出國報告（出國類別：其他(國際會議)）

赴美國參加第 133 屆公定分析化學家協會(AOAC)年會暨研討會

服務機關：衛生福利部食品藥物管理署
姓名職稱：施又寧助理研究員、李蕙君技士
派赴國家/地區：美國
出國期間：108 年 9 月 7 日至 108 年 9 月 13 日
報告日期：108 年 12 月 6 日

摘要

美國公定分析化學家協會(AOAC International)為一非營利化學協會，旨在發布有關食品過敏原、微生物、藥物殘留、污染物質及毒素等標準檢驗方法供全球檢驗機構參考，且每年皆有舉辦與食品中藥物殘留、污染物質及毒素、中草藥及非標的物分析相關會議，邀請各方專家學者發表近期科學新知，並提供全球產、官、學等不同領域技術交流平台。隨著科技日新月異，食品檢驗技術也日漸進步，為與國際接軌，本署每年皆會派員參加公定分析化學家協會(AOAC)年會暨研討會，與來自全球各地的產、官、學之專家學者互相交流，以開拓本署人脈，精進本署研究檢驗的實力，期能在未來開發方法與國際同步。本次年會共計有 23 個專題、近 1 百場專題演講及分為 3 天不同主題的壁報論文，本署李蕙君技士受邀於會中之「New Blood 2019-Developing Methods for the Detection of Important Chemical Analytes, Residues an Contaminants」時段發表口頭論文「The Detection and Identification of Tadalafil Analogues」，施又寧助理研究員則於「Poster Presentation: Analysis of Foodborne Contaminants and Residues, Analysis of Non-Foodborne Contaminants and Residues, and Microbiological Methods」時段發表壁報論文「Integration of Analytical Methods for Inorganic Arsenic in Food」，除與相關領域專家學者互相切磋外，亦提高臺灣在國際間的能見度，並展現本署之研究檢驗能力。另在臺灣分會上討論當前備受矚目的食安議題，討論熱烈，並在會後互相留下聯絡資訊，俾利未來更進一步的交流。

目次

目的

近年來，食品安全已成為國人重視的議題，公定分析化學家協會(AOAC)年會暨研討會每年皆會吸引國際產、官、學界人才出席，發表新穎的科學技術。本署執掌業務之一為開發並建立公告檢驗方法供各界使用，為瞭解國際趨勢，本署積極參與每年的研討會，除了聆聽及觀摩相關的檢驗技術及品質管制演講，亦發表本署近期的研究成果，達到國際間技術交流，展現傑出的檢驗能力，期能提高臺灣在國際間的能見度，同時精進國內的檢驗技術，厚植臺灣食品檢驗的基礎。

過程

與會同仁於 9 月 7 日自臺北啟程，經美國洛杉磯市轉乘國內線班機，於美國時間 9 月 7 日抵達美國丹佛市，並於同日至會場報到，於 9 月 8 日至 9 月 11 日出席第 133 屆 AOAC 年會，會上有來自全球各地的會員，包括產、官、學界的優秀研究人員共襄盛舉。與會同仁於會議期間參加不同主題的演講，並於 9 月 10 日之「New Blood 2019-Developing Methods for the Detection of Important Chemical Analytes, Residues an Contaminants」時段發表口頭論文「The Detection and Identification of Tadalafil Analogues」，於同日下午舉辦「Taiwan Section Business Meeting」，另於 9 月 11 日之「Poster Presentations: Analysis of Foodborne Contaminants and Residues, Analysis of Non-Foodborne Contaminants and Residues, and Microbiological Methods」時段發表壁報論文「Integration of Analytical Methods for Inorganic Arsenic in Food」，最終在 9 月 11 日下午議程結束後自丹佛機場搭機離開，經舊金山市轉機後，於臺灣時間 9 月 13 日抵達臺灣桃園。

在為期四天的年會中，同仁在 Scientific Session 聆聽多場專題演講、瀏覽連續三天不同主題的壁報論文、參觀今年最新分析儀器設備，獲益良多且成果豐碩。

一、專題演講：

今年主題包括中草藥、食品中藥物殘留、天然毒素、有害元素、食物過敏原、標準參考物質、品質管制、保健食品等檢驗技術，共分成 23 個專題，每一個專題分別有 3-4 場演講，內容豐富多元。其主題分列如下：

1. Wiley Award Symposium: Advances in Analytical Methods for Botanical Dietary Supplements and for Clinical Nutritional Assessment.
2. Recent Trends in Elemental Analysis Application
3. Application of DNA Technologies and Standards in the Authentication of Botanicals for Quality Control of Botanical Dietary Supplements
4. Multi-Class/Multi-Residue Veterinary Drug Methods-Which Strategies and for What Purpose?
5. Microbial Identification with Genomics and Proteomics in Food and Dietary Supplement
6. Applying Non-Target Data Acquisition for Target Analysis (nDATA) of Organic Contaminants and Biomarkers in Environment and Food Samples
7. Non-Target Testing for Food Authentication-Ideas, Challenges, Requirements
8. The Complexity of Validation STEC Methods to Address Varying Global Needs
9. New Blood 2019-Developing Methods for the Detection of Important Chemical Analytes, Residues an Contaminants
10. Food Fraud Detection Goes Mobile
11. How Can NGS-Based Methods Advance Food Safety and Quality Programs

12. Global Perspective on Mycotoxins in food

13. New Tools for Food Fraud, an Old Problem with Perpetually New Intricacy

14. Alternative Models for Characterizing Accuracy

15. Latest Development in Gluten Analysis

16. NMR Advancement in Quality Control and Compendial Applications

17. Reference Material Needs for Food Safety

18. Validation and Implementation of Emerging Methods for Food Allergen and Gluten Measurement

19. Improving the Measurement of Nutritional and Botanical Compounds-Strategies and Insights from NIST Quality Assurance Programs

20. Certified Reference Materials-Advancements in Manufacturing and Stability

21. Cannabis and Cannabis Byproducts-An Update on the State of Industry and Current Challenges

22. Prebiotic, an Evolving Nutrition Concept

23. Utilization of Enzymes for Analytical Analyses-Breakthroughs and Important Cautions

以上專題演講主題與本署業務息息相關，惟同一時段有 3 場演講同時進行，與會同仁僅將參與場次之內容概述如下：

(一) Wiley Award Symposium: Advances in Analytical Methods for Botanical Dietary Supplements and for Clinical Nutritional Assessment

植物營養補充品日益熱門，其中又以減肥與提升能量為大宗，為確保品質分析方法也須不斷精進。演講中列舉了 L-tryptophan 檢測方法、蕈類中 Agaritine 檢測方法、Yohimbe、Comfrey 及 Ephedra 成分分析方法，以 Chiral GC/NPD 檢測合成 ephedrine 的方法……等。另提到 American Herbal Products Association (AHPA)網頁有許多植物特性與鑑定方法相關資訊可供植物研究者或草藥產品生產者參考，而 National Institutes of Health (NIH)之 Dietary Supplement Analytical Methods and Reference Materials Program (AMRM)專頁上亦有針對營養補充品提供驗證參考物質(Certified Reference Materials, CRMs)之販售。

(二) Recent Trends in Elemental Analysis Application

1. Arsenic speciation in Krill Oil by Liquid Chromatography Inductively Coupled Plasma Mass Spectrometry

由 NOW FOODS 的 Katarzyna Banaszewski 發表磷蝦油中砷物種之檢驗方法。近年來，全球對於磷蝦油之需求逐漸上升，民眾開始食用與磷蝦油有關之保健食品，使政府機構對於磷蝦油產品需有更多的監控，而制定相關法規。本篇研究主要探討磷蝦油中無機砷檢驗方法開發，此方法之回收率及變異係數皆符合本署食品化學檢驗方法確效規範，前處理方式則與本署自行開發之無機砷檢驗方法有所不同，本篇研究之前處理方式係使用 0.28 M 硝酸微波消化法，而本署係使用超音波震盪萃取法，未來在本署開發水產動物油脂中無機砷之檢驗方法時，可將其前處理方式作為參考依據。

2. Arsenic, Iodine, and Bromine Speciation Analysis in Infant Formula, and Nutritional Products using HPLC-ICP-MS

由 Abbott Laboratories 的 Lawrence Pacquette 發表嬰幼兒食品及保健食品中砷、碘及溴物種之檢驗方法。在食品安全中，有害元素扮演著舉足輕重的地位，本篇研究係以兩種不同的方法，分別檢測嬰幼兒食品、保健食品及 NIST SRMs 中鹵素(如：溴及碘)物種及砷物種的含量。檢測結果，保健食品及 NIST SRMs 之鹵素物種的分析範圍介於 0~25660 μg/kg，而砷物種的分析範圍則介於 0~60 μg/kg，此方法之回收率及變異係數皆符合本署食品化學檢驗方法確效規範，顯示方法精準度佳。

3. Analysis of Thyroid Hormones in Dog Food by LC-ICP-MS

由 U.S. FDA 的 Robert Wilson 發表以 LC-ICP-MS 分析狗糧中甲狀腺激素含量。寵物的健康是飼主關注的議題，過去的文獻顯示，過量的甲狀腺激素會危害寵物的健康，因此本研究主要探討如何透過 LC-ICP-MS 定量狗糧中碘、碘酪氨酸(monoiodotyrosine, MIT)、二碘酪氨酸 (diiodotyrosine, DIT)、T3 及 T4 的含量。該方法已被用在許多狗糧中，並成功地找出危害寵物健康的狗糧，進而請廠商下架商品。

4. Heavy Metal Contaminants from Cannabis Vaporizer Cartridges: Valid Concern or Blowing Smoke?

由 CannaSafe 的 Ini Afia 發表，本篇研究主要闡述大麻霧化器卡匣中重金屬污染，測試 3 種不同品牌但皆係以標準化流程製造之大麻霧化器，檢測其砷、鉛、汞及鎘含量，他們依據此方法評估霧化器卡匣中會有多少重金屬因被霧化而溶出。初步數據顯示，重金屬的確會因霧化而從卡匣中溶出後被收集至凝集座中，本方法經測試後準確度佳，未來可應用於檢測大麻霧化器卡匣中重金屬含量。

(三) Application of DNA Technologies and Standards in the Authentication of Botanicals for Quality Control of Botanical Dietary Supplements

1. Validation Guidelines for PCR Based Botanical Species Identity Testing to Support Quality Control of Botanical Dietary Supplements

由 University of Guelph 的 Steven Newmaster 發表以 DNA 為基礎的植物基原鑑定方法。實驗操作通常使用均質機進行樣品前處理，後續加入界面活性劑與酵素以裂解細胞，最後使用 silica column 或酒精沉澱等方法純化 DNA。常用的鑑定方式可以分為:(1)目標性的，針對某種物種之序列設計的引子對與放大方法。(2)非目標性的，如NGS。比較常用的植物基原分析方法為 barcoding、NGS、pyrosequencing 及 probes nucleotides signature。本研究係以銀杏原料到產品之品種確認方法作為實例，並比較各基原鑑定發法的優劣。

2. Developing Species-Specific DNA Testing Methods to Identify Botanicals

由 USP 的 Ning Zhang 發表，介紹美國藥典中草本補充品來源鑑定之相關規範。由於草本補充品係源自於天然物，如有不慎攙偽或誤用有毒品種很有可能造成人體危害，故在美國藥典 (USP)中針對部分品項如：American ginseng 收載有檢驗方法。其中 DNA 鑑定方法使用通用引子 (universal primer)進行 PCR 增幅與定序比對，常用 marker 如: *rpo*2、*atp*A、*psb*D 及 *ndhc-atp*E 等。

3. Industry Experiences on Authentication of Botanical Materials using DNA-based Molecular Analysis

由 Herbalife International of America Inc.的 Zhangfei Lu 發表有關於草本產品之品種確認，常用方法不外乎形態上的鑑定、化學成分分析(如：HPTLC 成分分析)及 DNA 序列分析(如：ITS2 序列比對)。在有 NIST 標準品的情況下，DNA 層面的鑑定又可利用 targeting fragment 比對 PCR amplicon 大小與核酸序列，對於某些品種攙偽或誤用的情形都很有助益。

(四) Applying Non-Target Data Acquisition for Target Analysis (nDATA) of Organic Contaminants and Biomarkers in Environment and Food Samples

1. nDATA (non-target Data Acquisition Target Analysis) Workflow for Multiresidue Pesticide Screening by Ultra-High-Performance Liquid

Chromatography-Quadrupole Orbitrap Mass Spectrometry with a Compound Database

由 U.S. FDA 的 Jon Wong 發表有關農藥資料庫建置及相關驗證資訊。本篇研究主要分析儀器為Ultra-High-Performance Liquid Chromatography-Quadrupole Orbitrap Mass Spectrometry，前處理方式則是 QuEChERS，為驗證本篇研究之農藥資料庫，邀請超過 20 個實驗室針對 10 種不同基質檢測，而這些基質囊括 50 種農藥品項，截至目前為止，所有實驗室皆已提供比對數據，結果顯示，資料庫可以完成 50 種農藥品項的比對，未來會持續擴充，俾利提供更好的農藥檢測比對資料庫。

2. Improving Selectivity in Untargeted Contaminant Analysis with Scanning Quadrupole Data Independent Acquisition (sqDIA)

由U.S. FDA的William Cooke發表以 sqDIA 改善分析未知物之檢測技術。過去以 HRMS 執行例行食品中污染物檢測時，時常面臨許多挑戰，如：數據判讀，透過 DIA 的方式僅能依前驅離子及產物離子之滯留時間不同去分析待測物，如何解讀大量數據已成為一門重要的課題，因此被應用於 QToF 的 sqDIA 方法隨即產生，未來可將其應用在農藥檢測上。

3. Untargeted Mass Spectrometry Data Acquisition: An Emerging Approach to Detect Gelatin Adulteration Based on Targeted Proteotypic Peptides

由 University of Montreal 的 Francis Beaudry 發表以特定蛋白胜肽檢測明膠摻假。隨著交通科技進步，食品生產漸趨國際化，導致食品詐欺案例逐漸上升，為提高利潤，從假標示至使用非法添加物之案例層出不窮，食品詐欺已成為消費者關注的話題。明膠被廣泛使用在食品加工及化粧品，含有 I 型、II 型及 III 型膠原蛋白多胜肽。本研究提出兩種常見非目標物分析方法。未來能將非目標物分析方法與現有的檢測方法一同使用，使明膠摻雜能下降到 0.1%。

(五) New Blood 2019-Developing Methods for the Detection of Important Chemical

Analytes, Residues an Contaminants

本時段是由AOAC International邀請全球未曾於該年會發表口頭論文之研究人員分享自己的成果，內容多元豐富，本署受邀之李蕙君技士於本時段發表「The Detection and Identification of Tadalafil Analogues」，內容闡述本署摻假西藥檢驗方法及如何發現新西藥類緣物成分，此外，亦以近期2個tadalafil類緣物的例子說明新西藥類緣物成分分離鑑定流程，並分享本署新開發的tadalafil類緣物快速篩檢方法。其他講者之主題分別為「Simultaneous Targeted Quantification and Suspect Screening of Environmental Contaminates and Pesticide Residue in Food by High Resolution LC-QTOF」、「Seafood Reference Materials: Novel Production Methods and Analytical Assessment for Authentication of Food Products」及「Optimizing GC-MS and GC-MS/MS Analysis of 3-MCPD and glycidyl esters」，演講內容多元，涵蓋農藥、污染物質、中草藥以及品管物質，顯示國際間新生代研究人才輩出。

(六) NMR Advancement in Quality Control and Compendial Applications

1. NMR Advancements: Compendia Applications

由U.S. Pharmacopeia (USP)的Gabriel Giancaspro發表。隨著近年來qNMR的發展，相關應用方法也逐漸被納入美國藥典中。如beta-glucan中(1, 3)與(1, 6)鍵結的含量比例、最近當紅的磷蝦油31P檢測、蘆薈中acemannan檢測以及ginsenosied Rg1標準參考物質之標定。本研究亦以鑑定Cyanidin 3-o-glucoside之純度為例，若以HPLC分析在535 nm波長計算純度有90%，但以qNMR測定起來純度僅有78%，乃是因為pH值會影響該化合物之UV吸收。

2. 101 Uses for NMR in Your Home

由Steelyard Analytics Inc.的Kristie Adams及Spectral Service AG的Bernd Diehl發表。NMR係被廣泛運用於結構解析的高階儀器，其分析技術亦與常見的食品檢驗息息相關。例如：利用P核種的NMR分析phospholipid做為油品的來源鑑定、利

用13C天然同位素分布差異來確定糖來源(蔗糖或是甜菜之類)、蜂蜜或蜂糖摻假及複雜基質中的酒精含量檢測，如：血液中酒精濃度，並舉例以本技術發現一種軟性飲料含有很高濃度的酒精。

3. NMR Advancements: Food and Dietary Supplement Product's Applications

由 Herbalife International of American Inc 的 Congmei Cao 發表，主要是針對 NMR 技術在天然產物上的應用，例如：蘆薈產品之 *Aloe vera* 物種鑑定。又如鑑定茶萃取物，由於茶係屬相同品種不同製程之產物，較無法利用 PCR 進行物種鑑定，利用 NMR 測定其兒茶素反而有不錯的效果。

4. NMR Advancements: From Diverse Instrumentation to a Variety of Applications

由 Bruker Corporation 的 Amy Freund 發表，介紹 Bruker 在 NMR 技術的新進展與應用趨勢如下：近年來 NMR 除了不斷達到更高磁場(目前最高 1.1G Hz)，也開發了一些低磁場如桌上型約 60-100MHz 的 NMR 系統，提供較低成本的特殊應用。另提到新應用包括 cryogenically cooled probe 設計增加 NMR 感度、high resolution magic angle spinning (HR-MAS)設計增加 NMR 訊號解析度、dynamic nuclear polarization (DNP)技術以及快速實驗技術，如:NOAH (NMR by Ordered Acquisition using 1H detection)及 NUS(Non-Uniform Sampling)。此外，也討論了有關可利用的分析工具如：Human Metabolome Database (HMDB)及 Biological Magnetic Resonance Data Bank (BMRB)……等。

二、壁報論文

本次年會之壁報論文主題共分成三天，將其主題分列如下：

September 09

Botanical and Dietary Supplements, Food Nutrition and Food Allergens, Food Authenticity and Food Fraud, and Miscellaneous

September 10

Detection and Measurement of Natural Toxins, Agriculture and Environment, Cannabis, General Methods, Quality Assurance and Accreditation, and Performance Tested Methods℠

September 11

Analysis of Foodborne Contaminants and Residues, Analysis of Non-Foodborne Contaminants and Residues, and Microbiological Methods

三、參加臺灣分會會議：

每年年會皆會規劃專屬時段及場地提供 AOAC 臺灣分會舉辦「Taiwan Section Business Meeting」，今年由臺灣分會呂廷璋理事長、方銘志秘書長及同仁舉辦臺灣分會會議，本次約有 30 人參加，多為任職於美國官方及民間機構的臺灣人。會議期間皆以英語進行交流，首先由理事長介紹臺灣分會今年舉辦的活動及成果，並拋出食安議題供大家討論，例如：非目標物之分析、藥物殘留檢測、農藥資料庫及重金屬檢測…等，與會人員皆反應熱烈。

心得及建議

(一) 公定分析化學家協會(AOAC)年會暨研討會提供國際間各界開發之檢驗方法及品質管制一個互相交流的平台，本署研究檢驗組業務內容與其核心價值息息相關，建議未來持續派員參與以瞭解國際間新興檢驗技術，為民眾食品安全作最前端的把關。

(二) 近年國際間較受重視的議題包括非目標物檢測、天然藥物分析、NMR 檢測、磷蝦油中無機砷檢驗…等，未來皆可提供本署做為參考依據，俾利精進檢驗方法。

(三) 同仁在會中所發表之口頭論文及壁報論文，皆有來自不同國家及領域之專家學者前來討論，交流彼此意見及技術，建議未來持續派員參加並發表論文。

照片

會場入口


AOAC
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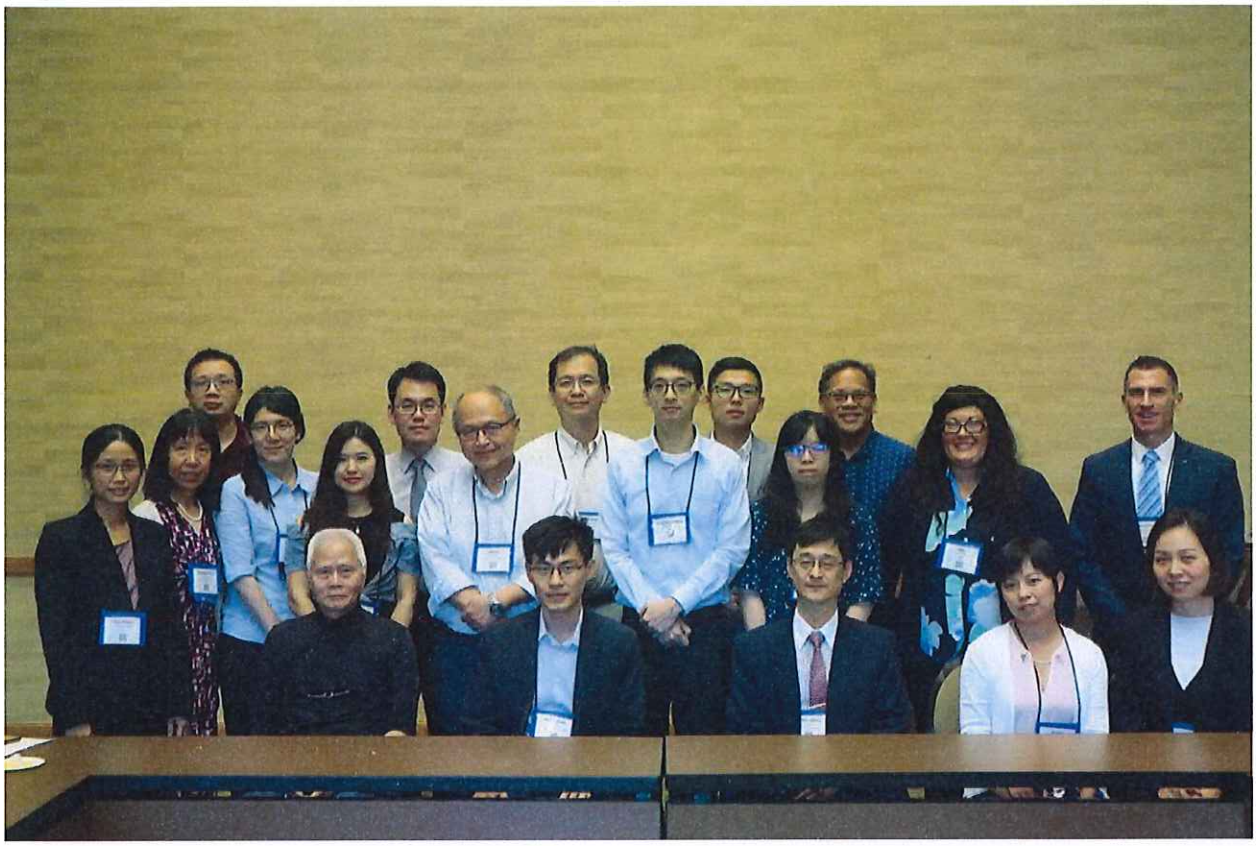
Opening Session

Keynote 演講

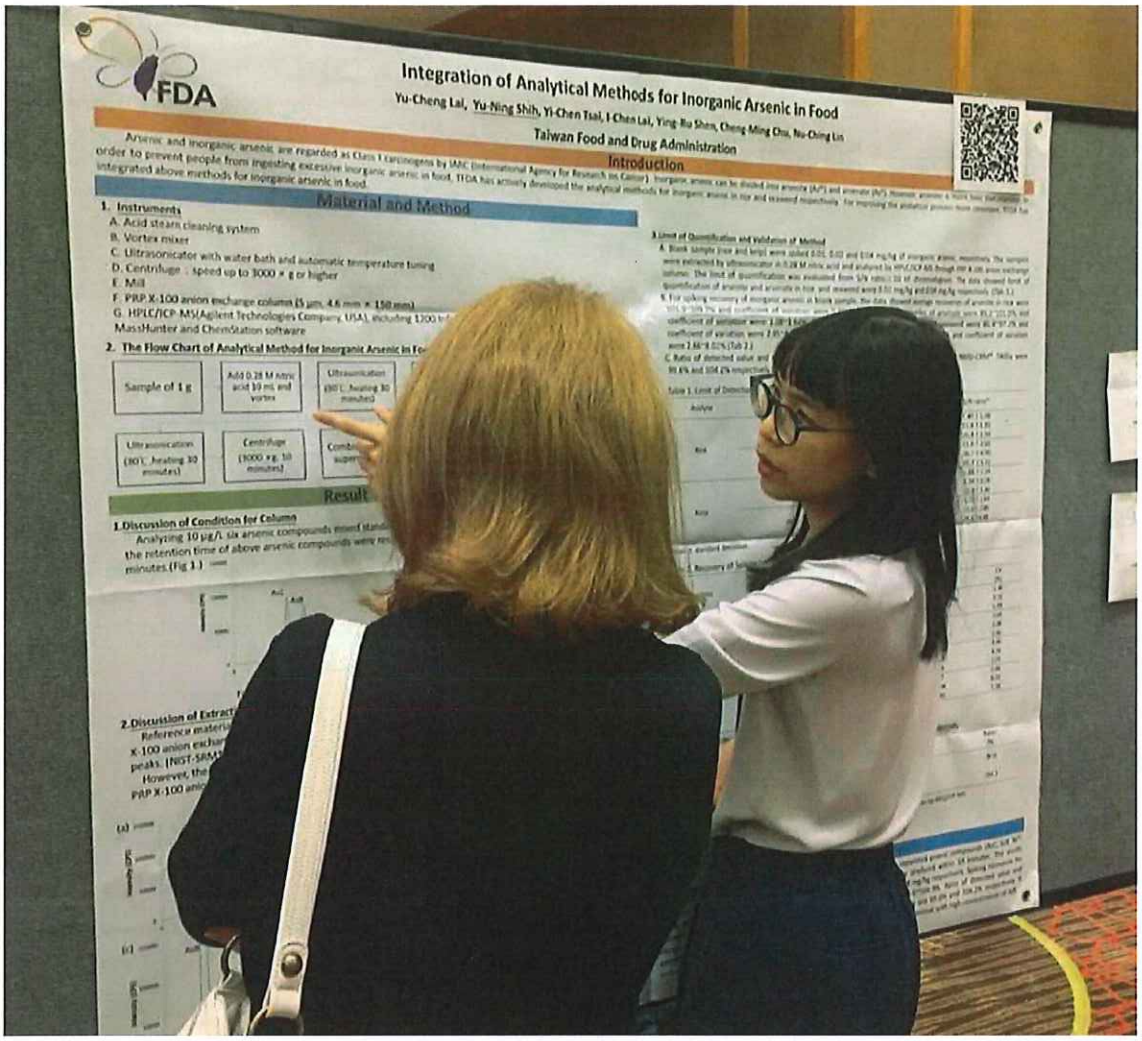
AOAC Taiwan Section Business Meeting

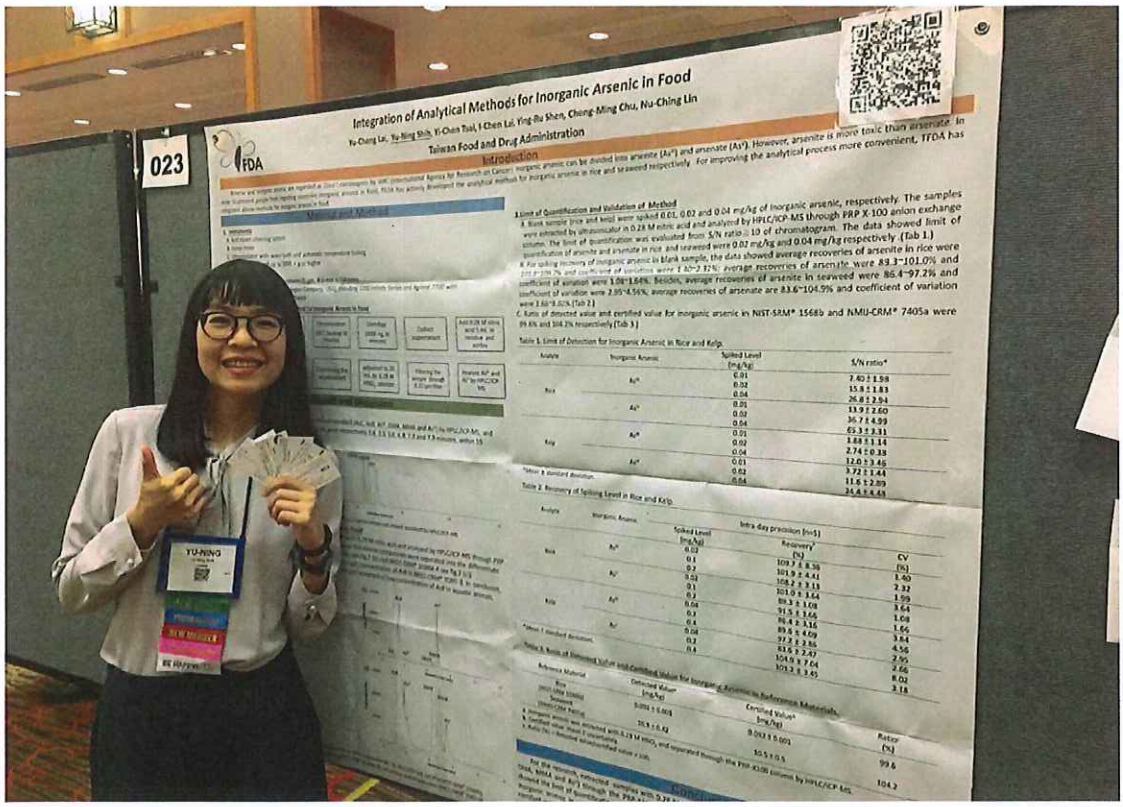


口頭報告

廠商佈展

壁報論文


FDA
Integration of Analytical Methods for Inorganic Arsenic in Food
Yu-Cheng Lai, Yu-Ning Shih, Yi-Chen Tsai, I-Chen Lai, Ying-Ru Shen, Cheng-Ming Chu, Nu-Ching Lin
Taiwan Food and Drug Administration
Introduction
Material and Method
Result


023
FDA
Integration of Analytical Methods for Inorganic Arsenic in Food
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Taiwan Food and Drug Administration
Introduction
YU-NING

The Detection and Identification of Tadalafil Analogues

Hui-Chun Lee
Taiwan Food and Drug Administration (TFDA)

衛生福利部 食品藥物管理署 Food and Drug Administration

http://www.fda.gov.tw/

Outline

- The detection of drug adulterants and their analogues
- The identification of tadalafil analogues
  - Case 1: Dipropylaminopretadalafil
  - Case 2: *N*-3-hydroxypropylnortadalafil
- The screening for tadalafil analogues

The detection of drug adulterants and their analogues

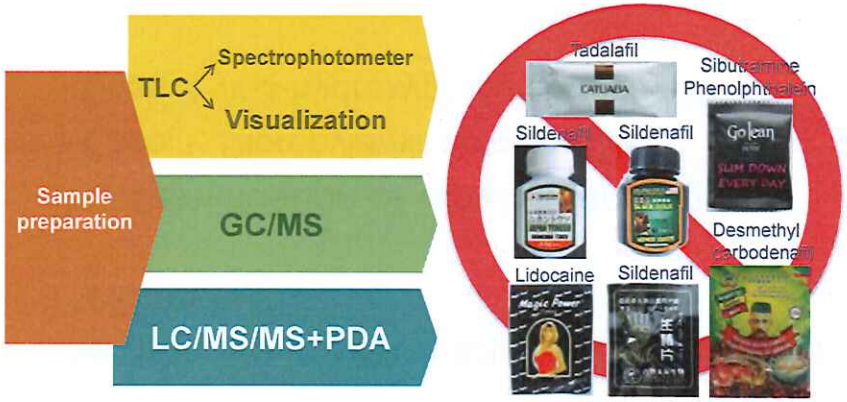
FDA

Drug-adulteration analysis

- Taiwan FDA tests samples from customs, police and public health bureaus.
- The high detection rate of drug adulterants indicates that many illegal products are intercepted at the border.

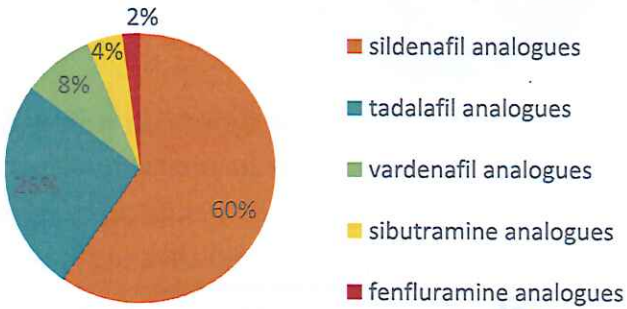
Drug-adulteration analysis

- Drug-adulterants screening



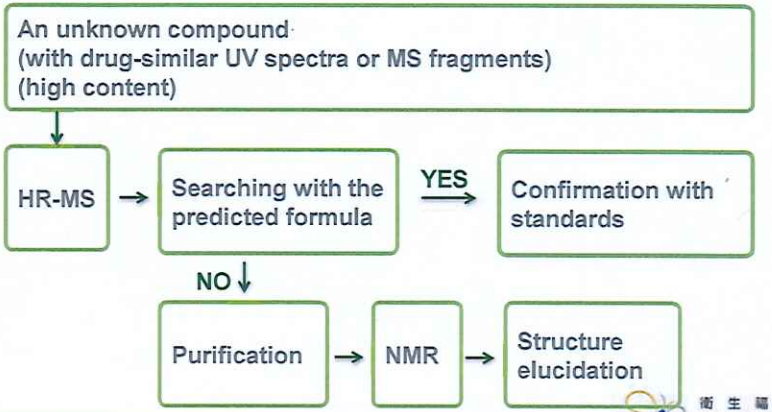
Novel analogues identification

- First detected analogue, homosildenfil, in 2002
- Total 47 analogues were determined until 2018



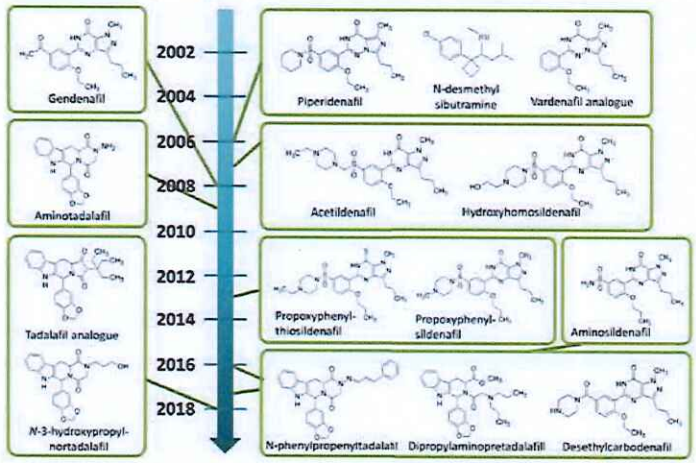
Novel analogues identification

- Drug analogues:
  a compound with structures like an approved drug but with structural modifications



Novel analogues identification

- Novel analogue publications



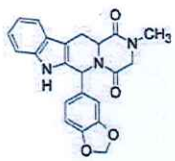
The identification of tadalafil analogues

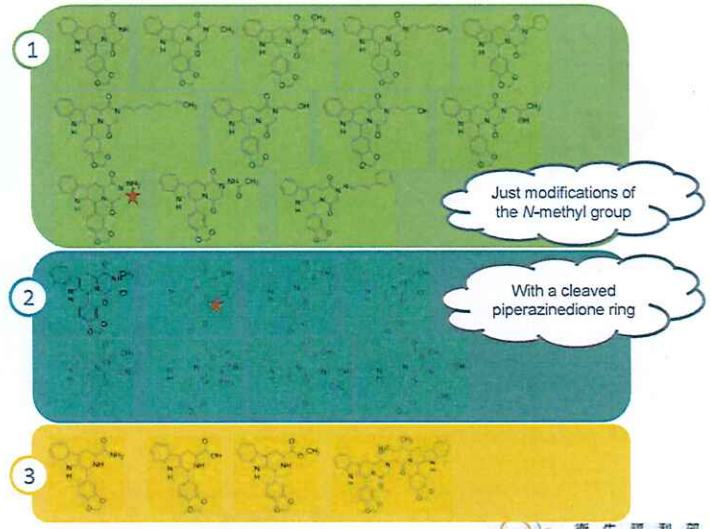
FDA

Tadalafil (Cialis)

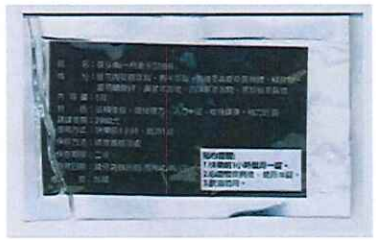
- Type V phosphodiesterase (PDE5) inhibitor
- For erectile dysfunction (ED)
- Improved PDE5/PDE6 selectivity
- Formula:$C_{22}H_{19}N_3O_4$, MW:389.4
- The first analogue was reported in 2006.
- 21 tadalafil analogues were reported until 2017.

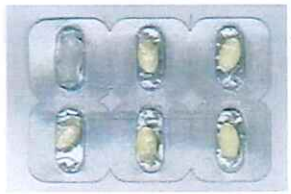
Tadalafil analogues





Case 1





Containing an obviously purple spot on the TLC plates but matched with no tadalafil analogue in the library

Case 1

20 Tabs (15.8 g)

↓ Extract with 50 mL MeOH *3

Crude extract (895 mg)

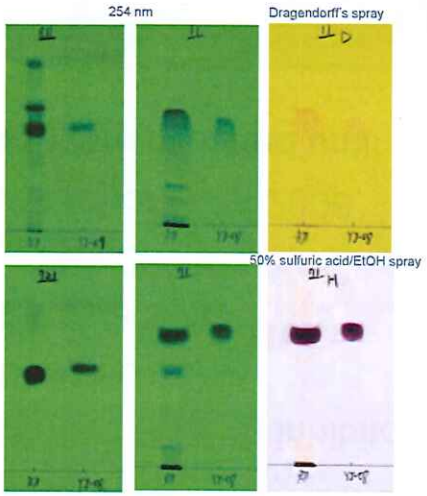
↓ Partition with Water / $CHCl_3$

Water layer

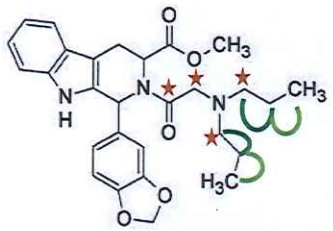
Organic layer ($CHCl_3$)

↓ Open column (silica gel)
$CHCl_3$: MeOH = 29 : 1

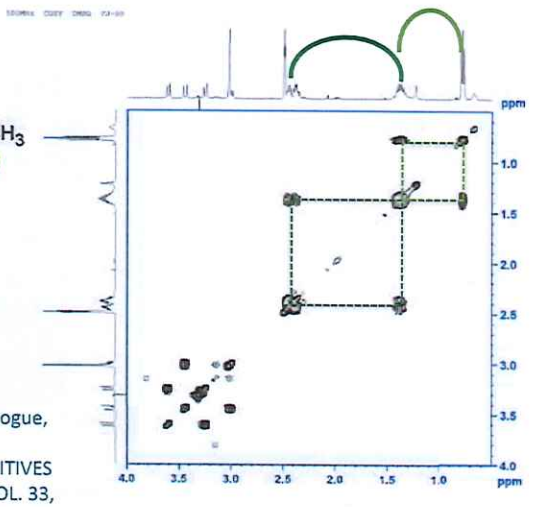
Compound YJ-08 (7.8 mg)



Case 1

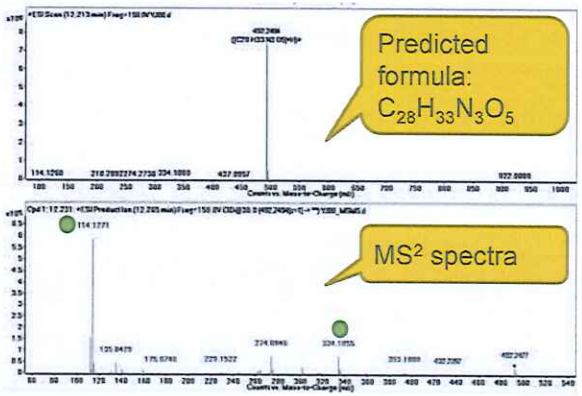


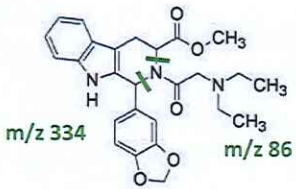
Dipropylaminopretadalafil
(YJ-08)



Identification of a new tadalafil analogue, dipropylaminopretadalafil, in a dietary supplement. FOOD ADDITIVES & CONTAMINANTS: PART A, 2016 VOL. 33, NO. 6, 953–958

Case 1

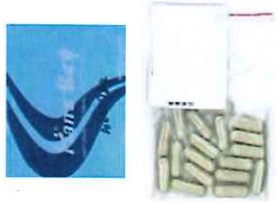




Diethylaminopretadalafil

Case 2

Containing a compound that has MS^2 data matched with 2-hydroxypropylnortadalafil but its *Rf* values shifted on the TLC plates.



2-hydroxypropylnortadalafil

Case 2

12 capsules (5.9 g)

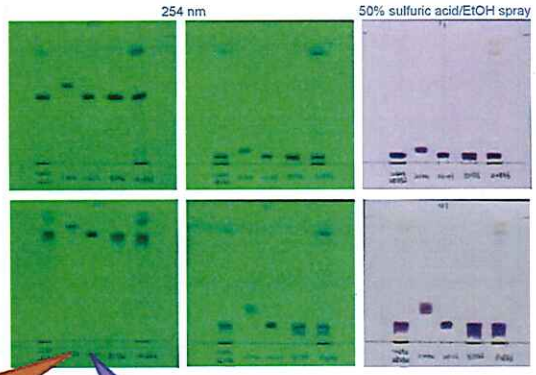
↓ Extracted with 15 mL EtOH for 30 min*3

↓ Extracted with 10 mL Chloroform for 30 min*3

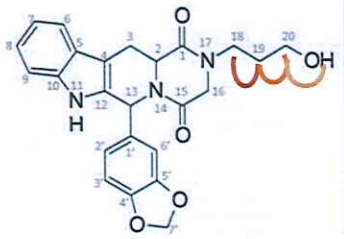
Chloroform crude extract (0.48 g)

↓ Column elution Recrystallized

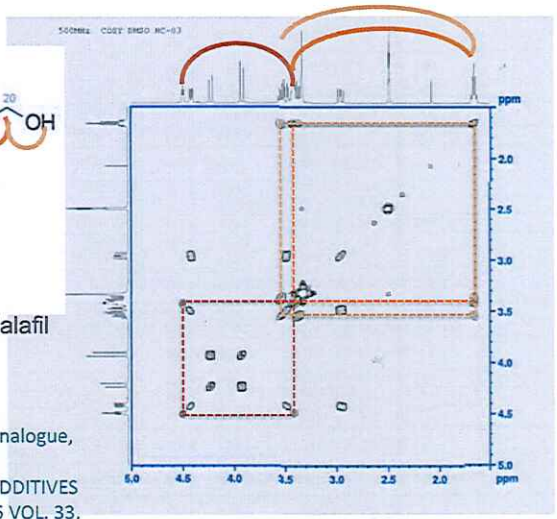
Compound HC-03 (138.3 mg)



Case 2

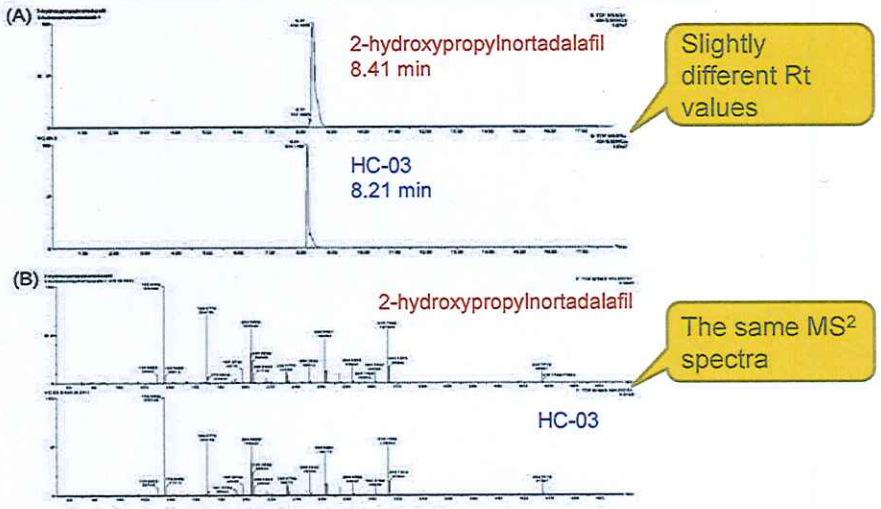


N-3-hydroxypropylnortadalafil (HC-03)



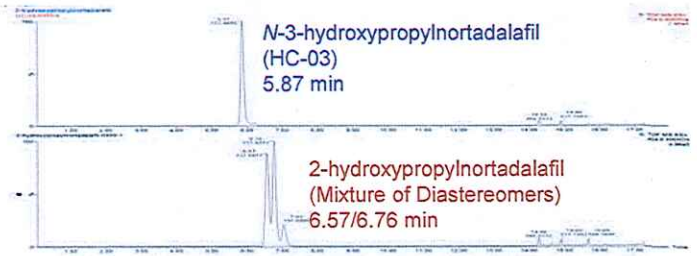
Identification of a new tadalafil analogue, dipropylaminopretadalafil, in a dietary supplement. FOOD ADDITIVES & CONTAMINANTS: PART A, 2016 VOL. 33, NO. 6, 953–958

Case 2



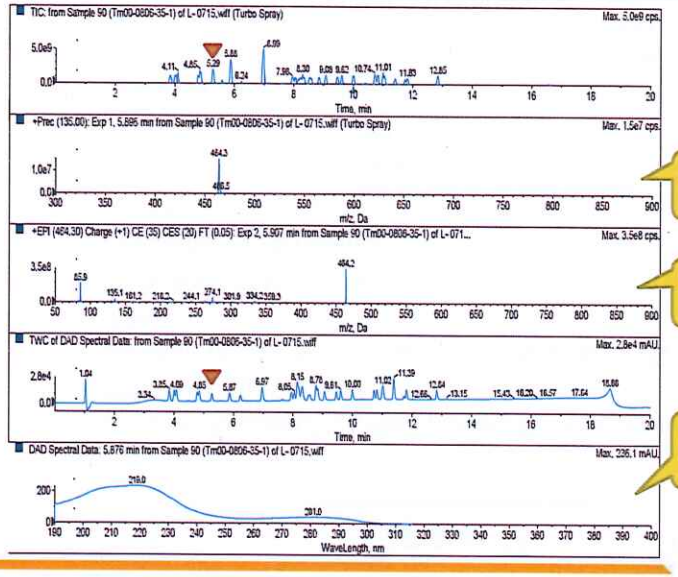
Case 2

- LC:
  - Column: AcquityUPLC®BEH shield RP 18 column (2.1 × 100 mm, 1.7 µm)
  - Solvent: (A) 0.1% formic acid in ACN & (B) 0.1% formic acid in ddH_2O
  - 70 % solvent B
  - Flow rate: 0.25 mL/min



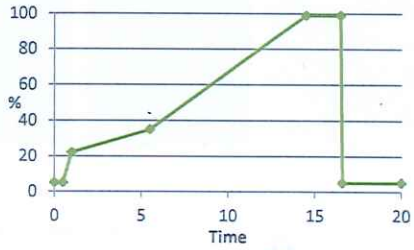
The screening for tadalafil analogues

Standard mix



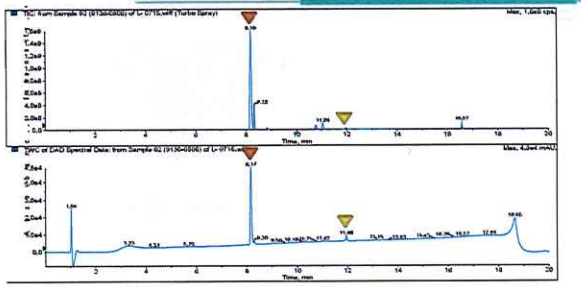
The screening method conditions

- Instrument: ABSciex QTRAP®5500
- LC:
  - Column: AcquityUPLC®BEH shield RP 18 column (2.1 × 100 mm, 1.7 µm)
  - Solvent: 0.1%FA in ACN & 0.1%FA in ddH_2O
  - Flow rate: 0.3 mL/min
- MS²: Prec+IDA+EPI
- PDA: 200-400 nm



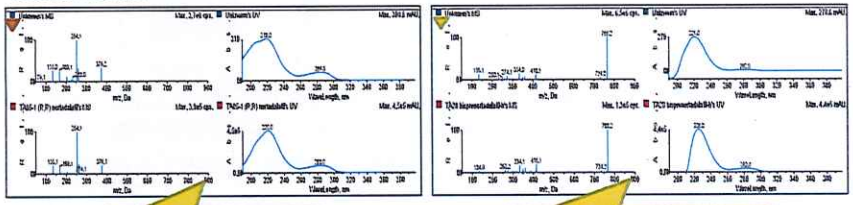
#	Name	Synonyms	Formula	MW	RT	Fragments
1	Tadalafil	Cialis/ Tardenafil	$C_{22}H_{19}N_3O_4$	389.4	8.82	268, 262, 205, 197, 169, 135, 77
2	Tadalafil impurity MW335	Tadalafil impurity 13	$C_{19}H_{17}N_3O_3$	335.4	3.86	319, 264, 250, 187, 150, 135
3	Tadalafil acid impurity MW336	Tadalafil acid impurity /Tadalafil impurity 20	$C_{19}H_{16}N_2O_4$	336.4	4.81	264, 250, 233, 135
4	Tadalafil impurity MW349	Tadalafil impurity 21,22 /Tadalafil impurity A , B	$C_{20}H_{19}N_3O_3$	349.4	4.03/4.12	333, 290, 274, 264, 206, 144, 87
5	Methyl ester tadalafil Impurity MW350	Tadalafil related impuirty 5, 6/ Tadalafil impurity 7, 31, 32	$C_{20}H_{18}N_2O_4$	350.4	4.85	264, 250, 206, 191, 135
6	Nortadalafil	N-Desmethyl tadalafil	$C_{21}H_{17}N_3O_4$	375.4	8.26/8.39	262, 254, 205, 169, 135
7	Aminotadalafil		$C_{21}H_{18}N_4O_4$	390.4	8.8	269, 262, 205, 169, 135
8	*N*-Ethyl tadalafil	Homotadalafil	$C_{23}H_{21}N_3O_4$	403.4	9.43	302, 282, 254, 169, 135
9	Tadalafil dimethoxy Impurity MW405		$C_{23}H_{23}N_3O_4$	405.5	8.21	268, 248, 233, 169, 151
10	*N*-Isopropyl tadalafil	HC-01	$C_{24}H_{23}N_3O_4$	417.5	9.97	296, 268, 262, 204, 169, 135
11	2-Hydroxyethyl nortadalafil		$C_{23}H_{21}N_3O_5$	419.4	8.05	298, 262, 250, 197, 169, 135
12	Tadalafil impurity MW425	Tadalafil impurity 23, 24 /Tadalafil impurity C, D	$C_{22}H_{20}ClN_3O_4$	425.9	9.15/9.63	395, 333, 276, 218, 159, 135
13	Chloropretadalafil	Tadalafil impurity 3, 4	$C_{22}H_{19}ClN_2O_5$	426.9	11.05	395, 334, 302, 274, 262, 135
14	*N*-Butyl tadalafil		$C_{25}H_{25}N_3O_4$	431.5	11.06	310, 262, 169, 135
15	Acetaminotadalafil		$C_{23}H_{20}N_4O_5$	432.4	8.19	311, 262, 233, 205, 169, 135
16	2-Hydroxypropylnortadalafil		$C_{24}H_{23}N_3O_5$	433.5	8.50/8.54	312, 262, 169, 135
17	*N*-3-Hydroxypropylnortadalafil	HC-03/Tadalafil impurity 37	$C_{24}H_{23}N_3O_5$	433.5	8.34	312, 262, 169, 135
18	Dimethylaminopretadalafil	Tadalafil related compound 2 /YC1218	$C_{24}H_{25}N_3O_5$	435.5	5.33	359, 334, 274, 135
19	Chloropropanoylpretadalafil		$C_{23}H_{22}ClN_3O_5$	440.9	11.01	409, 334, 302, 274, 262, 135
20	Tadalafil impurity MW440	Tadalafil impurity 25	$C_{23}H_{21}ClN_2O_5$	440.9	11.62/11.96	409, 334, 302, 274, 262, 135
21	Cylcopentyl nortadalafil	Tadalafil related impurity1	$C_{26}H_{25}N_3O_4$	443.5	10.84	322, 294, 262, 169, 135
22	Tadalafil dichloro impurity MW461	Tadalafil dichloro Impurity	$C_{22}H_{19}Cl_2N_3O_5$	461.3	11.82	334, 302, 274, 262, 135
23	Diethylaminopretadalafil	Tadalafil related compound 1 / YJ-01	$C_{26}H_{29}N_3O_5$	463.5	5.86	334, 274, 135, 86
24	*N*-Octyl nortadalafil		$C_{29}H_{33}N_3O_4$	487.6	12.85	266, 338, 262, 169, 135
25	Dipropylaminopretadalafil	YJ-08 / Tadalafil impurity 17, 18 /Tadalafil impurity 9 /Tadalafil N-dipropyl impurity	$C_{28}H_{33}N_3O_5$	491.6	6.97/6.99	334, 274, 135, 114, 86
26	*N*-phenylpropenyl tadalafil	YJ-02	$C_{30}H_{24}N_4O_4$	504.6	11.4	383, 262, 250, 232, 204, 169, 135
27	Bisprenortadalafil		$C_{41}H_{35}N_5O_9$	765.8	11.96	734, 416, 360, 349, 334, 302, 276, 262, 135

Sample analysis results



A white powder sample contains both nortadalafil and bisprenortadalafil.

Search result:



Nortadalafil

Bisprenortadalafil

Summary

- Tadalafil and its analogues are adulterants detected frequently in food products.
- Novel tadalafil analogues can be found by tadalafil-similar UV spectra and MS^2 fragments.
- Because there are many isomers, tadalafil analogues should be confirmed by not only MS^2 fragments but also Rt value.
- With the new screening method, we can detect at least 27 tadalafil-related compounds and find novel analogues at the same time.

Acknowledgement

Division of Research and Analysis, Taiwan Food and Drug Administration

國立臺灣大學 National Taiwan University

The Instrumentation Center, National Taiwan University

Yen-Chun Huang

Super Laboratory Company Ltd

Yun-Lian Lin

China Medical University

Food and Drug Administration Ministry of Health and Welfare

Thank you for your attention!

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衛生福利部 食品藥物管理署 Food and Drug Administration

http://www.fda.gov.tw/

Difficulties in analogues confirmation

- Chaotic analogue names
  - No single correspondent name
  - Name of analogue standards on suppliers' websites may change or have different meaning
- The same MS fragments ≠ the same compound
  - Structural isomers with the same molecular weight
- Different R*t* or R*f* values ≠ different compounds
  - Analogues with chiral centers

29

Case 1

- NMR data summary:

Table 1. NMR data of compounds YJ-01 and YJ-08 (500 MHz in DMSO-d_6, J in Hz).

YJ-01			YJ-08				
No.[a]	δ_H[b]	δ_C	No.[a]	δ_H[b]	δ_C	COSY	HMBC
1	5.79 (1 H, *d*, *J* = 6.9)	51.6	1	5.71(1 H, *d*, *J* = 6.8)	51.6	H-13	C-3, C-12, C-13, C-14, C-16
3	6.73 (1 H, *s*)	50.6	3	6.75(1 H, *s*)	50.4	–	C-1, C-4, C-12, C-16, C-1', C-2', C-6'
4	–	130.4	4	–	130.3	–	–
6	–	136.4	6	–	136.3	–	–
7	7.27 (1 H, *d*, *J* = 8.0)	111.2	7	7.27(1 H, *d*, *J* = 8.0)	111.2	H-8	C-9, C-11
8	7.08 (1 H, *t*, *J* = 7.3)	121.5	8	7.08(1 H, *t*, *J* = 7.6)	121.4	H-7, H-9	C-6, C-7, C-9, C-10
9	7.02 (1 H, *t*, *J* = 7.3)	118.7	9	7.01(1 H, *t*, *J* = 7.6)	118.6	H-8, H-10	C-7, C-8, C-11
10	7.52 (1 H, *d*, *J* = 8.0)	118.1	10	7.52(1 H, *d*, *J* = 8.0)	118.0	H-9	C-6, C-8, C-11, C-12
11	–	126.0	11	–	125.9	–	–
12	–	106.4	12	–	106.2	–	–
13	2.98, 3.47 (1 H each, *d*, *J* = 8.0)	21.2	13	2.98(1 H, dd, *J* = 13.8, 6.8) 3.43(1 H, *d*, *J* = 13.8)	21.2	H-1	C-1, C-4, C-11, C-12, C-14
14	–	171.1	14	–	171.0	–	–
15	3.00 (3 H, *s*)	51.6	15	3.01(3 H, *s*)	51.5	–	C-14
16	–	170.7	16	–	170.6	–	–
17	3.21, 3.61 (1 H each, *d*, *J* = 13.7)	58.0	17	3.25, 3.61 (1 H each, *d*, *J* = 13.8)	58.0	–	C-16, C-19, C-22
19/21	2.45, 2.54 (2 H each, *q*, *J* = 7.0)	46.7	19/22	2.37, 2.48 (2 H each, *q*, *J* = 7.3)	55.7	H-20, H-23	C-17, C-20, C-21, C-23, C-24
20/22	0.92 (6 H, *t*, *J* = 7.2)	11.4	20/23	1.38(4 H, *m*)	19.3	H-19, H-21, H-22, H-24	C-19, C-21, C-22, C-24
			21/24	0.78(6 H, *t*, *J* = 7.3)	11.7	H-20, H-23	C-19, C-20, C-22, C-23
1'	–	134.1	1'	–	134.1	–	–
2'	6.67 (1 H, *s*)	109.2	2'	6.66(1 H, *d*, *J* = 1.2)	109.1	–	C-3, C-1', C-3', C-4', C-6'
3'	–	146.5	3'	–	146.8	–	–
4'	–	146.9	4'	–	146.4	–	–
5'	6.78 (1 H, *d*, *J* = 8.2)	107.5	5'	6.77(1 H, *d*, *J* = 8.1)	107.4	H-6'	C-1', C-3', C-4', C-6'
6'	6.46 (1 H, *d*, *J* = 8.2)	122.3	6'	6.45(1 H, *dd*, *J* = 8.1, 1.2)	122.2	H-5'	C-3, C-2', C-4', C-5'
7'	5.95 (2 H, *d*, *J* = 8.0)	101.0	7'	5.95(2 H, *d*, *J* = 5.2)	100.9	–	C-3', C-4'
N-H	10.82 (1 H, *s*)	–	N-H	10.83(1 H, *s*)	–	–	C-4, C-6, C-11, C-12

Notes: [a]See Figure 1 for atom numbering.
[b]*d*, doublet; *dd*, doublet of doublet; *m*, multiplet; *q*, quartet; *s*, singlet; *t*, triplet.

Identification of a new tadalafil analogue, dipropylaminopretadalafil, in a dietary supplement. FOOD ADDITIVES & CONTAMINANTS: PART A, 2016 VOL. 33, NO. 6, 953–958

Case 2

- NMR data summary:

Table 1
NMR data of compound HC-03 (500 and 125 MHz in DMSO-d_6, J in Hz).

No.[a]	δ_H[b]	δ_C	COSY	HMBC
1	–	166.6	–	–
2	4.41 (1H, dd, *J* = 11.5, 4.0 Hz)	55.4	H-3	C-1, C-3, C-4
3	2.95 (1H, dd, *J* = 15.0, 11.5 Hz)3.50 (1H, dd, *J* = 15.0, 4.0 Hz)	22.7	H-2	C-1, C-2, C-4, C-5, C-12
4	–	104.6	–	–
5		125.7	–	–
6	7.53 (1H, d, *J* = 8.0 Hz)	118.1	H-7	C-4, C-5, C-8, C-10
7	6.98 (1H, td, *J* = 8.0, 1.0 Hz)	118.9	H-6, H-8	C-4, C-5, C-6, C-8, C-9, C-10
8	7.05 (1H, td, *J* = 8.0, 1.0 Hz)	121.2	H-7, H-9	C-6, C-7, C-9, C-10
9	7.29 (1H, d, *J* = 8.0 Hz)	111.3	H-8	C-5, C-7
10	–	136.1	–	–
12	–	133.8	–	–
13	6.16 (1H, s)	54.9	–	C-2, C-4, C-12, C-15, C-1', C-2', C-6'
15	–	167.5	–	–
16	3.92 (1H, d, *J* = 17.0 Hz) 4.22 (1H, dd, *J* = 17.0, 1.0 Hz)	59.1	–	C-15, C18
18	3.36/3.53 (1H each, m)	42.9	H-19	C-1, C-16, C-19, C-20
19	1.66 (2H, quintet, *J* = 6.5 Hz)	29.9	H-18, H-20	C-18, C-20
20	3.42 (2H, m)	58.2	H-19	C-18, C-19
1'	–	156.8	–	–
2'	6.75 (1H, dd, *J* = 1.5, 8.0 Hz)	119.0	H-3', H-6'	C-4', C-6'
3'	6.77 (1H, d, *J* = 8.0 Hz)	108.0	H-2'	C-4', C-5'
4'	–	146.1	–	–
5'	–	147.1	–	–
6'	6.84 (1H, d, *J* = 1.5 Hz)	106.8	H-2'	C-13, C-2'
7'	5.92 (2H, s)	100.9	–	C-4', C-5'
N–H	11.06 (1H, s)	–	–	C-4, C-5, C-9, C-10, C-12, C-13
O–H	4.49 (1H, t, *J* = 5.0 Hz)	–	H-20	C-19, C-20

[a] See Fig. 1 for atom numbering.
[b] *d*: doublet; *dd*: doublet of doublet; *m*: multiplet; *s*: singlet; *t*: triplet.

Identification of a new tadalafil analogue, *N*-3-hydroxypropylnortadalafil, in a supplement product. (2018)
Journal of Pharmaceutical and Biomedical Analysis 158 (2018) 257–261

Integration of Analytical Methods for Inorganic Arsenic in Food

Yu-Cheng Lai, Yu-Ning Shih, Yi-Chen Tsai, I-Chen Lai, Ying-Ru Shen, Cheng-Ming Chu, Nu-Ching Lin

Taiwan Food and Drug Administration

Introduction

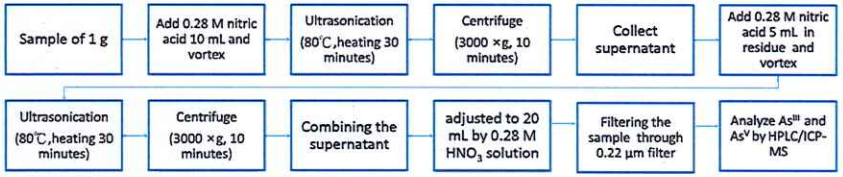
Arsenic and inorganic arsenic are regarded as Class I carcinogens by IARC (International Agency for Research on Cancer). Inorganic arsenic can be divided into arsenite (As^{III}) and arsenate (As^{V}). However, arsenite is more toxic than arsenate. In order to prevent people from ingesting excessive inorganic arsenic in food, TFDA has actively developed the analytical methods for inorganic arsenic in rice and seaweed respectively. For improving the analytical process more convenient, TFDA has integrated above methods for inorganic arsenic in food.

Material and Method

1. Instruments

A. Acid steam cleaning system
B. Vortex mixer
C. Ultrasonicator with water bath and automatic temperature tuning
D. Centrifuge：speed up to 3000 × g or higher
E. Mill
F. PRP X-100 anion exchange column (5 μm, 4.6 mm × 150 mm)
G. HPLC/ICP-MS(Agilent Technologies Company, USA), including 1200 Infinity Series and Agilent 7700 with MassHunter and ChemStation software

2. The Flow Chart of Analytical Method for Inorganic Arsenic in Food



Result and Discussion

1.Discussion of Condition for Column

Analyzing 10 μg/L six arsenic compounds mixed standard (AsC, AsB, As^{III}, DMA, MMA and As^{V}) by HPLC/ICP-MS, and the retention time of above arsenic compounds were respectively 1.6, 2.3, 3.6, 6.8, 7.3 and 7.9 minutes, within 15 minutes.(Fig 1.)

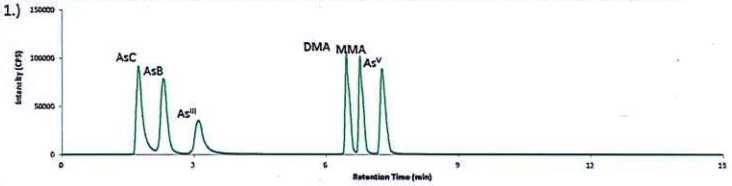


Figure 1.The chromatogram of 10 μg/L six arsenic compounds mixed standard by HPLC/ICP-MS

2.Discussion of Extraction Method for Inorganic Arsenic in Food

Reference materials were extracted by ultrasonicator with 0.28 M nitric acid and analyzed by HPLC/ICP-MS through PRP X-100 anion exchange column. The chromatogram showed the arsenic compounds were separated into the differentiate peaks. (NIST-SRM® 1568b see fig.2 (a), NMIJ-CRM® 7405a see fig.2 (b) and NRCC-CRM® DORM-4 see fig.2 (c))

However, the arsenite cannot be separated due to the high concentration of AsB in NRCC-CRM® TORT-3. In conclusion, PRP X-100 anion exchange column is appropriate for rice and seaweed or low concentration of AsB in aquatic animals.

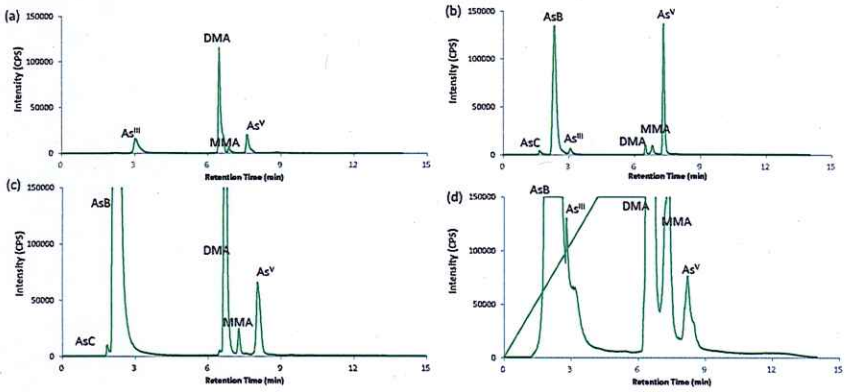


Figure 2. The chromatogram of 10 μg/L six arsenic compounds in reference materials by HPLC/ICP-MS. (a) Rice(NIST-SRM® 1568b), (b) Seaweed(NMIJ-CRM® 7405a), (c) Fish protein (NRCC-CRM® DORM-4), (d) Lobster hepatopancreas (NRCC-CRM® TORT-3).

3.Limit of Quantification and Validation of Method

A. Blank sample (rice and kelp) were spiked 0.01, 0.02 and 0.04 mg/kg of inorganic arsenic, respectively. The samples were extracted by ultrasonicator in 0.28 M nitric acid and analyzed by HPLC/ICP-MS through PRP X-100 anion exchange column. The limit of quantification was evaluated from S/N ratio≧10 of chromatogram. The data showed limit of quantification of arsenite and arsenate in rice and seaweed were 0.02 mg/kg and 0.04 mg/kg respectively .(Tab 1.)

B. For spiking recovery of inorganic arsenic in blank sample, the data showed average recoveries of arsenite in rice were 101.9~109.7% and coefficient of variation were 1.40~2.32%; average recoveries of arsenate were 89.3~101.0% and coefficient of variation were 1.08~3.64%. Besides, average recoveries of arsenite in seaweed were 86.4~97.2% and coefficient of variation were 2.95~4.56%; average recoveries of arsenate are 83.6~104.9% and coefficient of variation were 2.66~8.02%.(Tab 2.)

C. Ratio of detected value and certified value for inorganic arsenic in NIST-SRM® 1568b and NMIJ-CRM® 7405a were 99.6% and 104.2% respectively.(Tab 3.)

Table 1. Limit of Detection for Inorganic Arsenic in Rice and Kelp.

Analyte	Inorganic Arsenic	Spiked Level (mg/kg)	S/N ratio*
Rice	As^{III}	0.01	7.40 ± 1.98
		0.02	15.8 ± 1.83
		0.04	26.8 ± 2.94
	As^{V}	0.01	13.9 ± 2.60
		0.02	36.7 ± 4.99
		0.04	65.3 ± 3.31
Kelp	As^{III}	0.01	1.88 ± 1.14
		0.02	2.74 ± 0.38
		0.04	12.0 ± 3.46
	As^{V}	0.01	3.72 ± 1.44
		0.02	11.6 ± 2.89
		0.04	24.4 ± 4.48

*Mean ± standard deviation.

Table 2. Recovery of Spiking Level in Rice and Kelp.

Analyte	Inorganic Arsenic	Intra-day precision (n=5)		
		Spiked Level (mg/kg)	Recovery* (%)	CV (%)
Rice	As^{III}	0.02	109.7 ± 8.36	1.40
		0.1	101.9 ± 4.41	2.32
		0.2	108.2 ± 3.13	1.99
	As^{V}	0.02	101.0 ± 3.64	3.64
		0.1	89.3 ± 1.08	1.08
		0.2	91.5 ± 1.66	1.66
Kelp	As^{III}	0.04	86.4 ± 3.16	3.64
		0.2	89.6 ± 4.09	4.56
		0.4	97.2 ± 2.86	2.95
	As^{V}	0.04	83.6 ± 2.47	2.66
		0.2	104.9 ± 7.04	8.02
		0.4	101.2 ± 3.45	3.18

*Mean ± standard deviation.

Table 3. Ratio of Detected Value and Certified Value for Inorganic Arsenic in Reference Materials.

Reference Material	Detected Value[a] (mg/kg)	Certified Value[b] (mg/kg)	Ratio[c] (%)
Rice (NIST-SRM 1568b)	0.092 ± 0.001	0.092 ± 0.001	99.6
Seaweed (NMIJ-CRM 7405a)	10.9 ± 0.42	10.5 ± 0.5	104.2

a. Inorganic arsenic was extracted with 0.28 M HNO_3 and separated through the PRP-X100 column by HPLC/ICP-MS.
b. Certified value: mean ± uncertainty.
c. Ratio (%) = detected value/certified value x 100.

Conclusion

For the research, extracted samples with 0.28 M HNO_3 and completely separated arsenic compounds (AsC, AsB, As^{III}, DMA, MMA and As^{V}) through the PRP-X100 column by HPLC/ICP-MS can be analyzed within 15 minutes. The results showed the limit of quantification of rice and seaweed were 0.02 mg/kg and 0.04 mg/kg respectively. Spiking recoveries for inorganic arsenic in rice and seaweed were respectively 89.3~109.7% and 83.6~104.9%. Ratio of detected value and certified value for inorganic arsenic in NIST-SRM® 1568b and NMIJ-CRM® 7405a are 99.6% and 104.2% respectively. It indicated the analytical method is suitable for rice and seaweed but not for aquatic animal with high concentration of AsB.

第133屆公定分析化學家協會(AOAC)年會暨研討會

出國報告心得分享

出國人員：施又寧助理研究員、李蕙君技士
派赴國家：美國(科羅拉多州丹佛市)
出國期間：108年9月7日至108年9月13日
報告日期： 108年12月2日

衛生福利部 食品藥物管理署 Food and Drug Administration
http://www.fda.gov.tw/

報告大綱

簡介 | 專題分享 | 心得

- AOAC簡介
- 過程
- 口頭論文
- 壁報論文
- 臺灣分會會議
- 照片分享
- 建議

簡介

衛生福利部 食品藥物管理署 Food and Drug Administration
http://www.fda.gov.tw/

簡介

AOAC International 簡介

- 非營利化學協會。
- 由美國農業部創立於1884年，原為The Association of Official Agricultural Chemists (簡稱AOAC)，於1991年更改為AOAC International 。
- 旨在發布有關食品過敏原、微生物、藥物殘留、污染物質及毒素等標準檢驗方法供全球檢驗機構參考。
- 全球目前共有17個分會

AOAC INTERNATIONAL
AOAC INTERNATIONAL TAIWAN SECTION

衛生福利部 食品藥物管理署 Food and Drug Administration

簡介

全球分會

歐洲地區
- ☐ 歐洲分會
- ☐ 低地分會：比利時、盧森堡和荷蘭

亞太地區
- ☐ 臺灣分會
- ☐ 日本分會
- ☐ 中國分會
- ☐ 印度分會
- ☐ 泰國分會

北美地區
- ☐ 中部分會
- ☐ 大西洋中部分會
- ☐ 加拿大中部分會
- ☐ 中西部分會
- ☐ 大平洋西北部分會
- ☐ 太平洋西南部分會
- ☐ 南部分會
- ☐ 南加州分會

非洲地區
- ☐ 撒哈拉以南非洲分會

中南美地區
- ☐ 中南美洲加勒比分會

簡介

會議地點

Denver, Colorado

- 丹佛市於1858年成立。
- 又被稱為里高城(The Mile-High City) 。
- 位於大麻合法化的州別。
- 運動隊伍：NBA金塊隊及MLB科羅拉多洛磯隊
- 西面有洛磯山脈、東面則有北美大平原。
- 常見的知名景點： 大藍熊、鐘塔及聯合車站。

簡介

過程

簡介

行程與工作紀要

日期	行程與工作紀要
9月7日	去程 臺灣桃園-美國洛杉磯-美國丹佛-會場
9月8日	參加「第133屆AOAC年會」：開幕及儀器展示
9月9日	參加「第133屆AOAC年會」：演講及壁報展示 Taiwan Section Business Meeting
9月10日	參加「第133屆AOAC年會」 演講及壁報展示、發表口頭論文
9月11日	參加「第133屆AOAC年會」 演講及壁報展示、發表壁報論文 返程 會場-美國丹佛
9月13日	美國舊金山-臺灣桃園

專題分享

衛生福利部 食品藥物管理署 Food and Drug Administration

http://www.fda.gov.tw/

專題分享

本次年會共計有**23個**專題、**近1百場**專題演講，摘錄部分場次內容：

- Wiley Award Symposium: Advances in Analytical Methods for Botanical Dietary Supplements and for Clinical Nutritional Assessment
- Recent Trends in Elemental Analysis Application
- New Blood 2019-Developing Methods for the Detection of Important Chemical Analytes, Residues an Contaminants

專題分享

日期	壁報論文主題
9月9日	Botanical and Dietary Supplements, Food Nutrition and Food Allergens, Food Authenticity and Food Fraud, and Miscellaneous
9月10日	Detection and Measurement of Natural Toxins, Agriculture and Environment, Cannabis, General Methods, Quality Assurance and Accreditation, and Performance Tested Methods℠
9月11日	Analysis of Foodborne Contaminants and Residues, Analysis of Non-Foodborne Contaminants and Residues, and Microbiological Methods

專題分享

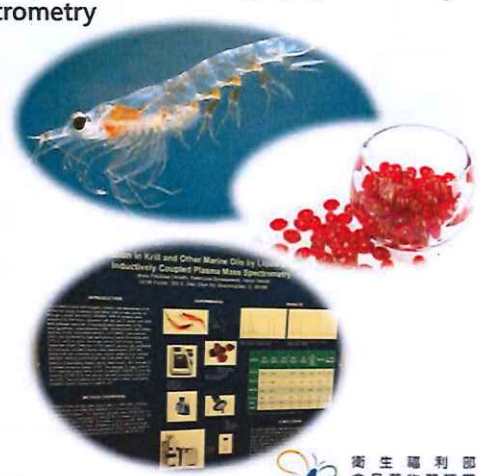
Wiley Award Symposium: Advances in Analytical Methods for Botanical Dietary Supplements and for Clinical Nutritional Assessment

專題分享

Recent Trends in Elemental Analysis Application

Topic: Arsenic speciation in Krill Oil by Liquid Chromatography Inductively Coupled Plasma Mass Spectrometry

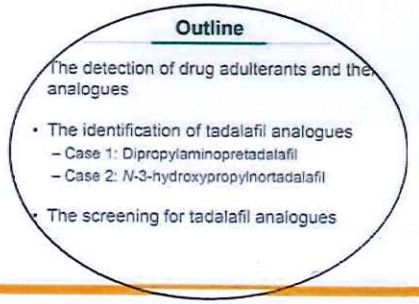
秤取檢體約0.25 g → 加入5 mL 0.28M硝酸及2.5 mL過氧化氫 → 微波消化法 → 以0.22 μm PVDF filter過濾 → HPLC/ICP-MS → 定容至50 mL



專題分享

口頭論文發表- The Detection and Identification of Tadalafil Analogues

- 李技士於「New Blood 2019-Developing Methods for the Detection of Important Chemical Analytes, Residues an Contaminants」時段發表。
- 闡述本署摻假西藥檢驗方法及如何發現新西藥類緣物成分。
- 以近期2個tadalafil類緣物的例子說明新西藥類緣物成分分離鑑定流程。
- 分享本署新開發的tadalafil類緣物快速篩檢方法。



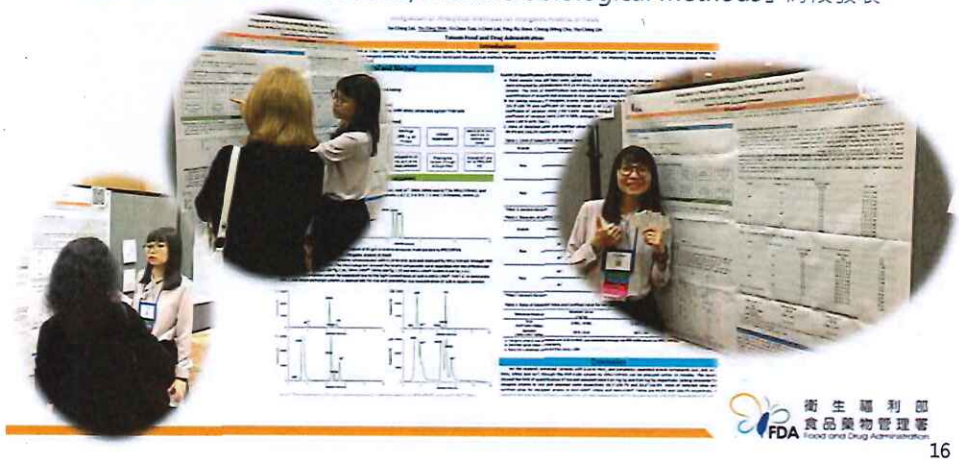
專題分享

		水產動物中食用油脂 無機砷之檢驗方法	NOW FOODS 磷蝦油中 無機砷之檢驗方法
檢測品項		無機砷	無機砷
檢體量		1 g	0.25 g
定量體積		20 mL	50 mL
前處理	使用試劑	10 mL 0.28 M硝酸溶液	5 mL 0.28M硝酸 2.5 mL 過氧化氫
	方式	超音波萃取法 80℃ 30 min(萃取2次)	微波消化爐
移動相	A	200 mM $(NH_4)_2CO_3$ (含3% (v/v)甲醇, pH 8.5)	2.5 mM $(NH_4)_2CO_3$ (含0.2 mM EDTA及1%甲醇, pH11)
	B	1 mM $(NH_4)_2CO_3$ (含3% (v/v)甲醇)	100 mM$(NH_4)_2CO_3$ (含0.2 mM EDTA及1%甲醇, pH11)
分析儀器		LC-ICPMS	LC-ICPMS

專題分享

壁報論文發表-Integration of Analytical Methods for Inorganic Arsenic in Food

- 施助理研究員於「Poster Presentation: Analysis of Foodborne Contaminants and Residues, Analysis of Non-Foodborne Contaminants and Residues, and Microbiological Methods」時段發表。



臺灣分會會議

- 每年AOAC年會皆會規劃專屬時段及場地提供AOAC臺灣分會舉辦「Taiwan Section Business Meeting」。
- 今年由臺灣分會呂廷璋理事長、方銘志秘書長及同仁舉辦臺灣分會會議，本次約有30人參加，多為任職於美國官方及民間機構的臺灣人。
- 會議期間皆以英語進行交流，首先由理事長介紹臺灣分會今年舉辦的活動及成果，並拋出食安議題供大家討論，現場並備有綠豆椪及鳳梨酥，以及具有紀念價值之臺灣造型金箔手工皂供與會者取用。

照片分享

會場入口

心得

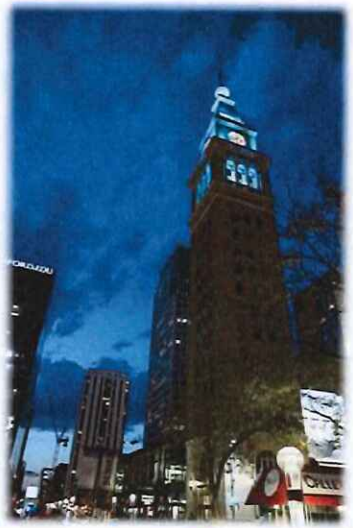
衛生福利部 食品藥物管理署 Food and Drug Administration

http://www.fda.gov.tw/

照片分享

Taiwan Section Business Meeting

照片分享



照片分享

建議

- 國際間各界開發之檢驗方法及品質管制一個互相交流的平台，本署研究檢驗組業務內容與其核心價值息息相關，**建議未來持續派員參與以瞭解國際間新興檢驗技術，為民眾食品安全作最前端的把關**。
- 近年國際間較受重視的議題包括非目標物檢測、天然藥物分析、NMR檢測磷蝦油中無機砷檢驗...等，未來**皆可提供本署做為參考依據，俾利精進檢驗方法**。
- 會中所發表之口頭論文及壁報論文，皆有來自不同國家及領域之專家學者前來討論，交流彼此意見及技術，**建議未來持續發表論文**。

恭請指教

衛生福利部 食品藥物管理署 FDA Food and Drug Administration

http://www.fda.gov.tw/