

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

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

Thierry Faye

Market Development Manager

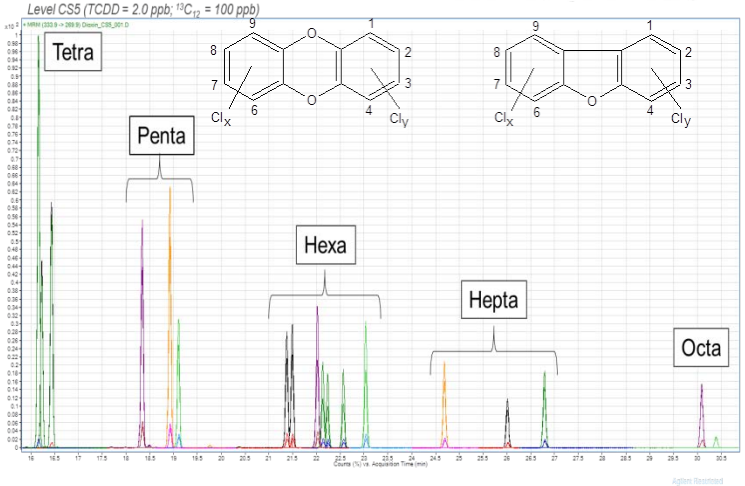
Agilent Technologies

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 compete 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

Université de Liège

CART Center for Analytical Research and Technology

OBIACHEM Organic and Biological Analytical Chemistry Group

Dioxins

Number of papers and reviews (Scopus)

1000
800
600
400
200

Yu-Che

Yusho

1968

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 <u>and</u> 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 Recommendati
- Commission Regulati /2006
- Commissio No 1881/2006
- Co ion (EC) No 1883/2006
- Regulation (EC) No 152/2009
- mission 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 ?

Evolutive 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 [1], Bert Baumann [2], Peter A. Behnisch [3], Richard Canady [4], Daniel Fraisse [5], Peter Fürst [6], Douglas Hayward [4], Ronald Hoogenboom [7], Ronald Hoogerbrugge [2], Djien Liem [2], Olaf Päpke [8], Wim Traag [7], Thomas Wiesmüller [9]

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

OHC 50 (2001) 59

www.dioxin20xx.org

→ **PBMS**

↓

- GC-IDHRMS *vs* CALUX
- ISO17025
- Validation @ LOQs (@1/5th 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

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 16215

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.

Xtraction

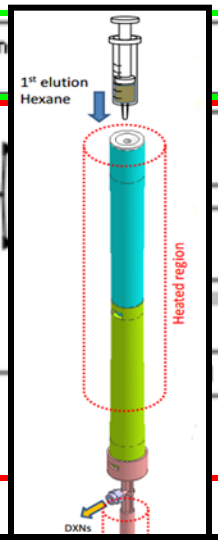
Clean-up & Fractionation

Analysis

9.1 General

9.2.1 Feed
10 g

10.2
B I- PFE



11.5.5
xtra clean up

11.2.3.1 Fr. A
for GCHRMS

11.2.3.2 Fr. B
for GCHRMS

EU Com. Reg.
589 & 709

GC-QQQ

11.2.3.1 Fr. A
for GCHRMS

EU Com. Reg.
589 & 709

Carbon

Fr. 1

Fr. 2,3,4

.3.2 Fr. B
for GCHRMS

Confirmatory Tool

- ✓ **HRMS sector or MS/MS instruments**

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

Kotz A[1], Malisch R[1*], Focant J[2], Eppe G[2], Cederberg TL[3], Rantakokko P[4], Fürst P[5], ...diadis L[6], Lovász C[7], Scortichini G[8], Diletti G[8], di Domenico A[9], Ingelido AM[9], ...ndes A[11]

[1]European Union Reference Laboratory (EU-RL) f... and Food, Freiburg, Germany; [2]CART, University of Liege, Belgium; [3]... ...nical University of Denmark, Søborg, Denmark; [4]National Institute f... Kuopio, Finland; [5]Chemisches und Veterinäruntersuch... ...-Lippe (CVUA-MEL), Münster, Germany; [6]NCSR Demokr... ...gricultural Office, Budapest, Hungary; [8]Istituto Zooprofilattico Spe... del Molise "G. Caporale, Teramo, Italy; [9]Istituto Superiore di Sanitá (ISS), Roma, Italy; ... Wageningen, The Netherlands; [11]The Food and Environment Research Agency (FERA), York, United Kingdom

EURL Working group recommendations

Organohalogen compound **74** (2012), 156-159

EU Regulation 252/2012 amended by 589/2014 (2nd of June 2014)

EU Regulation 152/2009 amended by 709/2014 (20th of June 2014)

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.

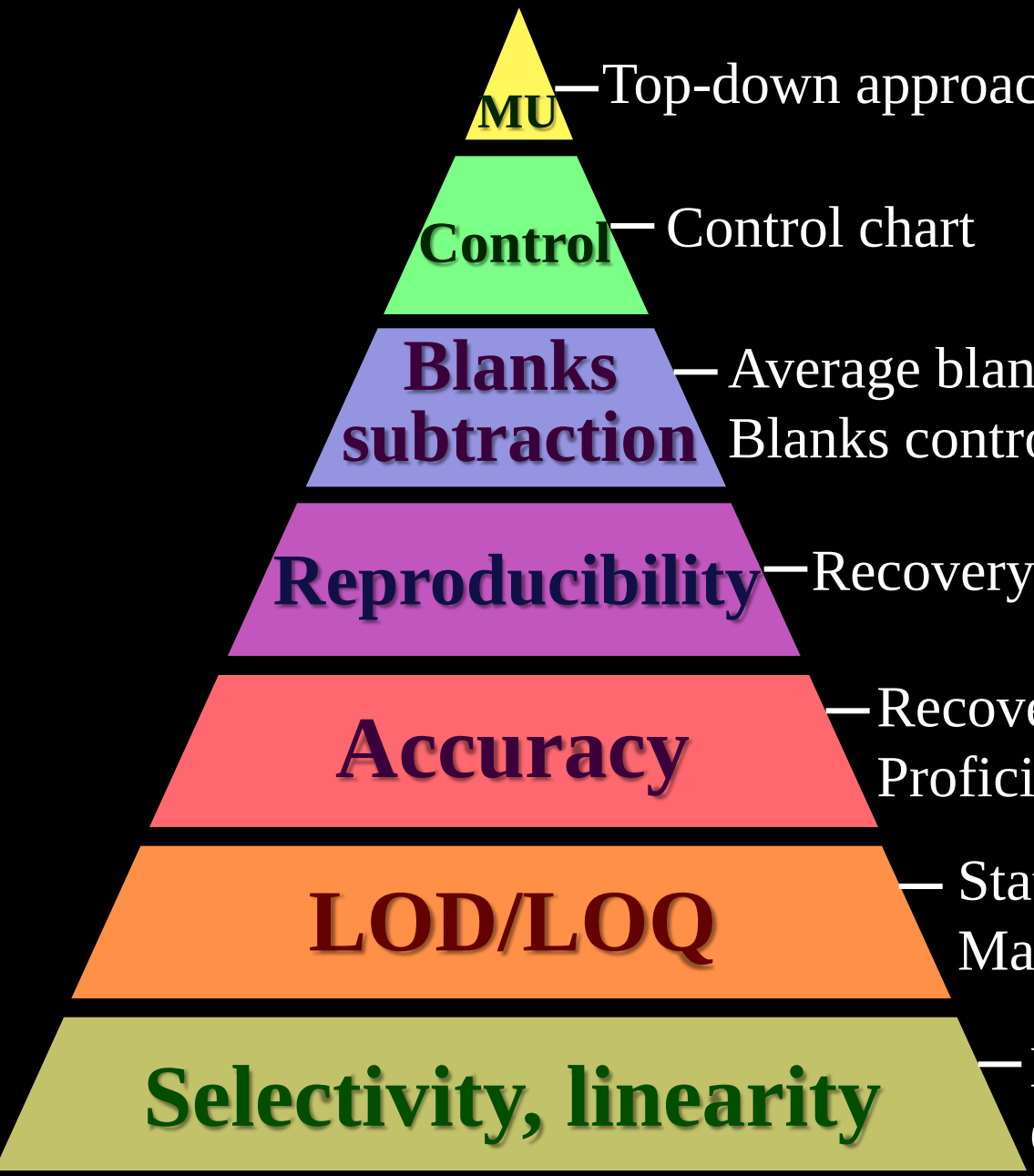
Criteria	**PCDD/Fs and DL-PCBs** *GC-MS/MS (589/2014)*	**NDL-PCBs** *GC-MS/MS (589/2014)*
Detectable quantity	-PCDD/F upper femtogram (10^-15g) -NO-PCB low picogram (10^-12g) -MO-PCB nanogram (10^-9g)	NDL-PCB nanogram (10^-9g)
Selectivity	-Chromatographic separation of 1,2,3,4,7,8-HxCDF and 1,2,3,6,7,8-HxCDF <25% valley peak to peak	Relative RT ±0.25% IS vs analyte
MRM tansitions	-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 maximul 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 caluclated 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

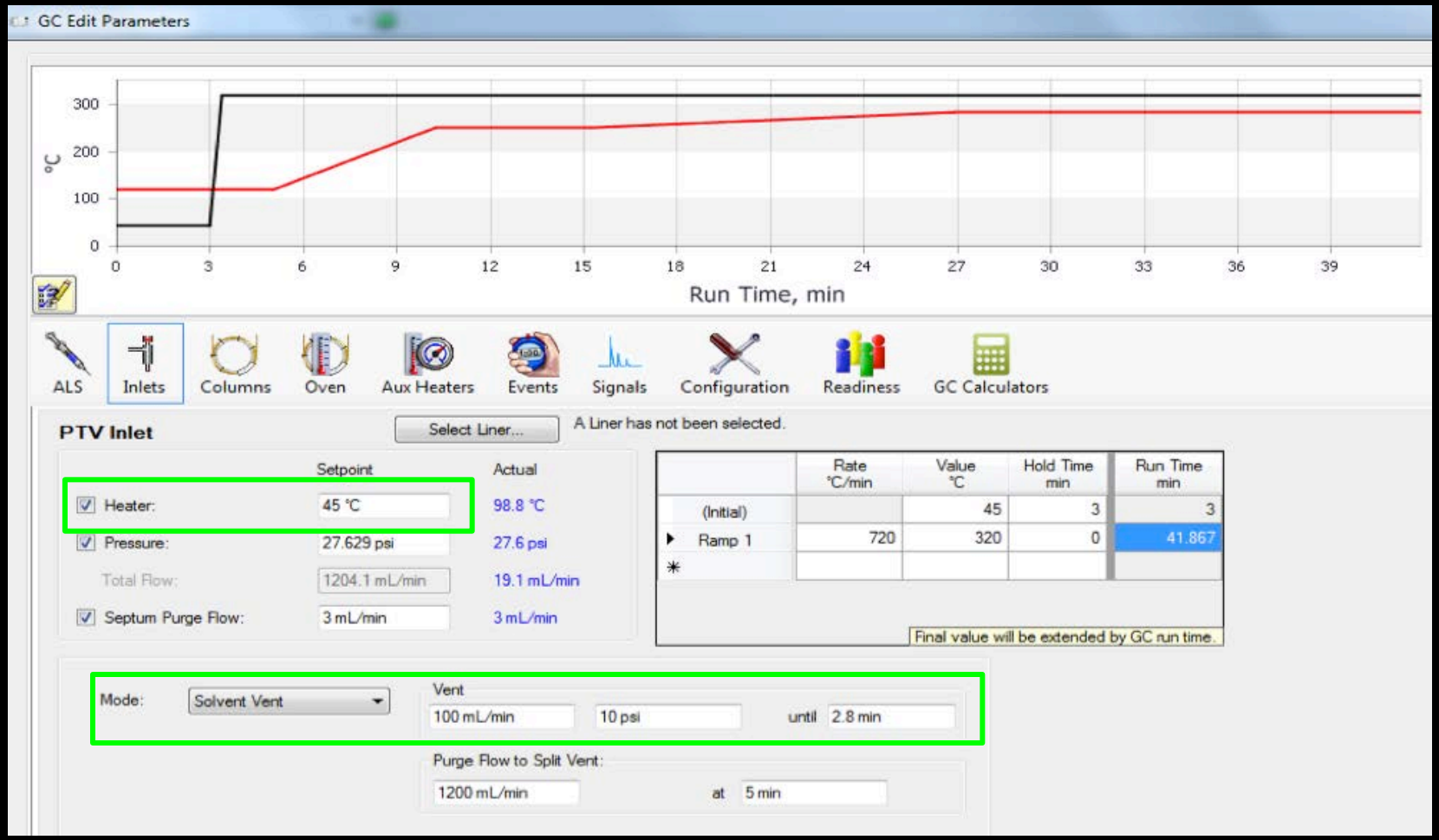
Full validation

Validation for vegetable oil (feed)

Agilent 7000C series
Triple Quad GC/MS



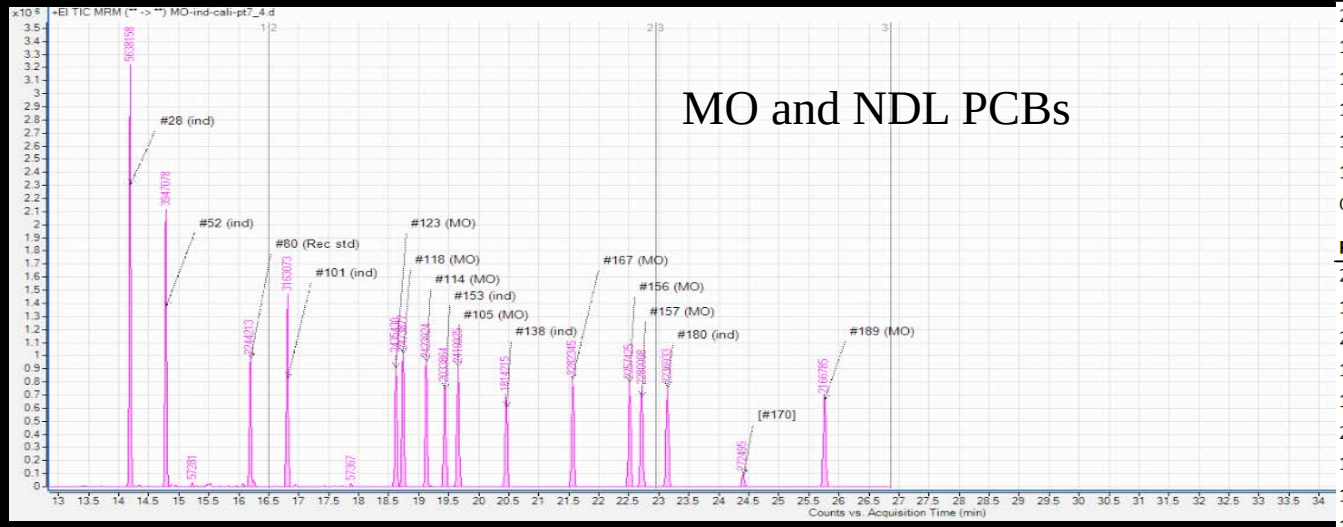
PTV Injections



Dioxin fraction runtime = 42min

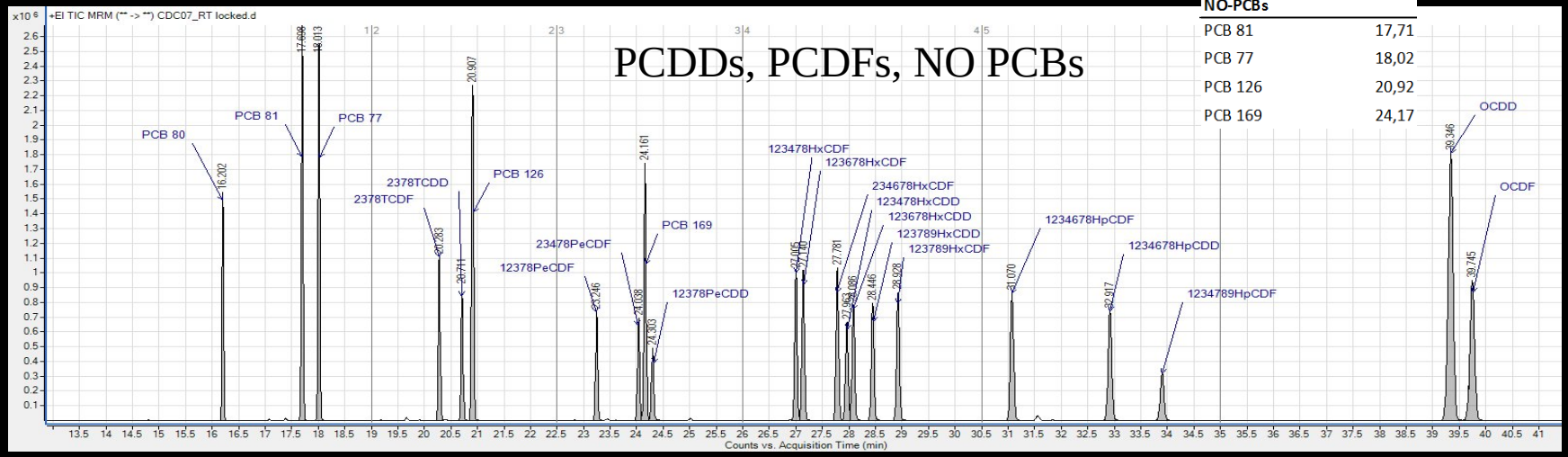
MO-PCBs fraction runtime = 28min

Chromatographic Profile



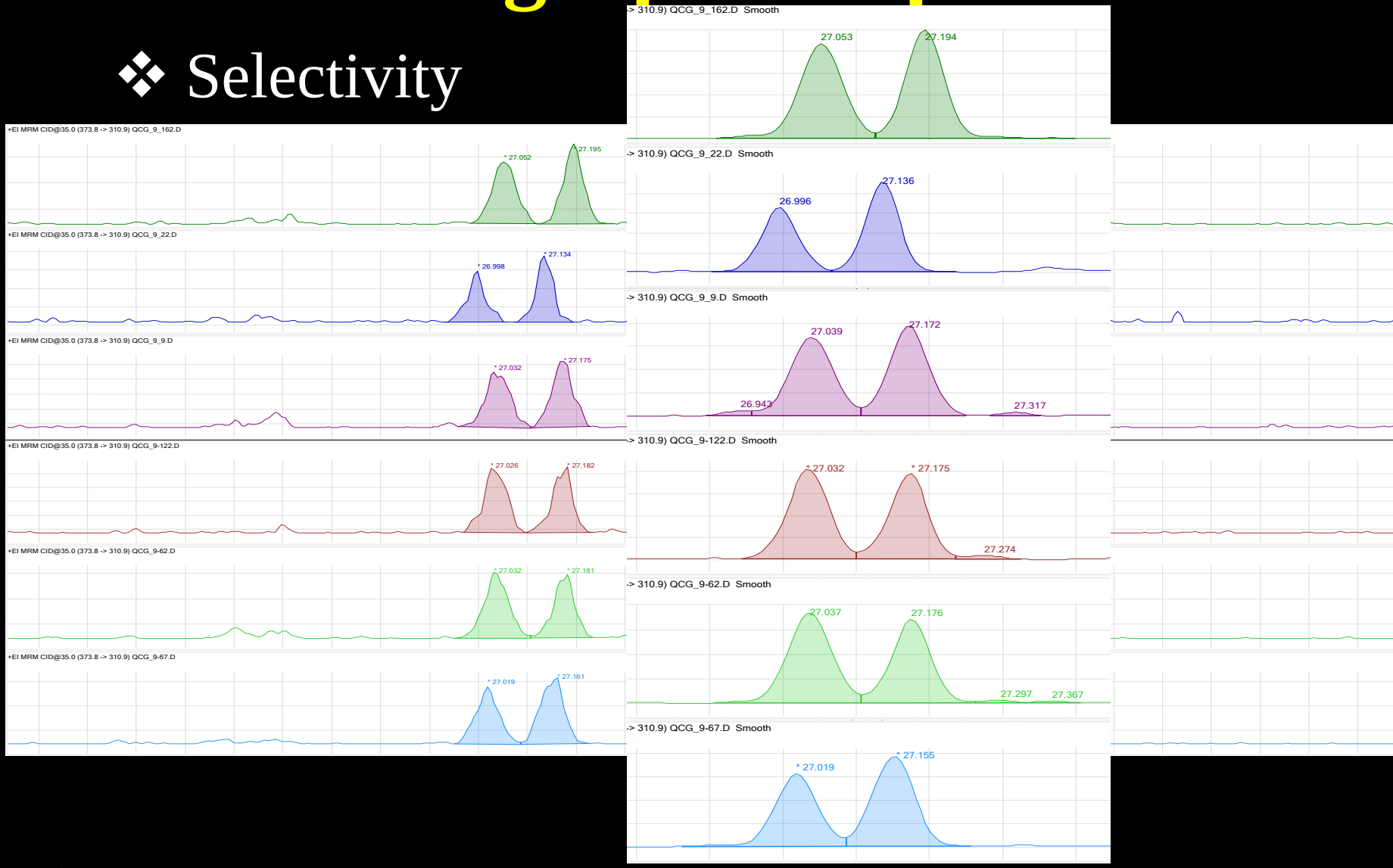
PCDDs	RT
2378-TCDD	20,72
12378-PeCDD	24,31
123478-HxCDD	27,97
123678-HxCDD	28,10
123789-HxCDD	28,46
1234678-HpCDD	32,93
OCDD	39,35
PCDFs	
2378-TCDF	20,30
12378-PeCDF	23,26
23478-PeCDF	24,06
123478-HxCDF	27,02
123678-HxCDF	27,15
234678-HxCDF	27,79
123789-HxCDF	28,94
1234678-HpCDF	31,09
1234789-HpCDF	33,92
OCDF	39,76
NO-PCBs	
PCB 81	17,71
PCB 77	18,02
PCB 126	20,92
PCB 169	24,17

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



Chromatographic Separation

- Selectivity



- ✓ 25% valley separation HxCDF

Tandem *in-space* MS

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

MRM transition Ratio

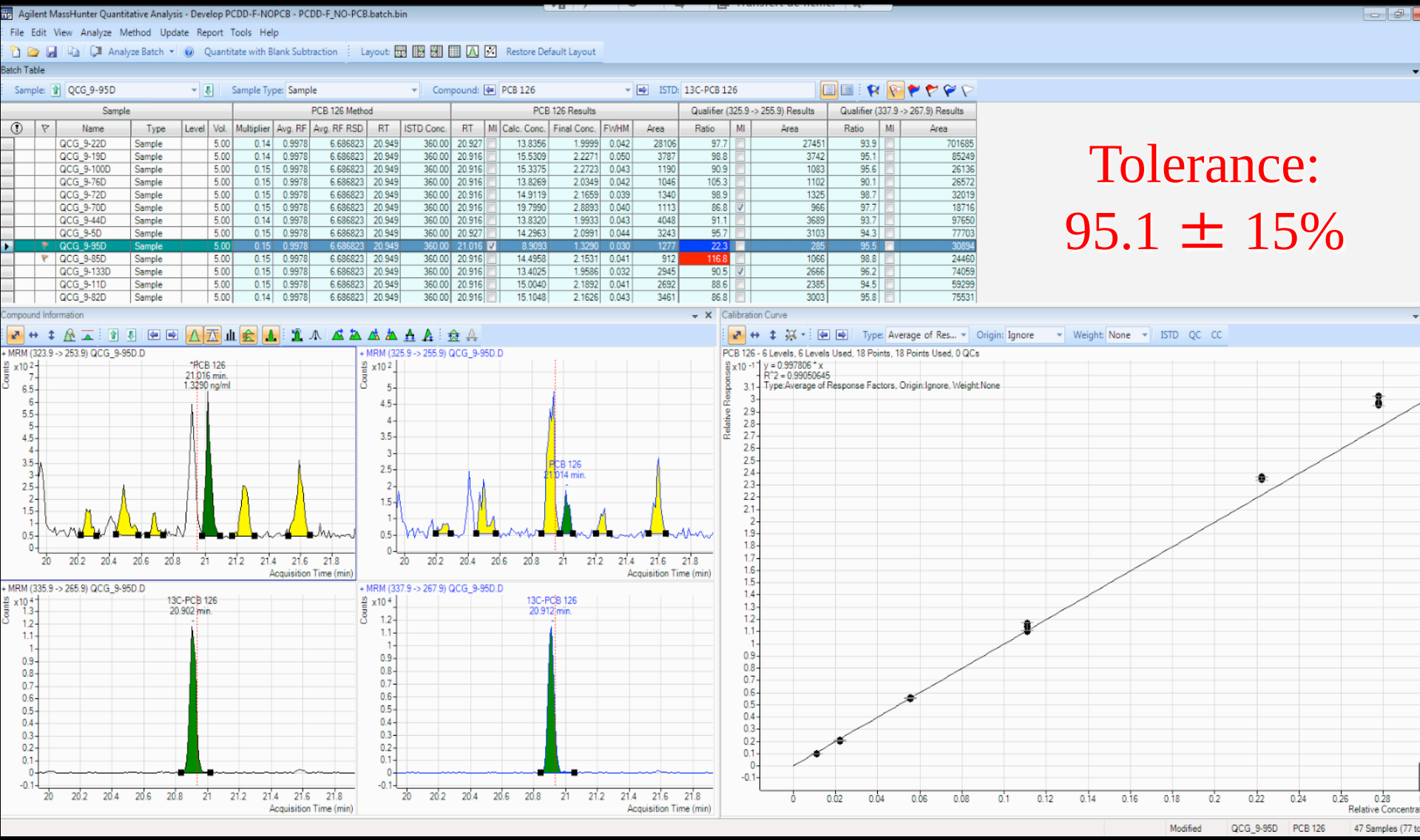
- Quant/Qual transition ratio

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



Tolerance:

95.1 ± 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[1], Malisch R[1*], Focant J[2], Eppe G[2], Cederberg TL[3], Rantakokko P[4], Fürst P[5], Bernsmann T[5], Leondiadis L[6], Lovász C[7], Scortichini G[8], Diletti G[8], di Domenico A[9], Ingelido AM[9], Traag W[10], Smith F[11], Fernandes A[11]

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 (≤ 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

< 15%

Av $RRF_{1\text{-}6}$ = 0.9622

R^2(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*stdev

- LOQ matrix

  Procedure blanks

  → 10 independent injections

  → Average + 6*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)

PCDD/Fs		Average	Stdev	RSD	Target	Bias
		ng WHO-TEQ/kg		%	ng WHO-TEQ/kg	%
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

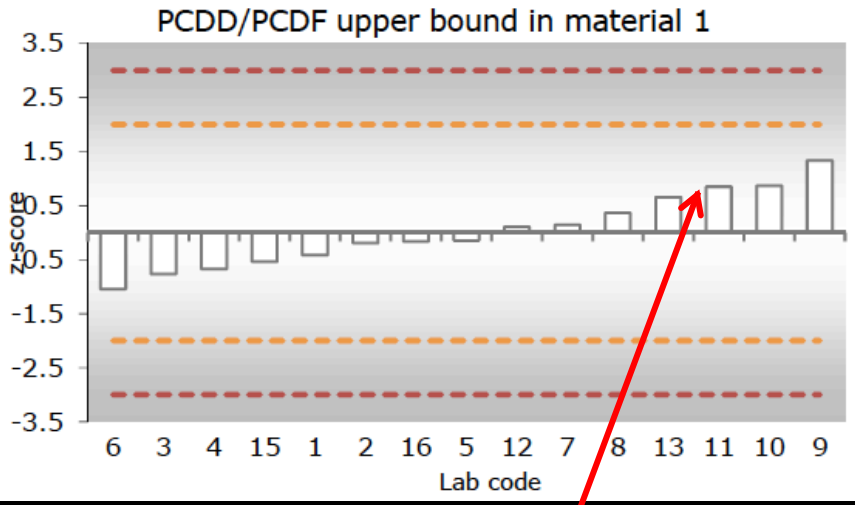
NO-PCBs		Average	Stdev	RSD	Target	Bias
		ng WHO-TEQ/kg		%	ng WHO-TEQ/kg	%
Spike level	ML/2	0,307	0,028	9,0	0,33	-7,00
	ML	0,595	0,020	3,4	0,65	-8,53
	2ML	1,256	0,021	1,6	1,30	-3,42

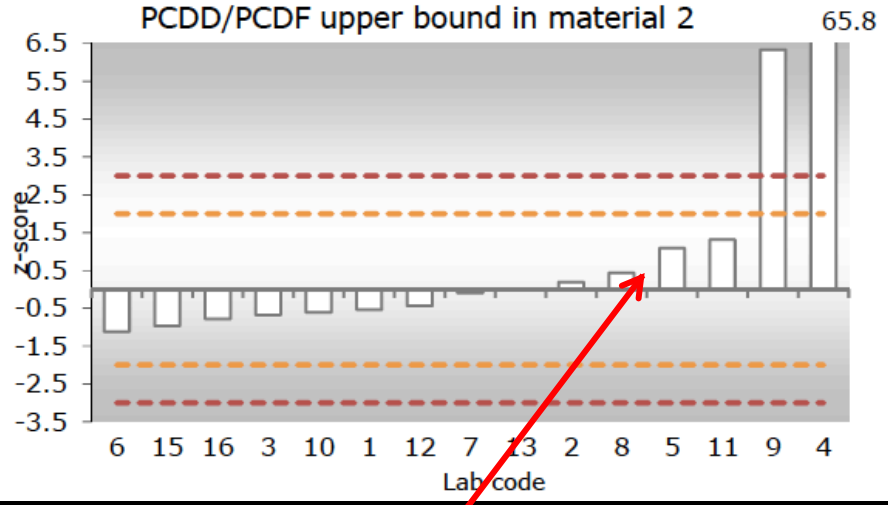
- ✓ Bias < 20%
- ✓ Within lab reproducibility < 15%

Accuracy & Reproducibility

Proficiency test (PT)

Material 1	Reported pg/g TEQ	Target Value pg/g TEQ	Accuracy
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%
Material 2			
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%





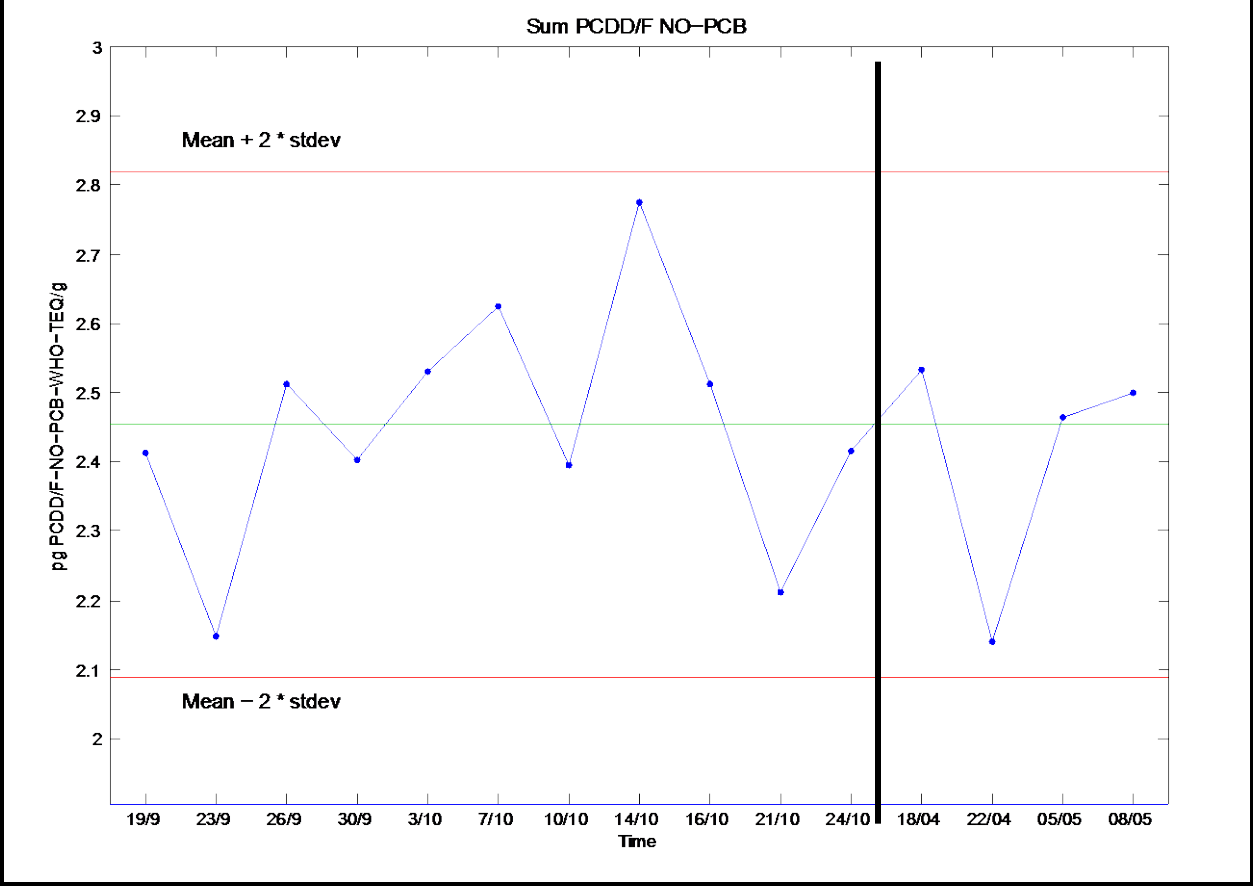
Wim Traag, Rikilt, NL

Z score = 0.80

Z score = 0.59

Within Lab Reproducibility

QC pork fat



MassHunter Report Generator

Batch Data Path	D:\MassHunter\GCMS\1\data\Develop PCDD-F-NOPCB\QuantResults\Laberca-PCDD-F-NO.batch.bin				
Analysis Time	2014-06-12 10:33	**Analyst**	admin		
Report Time	2014-06-12 15:30	**Reporter**	admin		
Last Calib Update	2014-05-12 16:56	**Batch**	Laberca-PCDD-F-NO.batch.bin		
2014-06-12T10:33:55.4621326+02:00	2014-06-12T15:30:36.0656107+02:00	2014-05-12T16:56:08.3478554+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
Total TEQ					**0,32144**	**0,16436**	**0,00729**	
Sum TEQ PCDD/F					**0,17534**	**0,09030**	**0,00526**	
Sum TEQ PCBs					**0,14610**	**0,07406**	**0,00203**	

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**