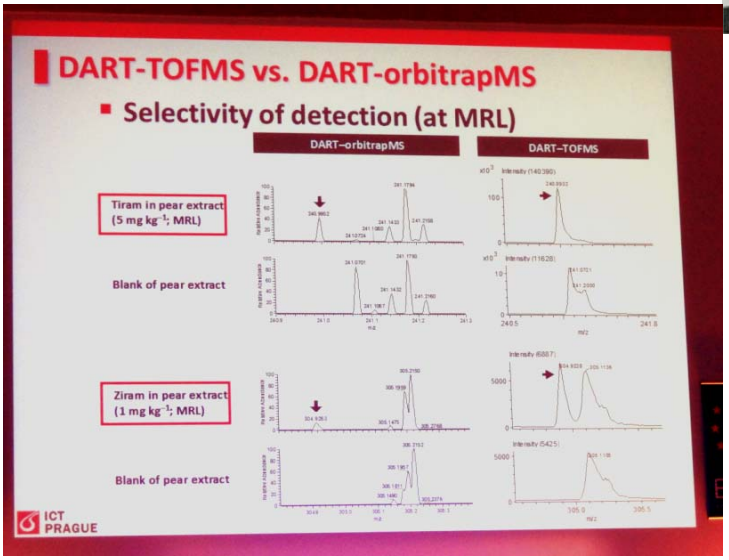
Jana Hajslova

Current challenges in the analysis of pesticide residues



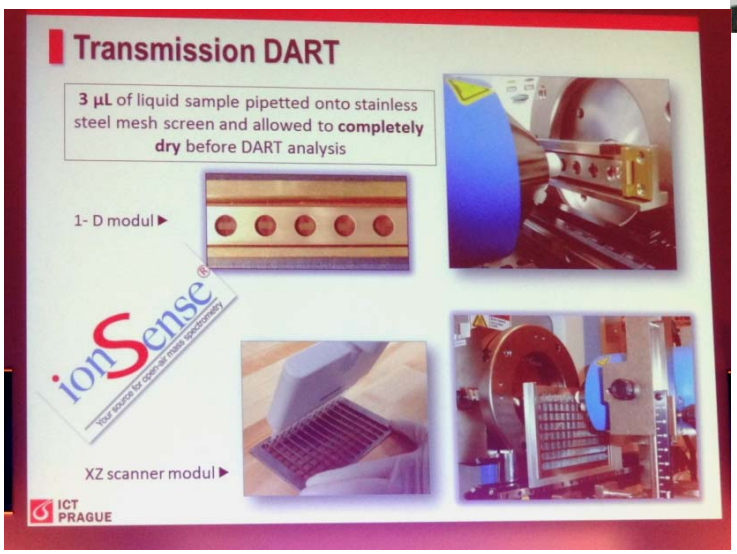
Jana Hajslova

Current challenges in the analysis of pesticide residues



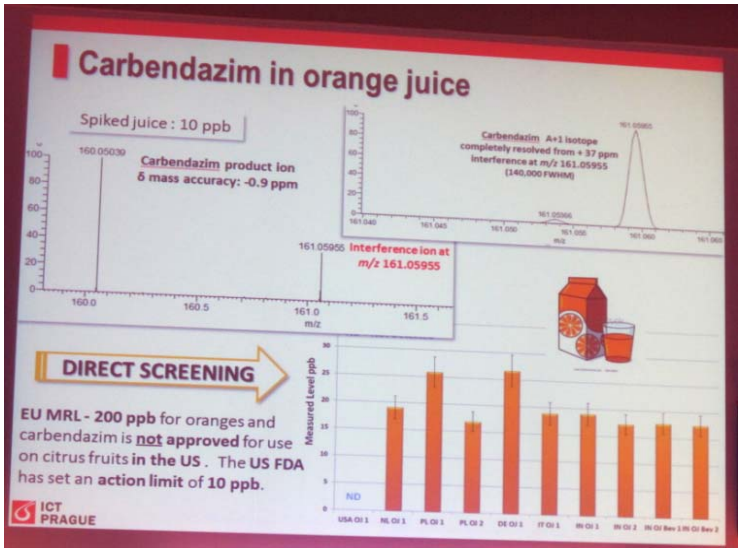
Jana Hajslova

Current challenges in the analysis of pesticide residues



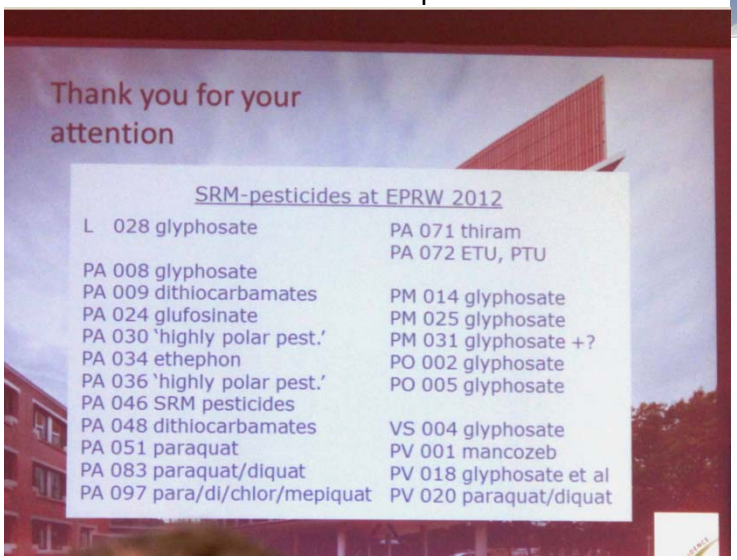
Jana Hajslova

Current challenges in the analysis of pesticide residues



Jana Hajslova

Flow injection – MS for rapid screening of pesticides not amenable to multi-residue methods: potential and limitations



Hans Mol

POSTER PRESENTATIONS

1. Animal
2. Polar pesticides
3. Special or interesting

PA023

Optimization of fat extraction for multiresidue pesticide analysis from meat samples

- Max 0.5g fat➔
- Extraction with ACN/acetone 2*3mL, centrifugation (4 min, 3500rpm), 15 min on ice➔
- Clean-up supernatant on a dispersive SPE tube (PSA:C18), centrifugation (4min, 3500rpm)➔
- GC/MS/MS, LC-QTOF

Kati Hakala and Riina Iitiä
Chemistry and Toxicology Research Unit, Evira, Helsinki, Finland

PA023

Optimization of fat extraction for multiresidue pesticide analysis from meat samples

Table 1. Optimization of fat extraction method. Samples do not contain pesticides. Planar mixer A = Edmund Bühler 7400 Tübingen KS 10, B = Heidolph Unimax 1000, Heidolph Incubator 1000

	Sample weight (g)	Extraction Volume (ml) Extraction Solvent: Hexane:Acetone (2:1)	Mixing equipment	FAT CONTENT (%) Swine meat (≈ 23% fat)	Poultry meat (≈ 7% fat)	Swine meat reference material* (20.51±0.08)
Method 1 (used before optimization)	8 (n=4)	10+10	Planar A	14.2	4.6	
Method 2	8 (n=4)	20+20	Planar A	18.3	5.9	
Method 3	8 (n=4)	10+10	Water bath , 45 °C	17.6	4.7	
Method 4	8 (n=4)	10+10	Planar B, 45 C	20.5	3.1	
Method 5	8 (n=4)	20	Planar B, 45 °C	17.6		
Method 6	8 (n=4)	20+20	Planar B, 45 °C	21.1	5.7	
Method 7	5 (n=4)	20+20	Planar B, 45 °C	23.1		18.0
Method 8	Standardized fat determination method [3] used in our laboratory for meat samples (n=2)			24.8±1.51	6.4±1.51	

*provided by EURL for pesticides in food of animal origin, contain no pesticides

Two proficiency test samples containing pesticides (EUPT-AO-05, EUPT AO-06) were re-analyzed by using optimized method for the extraction of fat (Tables 2-4).

Kati Hakala and Riina Iitiä
Chemistry and Toxicology Research Unit, Evira, Helsinki, Finland

PA023

Optimization of fat extraction for multiresidue pesticide analysis from meat samples

Table 3. Results for the re-analysed proficiency test sample (swine meat)

Compound	Concentration of pesticides µg/kg fat		
	Our result, new fat extraction	Median (EUPT-AO-05) [4]	Standard deviation (EUPT-AO-05)[4] µg/kg fat, coefficient of variation 25%
alpha-HCH	172	140	35
beta-HCH	232	201	50.2
diazinon	380	387	96.8
pirimiphosmethyl	1118	1104	279
alpha-endosulfan	301	307	76.8
DDE	252	252	62.9
beta-endosulfan	206	219	54.8
triazophos	286	285	71.3
cypermetrin	443	417	102
deltametrin	1151	1043	261

Kati Hakala and Riina Iitiä
Chemistry and Toxicology Research Unit, Evira, Helsinki, Finland

PA023

Optimization of fat extraction for multiresidue pesticide analysis from meat samples

Table 4. Results for the re-analysed proficiency test sample (poultry meat)

	Concentration of pesticides µg/kg meat		
Compound	Our result, new fat extraction	Median (EUPT-AO-06) [5]	Standard deviation (EUPT-AO-06)[5] ug/kg, coefficient of variation 25%
cis-chlordane	11.2	14	3.5
chlorpyriphos-ethyl	91.8	79	19.8
chlorpyriphos-methyl	14.7	13.3	3.33
cyfluthrin	199	124	31.0
TDE	38.1	39	9.75
diazinon	40.5	43.9	11.0
deltamethrin	81.5	88.2	22.1
fenvalerate	80.7	101.7	25.4
pirimiphos-methyl	48.4	37.9	9.48

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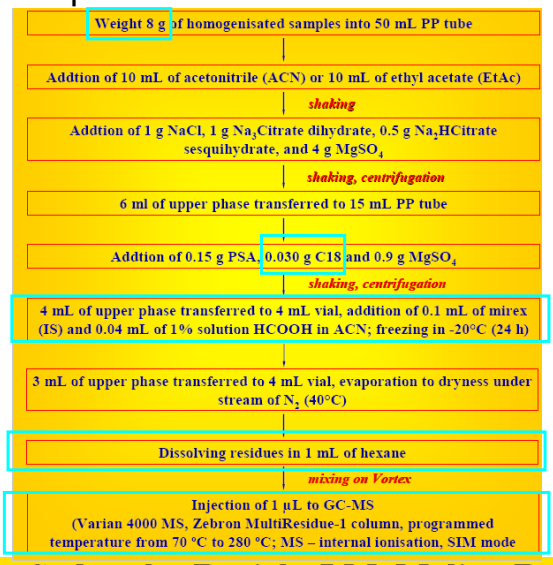
PA053

Application of QuEChERS method for the determination of organochlorine pesticide residues in food of animal origin

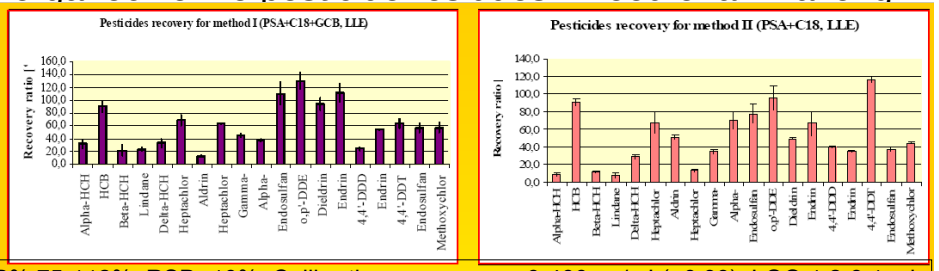


EN15662

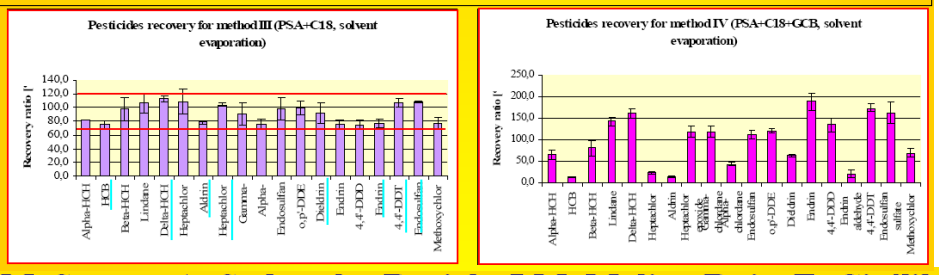
M. Surma, A. Sadowska-Rociek, J.M. Molina Ruiz, E. Cieślik
Malopolska Centre of Food Monitoring, Faculty of Food Technology, University of Agriculture in Krakow, Balicka 122, 30-149 Krakow, Poland

PA053

Application of QuEChERS method for the determination of organochlorine pesticide residues in food of animal origin



R%:75-112%; RSD<10%; Calibration curve range:0-400 ng/mL(>0.99); LOQ:1.2-3.1ppb



M. Surma, A. Sadowska-Rociek, J.M. Molina Ruiz, E. Cieślik

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PA055

Evaluation of different sample treatments for determination of pesticide residues in chicken liver samples by QuEChERS method and gas chromatography-mass spectrometry

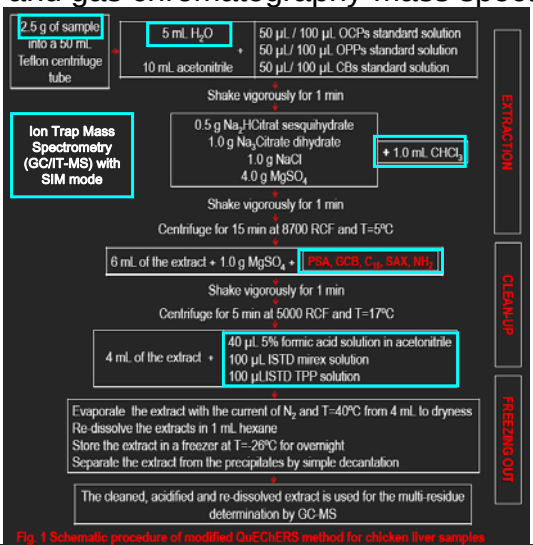


Fig. 1 Schematic procedure of modified QuEChERS method for chicken liver samples

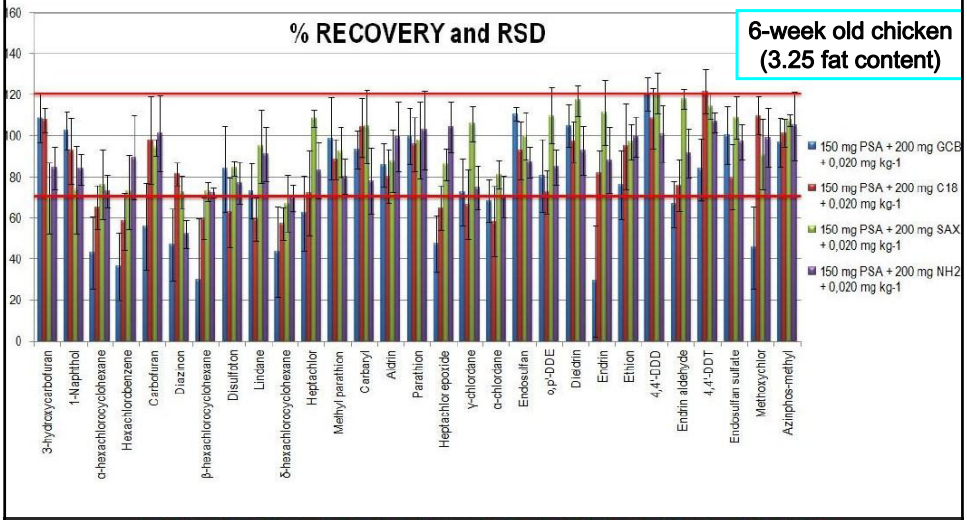
J. M. Molina-Ruiz[1], E. Cieslik[1], I. Walkowska[1], A. Sadowska-Rociek[1], W. Migdal[2]

[1]Malopolska Centre of Food Monitoring, University of Agriculture in Krakow, 122 Balicka St, 30-149, Krakow, Poland

[2]Department of Animal Products Technology, University of Agriculture in Krakow, 122 Balicka St, 30-149, Krakow, Poland

PA055

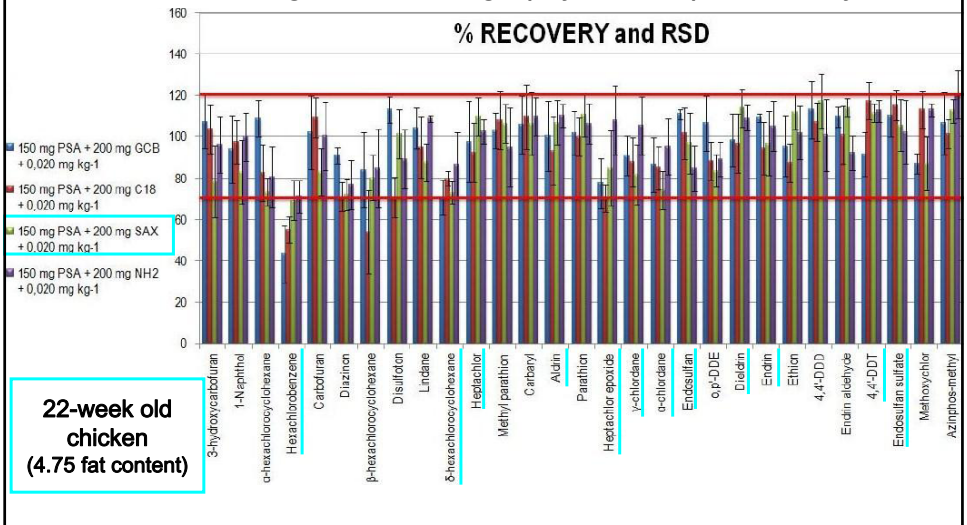
Evaluation of different sample treatments for determination of pesticide residues in chicken liver samples by QuEChERS method and gas chromatography-mass spectrometry



J. M. Molina-Ruiz[1], E. Cieslik[1], I. Walkowska[1], A. Sadowska-Rociek[1], W. Migdal[2]
[1]Malopolska Centre of Food Monitoring, University of Agriculture in Krakow, 122 Balicka St, 30-149, Krakow, Poland
[2]Department of Animal Products Technology, University of Agriculture in Krakow, 122 Balicka St, 30-149, Krakow, Poland

PA055

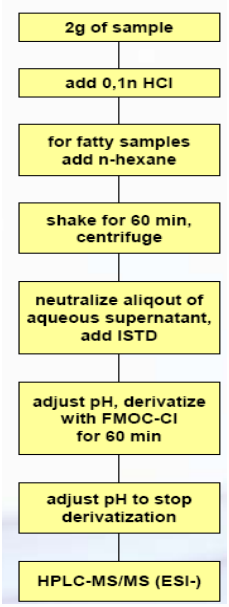
Evaluation of different sample treatments for determination of pesticide residues in chicken liver samples by QuEChERS method and gas chromatography-mass spectrometry



J. M. Molina-Ruiz[1], E. Cieslik[1], I. Walkowska[1], A. Sadowska-Rociek[1], W. Migdal[2]
[1]Malopolska Centre of Food Monitoring, University of Agriculture in Krakow, 122 Balicka St, 30-149, Krakow, Poland
[2]Department of Animal Products Technology, University of Agriculture in Krakow, 122 Balicka St, 30-149, Krakow, Poland

PA008

Analysis of glyphosate, AMPA and glufosinate in lentils, oil seeds, wheat and tea: method validation and analytical results



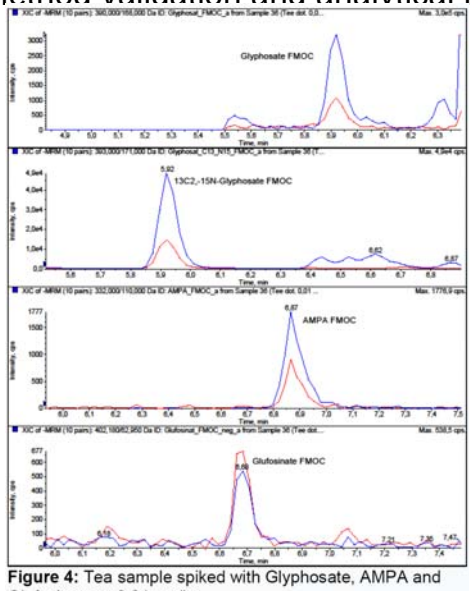


Figure 4: Tea sample spiked with Glyphosate, AMPA and Glufosinate at 0.01 mg/kg

Brockmeyer, R.; Lipinski, J.; SOFIA GmbH, Rudower Chaussee 29, 12489 Berlin

PA008

Analysis of glyphosate, AMPA and glufosinate in lentils, oil seeds, wheat and tea: method validation and analytical results

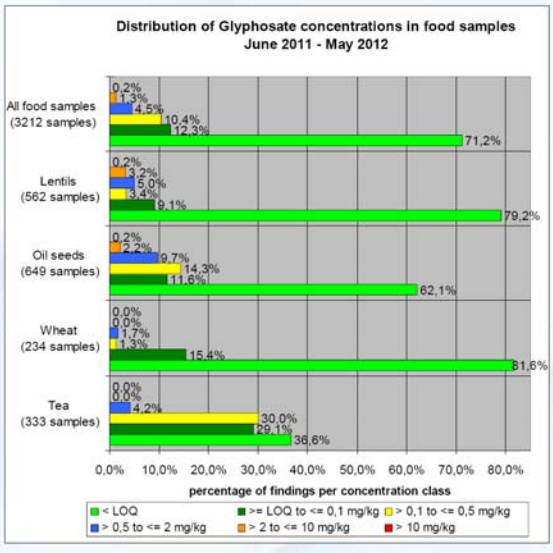


Figure 6: Distribution of Glyphosate concentrations in food

Brockmeyer, R.; Lipinski, J.; SOFIA GmbH, Rudower Chaussee 29, 12489 Berlin

EU Reference Laboratories for **Residues of Pesticides**

Single Residue Methods

Quick Method for the Analysis of Residues of Highly Polar Pesticides in Foods of Plant Origin Involving Simultaneous Extraction with Methanol and LC-MS/MS Determination (QuPPe-Method)

- **Version 6** (Aug 2011, Document History, see page 37)

Authors: M. Anastassiades; D. I. Kolberg; D. Mack; I. Sigalova; D. Roux; D. Fügel

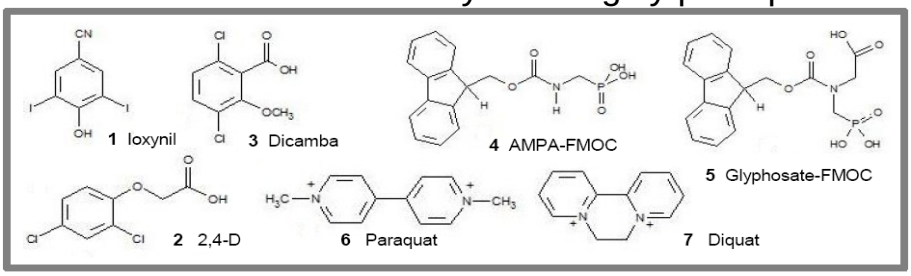
1. Scope and Short Description

A method is described for the residue analysis of very polar, non-QuEChERS-amenable, pesticides in foods of plant origin such as fruits (including dried fruits), vegetables, cereals and processed products thereof as well as honey.

Residues are extracted from the test portion following water adjustment and the addition of acidified methanol. The mixture is centrifuged, filtered and directly analyzed by LC-MS/MS. Various options for the simultaneous LC-MS/MS analysis of different combinations of pesticides are provided. Quantification is in most cases performed with the help of isotopically labeled analogues of the target analytes, which are used as internal standards (IL-ISTDs). So far available, these IL-ISTDs are added directly to the test portion at the beginning of the procedure to compensate for any factors having an influence on the recovery-rates such as volume-deviations, analyte losses during the sample preparation as well as matrix-effects during measurement.

PA030

Multi method for residue analysis of highly polar pesticides



A methanol
B methanol - water (1:1)
C ACN - buffer (9:1)
D buffer
E buffer - methanol (8:2)
F buffer - ACN (8:2)

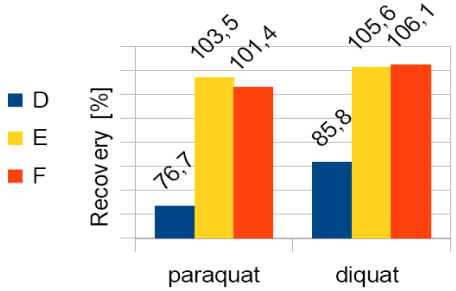


Figure 2: Recoveries of diquat and paraquat from ZIC-HILIC SPE cartridges.

Julia Helbling, Uwe Bohn, Wolfgang Schwack

PA030

Multi method for residue analysis of highly polar pesticides

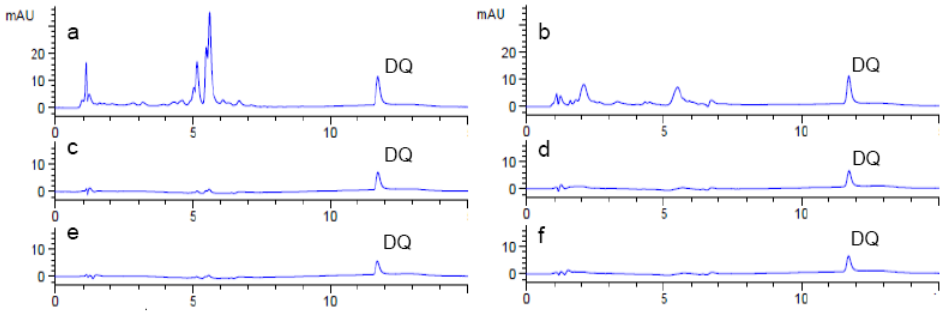


Figure 3: HILIC chromatograms of QuPPe extracts of lentils (left) and apples (right) before (a, b) and after cleanup on ZIC-HILIC cartridges eluted with buffer-methanol (c, d) or buffer-acetonitrile (e, f); UV at 310 nm for diquat (DQ).

Julia Helbling, Uwe Bohn, Wolfgang Schwack

PA006

Automation of the QuEChERS method for pesticide residue analysis

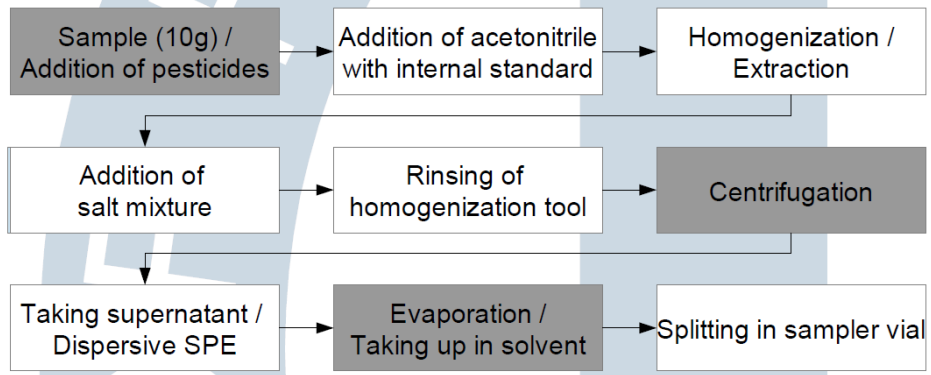


Fig 1: Chart of Extraction steps (grey steps performed manually)

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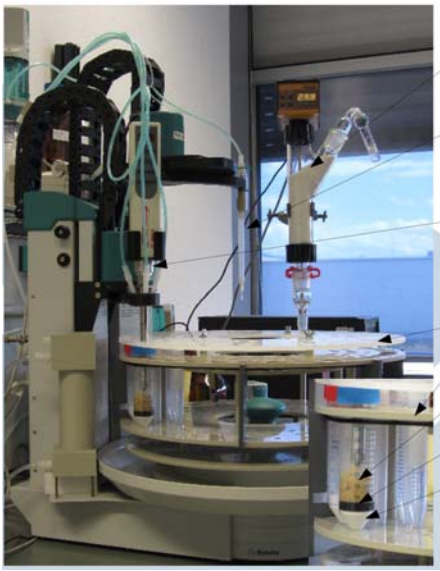
PA006

Automation of the QuEChERS method for pesticide residue analysis



LambdaDoser unit with salt mixture

Fresh DPX tip prior clean-up

Polytron homogenizer

Double bouncing restriction

Stored lemon sample (> 30 days)

Holder for separating film

Preweighted salt mixture

Uwe Bohn, Wolfgang Schwack

University of Hohenheim, Institute of Food Chemistry, Garbenstraße 28, D-70599 Stuttgart, www.ilc.uni-hohenheim.de

PA006

Automation of the QuEChERS method for pesticide residue analysis

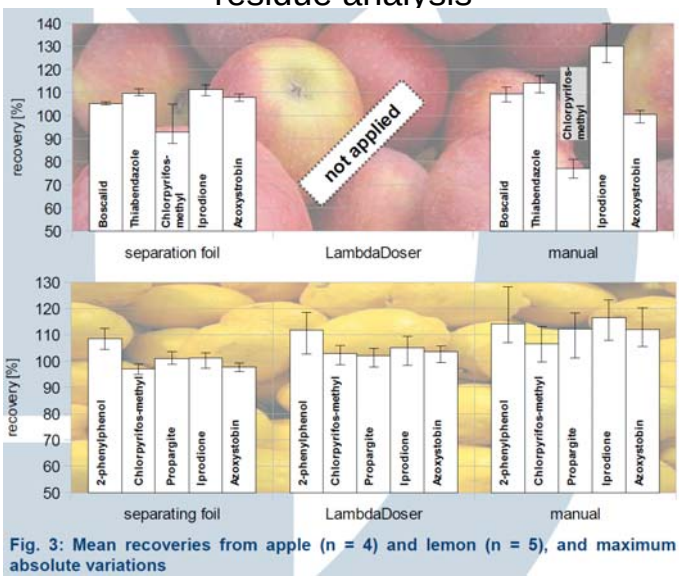


Fig. 3: Mean recoveries from apple (n = 4) and lemon (n = 5), and maximum absolute variations

Uwe Bohn, Wolfgang Schwack

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PA043

Automation of the ChemElut method for pesticide residue analysis

Extraction:

- apple (10 g) + 100 µL pesticide mix (setting for 5 min)
- addition of water to the sample (10 mL total water content)
- addition of methanol with internal standard (20 mL)
- homogenization with a polytron (2 min, 27000 rpm)
- thorough rinsing of the polytron system
- centrifugation (5 min, 3000 rpm)
- taking an aliquot of supernatant (3,75 mL) and mixing with a 20% sodium chloride solution (1,25 mL) directly in a disposable Rezorian cartridge
- applying the aliquot onto a ChemElut cartridge

Clean-up:

- elution with dichlormethane (4 x 6 mL)
- evaporation, taking up in solvent (ethyl acetate) and transfer into autosampler vials

After clean-up the samples were measured by GC-MS.

S. Linder, U. Bohn and W. Schwack
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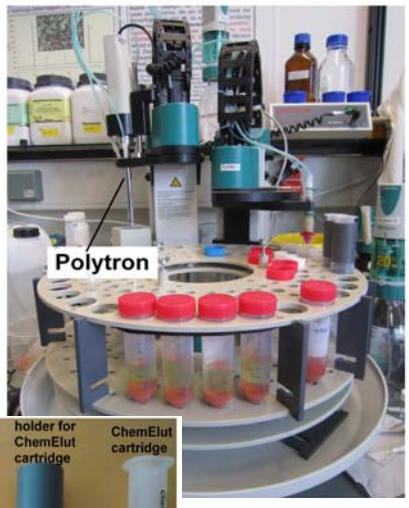
PA043

Automation of the ChemElut method for pesticide residue analysis



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PA043

Automation of the ChemElut method for pesticide residue analysis

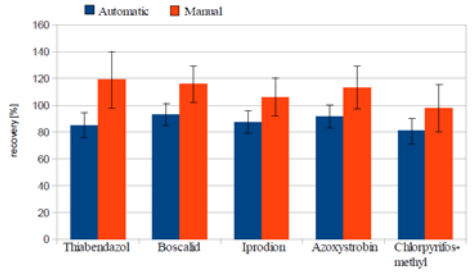

Figure 2 Comparison of recovery (n=5) for apple samples using automatic and manual sample preparation

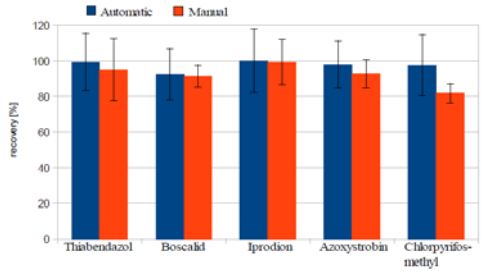

Figure 3 Comparison of recovery (n=5) for tomato samples using automatic and manual sample preparation

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PA007

Analysis of pesticide metabolites in food: Integration into an LC-MS/MS pesticide multiresidue method

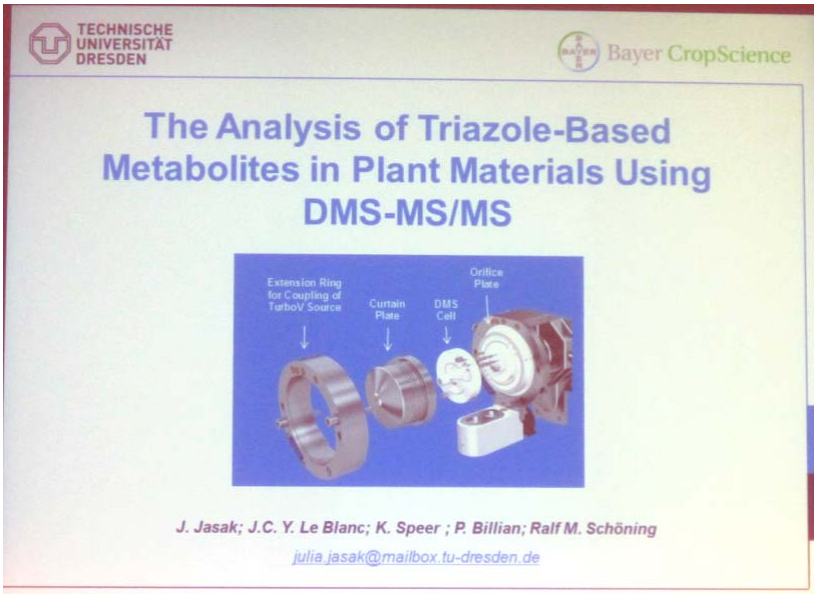
Parent compound	Metabolite	Regulation (EC) No 396/2005	reported LOQ [mg/kg]
Amitraz	2,4-Dimethylaniline	yes	0,01
Amitraz	2,4'-Formoxylidid (2,4-Dimethylphenylformamide)	yes	0,005
Amitraz	N-2,4-Dimethylphenyl-N'-methylformamidine	yes	0,005
Carbendazim	2-Aminobenzimidazole	no	0,01
Carbendazim	Benzimidazole	no	0,005
Dimethenamid	Dimethenamid ESA	no	0,01
Dimethenamid	Dimethenamid OA	no	0,01
Diuron	1-(3,4-Dichlorophenyl)-3-methyl-urea	yes	0,005
Diuron	1-(3,4-Dichlorophenyl)-urea	yes	0,005
Diuron	3,4-Dichloroaniline	yes	0,005
Ethofumesate	Ethofumesate-2-keto	yes	0,01
Fipronil	Fipronil-carboxamide	no	0,005
Fipronil	Fipronil-desulfinyl	no	0,005
Fipronil	Fipronil-sulfide	no	0,005
Fipronil	Fipronil-sulfone	yes	0,005
Fluazifop-p-butyl	Fluazifop	yes	0,005
Flufenacet	Flufenacet ESA	yes	0,01
Flufenacet	Flufenacet OA	yes	0,01
Haloxyfop-Ester	Haloxyfop	yes	0,005
Mepanipyrim	Mepanipyrim-2-hydroxypropyl	yes	0,005
Mepanipyrim	Mepanipyrim-2-oxopropyl	no	0,005
Metalaxyl	Metalaxyl CGA 108906	no	0,005
Metalaxyl	Metalaxyl CGA 62826	no	0,005
Methomyl	Methomyl-sulfone	no	0,005
Methomyl	Methomyl-sulfoxide	no	0,005
Metolachlor	Metolachlor CGA 357704	no	0,005
Metolachlor	Metolachlor CGA 368208	no	0,005
Metolachlor	Metolachlor CGA 37735	no	0,005
Metolachlor	Metolachlor CGA 50720	no	0,005
Metolachlor	Metolachlor ESA	no	0,005
Metolachlor	Metolachlor-mercapturate	no	0,005
Metolachlor	Metolachlor NOA 413173	no	0,01
Metolachlor	Metolachlor OA	no	0,005
Metolachlor	Metolachlor-2-ethoxy	no	0,005
Metolachlor	Metolachlor-2-hydroxy	no	0,005
Metolachlor	Metolachlor-deschloro	no	0,005
Metsulfuron-methyl	2-Amino-4-methoxy-6-methyltriazin	no	0,005
Phosmet	Phosmet-oxon	yes	0,005
Phosmet	Phthalimide	no	0,01
Phosmet	Phthalic acid	no	0,05
Pirimicarb	2-Amino-5,6-dimethyl-4-hydroxypyrimidine	no	0,01
Pirimicarb	Pirimicarb-desamido	no	0,01
Pirimicarb	Pirimicarb-desamido-desmethyl	no	0,05
Quizalofop ester	Quizalofop	yes	0,005
Spirotetramat	Spirotetramat-enol	yes	0,005
Spirotetramat	Spirotetramat-enol-glucoside	yes	0,01
Spirotetramat	Spirotetramat-ketohydroxy	yes	0,005
Spirotetramat	Spirotetramat-monohydroxy	yes	0,005

Brockmeyer, R.(1), Tietz, S.(2), Lipinski, J.(1)

1) SOFIA GmbH, D-12489 Berlin; 2) Beuth Hochschule für Technik, D-13353 Berlin

PA035

Winner



PA031

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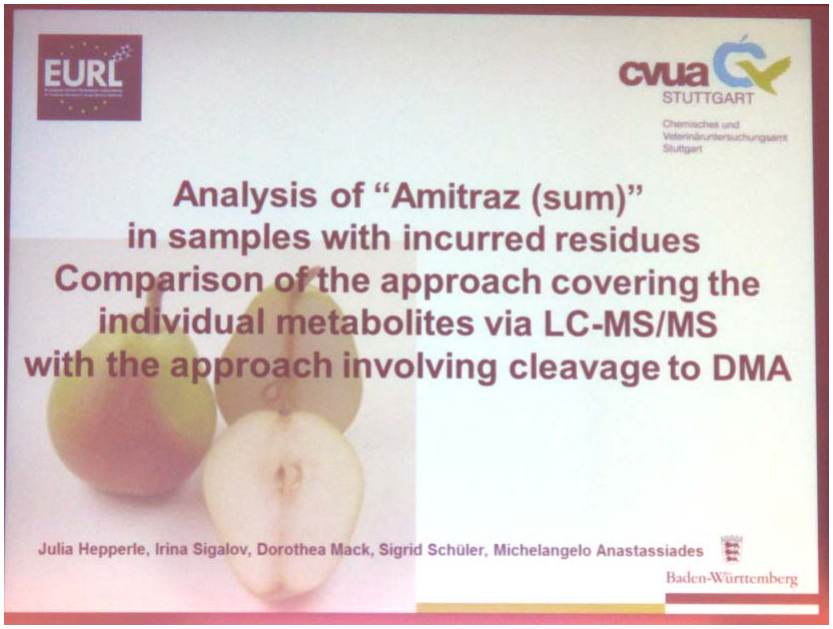

PA031

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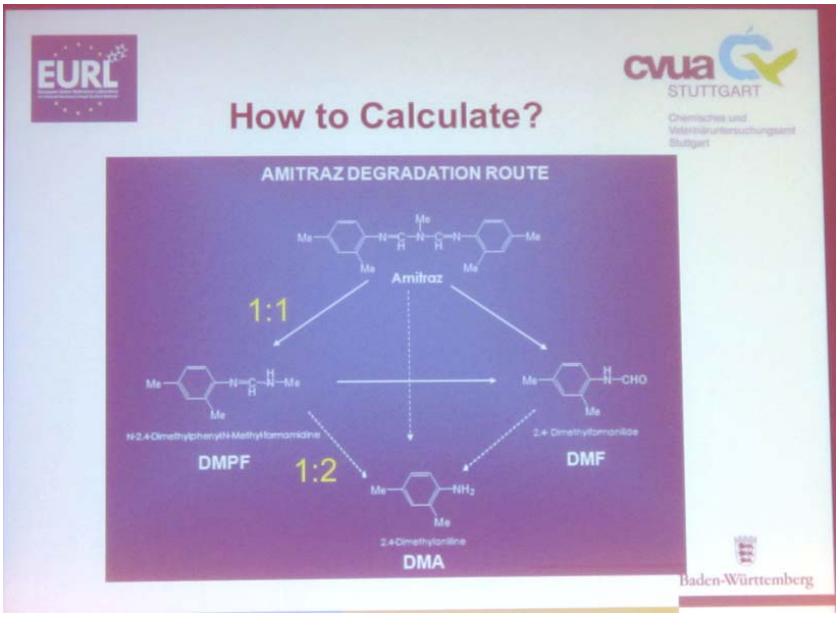

PA031

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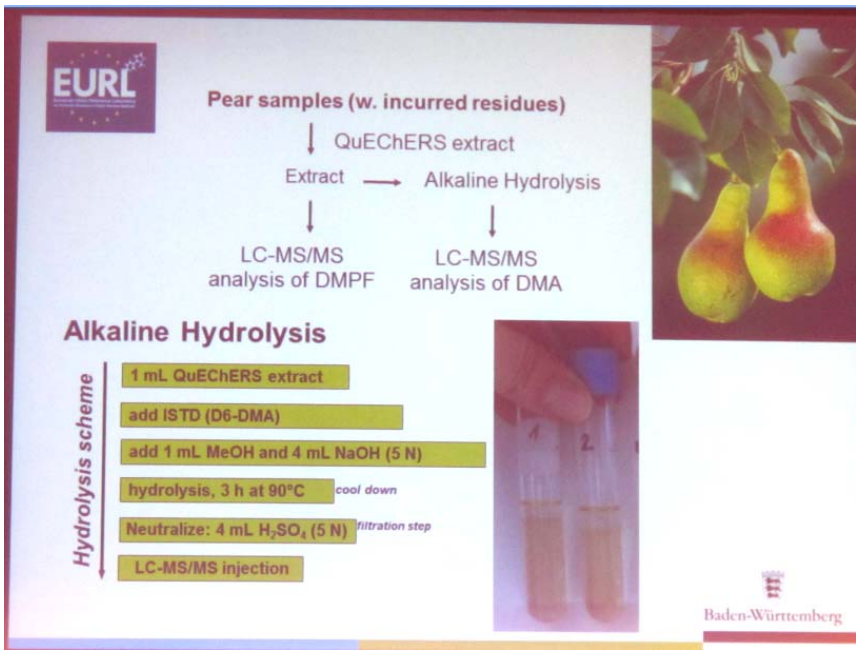

PA031

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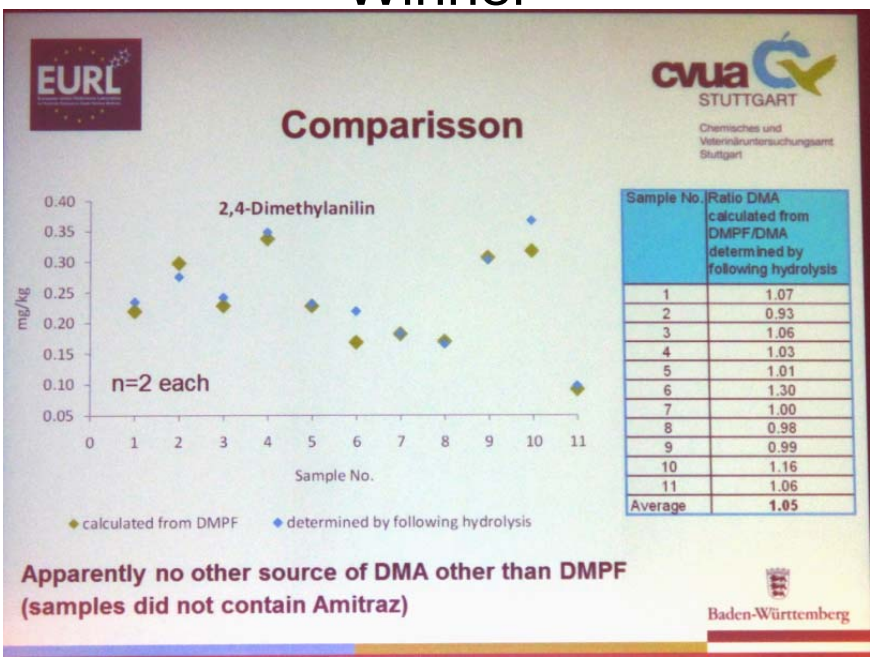

Sample No.	Ratio DMA calculated from DMPF/DMA determined by following hydrolysis
1	1.07
2	0.93
3	1.06
4	1.03
5	1.01
6	1.30
7	1.00
8	0.98
9	0.99
10	1.16
11	1.06
Average	**1.05**

PA031

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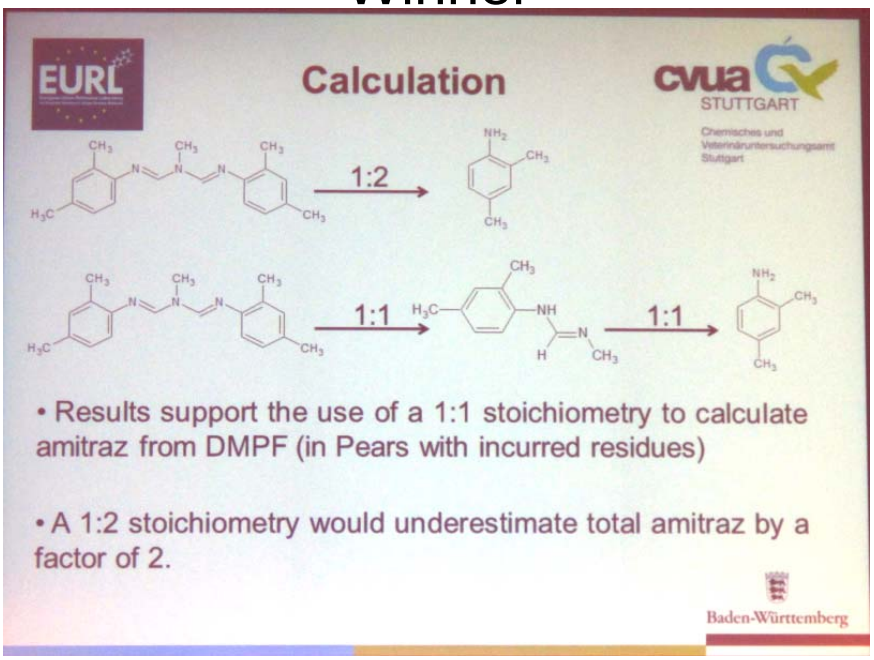

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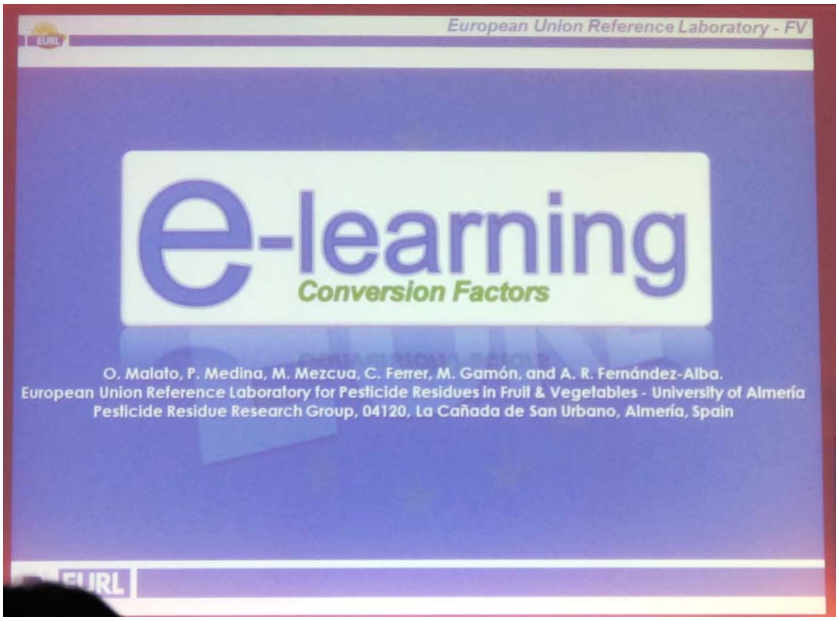

Winner

Winner

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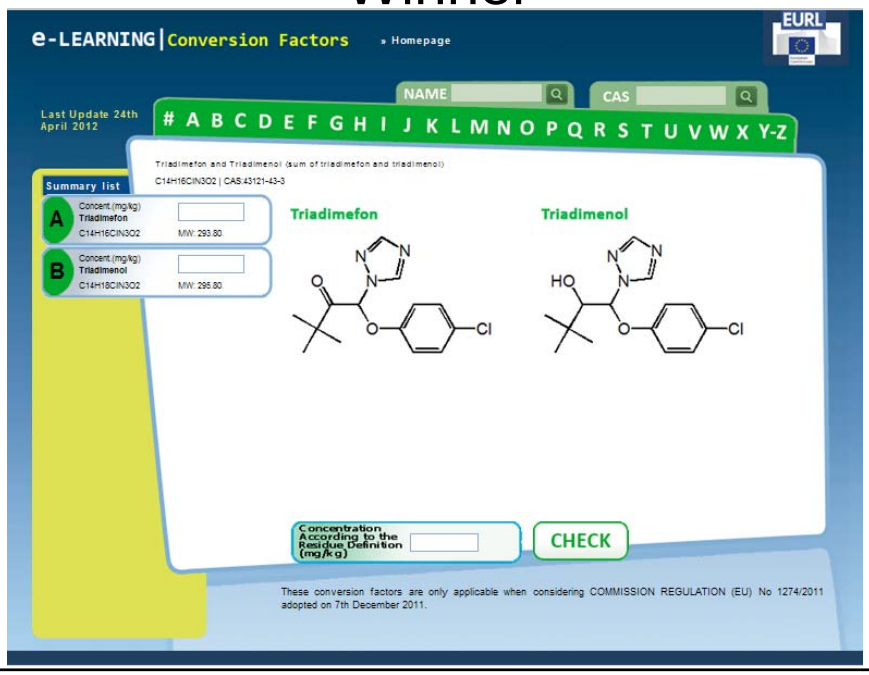

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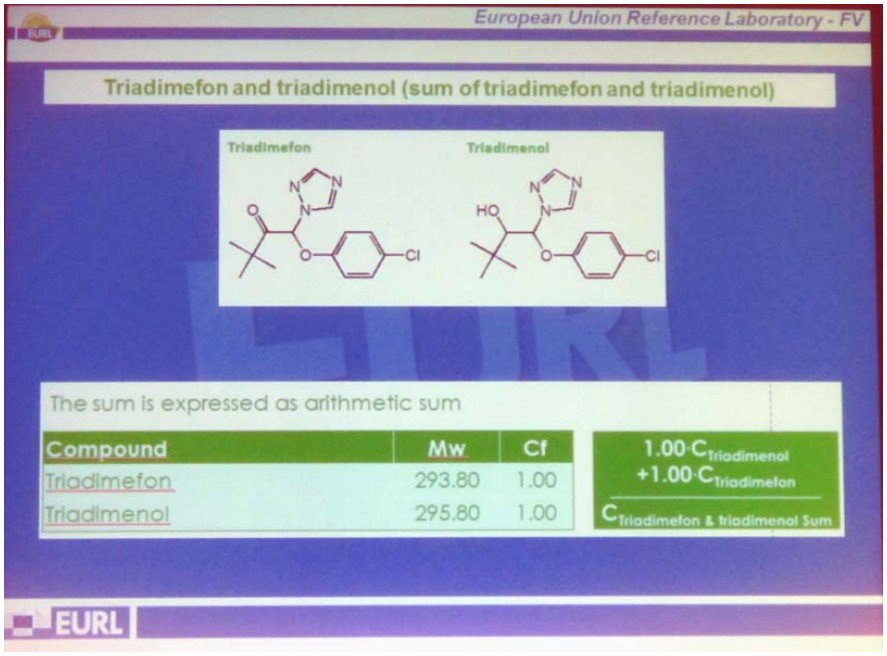

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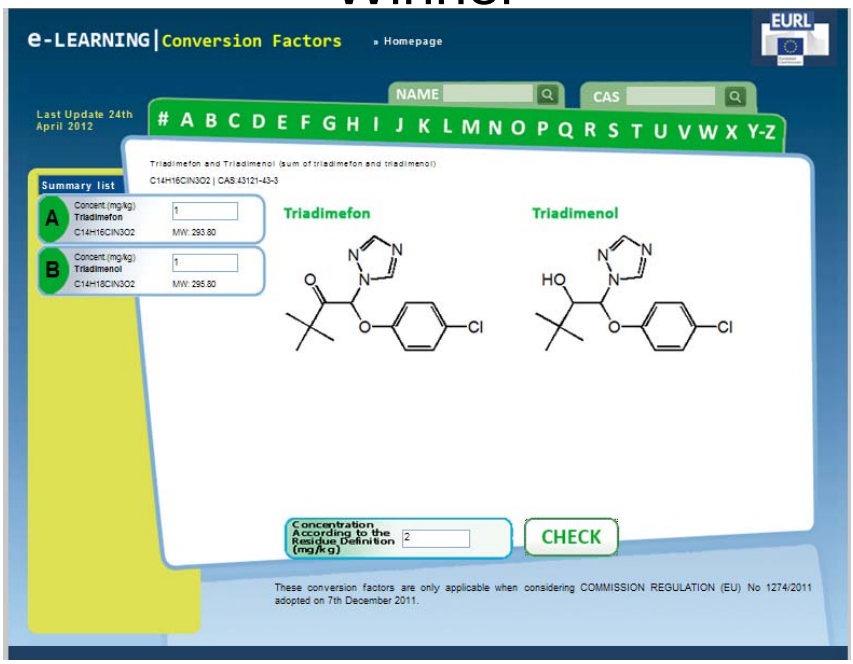

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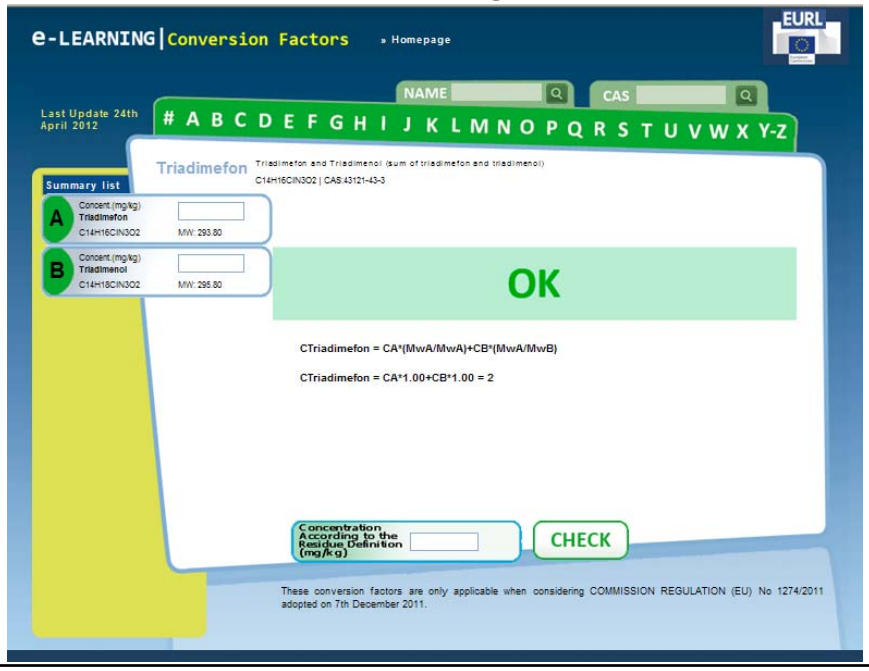

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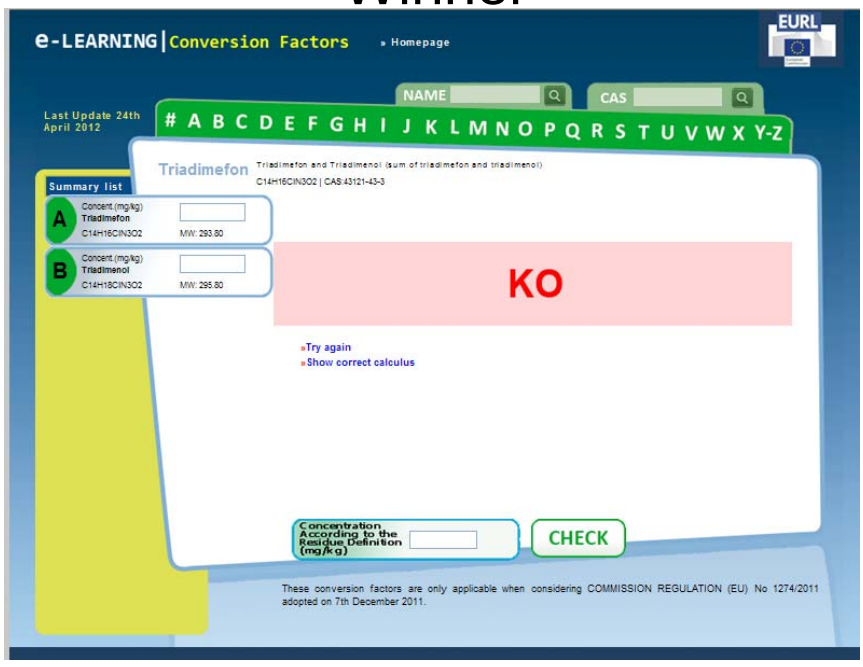

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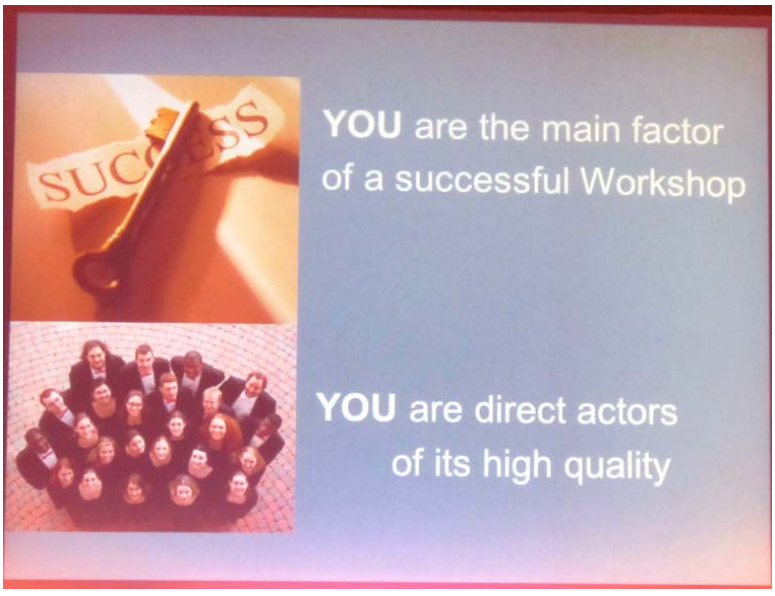
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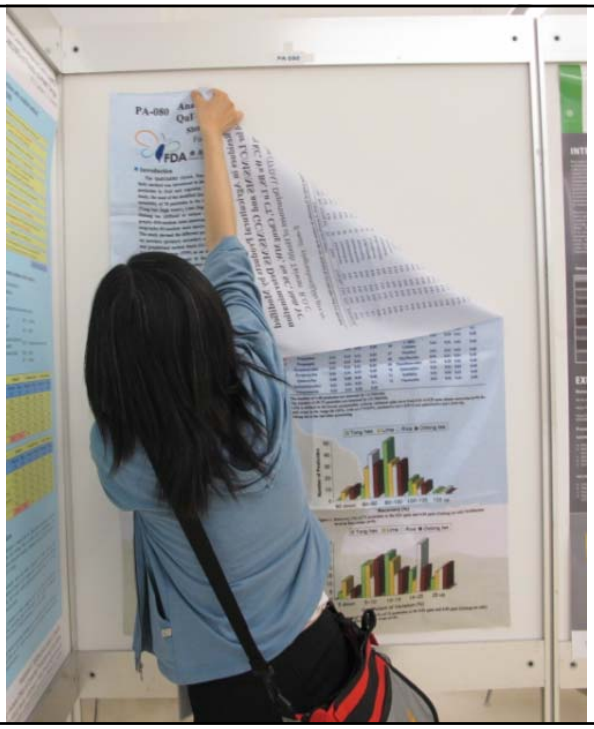
PA-080
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