



## ● Quantitative detection of low levels of dioxins for food and feed safety using the EVOQ GC-TQ

Reliable and relevant GC-MS/MS method to meet routine performance criteria according to current EU legislations

### Introduction

From the first published reports of adverse health effects from exposure to dioxins and furans in the 1970s, this class of Persistent Organic Pollutants (POPs), along

with other classes of compounds with similar effects, has been subject to increased global scrutiny. Although their production and use were banned by the Stockholm Convention in 2001, the continued detrimental

impact of exposure to these pollutants to human and animal health [1] has led to strict governmental regulations regarding their detection and quantitation.

*Keywords:*  
EVOQ GC-TQ, dioxins, persistent organic pollutants (POPs), pesticide detection, food and feed safety, environmental screening

Within the EU, the maximum levels (ML) of dioxins, dioxin-like PCBs, and non-dioxin-like PCBs in food have been established through Regulation EC 1881/2006 [2]. Under these regulations, both the sum of all dioxins and the sum of all dioxins and dioxin-like polychlorinated biphenyls (DL-PCBs) must be monitored according to their toxic equivalency factor (TEQ) as established by the WHO [3]. The MLs vary among specific food groups and feedstuffs in accordance with the understood adverse health effects from exposure. The current maximum levels according to Regulation EC 1881/2006 for example food groups are shown in Table 1.

The complex and varied nature of food and environmental samples adds to the analytical challenge of precise target differentiation and identification accuracy, as well as to meeting method sensitivity requirements. While GC-HRMS (based on magnetic sector instrumentation) has been broadly used within dioxin monitoring programs, the costs associated with this technique - the instruments themselves, maintenance, and the need for highly skilled personnel for their operation – can be prohibitive for routine analytical laboratories.

GC-MS/MS is also an EU-approved confirmatory technique for dioxin analysis [4,5], and triple quadrupole (TQ) MS instrumentation can offer unique analytical advantages. The EVOQ GC-TQ system has been engineered for sustained high sensitivity and selectivity, including an innovative "lens-free," elliptical ion-path design to provide the high sensitivity and chemical noise reduction essential for the reliable ultra-trace analysis of dioxins in complex matrices. (Figure 1) Together with intuitive operational software, precise and confident target detection and quantitation to meet strict regulatory guidelines can be easily achieved.

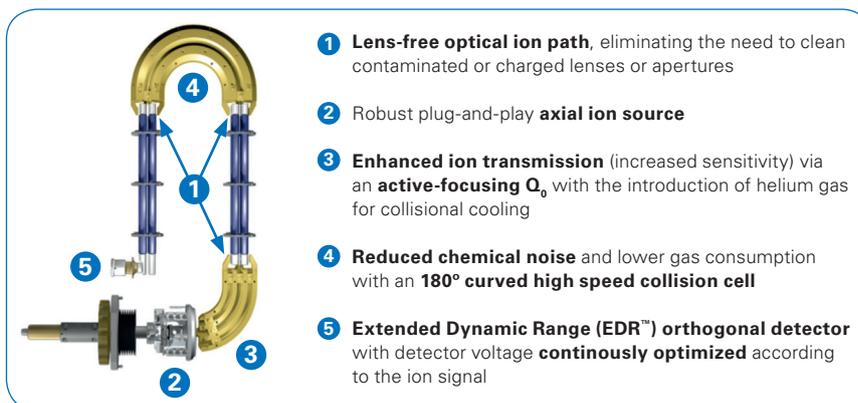


Figure 1: Unique hardware design features of the Bruker EVOQ GC-TQ, supporting instrument robustness and sustained high sensitivity.

Table 1: Maximum levels of dioxins in food (EU) [2].

Foodstuff	Sum of dioxins WHO-TEQ	Sum of dioxins and DL-PCBs WHO-TEQ
Bovine and sheep meat	2.5 pg/g fat	4.0 pg/g fat
Poultry meat	1.75 pg/g fat	3.0 pg/g fat
Pigs meat	1.0 pg/g fat	1.25 pg/g fat
Fish and fishery products	3.5 pg/g wet w	6.5 pg/g wet w
Marine oils	1.75 pg/g fat	6.0 pg/g fat
Milk, dairy products, butterfat	2.5 pg/g fat	5.5 pg/g fat
Hen eggs and egg products	2.5 pg/g fat	5.0 pg/g fat
Mixed animal fats	1.5 pg/g fat	2.5 pg/g fat
Vegetable oil and fats	0.75 pg/g fat	1.25 pg/g fat
Food for infants	0.1 pg/g wet w	0.2 pg/g wet w

Table 2: Analytical criteria of EU Regulation 644/2017 regarding the determination of dioxins, dioxin-like PCBs and non-dioxin-like PCBs in foodstuffs [4].

<b>MS Technique</b>	Confirmatory methods by GC-HRMS or GC-MS/MS
<b>Resolution</b>	Resolution for each quadrupole $\leq 1$ mass unit
<b>m/z ions</b>	Two precursor ions each with one specific MRM product ion
<b>Ion ratio QC</b>	Tolerance: +/- 15%
<b>GC separation</b>	Isomer separation (<25% peak to peak) between 1,2,3,4,7,8- HxCDF and 1,2,3,6,7,8-HxCDF
<b>Quantitation</b>	By isotope dilution technique. $^{13}\text{C}$ labeled isotope for each congener
<b>Sensitivity</b>	Detectable quantities in the upper femtogram ( $10^{-15}$ g) range
<b>LOQ</b>	LOQ = 1/5 of the maximum level, S/N>3 for less intensive signal
<b>Calibration</b>	To cover the relevant range of maximum or action levels
<b>Precision</b>	Intermediate precision RSD <sub>i</sub> <15%
<b>Recovery</b>	For internal standards: 60-120%
<b>Accuracy</b>	+/- 20%, expressed as trueness

Table 3: Instrument parameters

Gas chromatography	
Instrument	Bruker 436 GC
Carrier gas	Helium, 1.5 mL/min, constant flow
Injector	1177 split/splitless, 280°C
Injection volume	1 µL, pulsed splitless
Insert	4 mm ID fritted liner
Column	BR-Dioxins, 60 m x 0.25 mm, 0.25 micron film thickness
Column temp.	140°C, hold 1 min; 200°C at 20°C/min, hold 1min; 320°C at 5°C/min, hold 14 min
Autosampler	Bruker 8400
Mass spectrometry	
Instrument	Bruker EVOQ GC-TQ
Source	EI, 280°C
X-line temp.	280°C
Collision gas	Ar, 1.5 mTorr
MS mode	Dynamic MRM
Scan time	Automatic, EDR
Quad resolution	Q1 = 0.7 mass unit, Q3 = 0.7 mass unit
Software	Bruker Compass TQ

In this study, the capabilities of the EVOQ GC-TQ system for the determination of PCDDs and PCDFs in food, feed, and environmental samples were evaluated according to the analytical performance criteria within current EU regulations [4,5] (Table 2). Analyses were made on a set of validated reference materials and on an animal feed sample doped with a mixture of PCDDs/PCDFs at trace concentration levels. A subset of data collected on the EVOQ GC-TQ system was also compared to results obtained by GC-HRMS.

## Materials and Methods

Dioxins calibration standard (EPA 1613 CSV) was obtained from Wellington Laboratories, Inc (Ontario, Canada). Certified reference materials were obtained from the European Commission, Joint Research Centre (Geel, Belgium). Animal feed samples were prepared using the PowerPrep SPE system (Fluid Management Systems, Inc, Massachusetts, USA).

Target separation and analysis were made using an EVOQ GC-TQ Triple Quadrupole MS system coupled to a Bruker 436 GC. Instrument conditions are detailed in Table 3. GC-HRMS analyses of an identical aliquot of the certified reference materials were made by an external laboratory, and summary result reports provided.

Bruker Compass TQ software (Bruker Daltonics) was used for EVOQ GC-TQ data screening and quantitation. Data evaluation rules were set to meet the current EU regulations [4], such that any reported values higher than the setpoint would trigger a red flag to indicate that the compound requires review (Figure 2).

Ions	Ion Ratios	Rules			
		Rule name	Type	Value	
1	<input checked="" type="checkbox"/>	carryOverLimit	Implicit	0.05	
2	<input checked="" type="checkbox"/>	detectionLimit	Implicit	-0.05	
3	<input checked="" type="checkbox"/>	estimatedLowValue	Implicit		
4	<input checked="" type="checkbox"/>	falseNegative	Implicit		
5	<input checked="" type="checkbox"/>	lowerLimitOfQuantitation	Implicit	0.01	
6	<input checked="" type="checkbox"/>	maximumDeviation	Implicit	15.00	
7	<input checked="" type="checkbox"/>	maximumDeviationQC	Implicit	15.00	
8	<input checked="" type="checkbox"/>	minimumCorrelation	Implicit	0.99	
9	<input checked="" type="checkbox"/>	quantityAboveMaxQuantityOfCalibration	Implicit		
10	<input checked="" type="checkbox"/>	quantityBelowMinQuantityOfCalibration	Implicit		
11	<input checked="" type="checkbox"/>	quantityExceedsReportingLimit	Implicit	1,000.00	
12	<input checked="" type="checkbox"/>	responseRange	Implicit	10.00	
13	<input checked="" type="checkbox"/>	retentionTimeDrift	Implicit	10.00	
14	<input checked="" type="checkbox"/>	signalInBlank	Implicit	0.01	
15	<input checked="" type="checkbox"/>	standardDeviationResponseFactor	Implicit	15.00	
16	<input checked="" type="checkbox"/>	upperLimitOfQuantitation	Implicit	1,000.00	
		Ions	Ref. Ions	Ion Ratio Value	
				Ion Ratio Tolerance Value	
1	(+)	422.0 > 226.0 [70.0V]	(+) 424.0 > 361.0 [25.0V]	0.230	15.000
2	(+)	424.0 > 298.0 [42.0V]	(+) 424.0 > 361.0 [25.0V]	0.830	15.000

Figure 2: Data evaluation rules established within Bruker Compass TQ, including all analytical performance criteria outlined within EU 644/2017 [4].

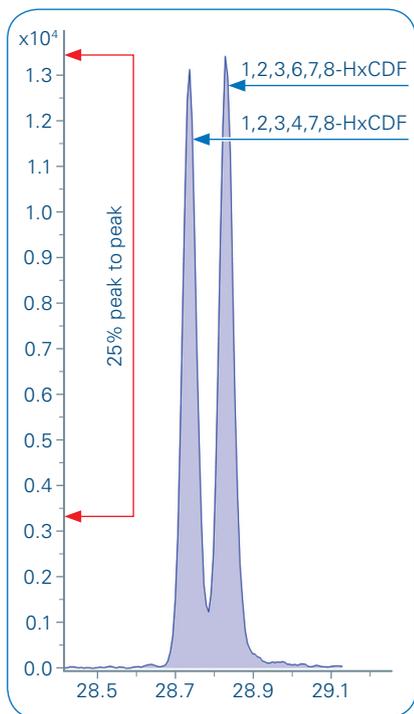


Figure 3: Isomer separation <25% peak to peak between 1,2,3,4,7,8- HxCDF and 1,2,3,6,7,8- HxCDF (CS2 level) via Bruker's 436 GC, meeting current EU regulatory criteria [4,5].

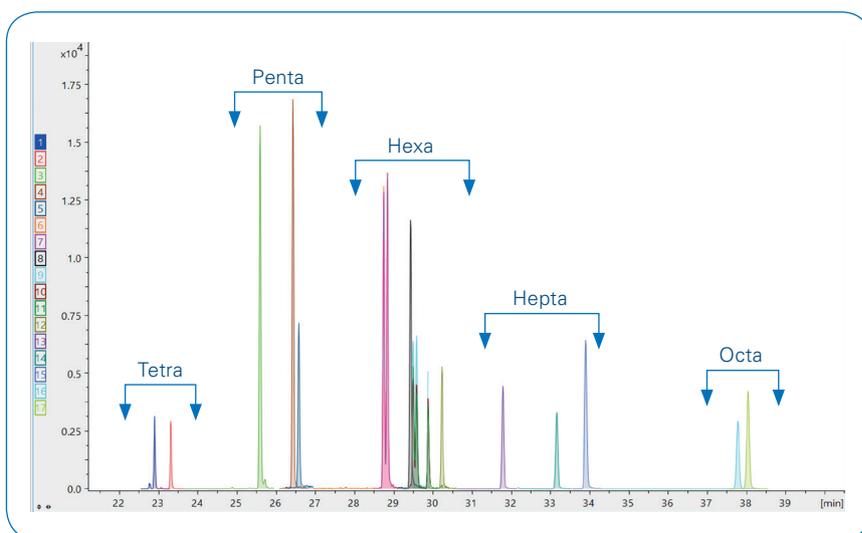


Figure 4: Overlaid MRM chromatogram of 17 native PCDD and PCDF compounds (calibration level CS2).

RQ	Score	Analyte Name	RT Score	Ions Score	Quantity [ng/ml]	Accuracy [%]
##	+++	1,2,3,4,6,7,8-HpCDD	++	++	51.04	102.08
##	+++	1,2,3,4,6,7,8-HpCDF	++	++	50.25	100.51
##	+++	1,2,3,4,7,8-HxCDD	++	++	47.59	95.18
##	+++	1,2,3,4,7,8-HxCDF	++	++	50.83	101.66
##	+++	1,2,3,6,7,8-HxCDD	++	++	49.38	98.77
##	+++	1,2,3,6,7,8-HxCDF	++	++	48.66	97.32
##	+++	1,2,3,7,8,9-HxCDD	++	++	52.42	104.85
##	+++	1,2,3,7,8,9-HxCDF	++	++	50.44	100.89
##	+++	1,2,3,7,8-PCDD	++	++	49.80	99.60
##	+++	1,2,3,7,8-PCDF	++	++	49.98	99.97
##	+++	1,2,3,4,7,8,9-HpCDF	++	++	52.04	104.09
##	+++	2,3,4,6,7,8-HxCDF	++	++	47.18	94.37
##	+++	2,3,4,7,8-PCDF	++	++	48.68	97.36
##	+++	2,3,7,8 (3,7Cl4)TCDD	++	++	49.71	99.42
##	+++	2,3,7,8-TCDD	++	++	9.75	97.47
##	+++	2,3,7,8-TCDF	++	++	10.31	103.13
##	+++	OCDD	++	++	105.74	105.74
##	+++	OCDF	++	++	107.74	107.74

Figure 5: Retention time and ion ratio scoring, precision and recoveries for target PCDDs/PCDFs compounds (CS3 verification level) as displayed within the Compass TQ software.

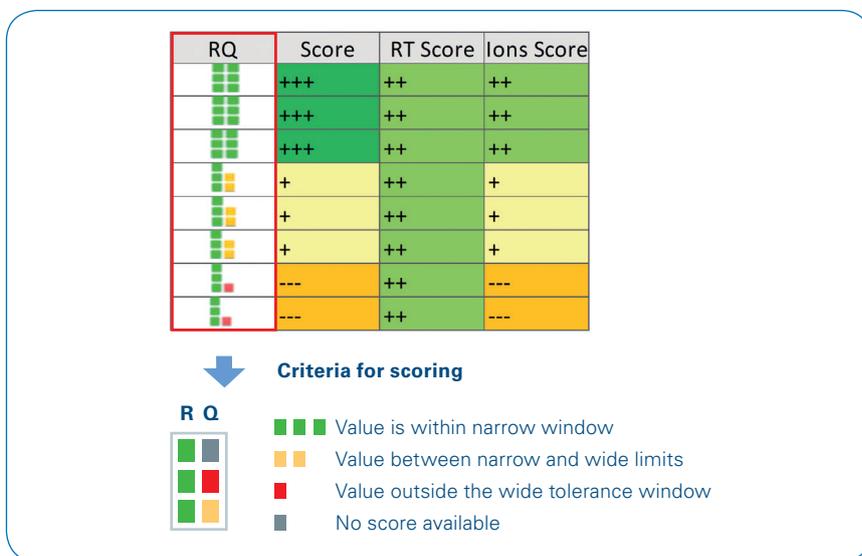


Figure 6: Visualization of retention time (R) and ion ratio (Q) criteria, as scored within Compass TQ software. RQ scores are indicated via color coding and bar height for fast and confident result review and elimination of false positives/negatives. Wide, narrow, and exclusion filter criteria may be individually defined for every target compound by the user.

Table 4: MRM transition table, indicating two precursor ions for each target, along with one specific MRM product ion. The loss of COCl (63 Da) is specific to dioxins, and this selective transition produces clean MRM chromatograms with very few interferences.

N°	Compounds	RT (min)	Quan. transition	CE (eV)	Confirm. transitions	CE (eV)
1	<sup>13</sup> C-1,2,3,4-TCDD	22.77	332 > 268	-22	334 > 270	-22
2	<sup>13</sup> C-2,3,7,8-TCDF	22.88	316 > 252	-30	318 > 254	-30
3	2,3,7,8-TCDF	22.89	304 > 171	-55	306 > 243 304 > 206	-30 -40
4	<sup>13</sup> C-2,3,7,8-TCDD	23.29	332 > 268	-22	334 > 270	-22
5	2,3,7,8-TCDD	23.30	322 > 259	-22	320 > 194 322 > 196	-40 -40
6	<sup>13</sup> C 1,2,3,7,8-PCDF	25.57	352 > 288	-32	350 > 286	-32
7	1,2,3,7,8-PCDF	25.58	340 > 277	-30	338 > 205 338 > 240	-55 -40
8	<sup>13</sup> C-2,3,4,7,8-PCDF	26.41	352 > 288	-32	350 > 286	-32
9	2,3,4,7,8-PCDF	26.42	340 > 277	-30	338 > 205 338 > 240	-55 -40
10	<sup>13</sup> C-1,2,3,7,8-PCDD	26.57	368 > 304	-22	370 > 306	-22
11	1,2,3,7,8-PCDD	26.57	356 > 293	-25	354 > 291 356 > 230	-25 -40
12	<sup>13</sup> C-1,2,3,4,7,8-HxCDF	28.73	386 > 322	-32	388 > 324	-32
13	1,2,3,4,7,8-HxCDF	28.74	374 > 311	-30	372 > 239 372 > 309	-55 -30
14	<sup>13</sup> C 1,2,3,6,7,8-HxCDF	28.83	386 > 322	-32	388 > 324	-32
15	1,2,3,6,7,8-HxCDF	28.83	374 > 311	-30	372 > 239 372 > 309	-55 -30
16	<sup>13</sup> C-2,3,4,6,7,8-HxCDF	29.41	386 > 322	-32	388 > 324	-32
17	2,3,4,6,7,8-HxCDF	29.42	374 > 311	-30	372 > 239 372 > 309	-55 -30
18	<sup>13</sup> C-1,2,3,4,7,8-HxCDD	29.48	402 > 338	-22	404 > 340	-22
19	1,2,3,4,7,8-HxCDD	29.49	390 > 264	-40	388 > 262 390 > 327	-40 -25
20	<sup>13</sup> C-1,2,3,6,7,8-HxCDD	29.56	402 > 338	-22	404 > 340	-22
21	1,2,3,6,7,8-HxCDD	29.58	390 > 264	-40	388 > 262 390 > 327	-40 -25
22	<sup>13</sup> C-1,2,3,7,8,9-HxCDD	29.87	402 > 338	-22	404 > 340	-22
23	1,2,3,7,8,9-HxCDD	29.87	390 > 327	-25	388 > 262 390 > 264	-40 -40
24	<sup>13</sup> C-1,2,3,7,8,9-HxCDF	30.21	386 > 322	-32	388 > 324	-32
25	1,2,3,7,8,9-HxCDF	30.22	372 > 309	-30	374 > 311 372 > 239	-30 -55
26	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	31.76	420 > 356	-35	422 > 358	-35
27	1,2,3,4,6,7,8-HpCDF	31.78	408 > 345	-35	410 > 347 408 > 275	-35 -55
28	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	33.15	436 > 372	-22	438 > 374	-22
29	1,2,3,4,6,7,8-HpCDD	33.15	424 > 361	-25	422 > 226 424 > 298	-70 -42
30	<sup>13</sup> C-1,2,3,4,7,8,9-HpCDF	33.87	420 > 356	-35	422 > 358	-35
31	1,2,3,4,7,8,9-HpCDF	33.9	408 > 345	-35	410 > 347 408 > 275	-35 -55
32	<sup>13</sup> C-OCDD	37.77	470 > 406	-25	472 > 408	-25
33	OCDD	37.78	458 > 395	-25	460 > 397 458 > 332	-25 -42
34	OCDF	38.04	444 > 381	-30	442 > 379 444 > 309	-30 -60

## Results

### Chromatographic separation and target identification

Using the EPA 1613 reference standard mix, the 17 target PCDD and PCDF compounds were easily separated and identified via the EVOQ GC-TQ workflow. Chromatographic separation of PCDD and PCDF isomers was very well-defined, with excellent peak symmetry for all native congeners, as shown in Figures 3 and 4. A minimum of two ions were detected for each target (Table 4).

### Retention time and ion ratio scoring, precision, and recovery

Method precision and recoveries were excellent for the targeted PCDDs/PCDFs using the EPA1613 method standards, easily meeting the established criteria. At the CS3 verification level, retention time variations (+/-) were less than 0.1 minute, and ion ratios were under 15%. Recoveries range from 94-107% (Figure 5). The color-coded RQ scoring of the Compass TQ software (Figure 6) enables rapid confirmation that the necessary data quality criteria for both retention time and ion ratio percentages (as established for each compound prior to analysis) were met.

### Calibration curve linearity

Using the isotope dilution method with <sup>13</sup>C labeled standards (in accordance with the current EU regulations), the calibration curves automatically generated within the Compass TQ software indicated excellent linearity across the monitored concentration range ( $R^2 > 0.99$ ,  $RSD < 13\%$ ) for all targeted compounds (Figure 7).

## LOD/LOQ

High method sensitivity is crucial to be able to confidently detect very low levels of dioxins in food and feed, as well as in environmental samples. Modern GC-MS/MS triple quadrupole systems, operating in MRM mode, generate very low background, with near-zero values for many transitions. Statistical calculations with near-zero values generate inaccurate estimates of limits of detection that are incongruous with the analytical reality. For this reason, Limit of Detection (LOD) and Limit of Quantitation (LOQ) have been experimentally determined by successive dilutions of the lowest calibration level.

The lowest calibration level within EPA1613 method standards was diluted 10-fold and 20-fold with n-nonane. With a 1  $\mu$ l injection, target compounds were easily detected in the lower femtogram ( $10^{-15}$  g) range, and LOQ criteria were met with  $S/N > 3$  for the least intense signals (Figure 8). For 2,3,7,8-TCDD, the LOD was determined to be 5 fg on-column, and the LOQ was 10 fg on-column.

Row	Analyte Name	Function Type	Origin	Weighting	Signal	R <sup>2</sup>	No. of data points	RSD RF
1	1,2,3,6,7,8-HxCDD	Linear	Ignore	1/x <sup>2</sup>	Area	0.99997	6	8.32
2	1,2,3,7,8-PCDD	Linear	Ignore	1/x <sup>2</sup>	Area	0.99995	6	8.69
3	2,3,4,7,8-PCDF	Linear	Ignore	1/x <sup>2</sup>	Area	0.99991	6	6.21
4	1,2,3,4,7,8-HxCDF	Linear	Ignore	1/x <sup>2</sup>	Area	0.99987	6	5.32
5	2,3,7,8-TCDD	Linear	Ignore	1/x <sup>2</sup>	Area	0.99971	6	8.64
6	1,2,3,4,6,7,8-HpCDF	Linear	Ignore	1/x <sup>2</sup>	Area	0.99970	6	11.3
7	OCDD	Linear	Ignore	1/x <sup>2</sup>	Area	0.99963	6	9.40
8	1,2,3,7,8,9-HxCDF	Linear	Ignore	1/x <sup>2</sup>	Area	0.99959	6	3.12
9	2,3,4,6,7,8-HxCDF	Linear	Ignore	1/x <sup>2</sup>	Area	0.99946	6	9.23
10	1,2,3,7,8-PCDF	Linear	Ignore	1/x <sup>2</sup>	Area	0.99932	5	4.46
11	1,2,3,6,7,8-HxCDF	Linear	Ignore	1/x <sup>2</sup>	Area	0.99925	6	7.94
12	2,3,7,8 (37Cl4)TCDD	Linear	Ignore	1/x <sup>2</sup>	Area	0.99899	6	5.31
13	OCDF	Linear	Ignore	1/x <sup>2</sup>	Area	0.99868	6	9.99
14	12,3,4,7,8,9-HpCDF	Linear	Ignore	1/x <sup>2</sup>	Area	0.99843	6	12.8
15	2,3,7,8-TCDF	Linear	Ignore	1/x <sup>2</sup>	Area	0.99825	6	8.64
16	1,2,3,7,8,9-HxCDD	Linear	Ignore	1/x <sup>2</sup>	Area	0.99785	6	5.80
17	1,2,3,4,6,7,8-HpCDD	Linear	Ignore	1/x <sup>2</sup>	Area	0.99544	6	12.9
18	1,2,3,4,7,8-HxCDD	Linear	Ignore	1/x <sup>2</sup>	Area	0.99227	6	8.98

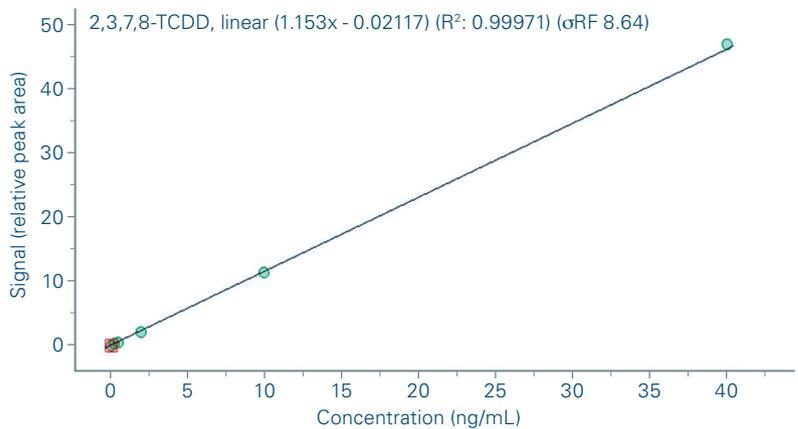


Figure 7: Calibration results for target PCDDs/PCDFs compounds, as shown within the Compass TQ software. An example calibration curve is shown for 2,3,7,8-TCDD (lower panel).

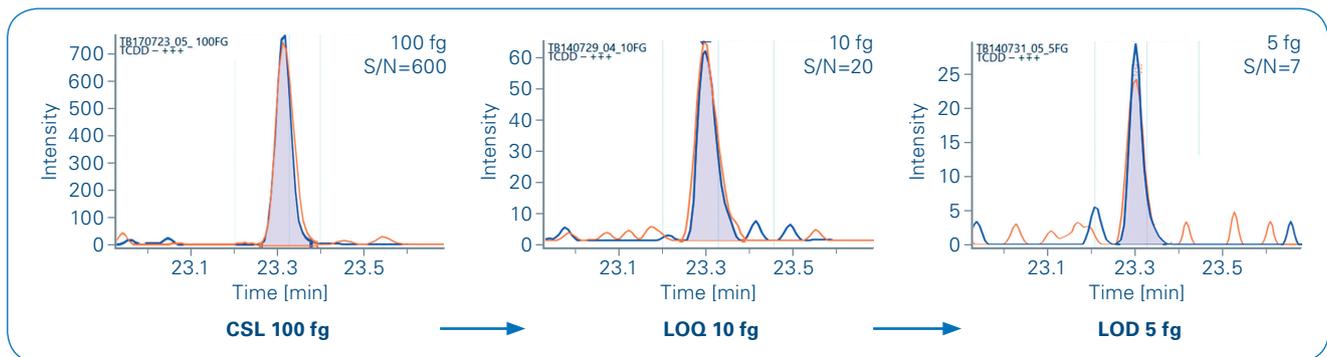


Figure 8: Method sensitivity for 2,3,7,8-TCDD (LOQ and LOD). Serial dilution from CSL standard level (on-column amounts).

## Analysis of certified milk powder

In analyses of certified milk powder (EC Joint Research Centre reference material BRC®-607), the concentrations of the target PCDDs/PCDFs obtained by the EVOQ GC-TQ system were accurate within (+/-) 20% of the product reference values (Table 5 and Figure 9) meeting current regulatory criteria. Further, values were comparable to those obtained by GC-HRMS.

## Analysis of doped animal feed

In the doped animal feed sample, all target PCDDs/PCDFs could be simultaneously detected via GC-MS/MS on the EVOQ GC-TQ system in the femtogram range (Figure 10). The % recoveries for the <sup>13</sup>C internal standards were within the 60-120% range necessary to meet current EU regulatory criteria (Figure 11).

Table 5: Summary of target compound determination in certified milk powder. Concentrations of each target PCDD or PCDF as indicated by product documentation, GC-HRMS, and GC-MS/MS are shown. The EVOQ GC-TQ method is accurate within +/- 20%, meeting regulatory criteria.

Compound	Certified value (ng/kg)	GC-HRMS (ng/kg)	GC-MS/MS (ng/kg)
2,3,7,8-TCDD	0.25	0.23	0.24
1,2,3,7,8-PeCDD	0.79	0.78	0.83
1,2,3,4,7,8-HxCDD	0.42	0.34	0.36
1,2,3,6,7,8-HxCDD	0.98	0.83	0.88
1,2,3,7,8,9-HxCDD	0.34	0.31	0.33
1,2,3,4,6,7,8-HpCDD	-	2.33	1.98
OCDD	-	10.29	9.03
2,3,7,8-TCDF	0.05	0.04	0.04
1,2,3,7,8-PeCDF	0.05	0.07	0.07
2,3,4,7,8-PeCDF	1.81	1.58	1.67
1,2,3,4,7,8-HxCDF	0.94	0.85	0.9
1,2,3,6,7,8-HxCDF	1.01	0.94	0.99
2,3,4,6,7,8-HxCDF	1.07	0.95	1.01
1,2,3,4,6,7,8-HpCDF	-	0.5	0.4
1,2,3,4,7,8,9-HpCDF	-	0.07	0.12
OCDF	-	0.17	0.34

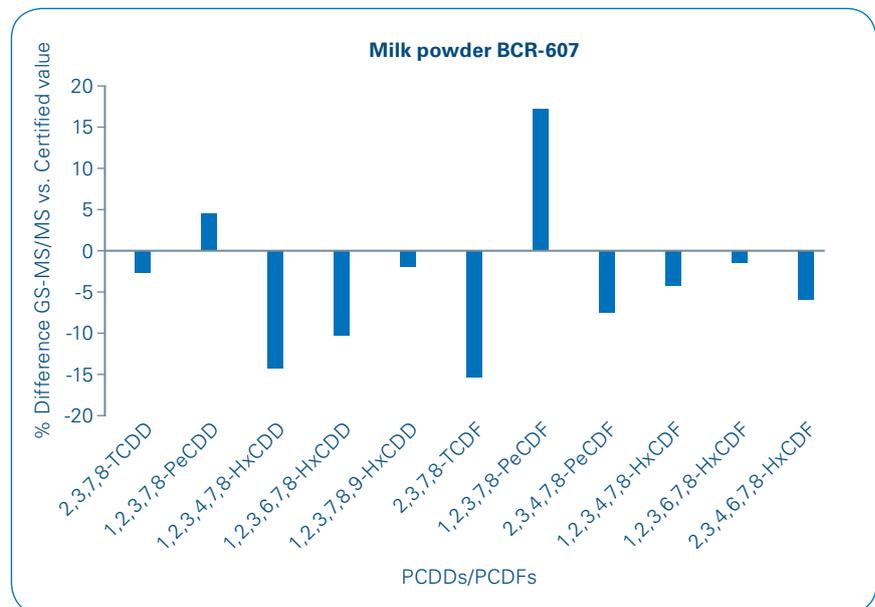


Figure 9: Difference in % of target PCDDs/PCDFs concentrations determined by GC-MS/MS versus the certified values in milk powder.

RQ	Score	Analyte Name	RT Score	Ions Score	Quantity [pg/g]
█	+++	2,3,7,8-TCDD	++	++	0.054
█	+++	2,3,7,8-TCDF	++	++	0.077
█	+++	1,2,3,7,8,9-HxCDD	++	++	0.149
█	+++	2,3,4,7,8-PCDF	++	++	0.218
█	+++	2,3,4,6,7,8-HxCDF	++	++	0.227
█	+++	1,2,3,7,8,9-HxCDF	++	++	0.236
█	+++	1,2,3,6,7,8-HxCDD	++	++	0.247
█	+++	1,2,3,7,8-PCDF	++	++	0.261
█	+++	1,2,3,4,7,8,9-HpCDF	++	++	0.264
█	+++	1,2,3,6,7,8-HxCDF	++	++	0.286
█	+++	1,2,3,4,6,7,8-HpCDF	++	++	0.300
█	+++	1,2,3,4,7,8-HxCDF	++	++	0.309
█	+++	1,2,3,7,8-PCDD	++	++	0.312
█	+++	1,2,3,4,7,8-HxCDD	++	++	0.337
█	+++	1,2,3,4,6,7,8-HpCDD	++	++	0.341
█	+++	OCDF	++	++	0.582
█	+++	OCDD	++	++	0.882

Figure 10: Automatic determination of PCDD/PCDFs concentrations in a doped animal feed sample via GC-MS/MS as summarized within the Compass TQ software.

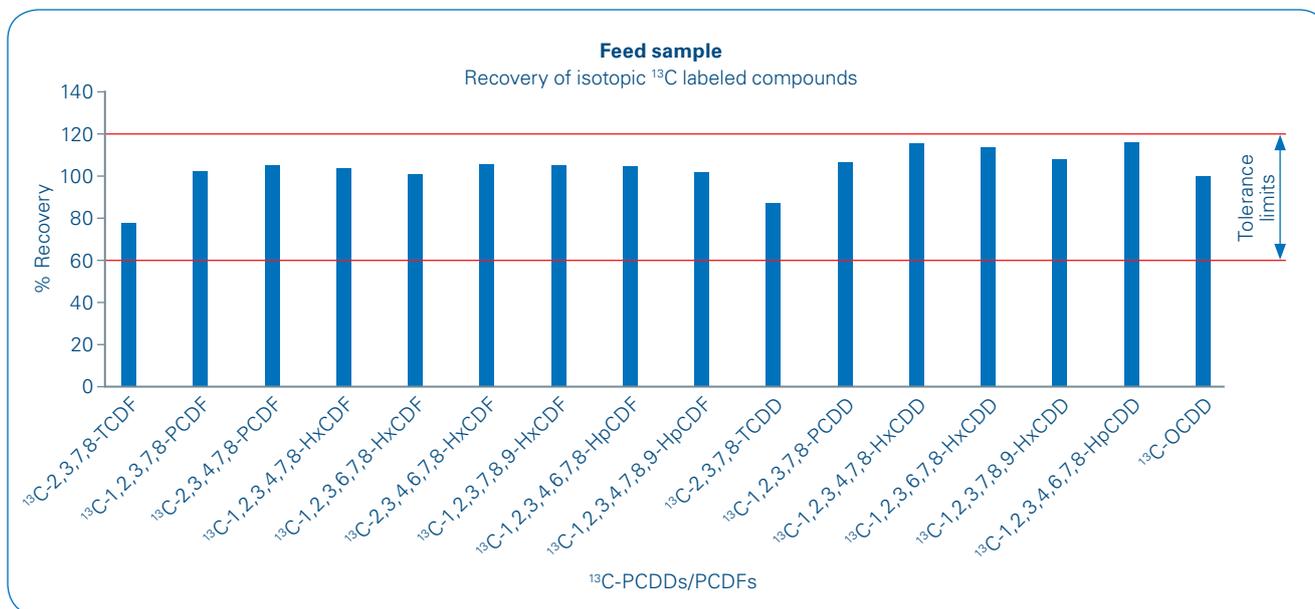


Figure 11: Recovery in % of doped PCDDs/PCDFs from an animal feed sample via GC-MS/MS.

Supplemental analyses – environmental samples

To further evaluate the EVOQ GC-TQ system, fly ash and sewage sludge (EC Joint Research Centre reference materials BCR®-615 and BCR®-677, respectively) were analyzed as a part of this study. As with the milk powder, the EVOQ GC-TQ method is accurate within +/- 20% and is comparable to data obtained via GC-HRMS (Table 6, Figure 12).

Table 6: Summary of target compound determination in certified reference environmental samples.

Compound	Fly Ash (BCR-615)			Sewage Sludge (BCR-677)		
	Certified value (ng/kg)	GC-HRMS (ng/kg)	GC-MS/MS (ng/kg)	Certified value (ng/kg)	GC-HRMS (ng/kg)	GC-MS/MS (ng/kg)
2,3,7,8-TCDD	27	31.5	25.8	1.5	1.2	1.3
1,2,3,7,8-PeCDD	92	97.5	108.6	4.1	3.3	3.7
1,2,3,4,7,8-HxCDD	74	69.9	70.7	-	2.3	1.6
1,2,3,6,7,8-HxCDD	103	100.4	100.9	235	213.5	221.7
1,2,3,7,8,9-HxCDD	108	104.7	89.0	79	79.5	65.7
1,2,3,4,6,7,8-HpCDD	870	845.6	885.4	3500	2887.2	3106.1
OCDD	1750	1907.5	2037.0	12,700	9932.7	12,675.7
2,3,7,8-TCDF	86	93.3	85.2	45.0	36.7	38.5
1,2,3,7,8-PeCDF	176	162.5	156.7	24.8	19.9	20.2
2,3,4,7,8-PeCDF	125	129.7	111.3	16.9	15.2	14.0
1,2,3,4,7,8-HxCDF	203	185.4	189.3	14.5	12.7	12.5
1,2,3,6,7,8-HxCDF	204	204.1	182.3	6.1	5.4	5.2
1,2,3,7,8,9-HxCDF	13.3	9.6	11.2	0.8	0.5	0.7
2,3,4,6,7,8-HxCDF	130	170.6	151.4	5.6	6.7	6.3
1,2,3,4,6,7,8-HpCDF	750	703.8	723.0	61.6	47.5	50.5
1,2,3,4,7,8,9-HpCDF	61	66.4	61.1	6.3	4.4	5.2
OCDF	290	274.7	318.3	177	129.2	145.8

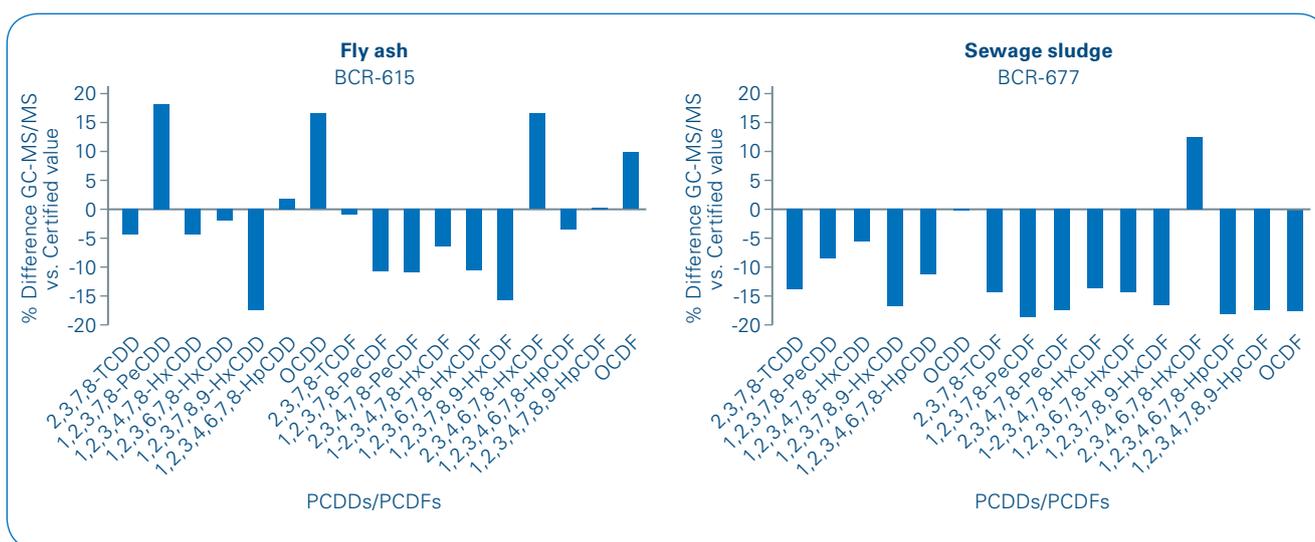


Figure 12: Difference in % of target PCDDs/PCDFs concentrations determined by GC-MS/MS versus the certified values in fly ash and sewage sludge.



Figure 13: Ion ratio stability of an example target compound (2,3,7,8-TCDD) throughout the study (left panel). 2,3,7,8-TCDD as detected in the milk powder reference sample at 0.24 ng/kg (right panel).

### Ion ratio stability

Ion ratio stability is an important factor for high confidence in regulatory compliance. Within the RQ score automatically determined for each target compound, the ion ratios for target compounds may be monitored across the complete batch of calibrants and samples. As shown in Figure 13, the ion ratio stability is excellent using the EVOQ GC-TQ system. For the 2,3,7,8-TCDD target, the average ion ratio was 0.64, with an RSD of 4.22% for the entire study. As previously indicated, all ion ratios were within the required +/- 15% tolerance.

## Discussion

Based on these results, the EVOQ GC-TQ system meets the current EU evaluation criteria [4,5] for the detection of PCDDs and PCDFs in food and feed. These persistent organic pollutant targets could be clearly differentiated via GC, and the assessment of MS/MS data subsequently obtained from each mixture component was rapidly and automatically made within the Compass TQ software. High accuracy in target identification and quantitation were demonstrated, and easily met the necessary evaluation criteria. The clear and confident detection of these compounds at very low levels, even within typically complex food and feed matrices, illustrates the high sensitivity of the method, as well as

its high analytical value considering the extremely high toxicity of many dioxins.

The detection accuracy of the EVOQ GC-TQ workflow was also demonstrated to be comparable to GC-HRMS in the analyses of certified milk, fly ash and sewage sludge samples, supporting the use of this system for diverse sample types to protect human and animal health.

Finally, the stability of the target ion ratios across the entire set of analyses – including a range of dioxin concentrations and potential matrix effects – supports the utility and reliability of this system for high confidence analyses to meet current regulatory demands.





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