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
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).
- 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
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
- Commission Directive 2006/13/EC
- Commission Recommendation 2006/88/EC
- 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
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 ⁷, Olaf Papke ⁸, Wim Traag ⁷, Thomas Wiesmüller ⁹

OHC 50 (2001) 53

PBMS

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

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

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

Clean-up & Fractionation

Analysis

EU Com. Reg. 589 & 709

GC-QQQ

EU Com. Reg. 589 & 709
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 A¹, Malisch R¹, Focant J², Eppe G², Cederberg TL³, Rantakokko P⁴, Fürst R⁵, Linn J⁶, Tadiadis L⁶, Lovasz C⁷, Scortichini G⁸, Diletti G⁸, di Domenico A⁹, Ingelido AM⁹, Horning M⁹, Steuber A⁹, Mehl S¹⁰, Schmid A¹¹

¹European Union Reference Laboratory (EU-RL) for Food and Feed, Freiburg, Germany; ²CART, University of Liege, Belgium; ³National Veterinary Institute, Uppsala, Sweden; ⁴National University of Denmark, Søborg, Denmark; ⁵National Institute of Public Health, Kuopio, Finland; ⁶Chemisches und Veterinäruntersuchungsamt des Landes Nordrhein-Westfalen (CVUA-MEL), Münster, Germany; ⁷NCSR Demokritos, Agricultural Office, Athens, Greece; ⁸Istituto Zooprofilattico Sperimentale delle Venezie del Molise "G. Caporale, Teramo, Italy; ⁹Istituto Superiore di Sanità (ISS), Roma, Italy; ¹⁰Wageningen UR, Wageningen, The Netherlands; ¹¹The Food and Environment Research Agency (FERA), York, United Kingdom

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

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)

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)

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.
<table>
<thead>
<tr>
<th>Criteria</th>
<th>PCDD/Fs and DL-PCBs GC-MS/MS (589/2014)</th>
<th>NDL-PCBs GC-MS/MS (589/2014)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Detectable quantity</td>
<td>-PCDD/F upper femtogram (10^-15g)</td>
<td>NDL-PCB nanogram (10^-9g)</td>
</tr>
<tr>
<td></td>
<td>-NO-PCB low picogram (10^-12g)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-MO-PCB nanogram (10^-9g)</td>
<td></td>
</tr>
<tr>
<td>Selectivity</td>
<td>-Chromatographic separation of 1,2,3,4,7,8-HxCDF and 1,2,3,6,7,8-HxCDF &lt;25% valley peak to peak</td>
<td>Relative RT ± 0.25% IS vs analyte</td>
</tr>
<tr>
<td>MRM transitions</td>
<td>-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</td>
<td>-Monitoring at least 1 precursor ion and 2 product ions -Tolerance ratio ± 20% if rel. intens. &gt;50% Tolerance ratio ± 25% if rel. intens. 20-50% -Resolution MS quadrupoles = unit</td>
</tr>
<tr>
<td>Blank</td>
<td>-Used for LOQ calculation</td>
<td>-Used for LOQ calculation</td>
</tr>
<tr>
<td></td>
<td>-Blank value &lt;30% of maximal level ML</td>
<td></td>
</tr>
<tr>
<td>iLOQ</td>
<td>-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 &lt;30%</td>
<td>-ditto</td>
</tr>
<tr>
<td>LOQ</td>
<td>-LOQ calculated from average blank level -LOQ &lt; 1/5 of maximum level ML -Difference ub and lb levels &lt;20%ML</td>
<td>-ditto</td>
</tr>
<tr>
<td>Accuracy Reproducibility</td>
<td>-Demonstrate performances at 0.5ML, ML, 2ML -Trueness (accuracy) ± 20% -Within-lab reproducibility (RSD) &lt;15%</td>
<td>-Demonstrate performances at 0.5ML, ML, 2ML -Trueness for sum ind-PCB @ ML ± 30% -Within-lab reproducibility (RSD) &lt;20%</td>
</tr>
<tr>
<td>Control</td>
<td>-QC chart for blanks</td>
<td>-QC chart for blanks</td>
</tr>
<tr>
<td></td>
<td>-QC charts control sample</td>
<td>-QC charts control sample</td>
</tr>
<tr>
<td>Recovery</td>
<td>-Individual internal std in range 60-120% -Out of range OK if contribu. to TEQ&lt;10%</td>
<td>-Individual internal std in range 50-120% -Out of range OK if contribu. to sum ind-PCB&lt;10%</td>
</tr>
<tr>
<td>Measurement uncertainty</td>
<td>-Expanded measurement uncertainty</td>
<td>-Expanded measurement uncertainty</td>
</tr>
<tr>
<td></td>
<td>-Coverage factor = 2 (CL=95%)</td>
<td>-Coverage factor = 2 (CL=95%)</td>
</tr>
<tr>
<td></td>
<td>-If separate determination of congeners, make sum of separate uncertainty for sum of PCDD/F and DL-PCBs</td>
<td></td>
</tr>
</tbody>
</table>

From Regulation

Full validation
Validation for vegetable oil (feed)

- Selectivity, linearity
  - MRM, Quant/qual transitions
- LOD/LOQ
  - Statistical determination
    - Matrix related
  - MRM, Quant/qual transitions
    - GC separation, Cali. curve
- Accuracy
  - Recovery experiments
    - Proficiency test
- Reproducibility
  - Recovery experiments, QCs
- Blanks subtraction
  - Average blank
  - Blanks control chart
- Control
  - Control chart
- MU
  - Top-down approach

Agilent 7000C series
Triple Quad GC/MS
PTV Injections

Dioxin fraction runtime = 42min
MO-PCBs fraction runtime = 28min
Chromatographic Profile

MO and NDL PCBs

<table>
<thead>
<tr>
<th>PCDDs</th>
<th>RT</th>
</tr>
</thead>
<tbody>
<tr>
<td>2378-TCDD</td>
<td>20.72</td>
</tr>
<tr>
<td>12378-PeCDD</td>
<td>24.31</td>
</tr>
<tr>
<td>123478-HxCDD</td>
<td>27.97</td>
</tr>
<tr>
<td>123678-HxCDD</td>
<td>28.10</td>
</tr>
<tr>
<td>123789-HxCDD</td>
<td>28.46</td>
</tr>
<tr>
<td>1234678-HpCDD</td>
<td>32.39</td>
</tr>
<tr>
<td>OCDD</td>
<td>39.35</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>PCDFs</th>
<th>RT</th>
</tr>
</thead>
<tbody>
<tr>
<td>2378-TCDF</td>
<td>20.30</td>
</tr>
<tr>
<td>12378-PeCDF</td>
<td>23.26</td>
</tr>
<tr>
<td>23478-PeCDF</td>
<td>24.66</td>
</tr>
<tr>
<td>123478-HxCDF</td>
<td>27.02</td>
</tr>
<tr>
<td>123678-HxCDF</td>
<td>27.15</td>
</tr>
<tr>
<td>234678-HxCDF</td>
<td>27.79</td>
</tr>
<tr>
<td>123789-HxCDF</td>
<td>28.94</td>
</tr>
<tr>
<td>1234678-HpCDF</td>
<td>31.09</td>
</tr>
<tr>
<td>OCDF</td>
<td>39.76</td>
</tr>
</tbody>
</table>

| 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

<table>
<thead>
<tr>
<th>PCB</th>
<th>RT</th>
</tr>
</thead>
<tbody>
<tr>
<td>81</td>
<td>17.71</td>
</tr>
<tr>
<td>77</td>
<td>18.02</td>
</tr>
<tr>
<td>126</td>
<td>20.92</td>
</tr>
<tr>
<td>169</td>
<td>24.17</td>
</tr>
</tbody>
</table>
Chromatographic Separation

Selectivity

✓ 25% valley separation HxCDF
# Tandem in-space MS

## 7000 MS/MS System
Optimized for gas chromatography

<table>
<thead>
<tr>
<th>Compound name</th>
<th>ISTD?</th>
<th>Precursor</th>
<th>MS1 resolution</th>
<th>Product ion</th>
<th>MS2 resolution</th>
<th>Dwell</th>
<th>Collision energy</th>
</tr>
</thead>
<tbody>
<tr>
<td>PePCB 126</td>
<td>⬜</td>
<td>325.9</td>
<td>Unit</td>
<td>⬜</td>
<td>255.9</td>
<td>⬜</td>
<td>75</td>
</tr>
<tr>
<td>PePCB 126</td>
<td>⬜</td>
<td>323.9</td>
<td>Unit</td>
<td>⬜</td>
<td>253.9</td>
<td>⬜</td>
<td>75</td>
</tr>
<tr>
<td>2378-TCDF</td>
<td>⬜</td>
<td>305.9</td>
<td>Unit</td>
<td>⬜</td>
<td>242.9</td>
<td>⬜</td>
<td>75</td>
</tr>
<tr>
<td>2378-TCDD</td>
<td>⬜</td>
<td>303.9</td>
<td>Unit</td>
<td>⬜</td>
<td>240.9</td>
<td>⬜</td>
<td>75</td>
</tr>
<tr>
<td>2378-TCDD</td>
<td>⬜</td>
<td>321.9</td>
<td>Unit</td>
<td>⬜</td>
<td>256.9</td>
<td>⬜</td>
<td>75</td>
</tr>
<tr>
<td>2378-TCDD</td>
<td>⬜</td>
<td>319.9</td>
<td>Unit</td>
<td>⬜</td>
<td>256.9</td>
<td>⬜</td>
<td>75</td>
</tr>
<tr>
<td>13C-TCDDs</td>
<td>⬜</td>
<td>333.9</td>
<td>Unit</td>
<td>⬜</td>
<td>263.9</td>
<td>⬜</td>
<td>25</td>
</tr>
<tr>
<td>13C-TCDDp</td>
<td>⬜</td>
<td>221.9</td>
<td>Unit</td>
<td>⬜</td>
<td>267.9</td>
<td>⬜</td>
<td>25</td>
</tr>
<tr>
<td><strong>13C-PePCB 126</strong></td>
<td>⬜</td>
<td>337.9</td>
<td>Unit</td>
<td>⬜</td>
<td>267.9</td>
<td>⬜</td>
<td>25</td>
</tr>
<tr>
<td><strong>13C-PePCB 126</strong></td>
<td>⬜</td>
<td>335.9</td>
<td>Unit</td>
<td>⬜</td>
<td>265.9</td>
<td>⬜</td>
<td>25</td>
</tr>
<tr>
<td>13C-6-TCDD (RS)</td>
<td>⬜</td>
<td>327.9</td>
<td>Unit</td>
<td>⬜</td>
<td>264.9</td>
<td>⬜</td>
<td>25</td>
</tr>
<tr>
<td>13C-6-TCDD (RS)</td>
<td>⬜</td>
<td>325.9</td>
<td>Unit</td>
<td>⬜</td>
<td>262.9</td>
<td>⬜</td>
<td>25</td>
</tr>
<tr>
<td>13C-2378-TCDF</td>
<td>⬜</td>
<td>317.9</td>
<td>Unit</td>
<td>⬜</td>
<td>253.9</td>
<td>⬜</td>
<td>25</td>
</tr>
<tr>
<td><strong>13C-2378-TCDF</strong></td>
<td>⬜</td>
<td>315.9</td>
<td>Unit</td>
<td>⬜</td>
<td>251.9</td>
<td>⬜</td>
<td>25</td>
</tr>
</tbody>
</table>
MRM transition Ratio

- Quant/Qual transition ratio

EU Reg 709/2014 says:

- **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

- **NDL-PCBs**: at least 1 precursor and 2 product ions
  - PCB 189: 393.8 > 323.8
  - 395.8 > 325.8
  - Average ratio = 0.627

Tolerance

- ± 15% R(quad)=unit
- ± 20% R(quad)=unit
In MassHunter

SANCO 11950 says:

- PCDD/Fs, DL-PCBs: 2 specific precursors with each specific product ions
  - PCB 189: 393.8 > 323.8
  - PCB 189: 395.8 > 325.8
  - 2378 TCDD: 319.9 > 256.9
  - 2378 TCDD: 321.9 > 258.9
  - Average ratio = 0.964

- NDL-PCBs: at least 1 precursor and 2 product ions
  - Average ratio = 0.627

Tolerance:
- ± 15%
- ± 20%
- R(quad)=unit

Tolerance: 95.1 ± 15%
Proper Estimation of LOQs

- LOD/LOQ

**iLOQ = 10*stdev (8 replicate injections – cali point)**

**LOQ = blank mean + 6*stdev (12 injections – blank)**

**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
Validation for vegetable oil (feed)

- iLOD/iLOQ

‘Acceptable and consistent deviation to the average RRF’

<table>
<thead>
<tr>
<th>Level</th>
<th>Conc.</th>
<th>Response</th>
<th>Enable</th>
<th>RF</th>
</tr>
</thead>
<tbody>
<tr>
<td>CDC01</td>
<td>0.0160</td>
<td>424</td>
<td></td>
<td>0.9341</td>
</tr>
<tr>
<td>CDC02</td>
<td>0.0400</td>
<td>968</td>
<td></td>
<td>0.9122</td>
</tr>
<tr>
<td>CDC03</td>
<td>0.0800</td>
<td>2596</td>
<td></td>
<td>0.9734</td>
</tr>
<tr>
<td>CDC04</td>
<td>0.0400</td>
<td>13128</td>
<td></td>
<td>0.9350</td>
</tr>
<tr>
<td>CDC05</td>
<td>0.0800</td>
<td>25648</td>
<td></td>
<td>0.9742</td>
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<tr>
<td>CDC06</td>
<td>2.8000</td>
<td>89229</td>
<td></td>
<td>0.9997</td>
</tr>
</tbody>
</table>

Mean of Calibrations

Av RRF<sub>1-6</sub> = 0.9622

Av RRF<sub>1</sub> = 0.9435

Deviation = -1.9%

R<sup>2</sup>(linear fit) = 0.9960

< 15%

> 0.9900

< 30%
Validation for vegetable oil (feed)

- **iLOD/iLOQ**
  
  Calibration curve (lowest level)
  
  8 replicates

- **LOQ matrix**
  
  Procedure blanks
  
  10 independent injections
  
  Average + 6*stdev.
## Validation for vegetable oil (feed)

**\( i \)LOQ**

<table>
<thead>
<tr>
<th>Compound</th>
<th>Avg Conc. pg/µL</th>
<th>Std. Dev.</th>
<th>iLOD pg/µL</th>
<th>iLOQ pg/µL</th>
<th>in blank?</th>
<th>Blanks</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>LOQ MS/MS pg/g</td>
</tr>
<tr>
<td>PCB 77</td>
<td>0.2917</td>
<td>0.0037</td>
<td>0.011</td>
<td>0.037</td>
<td>yes</td>
<td>49.66</td>
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<tr>
<td>PCB 81</td>
<td>0.2935</td>
<td>0.003</td>
<td>0.009</td>
<td>0.030</td>
<td>yes</td>
<td>3.67</td>
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<tr>
<td>2378-TCDD</td>
<td>0.0152</td>
<td>0.0018</td>
<td>0.005</td>
<td>0.018</td>
<td>no</td>
<td>0.02</td>
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<tr>
<td>2378-TCDF</td>
<td>0.0162</td>
<td>0.001</td>
<td>0.003</td>
<td>0.010</td>
<td>yes</td>
<td>0.10</td>
</tr>
<tr>
<td>PCB 126</td>
<td>0.2906</td>
<td>0.0077</td>
<td>0.023</td>
<td>0.077</td>
<td>yes</td>
<td>1.37</td>
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<tr>
<td>23478-PeCDF</td>
<td>0.0154</td>
<td>0.0021</td>
<td>0.006</td>
<td>0.021</td>
<td>yes</td>
<td>0.08</td>
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<tr>
<td>12378-PeCDD</td>
<td>0.0169</td>
<td>0.0029</td>
<td>0.009</td>
<td>0.029</td>
<td>no</td>
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<tr>
<td>PCB 169</td>
<td>0.3062</td>
<td>0.0071</td>
<td>0.021</td>
<td>0.071</td>
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<td>12378-PeCDF</td>
<td>0.0142</td>
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<td>0.007</td>
<td>0.022</td>
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<tr>
<td>123478-HxCDF</td>
<td>0.0164</td>
<td>0.0016</td>
<td>0.005</td>
<td>0.016</td>
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<td>0.07</td>
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<td>0.0009</td>
<td>0.003</td>
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<td>yes</td>
<td>0.06</td>
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<tr>
<td>234678-HxCDF</td>
<td>0.0166</td>
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Accuracy & Reproducibility

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

<table>
<thead>
<tr>
<th>PCDD/Fs</th>
<th>Average (ng WHO-TEQ/kg)</th>
<th>Stddev (ng WHO-TEQ/kg)</th>
<th>RSD (%)</th>
<th>Target (ng WHO-TEQ/kg)</th>
<th>Bias (%)</th>
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<tbody>
<tr>
<td>Spike level</td>
<td>ML/2</td>
<td>0.409</td>
<td>0.029</td>
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<table>
<thead>
<tr>
<th>NO-PCBs</th>
<th>Average (ng WHO-TEQ/kg)</th>
<th>Stddev (ng WHO-TEQ/kg)</th>
<th>RSD (%)</th>
<th>Target (ng WHO-TEQ/kg)</th>
<th>Bias (%)</th>
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</thead>
<tbody>
<tr>
<td>Spike level</td>
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- Bias < 20%
- Within lab reproducibility < 15%
Accuracy & Reproducibility

Proficiency test (PT)

<table>
<thead>
<tr>
<th>Material 1</th>
<th>Reported pg/g TEQ</th>
<th>Target Value pg/g TEQ</th>
<th>Accuracy</th>
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</thead>
<tbody>
<tr>
<td>PCDD/Fs</td>
<td>1.10±0.20</td>
<td>1.01</td>
<td>8.8%</td>
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<tr>
<td>DL-PCBs</td>
<td>0.80±0.19</td>
<td>0.89</td>
<td>-10.3%</td>
</tr>
<tr>
<td>Total TEQ</td>
<td>1.90±0.36</td>
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<td>-0.1%</td>
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</table>

<table>
<thead>
<tr>
<th>Material 2</th>
<th>Reported pg/g TEQ</th>
<th>Target Value pg/g TEQ</th>
<th>Accuracy</th>
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</thead>
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<tr>
<td>PCDD/Fs</td>
<td>0.55±0.11</td>
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<td>DL-PCBs</td>
<td>0.82±0.21</td>
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<td>-3.0%</td>
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<tr>
<td>Total TEQ</td>
<td>1.38±0.26</td>
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<td>3.7%</td>
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</table>
Within Lab Reproducibility

QC pork fat

![Graph showing PCDD/F NO-PCB over time with mean ± 2 * stdev markers.](image)
### 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

**Report Time**
2014-06-12 15:30

**Analyst**
admin

**Report Time**
2014-06-12 15:30

**Reporter**
admin

**Last Calib Update**
2014-06-12 16:56

**Batch**
Laberca-PCDD-F-NO.batch.bin

### Sample Information

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<tr>
<th>Sample Name</th>
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### Compound Table

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<tbody>
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</table>

**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