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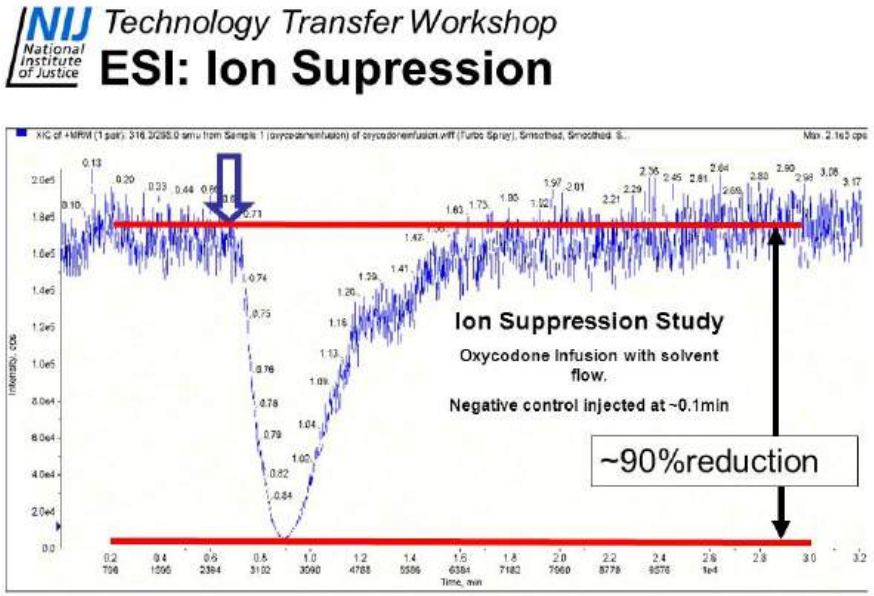
Technology & Science

Hiệu năng bền bỉ vượt thời gian

Hôm nay máy có ổn không?

Hôm nay ion source có phải vệ sinh không?



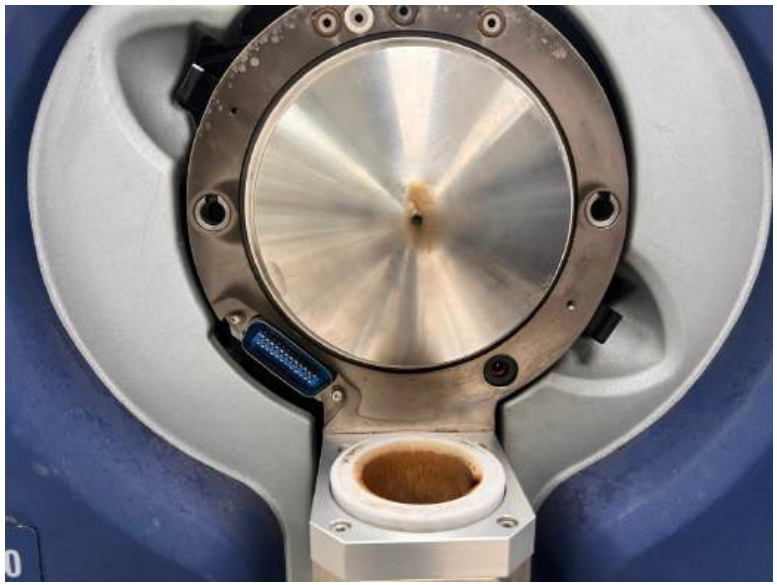
Năm thứ 5 của máy hiệu năng máy còn tốt không?

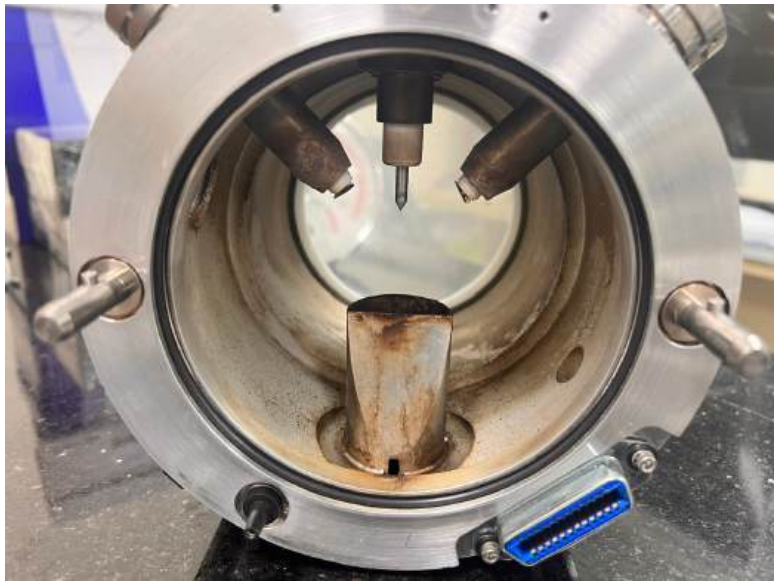
Năm thứ 10 của máy hiệu năng máy còn tốt không?

6/30/25

Hiệu năng bền bỉ vượt thời gian

- **Chức năng tự làm sạch** của nguồn ion hóa Turbo V Source
- Curtain gas chống nhiễm bẩn cho đường truyền dẫn ion
- Máy mới, máy đã hoạt động 5 năm, 10 năm….cùng một hiệu năng





HÌnh ảnh nguồn ion source chạy mẫu food sau **5 năm không vệ sinh**

NỘI DUNG

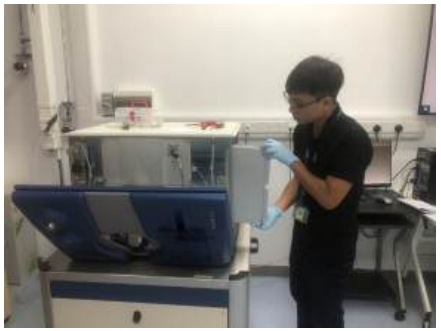
THIẾT KẾ

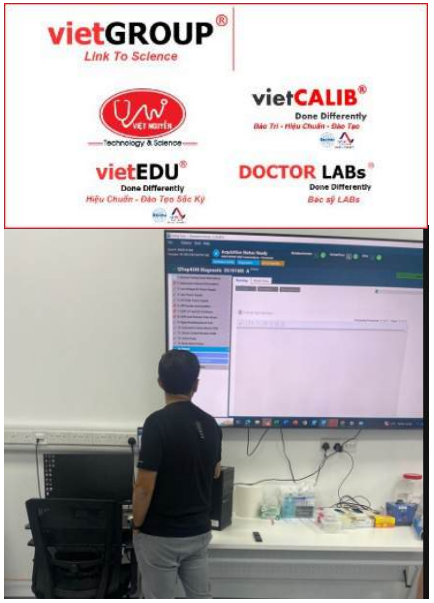
ỨNG DỤNG

SCIEX

PHẦN MỀM

KỸ THUẬT





SCIEX
Mass spectrometry portfolio

Triple Quad 3500

Triple Quad/
QTRAP 4500

Triple Quad
QTRAP Ready 5500+

Triple Quad/ QTRAP 6500+

Triple Quad
QTRAP Ready
7500/7500+

Pushing the limits

X500R
QTOF system

X500B QTOF system

ZenoTOF
7600 system

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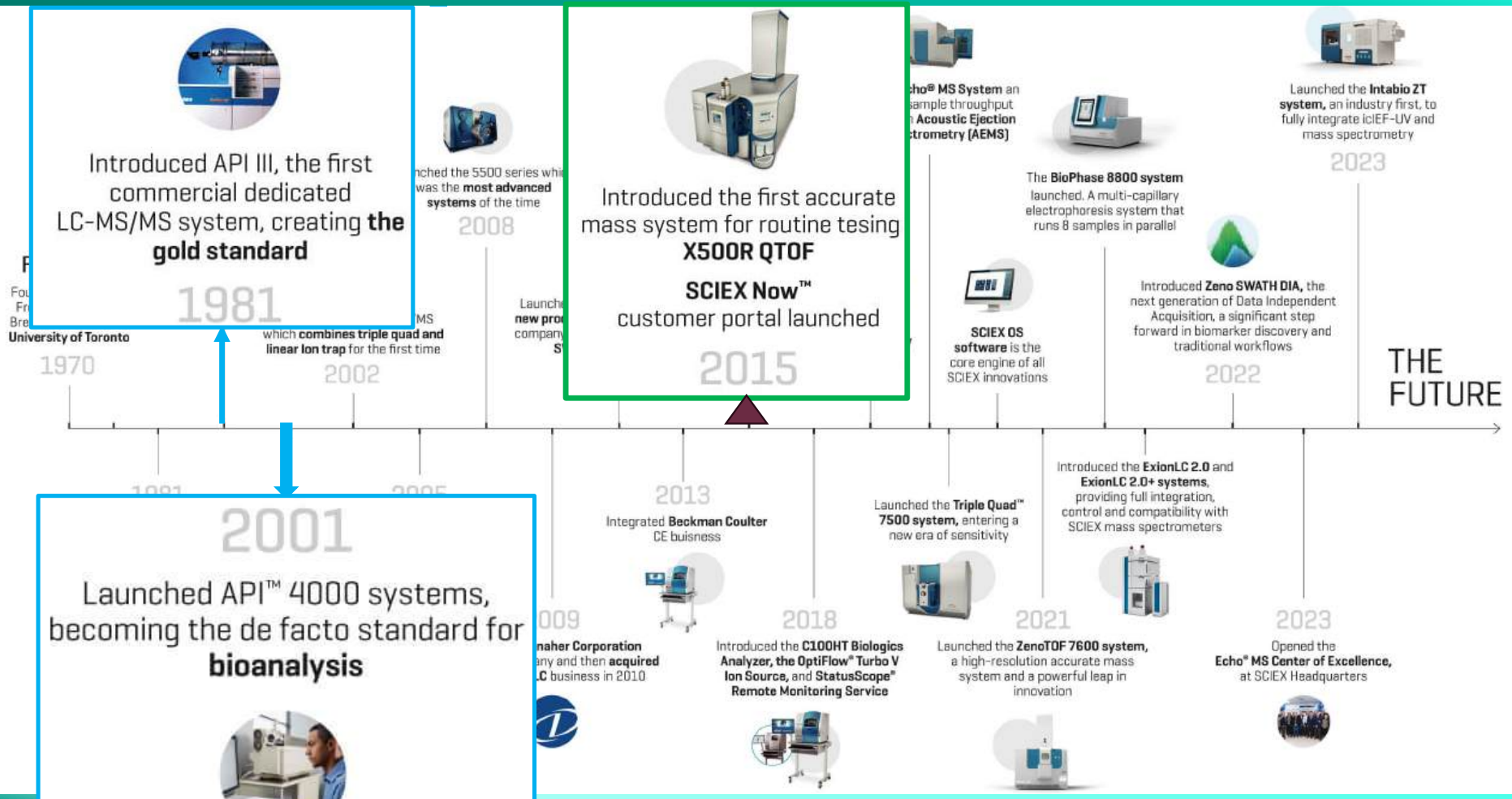

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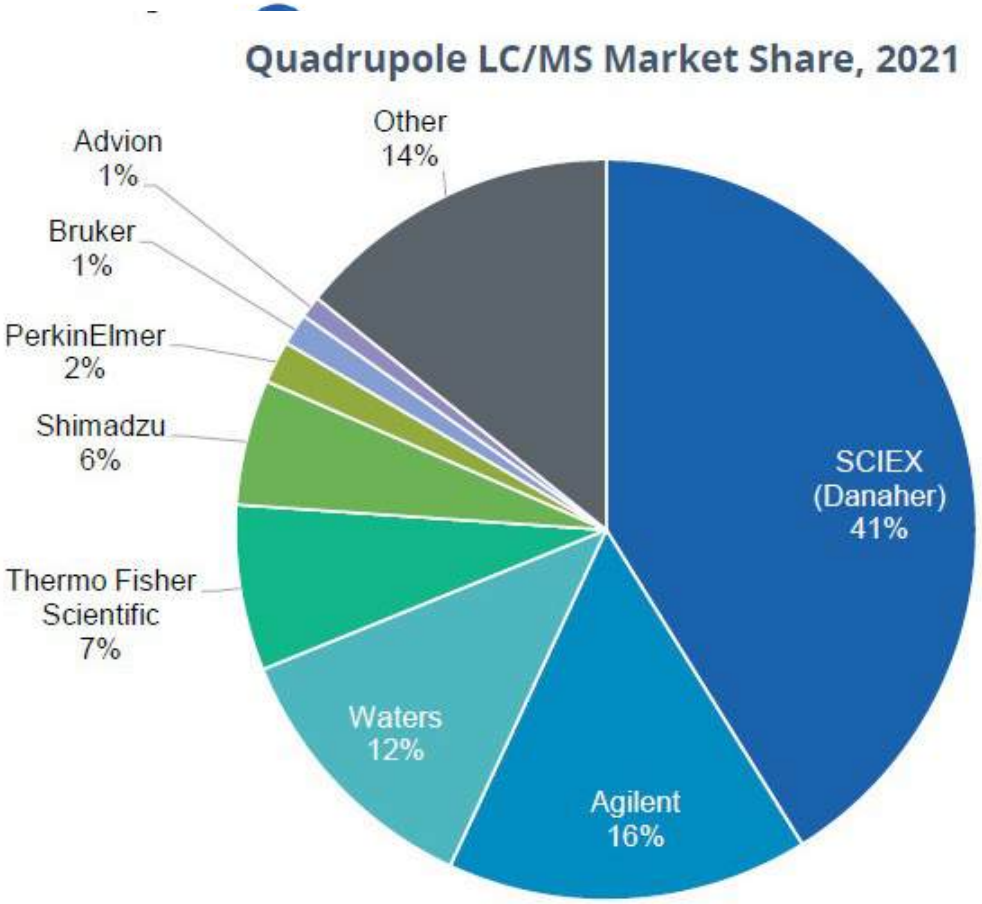

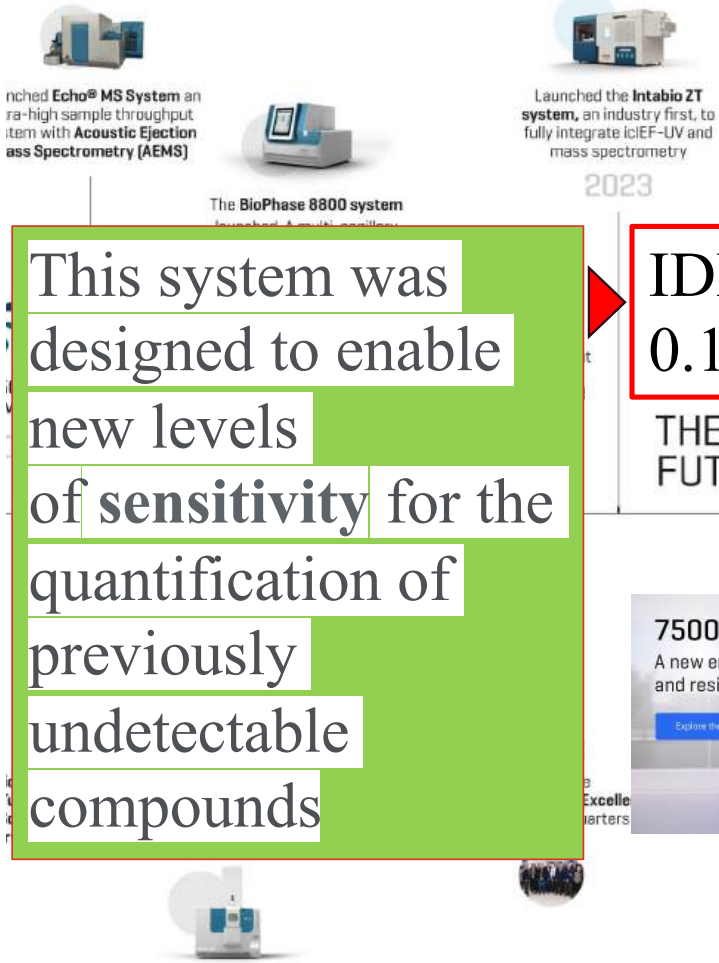

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U.S. FOOD & DRUG ADMINISTRATION

FDA Foods Program Compendium of Analytical Laboratory Methods: Chemical Analytical Manual (CAM)

METHOD NUMBER: C-003.02

POSTING DATE: November 14, 2023

POSTING EXPIRATION DATE: No expiration date (multi-laboratory validated method).

PROGRAM AREA: Mycotoxins

METHOD TITLE: Determination of Mycotoxins in Corn, Peanut Butter, and Wheat Flour Using Stable Isotope Dilution Assay (SIDA) and Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS).

2023.10. Method

The use of trade names in this method constitutes neither endorsement nor recommendation by the U. S. Food and Drug Administration (FDA). Citation of instrumentation is based on the original method validation and supporting method verification and platform extension studies.

A Shimadzu LC-30 system with a binary pump (LC-30AD), autosampler (SIL-30AC) and column oven (CTO-20AC) was used for method development and validation. A Sciex ExionLC and Shimadzu LC-40 have also been tested in method verification.

Mass spectrometric analysis is performed on a Sciex QTRAP® 4000, Sciex QTRAP® 6500/6500+, or Sciex Triple Quad 6500/6500+ platform in positive ionization mode. Source and compound dependent parameters are summarized below. MS/MS data are collected in a scheduled multiple reaction monitoring (MRM) mode using Analyst® or Sciex OS for instrument control (Sciex). Quantitative data analysis is performed using MultiQuant™ or Sciex OS Version 1.6 or higher (Sciex).

OXFORD

Journal of AOAC INTERNATIONAL, 104(3), 2021, 650–681

doi: 10.1093/jaoacint/qsaa168
Advance Access Publication Date: 22 December 2020
Article

FOOD CHEMICAL CONTAMINANTS

Screening of 154 Veterinary Drug Residues in Foods of Animal Origin Using LC–MS/MS: First Action 2020.04

Aurélien Desmarchelier, Thomas Bessaire, Marie-Claude Savoy, Adrienne Tarres, Claudia Mujahid, Andrea Beck, Pascal Mottier and Thierry Delatour*

(s) *LC–MS/MS instruments.*—

(1) For *Streams A, B, and D*, Agilent 1290 SL LC system coupled to a Sciex 5500 or 6500+ Triple-stage quadrupole mass spectrometer or equivalent. Warning: for *Streams A and B*, the LC–MS/MS system must also be capable to switch from positive to negative mode within less than 50 ms.

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European Commission EURL-SRM

Quick Met
Numerous Highly P
Extraction with Acidified M
I. Food of Plan

EURL-SRM EU Reference Laboratories for Residues of Pesticides
Single Residue Methods

***Table 5** : Methods mainly used by CVUA Stuttgart*

Method	Special remarks on Substances	LC-MS/MS	Comments
M3: Glyphosate & Co. Hypercarb (see 5.6.3)	Glyphosate AMPA N-Acetyl-AMPA N-Acetyl-Glyphosate Ethephon HEPA Glufosinate N-Acetyl-Glufosinate MPPA Fosetyl-Al Phosphonic acid (screening) Maleic Hydrazide Perchlorate (screening)) Chlorate (screening) Cyanuric acid Bialaphos Desmethyl-Dimethoate	Agilent 1200 Sciex QTRAP 5500	Evaluation via solvent calibration and ILISs except for Bialaphos and N-Acetyl-AMPA (ILIS not yet available) M 1.5 and M 1.6 are currently being tested for their suitability to replace M 1.3
M1.4: Method 1.4 (M 1.4): "Per-ChloPhos" **(see 5.6.4)**	Perchlorate (quantitative) Chlorate (quantitative) Phosphonic acid (quantitative) Bromide (Screening, quantitative) Bromate (quantitative) Thiocyanate	Agilent 1200 Sciex QTRAP 5500	Mostly employed directly (option: screening by M 1.3, if postive -> M 1.4) Dilution 5-fold Evaluation via solvent calibration and ILISs

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Please be aware that this old REACH registration data factsheet is no longer mainta

The new ECHA CHEM database has been released by ECHA, and it now contains all I transition of ECHA's published data to ECHA CHEM here.

Access ECHA CHEM

ECHA
https://echa.europa.eu

ECHA: Homepage

This is unique source of information on the chemicals manufactured and imported in Europe. It covers their hazardous properties, classification and labelling, ...

Di-"isononyl" phthalate

EC number: 249-079-5 | CAS number: 28553-12-0

- General information
- Classification & Labelling & PBT assessment
- Manufacture, use & exposure
- Physical & Chemical

- Toxicological Summary
- Toxicokinetics, metabolism and distribution
- Acute Toxicity
- Irritation / corrosion
- Sensitisation
- Repeated dose toxicity
- Genetic toxicity
- Carcinogenicity
- Toxicity to reproduction
- Specific investigations
- Exposure related observations in humans

Epider

Current

Administ

Admini

Endpoint:

Type of in

Details on exposure: Of the 2,582 pregnant women participating in the SELMA study, a first morning urine sample was obtained from 2,356 women (91%) during weeks 9–11 of the pregnancy. These samples were analyzed for the MBP, MEP, MBzP, MEHP, oh-MEHP, oxo-MEHP, cx-MEPP, sum DEHP, oh-MMeOP, oxo-MMeOP, cx-MMeHP, sum DiNP . Briefly, 0.2 mL of urine were added with 0.1 mL of ammonium acetate (pH 6.5) and 0.01 mL glucoronidase (Escherichia coli) and thereafter incubated at 37°C for 30 min. Then 0.05 mL was added of a 50:50 (vol:vol) water and acetonitrile solution of labeled (3H or 13C) internal standards of all analyzed compounds. A C18 column (2.1 mm i.d. × 50 mm; Genesis Lightn; Grace, Deerfield, IL, USA) was used before the injector to reduce interference. The phthalate metabolites in the samples were separated on a C18 column (1.5 µm, 2.0 mm i.d. × 30 mm VisionHT; Grace). The mobile phases were water and acetonitrile with 0.08% formic acid. The samples were analyzed on a Shimadzu UFLC system (Shimadzu Corporation, Kyoto, Japan) coupled to a **QTRAP5500 triple quadrupole linear ion trap mass spectrometer equipped with a Turbolon Spray source (AB Sciex, Foster City, CA, USA).** The samples were analyzed in triplicate, and the mean of the two closest were reported. All samples were analyzed in a randomized order. For quality control of the analyses, chemical blanks and two different in-house prepared quality control samples were analyzed in all sample batches. The limit of detection (LOD) was defined as the concentration corresponding to a peak area ratio of three times the standard deviation of the chemical blanks. The creatinine concentrations were analyzed.

Statistical methods: Urinary levels of phthalate metabolites were log-transformed to normalize distributions and geometric means (GMs) with 95% confidence interval (CIs)

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THE EUROPEAN FOOD RISK ASSESSMENT FELLOWSHIP PROGRAMME

SERIES 1
2017-2018

EFSA Journal 2018:16(8):e16083

www.efsa.europa.eu/efsajournal

European Food Safety Authority

2.2.2. Part 2

In the second part of the work programme, the fellow was trained to use a robotic workstation Hamilton MICROLAB® STAR equipped with a robotic arm connected to a Multilabel Plate Reader PerkinElmer Victor X3, to develop methods that were used for application of real world sample analysis and further method development and investigation stages. In particular, residual activities of each selected enzyme for the respective OPPs were determined. Assay analysis times were optimised to obtain prime reproducibility of data and to better investigate the effects of unique residual activities. To support the bioenzymatic method and confirm the robustness, a conventional mass spectroscopy method was developed and verified using an AB Sciex QTrap 4,500 triple quadrupole MS system coupled to a Shimadzu Nextera x2 liquid chromatography system.

6/30/25

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2022

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Abstract

Introduction

Key benefits of the method for ultra-

Achieving low parts-per-quadrillion detection limi water

[1]Eurofins Environment Testin

[2]SCIEX, USA; [3]Phenomenex

Achieving low parts-per-quadrillion detection limits for PFAS analysis in drinking water

A collaborative exploration of instrument sensitivity and sample preparation for ultra-trace analysis of PFAS on the SCIEX 7500 system

Thep Phomsopha[1] , Simon Roberts[2] , Craig M. Butt[2] , Karl Oetjen[2] , Matt Noestheden[2] , Sam Lodge[3] , Andrew N. Patterson[1]

[1]Eurofins Environment Testing West Sacramento;
[2]SCIEX, USA; [3]Phenomenex

Abstract

This technical note describes the collaboration between SCIEX and Eurofins Environment Testing to address the 2022 US EPA drinking water health advisory levels (HALs) for 4 per- and polyfluoroalkyl substances (PFAS).[1] Sample preparation and instrumental methods were developed to achieve low parts-perquadrillion (ppq) HALs —which is pg/L equivalent—for perfluorooctanoic acid (PFOA, 4 ppq), perfluor 0 ppq), perfluorobutane su hexafluoropropylene oxide dimer acid (HFPO-DA, or

Request information →

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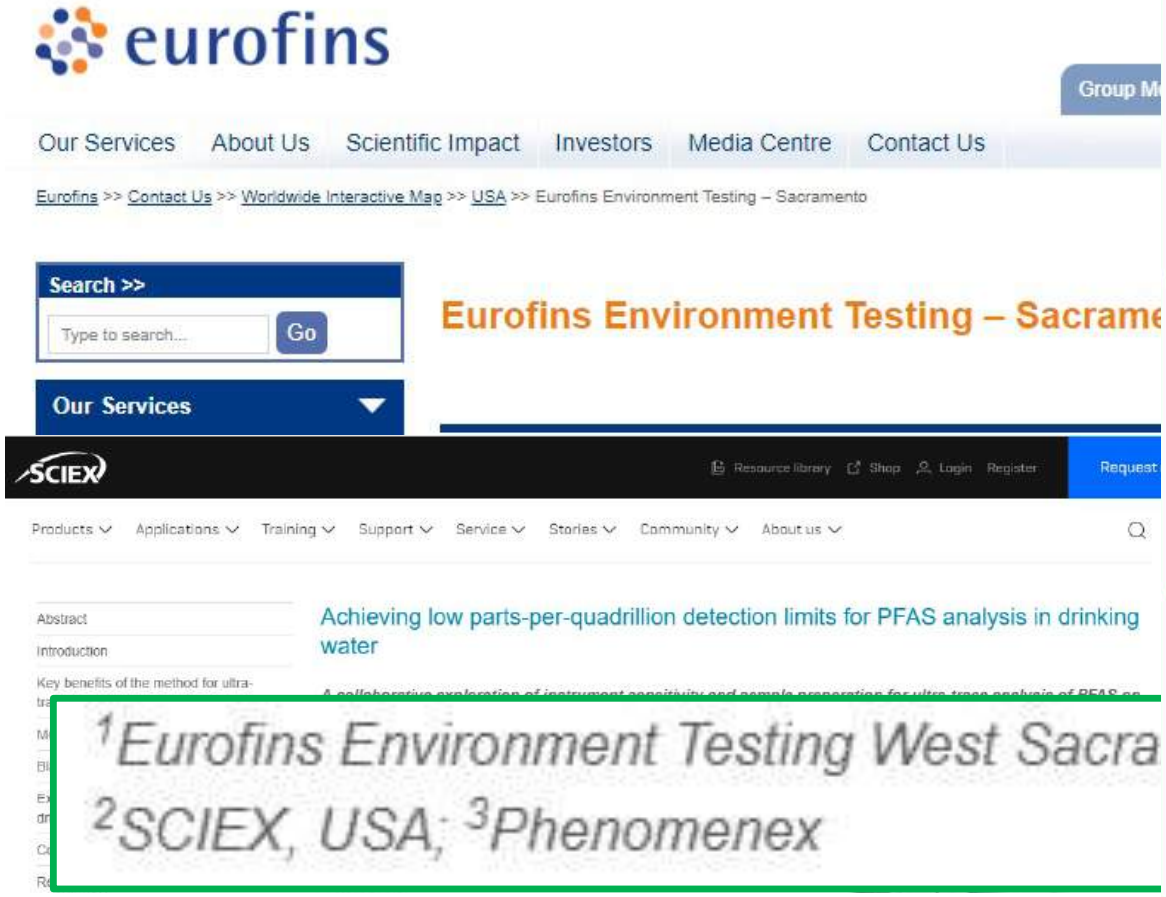
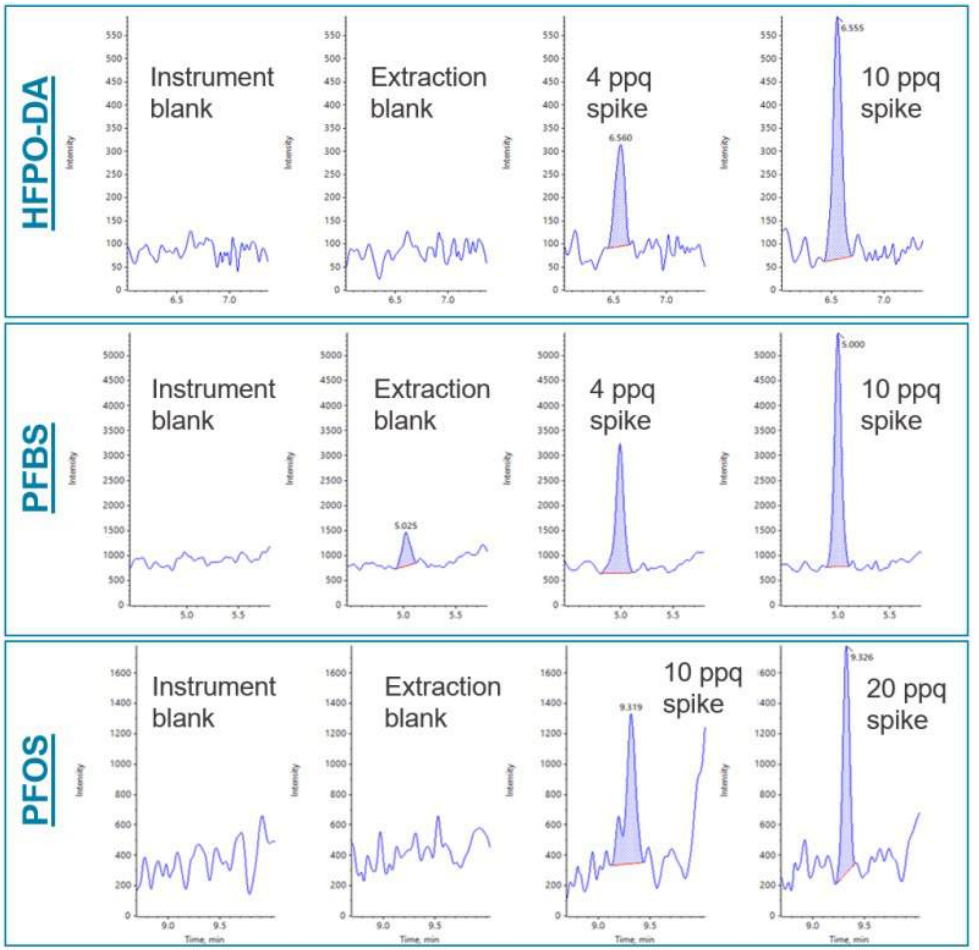
2022





The Best Choise – Niềm Tin

Testing Lab miền Bắc	SL
Viện kiểm nghiệm an toàn thực phẩm quốc gia	2
Trung tâm chứng nhận phù hợp Quacert	1
Viện kiểm nghiệm Thuốc	1
Vinmec (Test ATVS Thực phẩm)	2
Viện dinh dưỡng	3
Viện sức khỏe nghề nghiệp	1
TT Kiểm tra vệ sinh thú y TW1	1
CC quản lý hàng hóa nông hải sản Thái Nguyên	1
CDC Bắc Giang	1

The Best Choise – Niềm Tin

Testing Lab miền Nam	SL
Quatest 3	3
Quatest 2	1
Intertek	1
TUV Rheiland	2
TUV SUD	1
Viện Y tế công cộng HCM	3
Pasteur HCM	1
Pasteur Nha Trang	1
Case	1
SGS	1

The Best Choise – Niềm Tin

Sản xuất	SL
Thực phẩm Masan	3
Thức ăn chăn nuôi Deheus	1
Thức ăn chăn nuôi CJ Food	1
Thức ăn chăn nuôi CP	1
Thức ăn chăn nuôi Biomin - DSM	1
Thủy sản Gallant Ocean	1
Dược phẩm Domesco	1
SeaFood Nha Trang – F17	1

Sciex tại Phòng thí nghiệm Khách hàng

Quatest 2 – TQ5500

Pasteur HCM – TQ5500+

Sciex tại Phòng thí nghiệm Khách hàng

Viện VSDT Tây Nguyên – QTOF X500R

ĐẠI HỌC KHOA HỌC TỰ NHIÊN
ĐẠI HỌC QUỐC GIA THÀNH PHỐ HCM

SO SÁNH GIỮA CÁC MODEL HARDWARE

Triple Quad
QTRAP Ready
5500+

Triple Quad/
QTRAP 6500+

Triple Quad
QTRAP Ready 7500+

Thông số	TQ 3500	TQ 4500	TQ5500+	TQ6500+	TQ7500+
Ion source	Turbo V	Turbo V	Turbo V	IonDrive Turbo V	
Bơm roughing	1	1	1	2	2
Đường dẫn ion JET	QJET	**QJET**	**Dual JET**	**IonDrive QJet**	**D lJet**
Đầu dò	pulse counting detector CEM	pulse counting detector CEM	High Energy Dynode (HED)	High Energy Dynode (HED)	High Energy Dynode (HED)
Tốc độ chuyển cực	50ms	50ms	**5ms**	**5ms**	**5ms**
Tốc đọ đo MRM	500MRM/s	500MRM/s	500MRM/s	500MRM/s	500/800 MRM/s
Khoảng tuyến tính	Bậc 5	Bậc 5	Bậc 6	Bậc 6	Bậc 6

ĐỘ NHẠY CỦA CÁC DÒNG SẢN PHẨM
SCIEX mass spectrometry sensitivity

Thông số	3500	4500	5500+	6500+	7500+
ESI (+) S/N 1pg reserpine	100,000	300,000	**750,000**	**1,500,000**	**5,000,000**
ESI (-) S/N 1p grchloramphenicol	100,000	300,000	**750,000**	**1,500,000**	**5,000,000**
IDL fg reserpine	5.6 fg	2.8 fg	**1.4 fg**	**0.28 fg**	**0.14 fg**
Mass range	5-2000	5-2000	5-1250	5-2000	5-2000
Trap fuctionp	NA	Hardware ready	Trap ready	2 model	Trap ready

* 1 fg=10^{-3} pg =10^{-6} ng = 10^{-9} µg =10^{-12} mg =10^{-15} g

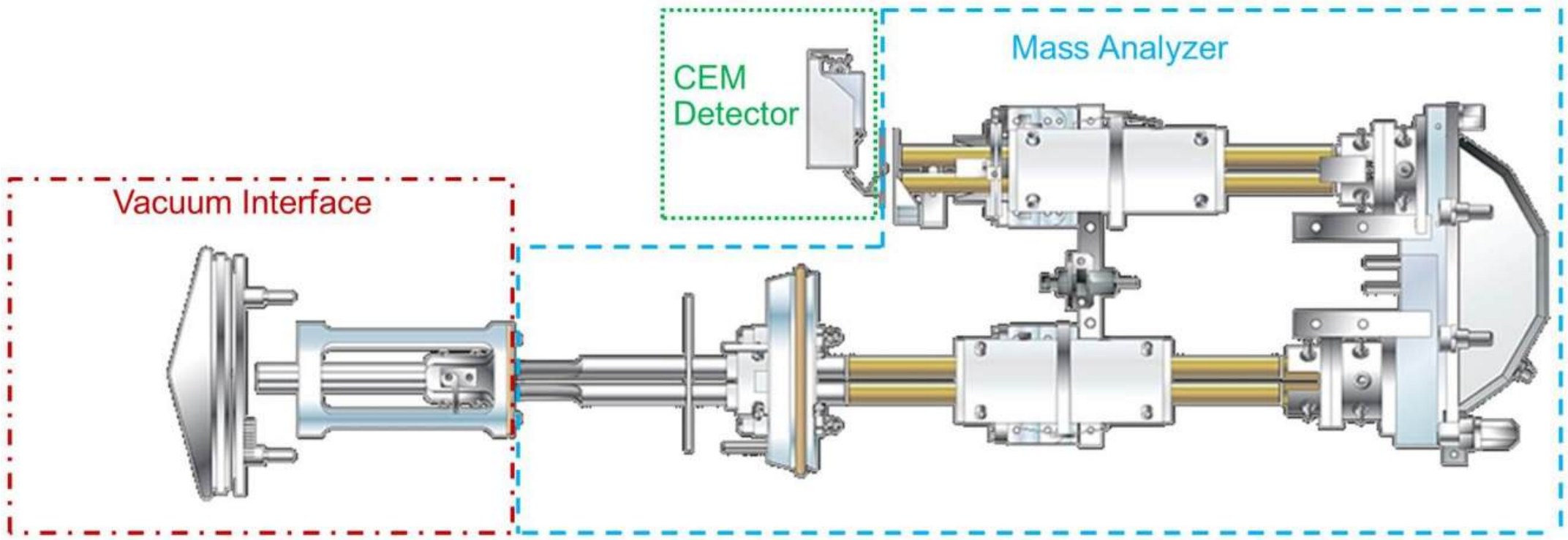
3/SO SÁNH VỚI CÁC HÃNG

Thông số	TQ 5500+	Agilent 6475
S/N	750,000:1	Pos S/N: >850.000:1 Neg S/N: >350.000:1
(IDL - ESI Positive mode)	5 fg on column (609/195) < **1.4 fg**	on column (609/195) < **4 fg**
ESI Negative mode	5 fg on column (321/152) < **1.4 fg**	on column (321/152) < **3.5 fg**

Thông số	Xevo TQ-S micro	Xevo TQ-XS
S/N	Pos S/N: >600.000:1 Neg S/N: >200.000:1	Pos S/N: >1.500.000:1 Neg S/N: >1.000.000
(IDL - ESI mode dương)	5 fg on column (609/195) < **2.5 fg**	on column (609/195) < **0.4 fg**
ESI mode âm	5 fg on column (321/152) < **2.5 fg**	on column (321/152) < **1 fg**

S/N: Tỉ lệ tín hiệu/nhiễu: phụ thuộc nhiều vào cách tính nhiễu của hãng. Giá trị không thật
IDL: giới hạn phát hiện thiết bị. Thông số có ý nghĩa thực tế, ví dụ 7500, hệ thống phải chạy mẫu chuẩn có nồng động 0.5fg và có RSD <10%
Mode âm Sciex có độ nhạy tương đương mode dương; đây là ưu điểm lớn của hãng sciex.

THIẾT KẾ QQQ - 50 Years of Innovation



frontiers in Chemistry

Front Chem. 2021; 9: 813359.
Published online 2021 Dec 21. doi: 10.3389/fchem.2021.813359

PMCID: PMC8724130
PMID: 34993180

Towards Higher Sensitivity of Mass Spectrometry: A Perspective From the Mass Analyzers

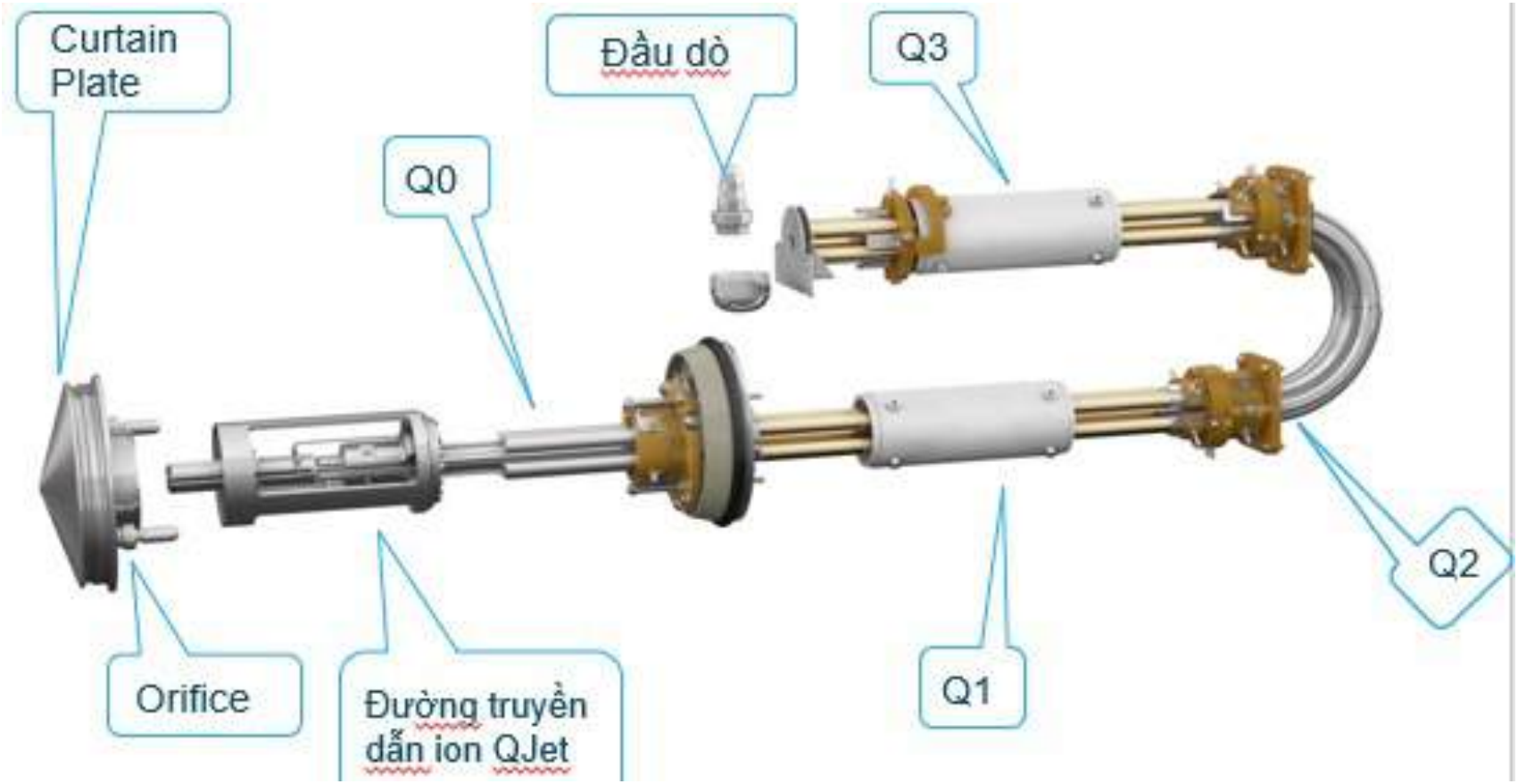
Chang Li,[1] Shiying Chu and Di Tian[1,*]

Abstract

Mass spectrometry
Recent developmen
measurement of sul
made consequently

Introducing a delayed DC ramp to quadrupole can improve the transmission efficiency of the quadrupole (Brubaker, 1968). A short RF-only quadrupole (pre-filter) is placed in front of the main quadrupole with RF and DC supply. The DC voltage is applied to the main quadrupole only after the ions enter the main quadrupole. Thus, the a and q parameters of the targeted ions always remain in the stable region. Losses of ions caused by electric fringe fields at the entrance of the main quadrupole are reduced. The sensitivity is significantly improved. This method is used in many modern commercial mass spectrometers. However, it is not useful when using higher stability regions. Huntley and Reilly (2020) simulated the acceptance and transmittance of mass filters driven by sine and rectangular waveforms. When a pre-filter was used or the duty cycle was delayed, the sensitivity of both systems is increased by four times.

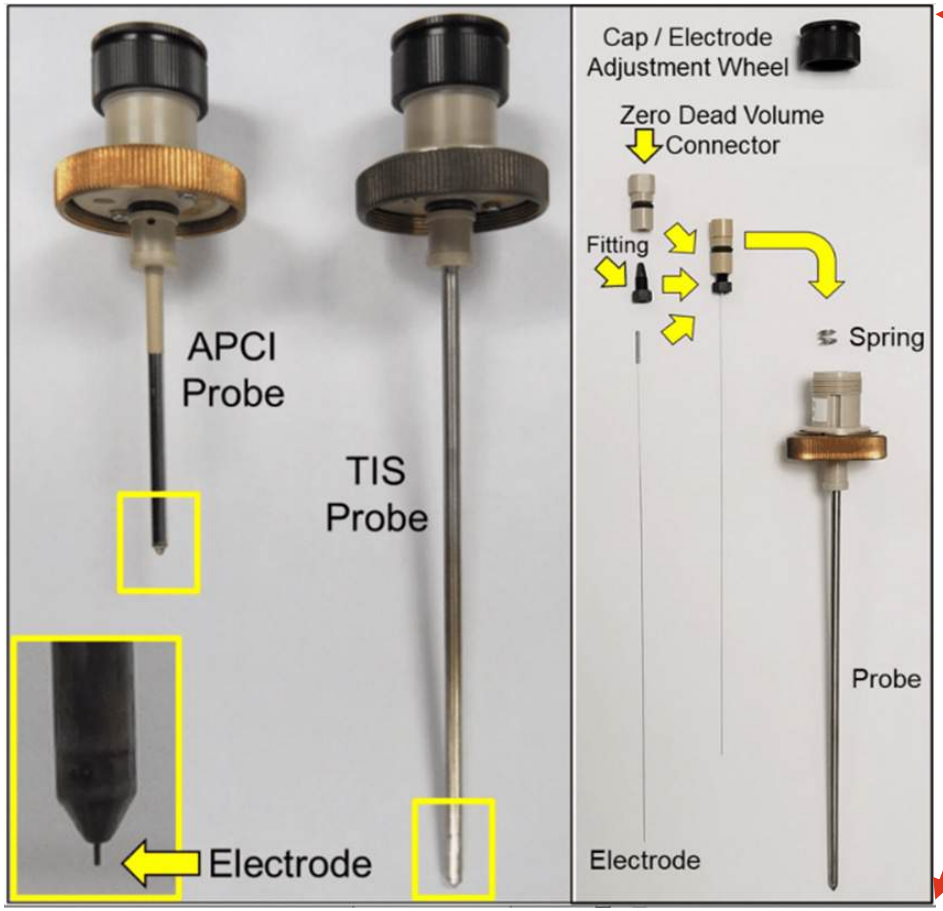
THIẾT KẾ QQQ - 50 Years of Innovation

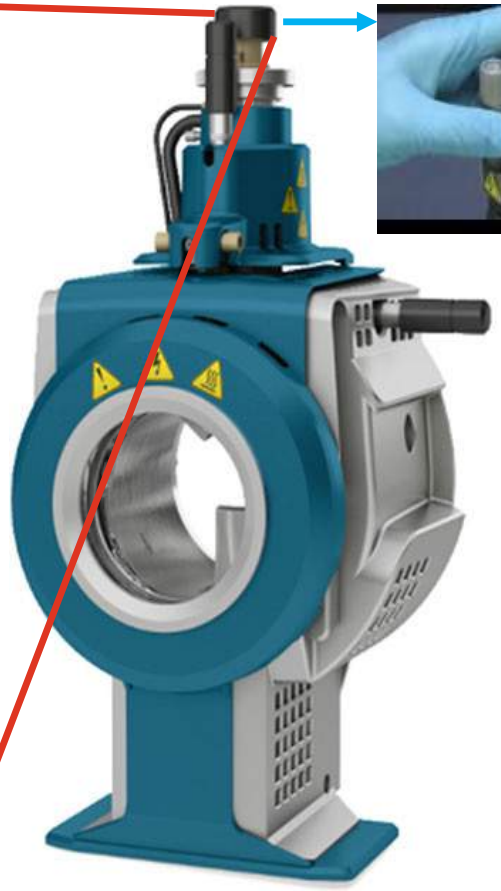


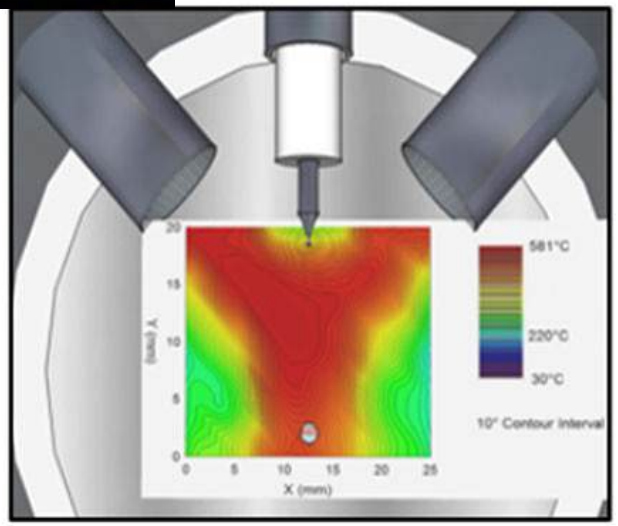
Increase pe

6/30/25

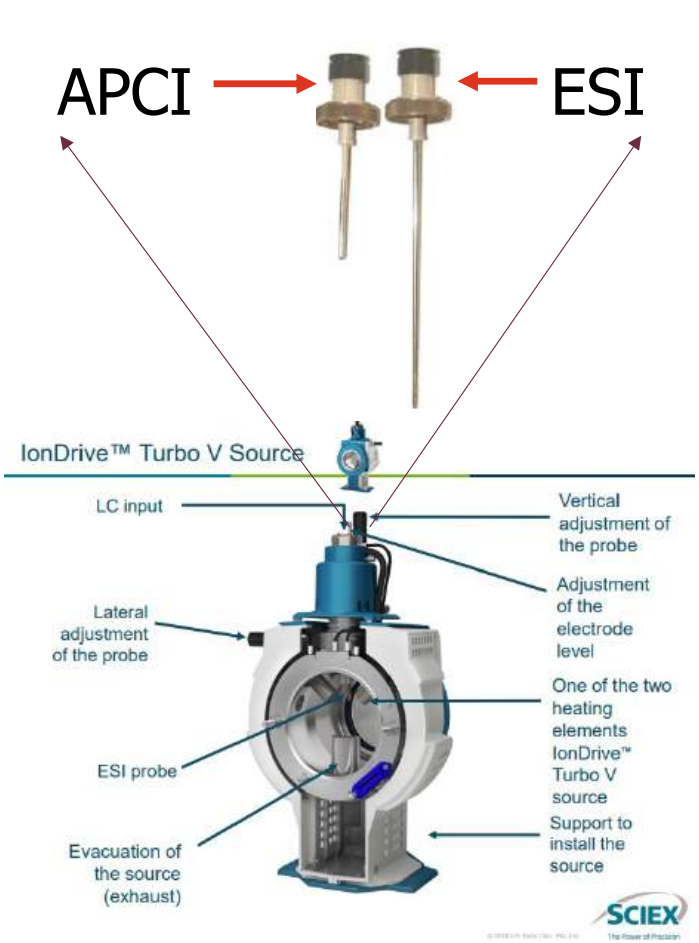
Pusing the limits - Turbo V – Ion source

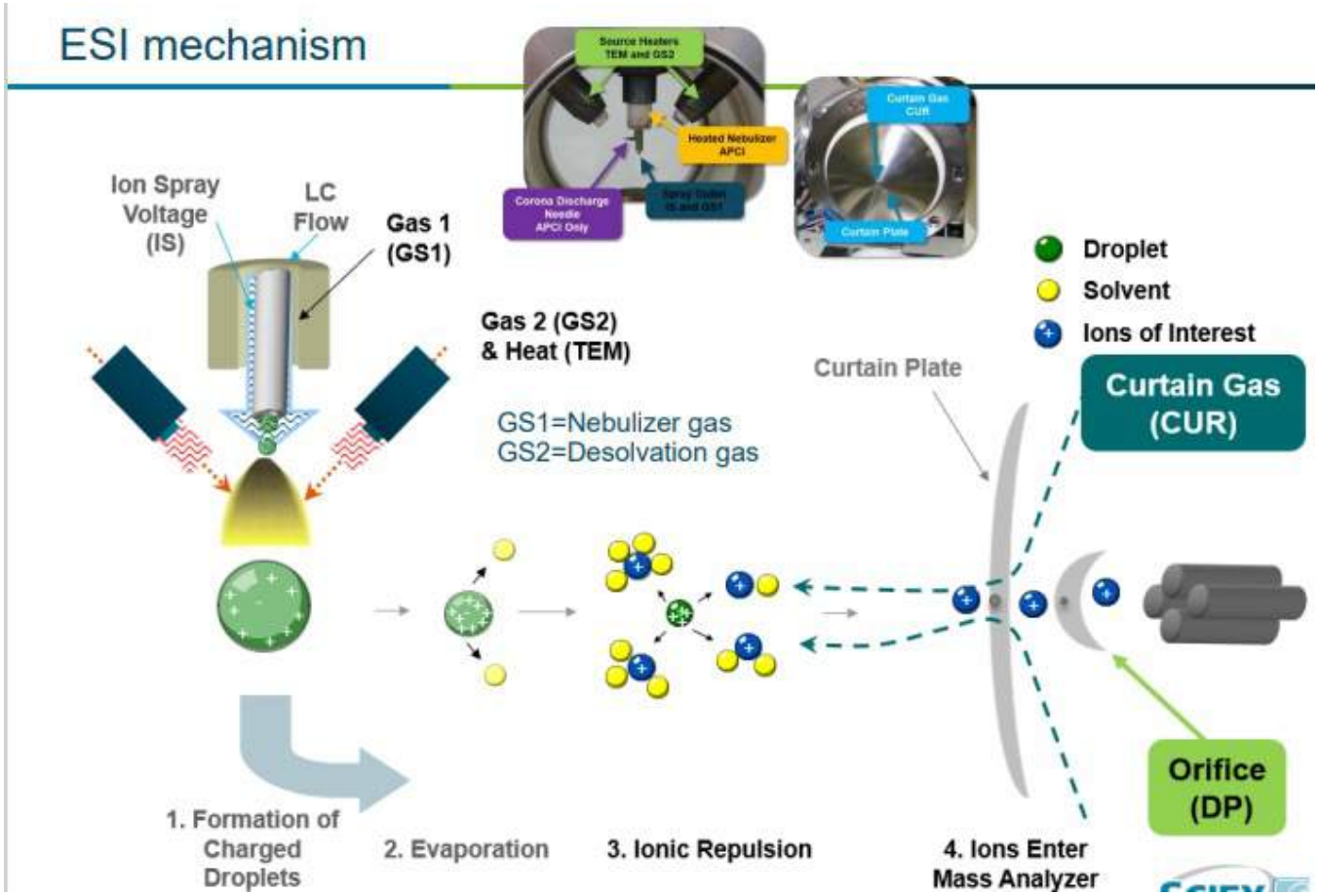




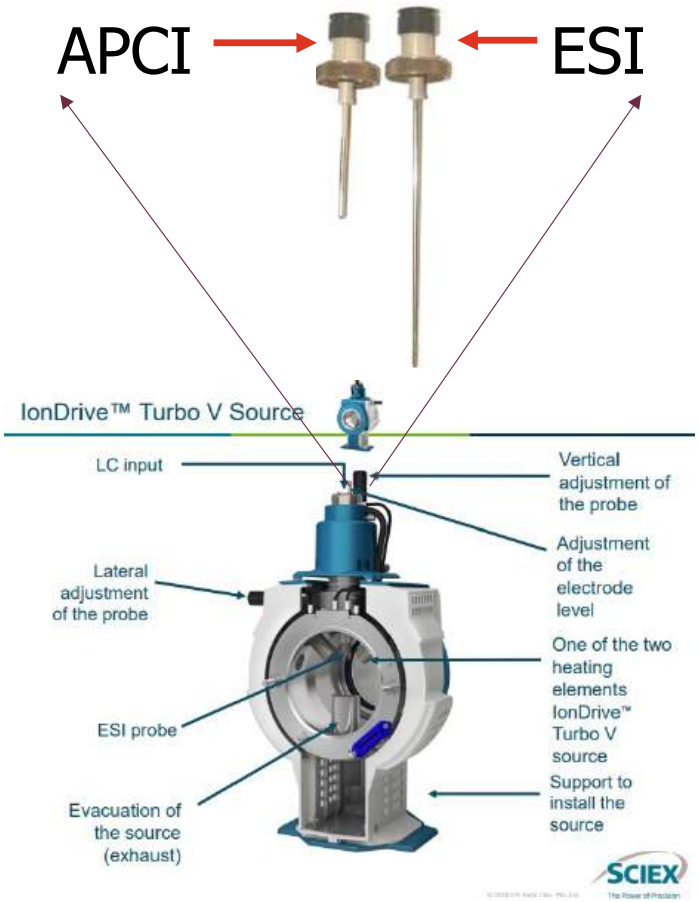


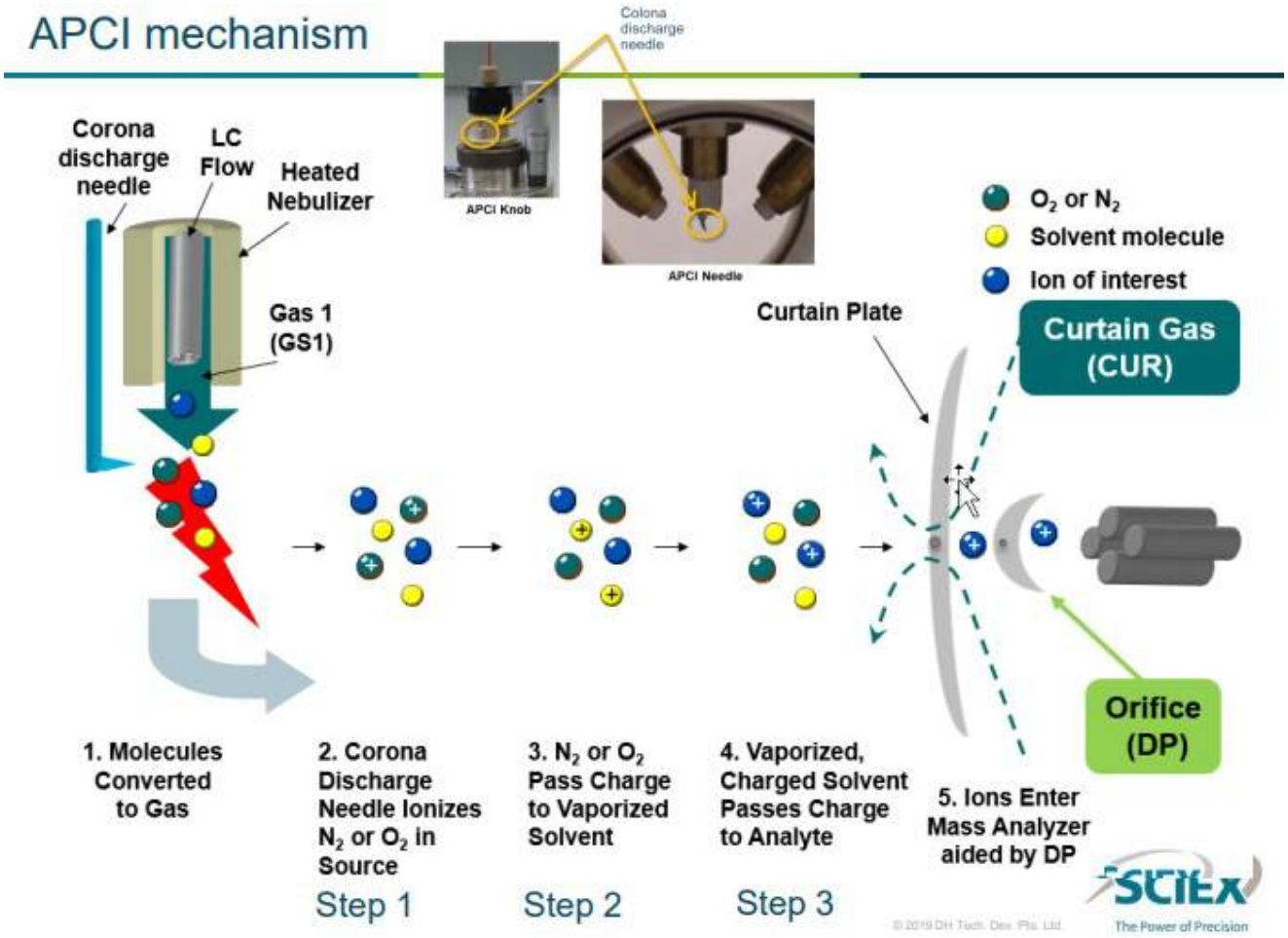
Turbo V – Ion source



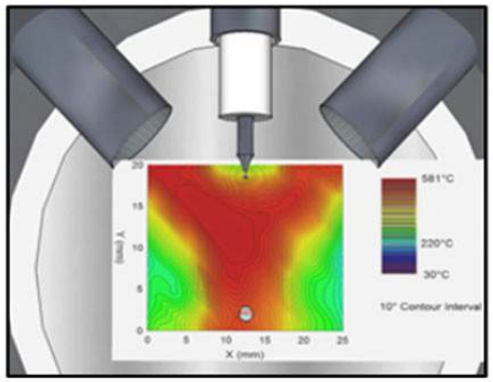


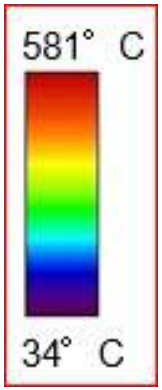
Turbo V – Ion source

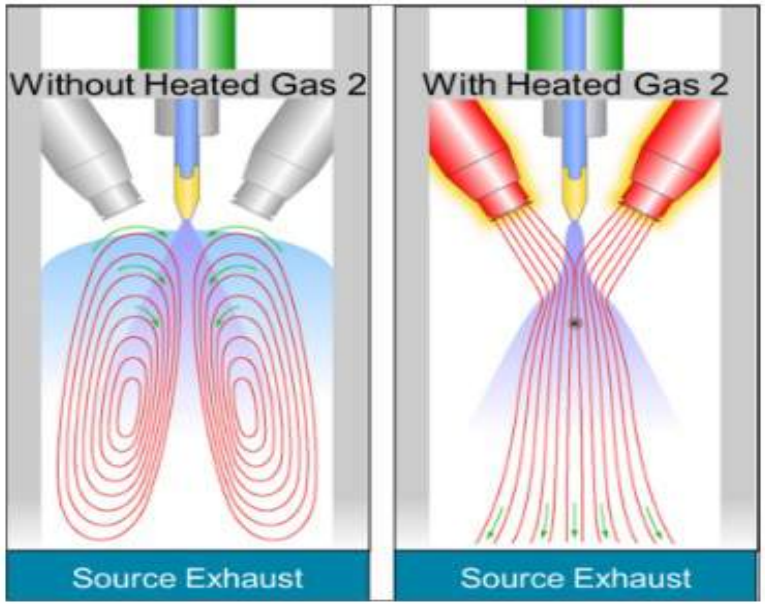




Pusing the limits - Turbo V – Ion source



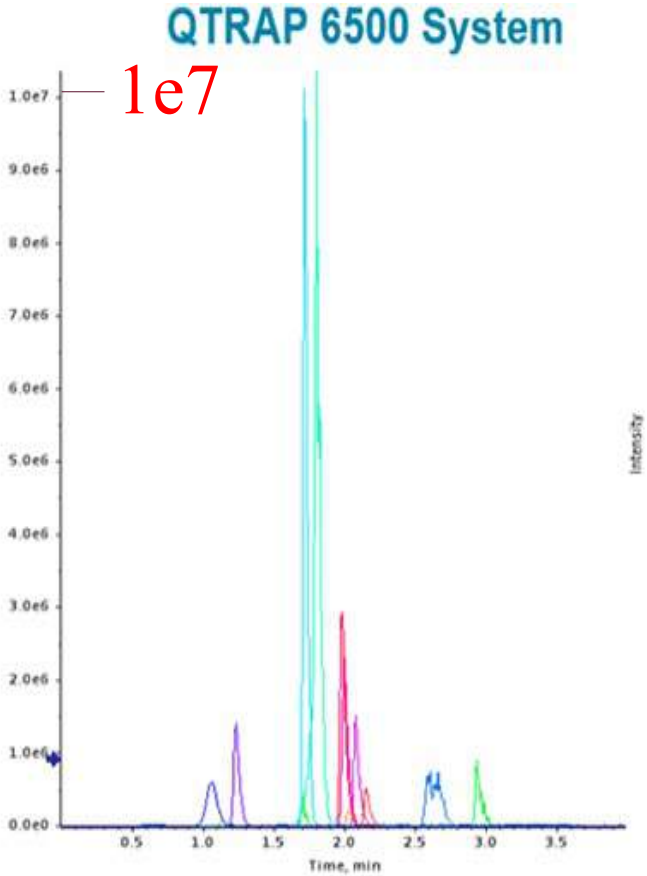


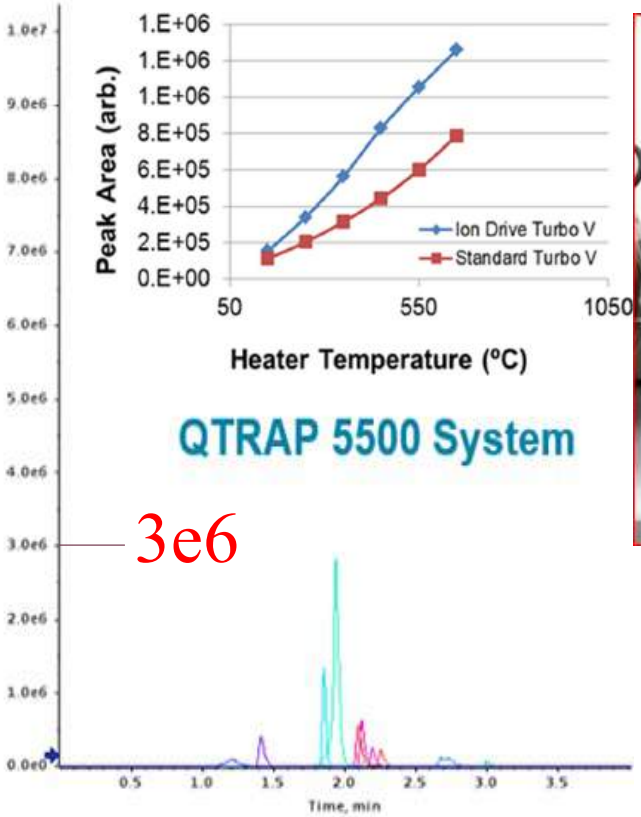


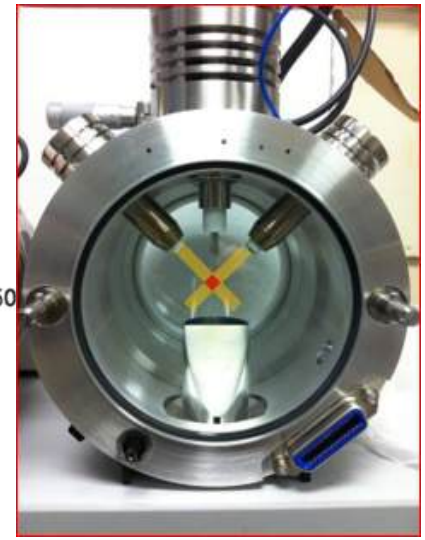
- Tốc độ dòng tối đa lên đến **3ml/phút**
- Đầu phun mẫu vuông góc với đầu vào bộ phân tích khối
- Gia nhiệt bằng khối sứ **tự làm sạch** lên đến **750°C**
- Dễ dàng tháo lắp, vệ sinh bảo trì đơn giản, **không cần tắt chân không**
- Hai chế độ ion hoá cơ bản **ESI** và **APCI**, thay đổi đầu phun dễ dàng, hệ thống tự nhận diện

Pushing the limits
IonDrive Turbo V – 6500 series

IonDrive Turbo V Ion Source

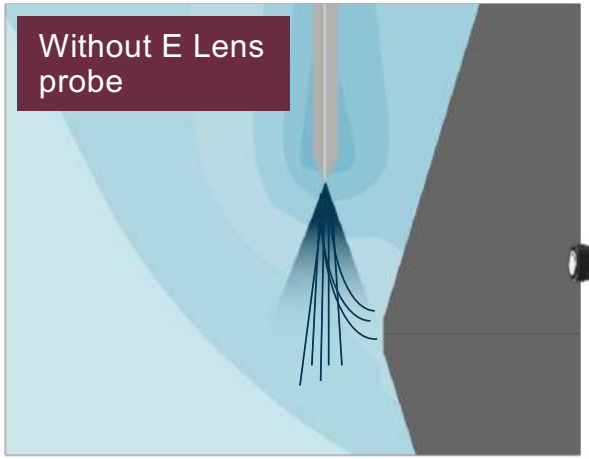


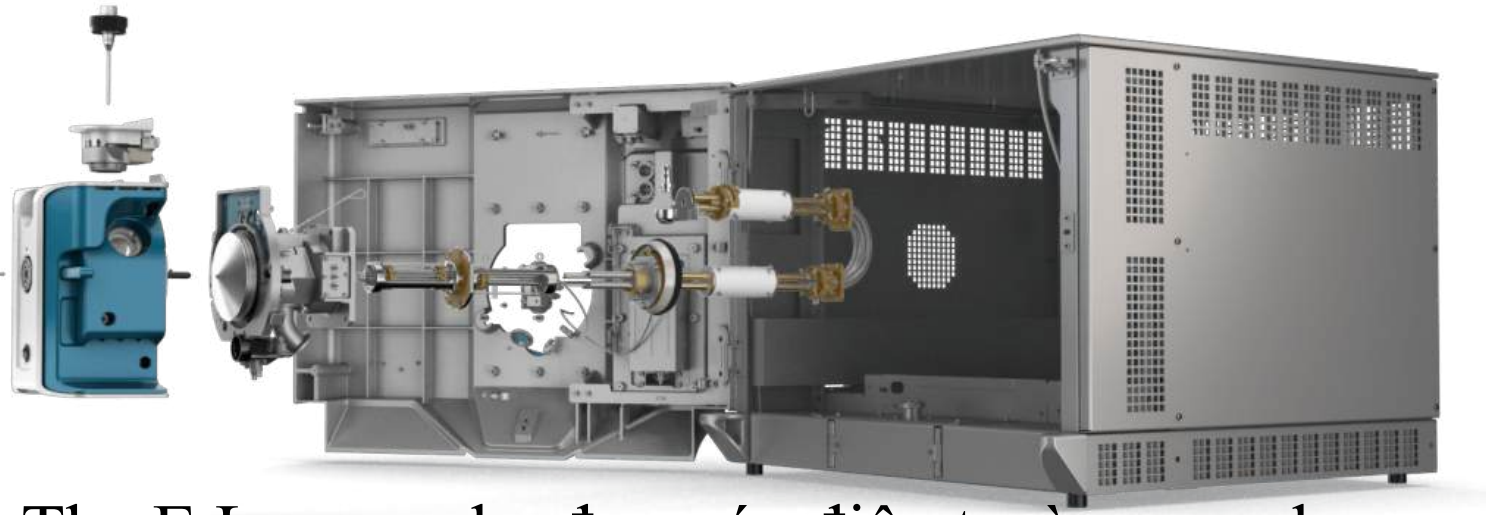


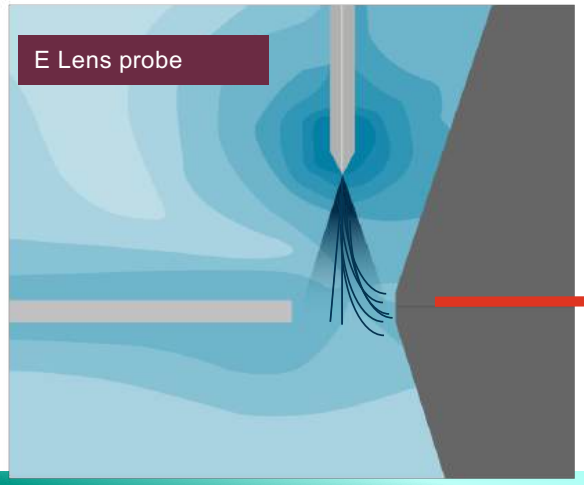


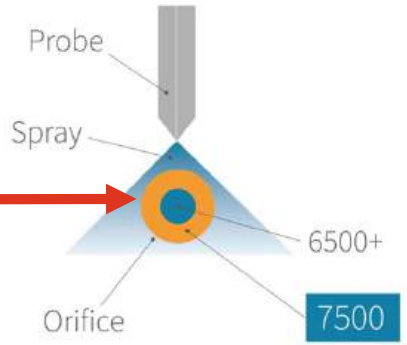
Turbo V Ion Source

Pushing the limits
E Lens technology for greater sensitivity **7500**





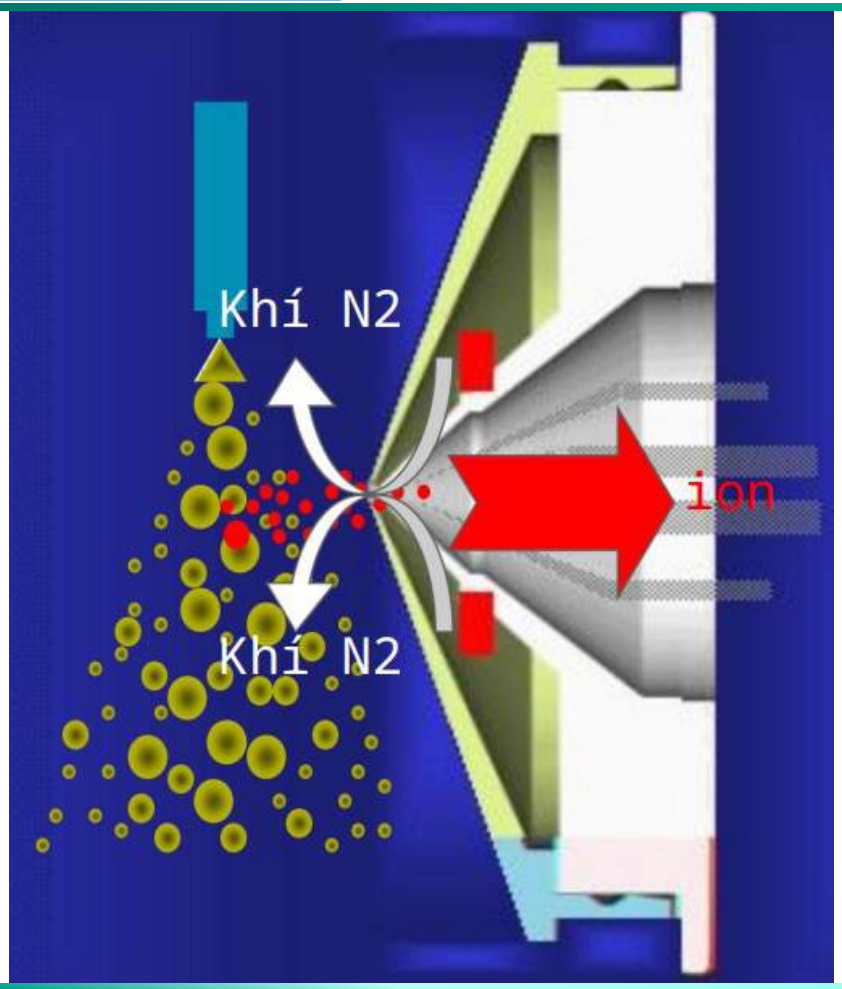




The E Lens probe được áp điện trường mạnh

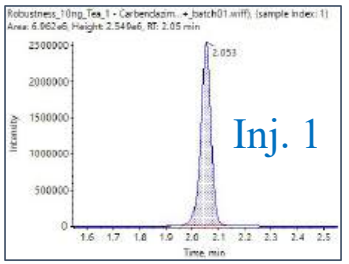
- ĐẨY ION TIẾN VỀ orifice
- Tách ion khỏi giọt sương hiệu quả
- Hiệu suất ion tăng lên hai lần

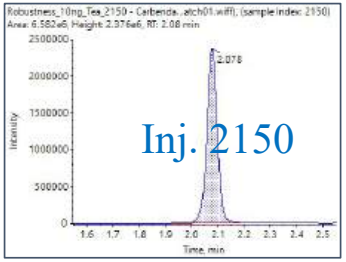
1/THIẾT KẾ Curtain gas

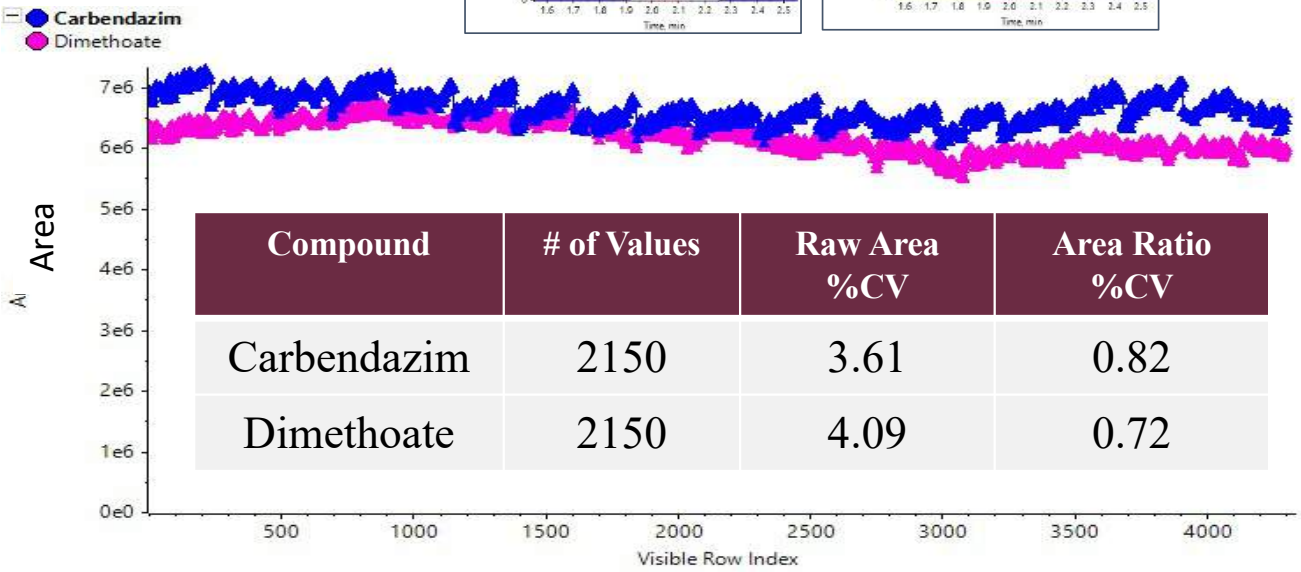


Thuốc trừ sâu trong trà đen trên 2150 lần tiêm

- Carbendazim và Dimethoate
- 10 ng
- % RSD <10%



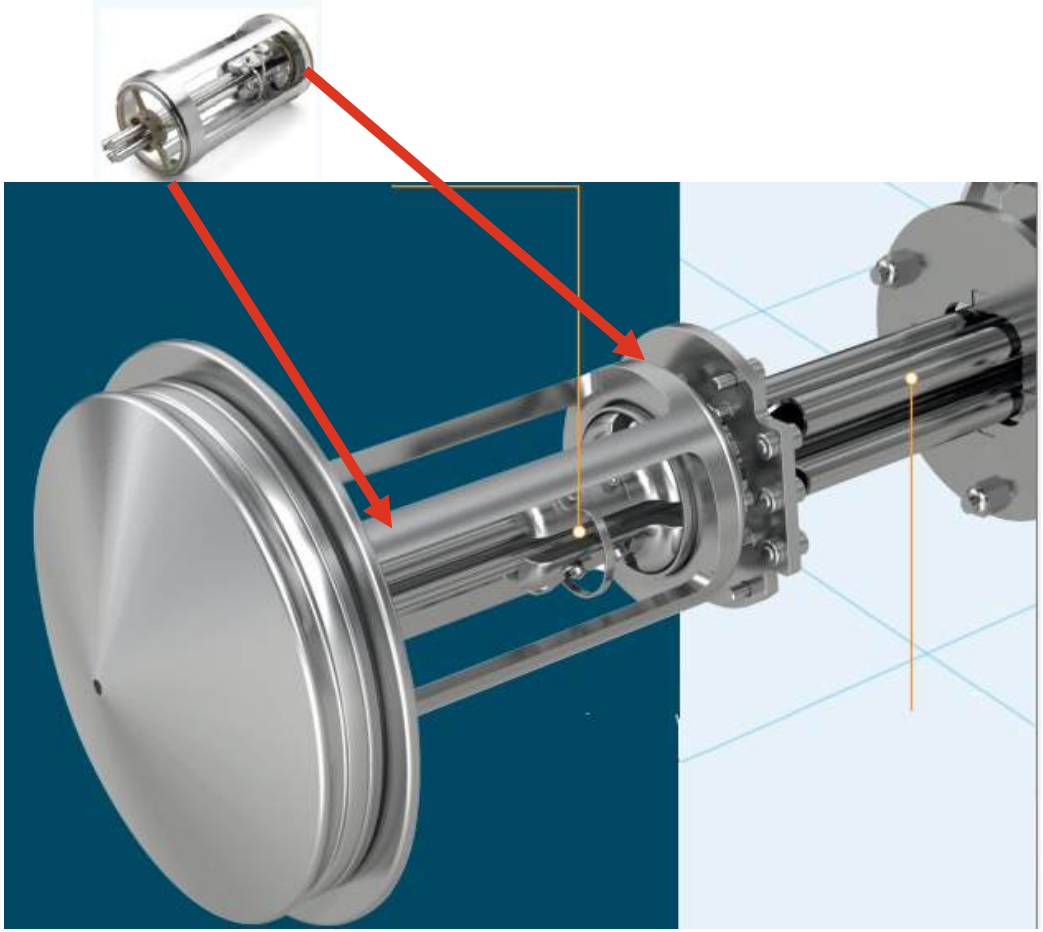




Compound	# of Values	Raw Area %CV	Area Ratio %CV
Carbendazim	2150	3.61	0.82
Dimethoate	2150	4.09	0.72

6/30/25

1/THIẾT KẾ Truyền dẫn ion QJET



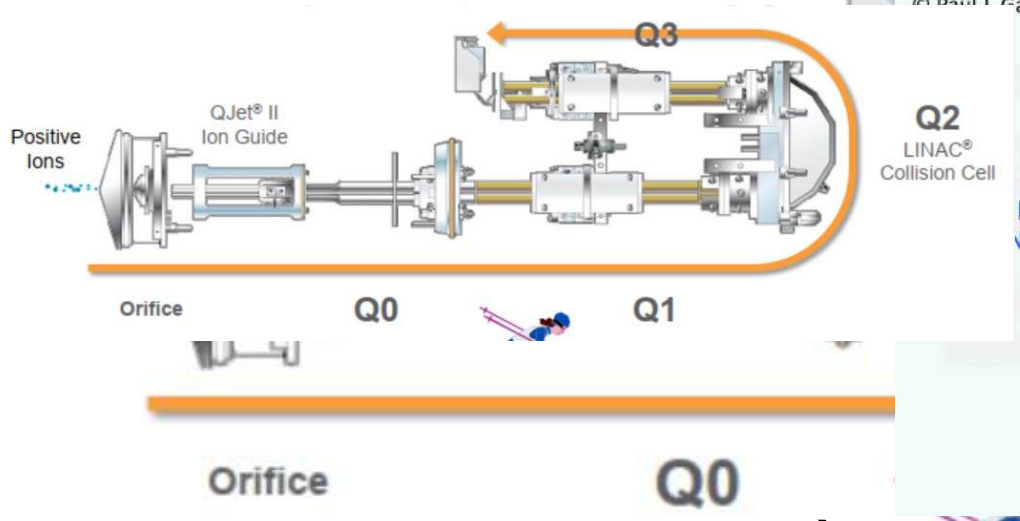
Sử dụng dao động RF để truyền dẫn và tập trung ion, giữ ion trong vùng Quỹ đạo bền, **tăng độ nhạy 4 lần**.

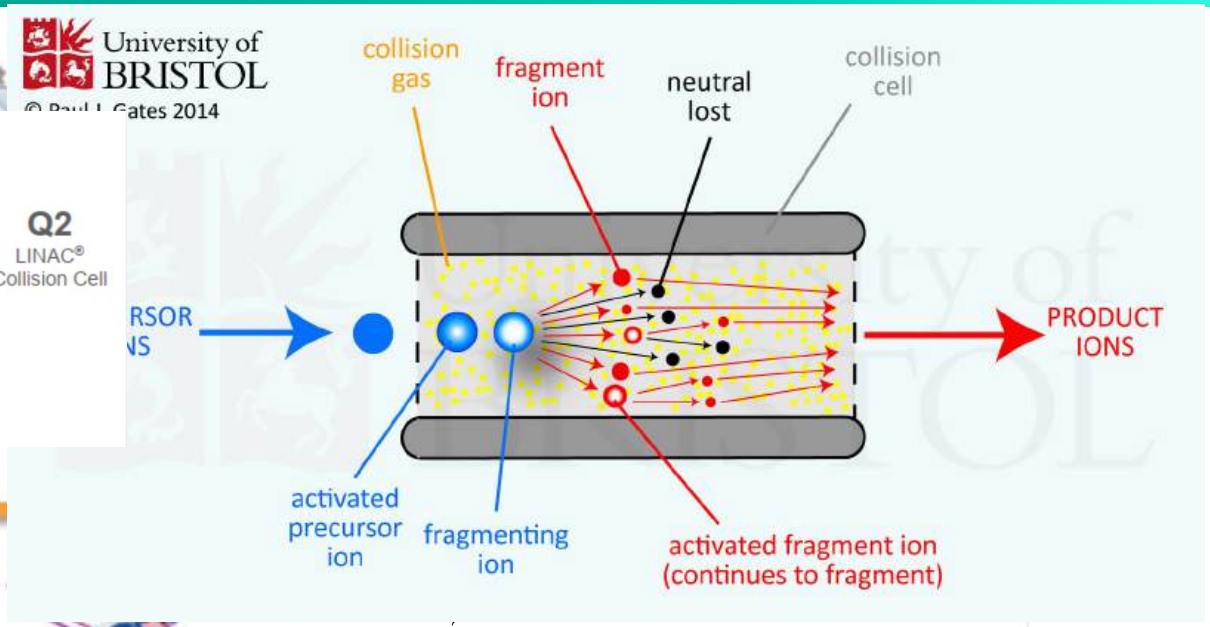
Thiết kế dạng tứ cực, không tiêu hao dễ dàng vệ sinh

Loại bỏ thành phần trung hòa, bảo vệ bộ tứ cực phân tích khối Q1

Làm bước đệm chân không giữa nguồn ion hóa ở áp suất thường và chân không sâu của bộ phân tích khối.

1/THIẾT KẾ Bộ cell va chạm – Q2 cong 180 độ

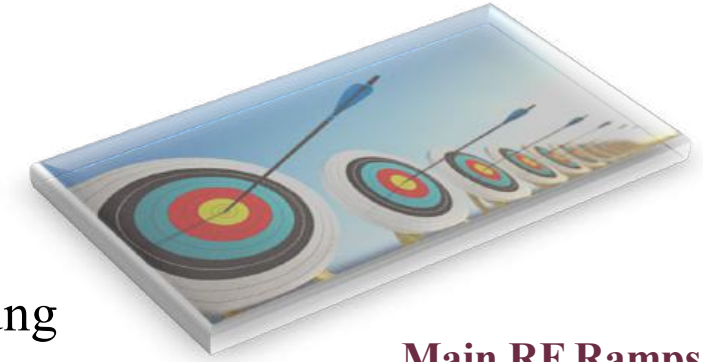


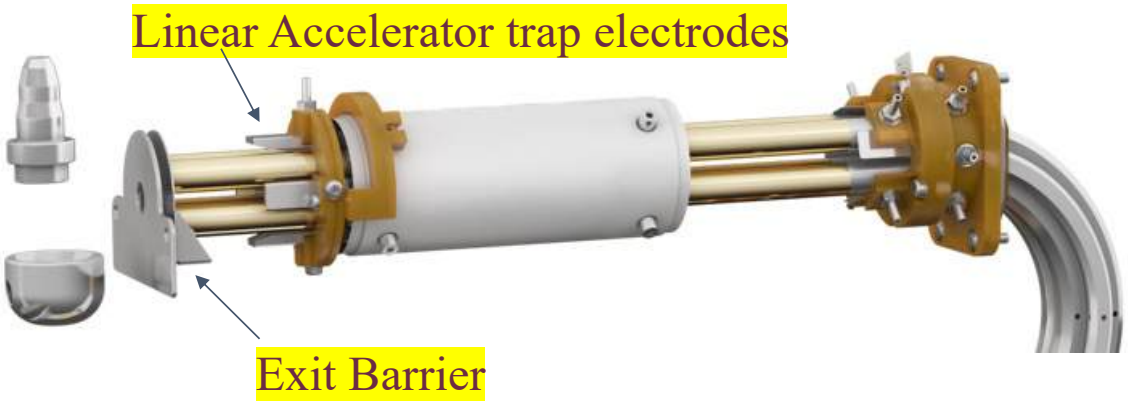


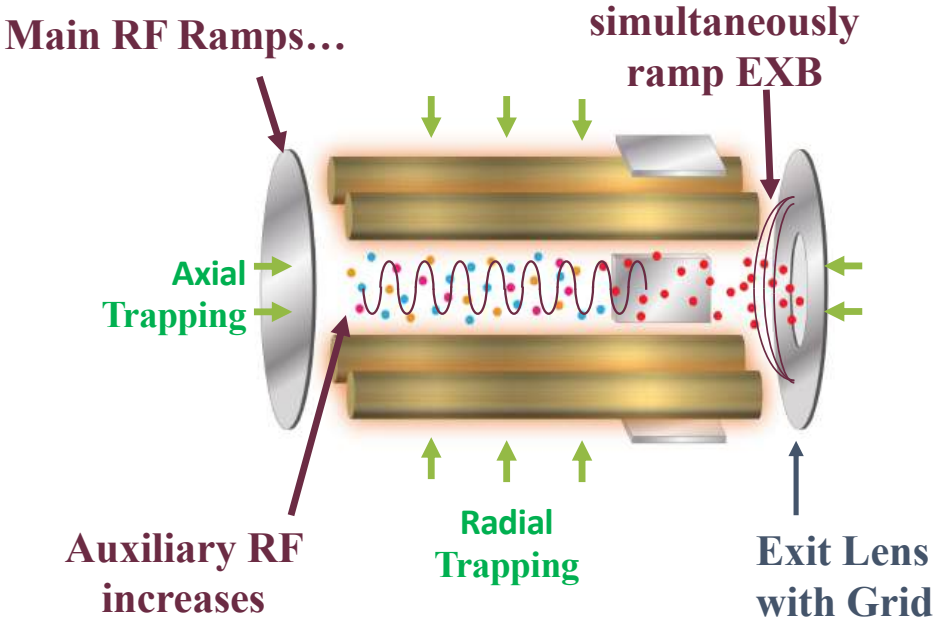
- **Hiệu năng phân mảnh cao** → tăng độ nhạy đáng kể
- Loại các thành phần trung hòa, **giảm nhiễu nền** và **tránh hiện tượng nhiễm chéo** ion con từ 2 ion mẹ khác nhau (crosstalk).
- Thiết kế cong **giảm không gian**, MS được thế kế nhỏ gọn hơn nhưng vẫn đảm bảo được hiệu năng.

THIẾT KẾ
QTRAP – Công nghệ độc quyền Sciex

- Bẫy ion tuyến tính trên Q3
- **QTRAP Ready** trong phần cứng
- => chỉ cần thêm **License** để sử dụng
  - ➢ Tăng độ nhạy chế độ scan hơn 20 lần



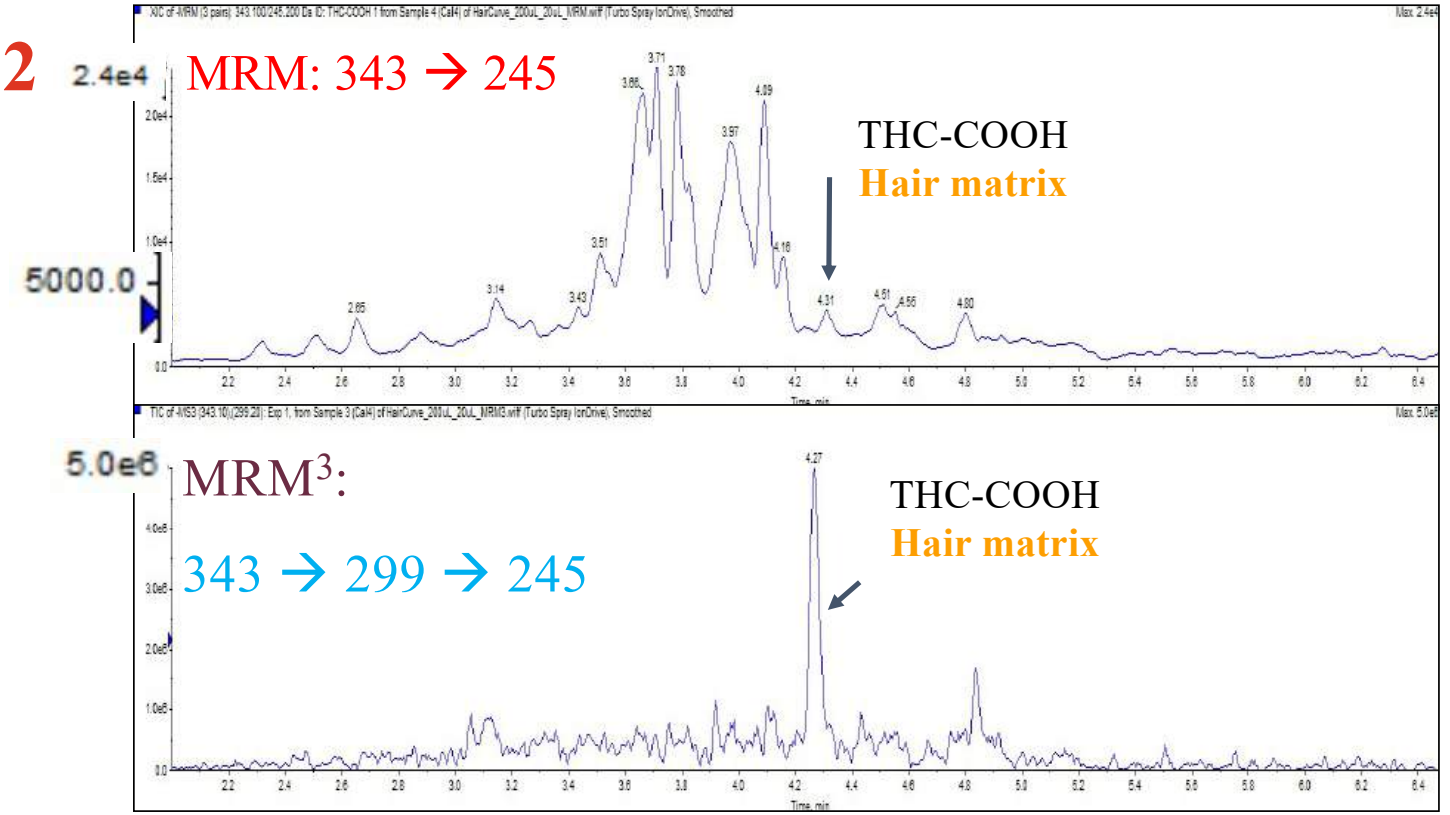




1/THIẾT KẾ
QTRAP® MS/MS – Hai thiết bị trong một

Tăng độ nhạy chế độ scan hơn 20 lần

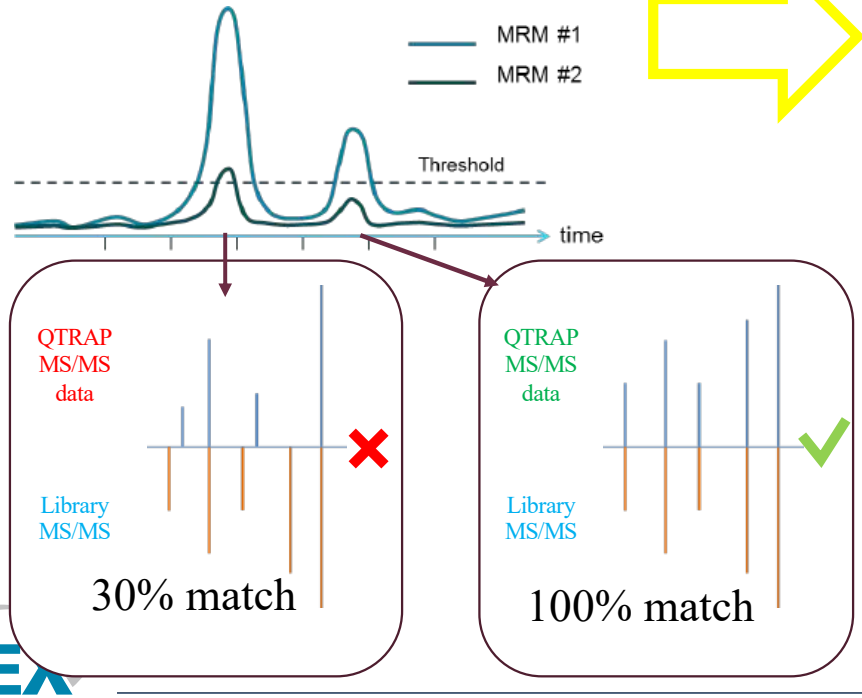
- **Phân mảnh lần 2 (MS/MS/MS)**
  - Tăng độ chọn lọc
  - Giảm nhiễu
  - Hạ LOD



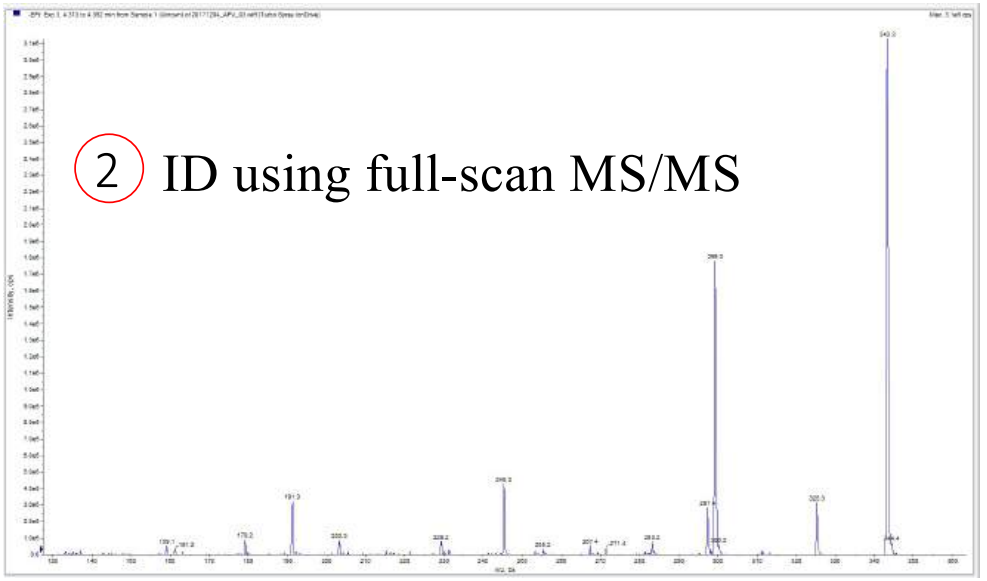
QTRAP Định danh chất phân tích

- Full-scan MS/MS
- **Định lượng và định tính chỉ trong một lần tiêm!**

1 Quantitation via MRM analysis



Enhanced Product Ion (EPI) scan of m/z 343.3 in negative ESI mode



2 ID using full-scan MS/MS

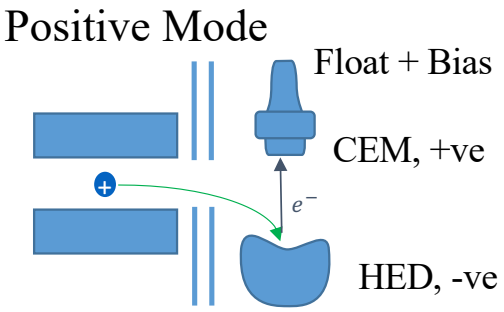
1/THIẾT KẾ
Đột phá tốc độ chuyển cực của đầu dò

- For Fast Polarity Switching

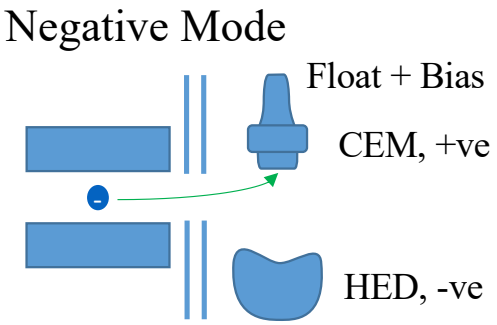
IonDrive High Energy Dynode detector

Positive Ions strike the HED first, Negative ions strike the magnum directly

Magnum to be kept at one polarity

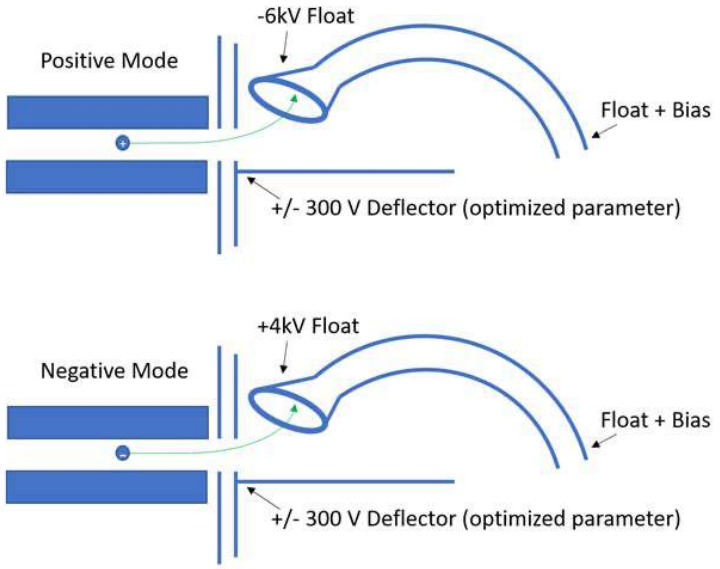


Positive ions first converted to an electron, then electrons are detected by electron multiplier



Negative ions directly detected by electron multiplier

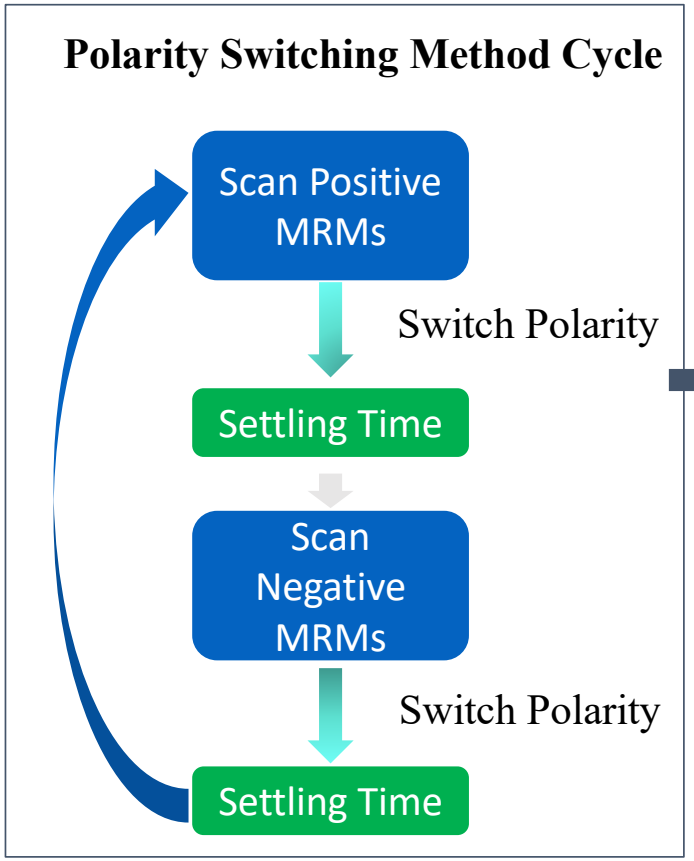
- **CEM detector**
- Both positive and negative ions strike to detector
- Detector voltage must be switched from positive to negative through ground for each cycle

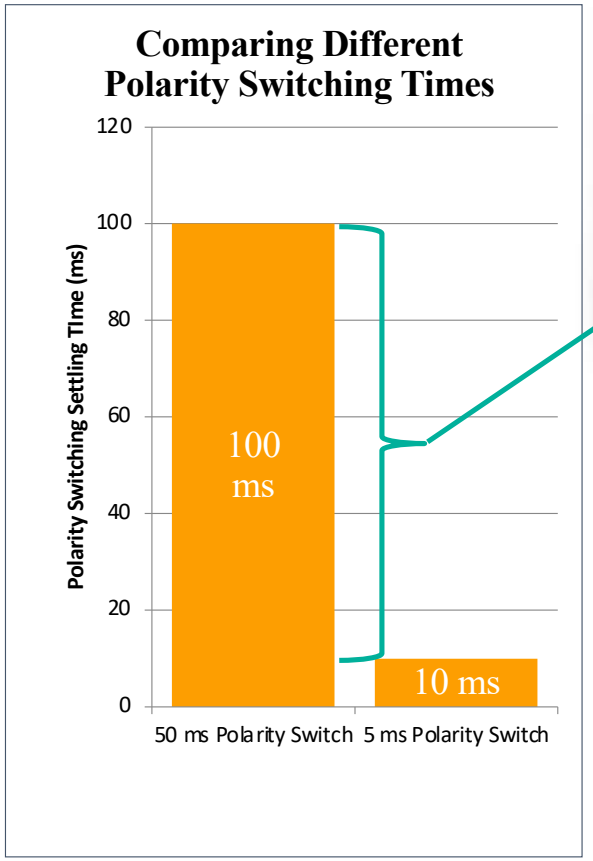


1/THIẾT KẾ

Polarity switching time decreased from 50 to 5 ms

TAKE ADVANTAGE BY SCANNING MORE COMPOUNDS





Gain of 90 msec can be used to add more compounds to each scan, or dwell longer on existing MRMs to improve ion statistics

4000 MRMs

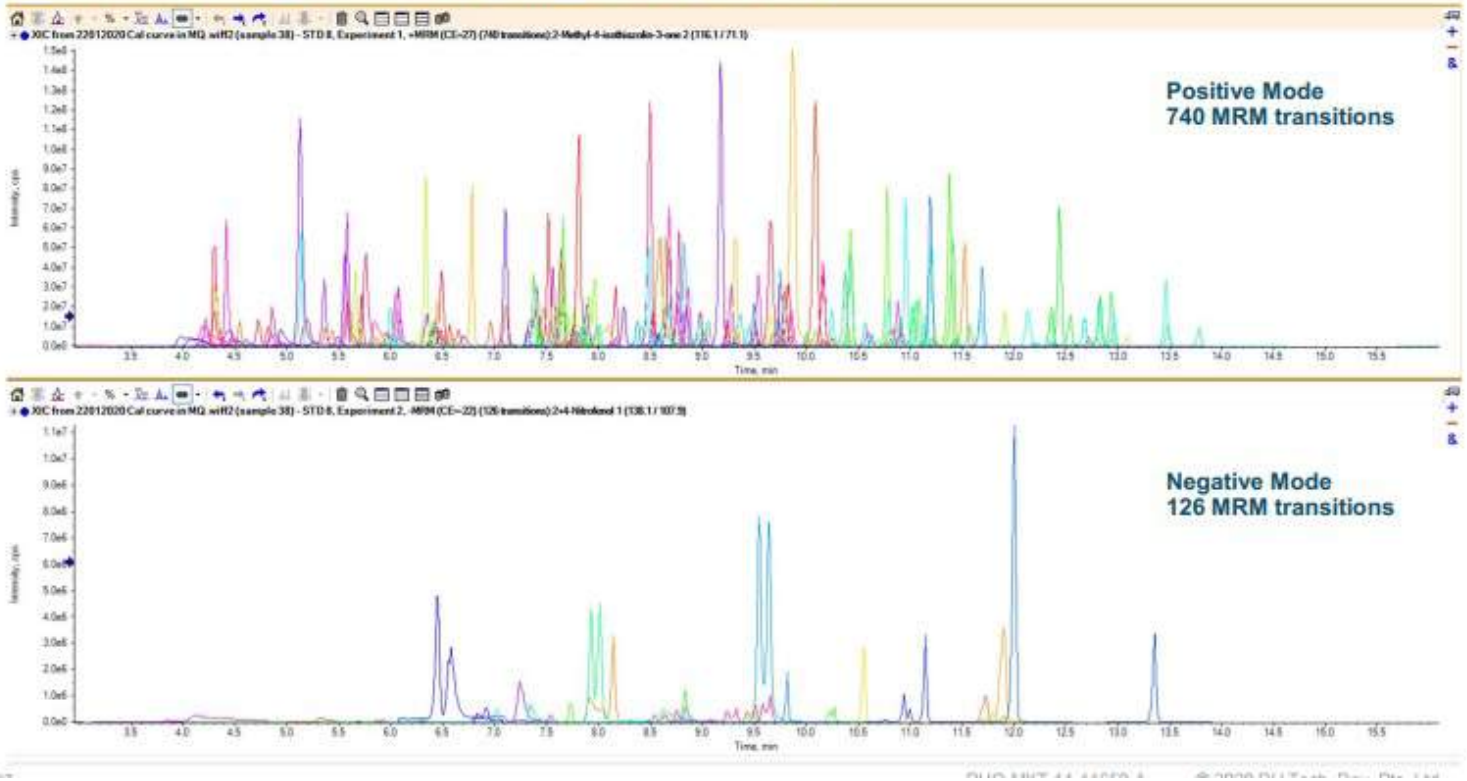
PHÂN TÍCH ĐỒNG THỜI POS VÀ NEG MODE

XIC of positive and negative ionization

SCIEX 7500 System

THE POWER OF PRECISION

ALL QUANTIFIER IONS IN MILLI-Q WATER

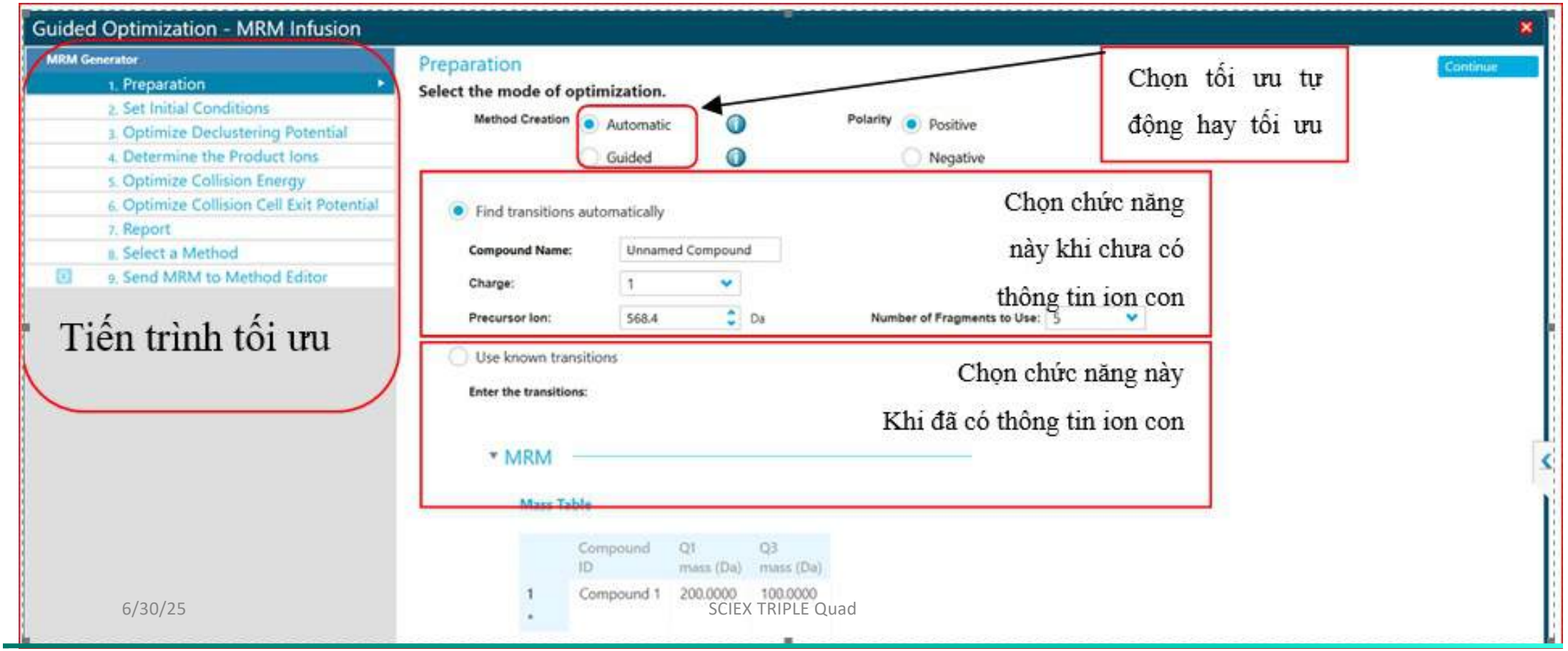


2- SCIEX OS SOFTWARE

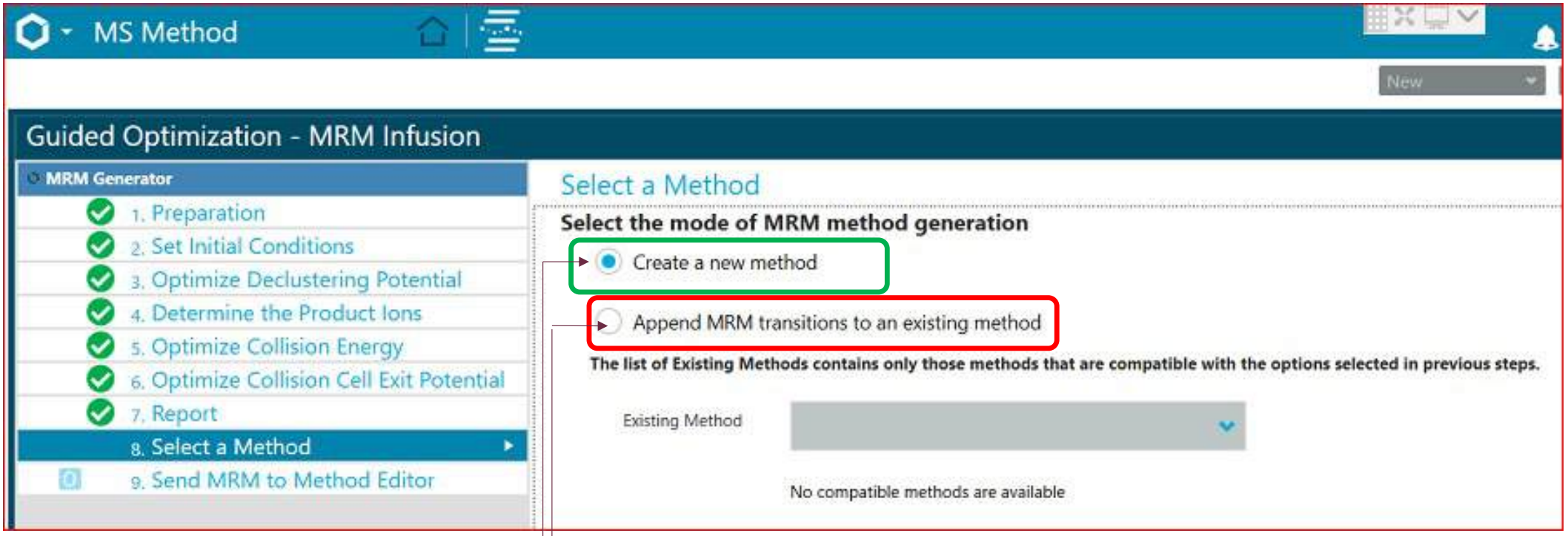
- ❑ Xây dựng phương pháp thần tốc
- ❑Thuật toán tích phân
- ❑Tự động hóa

Xây dựng và phát triển phương pháp thần tốc trên phần mềm SCIEX OS

Tối ưu và thiết lập MS method nhanh chóng, chính xác và tự động



Xây dựng và phát triển phương pháp thần tốc trên phần mềm SCIEX OS



Tạo phương pháp mới

Cập nhật thêm vào phương pháp đã có

PHẦN MỀM SCIEX

Thuật toán lấy tích phân

Project Default Settings

Quantitative Processing

Qualitative Processing

Workspace Layout

Set Project wide defaults for quantitative processing method parameters

Method Defaults

Signal to Noise Algorithm: Relative Noise

Integration Defaults

Integration Algorithm: AutoPeak

Retention Time (RT)

XIC width: 0.02 Da

RT Half Window: 30 sec

Optimize RT Half Window

Peak selection by: Expected RT

Advanced Integration

Pre-Processing

Smoothing: Low

Noise filter / Moving average

Integration

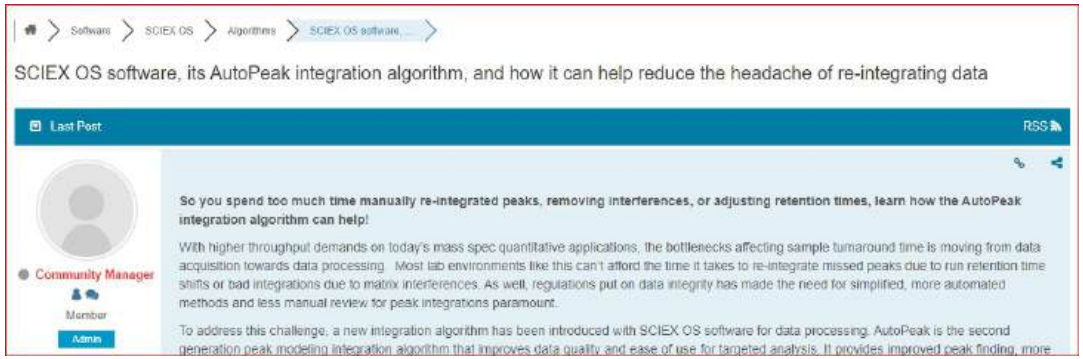
Process by group

Interference resolution: 50 %

Apply to current project | Use for new projects in current data root | More Projects... | Apply | Close | Help

PHẦN MỀM SCIEX

AutoPeak integration algorithm


Software > SCIEX OS > Algorithms > SCIEX OS software

SCIEX OS software, its AutoPeak integration algorithm, and how it can help reduce the headache of re-integrating data

Last Post RSS

Community Manager
Member
Admin

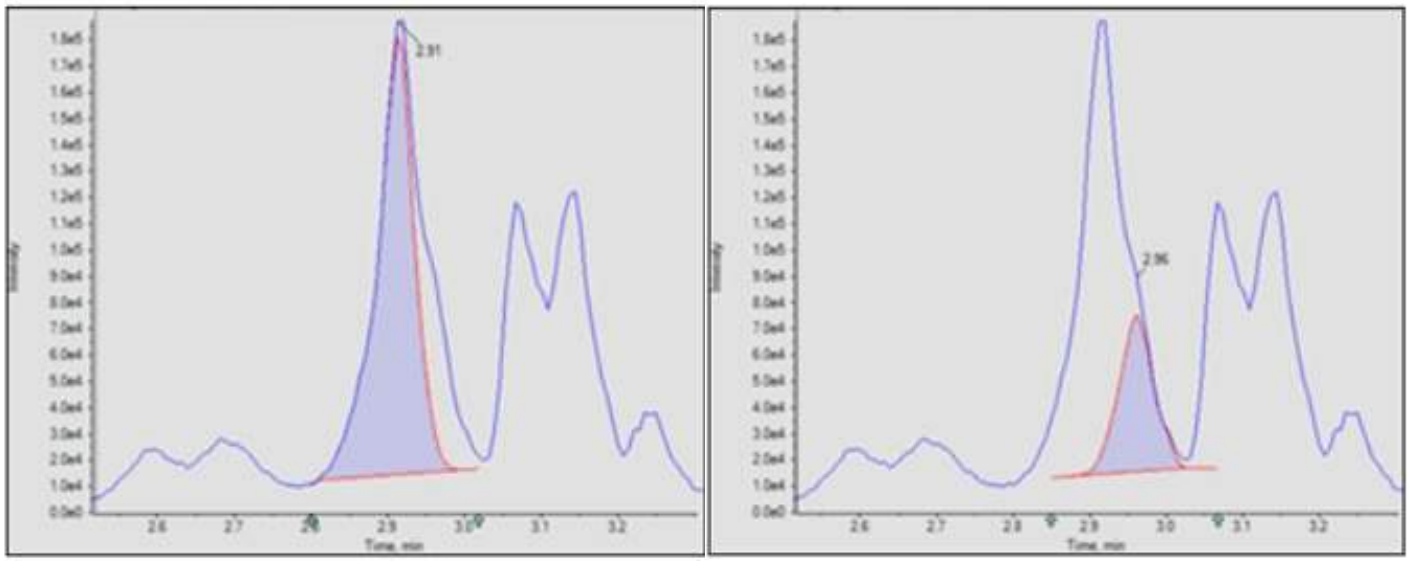
So you spend too much time manually re-integrated peaks, removing interferences, or adjusting retention times, learn how the AutoPeak integration algorithm can help!

With higher throughput demands on today's mass spec quantitative applications, the bottlenecks affecting sample turnaround time is moving from data acquisition towards data processing. Most lab environments like this can't afford the time it takes to re-integrate missed peaks due to run retention time shifts or bad integrations due to matrix interferences. As well, regulations put on data integrity has made the need for simplified, more automated methods and less manual review for peak integrations paramount.

To address this challenge, a new integration algorithm has been introduced with SCIEX OS software for data processing. AutoPeak is the second generation peak modeling integration algorithm that improves data quality and ease of use for targeted analysis. It provides improved peak finding, more

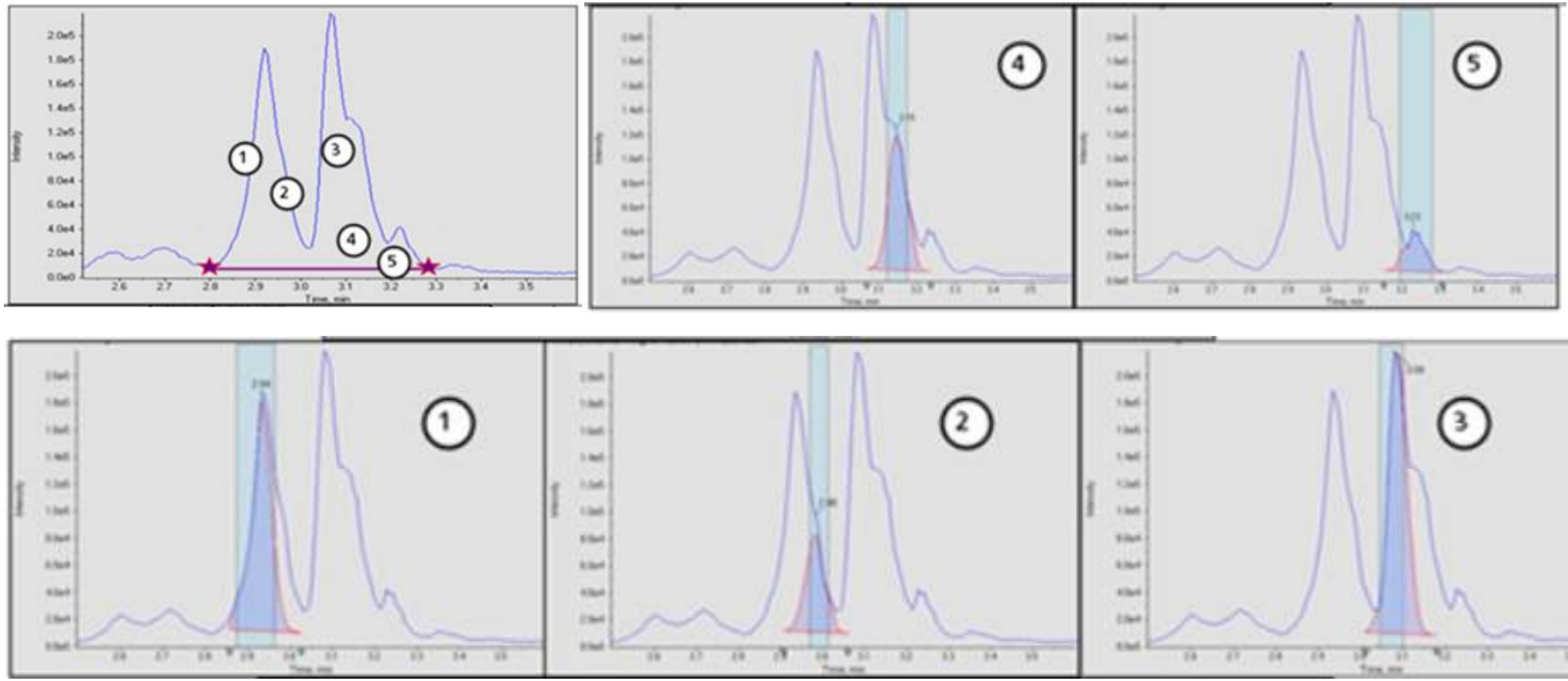
"over 90% of peaks integrated correctly, in the first pass"

"The AutoPeak integration algorithm showed a significant improvement for multiple data types compared to the other algorithms, with over 90% of peaks integrated correctly, in the first pass, without parameter optimization; whereas MultiQuant MQ4 algorithm left a lot of work to be done to optimize the integrations."



PHẦN MỀM SCIEX

AutoPeak integration algorithm

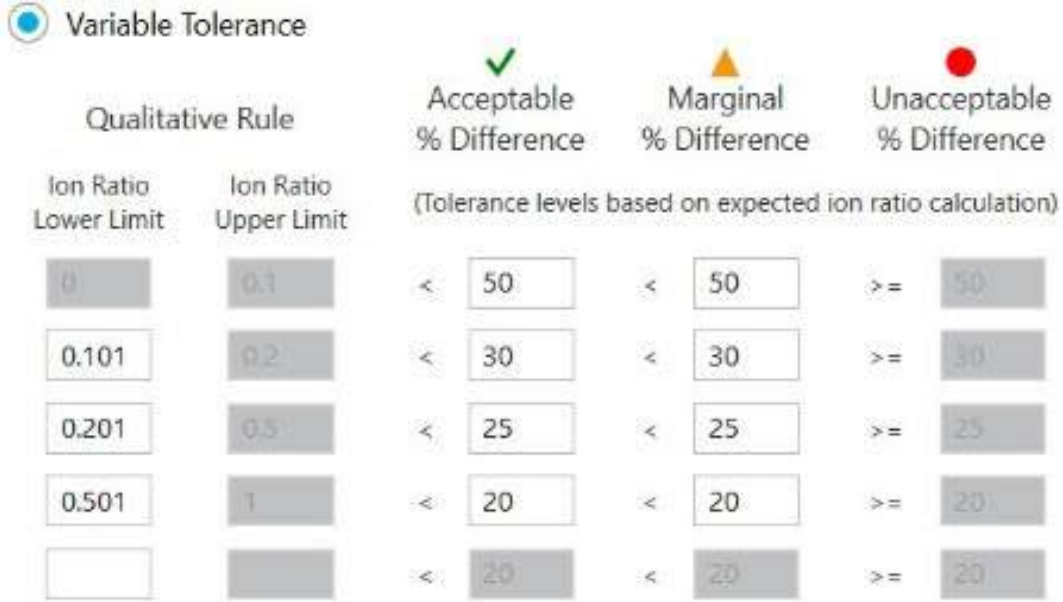


6/30/25

EC 657/2002 AOAC – EC 808

Relative intensity (% of base peak)	EI-GC-MS (relative)	CI-GC-MS, GC-MS^n LC-MS, LC-MS^n (relative)
> 50 %	± 10 %	± 20 %
> 20 % to 50 %	± 15 %	± 25 %
> 10 % t o 20 %	± 20 %	± 30 %
≤ 10 %	± 50 %	± 50 %

Ion Ratio Flagging Rules Set Up in SCIEX OS



PHẦN MỀM SCIEX

Tự động chỉ thị cảnh báo tỉ lệ m/z định lượng và định tính

Custom calculation and filtering packages for SCIEX OS software based on EU guidelines

Document N° SANTE/11312/2021

Table 3. *Identification requirements for different MS techniques.*[2]

MS detector/Characteristics		Acquisition	Requirements for identification	
Resolution	**Typical systems (examples)**		**minimum number of ions**	**additionally**
Unit mass resolution	Single MS quadrupole, ion trap, TOF	full scan, limited *m/z* range, SIM	3 ions	S/N ≥ 3[d] Analyte peaks from both product ions in the extracted ion chromatograms must fully overlap. Ion ratio from sample extracts should be within **±30% (relative)** of average of calibration standards from same sequence
	MS/MS triple quadrupole, ion trap, Q-trap, Q-TOF, Q-Orbitrap	selected or multiple reaction monitoring (SRM, MRM), mass resolution for precursor-ion isolation equal to or better than unit mass resolution	2 product ions	

Thiết bị Sắc ký lỏng Khối phổ Sciex

ExionLC AE - Sciex

Kết nối linh hoạt

- Áp suất tối đa: 15.200 PSI (1048 Bar)
- Độ nhiễm chéo *(carry over)* <0.0003%
- Thời gian tiêm mẫu *(cycle time)* dưới 7 giây
- Điều khiển đồng bộ từ phần mềm **SCIEX OS**

Agilent UHPLC

Shimadzu UHPLC

Chi phí vật tư tiêu hao LC-MSMS Sciex hàng năm

- **Phụ kiện UHPLC**

Maintenance Kit Bơm dung môi – khoảng 40 triệu

Maintenance Kit cho Autosampler – Khoảng 40 triệu

- **Phụ kiện cho đầu dò MS/MS**

Preventive Maintenence kit MS – Khoảng 20 triệu

Dầu cho bơm chân không – Khoảng 4 triệu (khuyến cáo thay 2 năm/lần)

- **Tổng: 105 triệu/năm**
- Do toàn bộ kết cấu truyền dẫn ion sau ion source của Sciex là các bộ tứ cực có thể vệ sinh được dễ dàng nên phụ kiện tiêu hao của đầu dò MS/MS hãng Sciex rất ít, chủ yếu các phụ kiện cơ bản ion source như điện cực, o-ring, ốc nối, dây dẫn dung môi… đã bao gồm trong PM kit nêu ở trên.

Chi phí vật tư tiêu hao LC-MSMS Sciex hàng năm

Tổng cộng: 828*2 + 1827 + 676 + 97.7 = 4159 usd ≈ 4257 usd = 106.4 triệu

Phụ kiện tiêu hao cần thay theo thực tế		số lượng			thời gian sử dụng thực tế
pump seal (Pump)	228-52711-93	4	289	1156	1-2 năm
LINE FILTER ASSY, (Pump)	228-57501-92	1	188	188	2 năm
Needle Seal, SIL-40	228-64364-42	1	177	177	2 năm
HPV rotor (seal)	228-71780-42	1	841	841	1-2 năm
Electrode for the TurboIonSpray® probe	25392	1	142	142	1 năm
Electrode for the APCI probe	25388	1	117	117	1 năm
Oil for Single Stage Roughing Pumps	5064867	1	97.7	97.7	2 năm
		Tổng cộng		2717.95	

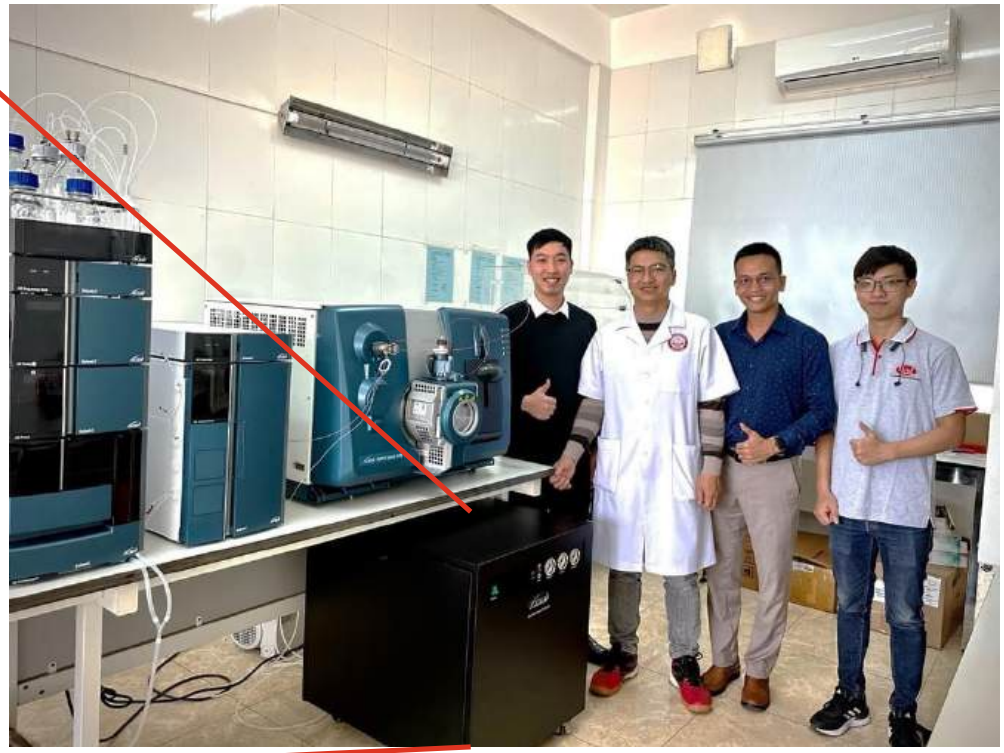
Chi phí vật tư tiêu hao LC-MSMS Sciex hàng năm

- **Phụ kiện máy sinh khí Nito**

  Maintenance kit khoảng 10 triệu

➢SCIEX sử dụng không khí sạch để làm khô mẫu → tiết kiệm khí nito

➢Khí phân mảnh Q2 dùng khí nito từ máy sinh khí (độ tinh khiết 95%), không cần thêm bình khí Argon hoặc Nito (99.99%)



6/30/25

3/ SCIEX - VIET NGUYEN Đồng hành cùng khách hàng

SCIEX

- Tối đa hóa hiệu năng phần cứng
- Phần mềm điều khiển và xử lý dữ liệu

SCIEX - VIET NGUYEN

Đồng hành cùng khách hàng

VIET NGUYEN

- Kỹ thuật giàu kinh nghiệm
- Ứng dụng chuyên sâu

SCIEX
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VIET GROUP

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- vietCALIB® (Hiệu chuẩn)
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SEAL – Mỹ/ Đức/ Hà Lan
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mP membraPure
WATER · TOC · AMINO · ION
membraPure – Đức
(Độc quyền)

BRUKER
BRUKER – Đức
(Đại diện chính hãng)

SI Analytics
a xylem brand
SI – Đức
(Đại diện chính hãng)

BS Bellingham + Stanley
BS – Anh
(Độc quyền)

TELEDYNE TEKMAR
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Certificate of Completion

Nguyen Khac Manh

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10/15/2023

SCIEX Certified LC/MS Practitioner

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Joe Lepore
Global Director
Commercial And Service Training

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Authorized Service Provider Certificate of Completion

Nguyen Khac Manh

Date Issued: 9/18/2023

Is Trained to Perform Installation Services

Mass Spectrometers: 3500/4500/5500/6500 Models
LC Systems: Shimadzu & Exlon Models

SCIEX confirms that the above-named engineer has been trained on Installation is eligible to receive continuing training on installation and has the ability to train customers on operation of the instruments listed in this record. Through the course of employment with SCIEX as an SCIEX Authorized Service Provider, this engineer has access to the latest SCIEX product information.

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Joe Lepore
Global Director
Commercial And Service Training

Satish Chandra Kumar Guttarlapalli
FSE Manager

3/Kỹ thuật Đào tạo chính hãng

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Tran The Duy

SCIEX Triple Quad 5500+ LCMS/MS Service Training

7/16/2023

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Tran The Duy

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9/21/2023

SCIEX Certified LC/MS Practitioner

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Joe Lepore
Global Director
Commercial And Service Training

Certificate of Completion

Le Anh Tuan

SCIEX Triple Quad 5500+ LCMS/MS Service Training

10/12/2023

SCIEX Certified LC/MS Practitioner

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Le Anh Tuan

Date Issued: 9/18/2023

Is Trained to Perform Installation Services

Mass Spectrometers: 3500/4500/5500/6500 Models
LC Systems: Shimadzu & Exlon Models

SCIEX confirms that the above-named engineer has been trained on installation is eligible to receive continuing training on installation and has the ability to train customers on operation of the instruments listed in this record. Through the course of employment with SCIEX as an SCIEX Authorized Service Provider, this engineer has access to the latest SCIEX product information.

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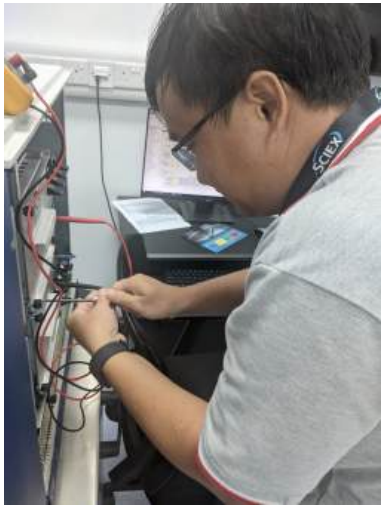
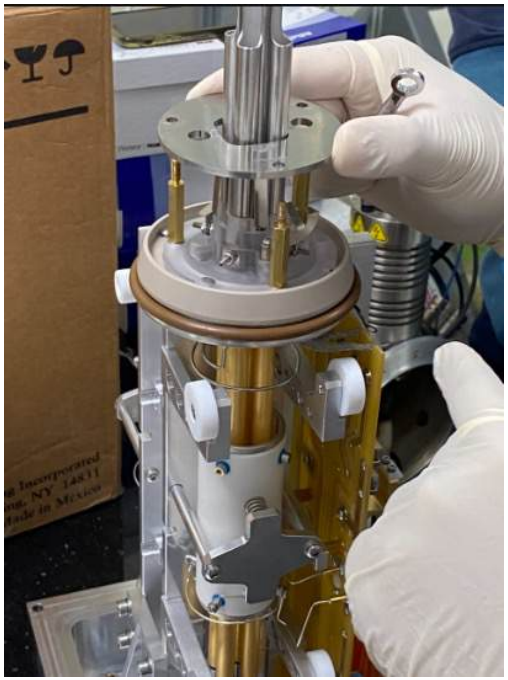
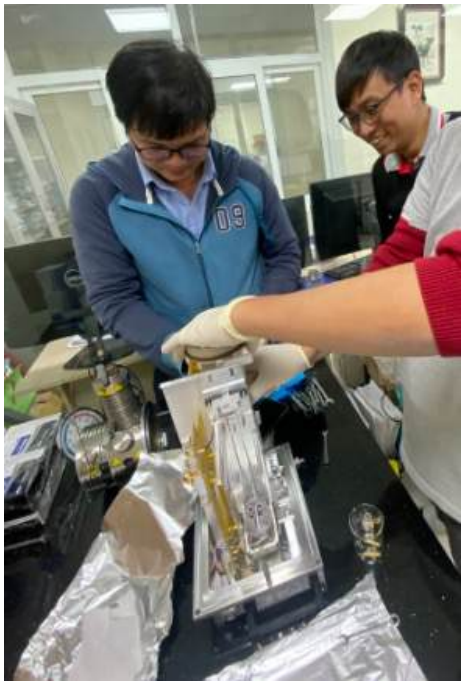
Joe Lepare
Global Director
Commercial And Service Training

Satish Chandra Kumar Guttarlapalli
FSE Manager

Kỹ thuật Đội ngũ Kỹ thuật tận tâm

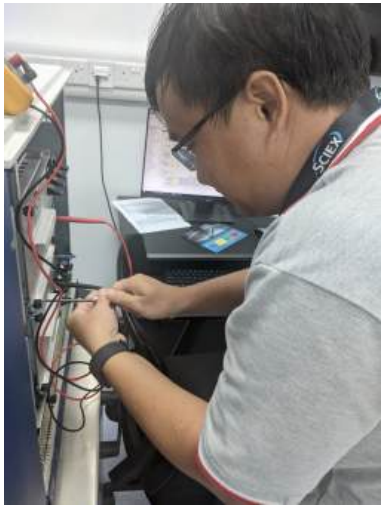


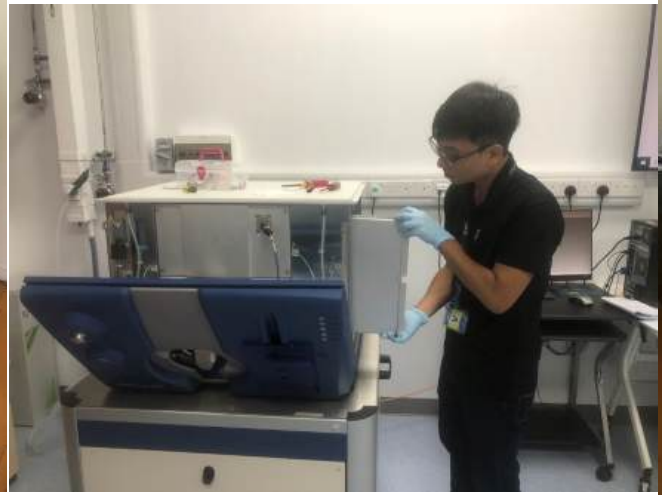




Phản hồi – kiểm tra và chuẩn đoán sự cố nhanh chóng, chính xác và hiệu quả

Đội ngũ Kỹ thuật tận tâm





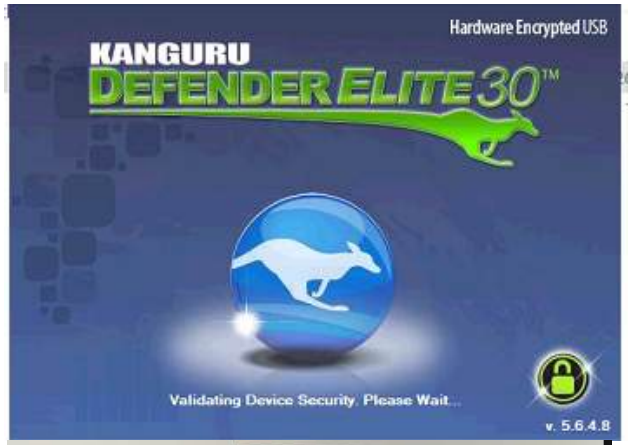
Phản hồi – kiểm tra và chuẩn đoán sự cố nhanh chóng, chính xác và hiệu quả

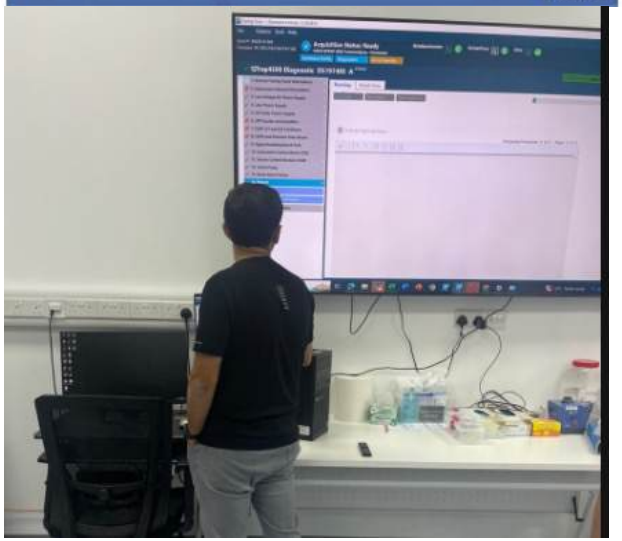
Field Service Support Software

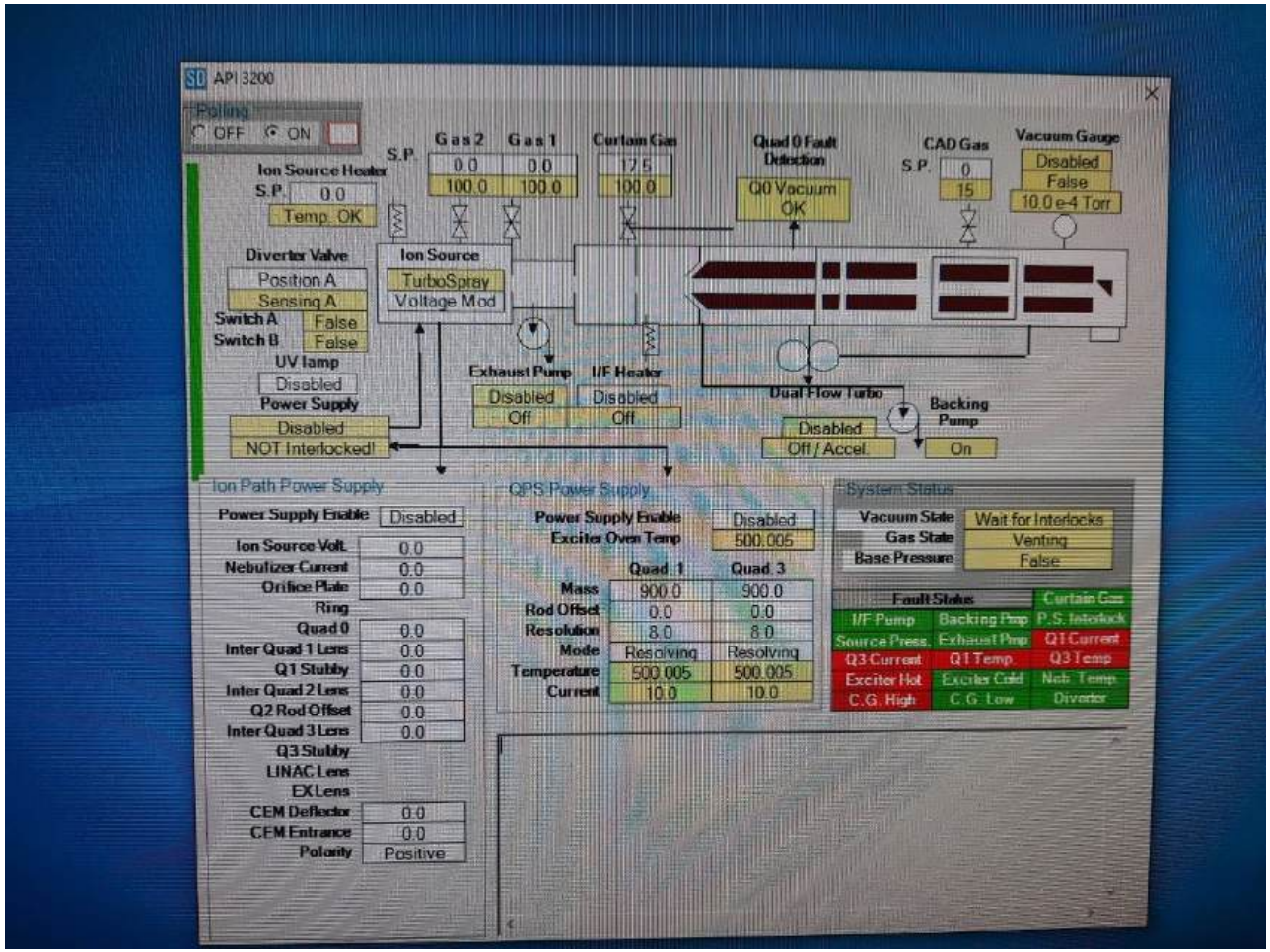
Provide an overview and supplemental information for the Field Service Support Software used by SCIEX Field Service Engineers (FSEs) and Channel Partners via a Kanguru™ Flash Drive to configure, test, troubleshoot and maintain the Blueline Series Mass Spectrometers.

- Analyst® Service Diagnostics (ASD)
- Tuning Tools (TT)
- Field Service Data Collector (FSDC)
- Agilent T-plus

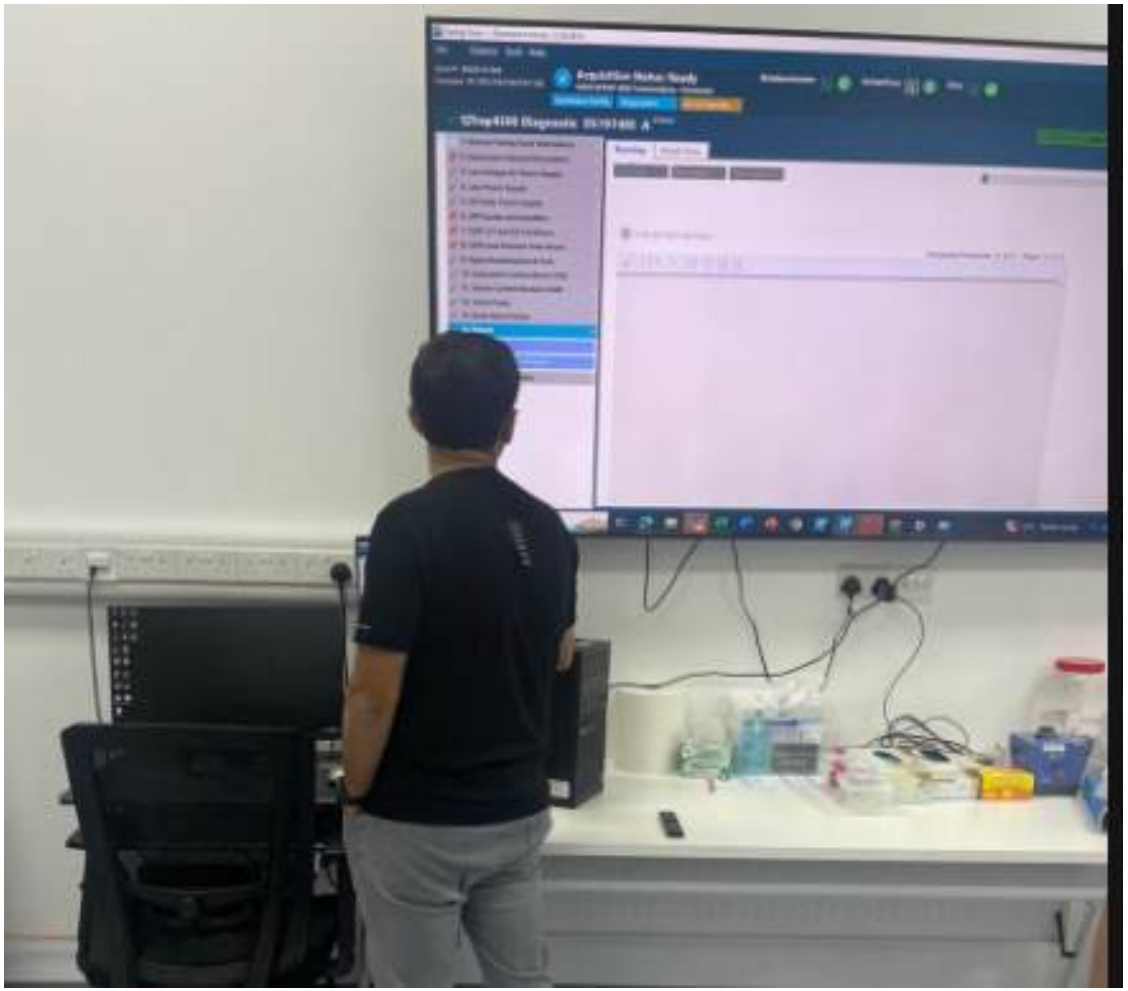
Công cụ hỗ trợ kỹ thuật, chuẩn đoán chính xác hiện trạng máy







Công cụ hỗ trợ kỹ thuật, chuẩn đoán chính xác hiện trạng máy



SCIEX **QTrap4500 Mass Spectrometer Diagnostic Report** 10/13/2023 3:55 PM

User Account	localadmin		
Instrument Model:	QTrap4500	Target Instrument:	QTrap4500
Ion Source:	TurbolonSpray	Optimization Set:	QTrap4500 Diagnostic
Serial Number:	BW20141504	Doc Num Revision:	D5197485 A

Name	Minimum	Maximum	Readback	Status
Ambient Temperature	15.00	35.00	23.87	Pass

Check the Pressure at CAD 0 **Pass** The pressure 6.9E-06 Torr was within tolerance levels

3. Low Voltage DC Power Supply

Step	Result	Details
Verify Low Voltage DC Power supplies	Pass	

Name	Set Point	Minimum	Maximum	Readback	Status
Electronics DC Power Supply +19.0	N/A	17.00	21.00	19.19	Pass
Electronics DC Power Supply +24.0	N/A	22.00	26.00	23.81	Pass
Electronics DC Power Supply +3.3	N/A	3.10	3.50	3.30	Pass
Electronics DC Power Supply +5.0	N/A	4.50	5.50	5.00	Pass
Electronics DC Power	N/A	-21.00	-17.00	-18.90	Pass

Disclaimer & copyright:

- This video is intended for MS Vision trained & certified customers only.
- Please observe applicable safety precautions; make sure the instrument is vented, the power is switched off, and disconnected from the mains. Please wear gloves when needed.
- Spectrometry Vision B.V. cannot be held liable for any damages or personal injuries that may occur.
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ISO/IEC 17025:2017

Fields of Accreditation: Metrology - Calibration
Accreditation No.: VLAC-1.0413

DIRECTOR

No.: AOSC-1.0413.1
Issue date: 18/3/2022
Expiration date: 17/3/2027

Assoc. Prof. NGUYEN THI KHANH TRAM

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Ứng dụng

Perfluorocarbons
PFCs

Pesticide

REGULATIONs

On April 10, 2024, EPA announced the final National Primary Drinking Water Regulation (NPDWR) for six PFAS.

EPA established legally enforceable levels, called Maximum Contaminant Levels (MCLs), for six PFAS in drinking water: PFOA, PFOS, PFHxS, PFNA, and HFPO-DA as contaminants with individual MCLs, and PFAS mixtures containing at least two or more of PFHxS, PFNA, HFPO-DA, and PFBS using a Hazard Index MCL to account for the combined and co-occurring levels of these PFAS in drinking water. EPA also finalized health-based, non-enforceable Maximum Contaminant Level Goals (MCLGs) for these PFAS.

Compound	Final MCLG	Final MCL (enforceable levels)
PFOA	Zero	4.0 parts per trillion (ppt) (also expressed as ng/L)
PFOS	Zero	4.0 ppt
PFHxS	10 ppt	10 ppt
PFNA	10 ppt	10 ppt
HFPO-DA (commonly known as GenX Chemicals)	10 ppt	10 ppt
Mixtures containing two or more of PFHxS, PFNA, HFPO-DA, and PFBS	1 (unitless) Hazard Index	1 (unitless) Hazard Index

PFAS/PFOS -SCIEX Triple Quad 4500 system

For Research Use Only. Not for Use In Diagnostic Procedures.

Food and Environmental

Analysis of PFAS at low ppt levels in drinking water via EPA method 533

Using the SCIEX Triple Quad 4500 system

Matthew Standland, Craig M. Butt, and Matthew Noestheden
SCIEX, USA

Per- and polyfluorinated alkyl substances (PFAS) are a group of manmade chemicals that have been used for decades in a large host of applications. A recent review focused on 1,400 unique PFAS species[1] although current OECD databases have upwards of 4,500 unique PFAS compounds.[2] The resilience of some PFAS to degradation and their potential for biological harm have spurred health and environmental concerns amongst regulatory agencies. The CDC's national biomonitoring program has found evidence of widespread presence of certain PFAS residues in human serum[3]. One possible route of human exposure is from PFAS contaminated water.[4]

PFAS are known to enter the water supply and bioaccumulate in watershed ecosystems. This can be a health issue for humans who directly consume contaminated water. The US EPA published the fifth unregulated contaminant monitoring rule

(UCMR5)[5], which proposes maximum residue limits (MRL) for PFAS residues in EPA method 533. Representative chemical structures are shown in Figure 1.

- EPA method 533. 6
- 250 mL of water was passed through Phenomenex Strata-X-AW Weak Anion 500 mg/6 mL.
- Eluted with methanol containing 2% ammonium hydroxide, dried down under nitrogen gas, and reconstituted in 1 mL of 80:20 methanol/ water

Làm giàu 250 lần

6/30/25

PFAS/PFOS SCIEX Triple Quad 4500 system

Chromatography: ExionLC AC system using a 12-minute gradient.

- The Phenomenex Gemini C18 (100 x 3.0 mm, 3.0 µm, 00D-4439-Y0)
- The delay column used was a Phenomenex Luna C18 (30 x 3.0 mm, 00A-4252-Y0).
- **The injection volume was 5 µL**
- Column temperature of 40°C using a flow rate of 0.6 mL/min.
- Mobile phases consisted of 10 mM ammonium acetate and methanol with 10 mM ammonium acetate.

PFAS/PFOS SCIEX Triple Quad 4500 system

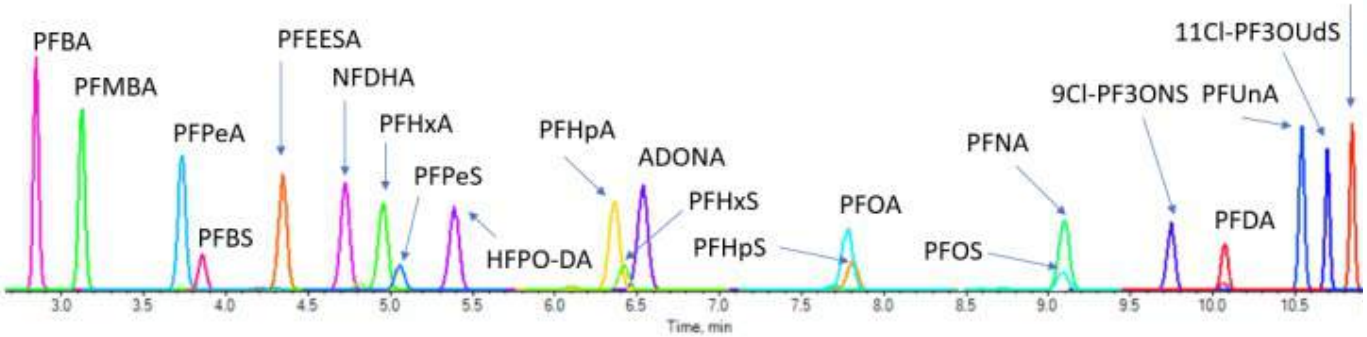

Figure 3. TIC chromatogram of 15 ng/mL solvent standard showing chromatographic separation of EPA method 533 PFAS analytes. Represent target residues are displayed here as extracted from their corresponding MRM in the mass spectrometry method.

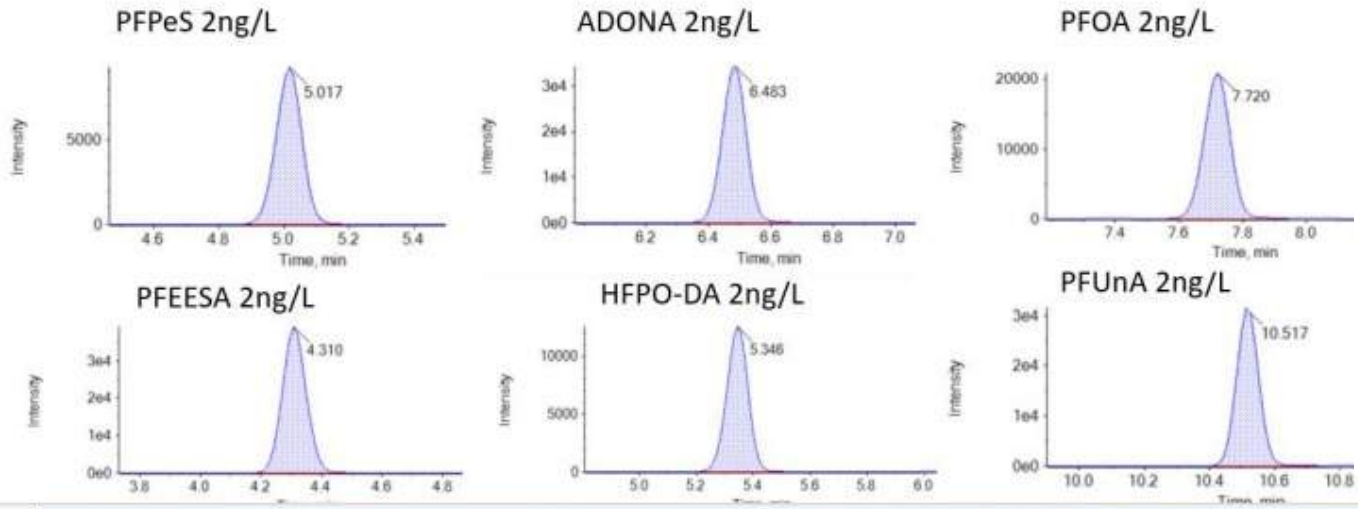

Table 1. Accuracy and precision of method. Precision CV measurements were based on the area response of the quantifier transition obtained from 7 replicates of water fortified at 6 ng/L. Mean % area values are based on 7 replicates of water fortified at 4 ng/L. Verified MRLs were established using EPA method 533 criteria. All residues passed data quality requirements in section 9 of EPA method 533.

Compound	Verified MRL (ng/L)	CV (%)	Mean accuracy (%)	UCMR5 MRL (ng/L)
11Cl-PF3OUdS	2	6.6	85.2	5
9Cl-PF3ONS	2	7.4	96.8	2
ADONA	2	3.5	94.9	3
HFPO-DA	2	5.4	94.5	5
NFDHA	2	4.3	91.1	20
PFBA	2	7.2	109.8	5
PFBS	2	6.6	97.9	3
8:2 FTS	3	9.5	117.0	5
PFDA	2	4.5	96.2	3
PFDoA	2	6.1	102.6	3
PFEESA	2	7.3	102.6	3
PFHpS	2	7.5	98.8	3
PFHpA	2	7.5	104.4	3
4:2 FTS	3	6.5	122.2	3
PFHxS	2	7.7	99.9	3
PFHxA	2	4.2	98.4	3

PFAS/PFOS SCIEX Triple Quad 4500 system

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HFPO-DA	2	5.4	94.5	5
NFDHA	2	4.3	91.1	20
PFBA	2	7.2	109.8	5
PFBS	2	6.6	97.9	3
8:2 FTS	3	9.5	117.0	5
PFDA	2	4.5	96.2	3
PFDoA	2	6.1	102.6	3
PFEESA	2	7.3	102.6	3
PFHpS	2	7.5	98.8	3
PFHpA	2	7.5	104.4	3
4:2 FTS	3	6.5	122.2	3
PFHxS	2	7.7	99.9	3
PFHxA	2	4.2	98.4	3
PFMPA	2	3.4	100.4	3
PFNA	2	4.2	93.3	4
6:2 FTS	3	5.1	112.9	5
PFOS	2	8.9	98.8	4
PFOA	2	8.0	100.0	4
PFPeA	2	4.2	101.4	3
PFPeS	2	7.1	98.5	4
PFUnA	2	4.1	100.2	2

Food and Environmental

SCIEX The Power of Precision

Quantitation of PFASs in water samples using LC-MS/MS large-volume direct injection and solid phase extraction

Simon Roberts[1], KC Hyland[1], Craig Butt[1], Scott Krepich[2], Eric Redman[3], and Christopher Borton[1]
[1]SCIEX, USA; [2]Phenomenex, USA; [3]TestAmerica Laboratories, Sacramento, USA

PFASs are unique chemicals whose physicochemical properties make them important for use in a variety of industrial and consumer products including carpets, cookware, food packaging, fire suppressants, and others[1]. Chemically, PFASs are aliphatic structures containing one or more C atoms on which H substituents have been replaced by F atoms. Classification and naming is typically by the particular functional group present, such as carboxylic acids, sulfonates, phosphonic acids, etc., as well as the length of the carbon chain. Desirable in various industrial applications for their chemical stability and low reactivity, these properties also make PFASs highly resistant to degradation in aquatic environments. Typical concentrations of PFASs found in various environmental water sources range from pg/L to µg/L levels[2].

This application note presents two methods for the quantitation

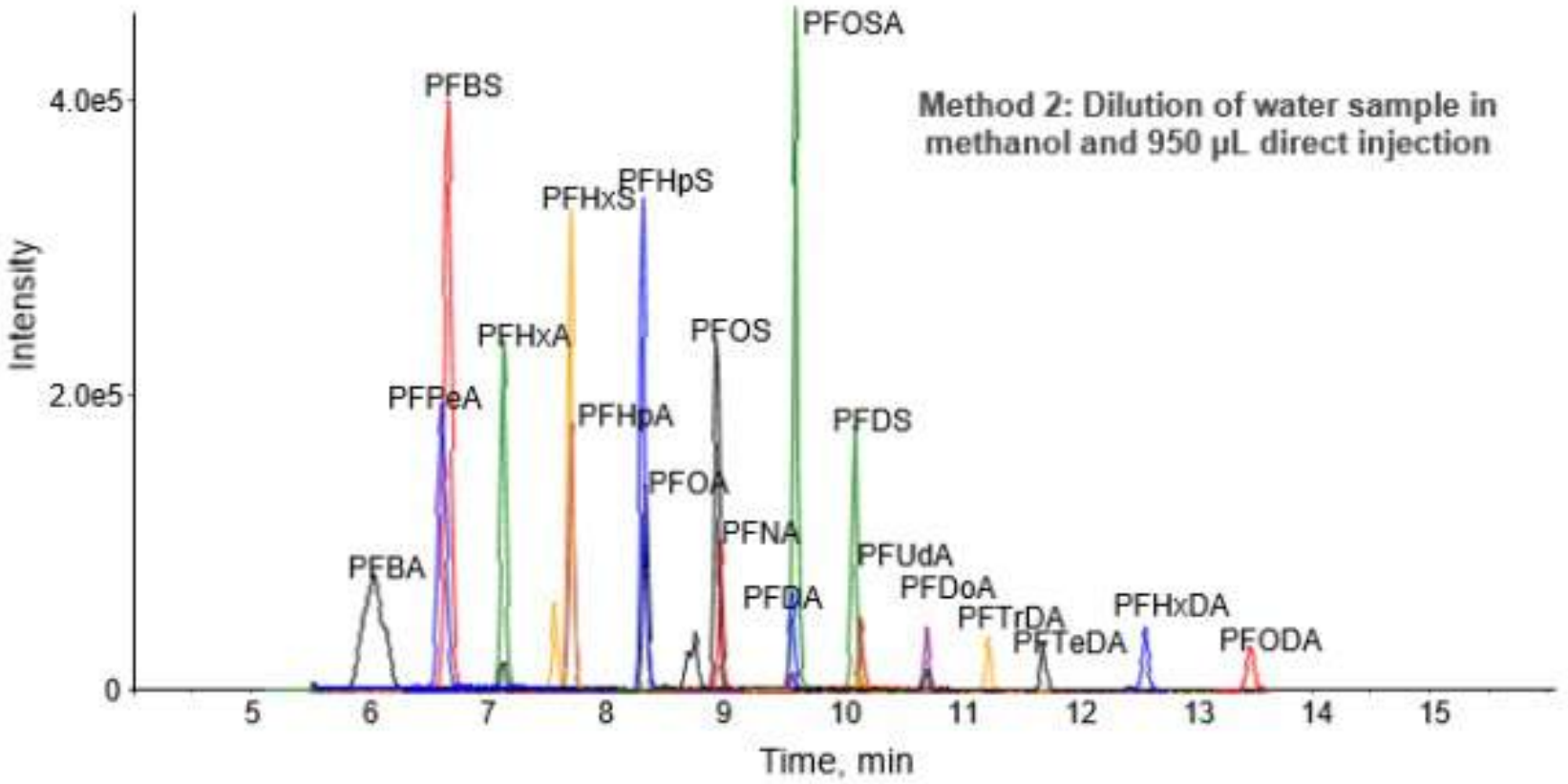


Figure 4. Method 2 chromatography. Chromatogram of a 10 ng/L matrix spike into groundwater that was diluted with methanol and injected according to Method 2.

Table 5. Calibration curves for method 1 and 2. Sensitivity and linearity from 25 to 20,000 ng/L and 1 to 200 ng/L (coefficient of regression, R2) using Method 1 and Method 2, respectively. S/N calculated using MultiQuant Software 3.0.2.

	Method 1				Method 2			
Compound	Calibration range (ng/L)	Linear correlation (R2)	S/N of 25 ng/L standard	Accuracy of 25 ng/L standard	Calibration range (ng/L)	Linear correlation (R2)	S/N of 1 ng/L standard	Accuracy of 1 ng/L standard
PFCAs								
PFBA	*25-20,000*	*0.997*	*108*	*104%*	*1-200*	*0.997*	*328*	*97%*
PFPeA	*25-20,000*	*0.998*	*88*	*103%*	*1-200*	*0.999*	*137*	*101%*
PFHxA	*25-20,000*	*0.998*	*104*	*93%*	*1-200*	*0.999*	*284*	*101%*
PFHpA	*50-20,000*	*0.999*	*116*	*101%*	*1-200*	*0.993*	*267*	*96%*
PFOA	*25-20,000*	*0.999*	*117*	*106%*	*1-200*	*0.999*	*113*	*99%*
PFNA	*25-20,000*	*0.990*	*91*	*109%*	*1-200*	*0.999*	*137*	*101%*
PFDA	*25-20,000*	*0.998*	*103*	*105%*	*1-200*	*0.997*	*176*	*96%*
PFUdA	*25-20,000*	*0.995*	*84*	*101%*	*1-200*	*0.998*	*168*	*99%*
PFDoA	*25-20,000*	*0.998*	*60*	*101%*	*1-200*	*0.994*	*127*	*94%*
PFTrDA	*25-20,000*	*0.998*	*32*	*104%*	*1-200*	*0.995*	*125*	*95%*
PFTeDA	*25-20,000*	*0.994*	*15*	*107%*	*1-200*	*0.998*	*56*	*98%*
PFHxDA	*25-20,000*	*0.999*	*21*	*103%*				
PFODA	*25-20,000*	*0.999*	*33*	*102%*				
PFSAs								
PFBS	*25-20,000*	*0.995*	*31*	*92%*	*2-200*	*0.994*	*1178*	*100%*
PFHxS	*25-20,000*	*0.999*	*604*	*103%*	*1-200*	*0.998*	*229*	*96%*
PFHpS	*25-20,000*	*0.997*	*103*	*105%*	*1-200*	*0.999*	*327*	*99%*
PFOS	*25-20,000*	*0.995*	*312*	*105%*	*1-200*	*0.999*	*251*	*99%*
PFDS	*25-20,000*	*0.998*	*88*	*102%*	*1-200*	*0.999*	*516*	*98%*

An ultra-high sensitivity analysis of PFAS compounds in multiple water sources

Using the SCIEX 7500 System

Bertram Nieland[1], Jack Steed[2], Rens-Jan Jaspers[3], Kay Hup[3], Abdessamad Chahbouni[3], Jianru Stahl-Zeng[4]
[1]SCIEX, The Netherlands; [2]SCIEX, UK; [3]Het Waterlaboratorium, [4]SCIEX, Germany

In this method sub-parts per trillion (ppt) levels of detection for 26 PFAS compounds was achieved with LOD values of 0.2 ng/L in diluent and calculated method detection limits in drinking, ground, and surface water ranging between 0.06 ng/L and 1.12 ng/L.

PFAS compounds are ubiquitous in our environment due to overuse and their lack of breakdown, ensuring that this will be a challenge for decades to come.[1] Therefore, it is imperative to provide rigorous and sensitive analytical testing to regulate these compounds and try to limit their possible effects on human health.

In December 2020, the European Parliament and Council of the European Union released a new directive that sets the limit of PFAS in drinking water to 0.5 µg/L for all PFAS compounds

Key features of PFAS analysis using the SCIEX 7500 System

PFAS/PFOS SCIEX 7500 System

- Sample preparation: **2.5 mL of the water sample + 2 mL of a 50:50 acetonitrile/methanol + 0.225 % formic acid**
- Chromatography: Phenomenex Luna Omega PS C18, 100 Å, 100 x 2.1 mm, 3 µm (PN: 00D-4758-AN),
  Phenomenex Gemini C18, 110 Å, 100 x 2.0 mm, 3 µm (PN: 00D-4439-B0) delay column.
- Mobile phase A was 20 mM ammonium acetate in water and mobile phase B was methanol.
- Mass spectrometry: The analysis was performed using the SCIEX 7500 System, operated in electrospray ionization in negative ion mode.

PFAS/PFOS SCIEX 7500 System

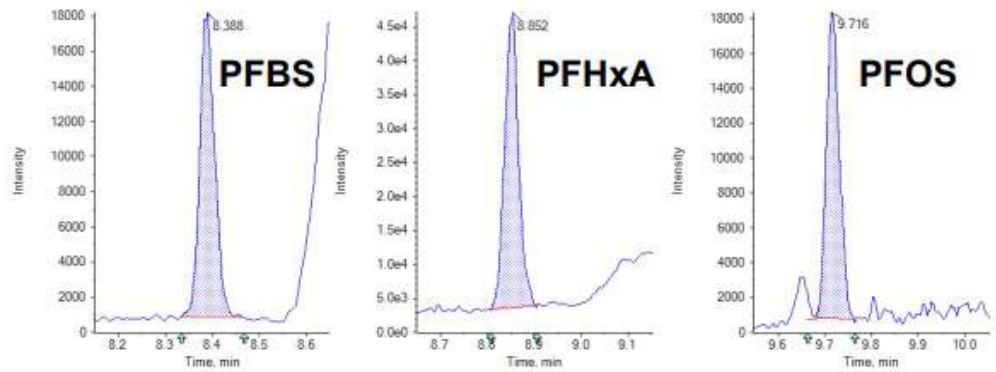


Figure 1. Extracted ion chromatograms (XICs) of three PFAS compounds at their respective LOQs. From left to right: PFBS, PFHxA and PFOS - all at 0.2 ng/L. The image above shows excellent sensitivity has been achieved with this method at low ng/L levels.

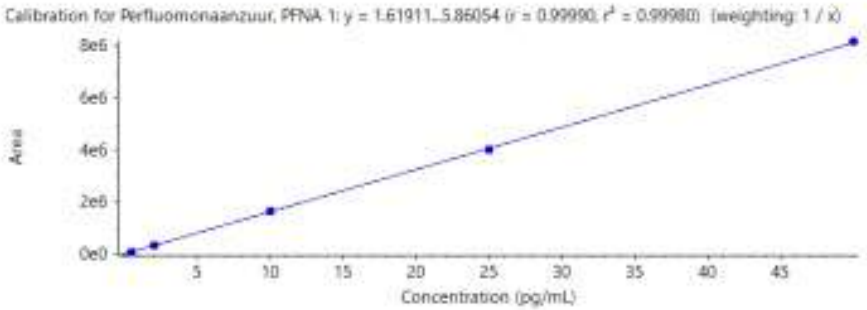


Figure 4. Calibration curve for PFNA. The image above shows the linear range of 0.5 – 50 ng/L for PFNA, highlighting an r value of 0.99990.

Table 1. LOQ of each compound analyzed in diluent with peak-to-peak S/N added to show that each peak is quantifiable, with S/N above 10 x.

Compound name	LOQ (ng/L)	Peak-to-peak S/N
PFHxS	0.20	34.5
PFNA	0.50	25.5
PFOS	0.20	34.5
PFOA	0.50	14.3
PFBA	0.20	11.7
PFPeA	0.20	19.6
PFHxA	0.20	24.7
PFHpA	0.20	23.6
PFDA	0.50	15.1
PFUdA	0.50	14.6
PFDoA	0.50	11.1
PFTrDA	1.00	55.4
HFPO-DA	0.20	31.2

Achieving low parts-per-quadrillion detection limits for PFAS analysis in drinking water

A collaborative exploration of instrument sensitivity and sample preparation for ultra-trace analysis of PFAS on the SCIEX 7500 system

Thep Phomsopha[1], Simon Roberts[2], Craig M. Butt[2], Karl Oetjen[2], Matt Noestheden[2], Sam Lodge[3], Andrew N. Patterson[1]

[1]Eurofins Environment Testing West Sacramento; [2]SCIEX, USA; [3]Phenomenex

This technical note describes the collaboration between SCIEX and Eurofins Environment Testing to address the 2022 US EPA drinking water health advisory levels (HALs) for 4 per- and polyfluoroalkyl substances (PFAS).[1] Sample preparation and instrumental methods were developed to achieve low parts-per-quadrillion (ppq) HALs—which is pg/L equivalent—for perfluorooctanoic acid (PFOA, 4 ppq), perfluorooctane sulfonic acid (PFOS, 20 ppq), perfluorobutane sulfonic acid (PFBS, 2 ppb) and hexafluoropropylene oxide dimer acid (HFPO-DA, or GenX, 10 ppt). The high sensitivity of the SCIEX 7500 system

EPA drinking water HALs demand an unprecedented level of cleanliness and instrumental sensitivity which ultimately necessitates newer, robust analysis techniques.

Key benefits of the method for ultra-trace level PFAS analysis

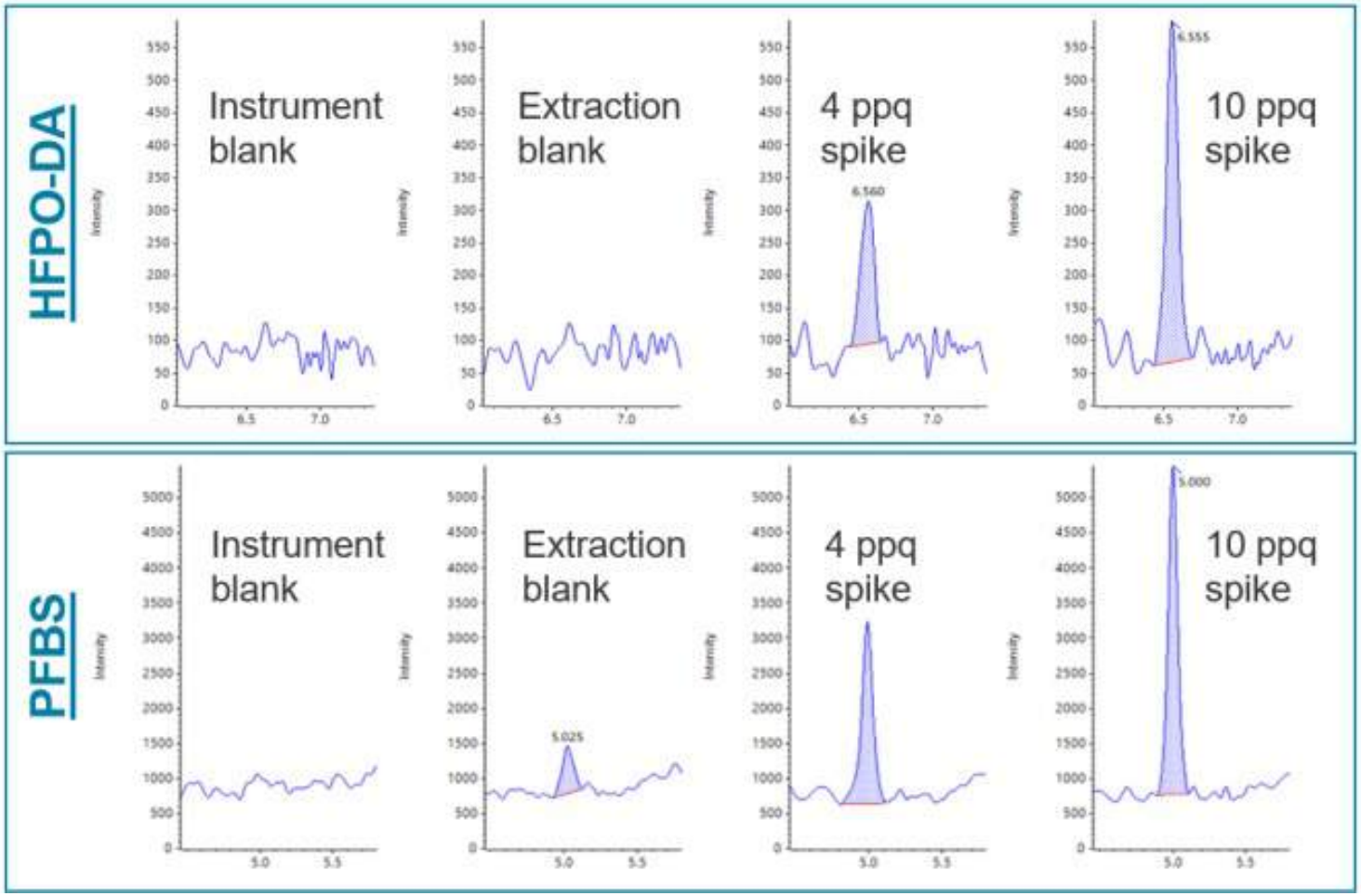
PFAS/PFOS SCIEX 7500 System

- Sample preparation: The sample extraction followed a modified EPA Method 533 procedure
- 250 mL of sample., load on (SPE) cartridges were the Phenonmenex Strata™ X-AW (500 mg).
- The final SPE eluent was adjusted to 10 mL of 80:20 methanol/water (0.1% ammonium hydroxide) and an aliquot was transferred to a polypropylene vial for analysis.

Chromatography: SCIEX ExionLC system

- The analytical column was the Gemini C18 (3 µm, 100 x 3 mm, Phenomenex, P/N: 00D-4439-Y0) with a Zorbax Diol guard cartridge (6 µm, 12.5 x 4.6 mm, P/N: 820950-911).
- The delay column was the Luna Omega PS C18 (5 µm, 50 x 4.6 mm, Phenomenex, P/N: 00B4753-E0).
- The mobile phases were water and methanol (modified with 5mM and 2mM ammonium acetate, respectively) with a flow rate of 0.7 mL/min. The column oven was 45 oC,
- The injection volume was 100 µL.

PFAS/PFOS SCIEX 7500 System



PFAS/PFOS SCIEX 7500 System

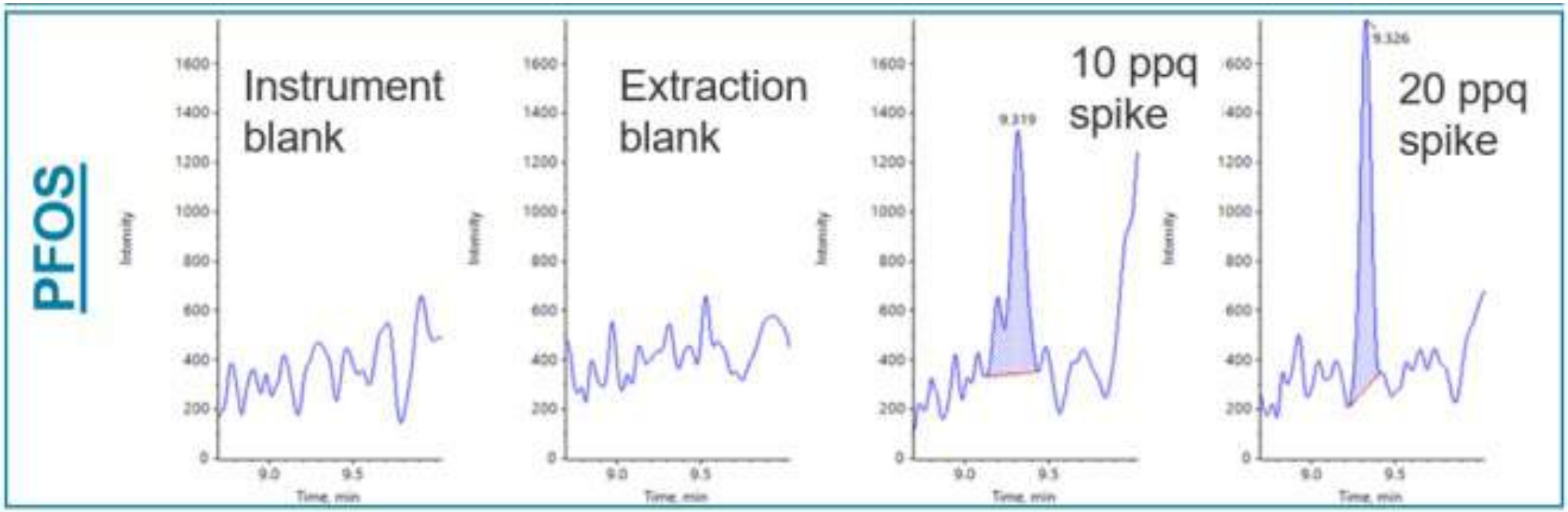


Figure 2. XIC chromatograms for HFPO-DA (m/z 285.0>169.0), PFBS (m/z 298.9>80.0) and PFOS (m/z 499>99.0) quantifier MRM transitions from method extraction samples. Instrument blank, extraction blank, and two spike levels are shown.

FDA U.S. FOOD & DRUG ADMINISTRATION

FDA Foods Program Compendium of Analytical Laboratory Methods: Chemical Analytical Manual (CAM)

METHOD NUMBER: C-010.03

POSTING DATE: 04/12/2024

POSTING EXPIRATION DATE: 04/12/2026

PROGRAM AREA: Chemical Contaminants

METHOD TITLE: Determination of 30 Per and Polyfluoroalkyl Substances (PFAS) in Food and Feed using Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS)

VALIDATION STATUS: Single Laboratory Validation (L2) per the Guidelines for the Validation of Chemical Methods for the FDA Foods Program 3rd Edition

2021.9 CALCULATIONS

Example calculation for concentration measured on LC-MS/MS to concentration in 5 grams of food with a final extract of 10 mL:

- The lowest calibration curve point is 0.01 ng/mL in 0.5 mL of solution.

$$\frac{0.01\ ng}{mL} * 0.5\ mL = 0.005\ ng$$

- Since there are 0.005 ng in 0.5 mL of extract, there would be 0.1 ng in the total 10 mL extract

$$0.005\ ng * \frac{10\ mL}{0.5\ mL} = 0.1\ ng$$

- With a 5 gram food sample, this is equivalent to 20 ng/kg in foods

$$\frac{0.1\ ng}{5\ g} = 0.02\frac{ng}{g}\ or\ 20\frac{ng}{kg}$$

Sciex OS software is used to prepare a linear standard curve where x is the concentration ratio (analyte/SS) and y is the instrument response ratio (analyte/SS) with 1/x weighting. Surrogates and their internal standard pairs are listed in Table 5, which are used to calculate absolute recoveries of the surrogate standards over the entire extraction method. Surrogates and their native analyte pairs are also listed in Table 5 with their curve fit. The calibration curve has surrogate and internal standard concentrations of 1 ng/mL.

Chuẩn thấp nhất **10ppt**

FDA Thẩm định Phương pháp Phân tích PFAS trên Sciex 6500+ QTRAP

2024.8.4 LC-MS/MS Analysis

All samples were analyzed using a liquid chromatograph (Nexera X2, (Shimadzu, Kyoto, Japan)). The MS/MS data was acquired using scheduled MRM with a Sciex QTRAP® 6500+.

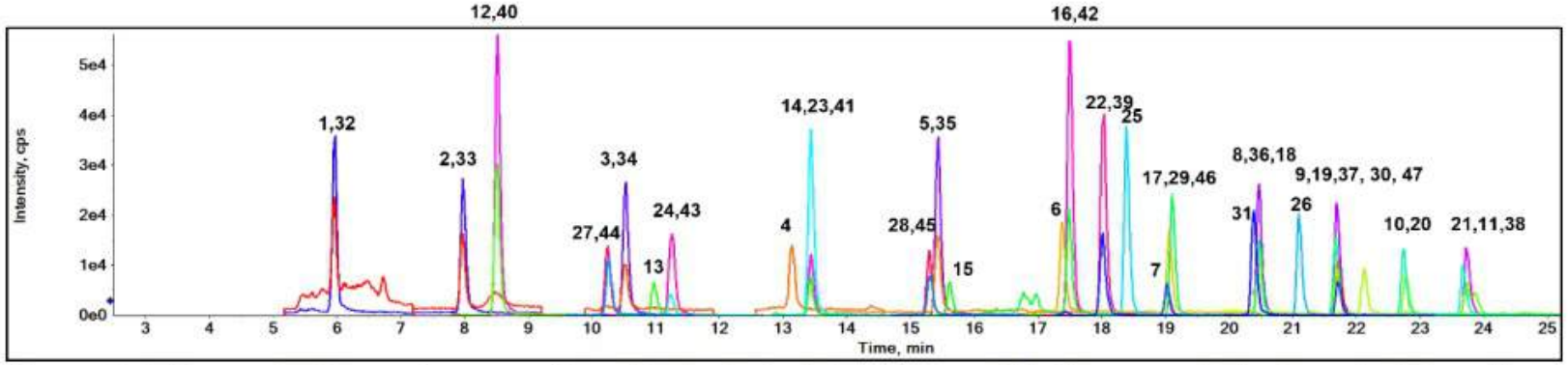


Figure 8-1. Example chromatogram of a spiked single-laboratory validation sample (salmon) with native and labeled PFAS concentrations at 5 µg/kg.

1. PFBA, 2. PFPeA, 3. PFHxA, 4. PFHpA, 5. PFOA, 6. PFNA, 7. PFDA, 8. PFUnA, 9. PFDoA, 10. PFTrDA, 11. PFTeDA, 12. PFBS, 13. PFPeS, 14. PFHxS, 15. PFHpS, 16. PFOS, 17. PFNS, 18. PFDS, 19. PFUnDS, 20. PFDoS, 21. PFTrDS, 22. FOSA, 23. DONA, 24. HFPO-DA, 25. 9Cl-PF3ONS, 26. 11Cl-PF3OUdS, 27. 4:2 FTS, 28. 6:2 FTS, 29. 8:2 FTS, 30. 10:2 FTS 31. d5 N-EtFOSAA, 32. M3 PFBA, 33. M3 PFPeA 34. M5PFHxA, 35. M8PFOA, 36. MPFUdA, 37. MPFDoA, 38. M2PFTeDA, 39. M8 FOSA 40. M3 PFBS, 41. M3PFHxS, 42. 13C PFOS, 43. M3 HFPO-DA 44. Labeled 4:2 FTS, 45. Labeled 6:2 FTS, 46. Labeled 8:2 FTS, 47. Labeled 10:2 FTS

2024.8.4 LC-MS/MS Analysis

All samples were analyzed using a liquid chromatograph (Nexera X2, (Shimadzu, Kyoto, Japan)). The MS/MS data was acquired using scheduled MRM with a Sciex QTRAP® 6500+.

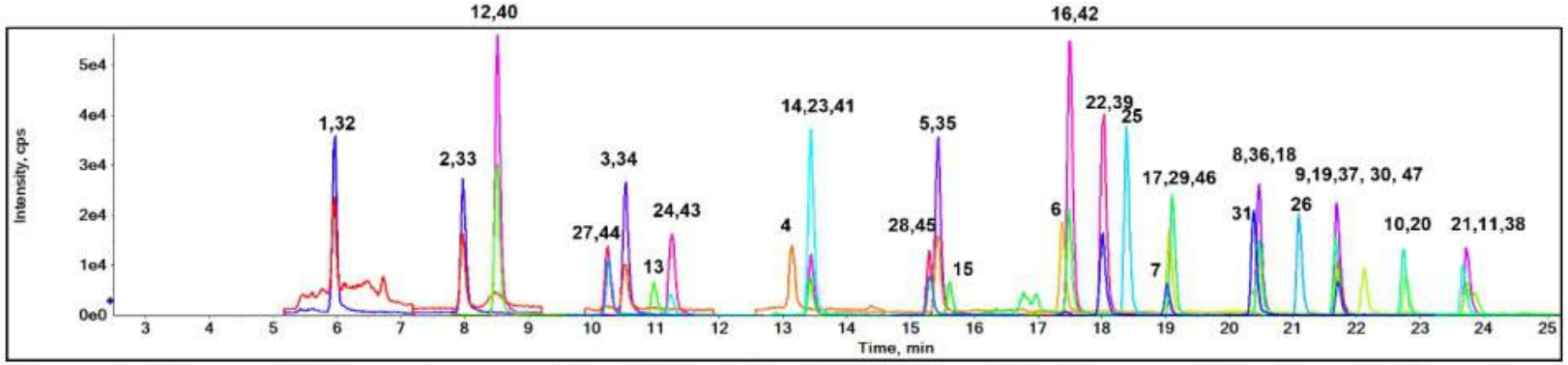


Figure 8-1. Example chromatogram of a spiked single-laboratory validation sample (salmon) with native and labeled PFAS concentrations at 5 µg/kg.

1. PFBA, 2. PFPeA, 3. PFHxA, 4. PFHpA, 5. PFOA, 6. PFNA, 7. PFDA, 8. PFUnA, 9. PFDoA, 10. PFTrDA, 11. PFTeDA, 12. PFBS, 13. PFPeS, 14. PFHxS, 15. PFHpS, 16. PFOS, 17. PFNS, 18. PFDS, 19. PFUnDS, 20. PFDoS, 21. PFTrDS, 22. FOSA, 23. DONA, 24. HFPO-DA, 25. 9Cl-PF3ONS, 26. 11Cl-PF3OUdS, 27. 4:2 FTS, 28. 6:2 FTS, 29. 8:2 FTS, 30. 10:2 FTS 31. d5 N-EtFOSAA, 32. M3 PFBA, 33. M3 PFPeA 34. M5PFHxA, 35. M8PFOA, 36. MPFUdA, 37. MPFDoA, 38. M2PFTeDA, 39. M8 FOSA 40. M3 PFBS, 41. M3PFHxS, 42. 13C PFOS, 43. M3 HFPO-DA 44. Labeled 4:2 FTS, 45. Labeled 6:2 FTS, 46. Labeled 8:2 FTS, 47. Labeled 10:2 FTS

2024.12.3 Method Detection Limits

Method detection limits were calculated by performing a minimum of 7 low-level spikes at 0.05 ng/g for all food and feed matrices. The standard deviation of the replicates was multiplied by 3.14 (t-value for seven replicates where 1-α =0.99). The MDL is defined as the statistically calculated minimum concentration that can be measured with 99% confidence that the measured concentration is distinguishable from method blank results. This procedure is published in the Code of Federal Regulations.[3] MDLs are recalculated yearly and are dependent on instrumental conditions at the time of the study. The limit of quantification (LOQ) can be calculated by multiplying the standard deviation of the replicates by 10. Both the MDLs and LOQs can be found in Appendix 1D.

Pesticides

PARAMETER *CHỈ TIÊU*	METHOD *PHƯƠNG PHÁP*
Agricultural chemical residues list of 1110 parameter	LC/MS/MS: Ref. EN 15662:2018 GC/MS/MS:Ref. AOAC 2007.01 (21st Ed., 2019 and EN 15662:2018)

LC-MS: 480 chỉ tiêu

GC-MS: 626 chỉ tiêu

HS- GC/MS: 4 chỉ tiêu

Selected product: Rice

Main product of the group or subgroup | Other products to which the same MRLs apply

Code Number	Name	Scientific Name

517 chỉ tiêu

Maximum residue level Min 0.001 (mg/kg) (2-amino-4-methoxy-6-(trifluormethyl)-1,3,5-triazine (AMTT))

https://ec.europa.eu/food/plant/pesticides/eu-pesticides-database/start/screen/mrls/latest

6/30/25

European Commission

EURL-SRM

EU Reference Laboratories for Residues of Pesticides

Single Residue Methods

Quick Method for the Analysis of Highly Polar Pesticides in Food Involving Extraction with Acidified Methanol and LC- or IC-MS/MS Measurement

I. Food of Plant Origin (QuPPe-PO-Method)

Version 12.2 (21.12.2023, Document History, see page **104**)

Check for latest version of this Method under https://www.quppe.eu; older versions: obsolete versions

Authors: M. Anastassiades; A.-K. Schäfer; D. I. Kolberg; E. Eichhorn; H. Dias; A. Benkenstein; S. Zechmann; D. Mack; C. Wildgrube; A. Barth; I. Sigalov; S. Goerlich; D. Dörk; G. Cerchia

EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL-SRM)

Contact: CVUA Stuttgart, Schaflandstr. 3/2, DE-70736 Fellbach, Germany, www.eurl-pesticides.eu; EURL@cvuas.bwl.de

Note: Changes from V12.1 to V12.2 are highlighted in green

http://www.eurl-pesticides.eu/

Pesticide phân cực

European Commission

EURL-SRM

EU Reference Laboratories for Residues of Pesticides
Single Residue Methods

Quick Method for the Analysis of Numerous Highly Polar Pesticides in Food Involving Extraction with Acidified Methanol and LC-MS/MS Measurement

II. Food of Animal Origin (QuPPe-AO-Method)

Version 3.2 (14.05.2019, Document History, see page 24)

Please check for the **latest version of this document** under www.quppe.eu

Changes from V2 to V3 are highlighted in yellow; from V3 to V3.1 in green and from V3.1 to 3.2 in grey

Authors: M. Anastassiades; A.-K. Wachtler; D. I. Kolberg; E. Eichhorn; A. Benkenstein; S. Zechmann; D. Mack; A. Barth; C. Wildgrube; I. Sigalov; S. Görlich; D. Dörk; G. Cerchia

EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL-SRM)
Address: CVUA Stuttgart, Schaflandstr. 3/2, DE-70736 Fellbach, Germany
Web: www.eurl-pesticides.eu,
E-Mail: EURL@cvuas.bwl.de

Pesticide phân cực – Không chiết được bằng QuEChRS

Pesticide phân cực

EURL-SRM — EU Reference Laboratories for Residues of Pesticides — Single Residue Methods

✓ = satisfactory chomatography and detection sensitivity achieved,
NT = Not tested under the conditions shown in the respective sections,
(✓) = possible but compromised due to matrix effects or lacking separation or limited sensitivity or limitations in the detection of qualifiers compromising identification.
"-" analysis was tested and found to be poor under the described conditions,
* Using a gradient (98% B -> 60% B in 5 min, hold 2 min)
** Different LC-conditions required to improve peaks (see M1.7 or M1.9)
*** Compromised quantitation of Phosphonic acid due to co-elution of Phosphonic acid and Fosetyl, see also General Hints 5.6.1
**** Quality of analysis may strongly depend on instrument type and condition

Table 6: Scope of QuPPe-LC-Methods of analytes analyzed in the ESI-neg. mode Part.I

QuPPe method code	M 1.1 (5.6.2)	M 1.2 (5.6.3)	M 1.3 (5.6.4)	M 1.4 (5.6.5)	M 1.5 (5.6.6)	M 1.6a/b (5.6.7)	M 1.7 a/b (5.6.8)	M 1.8 (5.6.9)	M 1.9 (5.6.10)	M 1.10 (5.6.11)	M 1.11 (5.6.12)	M 1.12 (5.6.13)
Separation principle	Anion Ex.	Anion Ex.	Carbon	Carbon	HILIC	HILIC	HILIC	HILIC	HILIC	HILIC	HILIC	HILIC
Column type	AS 11	AS 11-HC	Hypercarb	Hypercarb	Trinity Q1	a: Torus DEA/ b: APPC	a: Torus DEA/ b: APPC	APPC	Raptor PolarX	ObeliscN	Luna Polar Pesticides	Luna Polar Pesticides
ANALYTES COVERED BY LC-MS/MS IN THE ESI-NEGATIVE MODE												
Ethephon	✓	✓	✓	NT	✓	✓	(✓)	NT	✓	✓	✓	-
HEPA	✓	✓	✓	NT	✓	✓	(✓)	NT	✓	✓	✓	-
Glufosinate	✓	✓	✓	NT	✓	✓	(✓)	NT	✓	✓	✓	-
N-Acetyl-Glufosinate	✓	✓	✓	NT	✓	✓	(✓)	NT	✓	✓	✓	-
MPPA	✓	✓	✓	NT	✓	✓	(✓)	NT	✓	✓	✓	-
Glyphosate	✓	✓	✓	NT	✓	✓	(✓)	NT	✓	✓	✓	-
AMPA	✓	✓	✓	NT	✓	✓	(✓)	NT	✓	(✓)	(✓)	-
Phosphonic acid	(✓)	(✓)	✓	✓	✓	✓	✓	NT	✓	(✓)***	(✓)	✓
N-Acetyl-AMPA	NT	✓	✓	NT	✓	✓	(✓)	NT	NT	NT	NT	NT
Fosetyl-Al	-	✓	✓	NT	✓	✓	(✓)	NT	✓	(✓)***	✓	-
Maleic Hydrazide	-	-	✓	NT	-	-	-	(✓)	(✓)	(✓)	-	-
Perchlorate	NT	-	✓	✓	✓	(✓)**	✓	✓	✓	✓	-	✓
Chlorate	NT	-	✓	✓	✓	(✓)**	✓	✓	(✓)	✓	-	(✓)
Bialaphos	NT	NT	✓	NT	✓	NT	NT	NT	✓	NT	NT	NT
Cyanuric acid	NT	NT	✓	NT	-	-	-	✓	(✓)	(✓)	-	-
Bromide	NT	NT	-	✓	✓	(✓)**	✓	NT	✓	✓	(✓)	✓
Bromate	NT	NT	(✓)	✓	NT	NT	✓	NT	NT	NT	NT	NT
N-Acetyl-Glyphosate	NT	NT	✓	NT	(✓)**	✓	(✓)	NT	-	✓	✓	-
Difluoroacetic acid	NT	NT	NT	NT	NT	✓	✓	NT	✓	NT	NT	NT
Trifluoroacetic acid	NT	NT	NT	NT	NT	✓	✓	NT	✓	✓	-	(✓)
Thiocyanate	NT	NT	(✓)**	✓	NT	NT	✓	NT	✓	NT	-	(✓)
Desmethyl-Dimethoate	NT	NT	✓	-	NT	NT	NT	NT	NT	NT	NT	NT
Diethyl thiophosphate	NT	NT	NT	NT	NT	NT	✓	NT	NT	NT	NT	NT
Diethyl phosphate	NT	NT	NT	NT	NT	NT	✓	NT	NT	NT	NT	NT
O,O-dimethyl dithiophosphate	NT	NT	NT	NT	NT	NT	✓	NT	NT	NT	NT	NT
O,O-diemthyl thiophosphate	NT	NT	NT	NT	NT	NT	✓	NT	NT	NT	NT	NT
Diethyl dithiophosphate	NT	NT	NT	NT	NT	NT	✓	NT	NT	NT	NT	NT
Dimethyl phosphate	NT	NT	NT	NT	NT	NT	✓	NT	NT	NT	NT	NT

Pesticide phân cực

EURL-SRM — EU Reference Laboratories for Residues of Pesticides — Single Residue Methods

Table 8 : Methods mainly used by CVUA Stuttgart

Method	Special remarks on Substances	LC-MS/MS	Comments
M 1.3: Glyphosate & Co. Hyper-carb (see 5.6.4)	Glyphosate AMPA N-Acetyl-AMPA N-Acetyl-Glyphosate Ethephon HEPA Glufosinate N-Acetyl-Glufosinate MPPA Fosetyl-Al Phosphonic acid (screening) Maleic Hydrazide Perchlorate (screening)) Chlorate (screening) Cyanuric acid Bialaphos Desmethyl-Dimethoate	1290 Agilent Infinity II and Sciex QTRAP 6500+	Evaluation via solvent calibration and IL-ISs except for Bialaphos and N-Acetyl-AMPA (IL-IS not yet available) M 1.5 and M 1.6 are currently being tested for their suitability to replace M 1.3
M 1.4: Method 1.4 (M1.4): "Per-ChloPhos" (see 5.6.5)	Perchlorate (quantitative) Chlorate (quantitative) Phosphonic acid (quantitative) Bromide (Screening, quantitative) Bromate (quantitative) Thiocyanate	1290 Agilent Infinity II and Sciex QTRAP 6500+	Mostly employed directly (option: screening by M 1.3, if postive -> M 1.4) Dilution 5-fold Evaluation via solvent calibration and IL-ISs

Pesticide phân cực

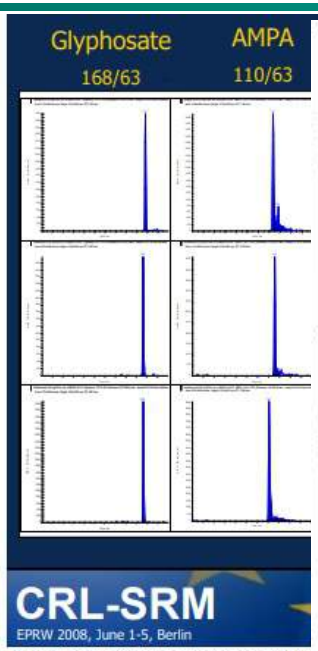


Figure 7: Typical chromatograms of Gl

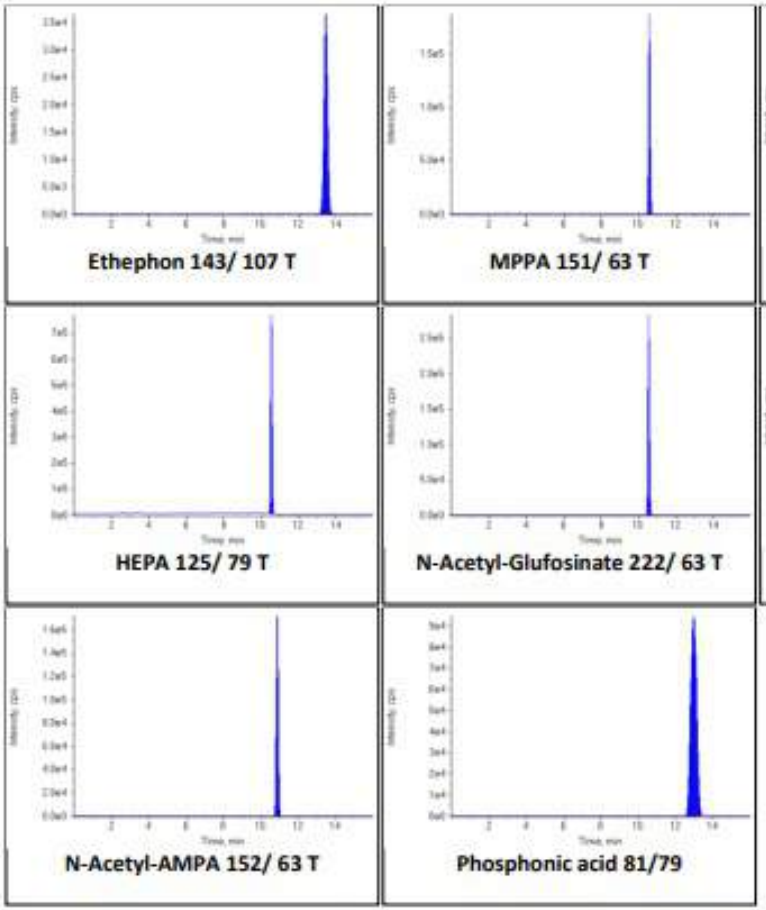


Figure 9: *Typical chromatograms of Ethephon, HEPA, Glyphosat, AMPA, Glufosinate, MPPA, N-Acetyl-AMPA, N-Acetyl-Glufosinate, Fosetyl-Al and Phosphonic acid at 0.1 mg/L in methanol with 1% formic acid.*

Pesticides

Biggest Problem with LC- and GC- MS(/MS)

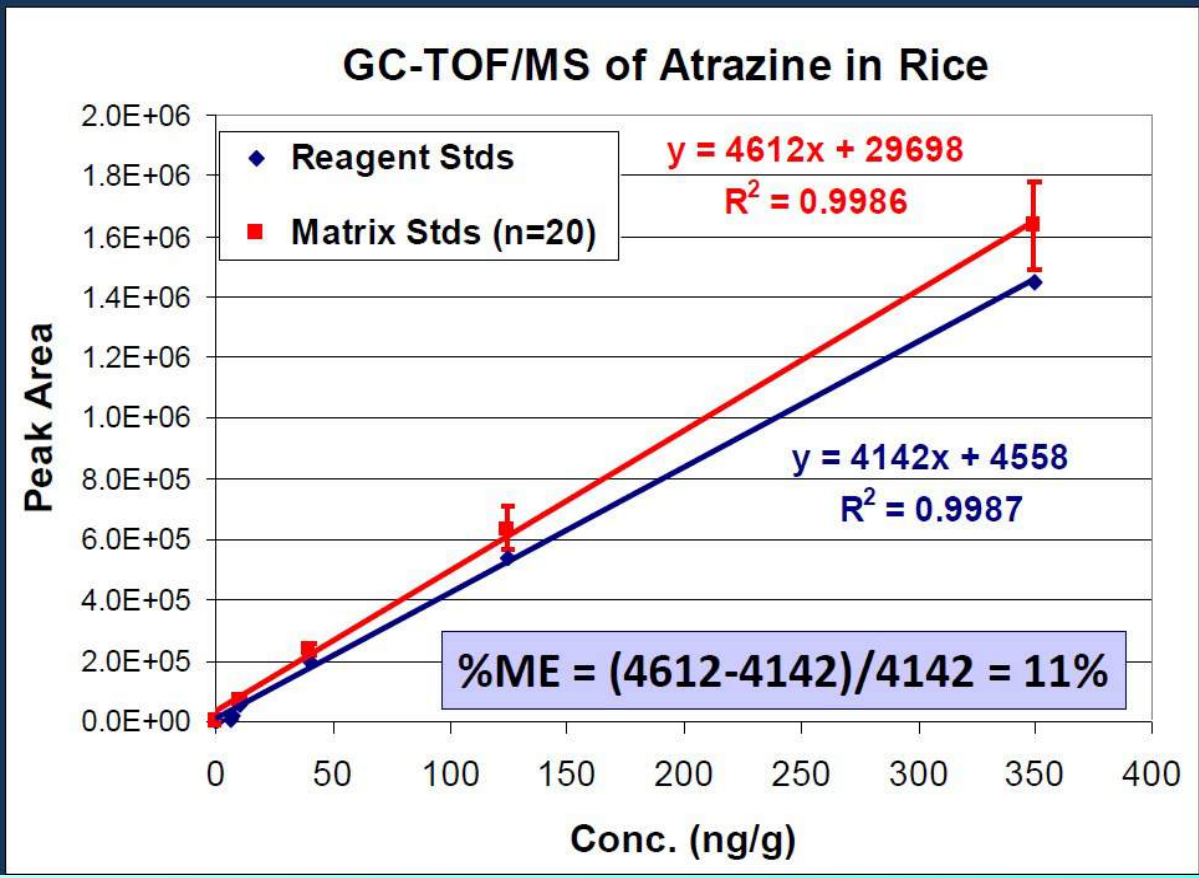
Matrix Effects

plus costs to purchase and maintain,
and facility requirements, and downtime,
and need for more expertise due to
greater complexity

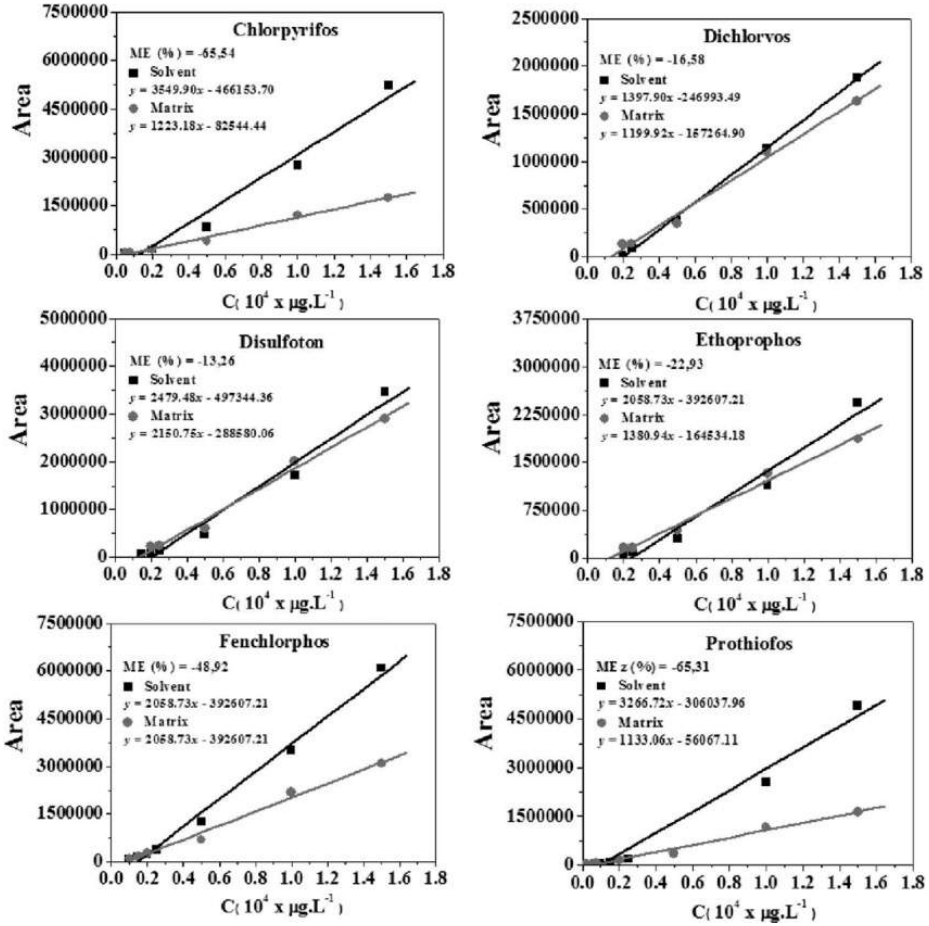
It is still a pain!

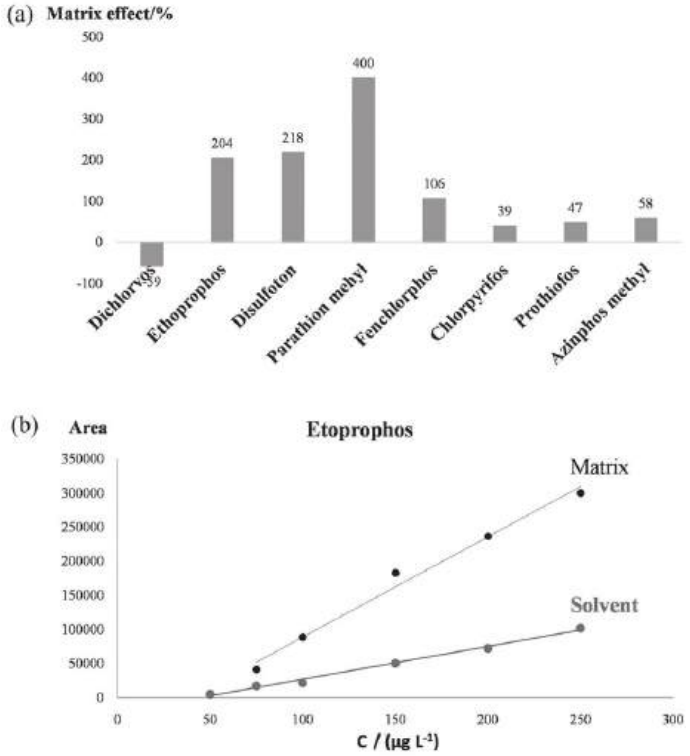
Pesticides

How to Calculate (Estimate) Matrix Effects



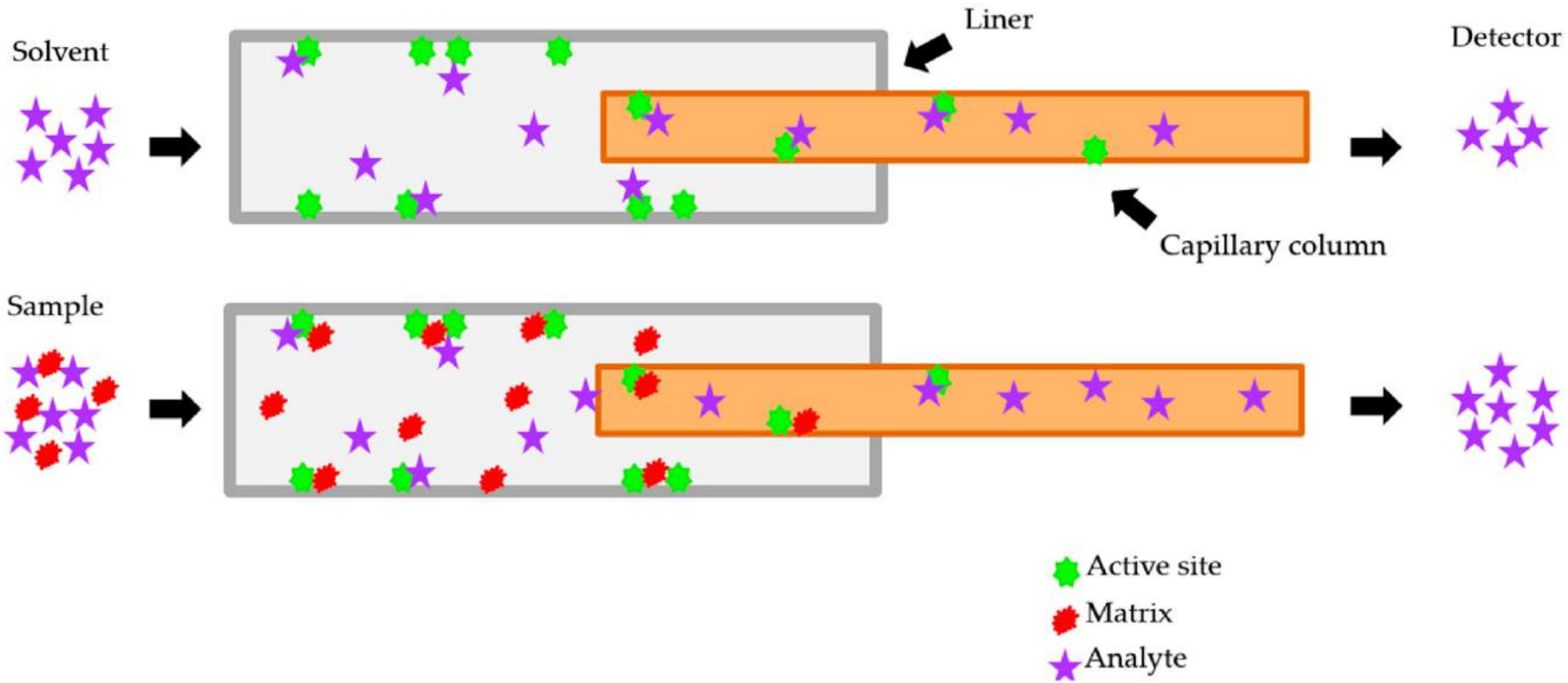
Pesticides





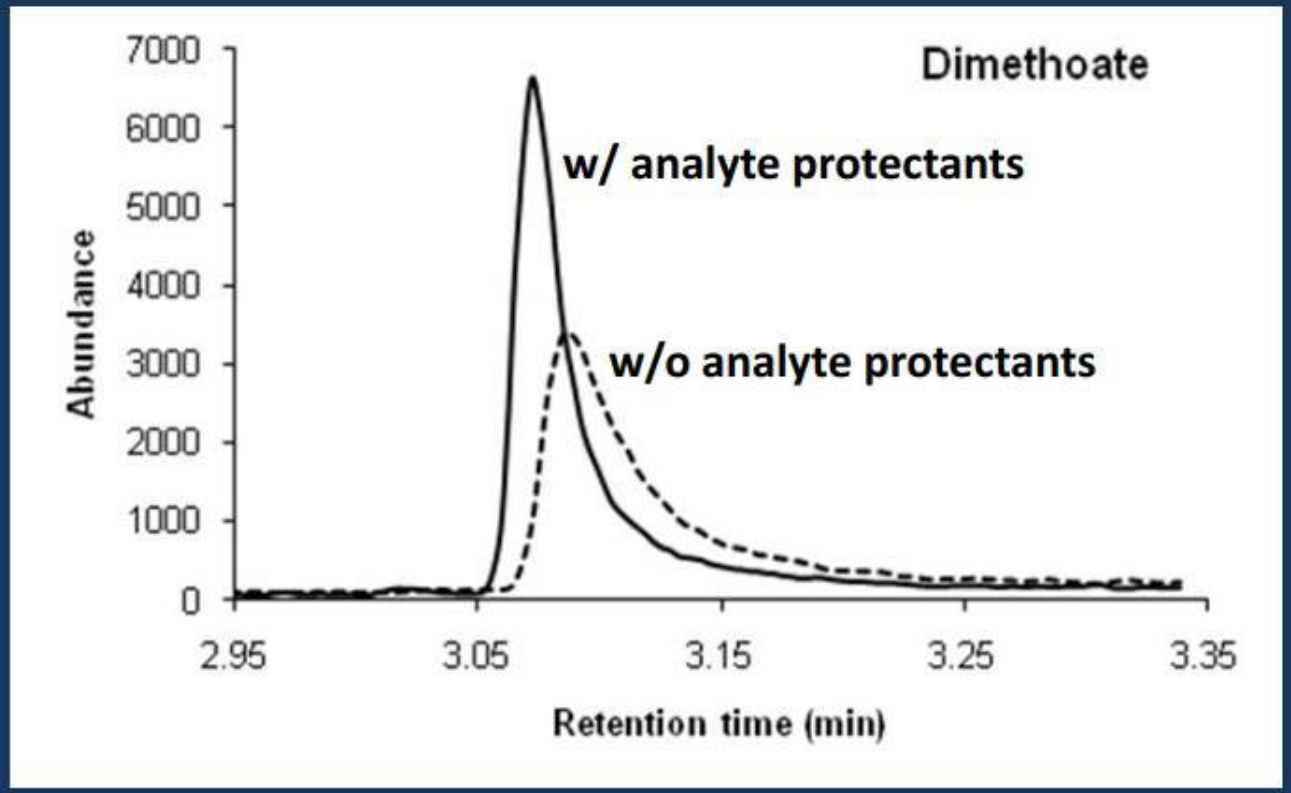
https://www.researchgate.net/figure/Matrix-effect-for-the-six-pesticides-studied-in-the-bell-pepper-matrix_fig2_335403723

Pesticides


Solvent
Liner
Detector
Capillary column
Sample
Active site
Matrix
Analyte

Pesticides

Effect of Analyte Protectants

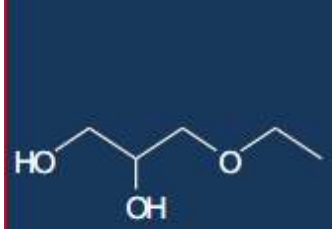


Pesticides

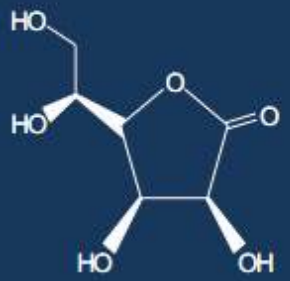
Analyte Protectants

Strongly interact with active sites in GC system (inlet, column and ion source) to decrease degradation and adsorption of co-injected analytes.

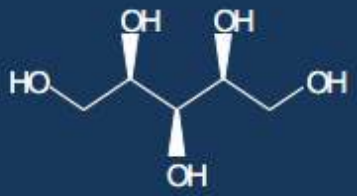
Sharper peaks, less tailing, more ruggedness, lower LOD



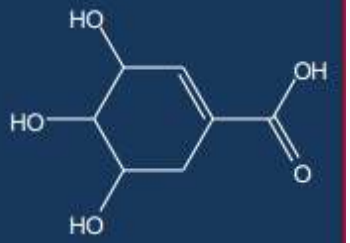
ethylglycerol
1 mg/mL



gulonolactone
0.1 mg/mL

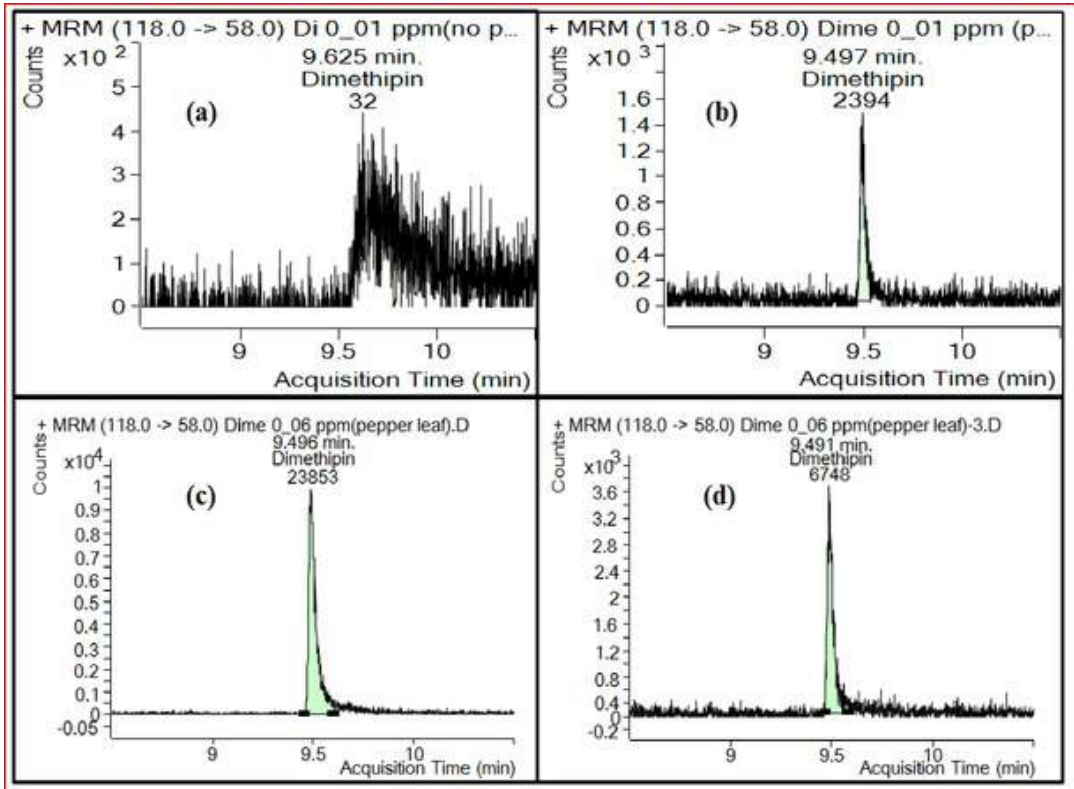


sorbitol
0.1 mg/mL



shikimic acid
0.05 mg/mL

Pesticides



Expand

Fig 2.

Optimization of analyte protectant (AP) for GC–MS/MS signal enhancement of dimethipin after priming with pepper leaf matrix (a) no priming (b) 10% AP after priming (c) 20% AP after priming (d) 30% AP after priming.

SCION 8900 TQMS APPLICATION NOTE

SCION INSTRUMENTS

Multiplexed pesticide analysis by GC-TQMS

ANALYSIS OF 450 PESTICIDES IN BLUEBERRIES

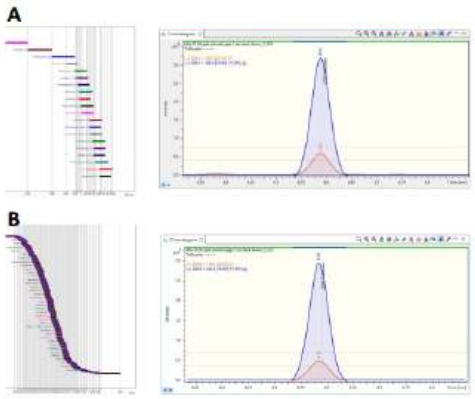
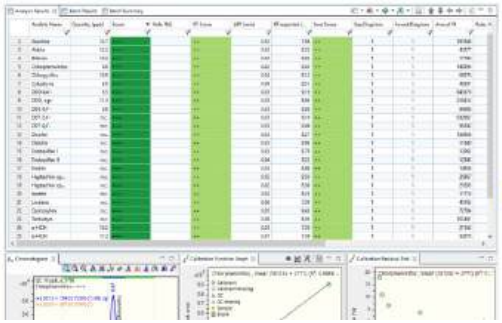


Figure 3. Increasing MRM does not impede sensitivity

Despite the increase in number of components measured, (Figure 3), the SCION 8900 TQMS maintains good accuracy and linearity at scan times of 4ms. The ion ratio stability is maintained when switched from 40ms to 7ms scan time. This is maintained even at the lowest concentration of calibration standards (down to 1ppb). Reproducibility is maintained over multiple runs.

minute and held for 14.3 minutes. The Backflush option was activated with a pressure of 5PSI and a start time of 14 minutes.



RESULTS

The data showed excellent linearity for standards and QC samples (Figure 5) with perfect ion scores observed throughout the batch. Retention time reproducibility was extremely consistent and good recovery and reproducibility. This reproducibility was consistent through all of the detected analytes. For example, chlorphenvinfos, an organophosphorous compound that is widely used as an insecticide, was detected at a concentration of 8ppb. The retention time of the compound was 8.45 minutes, which is within 0.02 minutes of the expected RT. The calibration curve had a R^2 of >0.998, with a residual of less than ± 20%.

Direct aqueous analysis of pesticides and PPCPs in drinking and bottled water at parts per trillion levels

Samples

- Pesticides and PPCP in water
- Compounds: positive mode 370, negative mode 61 (46 internal stds)
- The calibration standard concentrations were 0.1, 0.2, 0.5, 1, 2, 5, 10,
20, 50, 100, 200 and 500 ng/L. A calibration blank was also prepared.
- MilliQ water, tap water (drinking water), Evian, FIJI

Methods

LC system: ExionLC™ System

- Column: Luna Omega C18 1.6 µm, 100×2.1 mm
- Mobile phase:

– A : Water + 0.1% formic acid + 5mM NH4 formate

– B : MeOH + 0.1 % formic acid + 5mM NH4 formate

- Temperature: 40 ºC
- Injection volume: 500 µL
- MS: fast pos/neg switching with Scheduled MRM™ Algorithm

Total Time (min)	Flow Rate (µL/min)	A (%)	B(%)
0.00	500	99	1
0.50	500	99	1
16.00	500	1	99
21.00	500	1	99
21.10	500	99	1
25.00	500	99	1

SCIEX

Hiệu năng phân tích cực cao

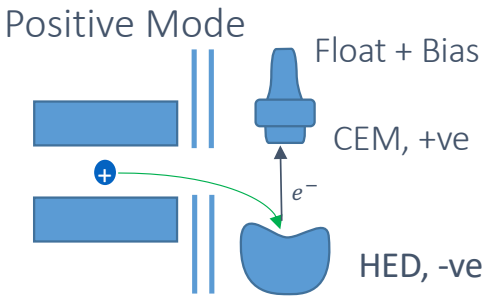
SCIEX QTRAP®® 6500+

Product	Glufosinate sum			Fosetyl sum			Glyphosate			Chlorate			Ethephon		
	LOD	MRL	%RSD at MRL	LOD	MRL	%RSD at MRL	LOD	MRL	%RSD at MRL	LOD	MRL	%RSD at MRL	LOD	MRL	%RSD at MRL
Fruit and Vegetables	16	30	11%	25	2000	13%	5	100	15%	8	10	15%	18	50	11%
Seeds	12	30	12%	90	2000	15%	8	100	15%	3	10	10%	6	50	14%
Vegetable oil, Fat and Fatty Acids	15	30	19%	40	2000	12%	7	100	22%	2	10	6%	3	50	7%
Grain	18	30	12%	71	2000	14%	8	100	7%	7	10	14%	9	50	6%
Herbs and spices	25	100	8%	87	2000	13%	23	100	6%	8	10	15%	8	100	16%
Meat and Seafood	19	30	15%	23	100	12%	9	50	23%	4	10	8%	4	50	10%
Animal Oil, Fat and Fatty Acids	14	30	20%	51	100	11%	9	50	25%	10	10	16%	7	50	12%
Eggs and Eggs products	18	30	12%	33	100	11%	4	50	13%	12	10	9%	6	50	17%
Milk and Milk products	17	30	9%	20	100	6%	8	50	22%	5	10	12%	5	50	13%
Fatty acids	21	100	14%	70	1000	14%	3	100	18%	4	10	9%	3	100	10%

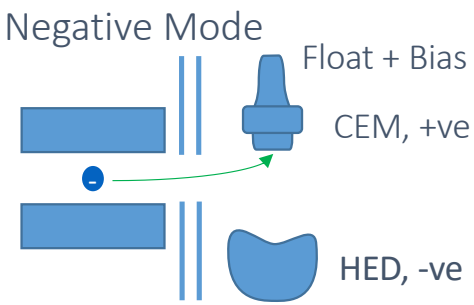
THIẾT KẾ
Đột phá tốc độ chuyển cực của đầu dò

- For Fast Polarity Switching IonDrive High Energy Dynode detector – 6500+

- **IonDrive HED detector**
- Positive Ions strike the HED first, Negative ions strike the magnum directly
- Magnum to be kept at one polarity

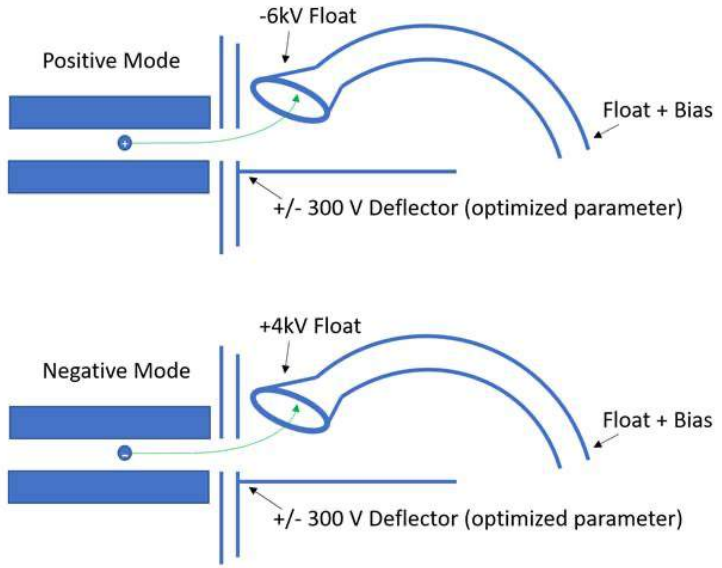


Positive ions first converted to an electron, then electrons are detected by electron multiplier



Negative ions directly detected by electron multiplier

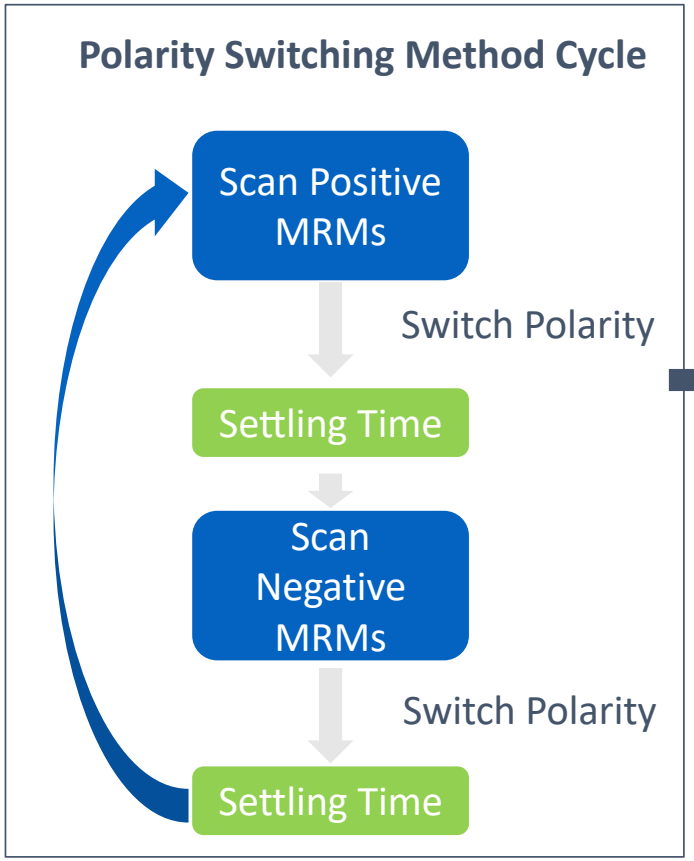
- **CEM detector**
- Both positive and negative ions strike to detector
- Detector voltage must be switched from positive to negative through ground for each cycle

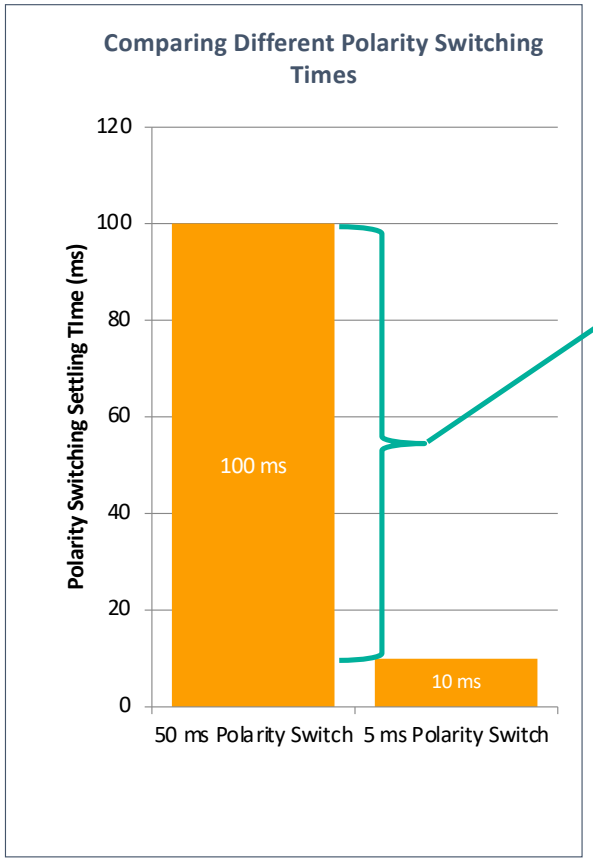


1/THIẾT KẾ

Polarity switching time decreased from 50 to 5 ms

TAKE ADVANTAGE BY SCANNING MORE COMPOUNDS





Gain of 90 msec can be used to add more compounds to each scan, or dwell longer on existing MRMs to improve ion statistics

Pesticides

PARAMETER *CHỈ TIÊU*	METHOD *PHƯƠNG PHÁP*
Agricultural chemical residues list of 1110 parameter	LC/MS/MS: Ref. EN 15662:2018 GC/MS/MS:Ref. AOAC 2007.01 (21st Ed., 2019 and EN 15662:2018)

LC-MS: 480 chỉ tiêu

GC-MS: 626 chỉ tiêu

HS- GC/MS: 4 chỉ tiêu

Triple Quad/
QTRAP 6500+

Thông số LC và MS cho 806 pesticide:
(+) 717 chỉ tiêu pos mode
(-) 89 chỉ tiêu neg mode

LC-MS: 323/ 480 chỉ tiêu

Chuyển đổi được 282 chỉ tiêu GCMS thành LC-MS

https://ec.europa.eu/food/plant/pesticides/eu-pesticides-database/start/screen/mrls/latest

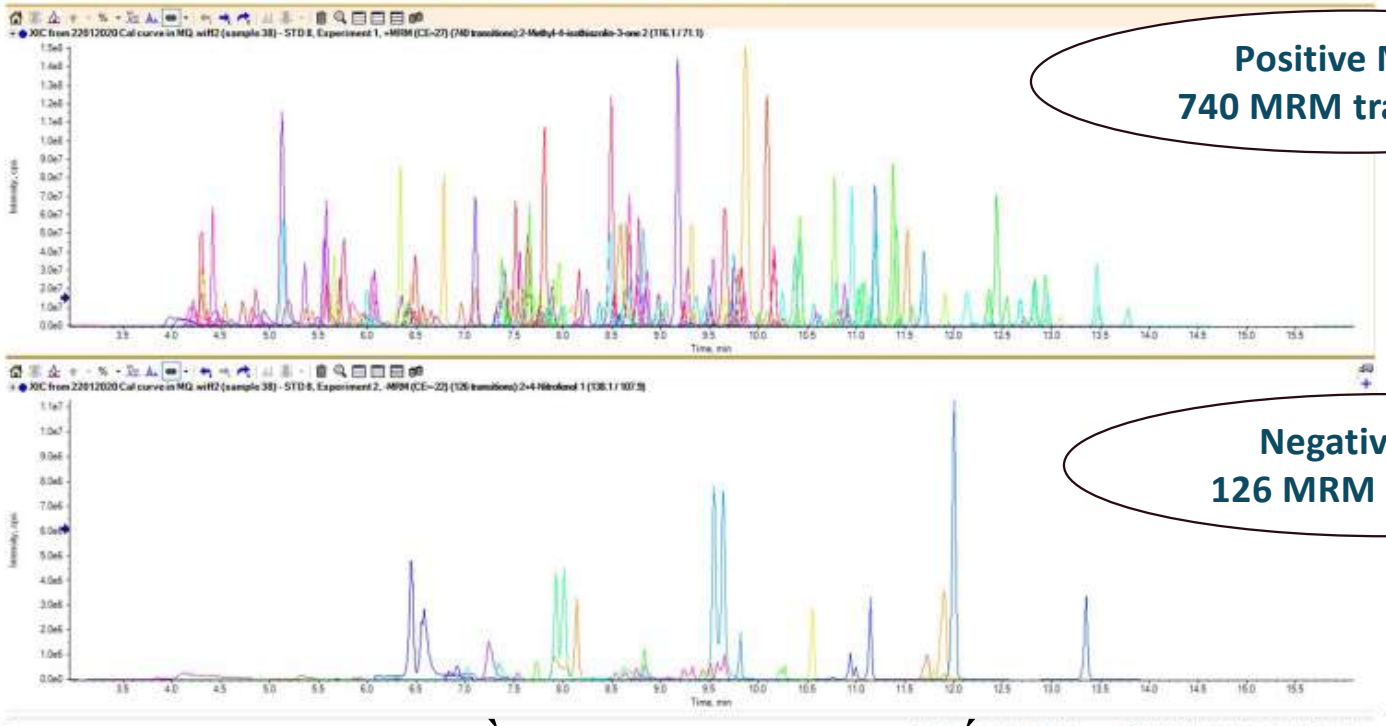
6/30/25

Hiệu năng phân tích cực cao

XIC of positive and negative ionization

SCIEX 7500 System

ALL QUANTIFIER IONS IN MILLI-Q WATER



Positive Mode
740 MRM transitions

Negative Mode
126 MRM transitions

- 4000 MRMs trong một lần phân tích trên thiết bị SCIEX TripleQuad™ và QTRAP®

Hiệu năng phân tích cực cao

For Research Use Only. Not for Use In Diagnostic Procedures.

Food and Environmental

Direct analysis of polar pesticides in water: a routine accredited method

Using the SCIEX QTRAP® 6500+ LC-MS/MS System

Y. Fillâtre[1], J. Prades[2], L. Ley[2], D. McMillan[1]
[1]SCIEX, France, [2]LDAR24, France

"The Glyphosate Paradox" is that, although glyphosate is the most widely used agrochemical in the world, it is also one of the least often determined by analytical methods.[1] Monitoring a highly polar, small organic pesticide such as glyphosate in food and water from diverse sources presents a significant challenge. Polar pesticides are not amenable to standard extraction procedures, are frequently poorly ionized, and demonstrate poor chromatographic separation. These pesticides, therefore, have historically required complex, single-residue methods to make them amenable to analysis—usually involving time-consuming derivatization steps and considerable clean-up procedures.

In 2018, Nofal ab, in collaboration with SCIEX, developed a

and other polar pesticides in water samples. In France, to become accredited by the COFRAC (Comité Français d'Accréditation), the laboratory must demonstrate their analytical method is fit for a routine use by achieving required

Hiệu năng phân tích cực cao

Key features

- Accredited method in drinking water with
  - ✓ LOQ = 20 ng/L for fosetyl-Al
  - ✓ LOQ = 30 ng/L for AMPA, glufosinate, glyphosate
- Flexibility and ease of use
- Long term stability and robustness

Methods Sample preparation: Chlorinated water samples were stabilized with sodium thiosulfate to neutralize the presence of chlorine and then analyzed with the LC-MS/MS system.

Surface water and underground water were directly injected.

Chromatography: Separation was achieved using an ExionLC™ System fitted with a 500 µL loop and a CTO-20A column oven.

A LC column of 150 x 4 mm with a guard column of the same material and a 0.5 µm filter was used for separation. This column allows the use of MS amenable mobile phases at around pH 9.

Hiệu năng phân tích cực cao

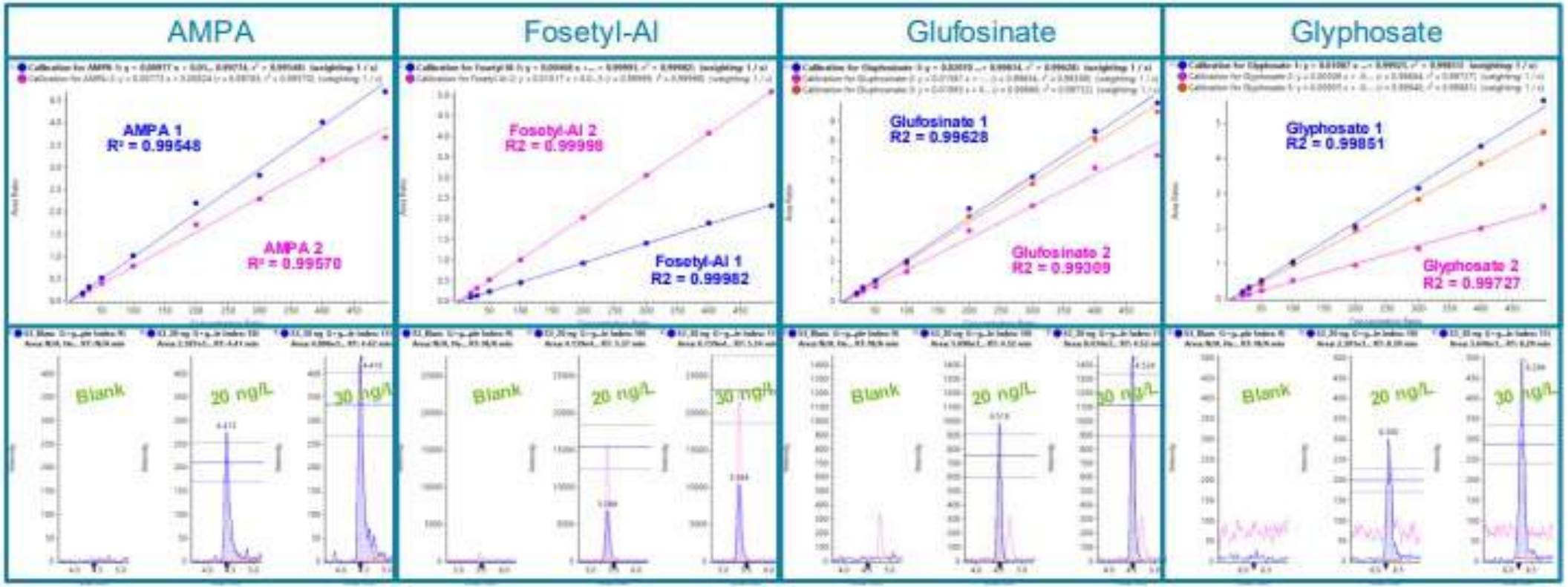


Figure 3. Calibration curves obtained for the third accreditation sequence. (Top) Calibration curves of quantification and qualification MRMs of AMPA, fosetyl-Al, glufosinate and glyphosate corrected with internal standards for S3. (Bottom) Extracted ion chromatogram (XIC) of MRM 1 and MRM 2 for the blank, 20 ng/L and 30 ng/L samples for the four compounds. The solid blue line shows the mean ion ratio calculated from the standards and the dotted lines the tolerance of 20%.

SCIEX

- Tối đa hóa hiệu năng phần cứng
- Phần mềm điều khiển và xử lý dữ liệu

SCIEX - VIET NGUYEN

Đồng hành cùng khách hàng

VIET NGUYEN

- Kỹ thuật giàu kinh nghiệm
- Ứng dụng chuyên sâu

And More


Thank you!

4/ Ứng dụng

Nguồn thông tin: https://www.cargill.com.vn/vi/trang-ch%E1%BB%A7

Nông nghiệp: thu mua, bảo quản, kinh doanh, xử lý cũng như phân phối ngũ cốc và hạt có dầu, bao gồm **lúa mì, ngô, các loại hạt có dầu, lúa mạch và cao lương, dầu thực vật và bã/bột thô** trên phạm vi toàn cầu, một cách đồng nhất.

Ngành Dinh dưỡng và Sức khỏe Vật nuôi: Các sản phẩm và dịch vụ của chúng tôi bao gồm **nguyên liệu thức ăn chăn nuôi**, công thức sản xuất thức ăn chăn nuôi, giải pháp quản lý, dịch vụ quản lý rủi ro, thức ăn chăn nuôi hỗn hợp, phụ gia và hỗn hợp chất bổ sung cho thức ăn chăn nuôi, và các giải pháp tùy chỉnh (đạt **Hazard Analysis and Critical Control Points -chứng nhận HACCP**).

Thực phẩm và nước giải khát

Ca cao và sô cô la; Các nhóm nguyên liệu chức năng; Nguyên liệu giúp tăng cường sức khỏe; Keo nước (Hydrocolloid); Lecithin; Đại mạch (Malt); Dầu và chất béo; Tinh bột và dẫn xuất; Chất làm ngọt; Chất tạo cấu trúc cho thực phẩm

Chăm sóc cá nhân & sắc đẹp

Dược Phẩm: cung cấp các thành phần dược phẩm hoạt tính sử dụng cho các giải pháp thẩm tách máu cũng như thẩm phân phúc mạc và các loại thuốc tiêm

KIỂM NGHIỆM THỰC PHẨM

Theo luật an toàn thực phẩm số 55/2010/QH12 của Quốc hội ngày 17/6/2010, kiểm nghiệm thực phẩm là hoạt động thử nghiệm, đánh giá sự phù hợp với quy chuẩn kỹ thuật và tiêu chuẩn tương ứng đối với **thực phẩm**, **phụ gia thực phẩm, chất hỗ trợ chế biến thực phẩm**, **chất bổ sung vào thực phẩm**, **bao gói, dụng cụ, vật liệu chứa đựng thực phẩm.**

Kiểm nghiệm thực phẩm hướng đến kiểm nghiệm chất lượng và kiểm nghiệm an toàn của sản phẩm thực phẩm

Kiểm nghiệm thành phần dinh dưỡng

Kiểm nghiệm phụ gia thực phẩm

Kiểm nghiệm cảm quan thực phẩm

Kiểm nghiệm độc tố vi nấm

Kiểm nghiệm độc tố tự nhiên

Kiểm nghiệm chất gây dị ứng

Kiểm nghiệm dư lượng thuốc thú y

Kiểm nghiệm dư lượng thuốc bảo vệ thực vật

Kiểm nghiệm kim loại nặng

Kiểm nghiệm vi khoáng

Kiểm nghiệm vi sinh vật

Đánh giá tính kháng kháng sinh của vi khuẩn

Kiểm nghiệm thực phẩm biến đổi gen

Kiểm nghiệm thành phần dinh dưỡng

- ***Thành phần dinh dưỡng đa lượng***: độ ẩm, tro, protein (đạm), lipid (béo), carbohydrat (glucid), đường tổng, đường khử, thành phần đường (glucose, fructose, saccarose, lactose…), xơ thô, xơ tổng, xơ tan, xơ không tan, muối, acid, năng lượng.
- ***Thành phần dinh dưỡng vi lượng***: Viện Kiểm nghiệm an toàn vệ sinh thực phẩm quốc gia liên tục phát triển các phương pháp phân tích thành phần dinh dưỡng vi lượng thực phẩm, đặc biệt, Viện đã thực hiện phân tích được các dạng khác nhau của các vitamin, như:

•Vitamin tan trong dầu: vitamin A (retinol, retinyl palmitate…), carotene (carotene tổng số, beta-caroten), vitamin D (D2, D3), vitamin E (alphatocopherol, tocopheryl acetat, beta-tocopherol, gama-tocopherol, delta-tocopherol), vitamin K (K1, K2, K3, MK4, MK7).

•Vitamin tan trong nước: vitamin nhóm B (B1, B2, B3, B5, B6, B9, B12, biotin), vitamin C (acid ascorbic, ascorbyl palmitate, ascorbyl glucoside, ascorbyl phosphate).

•Các khoáng chất: Natri, Kali, Calci, Sắt, Kẽm, Magie, Selen…

Giới thiệu về phụ gia thực phẩm

Theo Uỷ ban Tiêu chuẩn thực phẩm quốc tế (Codex Alimentarius Commission – CAC), phụ gia thực phẩm là: " Một chất, có hay không có giá trị dinh dưỡng, mà bản thân nó không được tiêu thụ thông thường như một thực phẩm và cũng không được sử dụng như một thành phần của thực phẩm, việc chủ ý bổ sung chúng vào thực phẩm để giải quyết mục đích công nghệ trong sản xuất, chế biến, bao gói, bảo quản, vận chuyển thực phẩm, nhằm cải thiện kết cấu hoặc đặc tính kỹ thuật của thực phẩm đó. Phụ gia thực phẩm không bao gồm các chất ô nhiễm hoặc các chất được bổ sung vào thực phẩm nhằm duy trì hay cải thiện thành phần dinh dưỡng của thực phẩm".

Như vậy, phụ gia thực phẩm không phải là thực phẩm, mà nó được bổ sung một cách chủ ý, trực tiếp hoặc gián tiếp vào thực phẩm nhằm cải thiện kết cấu hoặc đặc tính kỹ thuật của thực phẩm đó. Phụ gia thực phẩm tồn tại trong thực phẩm như một thành phần của thực phẩm với một giới hạn tối đa cho phép đã được quy định.

BỘ Y TẾ

CỘNG HÒA XÃ HỘI CHỦ NGHĨA VIỆT NAM
Độc lập - Tự do - Hạnh phúc

Số: 24/2019/TT-BYT

Hà Nội, ngày 30 tháng 8 năm 2019

THÔNG TƯ

QUY ĐỊNH VỀ QUẢN LÝ VÀ SỬ DỤNG PHỤ GIA THỰC PHẨM

Đối với nguyên liệu phụ gia thực phẩm, Bộ Y tế đã ban hành 23 quy chuẩn Việt nam quy định các yêu cầu kỹ thuật, bao gồm:

1. QCVN 4-1:2010/BYT: QCVN về chất điều vị
2. QCVN 4-2:2010/BYT: QCVN về chất làm ẩm
3. QCVN 4-3:2010/BYT: QCVN về chất tạo xốp
4. QCVN 4-4:2010/BYT: QCVN về chất chống đông vón
5. QCVN 4-5:2010/BYT: QCVN về chất giữ màu
6. QCVN 4-6:2010/BYT: QCVN về chất chống oxy hóa
7. QCVN 4-7:2010/BYT: QCVN về chất chống tạo bọt
8. QCVN 4-8:2010/BYT: QCVN về chất ngọt tổng hợp
9. QCVN 4-9:2010/BYT: QCVN về chất làm rắn chắc
10. QCVN 4-10:2010/BYT: QCVN về phẩm màu
11. QCVN 4-11:2010/BYT: QCVN về chất điều chỉnh độ acid
12. QCVN 4-12:2010/BYT: QCVN về chất bảo quản
13. QCVN 4-13:2010/BYT: QCVN về chất ổn định
14. QCVN 4-14:2010/BYT: QCVN về chất tạo phức kim loại
15. QCVN 4-15:2010/BYT: QCVN về chất xử lý bột
16. QCVN 4-16:2010/BYT: QCVN về chất độn
17. QCVN 4-17:2010/BYT: QCVN về chất khí đẩy
18. QCVN 4-18:2011/BYT: QCVN về nhóm chế phẩm tinh bột
19. QCVN 4-19:2011/BYT: QCVN về enzyme
20. QCVN 4-20:2011/BYT: QCVN về chất làm bóng
21. QCVN 4-21:2011/BYT: QCVN về chất làm dày
22. QCVN 4-22:2011/BYT: QCVN về chất nhũ hóa
23. QCVN 4-23:2011/BYT: QCVN về chất tạo bọt

Kiểm nghiệm độc tố vi nấm

+ Độc tố vi nấm (mycotoxin) là các độc tố do nấm mốc sinh ra, chúng là các sản phẩm phụ của quá trình trao đổi chất.

+400 loại độc tố vi nấm. Một số nhóm độc tố vi nấm chính : aflatoxin B & G, aflatoxin M1, ochratoxin A, patulin, fumonisin, deoxynivalenol và zearalenone.

+Tại các nước châu Âu, giới hạn cho phép của các độc tố vi nấm được quy định tại EC 1881/2006 và quy định sửa đổi EC 1126/2007.

Tại Việt Nam, Bô Y tế quy định mức giới hạn tối đa cho phép của độc tố vi nấm trong thực phẩm tại QCVN 8-1:2011/BYT.

Bộ Nông nghiệp và phát triển nông thôn quy định mức giới hạn tối đa cho phép của độc tố vi nấm aflatoxin trong thức ăn chăn nuôi tại QCVN 01-183:2016/BNNPTNT, QCVN 01 - 78: 2011/BNNPTNT.

Kiểm nghiệm độc tố tự nhiên

Độc tố nhóm Alkaloids độc gồm một số nhóm chính như sau:

•Alkaloid từ cây lá ngón: koumine, gelsemine

•Alkaloid từ cây mã tiền: brucine, strychine

•Alkaloid từ củ ấu tàu: aconitine

•Tropane alkaloid: Một số alkaloid họ cà như atropine , scopolamine (cà độc dược); cocaine (ca cao).

•Pyrrolizidine alkaloids: senecionine, echimidine, intermidine, jacobine…

•Glycoalkaloid: α-solanin, α-choconin, α-tomatin

Độc tố nhóm Cyanogenic glycosides: Một số thành phần cyanogenic glycoside trong sắn, măng, lúa miến, khoai môn: amygladin, hàm lượng tổng cyanide

Độc tố quả vải: HGA, MCPG

Độc tố cóc: resibufogenin, bufalin, cinobufagin

Độc tố nhuyễn thể gồm các nhóm:

•Độc tố gây liệt cơ (PSP)

•Độc tố gây tiêu chảy (DSP)

•Độc tố gây mất trí nhớ (ASP)

Độc tố cá nóc: Tetrodotoxin

Độc tố từ nấm độc: amatoxins, phalloidine, phallacidine

Kiểm nghiệm chất gây dị ứng

Chất gây dị ứng hay còn gọi là chất dị nguyên là những chất sau khi xâm nhập vào cơ thể sinh vật sẽ được hệ thống miễn dịch nhận biết và đáp ứng, tức là sinh ra các kháng thể tương ứng có đặc tính kết hợp với kháng nguyên ấy.

Chất gây dị ứng có thể là protein hoặc phức hợp của protein và polysaccharide, protein và lipid, lipid và polysaccharide hay protein và các hoá chất đơn giản. Cấu trúc hoá học, đặc biệt là cách sắp xếp acid amin trong dãy peptide là điều kiện quyết định tính kháng nguyên của peptide đó.

Ở châu Âu, Mỹ và nhiều nước phát triển, hiện đã có những quy định về việc ghi nhãn hàng hóa, công bố các thành phần thực phẩm có trong sản phẩm. Hàm lượng tối đa cho phép của các chất gây dị ứng trong thực phẩm tại châu Âu và Hoa Kỳ được quy định là 10 mg/kg.

Ở Việt Nam , theo thông tư liên tịch số 34/2014/TTLT-BYT-BNNPTNT-BCT về việc hướng dẫn ghi nhãn hàng hóa đối với thực phẩm, phụ gia thực phẩm và chất hỗ trợ chế biến bao gói sẵn cũng đã quy định việc công bố thành phần cấu tạo của thực phẩm, ví dị như công bố sự có mặt của trứng, lạc, đậu tương, sữa...

Kiểm nghiệm thuốc bảo vệ thực vật và thuốc thú y

Thuốc bảo vệ thực vật là những hợp chất hóa học (vô cơ hoặc hữu cơ), những chế phẩm sinh học (chất kháng sinh, vi khuẩn, nấm siêu vi trùng…) những chất có nguồn gốc động vật, thực vật được sử dụng để bảo vệ cây trồng và nông sản, chống lại sự phá hại của những sinh vật gây hại như côn trùng, nhện, tuyến trùng, chuột, chim, rong rêu cỏ dại

Một số quy định về thuốc trừ sâu và thuốc thú y tại Việt Nam:

- Thông tư số 10/2016/TT-BNNPTNT ban hành danh mục thuốc thú y được phép lưu hành, cấm sử dụng ở Việt Nam, công bố mã HS đối với thuốc thú y nhập khẩu được phéo lưu hành tại Việt Nam
- Thông tư số 13/2016/TT-BNNPTNT quy định về quản lý thuốc thú y của Bộ Nông nghiệp và phát triển nông thôn
- Quy chuẩn kỹ thuật quốc gia QCVN 01-188/BNNPTNT về chất lượng thuốc bảo vệ thực vật.
- Thông tư 10/2020/TT-BNNPTNT ban hành danh mục thuốc bảo vệ thực vật được phép sử dụng, cấm sử dụng tại Việt Nam

KIỂM NGHIỆM THỰC PHẨM

Thức ăn chăn nuôi là những sản phẩm thức ăn mà vật nuôi được cho ăn, uống (hoặc bổ sung vào môi trường đối với vật nuôi thủy sản) ở dạng tươi, sống, hoặc đã qua chế biến, bảo quản, bao gồm thức ăn dinh dưỡng và thức ăn chức năng ở các dạng: nguyên liệu, thức ăn đơn; thức ăn hỗn hợp hoàn chỉnh; thức ăn đậm đặc, thức ăn bổ sung; phụ gia thức ăn và các sản phẩm bổ sung vào môi trưởng nuôi (đối với thức ăn thủy sản) nhằm tạo thức ăn tự nhiên, ổn định môi trường nuôi, tăng hiệu quả sử dụng thức ăn.

kiểm nghiệm nguyên liệu thực phẩm

* Nhóm các chỉ tiêu Vật lý - Cảm quan:
•Cảm quan: màu sắc, trạng thái, mùi.
•Chỉ số acid, chỉ số peroxid
•Độ ẩm, mất khối lượng khi sấy khô.
•....
* Nhóm các chỉ tiêu chất lượng:
•Thành phần đa lượng: protein (đạm), lipid (béo), carbohydrat (glucid), tro
•Xác định hàm lượng hoạt chất chính:
+ Acid amin trong nguyên liệu và thành phẩm TĂCN
+ Các vitamin trong dạng premix hoặc thức ăn hỗn hợp
....

* Nhóm các chỉ tiêu an toàn:
•Phân tích độc tố vi nấm – mycotoxin (Aflatoxin, Ochratoxin, Citrinin,….)
•Phân tích dư lượng hóa chất bảo vệ thực vật (tất cả các nhóm Pesticide)
•Phân tích xác định giới hạn ô nhiễm vi sinh vật (E. coli, nấm men, mốc,….)
•Phân tích dư lượng kháng sinh, hormon tăng trưởng.
•Xác định hàm lượng kim loại nặng: Pb, Cd, As, Hg (vô cơ và hữu cơ)

For Research Use Only. Not for Use In Diagnostic Procedures.

Drug Discovery and Development

SCIEX

Quantitative LC-MS Solution for Targeted Analysis of Cell Culture Media

Featuring the SCIEX QTRAP® 6500+ LC-MS/MS System

Zuzana Demianova[1] and Lei Xiong[2]
[1]SCIEX, Brea, CA; [2]SCIEX, Redwood City, CA

Measurement of polar and nonpolar as well as positive and negative polarity components within a single method

Component group	Number of components
Amino acids	*39*
Vitamins	*15*
Carbohydrates	*4*
Fatty acids	*5*
Nucleic acids	*17*
Others	*32*

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Nucleic acids	*17*
Others	*32*

Mass Spec: MRM parameters for 110 components
+ a Triple Quad™ 6500+ system.
+ The scheduled MRM™ Algorithm Pro(sMRM) was used
+ Two MRM transitions were monitored for each analyte,
+This method contains 178 positive MRMs and 54 negative MRMs.

Chromatography: an ExionLC™ System
+ Phenomenex Kinetex® F5 column (150 mm x 2.1 mm ID, particle size 2.6 µm).
+ Total method time was 20 min
+ flowrate of 200 µl/min.
Mobile phase A was 0.1% formic acid in water, and mobile phase B was acetonitrile with 0.1% formic acid.
+ Column oven temperature was 40°C.
+ Injection volume was 5 µL.

***Sample preparation*:** A final master mix was prepared containing all standards (6.67-20 µg/ml) depending on analyte signal).
Cell culture medium (CD CHO medium, Gibco) was diluted 5-fold with 0.1% formic acid in 50% acetonitrile, and centrifuged. The supernatant was further diluted 60-fold with 0.1% formic acid.

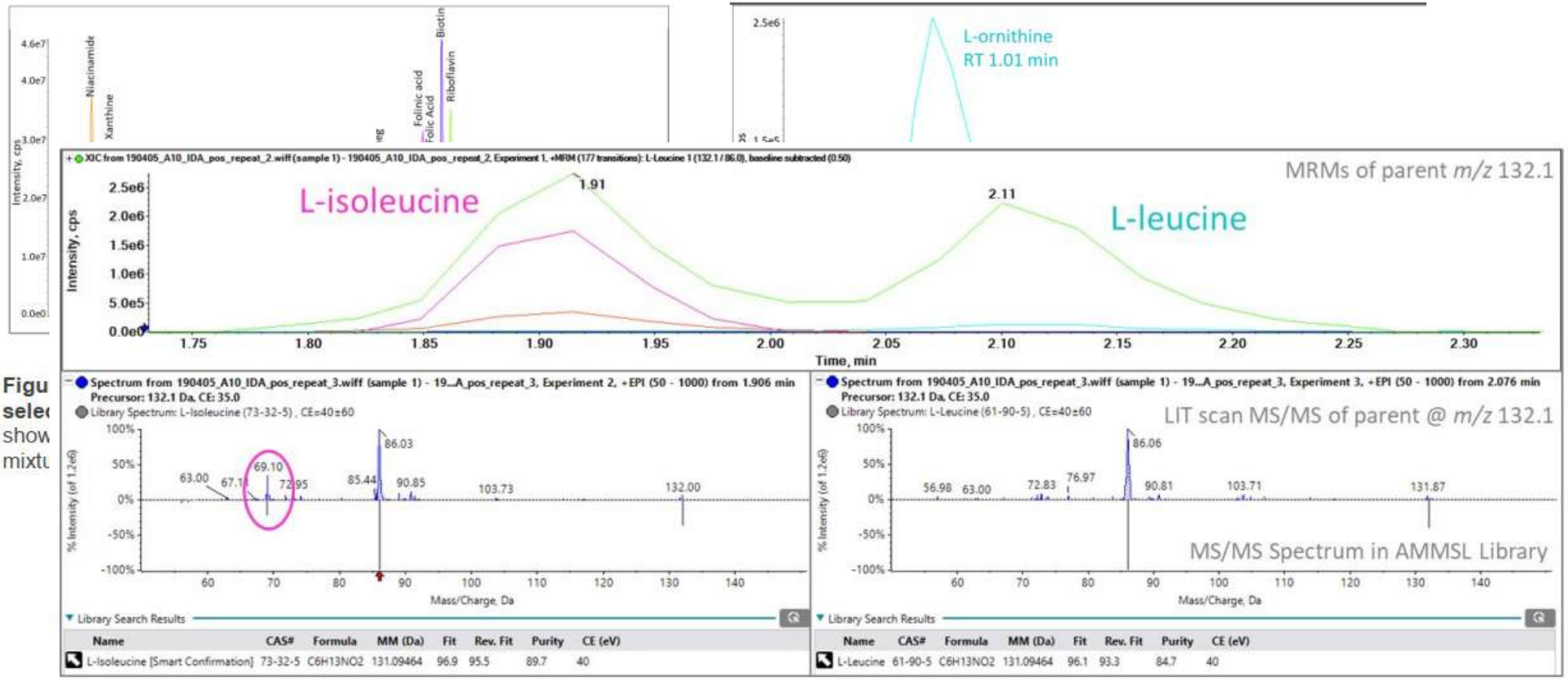
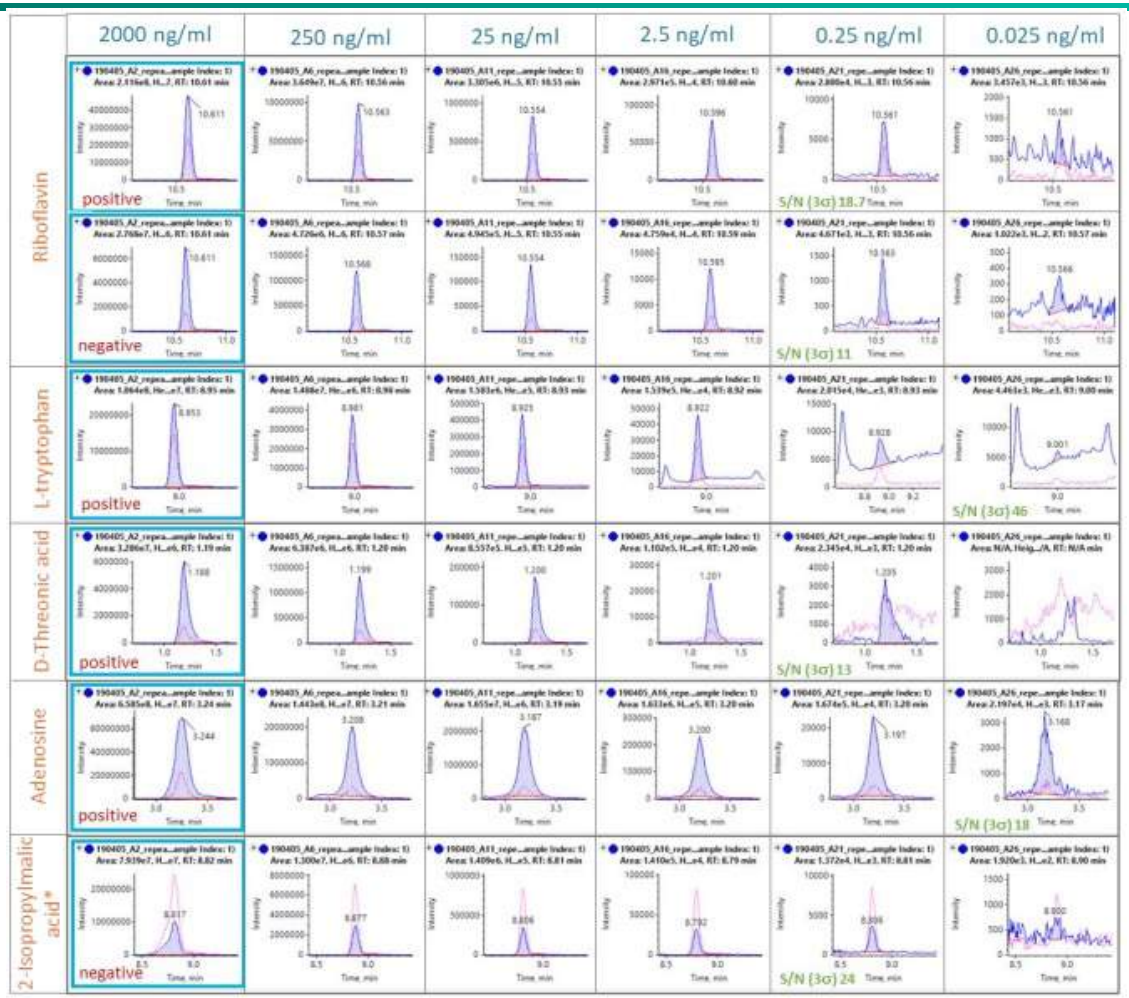


Figure 5. Representative MIDAS™ workflow for L-Leucine and L-Isoleucine with compound match from the Sciex Accurate Metabolite Spectra

KIỂM NGHIỆM THỰC PHẨM



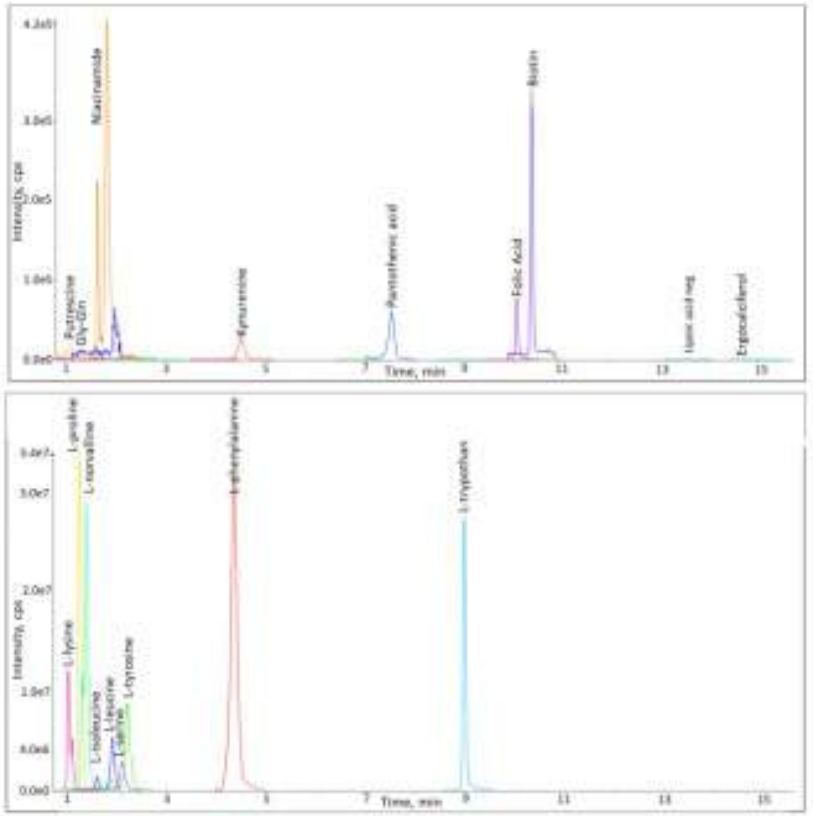
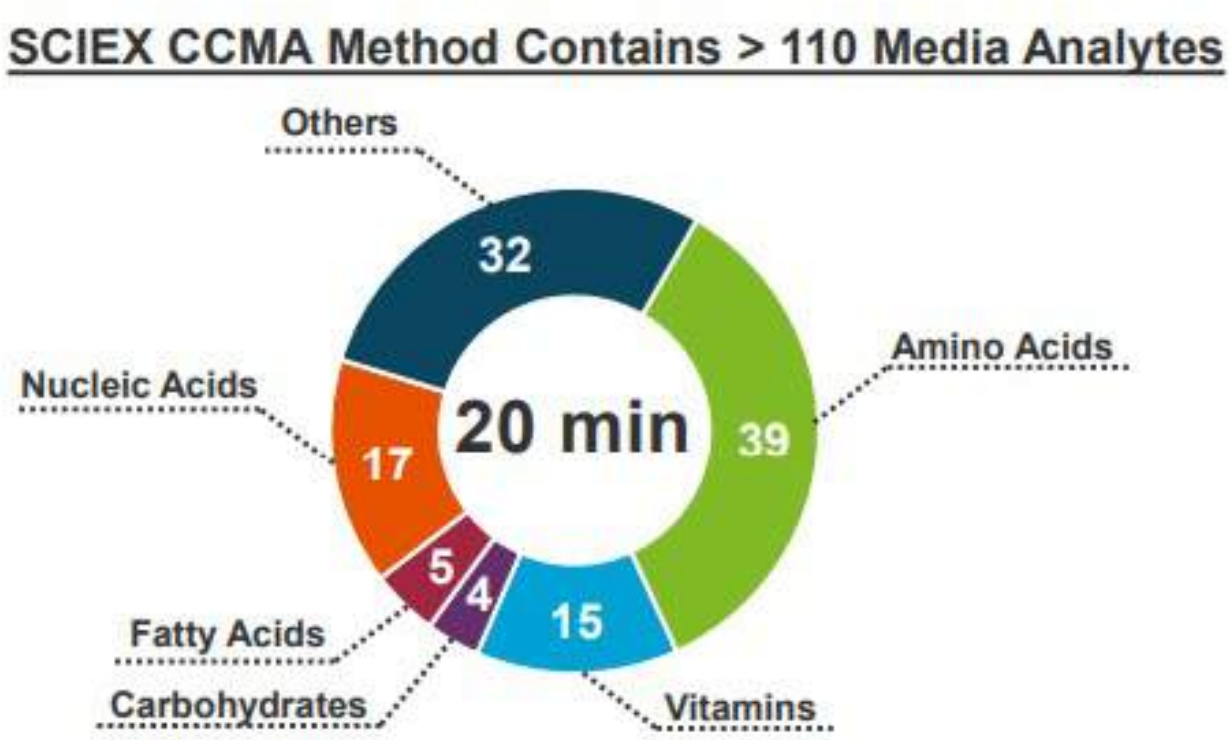


Figure 3. Top panel shows various XICs of low abundant components from Gibco's cell culture media (CD CHO medium). Bottom panel shows amino acids from Gibco's cell culture media (CD CHO medium).

KIỂM NGHIỆM THỰC PHẨM

High sensitivity
Selectivity
Speed
Robustness;
Enables unambiguous identification and quantification of a large number of analytes in a single analysis



KIỂM NGHIỆM THỰC PHẨM

Food and Environmental

Table 1. Daily required values (DV) of different B vitamins for a human adult as obtained from the FDA[1]

Vitamin		DV (mg)
Thiamine	B1	1.5
Riboflavin	B2	1.7
Niacin	B3	20
Pantothenic acid	B5	10
Pyridoxal	B6	2
Biotin	B7	0.3
Folic acid	B9	0.4
Cyanocobalamin	B12	0.006

mplex in Infant Formula

Concord, Ontario (Canada)

Vitamin B is a group of water-soluble vitamins

Food samples:matrices complex
Sensitive methods typically require highly selective sample clean up procedures.
Vitamin B is a complex mixture of highly polar compounds (Figure 1) whose pK_a range from 0.5 to 10.2, making their analysis challenging.

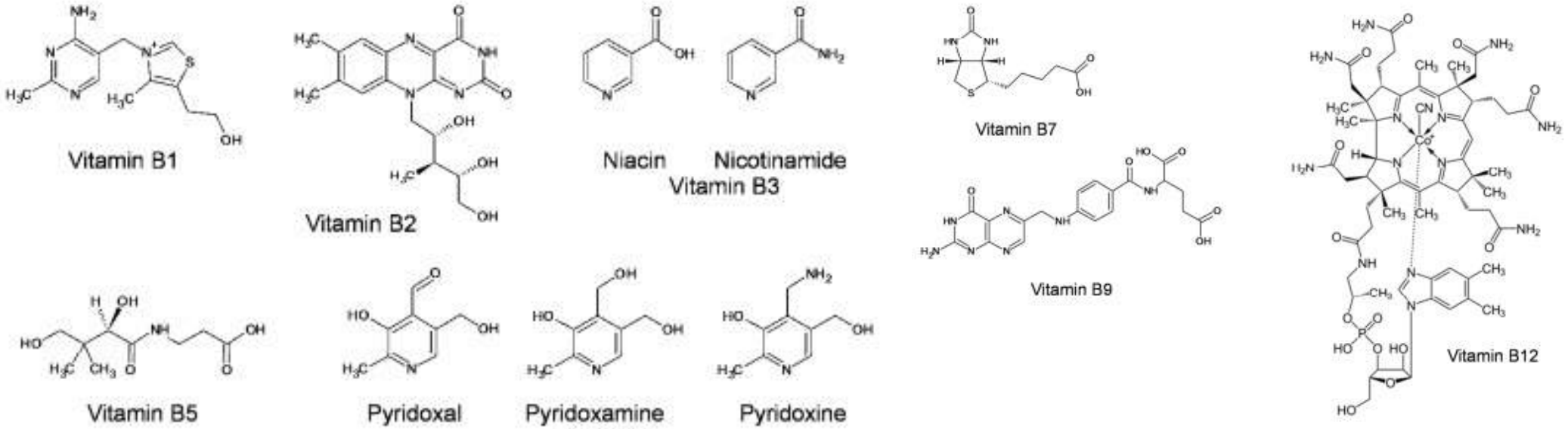


Figure 1. Chemical structures of B vitamins

Sample Preparation

Sample (1 g) was mixed with 50% acetonitrile in acidified water (containing an antioxidant) + IS.

Shaken vigorously for 1 minute, roller mixed for 10 minutes (protected from light).

After centrifugation, filtered and the filtrate diluted 20 times with water containing an ion paring reagent. The sample preparation

was kept as simple as possible to reduce possible vitamin breakdown, with SPE no longer needed for the late eluting B7,

B9, and B12 vitamins.

During the development work the effects of light, temperature,

and acidity on standard stability were tested and it was found

that the use of amber glass with a lower pH with the presence of

an antioxidant helped stabilize the extracts.