

出國報告 (出國類別：開會)

## 赴美國參加「2011 美國食品科技學會 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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出國期間：100 年 6 月 9 日至 6 月 16 日

報告日期：100 年 9 月 2 日

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## 摘要

本次奉派前往美國路易斯安納州紐奧良參加「2011 美國食品科技學會研討會」，本次研討會同時舉辦食品展，為期 4 天(100 年 6 月 11~14 日)，除了參加大會所舉辦的演講與食品展外，亦發表壁報論文，論文題目為「Simultaneous determination of 35 veterinary drugs in livestock, fishery product, and milk by UPLC-MS/MS」，獲得許多檢驗與食品科技發展之新知，可供國內食品相關產業及檢驗單位參考。僅提三點卓見供參：第一，積極派員參與此類國際研討會，收集各國食品檢驗與業界之發展趨勢；第二、增加檢驗人力並持續培訓專業檢驗人員，提升檢驗技術；第三、多鼓勵或贊助國內食品業參與國際之食品相關展覽，可將台灣之食品業推向國際。

## 壹、目的

「美國食品科技學會研討會」是每一個從事食品相關研究的人員，每年可以齊聚一堂的機會平台，亦是一個可以讓自己增廣見聞的最好機會。只要與食品領域相關之研究都會在此進行發表與交流，包含水產食品、生物技術、醱類、穀類食品、膳食營養補充品、食品發酵學、食品微生物學、食品化學、食品工程學、食品加工、法規、蔬菜水果產品、營養學、植物化學物質學、冷凍學、食品衛生安全...等，可獲得非常多之食品相關資訊。本人受派參加「2011 美國食品科技學會研討會」，除了學習各國之先進檢驗技術外，並以壁報發表本局研究成果，並期能建立與國際相關檢驗研究人員之聯絡溝通管道。

## 貳、過程

美國食品科技學會研討會 (IFT Annual Meeting) 為每年舉辦一次之國際性食品研討會，會議能提供現今流行之保健食品功能性探討與開發、食品科技與開發，以及食品衛生與安全，同時亦包含發展中之最新技術和未來可能發展層面和願景，因此本人將自己的研究成果摘要投稿於大會中，並有幸得到大會的接受函，能參加「2011 美國食品科技學會研討會」，並發表壁報論文 (poster)。本次 2011 年美國食品科技學會年會暨國際食品展於 6 月 11 日至 6 月 14 日舉行，本次年會於美國路易斯安那州紐奧良之 New Orleans Morial Convention Center 國際展覽會場舉行。在會議中至少有 15,000 來自全世界的學校、研究機構、食品公司、政府單位的人員參與，此外超過 100 個以上的 scientific sessions。在國際食品展部份，超過 900 個以上的單位參與此項展覽。會議從 6 月 11 日開始，展開一連四天的研討會暨食品展，會議開始時主要是讓會員辦理報到，取得會議相關手冊資料，同一時間已有部份的學術會議活動開始展開。6 月 11 日報到當天，拿到整個研討會之大會手冊後，利用一些時間去規劃會議期間想要參加之活動，由於大會安排許多的演講活動，在同一時間同時安排不同的研究內容，當下真的只能選擇對自己最有幫助與最感興趣的內容。美國食品科技學會年會是食品領域重要的年會之一，在參與人員的人數與展覽規模上，每年均保持一貫的高水準，可說是產、官、學的國際交流最好平台。此外，大會每年都會提供與會人員從飯店到會場之間的交通車接駁，因此從飯店到會場之交通是相當方便，也可以節省許多到會場的時間。

本次年會包含許多食品相關的研究主題，包括：水產食品、生物技術、醱類、穀類食品、膳食營養補充品、食品發酵學、食品微生物學、食品化學、食品工程學、食品加工、法規、蔬菜水果產品、營養學、植物化學物質學、冷凍學、食品衛生安全...等，內容包羅萬象，主要分為口頭宣讀、論文壁報以及特別演講等，可謂精采絕倫，身處於此會議中，更可體會出世界各國的科學家們在食品相關領域之努力，並藉此了解現今食品領域的研究發展趨勢，所以參加此次之研討

會真是獲益良多。在這四天當中能很充實且新奇的度過，累積許多新的想法與觀念。

本人在本次的會議中發表了一篇研究論文，以壁報形式發表，論文題目為「Simultaneous determination of 35 veterinary drugs in livestock, fishery product, and milk by UPLC-MS/MS」，可同時分析禽畜水產品中 15 項奎諾酮類與 20 項磺胺劑藥物，只要稱取適量檢體，加入含有 5% 甲醇的乙腈溶液進行均質與萃取，再利用無水硫酸鈉進行脫水及鹽析的作用，最後以乙腈飽和之正己烷溶液去除油脂，取乙腈層進行吹乾、回溶，即可利用 LC-MS/MS 進行分析。利用此方法進行分析，可節省許多時間及人力，提升檢驗量能。希望藉由此次研究論文發表機會，將研究成果與其他國際研究學者交流，提升自我的研究深度與國際視野。

## 參、心得

首先感謝本局長官推薦，本人有幸派赴美國參加 2011 年美國食品科技學會之國際性學術研討會，參加本次國際學術研討會，給了自己一個學習與成長之機會，能夠見識國際學術研討會之規模，各國學者齊聚一堂共同為學術研究而努力，讓我深刻體認到要繼續充實自己之學識，多累積自己的學術研究成果。期望未來能有機會繼續參加此類國際學術研討會，我相信從中能吸取不同之新知識與觀念，使自己能獲得更多之國際觀與想法。美國食品科技學會之年會是每一個從事食品相關研究的人員，每年可以齊聚一堂的機會平台，亦是一個可以讓自己增廣見聞的最好機會。本次年會吸引了超過 15,000 人食品相關人員參加，亦有超過 900 間以上廠商參加食品展的展出，整個會議內容非常的充實且精采。

論文壁報發表時，有國外學者問了一些問題，像是現在的檢驗方法都是朝向多重分析方法的方向作開發，有沒有比較過多重分析方法與單一方法的靈敏度、再現性及專一性等？我向國外學者解釋：「雖然我們沒有做這一類的評估，但是多重分析方法與個別檢驗方法比較，多重分析方法的靈敏度可能會不如單一的檢驗方法，但是多重分析方法可在一個檢驗方法中，同時檢測很多項藥物殘留，可節省時間、人力及降低試驗器具溶劑使用量，提升檢驗效率，亦可有更多的時間開發其他方法及進行相關檢驗，我們在檢驗方法開發上亦是朝向此方向進行。」，而他也相當同意我的說法，因為他們也正朝此方向前進，但是他們對於常有檢出微量藥物殘留且對人體健康影響重大之藥物，還是會保留它個別的檢驗方法，以備進行複驗及確認用。

在其他學者的研究發表中，有看到一篇比較特別的論文，是由上海海洋大學所發表的「Rapid Determination of Ractopamine in Swine Urine Using Surface Enhanced Raman Spectroscopy」，論文中以表面增強拉曼光譜儀檢測豬隻尿液中的 ractopamine 殘留量，其中探討兩種前處理方式：1. 檢體過濾後進行液/液萃取；2. 檢體過濾後進行液/液萃取，再經過 SPE 管柱淨化。評估後得到方法偵測極限分別為 800 ng/mL 及 400 ng/mL。我們現行公告方法之偵測極限為 0.3~0.5

ppb，雖然這篇論文之研究結果對我們在殘留量檢測來說有點高，用來當作改善我們現行方法之參考論文可行性不高，但是也提供給我們拉曼光譜儀一個新的應用方法。

大會亦安排多場不同主題研討會在不同的演講廳同時間進行，與會者可擇一參加。以下資訊與大家分享：

1. Dr. Ofori 提到有關牛奶中過敏原的檢測，現在很多人會對牛奶、羊奶及奶製品過敏，Dr. Ofori 利用 ELISA 進行奶類及奶製品中過敏原的測定，並將 ELISA 結果與 Western blot 的結果進行比較，發現 ELISA 既靈敏又可節省許多時間及試藥是不錯的檢驗工具。

2. Dr. Awad 分享有關蕃茄中陶斯松 (Chlorpyrifos) 及亞特松 (Pirimiphos-Methyl) 之檢驗，主要研究目的為開發靈敏解快速的檢驗方法，並做田間試驗，評估施藥後於開放田間及儲存期間藥品消退或其他變化情形。研究結果發現於開放田間中陶斯松在施藥後 1 天即消退 80%，14 天後即無法檢測到，亞特松則是施藥後 24 天即無法偵測。

3. 有關多重抗藥性沙門氏菌(multi-drug resistant Salmonella)的研究，講者自 33 種的食品進行菌株的分離，研究發現從 13% 的食品中可分離出多達 22 種的沙門氏菌，再進行抗藥性分析，其中利用的許多新興的生化分析方法，包含 Real-time PCR、16s rDNA 定序分析及生物晶片快速篩檢法等。

4. 在奈米科技方面，除了持續研究奈米化顆粒的不同物化性質之外，更發表了許多奈米包材用於食品保鮮、抗菌、抗氧化等功效。此外亦討論到奈米材料之安全性，因顆粒被奈米化，其可能通過血腦障壁，另也因表面積變大增加了暴露劑量的危險，因此奈米食品與藥品的安全性都須重新評估。

5. 由於現代人對健康越來越重視，天然營養補充品的市場也越來越大，這個現象在國內也有發現，因此很多學者都投入 nutraceuticals 的相關研究，例如天然植物精油萃取、多酚化合物之抗氧化與抗癌特性等，且在食品展中也看到許多跟保健食品相關的廠商。

6. 大會之一係針對食品履歷進行介紹，要從農場將資訊串連到餐桌上，需

要很多人力、物力及資訊系統的整合，以台灣的食品業來看，大多為家庭式小企業，相對資金與人力較為不足，可能需要較多的資源補助及輔導才有辦法徹底施行。

除了學術研究上之收穫外，在食品展中亦有很多收穫。在場中發現展出的多為食品添加物之原料廠商，而且都強調這些添加物都是由天然動植物中所萃取而來的，並不是化學合成；國內的食品展大多是以終產品呈現，較少原料商參展。另也發現展場中有一個區塊皆是大陸的食品廠商，詢問後得知，他們是由政府及工協會贊助出國參展的，大約有 100~150 家，而且這樣的贊助方式已經持續很多年了，看來是大陸政府有計劃性的要將大陸食品推向國際化。

## 肆、建議

僅提三點卓見供參：第一，近年保健食品市場蓬勃發展與食品衛生安全為世界各國首要注重的議題，因此美國食品科技學會研討會(IFT Annual Meeting)中主要議題含蓋的有營養學、生物技術、食品化學、食品微生物學、食品法規及肉品加工等等。建議積極參與此類國際研討會，除可與世界各地的專家學者互動，建立交流平台外，更可獲知最新的科技發展，對日後自己的研究發展工作有絕對的好處；第二、國人對食品衛生安全越來越關注，相較於其他國家投注於食品安全之經費與人力，建議可再增加檢驗人力並持續培訓專業檢驗人員，提升檢驗技術，為民眾食的安全而努力；第三、在食品展中看到各式各樣的食品業者，也驚嘆食品業的蓬勃發展，領域已從食品跨足到機械、加工、醫藥、衛生、保健、化學、工程等不同領域的整合，建議可多鼓勵或補助國內食品業參與此類國際食品展覽，可提高國家知名度，也可增廣見聞，獲得食品也最新發展情勢，有助於國內食品產業之發展，亦可將台灣食品業推向國際，增加外銷機會與產值。

## 附錄一、議程表



# Program & Exhibit Directory

June 11-14, 2011  
New Orleans Morial Convention Center  
New Orleans, LA USA

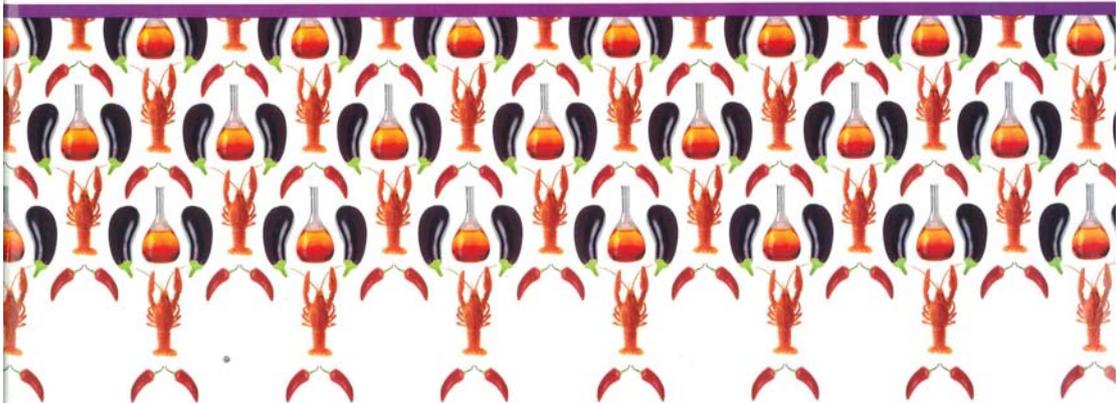


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- **What's New!:** A Listing of New Products & Services



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TOPICS - AT - A - GLANCE

SESSIONS CATEGORIZED BY TRACK

This grid references all sessions by track. At time of submission, session organizers indicated a primary track and sometimes a secondary track (session numbers indicated in bold below) for their session. These classifications are subject to change. Detailed session information may be found in the Scientific Program section of this tab.

Track	Sunday 7:15AM	Sunday 10:30AM	Sunday 1:00PM	Sunday 3:00PM	Sunday 4:30PM	Monday 8:15AM	Monday 10:30AM	Monday 1:15PM
Education & Professional Development	013	066				119, 114	129, 137	138
Food Chemistry	014	062				119	130	
Food Engineering	015	067, 068, 071			101	111	131, 133	
Food Health & Nutrition	016, 026, 021, 022, 025	073, 074, 075, 076, 078, 079	085, 086, 087, 093, 090, 091, 092, 093, 094, 095, 096, 097, 098, 099, 100			115, 117, 118, 121, 124	136, 137, 142	
Food Microbiology		095, 097				112	133	
Food Processing & Packaging	007	015, 017, 021, 022, 026	050, 045, 046, 047, 048, 049, 050, 051, 052, 053, 054, 055, 056, 057			111, 112, 119, 120	134, 138, 139	
Food Safety & Allergens	017, 018, 024	028, 027	031, 032, 033, 034, 035, 036, 037, 038, 039, 040, 041, 042, 043			114, 115, 116	132, 134, 135	
Product Development & Innovation	006, 007	013, 014, 016, 020, 021, 022, 023	041, 042, 046, 047, 049, 048, 050, 051, 052, 053, 054, 055, 058, 057	101	100	118, 119, 120, 123	131, 135, 136, 138, 138, 141	
Public Policy, Food Laws & Regulations	023, 024	074, 078				117, 121	140	
Sensory Science	025	028, 029, 060, 061				127	141	
Sustainability	060	026				124	142, 163, 164, 165, 166, 167, 168	

Scientific Program Tracks

Symposia, workshops, panel discussions, sunrise, poster, oral, and general sessions have been categorized into new tracks to help you efficiently choose from the diverse scientific and educational offerings available to you.

Education & Professional Development

This track will help enhance professional skills for career growth and the effective delivery of food science education. Topics such as project management, communication skills, and career management will be discussed.

Food Chemistry

This track will showcase presentations of novel basic and applied research relating to chemistry and the analysis of food.

Food Engineering

This track will present basic research pertaining to food engineering, including measurement, modeling, and control of food processing systems.

Food, Health & Nutrition

This track will present applied nutrition research, new science, and market trends associated with the development of foods to maintain and/or improve health.

Food Microbiology

This track features sessions highlighting cutting-edge research in food microbiology, including detection and quantification methods, survival of microorganisms in the food and processing environment, characterization of emerging pathogens, and more.

Food Processing & Packaging

This track will focus on food processing and packaging in order to improve quality, efficiency, and sustainability, or to lead to development of new products, processes, packaging materials, and techniques.

TOPICS - AT - A - GLANCE

Track	Monday 1:30PM	Monday 3:30PM	Monday 4:30PM	Tuesday 7:15AM	Tuesday 10:30AM	Tuesday 1:00PM	Tuesday 1:30PM
Education & Professional Development	051, 061	192				217	253
Food Chemistry	114, 129		164	211, 242	232, 251, 254	269, 274	
Food Engineering	142					249	267
Food Health & Nutrition	160, 181, 183			213	218, 224, 225, 229	251, 254, 256, 257, 258	272, 273, 276
Food Microbiology	173	193, 194, 195, 196, 197, 198, 199, 200, 201, 202, 203, 204, 205				249	266, 267, 270
Food Processing & Packaging	182, 183		265	278, 279	280, 289	284, 295, 275, 271	
Food Safety & Allergens	174, 175, 176, 179, 184			212, 213	231, 231, 238	251, 252	278, 271, 277
Product Development & Innovation	177, 182, 183, 185				235, 236, 227	256	273, 274, 275
Public Policy, Food Laws & Regulations	178, 184	206			231, 228	257	271, 272, 274, 277
Sensory Science	185				279	288	278
Sustainability	186				282	293	

Food Safety & Defense

This track will address current/hot topics in a number of areas, including food safety and defense systems, and their components (e.g., risk assessment and management, traceability, HACCP, product testing, auditing, crisis management, recall, and regulations).

Product Development & Ingredient Innovations

This track will focus on the primary aspects of the development and introduction of new food and beverage product innovation to the global marketplace. This category includes top-level innovations, consumer research, product innovation, consumer and related business information, as well as the technical and marketing aspects of product development.

Public Policy, Food Laws & Regulations

This track will discuss the practical, real-world implications for the food industry of legislative, regulatory, and judicial developments on both a national (U.S.) and international scale.

Sensory Science

This track will address the latest advancements in the science of sensory and consumer research as they apply to food, and will explore such topics as new and improved sensory methods, sensory's role within the food product development process, the relationship between sensory and market research, and more.

Sustainability

This track will showcase the efforts of food industry, academia, and government to create a more sustainable food supply.

# Simultaneous determination of 35 veterinary drugs in livestock, fishery product, and milk by UPLC-MS/MS

Yu-Shan Yu, Pei-Ju Chou, Yuan-Fong Ku, Su-Hsiang Tseng, Shu-Chu Su, Yang-Chih Shih  
Food and Drug Administration, Department of Health, Executive Yuan, Taiwan

## Introduction

Veterinary drugs are widely used for the treatment and prevention of disease in livestock and fishery. Inadequate use of these drugs can leave residues in edible tissues, which can cause health problems. In order to ensure human food safety, the Department of Health (DOH) in Taiwan has set maximum residue limits (MRLs) for veterinary drugs. Monitoring programs for veterinary drugs in livestock, fishery product and milk have been the routine work of food safety related authorities. For this type of target analysis, multi-residue analytical methods are preferred to reduce workload and costs.

Quinolones constitute the main group of antibiotics used both in human and veterinary medicine for therapeutic purposes as effective against both Gram-positive and Gram-negative bacteria. The mechanism of action of quinolones is bactericidal; in most cases they inhibit the DNA enzyme gyrase of the bacteria cell, which is indispensable in the duplication of DNA. However their extensive administration may lead to residues in edible tissues.

Sulfadiazines are often used to prevent and control a number of diseases in veterinary practice. They are administered orally or mixed with animal feed. About 30 sulfadiazines have been found so far and some of them have a good antibacterial effect. However, such treatment leads to the sulfadiazine residues in edible tissues. Sulfadiazines will not be accumulated in humans because the leave the body after acetylation. The abuse of sulfadiazines can induce various bacteria to generate strong resistance to drugs and greatly harm human health, which may lead to many diseases such as thyroid cancer, anaplastic reaction and resistance to drugs.

The aim of this study was to optimize and establish a reliable and rapid UPLC-MS/MS method for the simultaneous analysis of 15 quinolones and 20 sulfadiazines in livestock, fishery product and milk using multiple reaction monitor (MRM) mode for detection.

## Materials & Methods



Muscle



Liver



Fishery product



Milk

### Sample preparation

Five grams of homogenized sample was weighed into a 50 mL centrifuge. To perform the extraction process, 25 mL of acetonitrile with 5% methanol was added and the sample was homogenized with high speed homogenizer for 3 minutes. The 10 g of anhydrous sodium sulfate was added. The sample was shaken vigorously for 10 minutes, then centrifuged at 4000 rpm for 10 minutes at 4°C. The supernatant was transferred into a 250 mL separatory funnel. The pellet was re-extracted by above process, and supernatants were combined together. After de-fatted by 30 mL of acetonitrile-saturated heptane, the sample solution was liquid-liquid extracted. The collected acetonitrile phase was dried by the rotary evaporator. The residue was dissolved with 1 mL of 20% methanol. Finally, the sample was filtered via 0.22 µm microporous film and injected into the UPLC-MS/MS system.

### UPLC conditions

Liquid Chromatography:  
Alliance® ACQUITY UPLC System

Column:  
ACQUITY UPLC HSS T3  
(2.1x100 mm, 1.8 µm)

Mobile phase:  
A: Water with 0.1% formic acid  
B: Methanol with 0.1% formic acid

Flow rate: 0.3 mL/min  
Injection volume: 10 µL

Gradient LC Conditions:

Time (min)	A (%)	B (%)
0.0 → 2.0	95 → 95	4 → 5
2.0 → 3.0	95 → 80	5 → 20
3.0 → 6.0	80 → 75	20 → 25
6.0 → 8.6	75 → 75	25 → 27
8.6 → 14.5	73 → 63	27 → 37
14.5 → 14.7	63 → 0	37 → 100
14.7 → 18.7	0 → 96	100 → 4
18.7 → 20.0	96 → 96	4 → 4

### MS/MS conditions

Waters Micromass Quattro Premier™ XE System

[ESI(+)]

Capillary voltage: 3.3 kV

Source temperature: 120 °C

Dissolution temperature: 450 °C

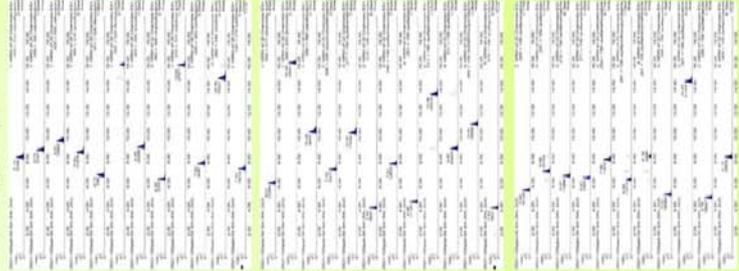
Disolution gas flow rate: 800 L/hr

Cone gas flow rate: 100 L/hr

Multiplier: 650 V

## Results

Figure 1. MRM chromatograms of 15 quinolones and 20 sulfadiazines (100 ng/mL).



Samples including chicken muscle, chicken liver, fish, shrimp and milk were fortified with 0.1 and 0.05 ppm. The results show the satisfactory recoveries and repeatability. The recoveries of 35 veterinary drugs were mostly ranged 60~120% and the coefficients of variation were below 20%, and LOQs were ranged 0.001~0.005 ppm in tested matrices. The MRM parameters, correlation coefficient, recoveries, CV, LOQs of 35 veterinary drugs in pork shown in Table 1. The MRM chromatograms of 15 quinolones and 20 sulfadiazines (100 ng/mL) were shown in Figure 1.

Table 1. MRM parameters, correlation coefficient, recoveries, CV, LOQs of 35 veterinary drugs in pork (spiked at 0.05 ppm, n=5)

Compound	Quantification (ng)	Quantification (ng/mL)	Correlation coefficient (r <sup>2</sup> )	Recovery (%)	CV (%)	LOQ (ppm)
Ciprofloxacin	332 ± 314	332 ± 231	0.998	71.2	12.2	0.001
Danofloxacin	358 ± 240	358 ± 283	0.996	71.1	4.3	0.001
Difloxacin	400 ± 356	400 ± 269	0.998	80.1	5.5	0.001
Enoxacin	360 ± 316	360 ± 245	0.998	92.8	8.8	0.001
Flumequine	370 ± 326	370 ± 280	0.994	79.2	5.6	0.001
Flumequine	282 ± 244	282 ± 202	0.996	92.1	86.0	0.001
Lomefloxacin	383 ± 345	383 ± 308	0.999	71.4	70.3	0.001
Morfloxacin	333 ± 275	333 ± 217	1.000	72.3	5.3	0.001
Nalidixic acid	233 ± 215	233 ± 187	0.995	63.6	7.4	0.001
Norfloxacin	320 ± 302	320 ± 276	0.998	76.3	7.5	0.001
Oxolinic acid	282 ± 244	282 ± 218	0.999	98.7	5.0	0.001
Pivofloxacin	334 ± 316	334 ± 233	0.998	78.5	6.5	0.001
Pipemidic acid	304 ± 277	304 ± 189	0.998	51.0	8.3	0.001
Prometic acid	289 ± 243	289 ± 271	0.994	80.2	7.6	0.001
Sarafloxacin	386 ± 368	386 ± 242	0.993	83.1	9.2	0.001
Sucramylsulfazobol	366 ± 266	366 ± 192	0.997	74.9	9.0	0.001
Sulfacetamide	271 ± 156	271 ± 92	0.997	102.1	75.1	0.001
Sulfacarbonyl	271 ± 156	271 ± 92	0.990	70.9	78.8	0.005
Sulfadiazine	285 ± 156	285 ± 92	0.999	95.3	3.0	0.005
Sulfadiazine	251 ± 156	251 ± 92	0.996	107.6	4.9	0.001
Sulfadiazine	311 ± 156	311 ± 92	0.999	97.5	7.8	0.001
Sulfadiazine	295 ± 156	295 ± 92	0.997	108.7	12.2	0.001
Sulfadiazine	271 ± 156	271 ± 92	0.991	112.8	6.8	0.005
Sulfadiazine	265 ± 156	265 ± 92	0.997	96.8	5.6	0.001
Sulfameter	281 ± 156	281 ± 92	0.990	84.5	5.5	0.001
Sulfamethazine	279 ± 156	279 ± 188	0.995	109.0	11.6	0.001
Sulfamethazole	271 ± 156	271 ± 92	0.996	92.5	6.5	0.001
Sulfamonomethoxazole	254 ± 156	254 ± 92	0.991	94.5	6.5	0.001
Sulfamonomethoxazole	281 ± 156	281 ± 92	0.996	98.8	14.9	0.001
Sulfamonomethoxazole	281 ± 156	281 ± 92	0.997	106.4	17.5	0.001
Sulfapyridine	260 ± 156	260 ± 92	0.991	102.2	6.4	0.001
Sulfquinolone	301 ± 156	301 ± 92	0.997	81.9	8.9	0.001
Sulfisoxazole	256 ± 156	256 ± 92	0.991	91.5	5.1	0.001
Sulfisoxazole	288 ± 156	288 ± 92	0.995	95.4	4.5	0.001

## Conclusion

This study described and validated a UPLC-MS/MS methods for simultaneous quantification of 15 quinolones and 20 sulfadiazines in livestock, fishery product and milk. The LODs were found to be low enough to determine in sample below the permissible MRLs established by Department of Health (DOH) in Taiwan. In conclusion, the proposed method is considered satisfactory for routine monitoring of veterinary drugs residues in livestock, fishery product and milk.

### 附錄三、論文集掃描



by Soxhlet and tested for oil contents, fatty acid profiles, and tocopherols. Hazelnut samples were extracted with 50% acetone and examined for total phenolic contents (TPC), antioxidant activities using ORAC, and DPPH. Extracts were also tested on the HT-29 human colon cancer cell line for determination of antiproliferative effects. The Turkish hazelnuts contained over 65% oil while the Ore. roasted sample contained 43.8%. The primary fatty acid was oleic acid, comprising 76.7 g/100 g oil in the Ore. grown and 83.3 g/100 g oil in the Turkish grown samples. The TPC values were 102.16 and 91.4 mg gallic acid equivalents/g for the Ore. roasted hazelnut skin and Turkish roasted hazelnut skin respectively and were 30 to 100 times higher than their respective meals without skin. The Turkish roasted hazelnut skin had the highest ORAC value of 1166.3 Trolox equivalents (TE)  $\mu\text{mol/g}$  sample (TE  $\mu\text{mol/g}$ ) and was 36 times higher than the Turkish roasted hazelnut with no skin. The range of DPPH  $\text{ED}_{50}$  was from 0.075 to 118.2 mg sample equivalents/mL among the samples. The Ore. roasted hazelnut skin and Turkish raw hazelnut no skin exhibited the strongest and weakest ability to reduce DPPH, respectively. At 6 mg/mL media Ore. roasted hazelnut skin extract significantly inhibited the growth of the HT-29 cells by 96% following 4 d of treatment, and no effect was seen from the Turkish roasted skinless hazelnut extract. The Turkish raw hazelnut had significantly higher tocopherol contents and antioxidant activities compared to the Ore. roasted variety, which may be explained by chemical changes during heating or possibly the total oil-to-flour ratio.

### 089-13

#### Modulation of bulk physicochemical properties of emulsions by hetero-aggregation of oppositely charged protein-coated lipid droplets

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Structural design principles can be used to create novel food properties, such as rheology, optical properties, stability, flavor profile, or nutrition. Functional micro-clusters can be assembled from oppositely-charged protein-coated lipid droplets. Micro-cluster characteristics can be controlled by precisely controlling the pH and ratio of different kinds of particles. In our study, micro-clusters were formed by mixing a 10% oil-in-water emulsion containing  $\beta$ -lactoglobulin-coated lipid droplets with another 10% oil-in-water emulsion containing lactoferrin-coated lipid droplets. Under a particular pH range (pH 5 to 8.5),  $\beta$ -lactoglobulin and lactoferrin have opposite charges, which promotes micro-cluster formation. The influence of pH, ionic strength, and dilution on micro-cluster properties was characterized by  $\zeta$ -potential, particle size, and confocal microscopy measurements. The net charge and size of the micro-clusters depended on pH and the ratio of  $\beta$ -lactoglobulin- to lactoferrin-coated lipid droplets. The largest clusters tended to be formed when their net charge was close to zero. The addition of NaCl to increase the ionic strength caused a decrease in micro-cluster formation, which was attributed to screening of the electrostatic attraction between lactoferrin- and  $\beta$ -lactoglobulin-coated lipid droplets. Systems with extensive micro-cluster formation tended to have gel-like characteristics, which was attributed to the formation of a three-dimensional network of aggregated droplets. These results suggest that the rheology and stability of colloidal dispersions consisting of mixtures of negatively and positively charged protein-coated oil droplets can be modulated by varying the ratios of the two types of droplets. These micro-clusters may provide useful for modifying the functional properties of foods.

### 089-14

#### Simultaneous determination of 35 veterinary drugs in livestock, fishery product, and milk by UPLC/MS/MS

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A simple, selective, and fast multi-residue method was developed to determine 35 veterinary drugs (15 quinolones and 20 sulfadiazones) in livestock, fisher, and milk by an ultra-high-pressure liquid chromatography coupled to tandem quadrupole mass spectrometry. Samples were extracted with methanol/acetonitrile solvents (5 : 95, v/v) and defatted by partition with acetonitrile-saturated hexane. The quinolones and sulfadiazones from the extracts were separated on an ACQUITY UPLC T3 column using a gradient elution. Samples were detected by electrospray ionization in positive ion mode with MRM, and mass spectrometric conditions were optimized in order to increase sensitivity and selectivity. The developed method was validated in terms of linearity, trueness, precision, limit of detection (LOD) and quantification. The values of recoveries ranged, coefficients of variation (CV), and LODs from all matrices were 60.4~119.3%, 5.7~20.2%, and 0.001~0.005 ppm, respectively. Finally, 32 real samples were analysed via this method, and no veterinary drugs were detected among them. Hence, this satisfactory method can be applied in the routine monitoring of veterinary drugs residues in livestock, fishery product, and milk.

### 089-15

#### Comparison of the antioxidant activity of four commercial unifloral honey samples obtained from Portugal

P. Ulloa, V. Gonçalves, T. Cavaco, A. Figueira, A. Brigas, Univ. do Algarve, Faro, Portugal, Email: pablo.ulloa.f@gmail.com.

Honey and derivatives have been used since prehistoric times, because of their biological and therapeutic actions. It is a source of antioxidants, proven to be effective against deteriorative oxidation reactions.

The objective of this work was to determine the antioxidant capacity of four unifloral honey samples Citrus (orange blossom, L-1), Helianthus (sunflower, G-1), Rosmarinus (rosemary, R-1) and Arbutus (strawberry-tree, B-1) that were obtained commercially. Some quality parameters namely moisture, color, hydroxymethylfurfural (HMF), and diastase activity (DN) were determined. In addition the antioxidant capacity (DPPH, expressed in mg of ascorbic acid/100g of honey) and phenol content (expressed in mg of gallic acid/100g of honey) were analyzed.

Moisture content of the all samples showed values lot of sample L-1 was classified as extra light amber (34-50 m. amber (50-85) and samples G-1 and B-1 classified as dark a 114) according to White (1984). Regarding the DN parameter higher than 8 (Gothe unit), which is in accordance to the leg. only three of the samples showed values below the 40 meq/L Legislation.

Results for the antioxidant capacity and phenol contents were values for the B-1 sample (15.6 and 81.0, respectively), when compared with other samples. A correlation was found (linear regression) between phenol content and antioxidant capacity ( $R^2=0.948$ ), with higher values of phenol content by samples with more antioxidant capacity. The B-1 sample is known as "bitter honey"; this isn't very appetizing for the consumer, in contrast with all the other samples analyzed. These results can generate an added value to this type of honey (presence of bioactive compounds), which might result in a greater demand from the population.

### 089-16

#### The effect of cultivating conditions of radish sprouts on sulforaphane content

M. Shimizu, M. Akao, F. Shinmachi, A. Noguchi, I. Hasegawa, H. Kumagai, Nihon Univ., Fujisawa-shi, Japan, Email: bun-motomi@cosmos.ocn.ne.jp.

In our previous study, we have found that the amount of 4-methylsulfinyl-3-butenyl isothiocyanate (sulforaphane) from radish sprouts was approximately 25 times higher than that of 4-methylsulfinylbutyl isothiocyanate (sulforaphane) from broccoli sprouts. Sulforaphane has similar structure to sulforaphane, and had almost the same activity to induce phase II enzymes as sulforaphane. Localization analysis of sulforaphane in radish sprouts indicated that the leaf mainly contained sulforaphane in the plant body. When sprouts were cultivated in water under short irradiation, the amount of sulforaphane retained high. As sulforaphane contains sulfur (S) and nitrogen (N) in its structure, the amount of S and N in cultivation solution may affect its content. Therefore, in this study, we examined if alteration in S and N composition in Hogland's solution might affect sulforaphane content. Moreover, we also examined the influence of plant physiological status on sulforaphane content.

Radish sprouts were cultivated with different compositions of S and N in Hogland's solution under light or dark conditions. Light/dark condition was controlled by tin foil during the whole period. Irradiation started after 4 days of cultivation. Sulforaphane content in sprouts was measured by HPLC analysis. Physiological status of sprouts was evaluated by the 2,3,5-triphenyltetrazolium chloride (TTC) reducing activity.

Under the light condition, sulforaphane content decreased with the increase in nitrate salt concentration, while it increased with the increase in ammonium salt concentration. Sulforaphane content in sprouts cultivated under the dark condition was higher than that cultivated in water or with nitrate salt under the light condition. TTC reducing activity was inversely related to sulforaphane content. Therefore, our results suggest sulforaphane content would decrease with the activation of plant metabolism.

### 089-17

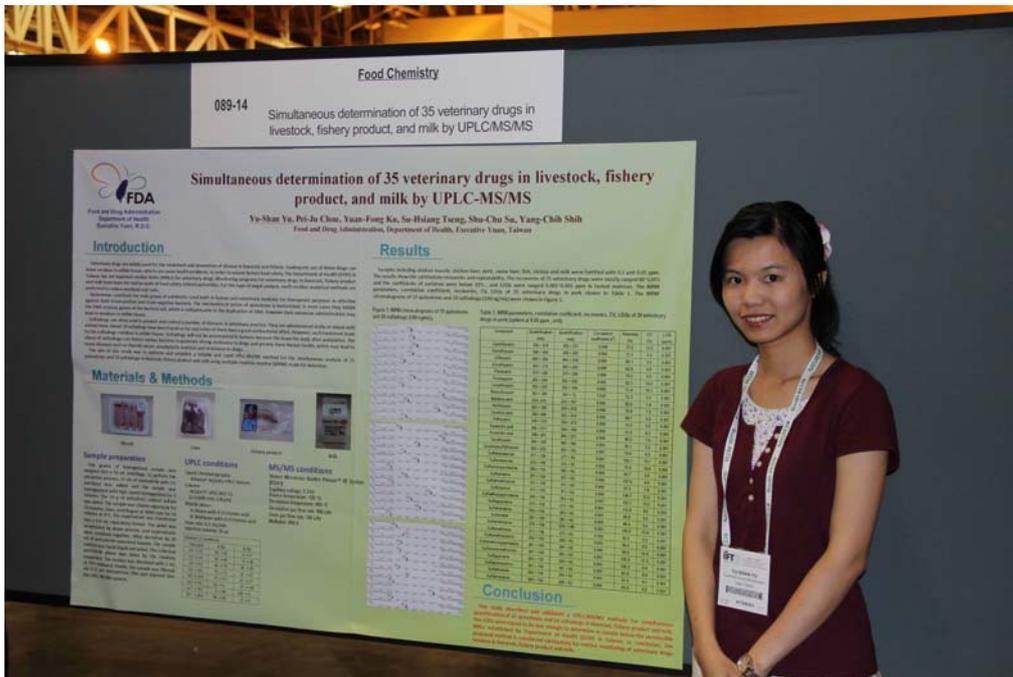
#### Determination of antioxidant capacity and bioactive compounds from the passion fruit (*Passiflora mollissima*) grown in Colombia: An alternative as a natural additive microencapsulated by spray drying

A. Gil, L. Alzate, D. Sánchez, A. Soto, A. Restrepo, M. Alvarez, L. Millan, Corp. Univ. Lasallista, Caldas, Colombia; B. Rojano, Univ. Nacional de Colombia, Medellín, Colombia; C. Restrepo, D. Rodas, Enviado, Colombia; N. Sanchez, Univ. Nacional de Colombia, Caldas, Colombia, Email: magil@lasallista.edu.co.

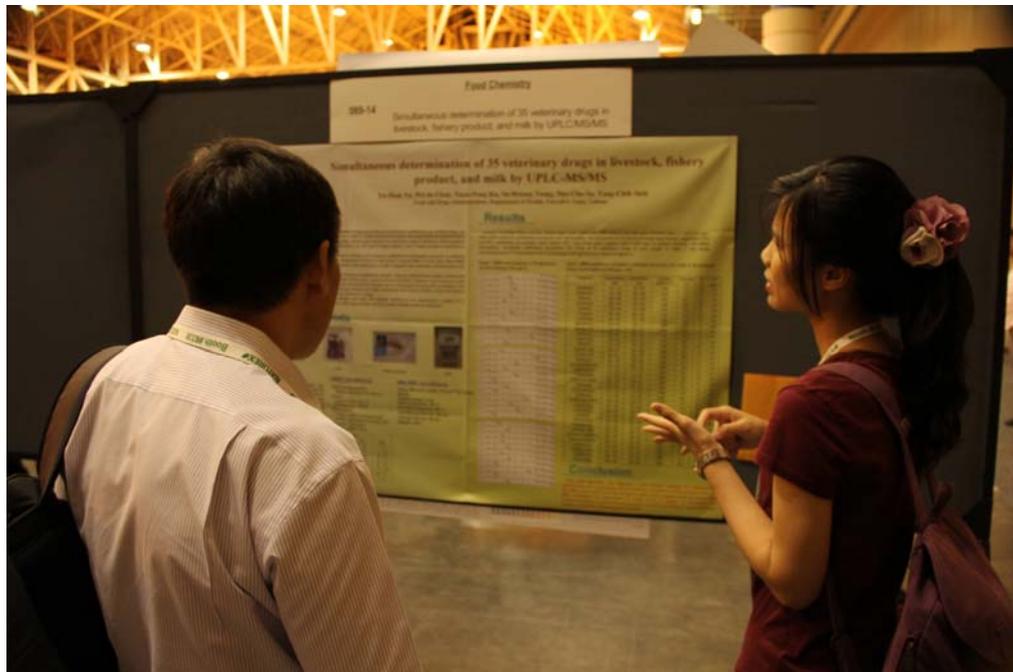
The passion fruit (*Passiflora mollissima*) is an exotic fruit from Colombia, with a high content of phenolic compounds, that is why it is one of the best natural sources of antioxidants and is an alternative to replace synthetic additives. Their antioxidant capacity has only been reported by FRAP. In addition there are few scientific studies that report the contents of the metabolites responsible for the antioxidant function of the passion fruit to use it in different food matrices and control the variables that affect its stability. One of the best ways to preserve the antioxidant activity of this fruit is microencapsulating by spray-drying. The aim of this study is to quantify the antioxidant capacity and bioactive compounds before being microencapsulated to offer as an alternative natural additive. The fruit was subjected to two thermal treatments before testing to ensure safety: blanching (3 min.), pasteurized (92°C). The pulp was obtained by a mechanical pulper to be characterized physicochemical and microbiologically. The antioxidant capacity was evaluated by ABTS, FRAP, DPPH and total phenols. The bioactive compounds were quantified by HPLC. Microencapsulation was carried out with maltodextrin and lecithin to 140°C and 160°C, 600L/h and 10mL/min. The safest treatment according to microbiological results was the pasteurization (below thresholds), also for the best yield (60%) and the results of its antioxidant capacity (DPPH: 6.6320234  $\mu\text{mol}$  Trolox/g; ABTS: 187, 6431682  $\mu\text{mol}$  Trolox/g; FRAP: 134633.4584 mgAA/100g, Total Phenols: 88.62216429 galic ac. mg/g), maybe due to the phenols were condensed before treatment. Bioactive compounds found were (Chlorogenic acid 9.450 mg/mL and Acid Coumaros 31.052 mg/mL) values close to the coffee. The microcapsules should be submitted to SEM and verify the efficiency of the total phenols encapsulated using the Fagen's method. The results showed a higher antioxidant capacity can make passion fruit a potential raw material for natural additives industry.

Functional properties of light for up-irradiation, using a resonance frequency of 100 kHz. H. Jang, N. Kim, Email: hjang@knu.ac.kr

## 附錄四、照片分享



壁報發表



有其他的學者對我們的研究有興趣，討論 poster 內容細節的部分。



食品展中的香料廠商，展示香料的萃取原料。



食品展中有一個區塊，皆是來自大陸的食品業者。

## 附錄五、心得分享投影片



中華民國精彩一百

藥事安全 食在安心



# 2011年 美國食品科技學會年會暨食品展

2011年6月9日~16日

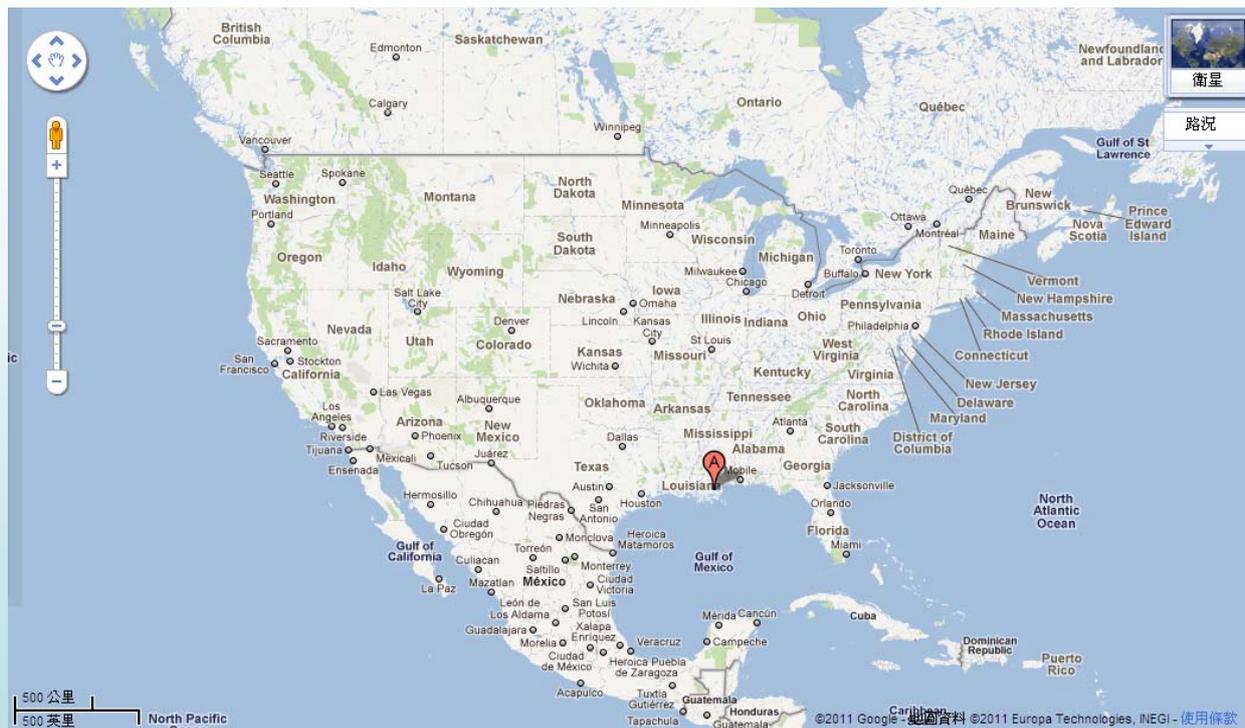
研究檢驗組

尤譽姍

2011年8月16日

## 前言

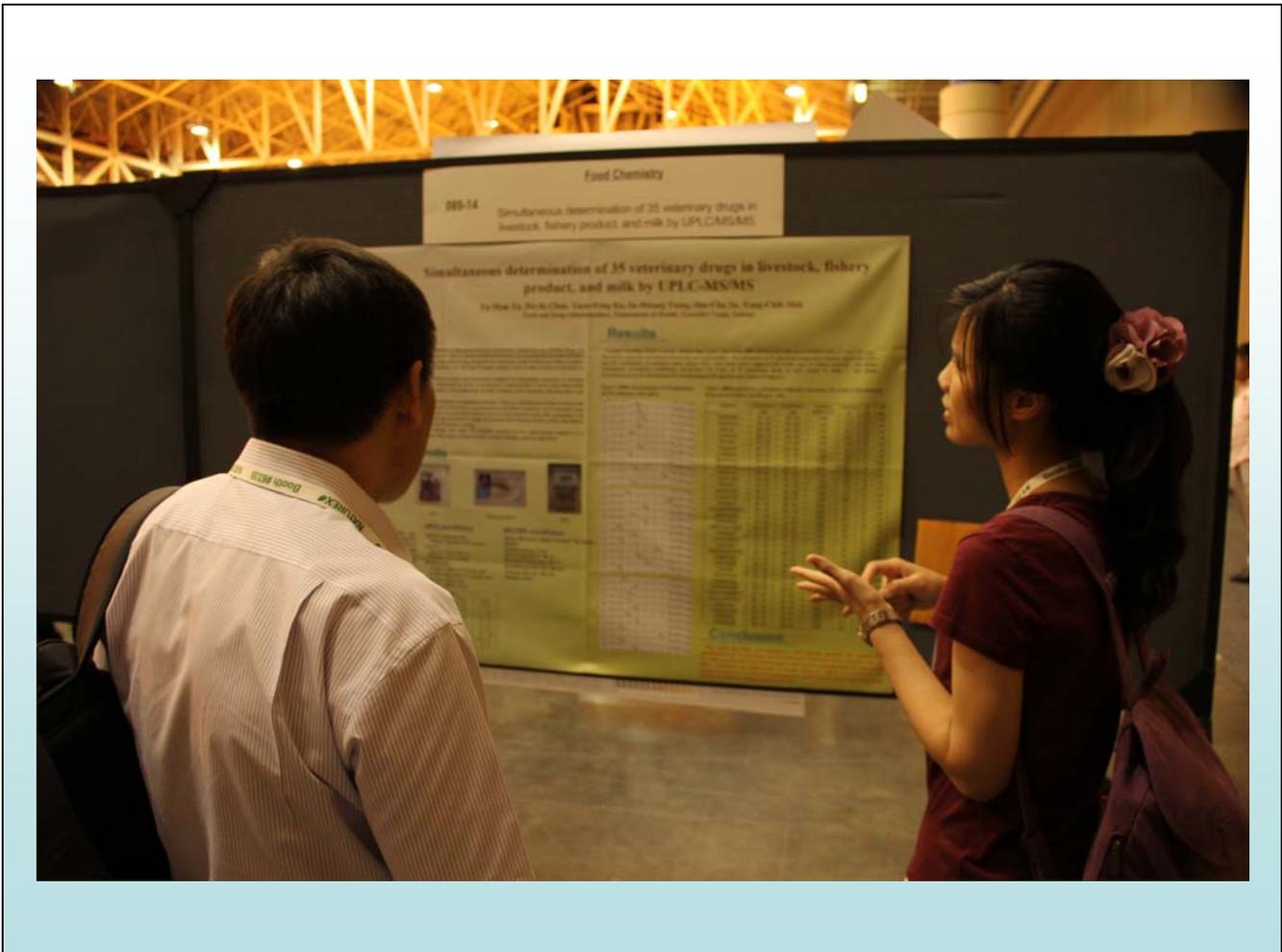
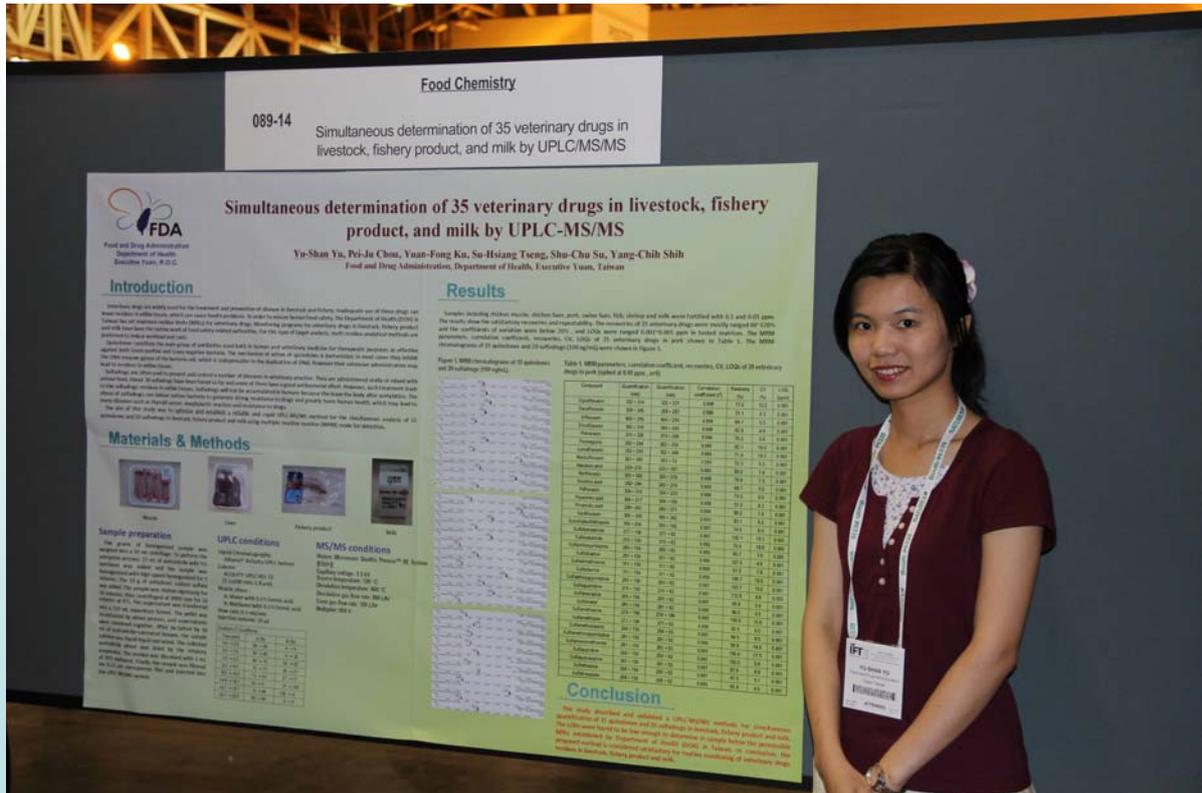
- 美國食品科技學會(Institute of Food Technologists, IFT)為國際知名之食品相關組織，今年在美國路易斯安那州新奧爾良(New Orleans)舉辦年會暨食品展，期藉由本次會議收及食品相關資訊，了解國際間食品安全及食品檢驗技術之最新發展趨勢。



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## Certificate of Participation

This certifies that

YU-SHAN YU, PEI-JU CHOU, YUAN-FONG KU, SU-HSIANG TSENG,  
SHU-CHU SU, YANG-CHIH SHIH

**SIMULTANEOUS DETERMINATION OF 35 VETERINARY DRUGS IN  
LIVESTOCK, FISHERY PRODUCT, AND MILK BY UPLC-MS/MS**

attended the 2011 IFT Annual Meeting & Food Expo® June 11-14, 2011  
held in New Orleans, Louisiana, USA

Heather C. Joireman, CMP, Meetings Manager  
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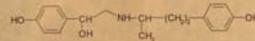


## Rapid Determination of Ractopamine in Swine Urine Using Surface Enhanced Raman Spectroscopy

Fuli Zhai, Keqiang Lai, Chunying Li, Yungang Shen, Yiqun Huang  
College of Food Science and Technology, Shanghai Ocean University, Shanghai, China

### Abstract

Ractopamine is approved for use in swine to improve carcass leanness in the USA, but banned in the European Union and China since ractopamine residues may pose health risks. This study investigated the possibility of applying surface enhanced Raman spectroscopy (SERS) for analysis of ractopamine in swine urine. Ractopamine (400 ng·mL<sup>-1</sup> to 10 µg·mL<sup>-1</sup>) was added to urine samples collected from 20 pigs. A liquid-liquid extraction (LLE) method and a more complicated method involving liquid-liquid extraction and solid phase extraction (LLE-SPE) were used to extract ractopamine from urine samples. Results indicated that the limit of detection for ractopamine from LLE and LLE-SPE extraction methods was 800 ng·mL<sup>-1</sup> and 400 ng·mL<sup>-1</sup>, respectively. For the quantitative analyses, the correlation coefficient (R<sup>2</sup>) of ractopamine values vs. their values predicted by partial least square models was 0.8184 for LLE, and 0.8166 for LLE-SPE extraction methods. SERS method with simple sample preparation had great potential for rapid analysis of ractopamine in swine urine.



### Materials and Methods

**Preparation of ractopamine solution.** Ractopamine (Fig 1) standard solutions (0.1 - 5 µg·mL<sup>-1</sup>) in 50% methanol water solution were prepared.

**Urine sample preparation.** Urine samples (n=20) were collected from 20 pigs, and spiked with ractopamine to prepare samples containing ractopamine ranging from 400 ng·mL<sup>-1</sup> to 10 µg·mL<sup>-1</sup>. A liquid-liquid extraction (LLE) and a more complicated method involving liquid-liquid extraction and solid phase extraction (LLE-SPE) was used to extract ractopamine, respectively, before SERS analysis.

**LLE-SPE extraction method<sup>[1-2]</sup>** A urine sample (5 ml) was mixed well with sodium borate buffer (2 mL, 80 mmol·L<sup>-1</sup>, pH 10.3±0.1), extracted twice with ethyl acetate (15mL), centrifuged at 5000 rpm (300 g) for 10 min. The collected supernatant extracts were dried and reconstituted in acetic acid (4 mL, 1 mol·L<sup>-1</sup>). Then the sample was loaded onto a preconditioned MCX-SPE column (3mL, 60mg; Agela technologies, Tianjin, China), rinsed with acetic acid (3 mL, 1 mol·L<sup>-1</sup>) and acetonitrile (6 mL), eluted with 6 mL 2% ammonia in ethyl acetate:methanol (30:70, v/v), and the elution was dried and dissolved in 50% MeOH:H<sub>2</sub>O (1 mL).

**LLE extraction method.** The procedure was the same as the LLE in the LLE-SPE method except that the reconstruct solution was 1 mL of 50% methanol water instead of 4 mL of acetic acid.

**Raman spectral acquisition.** SERS spectra were recorded with a DXR microscopy Raman spectrometer (Thermo Fisher Scientific Inc., USA) and Klarite™ SERS-active substrates (D3 technologies Ltd., U.K.). A tested sample (1.0 µL) was deposited onto the substrate, and its SERS spectrum was recorded after the solvent being evaporated.

### Results and Discussion

#### Standard solution of ractopamine

The three most prominent peaks at around 642, 857 and 1616 cm<sup>-1</sup> in the spectrum of ractopamine were attributed to the C-C aromatic stretching vibration, C-H aromatic out-of-plane bending and C-H wagging, respectively<sup>[3,4]</sup> (Fig 2A).

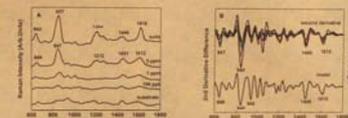


Fig. 2. (A) Raman spectra and representative SERS spectra of ractopamine (B) Second derivative transformation of SERS spectra and PLS model for ractopamine standard solution

The peaks at around 642, 857 and 1616 cm<sup>-1</sup> exhibited an increase in intensity with the increase of ractopamine concentration (Fig 2B). For quantitative analysis, PLS model (n = 36) with five latent variables yielded the best result (R<sup>2</sup> = 0.9368, RPD = 3.971) (Table 1).

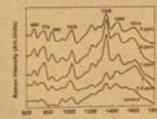


Fig. 3. SERS spectra of ractopamine extracted with LLE-SPE

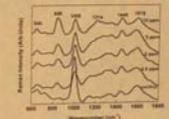


Fig. 4. SERS spectra of ractopamine extracted with LLE

#### SERS analysis of urine samples prepared with LLE-SPE

The prominent peaks at around 840 and 660 cm<sup>-1</sup> in the SERS spectra of ractopamine (from standard solution) were still among the most prominent peaks in the SERS spectra of LLE-SPE extraction of urine spiked with ractopamine (Fig 3). The lowest level of ractopamine in urine could be detected with SERS was about 400 ng·mL<sup>-1</sup>. The best PLS model (n = 180) for determination of ractopamine in urine yielded R<sup>2</sup> of 0.8166 and RPD of 2.328 (Table 1).

Table 1. Results of PLS models for ractopamine determination

	Standard solution	LLE method	LLE-SPE method
Concentration range (µg·mL <sup>-1</sup> )	0.1-5	0.4-8	0.8-10
LOD(ng·mL <sup>-1</sup> )	100	800	400
R <sup>2</sup>	0.9368	0.8184	0.8166
RPD	3.971	2.347	2.328

#### SERS analysis of urine samples prepared with LLE

The SERS spectra of ractopamine LLE extractions showed consistency with that of ractopamine standard solutions, though the interference of other compounds in the urine extracts was quite obvious (Fig 4). The very strong band at 1038 cm<sup>-1</sup> corresponded to symmetrical C-N stretch of urea<sup>[5]</sup>. Due to the strong interference of urea, the intensities of characteristic bands of ractopamine were seriously weakened, but the band at about 846 cm<sup>-1</sup> could still be discernible at the ractopamine level of 800 ng·mL<sup>-1</sup> in urine.

The best PLS model (n = 160) for ractopamine extracted with LLE (R<sup>2</sup> = 0.8184, RPD = 2.347) yielded similar results as that extracted with LLE-SPE method.

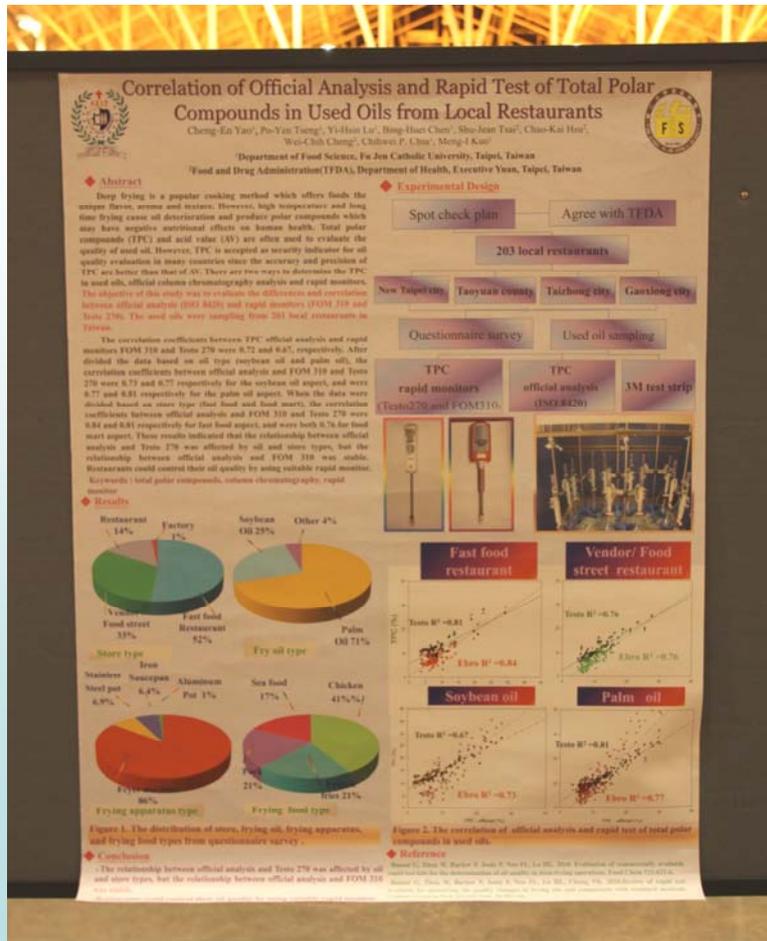
### Conclusions

Sample lost during ractopamine extraction led to an increase in the detection limit. Interference of the other components in urine extraction also adversely affected SERS analysis for ractopamine in urine.

It took less than 30 min for the LLE extraction method, but about an hour for the LLE-SPE method. The SPE step was time consuming and the disposable columns used were quite costly. Although SPE was the necessary step for determination of ractopamine in urine with chromatography-based analytical methods, this study demonstrated that it did not affect the accuracy of SERS method for quantitative analysis of ractopamine without SPE step during urine sample preparation.

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**Thanks for your attention !**