¿Cómo analizar residuos de contaminantes en matrices alimenticias utilizando innovaciones tecnológicas?

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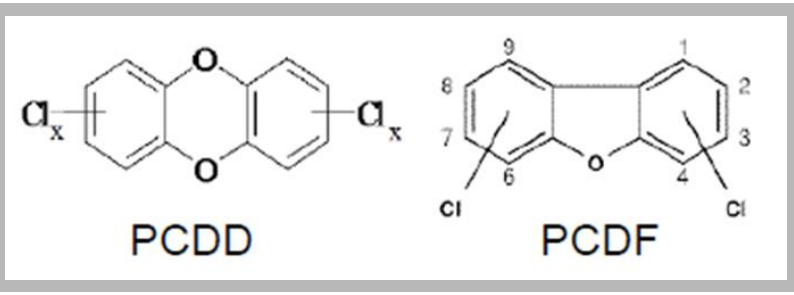
Qué son las Dioxinas



- Las Dioxinas son un grupo de contaminantes orgánicos persistentes altamente tóxicos
- No sintetizadas deliberadamente – son productos de procesos de fabricación en la industria de pulpa y papel como el blanqueo, de la incineración de desechos y de la fabricación de algunos herbicidas y plaguicidas.
- 80% de la exposición en Humanos proviene de alimentos de origen animal
- Las Dioxinas se acumulan en el tejido adiposo de los animales.
- Monitoreo por parte de Agencias Ambientales como European Commission (EC), EPA, WHO, etc

Legislación de Dioxinas

US EPA 1613 (1994)

Legislación UE EC 1883/2006

- Reglamento (CE) 1883/2006 de 19 de diciembre de 2006 (DOL 364, de 20-12-2006)
- Reglamento (CE) 565/2008 de 18 de Junio de 2008 (DOL 160 de 19-06-08)
- Reglamento (UE) 420/2011 de 19 de diciembre de 2011 (DOL 111 de 30-04-2011)
- Reglamento (UE) 1259/2011 de 02 de diciembre de 2011 (DOL 320 de 03-12-2011)
- Reglamento (UE) 594/2012 de 05 de julio de 2012 (DOL 176 de 06-07-2011)
- Reglamento (UE) 2015/704 de 30 de abril de 2015 (DOL 113 de 01-05-2015

GC/MS/MS es ahora una técnica confirmatoria en Análisis de Dioxinas/Furanos/PCB´s!

European Market for Dioxin analysis in [Animal] Feed and Foodstuffs

Commission Regulation (EU) No 589/2014 (of 2 June 2014)
laying down methods of sampling and analysis for the control of levels of dioxins, dioxin-like PCBs and non-dioxin-like PCBs in certain foodstuffs and repealing Regulation (EU) No 252/2012

Commission Regulation (EU) No 709/2014 (of 20 June 2014)
amending Regulation (EC) No 152/2009 as regards the determination of the levels of dioxins and polychlorinated biphenyls in feed

Except from EU No 589/2014

(9) In addition to the gas chromatography/high resolution mass spectrometry (GC-HRMS), technical progress and developments have shown that also gas chromatography/tandem mass spectrometry (GC-MS/MS) can be used as a confirmatory method for checking compliance with the maximum level (ML). Regulation (EU) No 252/2012 should therefore be replaced by a new Regulation providing for the use of gas chromatography/tandem mass spectrometry (GC-MS/MS) as an appropriate confirmatory method for checking compliance with the maximum level.

In force as of June 20th 2014

Junio/Julio 2014: breaking news in Europe for GC/MS/MS!

L 164/18 EN Official Journal of the European Union 3.6.2014

COMMISSION REGULATION (EU) No 589/2014

of 2 June 2014

laying down methods of sampling and analysis for the control of levels of dioxins, dioxin-like PCBs and non-dioxin-like PCBs in certain foodstuffs and repealing Regulation (EU) No 252/2012

(Text with EEA relevance)

THE EUROPEAN COMMISSION,

Having regard to the Treaty on the Functioning of the European Union,

Having regard to Regulation (EC) No 882/2004 of the European Parliament and of the Council of 29 April 2004 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules (1), and in particular Article 11(4) thereof,

3.6.2014 EN Official Journal of the European Union L 164/19

(7) In fish of the same species originating from the same region, the level of dioxins, dioxin-like PCBs and non-dioxin-like PCBs can be different depending on the size and/or the age of the fish. Moreover, the level of dioxins, dioxin-like PCBs and non-dioxin-like PCBs is not necessarily the same in all parts of the fish. Therefore, it is necessary that the sampling and sample preparation is specified in order to ensure a harmonised approach throughout the Union.

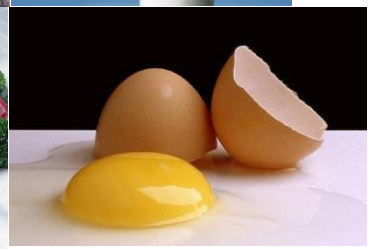
(8) It is important that analytical results are reported and interpreted in a uniform way in order to ensure a harmonised enforcement approach throughout the Union.

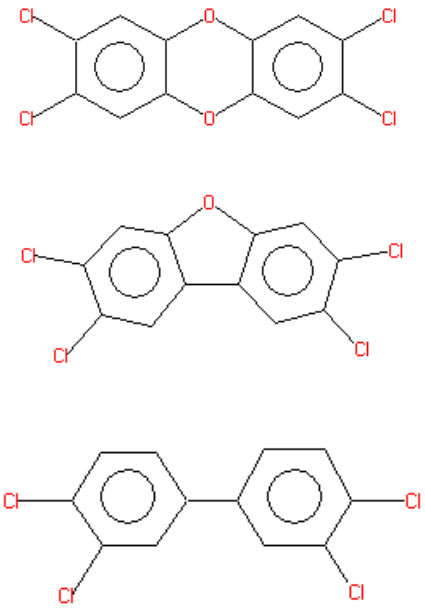
(9) In addition to the gas chromatography/high resolution mass spectrometry (GC-HRMS), technical progress and developments have shown that also gas chromatography/tandem mass spectrometry (GC-MS/MS) can be used as a confirmatory method for checking compliance with the maximum level (ML). Regulation (EU) No 252/2012 should therefore be replaced by a new Regulation providing for the use of gas chromatography/tandem mass spectrometry (GC-MS/MS) as an appropriate confirmatory method for checking compliance with the maximum level.

(10) The measures provided for in this Regulation are in accordance with the opinion of the Standing Committee on the Food Chain and Animal Health,

(5) A screening method of analysis with widely acceptable validation and high throughput can be used to identify the samples with significant levels of PCDD/Fs and dioxin-like PCBs (preferably selecting samples exceeding action levels and ensuring the selection of samples exceeding maximum levels). The levels of PCDD/Fs and dioxin-like PCBs in these samples need to be determined by a confirmatory method of analysis. It is therefore appropriate to establish appropriate requirements for the screening method making sure that the false-compliant rate with respect to maximum levels is below 5 % and strict requirements for the confirmatory methods of analysis. Furthermore, confirmatory methods with sufficient sensitivity allow the determination of levels also in the low background range. That is important for to follow time trends, exposure assessment and for the re-evaluation of maximum and action levels.

Determinación de policlorodibenzodioxinas (PCDDs) y policlorodibenzofuranos (PCDFs) en matrices alimenticias usando el sistema GC-MS/MS





Analizador GC/MS/MS de Agilent para la detección de Dioxinas en alimentos

Printed in U.S.A., February 10, 2015
5991-5471EN

DETECT AND REPORT
TRACE-LEVEL DIOXINS AND DIOXIN-LIKE PCBs

The Measure of Confidence

Agilent GC/MS/MS Dioxins in Feed and Food Analyzer

Dioxin and dioxin-like PCBs are environmental pollutants and persistent organic pollutants (POPs) that originate as by-products of industrial processes such as paper bleaching, pesticide manufacturing, and waste incineration. These compounds accumulate in the food chain, mainly in the fatty tissue of animals. Humans can ingest these highly toxic compounds from eating meat, dairy, fish, and other animal products.

The Agilent Dioxins in Feed and Food Analyzer leverages the latest GC and MS/MS innovations and reflects our focus on quality and performance and includes:

Factory

Matrices?

El Sistema es utilizado para:

Todas las matrices alimenticias
(ej. pescado, carne, aceite de pescado, huevo, aceites vegetales)

Plantas (ej. Alfalfa)**, hierbas** & e**specias** (ej. oregano)

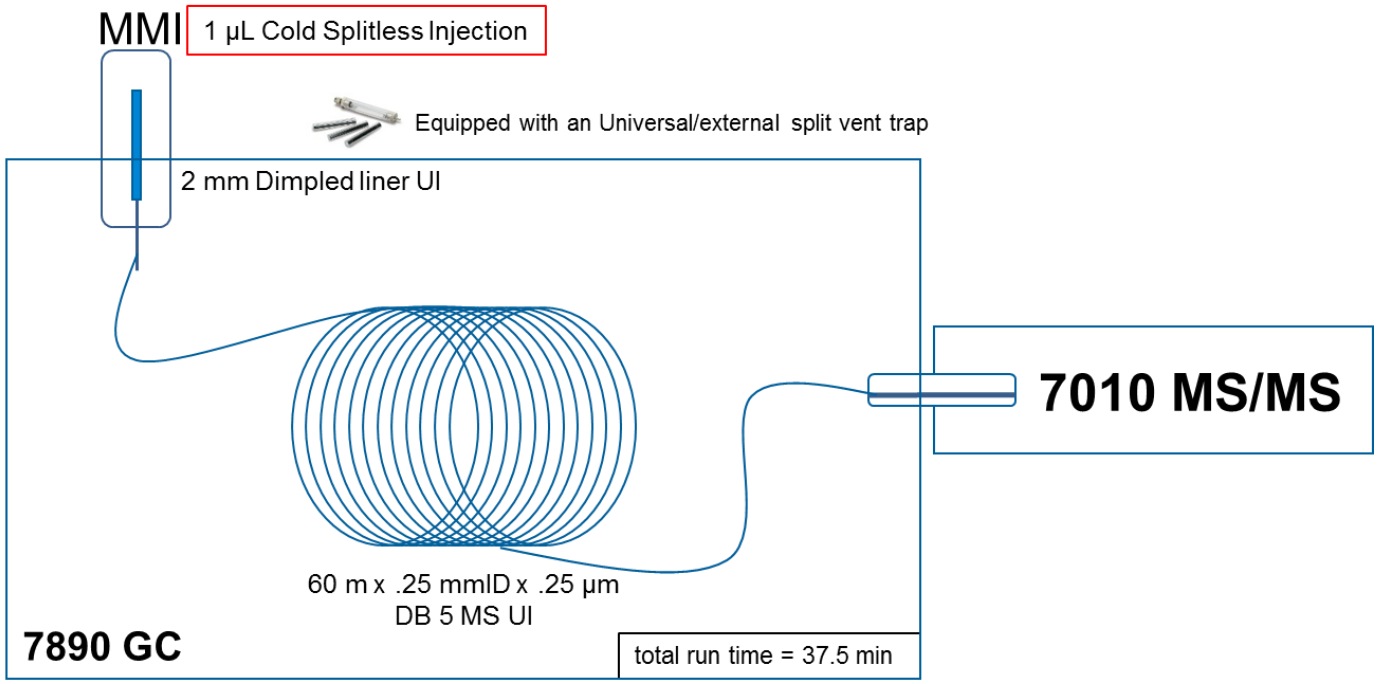
Todas las matrices ambientales
(ej. suelo, lodos, aguas residuales, sedimentos)

Configuración del Analizador de Dioxinas GC/MS/MS

Configuración Simple, ya aceptada por Laboratorios de Dioxinas de la Unión Europea!

El Analizador utiliza:

- Nuevo sistema 7010 GC-MS/MS – Aumenta sensibilidad
- Iguales parámetros de método GC para ambas fracciones de muestra – Aumenta Productividad
- MMI – Versatilidad con técnicas/volumenes de inyección
- Columna GC – Validada para análisis de dioxinas
- RT locked MRM transitions – Optimizado & Validado en un Lab de Referencia de Dioxinas en Europa



RT Locking compound – **PCB 105** @ 15.700 min

Condiciones del Método/Set points

GC Conditions			
Column	DB 5MSUI 60 m x 0.25 mmID x 0.25 µm		
Injection port liner	2mm id dimpled splitless liner, UI		
Injection mode	Cold-splitless (compressed air/CO_2 cooled MMI)		
Injection volume	1 µL		
Inlet temperature program		60 °C	0.31 min
	600 °C/min	330 °C	5 min
Carrier gas	He, constant flow 0.700 mL/min		
Oven program		60 °C	1 min
	30 °C/min	270 °C	1 min
	2 °C/min	310 °C	0 min
	5 °C/min	350 °C	0.5 min
MS transfer line temperature	350 °C		

MS set points	
Electron Energy	70 eV
Tune	eihs.tune.xml
EM gain	10
MS1 resolution	Unit
MS2 resolution	Unit
Collision Cell	1.5 mL/min N_2 4 mL/min He
Quant/Qual transitions	Fraction Specific
Dwell times	Fraction Specific
Collision energies	Optimized
Source temperature	350 °C
Quad temperatures	150 °C

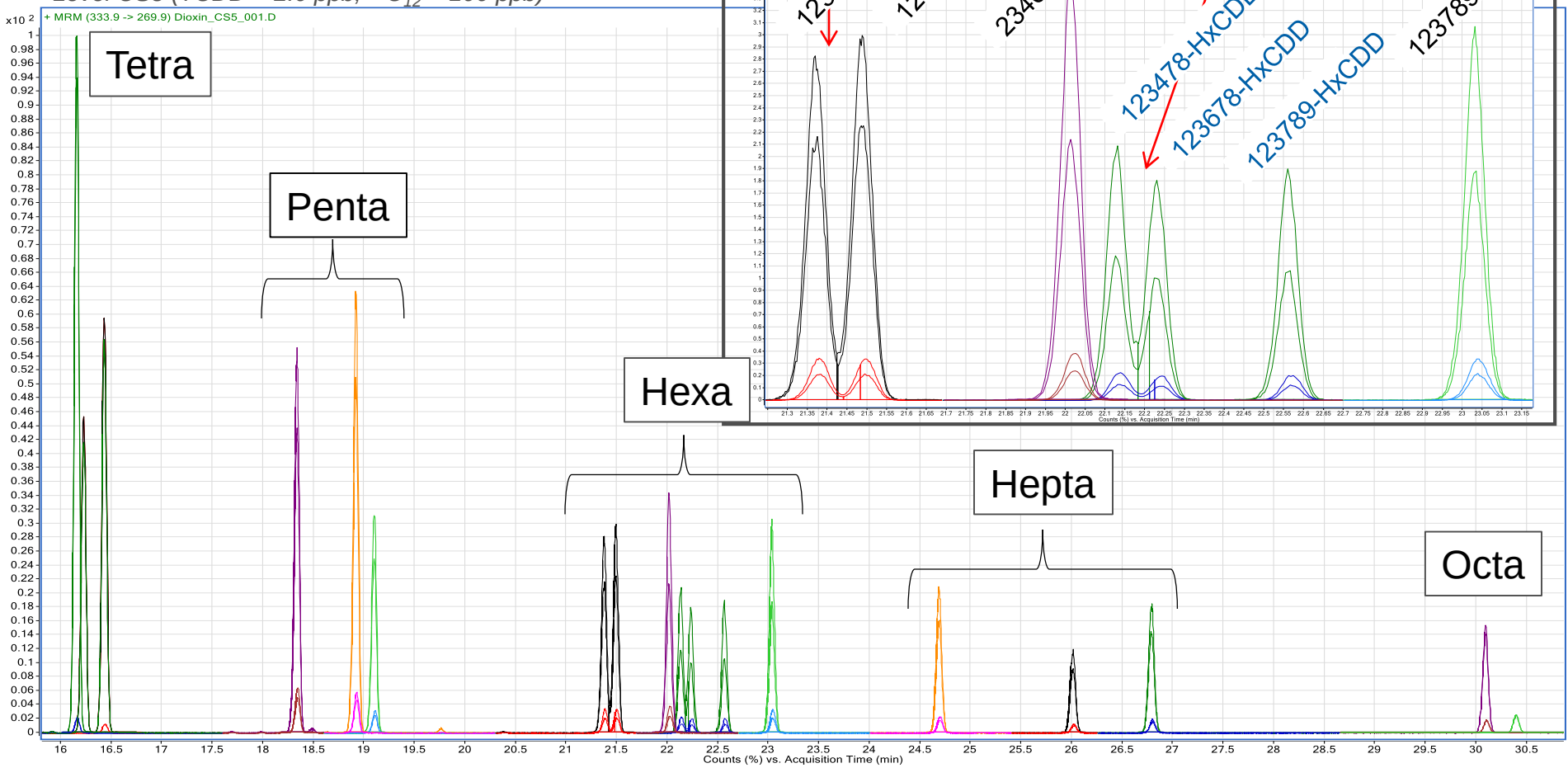
Las Condiciones del GC son las mismas para ambas fracciones

Dioxinas/Furanos – Cromatograma

Excelente separación de los isomeros *hexa*-dioxinas/furanos

Columna GC– DB5MS UI (*60m x 0.25mm x 0.25 µm*)

Level CS5 (TCDD = 2.0 ppb; $^{13}C_{12}$ = 100 ppb)

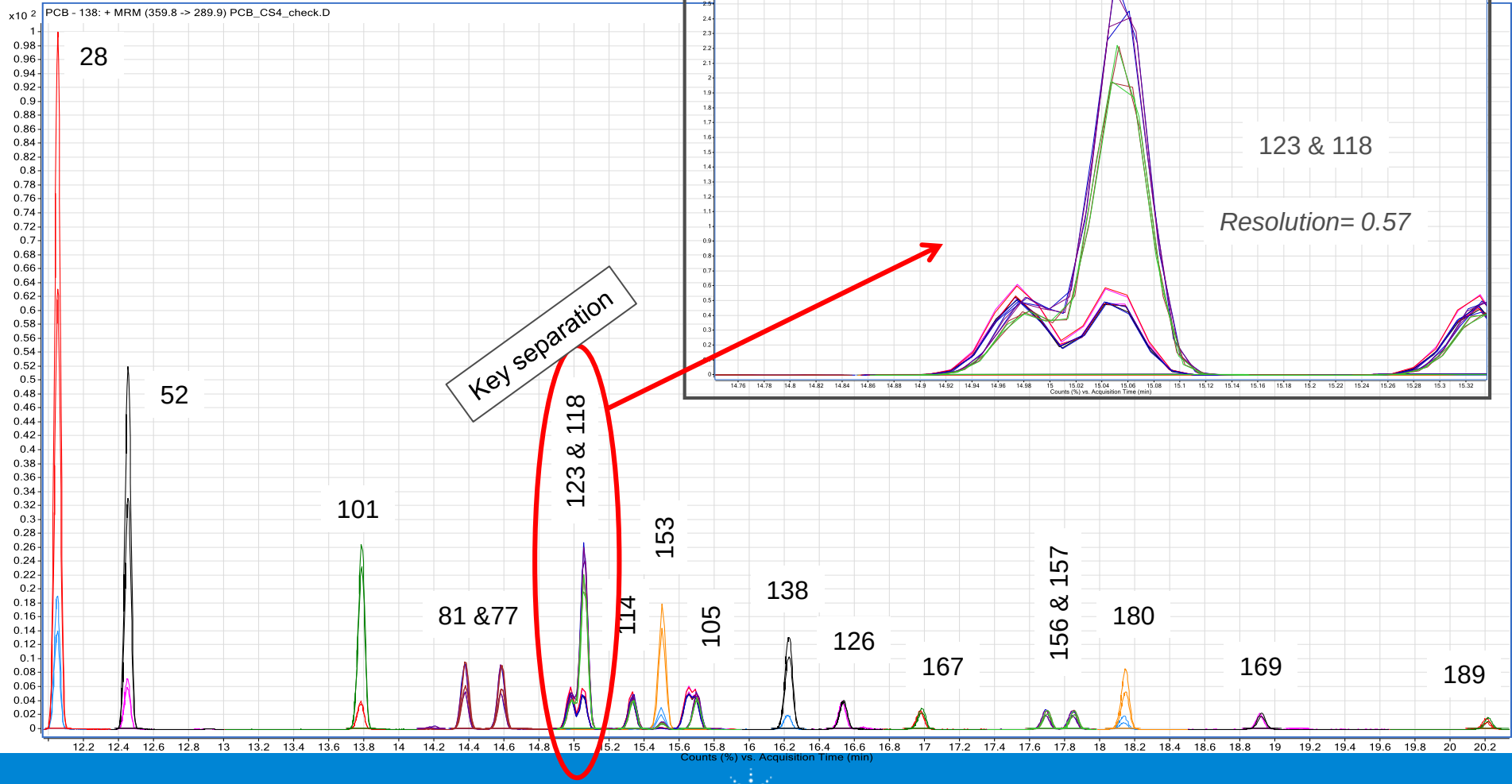


PCBs – Cromatograma

La clave para separar PCBs mono-orto substitutidos 123 & 118 se logra con el mismo método de Dioxinas con los mismos parámetros.

GC Column – DB 5MS UI (60m x 0.25mm x 0.25 µm)

Level CS4 (PCB 123/118 = 10/50 ppb; $^{13}C_{12}$ = 10 ppb)



GC/MS/MS Límite de Detección Instrumental (IDL_{RSD}) ~ ***fg***

$$IDL_{RSD} = \frac{t_{\alpha,n-1} \times RSD \times c}{100}$$

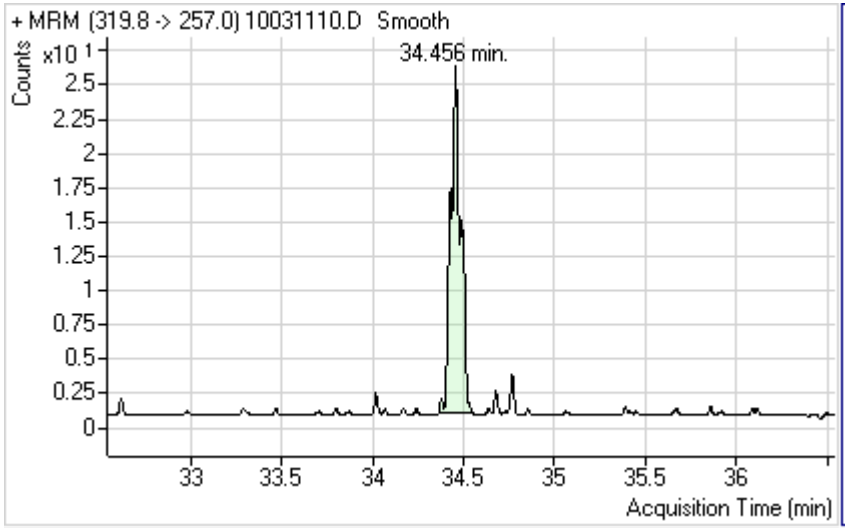
$t_{\alpha,n-1}$ = t value (coefficient) at the level of α with the sample size of n-1

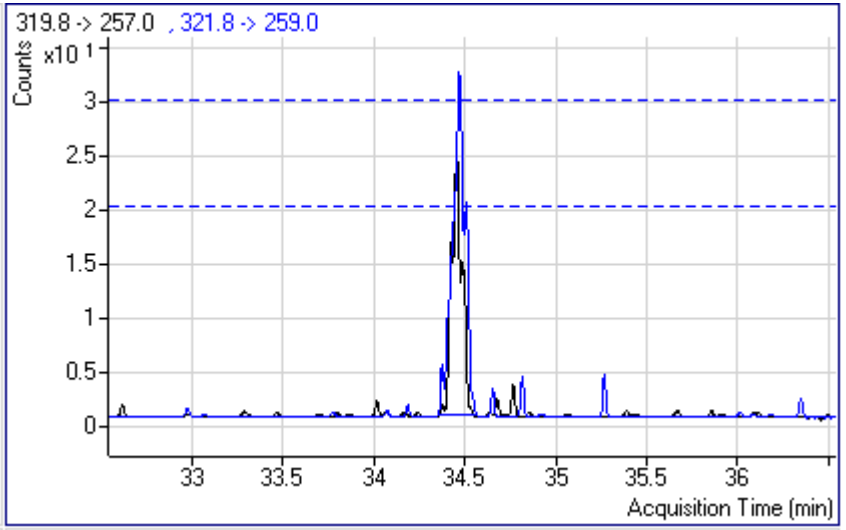
c = concentration of the std sample injected

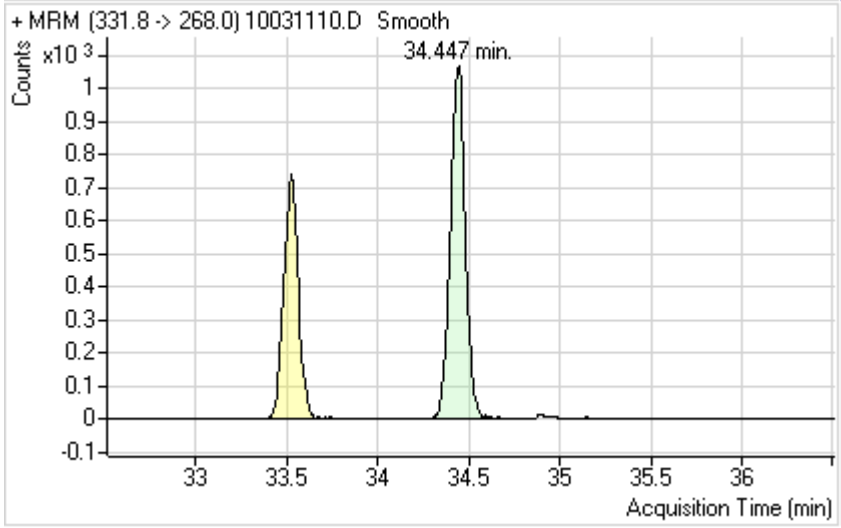
CMPD	RRF	10 reps (CS1)	
		%RSD	IDL_{RSD} (fg)
2378-TCDF	1.180	4.92	6.8
2378-TCDD	1.258	4.28	**5.9**
12378-PeCDF	1.206	2.39	16.5
23478-PeCDF	0.961	2.98	20.6
12378-PeCDD	1.080	3.91	27.0
123478-HxCDF	1.278	3.33	23.0
123678-HxCDF	1.194	2.58	17.8
234678-HxCDF	1.171	2.71	18.7
123478-HxCDD	1.184	4.83	33.4
123678-HxCDD	1.183	4.40	30.4
123789-HxCDD	1.178	4.92	34.0
123789-HxCDF	1.906	2.24	15.5
1234678-HpCDF	1.183	2.54	17.6
1234678-HpCDD	1.171	3.37	23.3
1234789-HpCDF	0.875	5.44	37.6
OCDD	1.391	3.69	51.0
OCDF	1.963	3.04	42.0

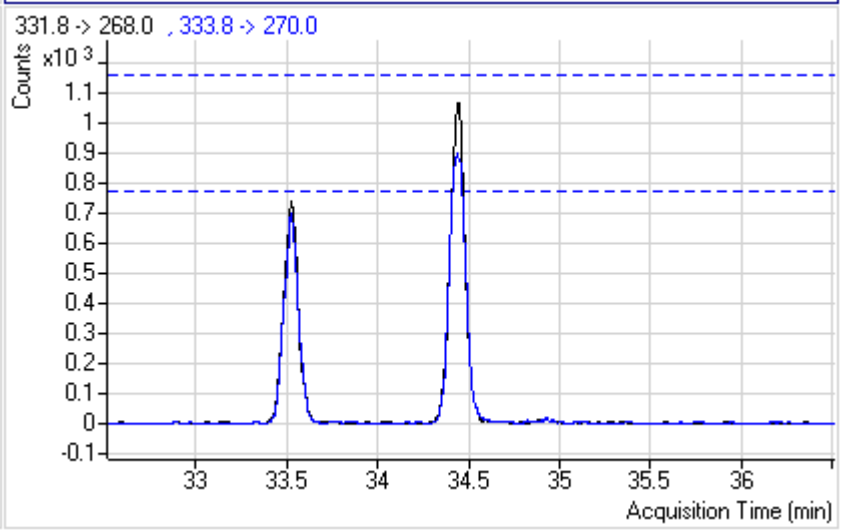
CMPD	RRF	10 reps (CS1)	
		%RSD	IDL_{RSD} (fg)
PCB – 28	1.077	2.40	33.2
PCB – 52	1.465	1.91	26.3
PCB – 101	1.276	1.57	21.6
PCB – 77	1.024	1.71	4.7
PCB – 81	1.040	1.41	**3.9**
PCB – 118	0.620	1.43	19.8
PCB – 123	2.854	16.11	44.5
PCB – 105	0.671	19.44	53.7
PCB – 114	3.316	9.89	27.3
PCB – 153	0.883	1.97	27.3
PCB – 138	1.402	1.17	16.2
PCB – 126	1.061	5.43	15.0
PCB – 167	1.168	2.11	5.8
PCB – 156	1.053	4.24	11.7
PCB – 157	1.025	3.49	9.6
PCB – 180	0.930	1.24	17.2
PCB – 169	1.228	2.12	5.9
PCB – 189	1.095	3.13	8.7

Tetra-clorodibenzo-dioxinas, TCDD en Carne Roja (0.28 pg TEQ /g Fat)







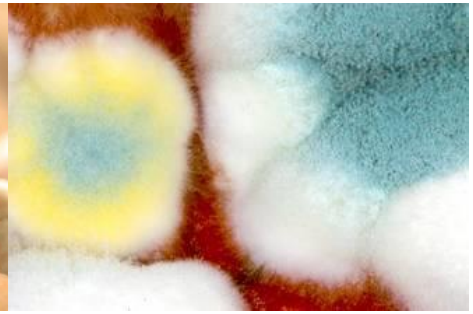


Dioxinas / DBFs Total TEQ* en extracto de carne

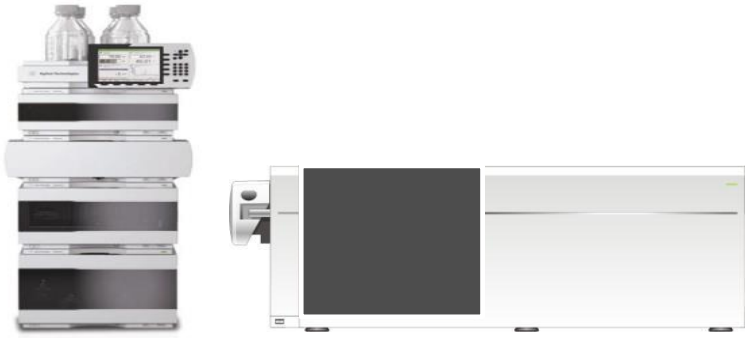
	Analyte	Analytical result	TEF Value	TEQ
		pg/g Fat		pg/g Fat
Dioxins	2378-TCDD	0.281	1	0.281
	12378-PCDD	0.237	1	0.237
	123478-HxCDD	0.001	0.1	0.0001
	123678-HxCDD	0.575	0.1	0.0575
	123789-HxCDD	0.040	0.1	0.004
	1234678-HpCDD	0.663	0.01	0.00663
	OCDD	0.209	0.0001	0.000021
			Dioxins Total TEQ pg/g fat	0.586
Furans	2378-TCDF	0.037	0.1	0.0037
	12378-PCDF	0.009	0.05	0.00045
	23478-PCDF	3.289	0.5	1.6445
	123478-HxCDF	1.475	0.1	0.1475
	123678-HxCDF	1.539	0.1	0.1539
	234678-HxCDF	1.584	0.1	0.1584
	123789-HxCDF	0.002	0.1	0.0002
	1234678-HpCDF	0.439	0.01	0.00439
	1234789-HpCDF	0.108	0.01	0.00108
	OCDF	0.356	0.0001	0.000036
			Furans Total TEQ pg/g fat	2.114
			Total (Dioxins + Furans)TEQ pg/g fat	2.70
			Max permitted level TEQ pg/g fat	3

*TEQ = Concentración Equivalente de Toxicidad

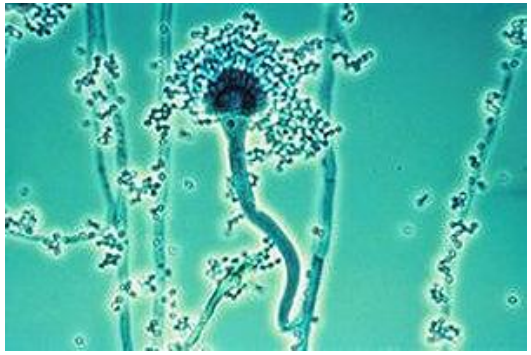
Total TEQ = Suma de resultados de 17 Dx/DBFs, expresada como pg-TEQ/g grasa

Análisis de Micotoxinas en Fórmula Infantil utilizando LC/MS/MS

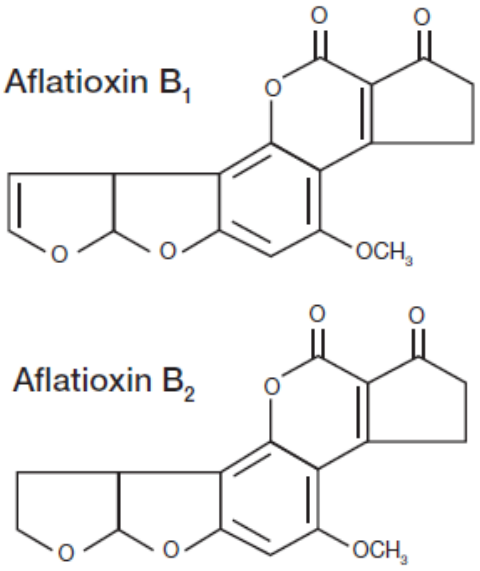


Micotoxinas

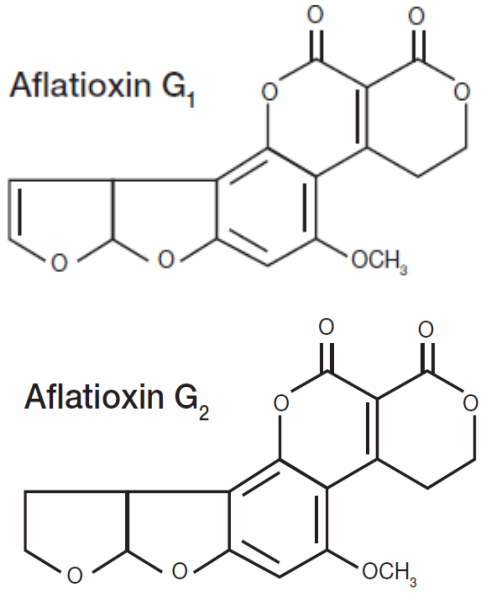


Metabolitos secundarios tóxicos producidos por organismos del reino fungi, principalmente hongos. Las Micotoxinas más comunes son producidas por especies de los géneros *Aspergillus, Penicillium y Fusarium*

Aflatoxinas B_1 & B_2 :
(producidas por *Aspergillus flavus* y *A. parasiticus*.)



Aflatoxinas G_1 & G_2 :
(producidas por *Aspergillus parasiticus.)*



Micotoxinas reguladas

- **Aflatoxinas: B_1, B_2, G_1, G_2 y M_1**
- **Tricotecenos Tipo A y B: fusX, DON, DON-1, 3–acetil DON, 15–acetil DON, HT-2, DAS, T-2, T-2 Triol, NIV y NEO**
- **Fumonisinas: B_1 and B_2**
- **Zeralenona: ZON**
- **Ocratoxinas: A y B**
- **Otras toxinas: Patulina, citrinina, sterigmacistina**

Preparación de muestra

0.5 g muestra
(representativa y homogeneizada)

Extracción con 2 ml de solvente
(CH_3CN/H_2O/HAc 79 + 20 + 1)

Agitación (90 min) y centrifugación
(2 min @ 3000 $Umin^{-1}$)

Dilución (0.5 ml muestra + 0.5 ml solvente)
(CH_3CN/H_2O/HAc 20 + 29 + 1)

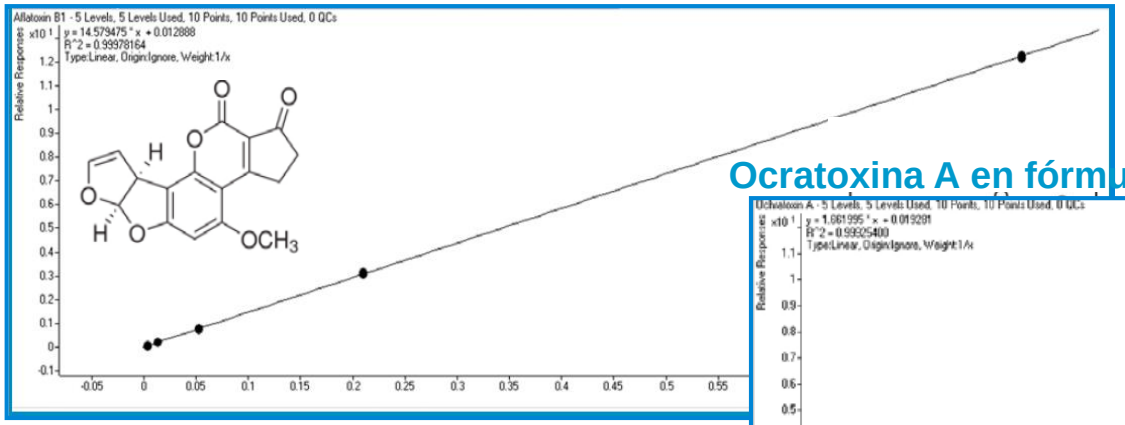
LC-MS/MS

Condiciones para Micotoxinas, transiciones MRM

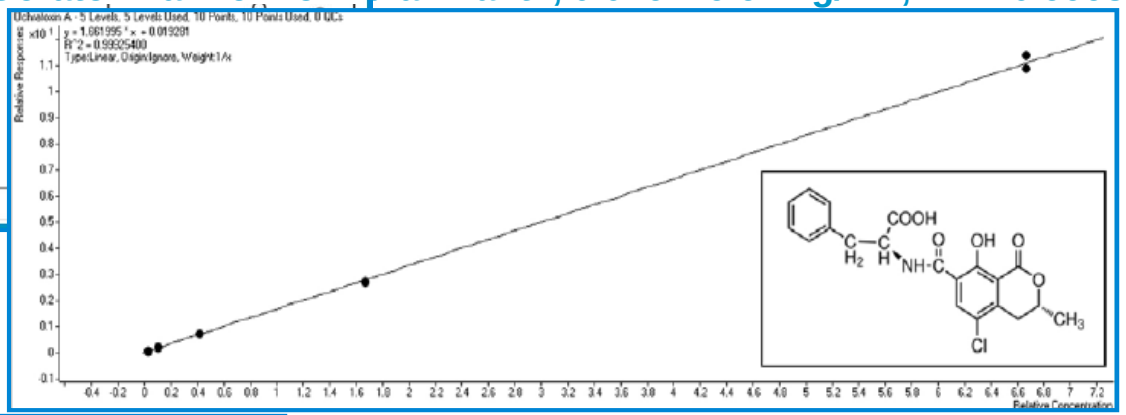
Compuesto	TS	Prec Ion	Frag (V)	Prod Ion 1	CE (V)	Prod Ion 2	CE (V)	Dwell	Polarity
Fus-X	1	413.1	95	263	10	59	28	100	Negative
NIV	1	371.1	95	281	12	59.1	32	100	Negative
DON	1	355.1	100	265.1	8	59.2	20	100	Negative
Neosolaniol	2	400.2	95	305.1	4	185.1	16	100	Positive
DAS	2	384.2	95	307.1	2	247.1	6	40	Positive
3-Acetyl DON	2	397.3	85	337.1	4	59.1	20	40	Negative
15-Acetyl DON	2	356	95	321	5	137	8	40	Positive
AFG2	2	331.1	175	313	20	245	24	40	Positive
AFG1	2	329	145	243	24	200	44	40	Positive
AFB2	2	315.1	165	287	24	259	26	40	Positive
AFB1	2	313	165	285	18	241	40	40	Positive
FB1	3	722.4	220	352.3	40	334.3	44	50	Positive
FB2	3	706.4	200	336	40	318	40	50	Positive
T-2	3	484.2	90	215.1	14	185.1	14	50	Positive
HT-2	3	447.1/442.1*	100	345.1	12	263*	4	50	Positive
OTA	3	404.1	120	358	6	239	18	50	Positive
ZON	3	317.1	170	175	30	131	28	50	Negative

Sensibilidad, Rango Dinámico Lineal y Precisión

Aflatoxina B1 en fórmula infantil, 0.003 – 0.83 ng/mL, R2 = 0.9998



Ocratoxina A en fórmula infantil, 0.026 – 6.67 ng/mL, R2 = 0.9993



Recobros promedio y desviación estándar relativa (RSDs, n = 5)

Mycotoxin	Calibration Range (ng/mL)	EU Regulated Level (µg/kg)	Fortification Level (µg/kg)	Recovery (%)	RSD (%)
Aflatoxin M1	0.003 - 0.83	0.025	0.02	99.4	5.7
Aflatoxin B1	0.003 - 0.83	0.1	0.02	86.0	2.2
Aflatoxin B2	0.003 - 0.83		0.02	90.8	3.7
Aflatoxin G1	0.003 - 0.84		0.04	99.2	12.1
Aflatoxin G2	0.003 - 0.85		0.04	94.8	8.8
Aflatoxin M2	0.003 - 0.86		0.02	82.9	7.3
Fumonisin B1	0.17 - 43.0	200 FB1+FB2	1.0	93.1	11.2
Fumonisin B2	0.17- 43.0		1.0	86.2	7.9
Deoxynivalenol	0.65 - 167	200	4.0	93.9	4.5
Ochratoxin A	0.026 - 6.67	0.5	0.16	85.8	8.3
Zearalenone	0.21 - 53.1	20	2.6	90.5	10.6
HT-2	0.67 - 169		4.0	87.8	4.8
T2	0.067 - 16.9		0.4	93.4	3.4

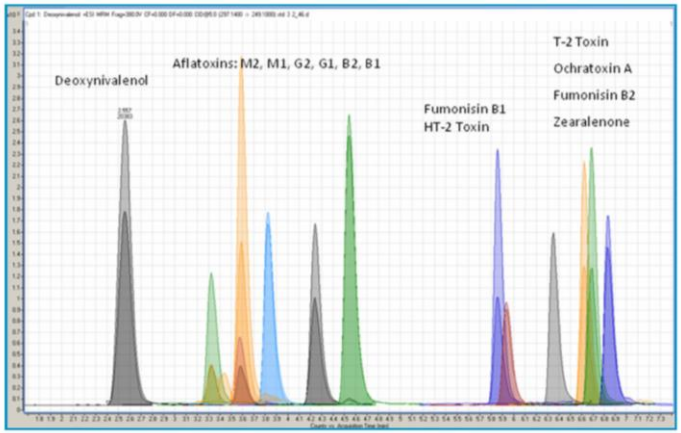
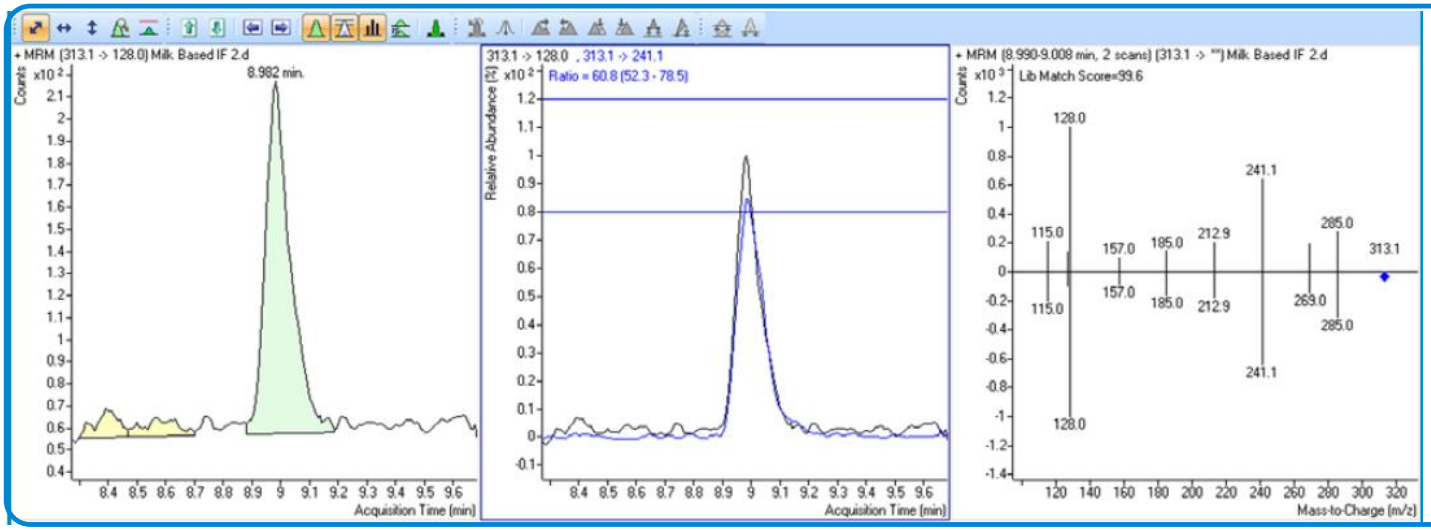


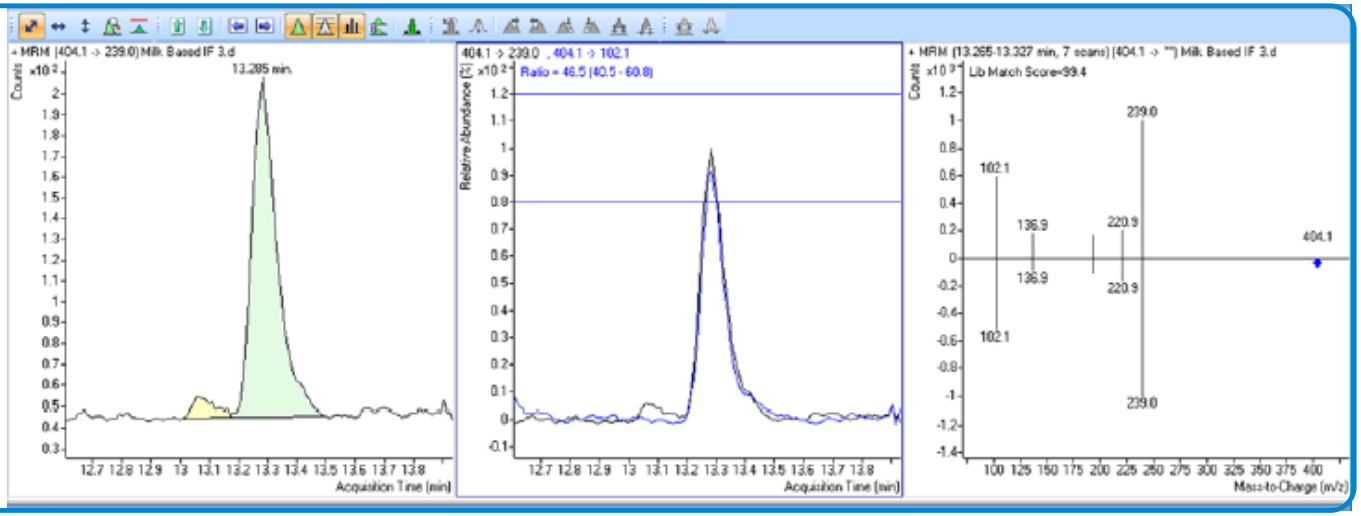
Figure 1. Extracted ion chromatogram (XIC) of a mycotoxin standard.

tMRM – Confianza en confirmación de compuestos a sub-ppb en muestras de Fórmula Infantil



Aflatoxina B1 en fórmula infantil, fortificada a 0.02 µg/kg

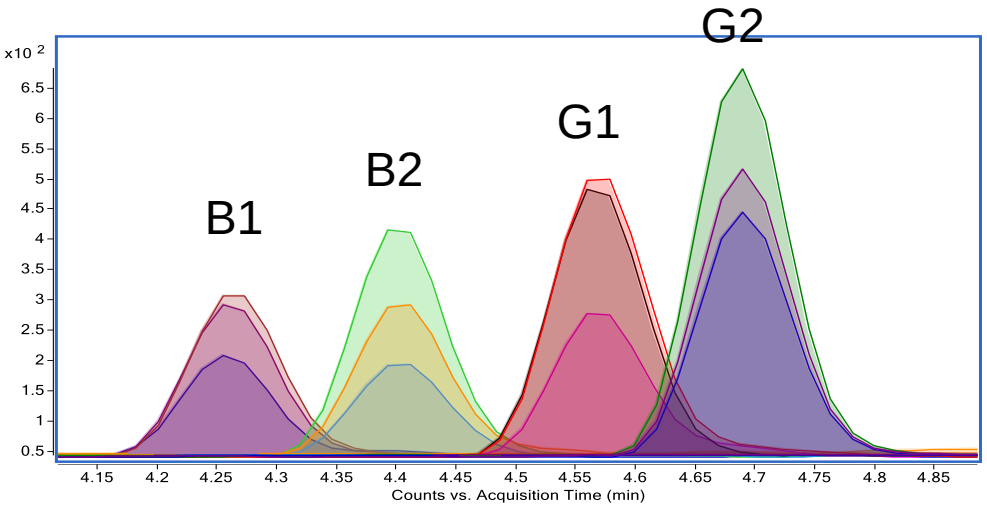
Ocratoxina A en fórmula infantil, fortificada a 0.16 µg/kg



Aflatoxinas

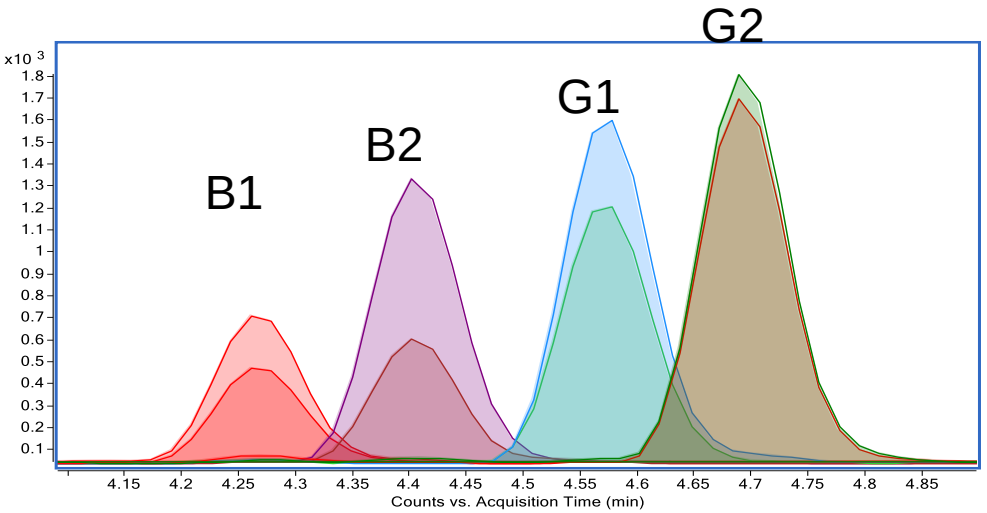
Aflatoxinas

EICs
1 Ion Cuantificador & 2 Cualificadores
(1ppb)



Aflatoxinas marcadas isotópicamente

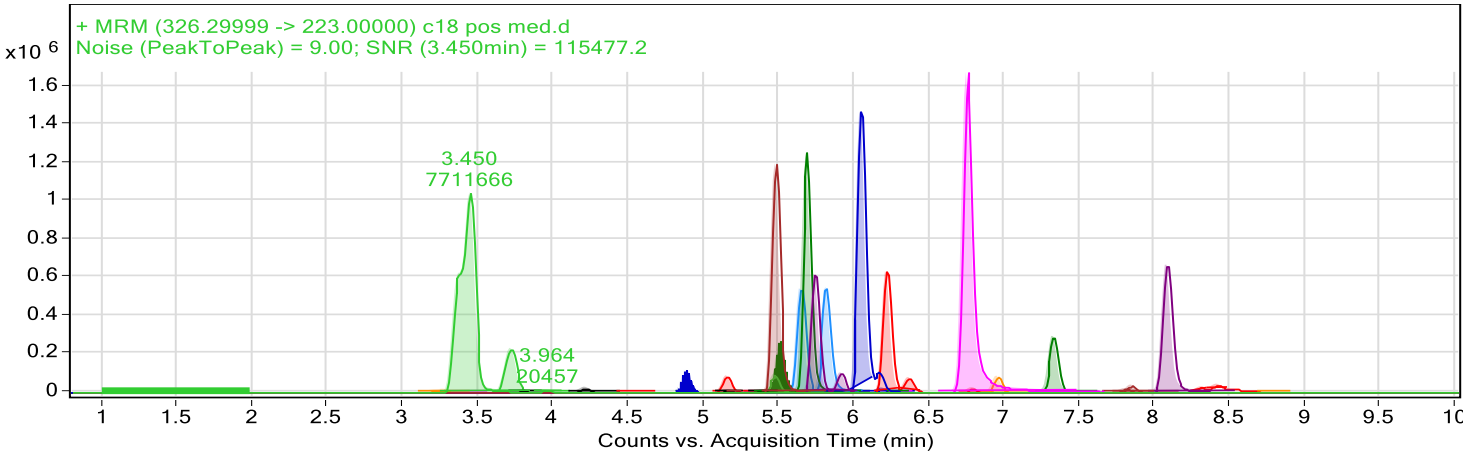
EICs
Iones Cuali y Cuantificadores
(2.5ppb)



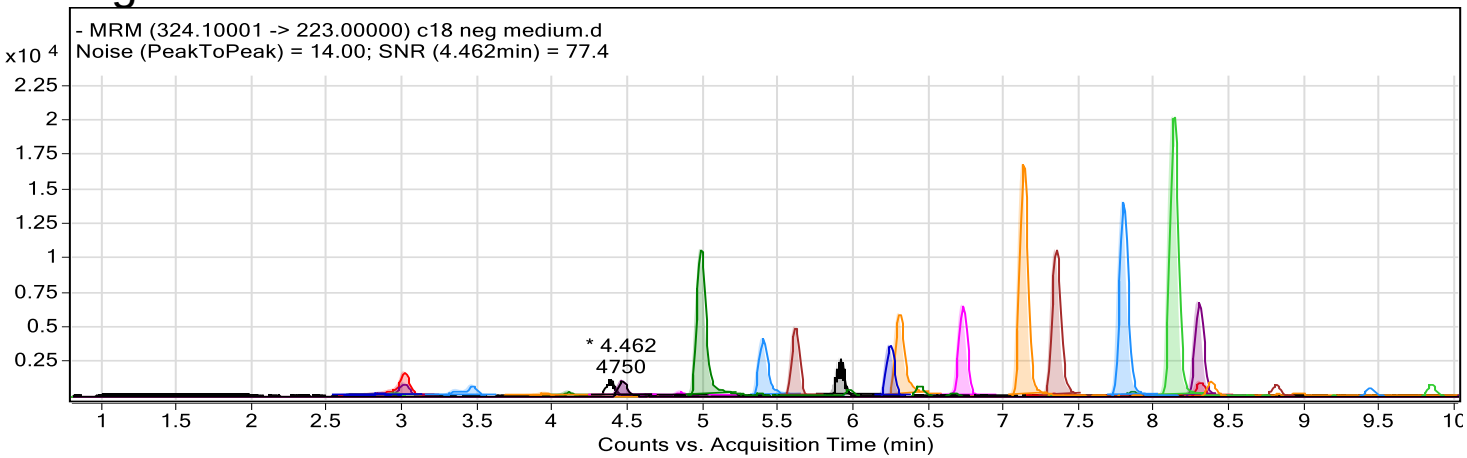
Análisis de 31 micotoxinas por LC/MS/MS modo ESI pos y neg

Condiciones: poroshell 120 2.1x50 mm 2.7 um
gradiente 5-95% ACN 12 min (0.025% TFA para ESI pos, 20 mM am Act ESI neg), 0.3 ml/min

Positive ESI MRM transitions



Negative ESI MRM transitions



Pesticidas por GC/QTOF de Alta Resolución

El Análisis de Residuos de Pesticides es un desafío

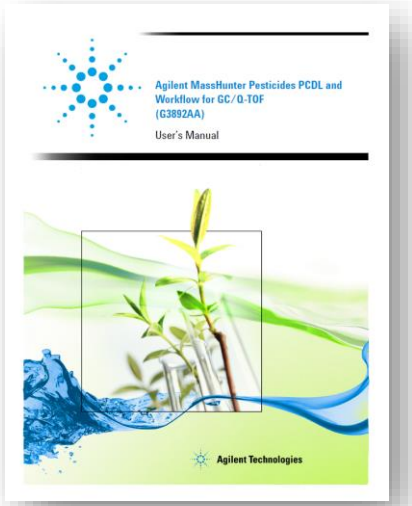
1000+ pesticides in use or remain in environment

Thousands of possible pesticide/food pairs

- Los MRLs “Default” MRLs en varias regiones del mundo es de 10 µg/kg entonces se requiere alta sensibilidad
- Los extractos son “sucios” entonces se requiere alta sensibilidad

Srceening de Pesticidas utilizando GC/Q-TOF

Agilent 7200 GC/Q-TOF



Adquisición por Full Scan
Full-Spectrum Data

Screening Cuantitativo

Screening Cualitativo

Preguntas que se pueden responder con GC/Q-TOF

Cuantos compuestos Target están presentes?

Dozens's Compounds
e.g. ~120 Targets

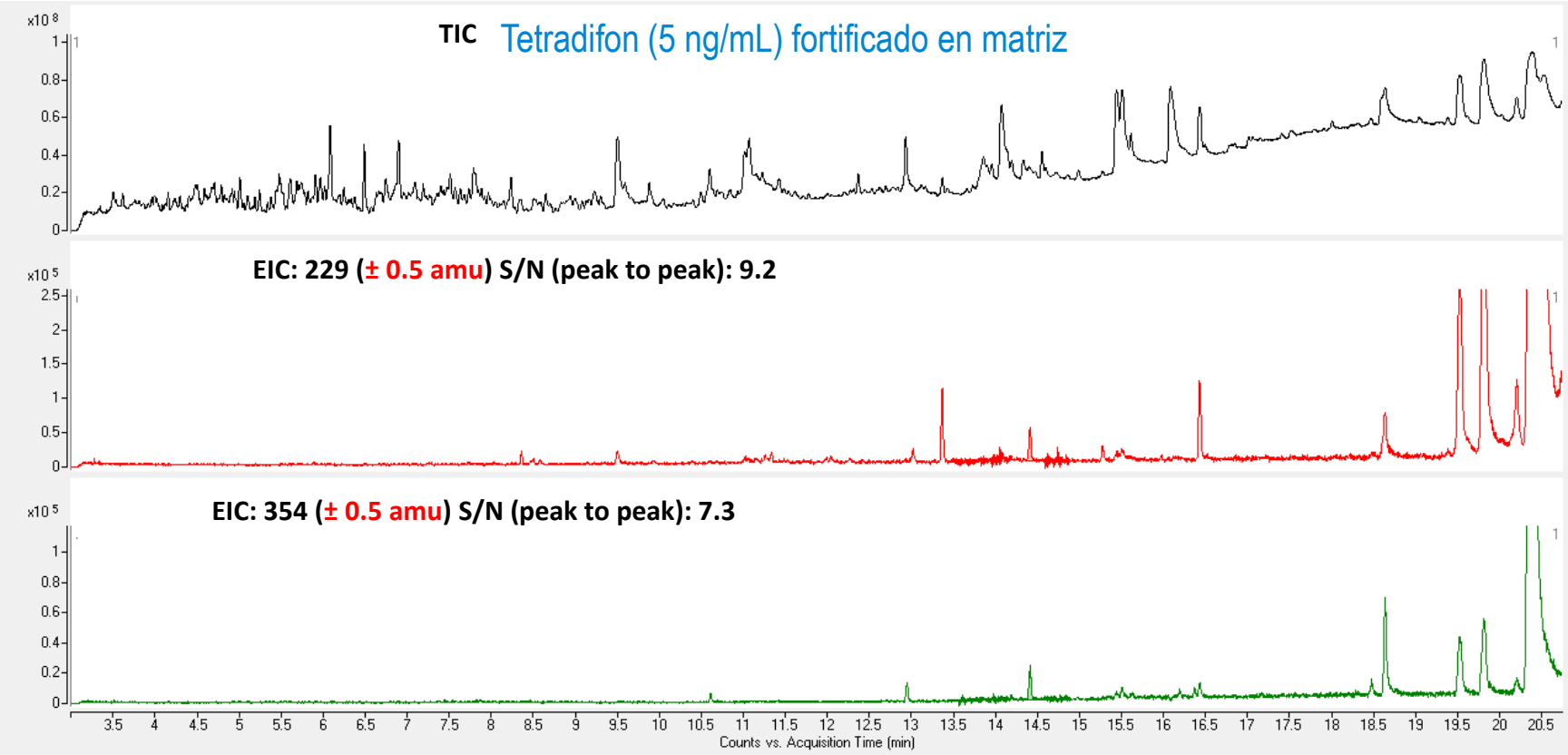
Hay presencia de algún otro residuo Target?

100's compounds
e.g. 800 Targets

Hay algo más en mi muestra?

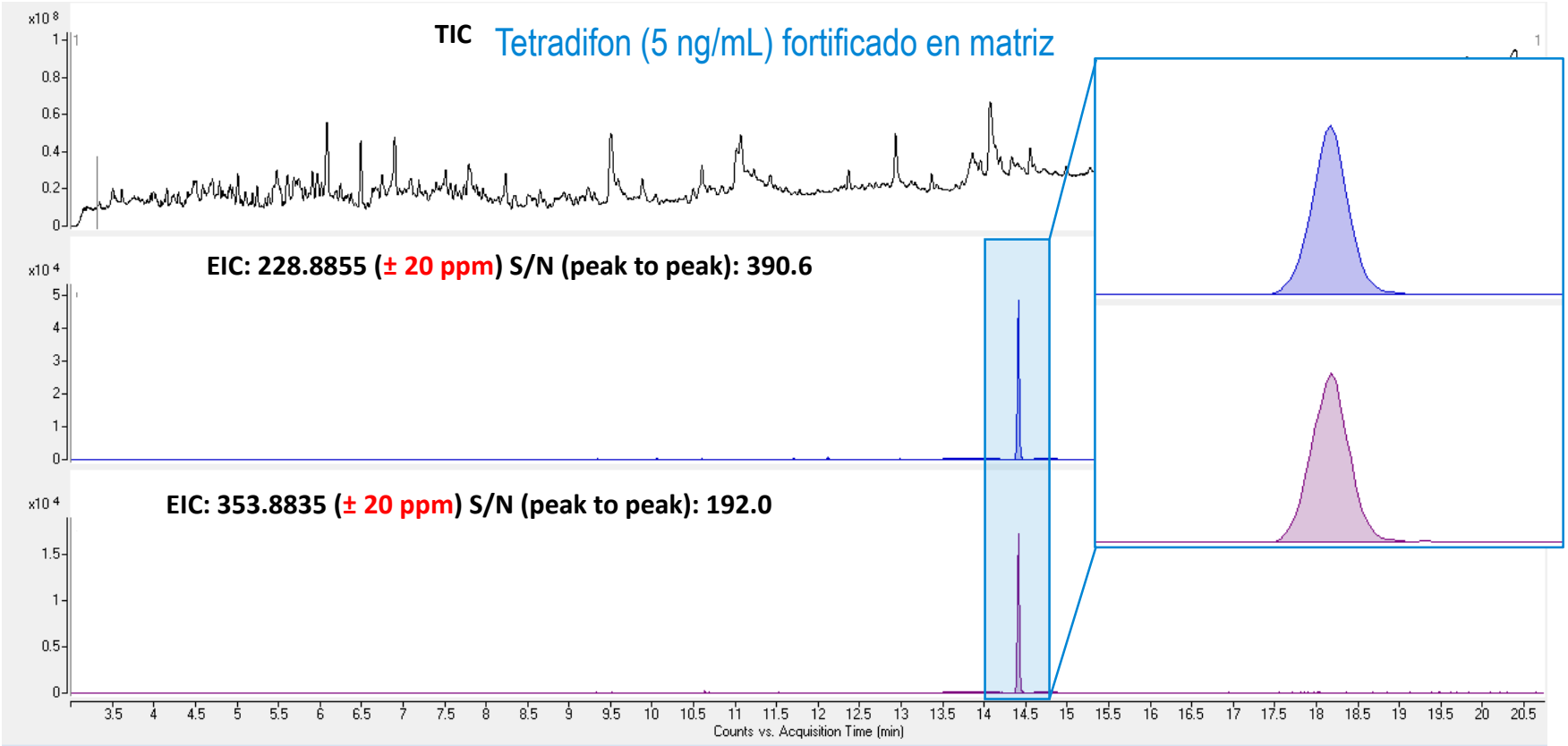
1000's compounds
e.g. 8000 Targets

Ventajas de GC/Q-TOF



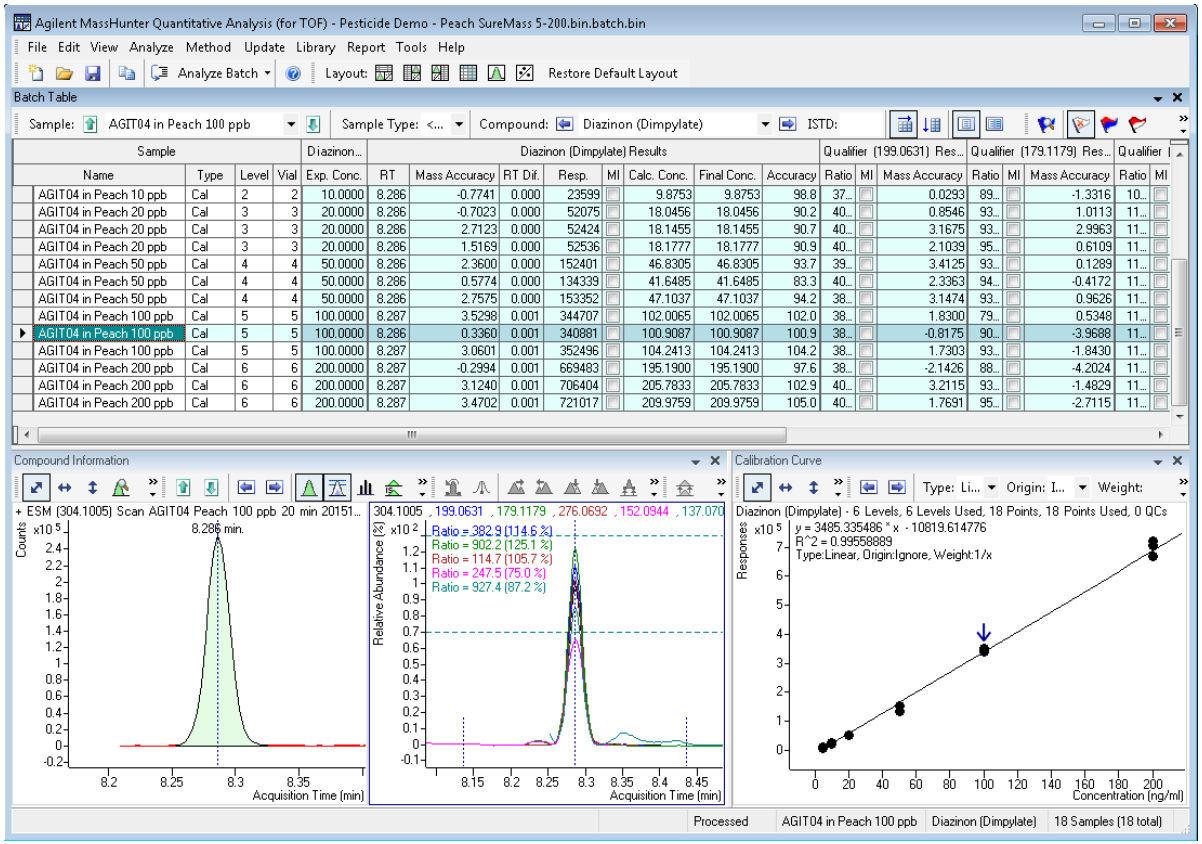
Ventajas de GC/Q-TOF

Aumento de Selectividad utilizando Masa Exacta

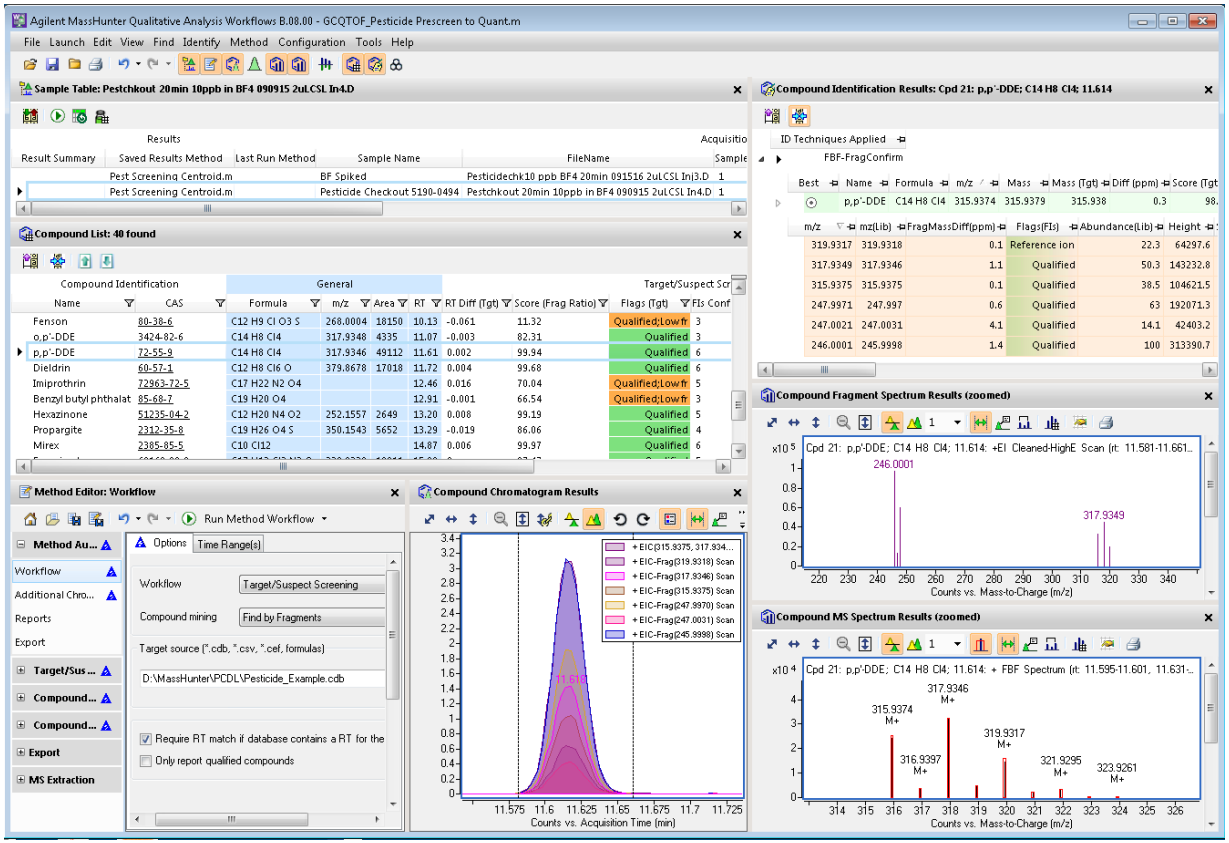


Screening Cuantitativo

Aplicar el método cuantitativo para un Screening de rutina



Screening Cualitativo



Screening Cualitativo

Qualitative Result Highlights

Screening Results of 17 representative pesticides (10 ng/mL) spiked in blend of food

Name	中文名	CAS	Formula	Identified?	RT (min)	RT Diff(min)	FIs Conf.	Qualified Ion (I)		Qualified Ion (II)	
								m/z	diff (ppm)	m/z	diff (ppm)
Dichlorvos	敌敌畏	62-73-7	C4H7Cl2O4P	Yes	4.679	**0.014**	6	219.9464	**4.7**	184.9744	**3.0**
Mevinphos	速灭磷	7786-34-7	C7H13O6P	Yes	5.610	**0.001**	4	192.0198	**0.3**	164.0233	**1.8**
Ethalfluralin	丁氟消草	55283-68-6	C13H14F3N3O4	Yes	7.139	**0.001**	5	316.0911	**2.4**	292.0548	**2.8**
Trifluralin	氟乐灵	1582-09-8	C13H16F3N3O4	Yes	7.247	**0.003**	6	306.0709	**4.2**	290.0755	**2.8**
Atrazine	莠去津	1912-24-9	C8H14ClN5	Yes	7.887	**0.000**	6	215.0932	**0.2**	202.0680	**3.8**
Chlorpyrifos-methyl	甲基毒死蜱	5598-13-0	C7H7Cl3NO3PS	Yes	9.143	**0.001**	6	285.9267	**3.9**	287.9236	**1.5**
Heptachlor	七氯	76-44-8	C10H5Cl7	Yes	9.339	**-0.006**	6	336.8496	**2.3**	271.8106	**3.7**
Malathion	马拉硫磷	121-75-5	C10H19O6PS2	Yes	9.729	**-0.002**	5	124.9824	**2.6**	99.0077	**2.4**
DDE, *p,p'*-	滴滴伊, *p,p'*-	72-55-9	C14H8Cl4	Yes	11.612	**0.002**	6	315.9375	**0.1**	317.9349	**1.1**
Dieldrin	狄氏剂	60-57-1	C12H8Cl6O	Yes	11.717	**0.004**	6	276.8722	**0.5**	260.8595	**0.6**
Hexazinone	环嗪酮	51235-04-2	C12H20N4O2	Yes	13.195	**0.008**	5	171.0877	**0.5**	172.0896	**4.8**
Propargite	克螨特	2312-35-8	C19H26O4S	Yes	13.318	**-0.019**	4	173.0955	**3.6**	136.0835	**2.4**
Mirex	灭蚁灵	2385-85-5	C10Cl12	Yes	14.874	**0.006**	6	269.8127	**0.6**	236.8409	**0.6**
Fenarimol	氯苯嘧啶醇	60168-88-9	C17H12Cl2N2O	Yes	15.084	**0.000**	5	330.0339	**5.5**	219.0317	**1.2**
Coumaphos	蝇毒磷	56-72-4	C14H16ClO5PS	Yes	15.853	**0.013**	5	362.0139	**0.1**	333.9822	**1.3**
Etofenprox	醚菊酯	80844-07-1	C25H28O3	Yes	16.777	**0.015**	3	163.1124	**4.2**	164.1155	**0.8**
Deltamethrin	溴氰菊酯	52918-63-5	C22H19Br2NO3	Yes	18.117	**0.018**	3	252.9053	**3.3**	250.9056	**3.9**

Screening Cualitativo

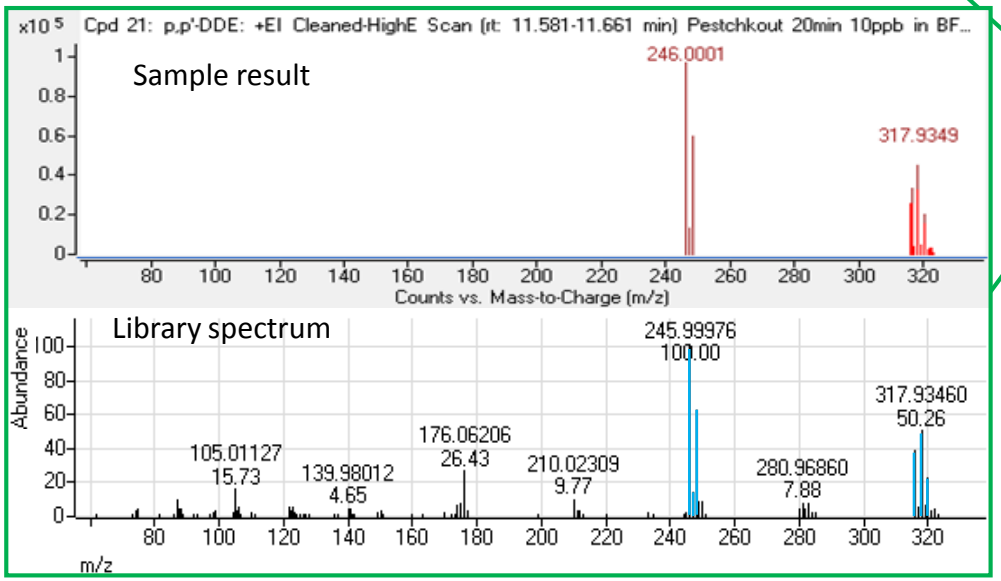
Confianza en la Identificación – Compuestos Desconocido

p, p'-DDE (10 ng/mL Spiked)

Compound Identification		General			Target Screening	
Name	CAS	Formula	RT	RT Diff (Tgt)	Score (Frag Ratio)	Flags (Tgt)
o,p'-DDE	3424-82-6	C14 H8 Cl4	11.074	-0.003	82.31	Qualified
p,p'-DDE	72-55-9	C14 H8 Cl4	11.614	0.002	99.94	Qualified
Dieldrin	60-57-1	C12 H8 Cl6 O	11.721	0.004	99.68	Qualified
Imiprothrin	72963-72-5	C17 H22 N2 O4	12.46	0.016	70.04	Qualified;Low fra
Benzyl butyl phthalate	85-68-7	C19 H20 O4	12.918	-0.001	66.54	Qualified;Low fra
Hexazinone	51235-04-2	C12 H20 N4 O2	13.203	0.008	99.19	Qualified
Propargite	2312-35-8	C19 H26 O4 S	13.299	-0.019	86.06	Qualified
Mirex	2385-85-5	C10 Cl12	14.878	0.006	99.97	Qualified
Fenarimol	60168-88-9	C17 H12 Cl2 N2 O	15.084	0	87.47	Qualified

Best	Name	Formula	m/z	Mass	Mass (Tgt)	Diff (ppm)
◉	p,p'-DDE	C14 H8 Cl4	315.9374	315.9379	315.938	0.3

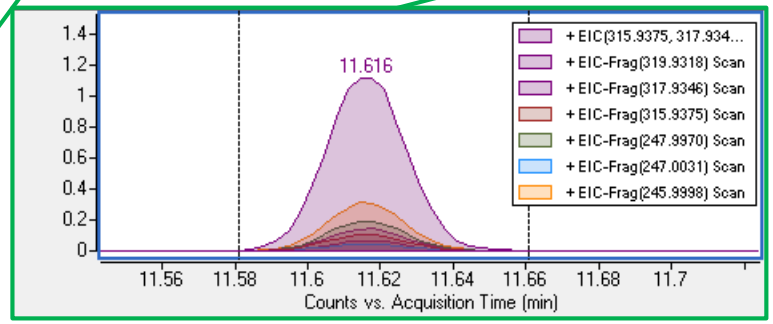
m/z	mz(Lib)	FragMassDiff(ppm)	Flags(FIs)	Abundance(Lib)
319.9317	319.9318	0.1	Reference ion	22.3
317.9349	317.9346	1.1	Qualified	50.3
315.9375	315.9375	0.1	Qualified	38.5
247.9971	247.997	0.6	Qualified	63
247.0021	247.0031	4.1	Qualified	14.1
246.0001	245.9998	1.4	Qualified	100



✓ Excellent mass error

✓ High fragment ratio score

✓ Good EIC Alignment



ANALISIS DE ACRILAMIDA Y SUS POSIBLES PRECURSORES

En colaboración con PEPSICO

Product Range

Fun-for-You Portfolio

These products are part of PepsiCo's core food and beverage businesses.

Pepsi: The bold, refreshing, robust cola

Red Rock Deli Potato Chips: Seasoned with delicious deli-inspired flavors

Better-for-You Portfolio

These are foods and beverages that have levels of total fat, saturated fat, sodium and/or added sugar that are in line with global dietary intake recommendations. Included in this category are products such as baked snacks with lower-fat content and beverages with fewer or no calories and less added sugar.

Baked! Lay's: Baked potato crisps with zero trans fats

Propel Zero: Zero-calorie enhanced water beverage with antioxidant and other vitamins

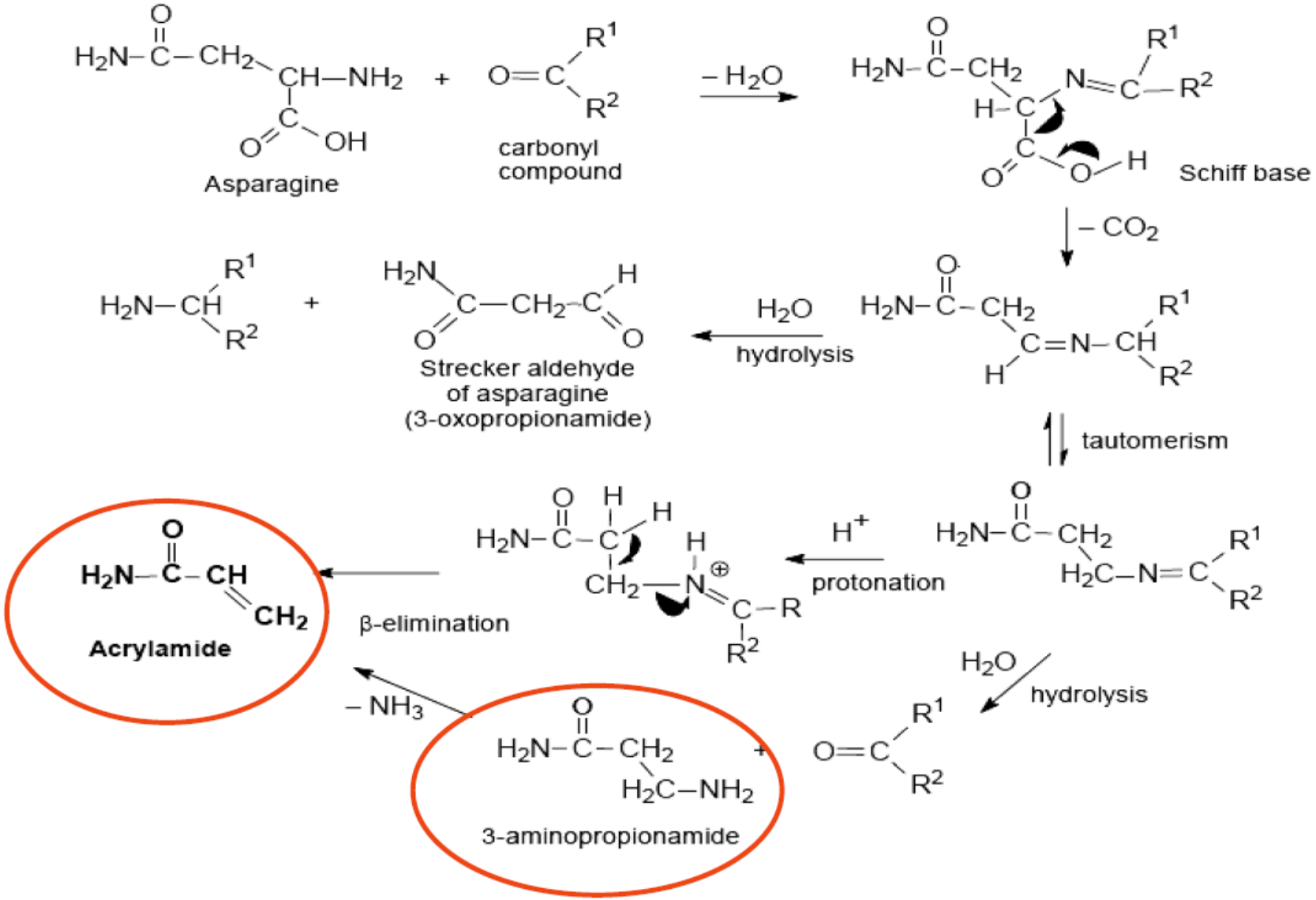
Good-for-You Portfolio

These are foods and beverages that deliver positive nutrition through the inclusion of whole grains, fruits, vegetables, low-fat dairy, nuts and seeds or significant amounts of important nutrients, while moderating total fat, saturated fat, sodium and/or added sugar. We also include products that have been specifically formulated to provide a functional benefit, such as addressing the performance needs of athletes.

Quaker Instant Oatmeal: Made with heart-healthy whole-grain oats

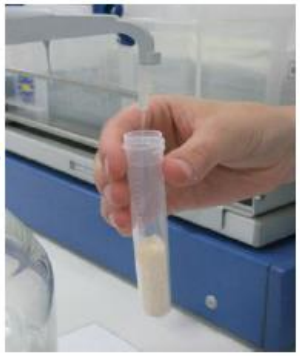
Naked Juice: 100 percent juice smoothie made from real fruit

Acrylamide (AA) Formation

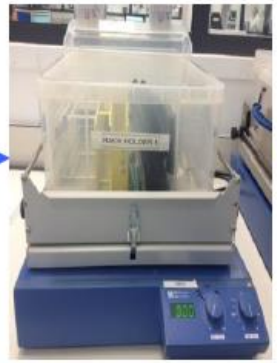


US FDA LCMSMS Acrylamide Draft Method – Dual Cartridge Approach

Weigh 1g of homogenous sample



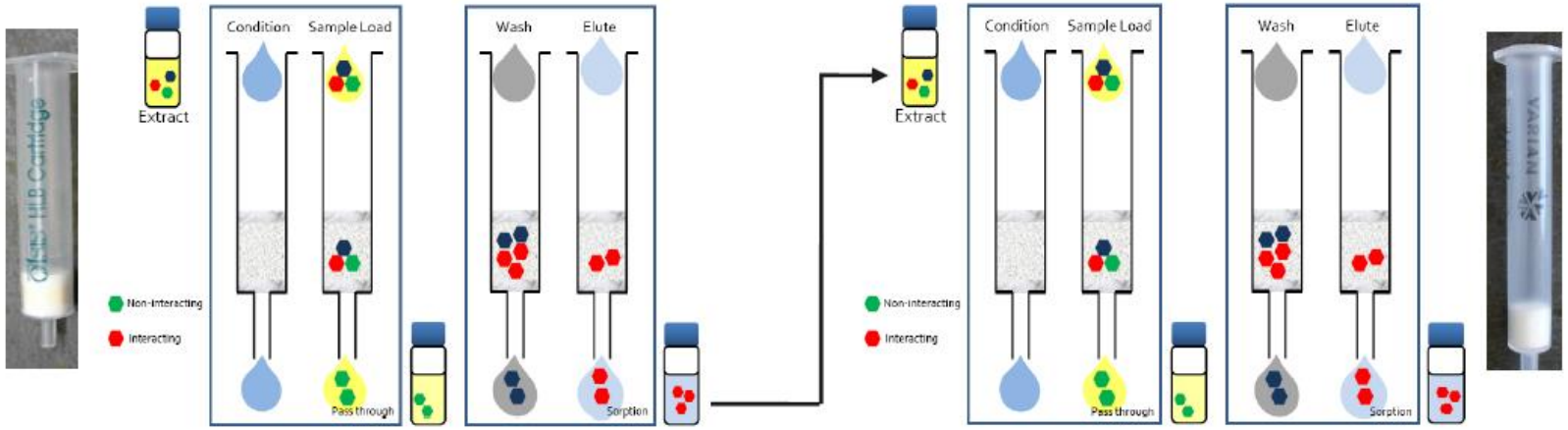
Add 10 ml Water with I.S.



Shake for 20mins

Centrifuge for 20mins

Dual cartridge clean up

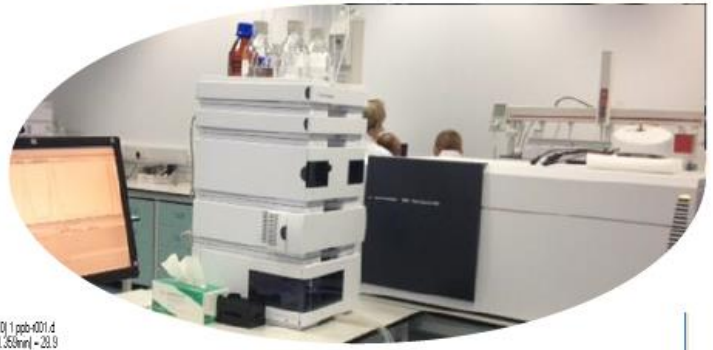


Stage 1
Removal of hydrophobic components within the solution

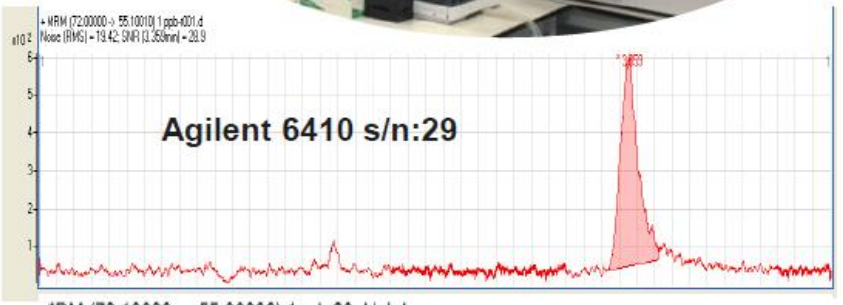
Stage 2
Removal of ionic components within the solution

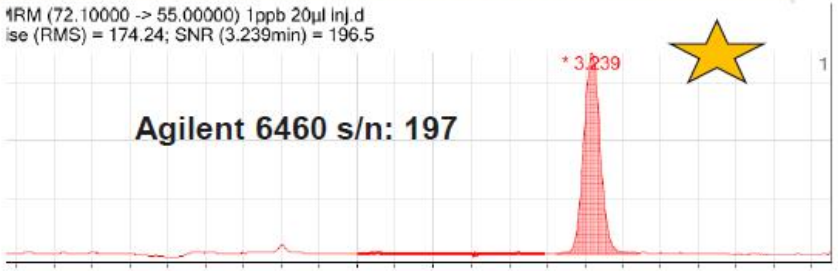
Acrylamide LCMSMS Analysis

Mobile phase: water: methanol: formic acid : 900:100:1.0 (v:v:v)
Flow rate: 0.25 ml/min
Run Time: 6 min
Injection volume: 5 µL
Temperature: 60°C
Column: Hypercarb, 100 mm x 3 mm, 5 µm particle size



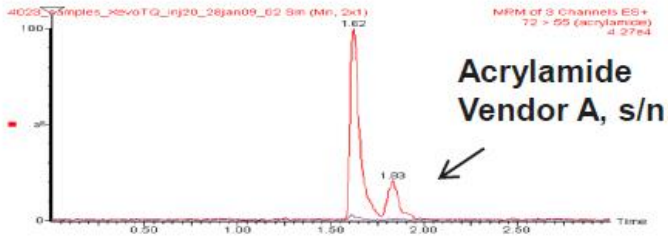
6460 Parameters:
Gas temperature: 350°C
Gas flow: 12 l/min
Nebuliser: 60 psi
Sheath gas temperature: 350°C
Sheath gas flow: 12 l/min
Capillary voltage: 2500 V
Nozzle voltage: 1000V

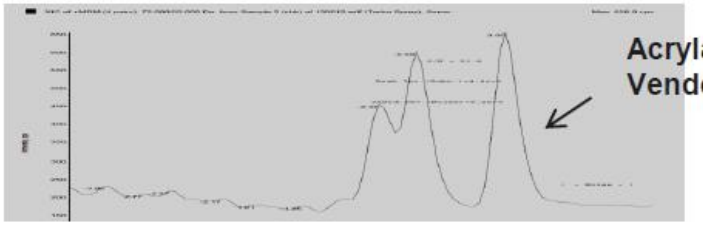




Acquisition parameters:

Transitions	Fragmentor	Collision energy
72 ⇒ 55	80	5
72 ⇒ 44	80	5
75 ⇒ 58	80	5





Gracias por atender este seminario!

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