



# Analysis of Dioxins and Dioxin-like PCBs in Feed and Food

---

**Thierry Faye, Agilent Technologies**

**Jef Focant, University of Liege, Belgium**





# GC/MS/MS Analyzer for Analysis of Dioxins and Dioxin-like PCBs in Feed and Food

Thierry Faye

Market Development Manager

Agilent Technologies



Agilent Technologies

# NEW Introducing the Agilent GC/MS/MS Dioxin Analyzer

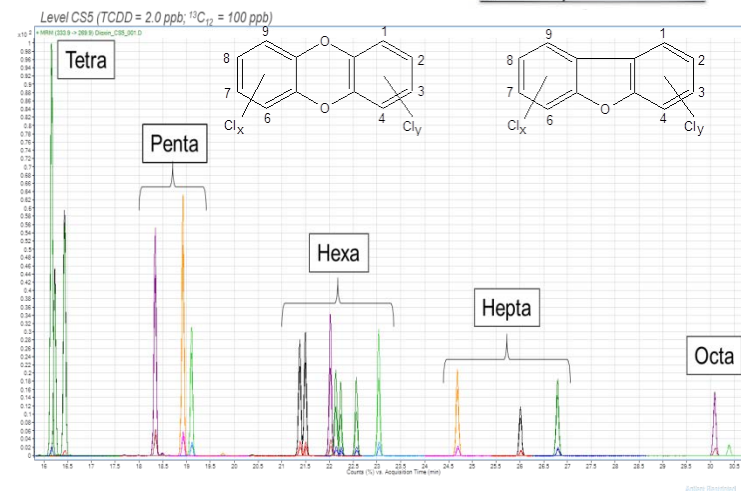
⇒ Cutting edge technology enables fast & reliable Dioxin analysis at low levels:

- New standard for GC/MS/MS sensitivity with 7010 Triple Quadrupole EI source = An instrument with sensitivity of GC Sector MS.
- MultiMode Inlet (MMI) for effective cold split less injections and more.



⇒ Developed from successful collaborations with leading Dioxin Labs in Europe:

- The Agilent platform already validated according to new regulations in Europe for both food and feed (EC 589/2014, 709/2014).
- Refer to L'Homme/Focant's publication: ***Journal of Chromatography A*, 1376 (2015) 149–158.**
- Custom reporting with complete calculations have been developed and automated in MassHunter.



⇒ Ready for analysis:

- Pre-configured and pre-tested at our factory so installation in your lab is fast and efficient.
- **The RTL advantage:** guarantees the exact matching of our reference method on a new instrument.
- A service engineer runs a complete check out standard so validation can begin.
- Method never needs altering even when column maintenance is performed.

# Validation of GC/MS/MS confirmatory method for the European official control of levels of dioxins, furans, and dioxin-like PCBs in foodstuffs

L'Homme B., Focant J.-F.



Organic and Biological Anal. Chem.  
University of Liege, Belgium





■ Dioxins

Number of papers and reviews (Scopus)

1000  
800  
600  
400  
200



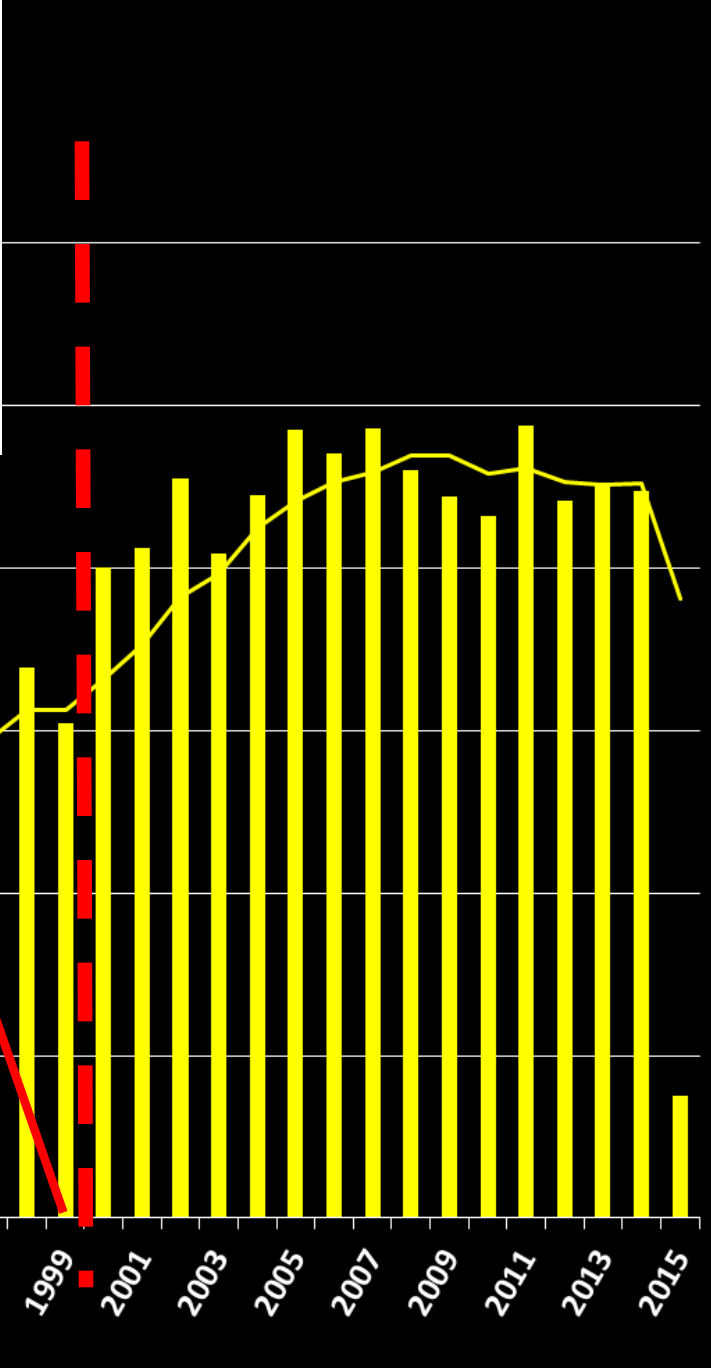
**Yu-Chen**



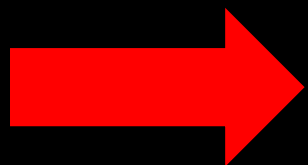
**1968**  
**Yusho**

**1957**

1977 1979 1981 1983 1985 1987 1989 1991 1993 1995 1997 1999 2001 2003 2005 2007 2009 2011 2013 2015



# 95% Exposure by Food Consumption



**Food and Feed control...**



**Dioxin EU Regulation for  
food-feed ‘started’ with the  
Belgian Dioxin Crisis in  
1999...**

# EU Commission Documents

- Council Regulation 2001/102/EC
- Council Regulation (EC) No 2375/2001
- Commission Directive 2002/69/EC
- Commission Directive 2002/70/EC
- Commission Directive 2006/13/EC
- Commission Recommendation 2006/350/EC
- Commission Regulation (EC) No 593/2006
- Commission Regulation (EC) No 1881/2006
- Commission Regulation (EC) No 1883/2006
- Commission Regulation (EC) No 152/2009
- Commission Regulation (EU) No 1259/2011
- Commission Recommendation 2011/516/EU
- Commission Regulation (EC) No 252/2012
- Commission Regulation (EC) No 277/2012
- Commission Regulation (EC) No 278/2012
- **Commission Regulation (EC) No 589/2014**
- **Commission Regulation (EC) No 709/2014**

<http://eur-lex.europa.eu>



# EU Commission Strategy (food-feed)

- ✓ Continuous monitoring
- ✓ Maximum-Action-(Target) level strategy
- ✓ Screening-Confirmatory approach
- ✓ RASFF (high capacities)

 Based on state-of-the-art methods

# **Analytical Methods ?**

# Evolutionary Guidelines

## HARMONISED QUALITY CRITERIA FOR CHEMICAL AND BIOASSAYS ANALYSES OF PCDDs/PCDFs IN FEED AND FOOD

### PART 1: GENERAL CONSIDERATIONS, GC/MS METHODS

Rainer Malisch<sup>1</sup>, Bert Baumann<sup>2</sup>, Peter A. Behnisch<sup>3</sup>, Richard Canady<sup>4</sup>, Daniel Fraisse<sup>5</sup>, Peter Fürst<sup>6</sup>, Douglas Hayward<sup>4</sup>, Ronald Hoogenboom<sup>7</sup>, Ronald Hoogerbrugge<sup>2</sup>, Djien Liem<sup>2</sup>, Olaf Pöpke<sup>8</sup>, Wim Traag<sup>7</sup>, Thomas Wiesmüller<sup>9</sup>

OHC 50 (2001) 53

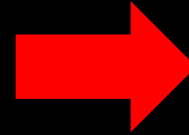
## HARMONISED QUALITY CRITERIA FOR CHEMICAL AND BIOASSAYS ANALYSES OF PCDDs/PCDFs IN FEED AND FOOD

### PART 2: GENERAL CONSIDERATIONS, BIOASSAY METHODS

Peter A. Behnisch<sup>1</sup>, Randy Allen<sup>2</sup>, Jack Anderson<sup>3</sup>, Abraham Brouwer<sup>4</sup>, David J. Brown<sup>5</sup>, T. Colin Campbell<sup>6</sup>, Leo Goeyens<sup>7</sup>, Robert O. Harrison<sup>8</sup>, Ron Hoogenboom<sup>9</sup>, Ilse Van Overmeire<sup>7</sup>, Wim Traag<sup>7</sup> and Rainer Malisch<sup>10</sup>

OHC 50 (2001) 59

[www.dioxin20xx.org](http://www.dioxin20xx.org)



# PBMS



- GC-IDHRMS vs CALUX
- ISO17025
- Validation @ LOQs  
(@ 1/5<sup>th</sup> level of interest)
- ≠Upper-Lowerbound < 20%  
@ 1pg TEQ/g fat level
- Z-scores @ PTs
- Recovery rates...

# Recent EN 16215

NEN-EN 16215:2012

EUROPEAN STANDARD

**EN 16215**

NORME EUROPÉENNE

EUROPÄISCHE NORM

April 2012

---

ICS 65.120

**PBMS philosophy**

English Version

**Animal feeding stuffs - Determination of dioxins and dioxin-like  
PCBs by GC/HRMS and of indicator PCBs by GC/HRMS**

Aliments des animaux - Dosage des dioxines, des PCB de  
type dioxine et des PCB indicateurs par GC/HRMS

Futtermittel - Bestimmung von Dioxinen und dioxin-  
ähnlichen PCBs mittels GC/HRMS und von Indikator-PCBs  
mittels GC/HRMS

This European Standard was approved by CEN on 9 March 2012.

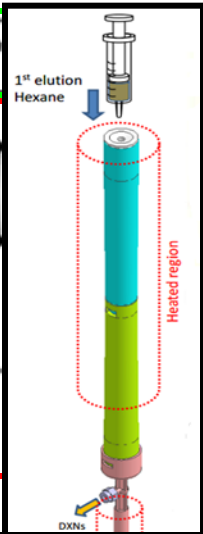
# 9.1 General Xtraction

9.2.1 Feed  
10 g

10.2  
B I- PFE



# Clean-up & Fractionation



# Analysis



**EU Com. Reg. 589 & 709**

**GC-QQQ**

**EU Com. Reg. 589 & 709**



# New EU Regulation

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)

27.6.2014

EN

Official Journal of the European Union

L 188/1

## COMMISSION REGULATION (EU) No 709/2014

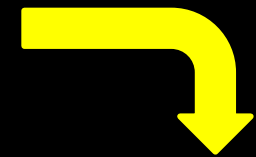
of 20 June 2014

amending Regulation (EC) No 152/2009 as regards the determination of the levels of dioxins and polychlorinated biphenyls

(Text with EEA relevance)

- (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.

<i>Criteria</i>	<b>PCDD/Fs and DL-PCBs GC-MS/MS (589/2014)</b>	<b>NDL-PCBs GC-MS/MS (589/2014)</b>
Detectable quantity	-PCDD/F upper femtogram (10 <sup>-15</sup> g) -NO-PCB low picogram (10 <sup>-12</sup> g) -MO-PCB nanogram (10 <sup>-9</sup> g)	NDL-PCB nanogram (10 <sup>-9</sup> g)
Selectivity	-Chromatographic separation of 1,2,3,4,7,8-HxCDF and 1,2,3,6,7,8-HxCDF <25% valley peak to peak	Relative RT ±0.25% IS vs analyte
MRM transitions	-Monitoring 2 specific precursors with each specific product ion transition for all labeled and unlabeled analytes -Relative ion intensities max ±15% -Resolution MS quadrupoles = unit	-Monitoring at least 1 precursor ion and 2 product ions -Tolerance ratio ±20% if rel. intens. >50% Tolerance ratio ±25% if rel. intens. 20-50% -Resolution MS quadrupoles = unit
Blank	-Used for LOQ calculation	-Used for LOQ calculation -Blank value <30% of maximum level ML
iLOQ	-iLOQ calculated from lowest cali. point -lowest concentration point on cali. must give acceptable and consistent deviation to the average RRF -Average RRF calculated for all points -Deviation to average RRF <30%	-ditto
LOQ	-LOQ calculated from average blank level -LOQ < 1/5 of maximum level ML -Difference ub and lb levels <20%ML	-ditto -Diff. ub and lb for sum ind-PCB @ ML <20%
Accuracy Reproducibility	-Demonstrate performances at 0.5ML, ML, 2ML -Trueness (accuracy) ±20% -Within-lab reproducibility (RSD) <15%	-Demonstrate performances at 0.5ML, ML, 2ML -Trueness for sum ind-PCB @ ML ±30% -Within-lab reproducibility (RSD) <20%
Control	-QC chart for blanks -QC charts control sample	-QC chart for blanks -QC charts control sample
Recovery	-Individual internal std in range 60-120% -Out of range OK if contribu. to TEQ<10%	-Individual internal std in range 50-120% -Out of range OK if contribu. to sum ind-PCB<10%
Measurement uncertainty	-Expanded measurement uncertainty -Coverage factor = 2 (CL=95%) -If separate determination of congeners, make sum of separate uncertainty for sum of PCDD/F and DL-PCBs	-Expanded measurement uncertainty -Coverage factor = 2 (CL=95%)

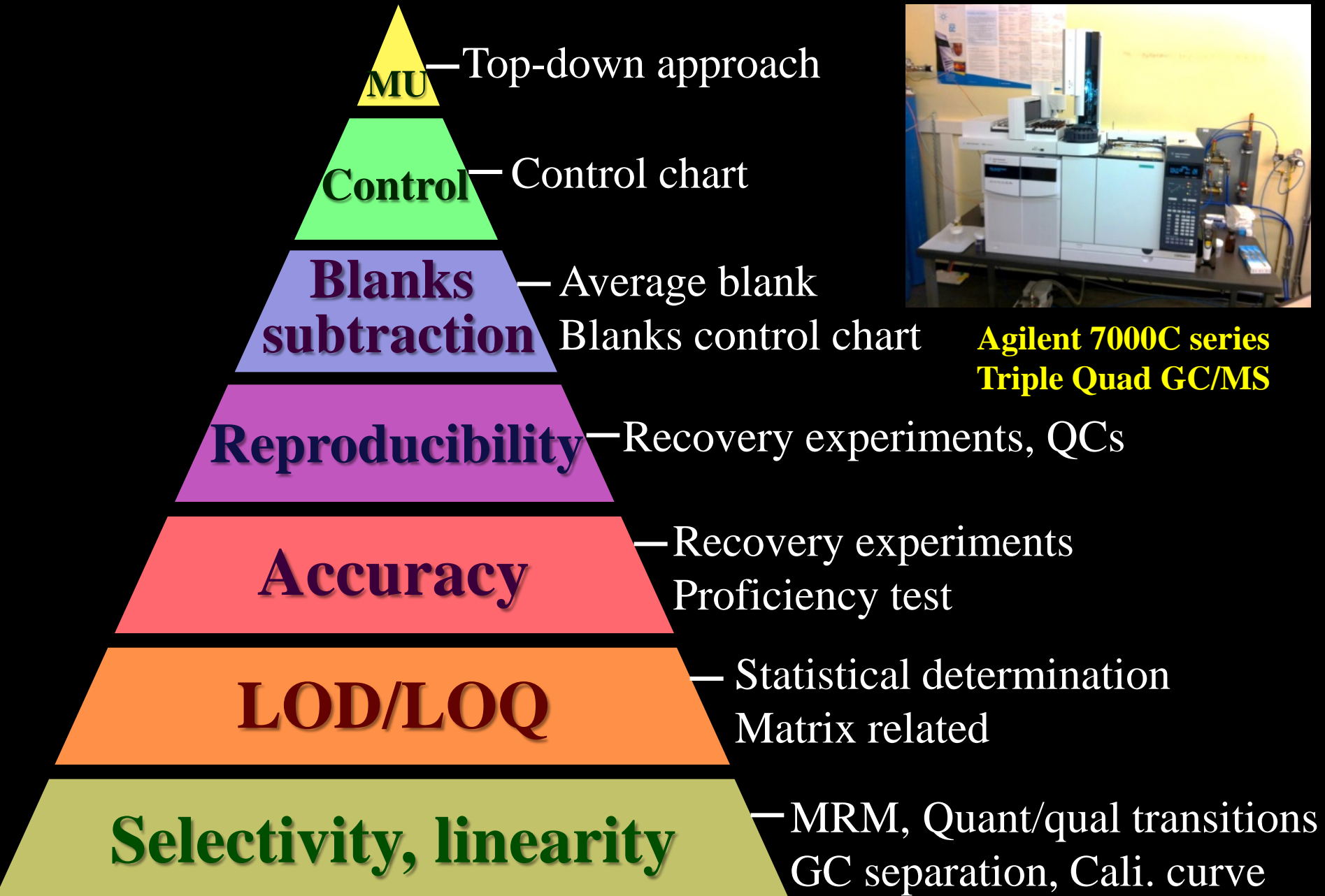


**From Regulation**

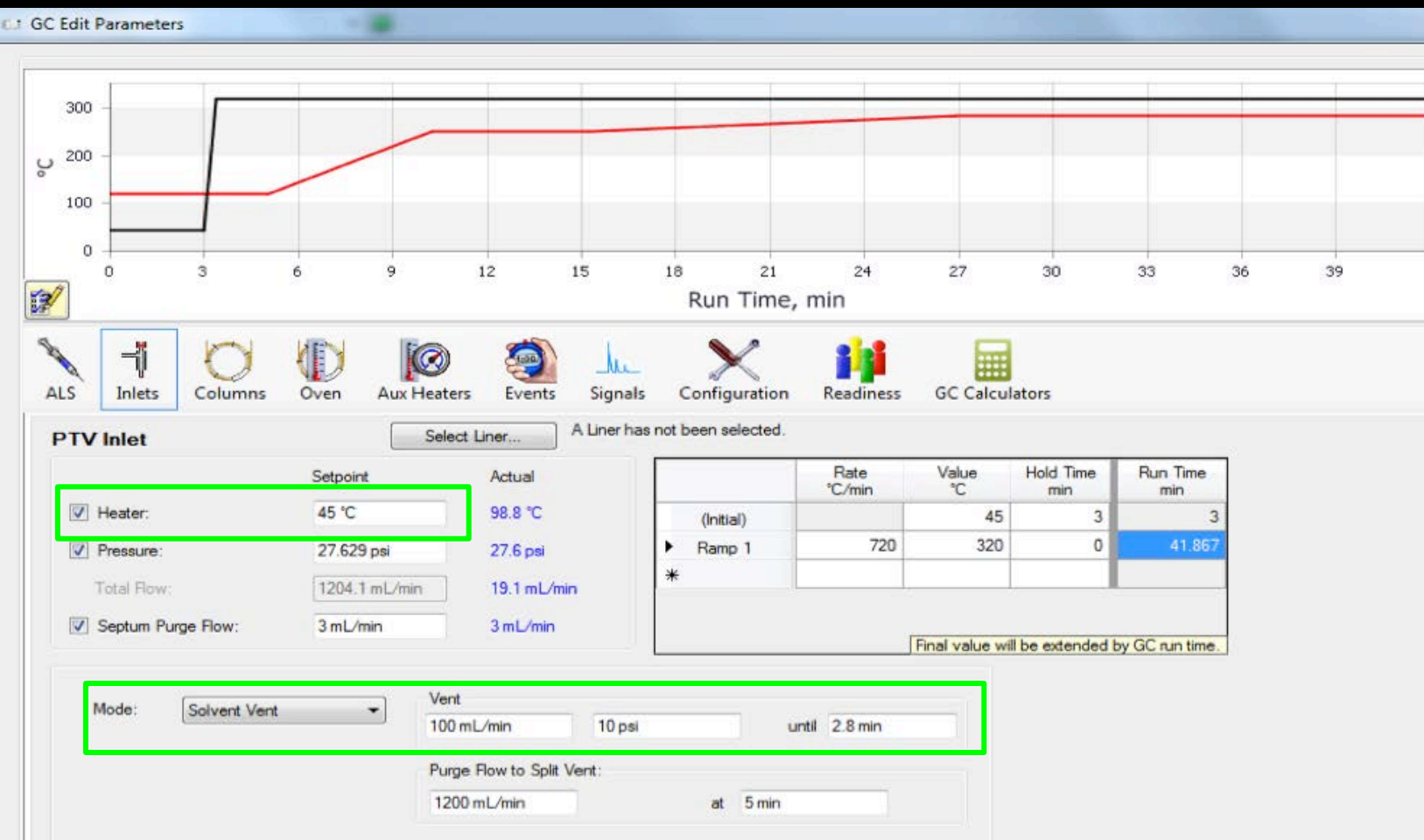


**Full validation**

# Validation for vegetable oil (feed)



# PTV Injections

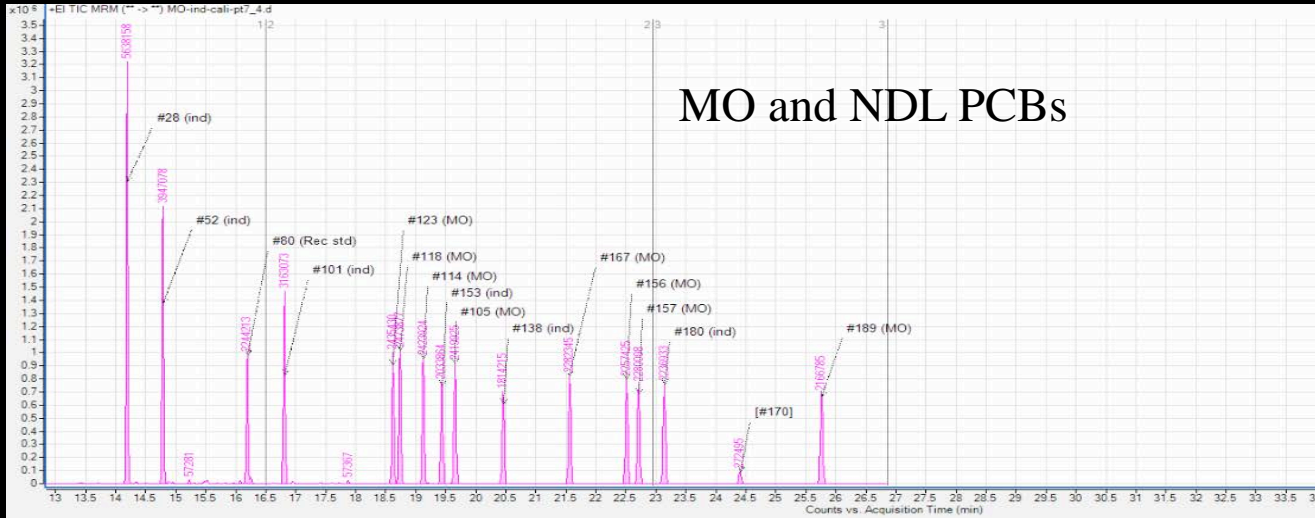


Dioxin fraction runtime = 42min

MO-PCBs fraction runtime = 28min

# Chromatographic Profile

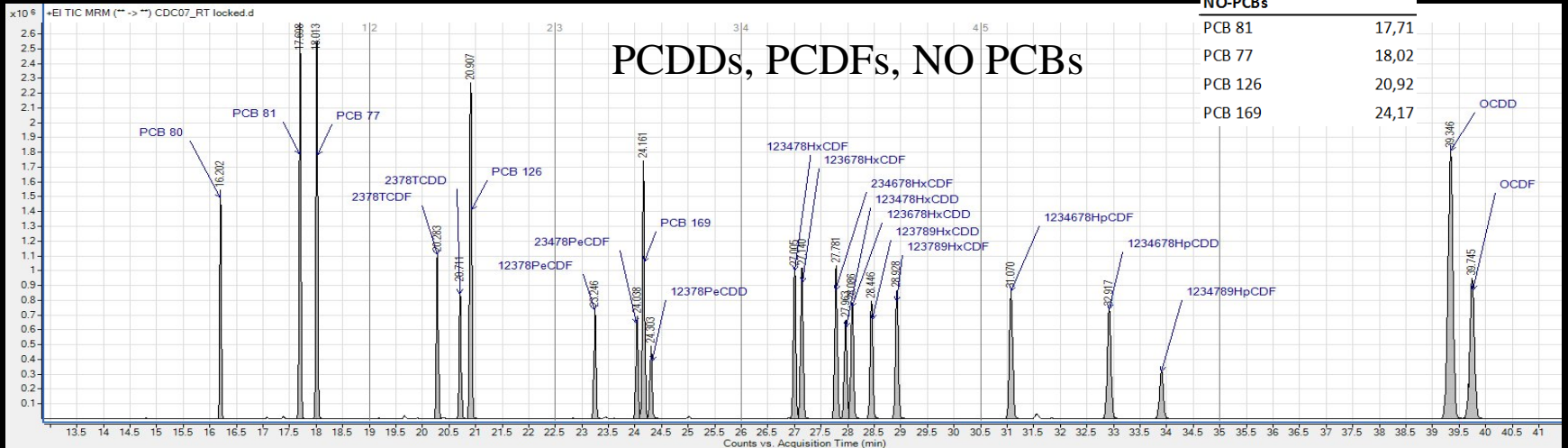
## MO and NDL PCBs



PCDDs	RT
2378-TCDD	20,72
12378-PeCDD	24,31
123478-HxCDD	27,97
123678-HxCDD	28,10
123789-HxCDD	28,46
1234678-HpCDD	32,93
OCDD	39,35
PCDFs	
2378-TCDF	20,30
12378-PeCDF	23,26
23478-PeCDF	24,06
123478-HxCDF	27,02
123678-HxCDF	27,15
234678-HxCDF	27,79
123789-HxCDF	28,94
1234678-HpCDF	31,09
1234789-HpCDF	33,92
OCDF	39,76

MO-PCBs	RT
PCB-123	18,62
PCB-118	18,74
PCB-114	19,12
PCB-105	19,66
PCB-167	21,56
PCB-156	22,51
PCB-157	22,71
PCB-189	25,76
NDL-PCBs	
PCB-28	14,19
PCB-52	14,79
PCB-101	16,81
PCB-153	19,43
PCB-138	20,46
PCB-180	23,14

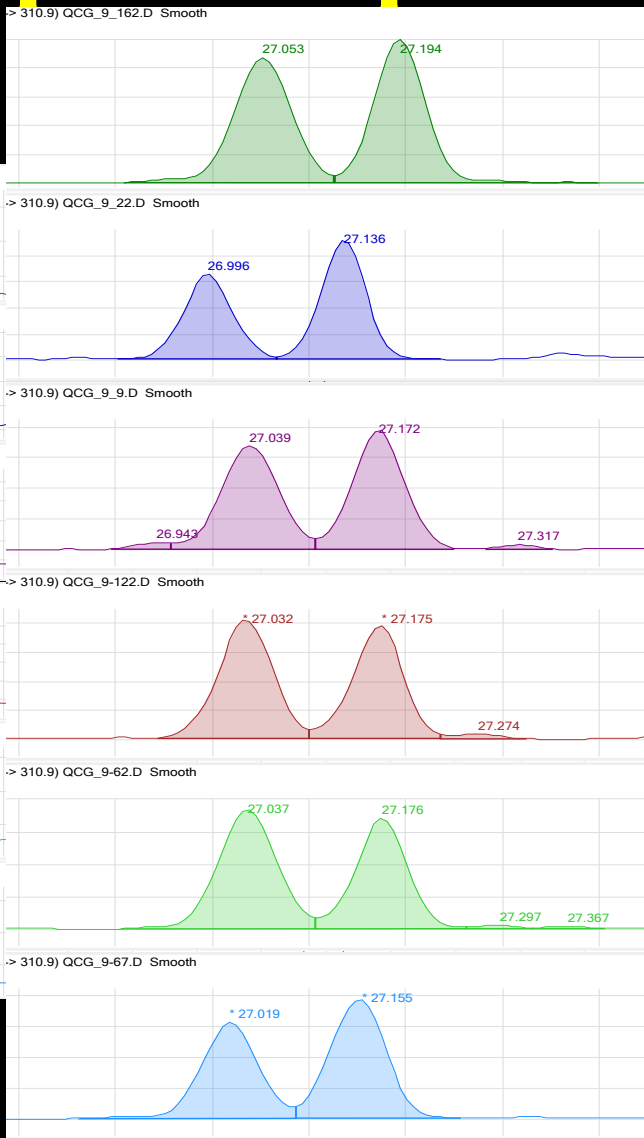
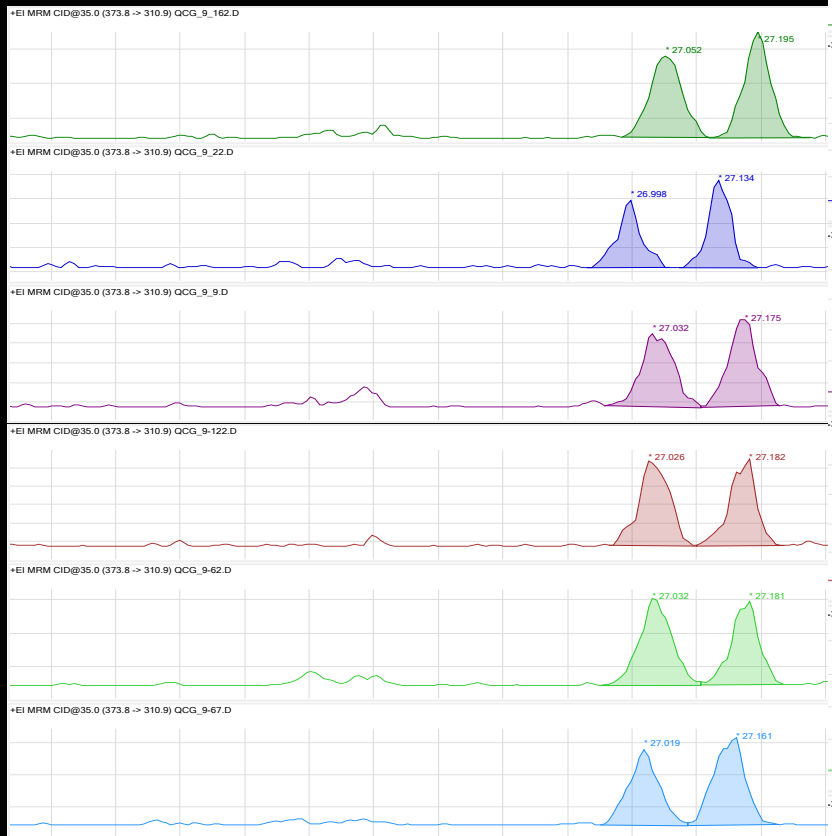
## PCDDs, PCDFs, NO PCBs



NO-PCBs	RT
PCB 81	17,71
PCB 77	18,02
PCB 126	20,92
PCB 169	24,17

# Chromatographic Separation

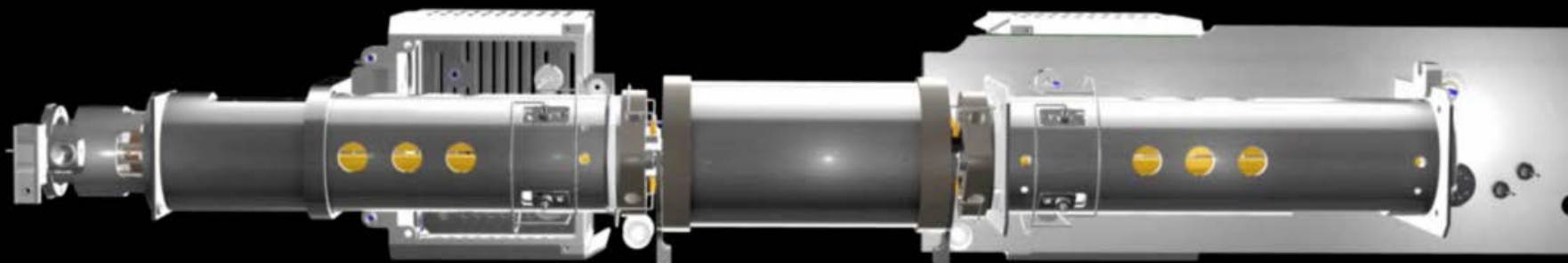
## ❖ Selectivity



✓ 25% valley separation HxCDF

# Tandem *in-space* MS

**7000 MS/MS System**  
**Optimized for gas chromatography**



Compound name	ISTD?	Precursor ion	MS1 resolution	Product ion	MS2 resolution	Dwell	Collision energy
PePCB 126	<input type="checkbox"/>	325.9	Unit	255.9	Unit	75	28
PePCB 126	<input type="checkbox"/>	323.9	Unit	253.9	Unit	75	28
2378-TCDF	<input type="checkbox"/>	305.9	Unit	242.9	Unit	75	33
2378-TCDF	<input type="checkbox"/>	303.9	Unit	240.9	Unit	75	33
2378-TCDD	<input type="checkbox"/>	321.9	Unit	258.9	Unit	75	24
2378-TCDD	<input type="checkbox"/>	319.9	Unit	256.9	Unit	75	24
13C-TCDDs	<input checked="" type="checkbox"/>	333.9	Unit	269.9	Unit	25	24
13C-TCDDs	<input checked="" type="checkbox"/>	331.9	Unit	267.9	Unit	25	24
13C-PePCB 126	<input checked="" type="checkbox"/>	337.9	Unit	267.9	Unit	25	28
13C-PePCB 126	<input checked="" type="checkbox"/>	335.9	Unit	265.9	Unit	25	28
13C6-TCDD (RS)	<input checked="" type="checkbox"/>	327.9	Unit	264.9	Unit	25	28
13C6-TCDD (RS)	<input checked="" type="checkbox"/>	325.9	Unit	262.9	Unit	25	28
13C-2378-TCDF	<input checked="" type="checkbox"/>	317.9	Unit	253.9	Unit	25	33
13C-2378-TCDF	<input checked="" type="checkbox"/>	315.9	Unit	251.9	Unit	25	33

# MRM transition Ratio

## ❖ Quant/Qual transition ratio

EU Reg 709/2014 says:

Tolerance

✓ PCDD/Fs, DL-PCBs: 2 specific precursors with each specific product ions

- 2378 TCDD:  $319.9 > 256.9$   
 $321.9 > 258.9$  → Average ratio = 0.964

± 15%  
R(quad)=unit

✓ NDL-PCBs: at least 1 precursor and 2 product ions

- PCB 189:  $393.8 > 323.8$   
 $395.8 > 325.8$  → Average ratio = 0.627

± 20%  
R(quad)=unit

# In MassHunter

Agilent MassHunter Quantitative Analysis - Develop PCDD-F-NOPCB - PCDD-F-NO-PCB.batch.bin

File Edit View Analyze Method Update Report Tools Help

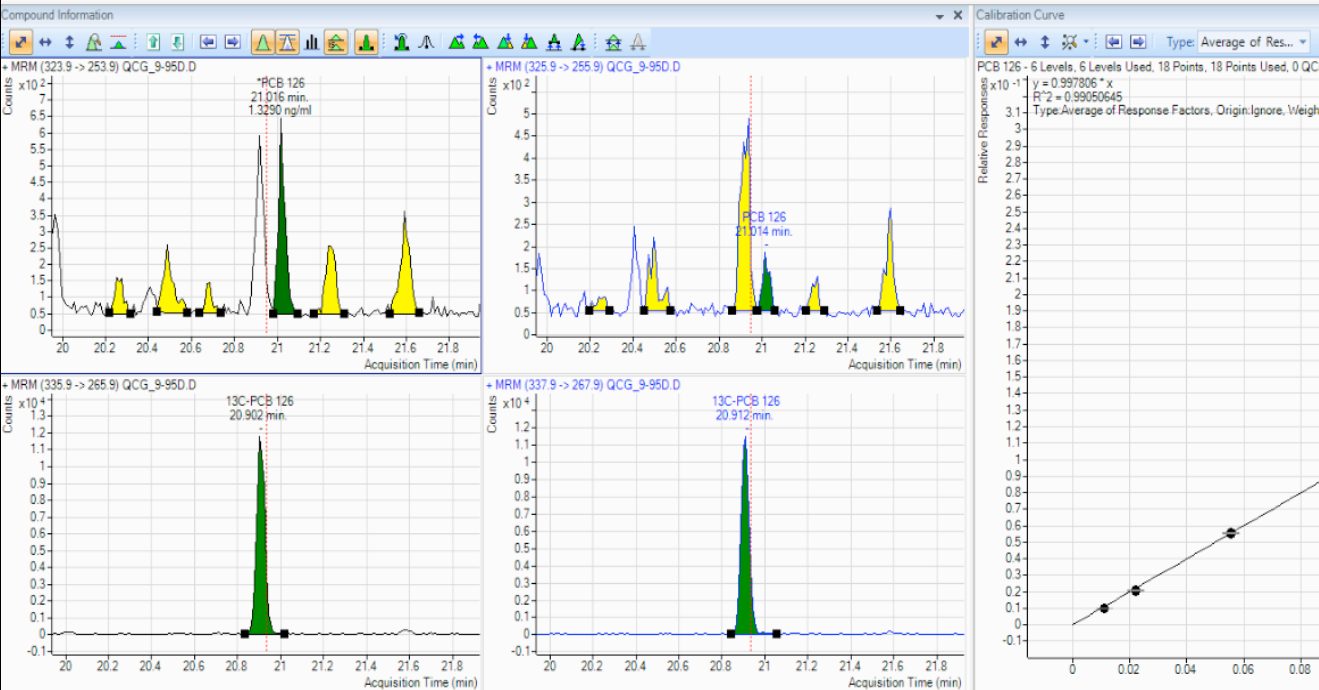
Analyze Batch Quantitate with Blank Subtraction Layout: Restore Default Layout

Batch Table

Sample: QCG\_9-95D Sample Type: Sample Compound: PCB 126 ISTD: 13C-PCB 126

Sample				PCB 126 Method						PCB 126 Results						Qualifier (325.9 -> 255.9) Results			Qualifier (337.9 -> 267.9) Results		
Name	Type	Level	Vol.	Multiplier	Avg. RF	Avg. RF RSD	RT	ISTD Conc.	RT	MI	Calc. Conc.	Final Conc.	FWHM	Area	Ratio	MI	Area	Ratio	MI	Area	
QCG_9-22D	Sample	5.00	0.14	0.9978	6.686823	20.949	360.00	20.927	13.8356	1.9999	0.042	28106	97.7	27451	93.9	701685					
QCG_9-19D	Sample	5.00	0.14	0.9978	6.686823	20.949	360.00	20.916	15.5309	2.2271	0.050	3787	98.8	3742	95.1	85249					
QCG_9-100D	Sample	5.00	0.15	0.9978	6.686823	20.949	360.00	20.916	15.3375	2.2723	0.043	1190	90.9	1083	95.6	26136					
QCG_9-76D	Sample	5.00	0.15	0.9978	6.686823	20.949	360.00	20.916	13.8269	2.0349	0.042	1046	105.3	1102	90.1	26572					
QCG_9-72D	Sample	5.00	0.15	0.9978	6.686823	20.949	360.00	20.916	14.9119	2.1659	0.039	1340	98.9	1325	98.7	32019					
QCG_9-70D	Sample	5.00	0.15	0.9978	6.686823	20.949	360.00	20.916	19.7990	2.8893	0.040	1113	86.8	966	97.7	18716					
QCG_9-44D	Sample	5.00	0.14	0.9978	6.686823	20.949	360.00	20.916	13.8320	1.9933	0.043	4048	91.1	3689	93.7	97650					
QCG_9-5D	Sample	5.00	0.15	0.9978	6.686823	20.949	360.00	20.927	14.2963	2.0991	0.044	3243	95.7	3103	94.3	77703					
QCG_9-95D	Sample	5.00	0.15	0.9978	6.686823	20.949	360.00	21.016	8.9093	1.3290	0.030	1277	22.3	285	95.5	30894					
QCG_9-85D	Sample	5.00	0.15	0.9978	6.686823	20.949	360.00	20.916	14.4958	2.1531	0.041	912	116.8	1066	98.8	24460					
QCG_9-133D	Sample	5.00	0.15	0.9978	6.686823	20.949	360.00	20.916	13.4025	1.9586	0.032	2945	90.5	2666	96.2	74059					
QCG_9-11D	Sample	5.00	0.15	0.9978	6.686823	20.949	360.00	20.916	15.0040	2.1892	0.041	2692	88.6	2385	94.5	59299					
QCG_9-82D	Sample	5.00	0.14	0.9978	6.686823	20.949	360.00	20.916	15.1048	2.1626	0.043	3461	86.8	3003	95.8	75531					

Tolerance:  
 $95.1 \pm 15\%$



# Proper Estimation of LOQs

## ❖ LOD/LOQ

### ANALYTICAL CRITERIA FOR USE OF MS/MS FOR DETERMINATION OF DIOXINS AND DIOXIN-LIKE PCBS IN FEED AND FOOD

Kotz A<sup>1</sup>, Malisch R<sup>1\*</sup>, Focant J<sup>2</sup>, Eppe G<sup>2</sup>, Cederberg TL<sup>3</sup>, Rantakokko P<sup>4</sup>, Fürst P<sup>5</sup>, Bernsmann T<sup>5</sup>, Leondiadis L<sup>6</sup>, Lovász C<sup>7</sup>, Scortichini G<sup>8</sup>, Diletti G<sup>8</sup>, di Domenico A<sup>9</sup>, Ingelido AM<sup>9</sup>, Traag W<sup>10</sup>, Smith F<sup>11</sup>, Fernandes A<sup>11</sup>

*Approach 1:* The LOQ can be calculated from the signal-to-noise ratio as already defined in the current regulations.

*Approach 2:* As an alternative approach, if the signal-to-noise ratio does not provide reliable results due to a very low, or no discernable noise level, the limit of quantification is based on the calibration curve. The limit of quantification is then defined as the lowest concentration point on a calibration curve that gives an acceptable ( $\leq 30\%$ ) and consistent (measured at least at the start and at the end of an analytical series of samples) deviation to the average relative response factor calculated for all points on the calibration curve in each series of samples.

For conversion of the limit of quantification from the calibration curve to the sample, the recovery of the internal standards of the respective congener and the sample intake has to be taken into account.

Organohalogen compound 74 (2012), 156-159

- ✓  $iLOQ = 10 * stdev$  (8 replicate injections – cali point)
- ✓  $LOQ = blank\ mean + 6 * stdev$  (12 injections – blank)

# Validation for vegetable oil (feed)

## ❖ iLOD/iLOQ

‘Acceptable and consistent deviation to the average RRF’

Quantifier						
Name	TS	RT	Transition	Scan	Type	
2378-TCDD	2	20.753	319.9 -> 256.9...	MRM	Target	
Calibration						
Level	Conc.	Response	Enable	RF	Level RSD	
1	CDC01	0.0160	424	✓	0.9341	2.534717
	CDC01	0.0160	459	✓	0.9707	2.534717
	CDC01	0.0160	450	✓	0.9257	2.534717
2	CDC02	0.0400	968	✓	0.9107	7.259317
	CDC02	0.0400	958	✓	0.9098	7.259317
	CDC02	0.0400	944	✓	1.0297	7.259317
3	CDC03	0.0800	2596	✓	0.9122	3.487891
	CDC03	0.0800	2710	✓	0.9728	3.487891
	CDC03	0.0800	2573	✓	0.9216	3.487891
4	CDC04	0.4000	13128	✓	0.9350	2.407250
	CDC04	0.4000	13004	✓	0.9408	2.407250
	CDC04	0.4000	7104	✓	0.9772	2.407250
5	CDC05	0.8000	28604	✓	0.9742	1.207290
	CDC05	0.8000	28255	✓	0.9670	1.207290
	CDC05	0.8000	28548	✓	0.9901	1.207290
6	CDC06	2.8000	89229	✓	0.9997	1.368236
	CDC06	2.8000	89444	✓	1.0241	1.368236
	CDC06	2.8000	88721	✓	1.0235	1.368236

$Av RRF_{1-6} = 0.9622$   
 $R^2(\text{linear fit}) = 0.9960$   
 $> 0.9900$

$Av RRF_1 = 0.9435$

Deviation = -1.9%  
 $< 30\%$

# Validation for vegetable oil (feed)

## ❖ iLOD/iLOQ

Calibration curve (lowest level)

 8 replicates

  $10 * \text{stdev}$

## ❖ LOQ matrix

Procedure blanks

 10 independent injections

 Average +  $6 * \text{stdev}$ .

# Validation for vegetable oil (feed)

❖ (i)LOQ

Compound	Lowest cali point				Blanks		
	Avg Conc. pg/μL	Std. Dev.	iLOD pg/μL	iLOQ pg/μL	in blank?	LOQ MS/MS pg/g	LOQ HRMS pg/g
PCB 77	0,2917	0,0037	0,011	0,037	yes	49,66	64,59
PCB 81	0,2935	0,003	0,009	0,030	yes	3,67	9,53
2378-TCDD	0,0152	0,0018	0,005	0,018	no	0,02	0,06
2378-TCDF	0,0162	0,001	0,003	0,010	yes	0,10	0,12
PCB 126	0,2906	0,0077	0,023	0,077	yes	1,37	1,21
23478-PeCDF	0,0154	0,0021	0,006	0,021	yes	0,08	0,08
12378-PeCDD	0,0169	0,0029	0,009	0,029	no	0,01	0,06
PCB 169	0,3062	0,0071	0,021	0,071	no	0,02	0,27
12378-PeCDF	0,0142	0,0022	0,007	0,022	yes	0,56	0,09
123478-HxCDF	0,0164	0,0016	0,005	0,016	yes	0,07	0,10
123678-HxCDF	0,0169	0,0009	0,003	0,009	yes	0,06	0,06
234678-HxCDF	0,0166	0,0007	0,002	0,007	yes	0,08	0,09
123789-HxCDF	0,0156	0,002	0,006	0,020	yes	0,18	0,10
123789-HxCDD	0,0774	0,0061	0,018	0,062	yes	0,06	0,06
123478-HxCDD	0,017	0,0022	0,007	0,022	yes	0,02	0,06
123678-HxCDD	0,0426	0,0032	0,010	0,032	yes	0,14	0,08
OCDD	3,5201	0,0465	0,140	0,465	yes	1,99	2,02
OCDF	0,0158	0,0027	0,008	0,027	yes	0,60	0,77

# Accuracy & Reproducibility

Spiked materials at 0.5 ML, ML, 2 ML (6 series, 3 days)

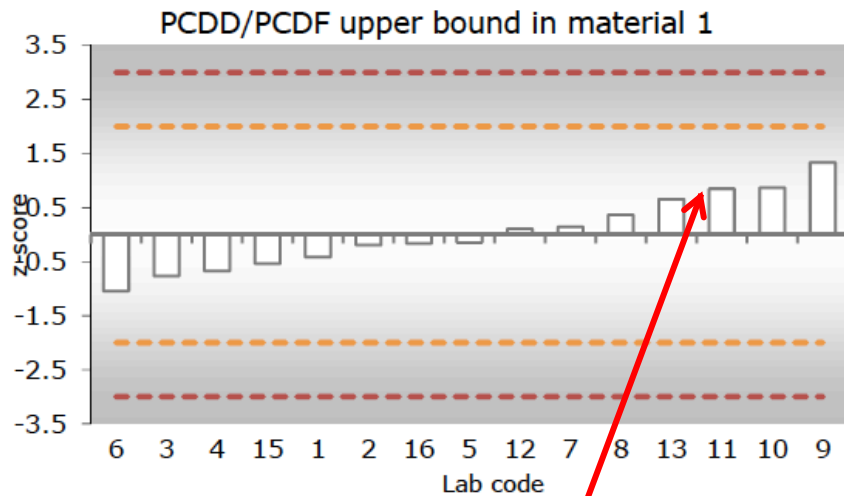
<b>PCDD/Fs</b>		<b>Average</b> ng WHO-TEQ/kg	<b>Stdev</b>	<b>RSD</b> %	<b>Target</b> ng WHO-TEQ/kg	<b>Bias</b> %
Spike level	ML/2	0,409	0,029	7,1	0,40	2,36
	ML	0,778	0,045	5,7	0,79	-1,54
	2ML	1,600	0,035	2,2	1,58	1,30
<b>NO-PCBs</b>		<b>Average</b> ng WHO-TEQ/kg	<b>Stdev</b>	<b>RSD</b> %	<b>Target</b> ng WHO-TEQ/kg	<b>Bias</b> %
Spike level	ML/2	0,307	0,028	9,0	0,33	-7,00
	ML	0,595	0,020	3,4	0,65	-8,53
	2ML	1,256	0,021	1,6	1,30	-3,42

- ✓ Bias < 20%
- ✓ Within lab reproducibility < 15%

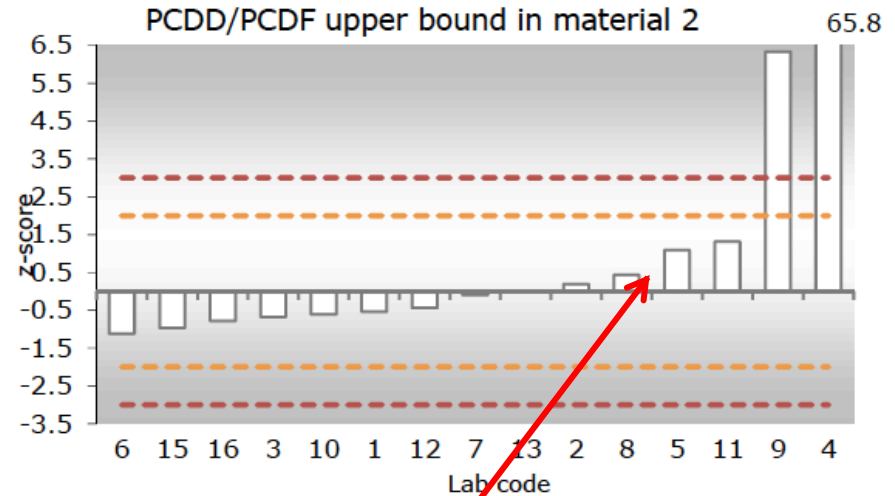
# Accuracy & Reproducibility

## Proficiency test (PT)

	Reported pg/g TEQ	Target Value pg/g TEQ	Accuracy
<b>Material 1</b>			
PCDD/Fs	1.10±0.20	1.01	8,8%
DL-PCBs	0.80±0.19	0.89	-10,3%
Total TEQ	1.90±0.36	1.90	-0,1%
<b>Material 2</b>			
PCDD/Fs	0.55±0.11	0.48	15,6%
DL-PCBs	0.82±0.21	0.85	-3,0%
Total TEQ	1.38±0.26	1.33	3,7%



Z score = 0.80

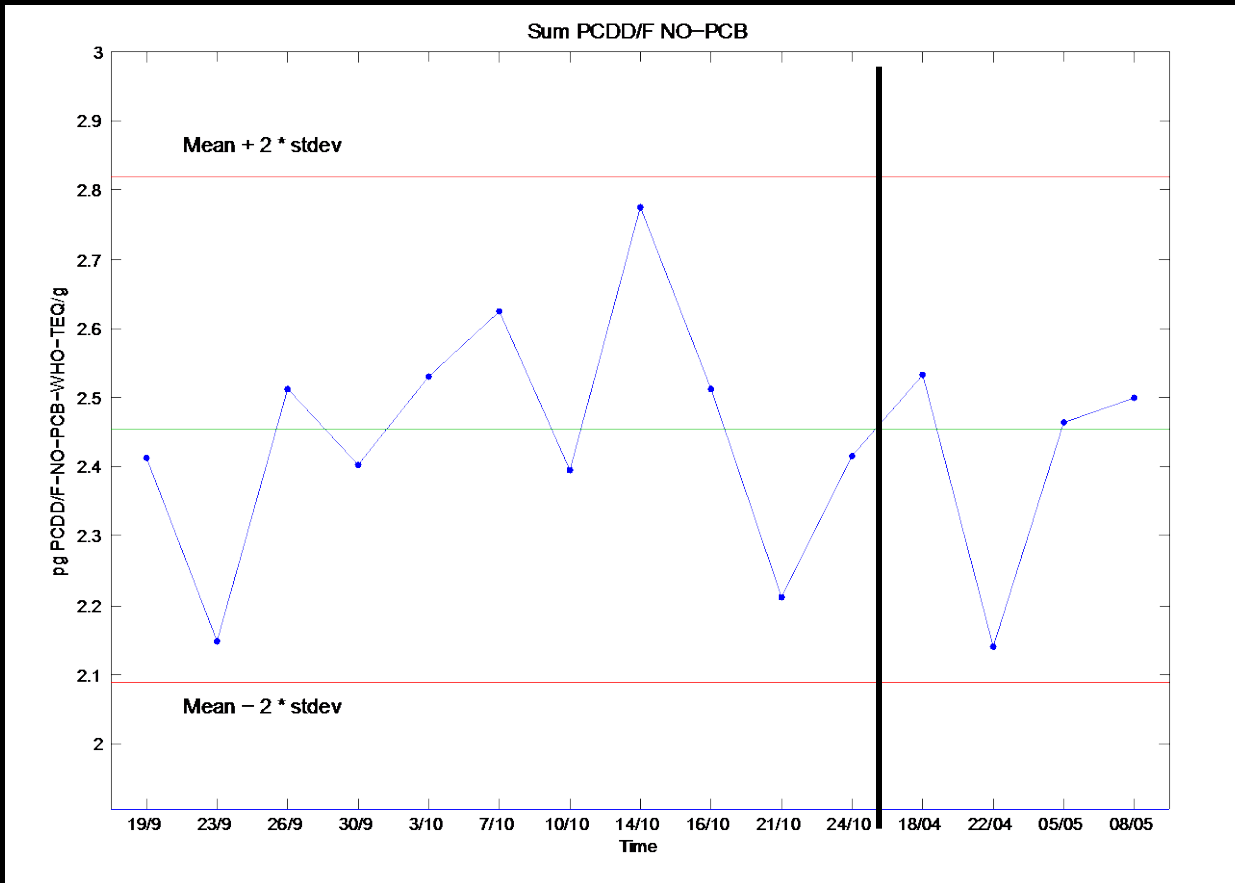


Z score = 0.59

Wim Traag, Rikilt, NL

# Within Lab Reproducibility

## QC pork fat



# MassHunter Report Generator

D:\MassHunter\GCMS\1\data\Develop PCDD-F-NOPCB\QuantResults\Laberca-PCDD-F-NO.batch.bin

**Batch Data Path**  
**Analysis Time** 2014-06-12 10:33 **Analyst** admin  
**Report Time** 2014-06-12 15:30 **Reporter** admin  
 2014-05-12 16:56 Laberca-PCDD-F-NO.batch.bin  
**Last Calib Update** **Batch**  
 2014-06- 2014-06- 2014-05-12T16:56:08.3478554+02:00  
 12T10:33:55.4621326+02:0 12T15:30:36.0656107  
 0 +02:00

**Sample Name** 14.4441\_diox-1 **Type** Sample **Vial** 62  
**Vol. [µl]** 5 **Comment** Diox

Compound	RT [min]	Conc [ng/ml]	TEQ Conc [ng/ml]	LOQ	Upper Bound [ng/ml]	Medium Bound [ng/ml]	Lower Bound [ng/ml]	WHO-TEF 2005
2378-TCDD	20,94	<LOQ	<LOQ	0,0200	0,02000	0,01000	0,00000	1
12378-PeCDD	24,27	<LOQ	<LOQ	0,0300	0,03000	0,01500	0,00000	1
123478-HxCDD	28,02	0,0376	0,00376	0,0200	0,00376	0,00376	0,00376	0,1
123678-HxCDD	28,15	<LOQ	<LOQ	0,1400	0,01400	0,00700	0,00000	0,1
123789-HxCDD	28,51	<LOQ	<LOQ	0,0600	0,00600	0,00300	0,00000	0,1
1234678-HpCDD	33,08	<LOQ	<LOQ	0,4400	0,00440	0,00220	0,00000	0,01
OCDD	39,46	4,9895	0,00150	1,9900	0,00150	0,00150	0,00150	0,0003
2378-TCDF	19,90	<LOQ	<LOQ	0,1000	0,01000	0,00500	0,00000	0,1
12378-PeCDF	23,26	<LOQ	<LOQ	0,5600	0,01680	0,00840	0,00000	0,03
23478-PeCDF	24,08	<LOQ	<LOQ	0,0800	0,02400	0,01200	0,00000	0,3
123478-HxCDF	27,05	<LOQ	<LOQ	0,0700	0,00700	0,00350	0,00000	0,1
123678-HxCDF	27,19	<LOQ	<LOQ	0,0600	0,00600	0,00300	0,00000	0,1
123789-HxCDF	29,06	<LOQ	<LOQ	0,1800	0,01800	0,00900	0,00000	0,1
234678-HxCDF	27,84	<LOQ	<LOQ	0,0800	0,00800	0,00400	0,00000	0,1
1234678-HpCDF	31,29	<LOQ	<LOQ	0,5500	0,00550	0,00275	0,00000	0,01
1234789-HpCDF	34,01	<LOQ	<LOQ	0,0200	0,00020	0,00010	0,00000	0,01
OCDF	39,89	<LOQ	<LOQ	0,6000	0,00018	0,00009	0,00000	0,0003
PCB 77	18,01	<LOQ	<LOQ	49,6600	0,00497	0,00248	0,00000	0,0001
PCB 81	18,01	6,7691	0,00203	3,6700	0,00203	0,00203	0,00203	0,0003
PCB 126	21,04	<LOQ	<LOQ	1,3700	0,13700	0,06850	0,00000	0,1
PCB 169	24,22	<LOQ	<LOQ	0,0700	0,00210	0,00105	0,00000	0,03
<b>Total TEQ</b>					<b>0,32144</b>	<b>0,16436</b>	<b>0,00729</b>	
<b>Sum TEQ PCDD/F</b>					<b>0,17534</b>	<b>0,09030</b>	<b>0,00526</b>	
<b>Sum TEQ PCBs</b>					<b>0,14610</b>	<b>0,07406</b>	<b>0,00203</b>	

Sum TEQ PCDD/F	0,175 ±0,032
Sum TEQ PCBs	0,146 ±0,035

# Take Home Message

- ✓ **PTV-GC/MS/MS accepted as a confirmatory tool under EU Regs**
- ✓ **Full validation on challenging matrix**
- ✓ **MS/MS, but still dioxin analyses...**
- ✓ **MS/MS & sectors to be properly perceived**