



# Determination of Dioxin-Like and Non-Dioxin-Like Polychlorinated Biphenyl Congeners in Foodstuffs and Animal Feed Using the Agilent 7000 Triple Quadrupole GC/MS System

## Application Note

Food Safety

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### Abstract

Two methods have been developed on the Agilent 7000 Triple Quadrupole GC/MS system for the analysis of polychlorinated biphenyl (PCB) congeners in foodstuffs and animal feed. The methods were shown to give linear response over the required concentration ranges. In addition, quantitative results for dioxin-like PCB (dl-PCB) congeners down to low pg TEQ/g levels and non-dioxin-like PCB (ndl-PCB) congeners at levels below 1 ng/g product were in good agreement with values obtained using a GC-High Resolution mass spectrometer. This application note demonstrates the determination of the 12 dl-PCB comprising eight mono-*ortho* PCB congeners (# 105, 114, 118, 123, 156, 157, 167 and 189) and four non-*ortho* PCB congeners (# 77, 81, 126 and 169) as well as the six ndl-PCB congeners (# 28, 52, 101, 138, 153 and 180) that are also known as "Indicator PCB" congeners.



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## Introduction

Polychlorinated biphenyls (PCB) are highly toxic Persistent Organic Pollutants (POP) with properties that are detrimental to human health. They have been linked to cancer, endocrine disruption and reproductive disorders. Until their ban in the late 20th Century, PCBs were widely manufactured for use in hundreds of industrial and commercial applications including electrical products and hydraulic equipment and as plasticizers in paints, plastics and, rubber products. PCB congeners that have been released into the environment can bio-accumulate in animal tissues and thereby enter the human food chain.

Current legislation in the United States [1] and the European Union (EU) [2], [5] require the confirmation and quantitation of polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF) and dioxin-like polychlorinated biphenyl congeners (dl-PCB) in foodstuffs and animal feed by isotope dilution capillary gas chromatography–high resolution mass spectrometry (GC-HRMS). The analysis of dioxins and furans in foodstuffs and animal feed by gas chromatography-triple quadrupole mass spectrometry is shown in a previously published Agilent application note [3].

Maximum levels for PCDD, PCDF and dl-PCB congeners in foodstuffs and animal feed are given in additional EU regulations [4], [6]. dl-PCB congeners have each been assigned a Toxic Equivalency Factor (TEF) that relates the toxicity of each individual dl-PCB congener to 2,3,7,8-Tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), which itself is assigned a TEF of 1. The individual concentration of each dl-PCB found in foodstuffs and animal feed samples is multiplied by its respective TEF and after summation the total concentration is expressed as the Toxic Equivalent (TEQ) in terms of pg TEQ/g fat, pg TEQ/g fresh weight (fish) or ng TEQ/kg in 88% dry feed.

The World Health Organization (WHO) through the International Program on Chemical Safety (IPCS) originally established and then re-evaluated TEF for PCDD, PCDF and dl-PCB. Original TEF values were established by WHO/IPCS expert consultation in 1997 and re-evaluated in 2005. As a result, some TEF values have been changed and it is important to clearly state the set of TEF values used by indicating the year in which they were first expressed ( $TEF_{WHO98}$  or  $TEF_{WHO05}$ ) and the resultant  $TEQ_{WHO98}$  and  $TEQ_{WHO05}$  values. TEF values assigned to PCDD, PCDF and dl-PCB are shown in Table 1.

The maximum levels (based on  $TEF_{WHO98}$  values) for PCDD, PCDF and dl-PCB in certain foodstuffs as prescribed by EU legislation are given in Table 2.

Table 1. WHO Toxic Equivalency Factors ( $TEF_{WHO98}$  and  $TEF_{WHO05}$ ) for PCDD, PCDF and dl-PCB Congeners from Assessments Made in 1998 and 2005 (Changed Values in Italics)

Compound	TEF WHO <sub>98</sub>	TEF WHO <sub>05</sub>	Compound	TEF WHO <sub>98</sub>	TEF WHO <sub>05</sub>
<b>Chlorinated dibenzo-p-dioxins</b>			<b>Non-ortho substituted PCB</b>		
2378-TCDD	1	1	PCB-77	0.0001	0.0001
12378-PeCDD	1	1	PCB-81	0.0001	<i>0.0003</i>
123478-HxCDD	0.1	0.1	PCB-126	0.1	0.1
123678-HxCDD	0.1	0.1	PCB-169	0.01	<i>0.03</i>
123789-HxCDD	0.1	0.1			
1234678-HpCDD	0.01	0.01			
OCDD	0.0001	<i>0.0003</i>			
<b>Chlorinated dibenzofurans</b>			<b>Mono-ortho substituted PCB</b>		
2378-TCDF	0.1	0.1	PCB-105	0.0001	<i>0.00003</i>
12378-PeCDF	0.05	<i>0.03</i>	PCB-114	0.0005	<i>0.00003</i>
23478-PeCDF	0.5	<i>0.3</i>	PCB-118	0.0001	<i>0.00003</i>
123478-HxCDF	0.1	0.1	PCB-123	0.0001	<i>0.00003</i>
123678-HxCDF	0.1	0.1	PCB-156	0.0005	<i>0.00003</i>
234678-HxCDF	0.1	0.1	PCB-157	0.0005	<i>0.00003</i>
123789-HxCDF	0.1	0.1	PCB-167	0.00001	<i>0.00003</i>
1234678-HpCDF	0.01	0.01	PCB-189	0.0001	<i>0.00003</i>
1234789-HpCDF	0.01	0.01			
OCDF	0.0001	<i>0.0003</i>			

Table 2. Maximum Levels (Upperbound concentrations) for PCDD, PCDF and dl-PCB Congeners in Certain Foodstuffs, as Specified in EU Regulation (EC) No 1881/2006

Foodstuff	Maximum levels Sum of dioxins (WHO-PCDD/F-TEQ)	Maximum levels Sum of dioxins and dl-PCB (WHO-PCDD/F-PCB-TEQ)
Meat and meat products (excluding edible offal) of the following animals :		
- Bovine animals and sheep	3.0 pg/g fat	4.5 pg/g fat
- Poultry	2.0 pg/g fat	4.0 pg/g fat
- Pigs	1.0 pg/g fat	1.5 pg/g fat
- Raw milk and dairy products, including butter fat	3.0 pg/g fat	6.0 pg/g fat
- Hens eggs and egg products	3.0 pg/g fat	6.0 pg/g fat

The action levels for the sum of the 12 dl-PCB congeners in certain foodstuffs and certain feedstuffs are shown in Tables 3 and 4, respectively.

The six ndl-PCB congeners do not have TEF values and their results are expressed simply as the sum of the six individual congeners.

The chemical structures of the 12 dl-PCB congeners and 6 ndl-PCB congeners are shown in Figures 1 and 2, respectively.

This application note describes sensitive and reproducible methods for the screening of dl-PCB congeners and ndl-PCB congeners in foodstuffs and animal feed using the Agilent 7000 Triple Quadrupole GC/MS system that meets the requirements of EU Legislation for a screening method.

Table 3. Action Levels (Upperbound Concentrations) for the Sum of dl-PCB Congeners in Certain Foodstuffs, as Specified in EU Commission recommendation 2006/88

Food	Action level for dioxin-like PCBs (TEQ <sub>WHO98</sub> )
Meat and meat products of Ruminants (bovine animals, sheep) Poultry and farmed game Pigs	1.0 pg/g fat 1.5 pg/g fat 0.5 pg/g fat
Liver and derived products of terrestrial animals	4.0 pg/g fat
Muscle meat of fish and fishery products and products thereof with the exception of eel	3.0 pg/g fresh weight
Muscle meat of eel ( <i>Anguilla anguilla</i> ) and products thereof	6.0 pg/g fresh weight
Milk and milk products, including butter fat	2.0 pg/g fat
Hens eggs and egg products	2.0 pg/g fat

Table 4. Action Levels (Upperbound Concentrations) for the Sum of dl-PCB Congeners in Certain Feedstuffs, as Specified in EU Regulation 2002/32/EC

Product intended for animal feed	Action threshold relative to a feeding stuff with a moisture content of 12%
Feed materials of plant origin with the exception of vegetable oils and their byproducts	0.35 ng TEQ/kg
Vegetable oils and their byproducts	0.5 ng TEQ/kg
Feed materials of mineral origin	0.35 ng TEQ/kg
Animal fat, including milk fat and egg fat	0.75 ng TEQ/kg
Other land animal products including milk and milk products and eggs and egg products	0.35 ng TEQ/kg
Fish oil	14.0 ng TEQ/kg

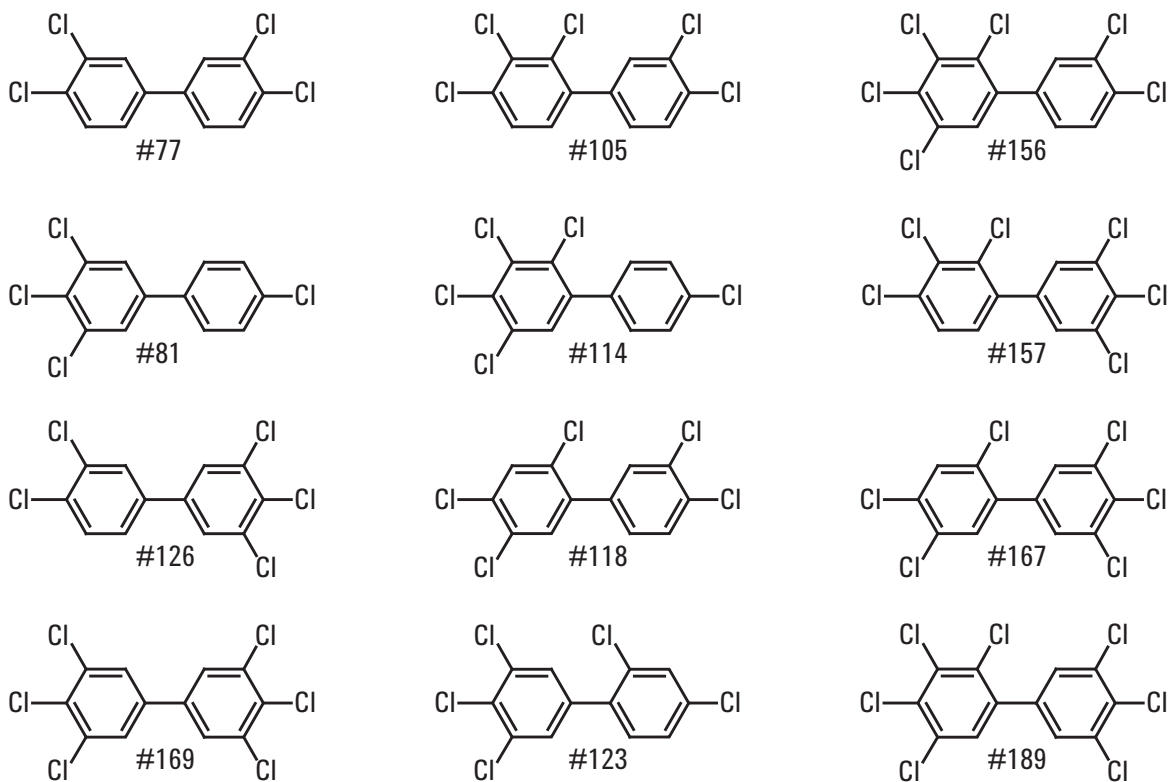


Figure 1. Chemical structures of dl-PCB congeners.

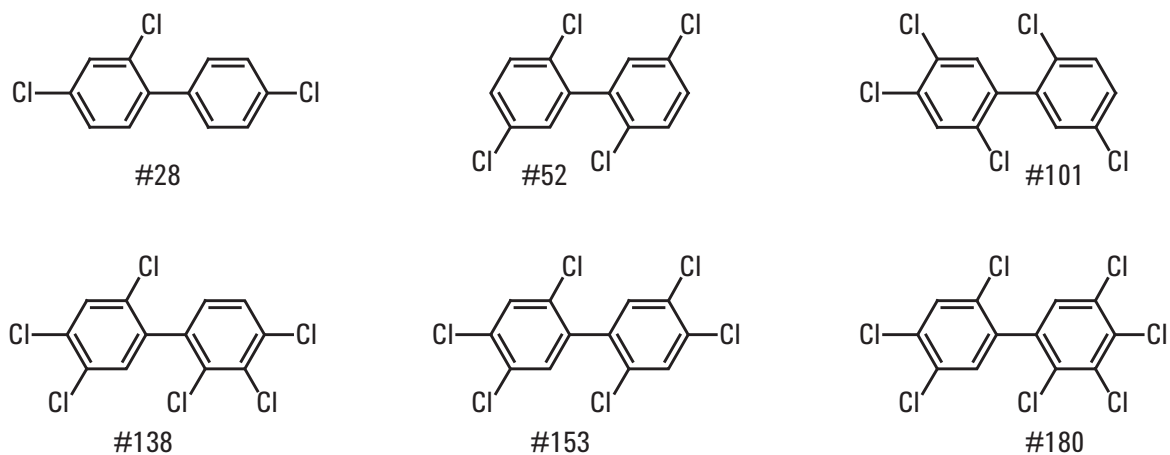


Figure 2. Chemical structures of ndl-PCB congeners.

## Experimental

### Calibration Standards

Calibration mixtures of native PCB congeners and their  $^{13}\text{C}$ -isotope labelled internal standards were obtained from Cambridge Isotope Laboratories and Wellington Laboratories Inc.

### Sample Preparation and Analysis

The most frequently used methods for the determination of PCDD, PCDF, dl-PCB congeners and ndl-PCB congeners in foodstuffs and animal feed combine fat extraction (for example, Soxhlet or extraction with organic solvents) with cleanup steps using different column chromatographies such as silica gel coated with sulfuric acid, florisil, alumina, and active carbon. The final extracts are collected as three fractions containing the mono-*ortho* PCB congeners and indicator PCB congeners (1a, Figure 3), non-*ortho* PCB congeners (1b, Figure 3) and PCDD/F (2, Figure 3), by eluting with various solvents. After addition of a syringe spike of  $^{13}\text{C}$ -labelled PCB internal standards, the extracts were evaporated under a gentle stream of nitrogen and subsequently reconstituted with toluene and analyzed with GC/MS/MS. The PCDD/F fraction was reconstituted with 20  $\mu\text{L}$  of toluene, the non-*ortho* PCB fraction with 40  $\mu\text{L}$  of toluene and the mono-*ortho*/indicator PCB fraction with 250  $\mu\text{L}$  of toluene.

A flow diagram summarizing the sample preparation steps is shown in Figure 3.

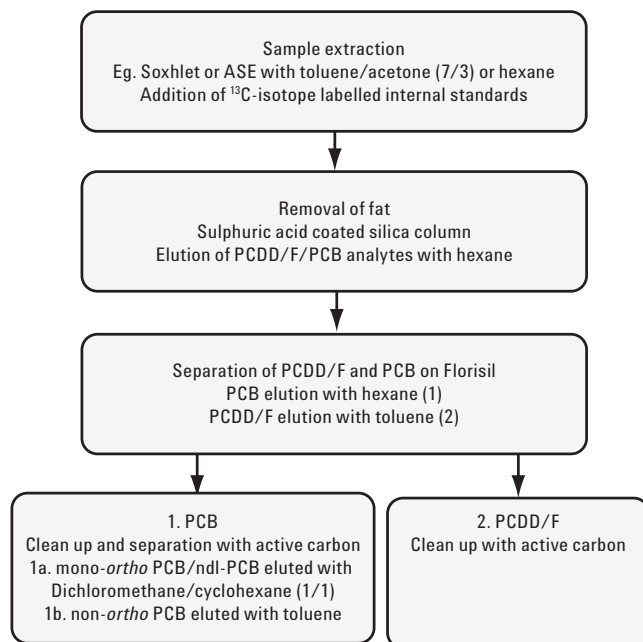


Figure 3. Flow diagram of the sample extraction and cleanup procedures.

The analyses were performed on an Agilent 7890 GC and an Agilent 7000 Triple Quadrupole GC/MS system. The 7890 Series GC was configured with a carbon dioxide cooled Multimode Inlet (MMI) and an HT-8 50 m  $\times$  0.22 mm, 0.25  $\mu\text{m}$  capillary column.

The GC instrument conditions for the mono-*ortho* PCB congeners are listed in Table 5 and the GC instrument conditions for the non-*ortho* PCB congeners are given in Table 6. MS parameters, common to both sets of PCB congeners, are shown in Table 7. The 7000 Triple Quadrupole GC/MS was operated in MS/MS-EI (electron ionization) Multiple Reaction Monitoring (MRM) mode. Each analyte and its associated  $^{13}\text{C}$ -Internal standard (ISTD) were measured using two different precursor ions and two different product ions.

Table 5. GC Conditions for Mono-*ortho* and dl-PCB Congeners

Column	HT-8 50 m $\times$ 0.22 mm id, 0.25 $\mu\text{m}$
Injection	2 $\mu\text{L}$ cold splitless using $\text{CO}_2$ cooled Multimode Inlet (MMI)
Injection port liner	4 mm id, unpacked
Inlet temperature program	100 $^\circ\text{C}$ (0.02 min), 500 $^\circ\text{C}/\text{min}$ to 300 $^\circ\text{C}$
Purge flow to split vent	50 mL/min at 1.0 min
Carrier gas	Helium, constant flow 1.2 mL/min
Oven program	80 $^\circ\text{C}$ (3.0 min hold), 20 $^\circ\text{C}/\text{min}$ to 160 $^\circ\text{C}$ (0 min), 4 deg $^\circ\text{C}/\text{min}$ to 300 $^\circ\text{C}$ (8 min), (Total run time = 50.0 minutes)
MS transfer line temp	280 $^\circ\text{C}$

Table 6. GC Conditions for Non-*ortho*-PCB Congeners

Column	HT-8 50 m $\times$ 0.22 mm id, 0.25 $\mu\text{m}$
Injection	2 $\mu\text{L}$ cold splitless using $\text{CO}_2$ cooled Multimode Inlet (MMI)
Injection port liner	4 mm id with glass wool
Inlet temperature program	100 $^\circ\text{C}$ (0.02 min), 500 $^\circ\text{C}/\text{min}$ to 300 $^\circ\text{C}$
Purge flow to split vent	50 mL/min at 1.0 min
Carrier gas	Helium, constant flow 1.2 mL/min
Oven program	120 $^\circ\text{C}$ (2.0 min hold), 40 $^\circ\text{C}/\text{min}$ to 160 $^\circ\text{C}$ (0 min), 7 deg $^\circ\text{C}/\text{min}$ to 300 $^\circ\text{C}$ (10 min), (Total run time = 33.0 minutes)
MS transfer line temp	280 $^\circ\text{C}$

Table 7. MS Setpoints for all PCB Congeners

Electron energy	-78 EV
Tune	EI Autotune
EM gain	100
MS1 resolution	Unit
MS2 resolution	Wide
Quant/Qual transitions	Table 8/Table 9
Dwell times	Table 8/Table 9
Collision energies	Table 8/Table 9
Collision cell gas flows	Nitrogen at 1.5 mL/min, Helium at 2.25 mL/min
MS temperatures	Ion source 280 $^\circ\text{C}$ , MS1 150 $^\circ\text{C}$ , MS2 150 $^\circ\text{C}$

A full list of the analyte retention times, MRM settings and dwell times for the mono-*ortho* and ndl-PCB congeners and the non-*ortho* PCB congeners are given in Tables 8 and 9, respectively.

An Agilent 7693A Automatic Liquid Sampler with the sampler tray cooled to 5 °C was used and 2 µL cold splitless injections were made using a 10-µL syringe.

Table 8. MS/MS Settings for Native Mono-Ortho and ndl-PCB Congeners (ndl-PCB Congeners Shown in Bold Italics) and <sup>13</sup>C-Internal Standards

TS	Segment start time (min)	Analyte	RT (min)	Quant pre-cursor	Product	Dwell (ms)	CE (V)	Qual pre-cursor	Product	Dwell (ms)	CE (V)
1	22.0	<i><sup>13</sup>C-PCB 28</i>	<b>24.34</b>	<b>268.0</b>	<b>198.1</b>	<b>25</b>	<b>26</b>	<b>270.0</b>	<b>198.1</b>	<b>25</b>	<b>26</b>
		<b>PCB 28</b>	<b>24.35</b>	<b>256.0</b>	<b>186.0</b>	<b>75</b>	<b>26</b>	<b>258.0</b>	<b>186.0</b>	<b>75</b>	<b>26</b>
		<i><sup>13</sup>C-PCB 52</i>	<b>25.66</b>	<b>302.0</b>	<b>232.0</b>	<b>25</b>	<b>28</b>	<b>304.0</b>	<b>234.0</b>	<b>25</b>	<b>28</b>
		<b>PCB 52</b>	<b>25.67</b>	<b>289.9</b>	<b>220.0</b>	<b>75</b>	<b>28</b>	<b>291.9</b>	<b>222.0</b>	<b>75</b>	<b>28</b>
2	29.0	<i><sup>13</sup>C-PCB 101</i>	<b>30.15</b>	<b>335.9</b>	<b>266.0</b>	<b>25</b>	<b>28</b>	<b>337.9</b>	<b>268.0</b>	<b>25</b>	<b>28</b>
		<b>PCB 101</b>	<b>30.16</b>	<b>323.9</b>	<b>253.9</b>	<b>75</b>	<b>28</b>	<b>325.9</b>	<b>255.9</b>	<b>75</b>	<b>28</b>
		<sup>13</sup> C-PCB 123	33.55	335.9	266.0	25	28	337.9	268.0	25	28
		PCB 123	33.56	323.9	253.9	75	28	325.9	255.9	75	28
		<sup>13</sup> C-PCB 118	33.76	335.9	266.0	25	28	337.9	268.0	25	28
		PCB 118	33.77	323.9	253.9	75	28	325.9	255.9	75	28
		<sup>13</sup> C-PCB 141	34.00	371.9	301.9	25	28	369.9	299.9	25	28
		<sup>13</sup> C-PCB 114	34.19	335.9	266.0	25	28	337.9	268.0	25	28
		PCB 114	34.20	323.9	253.9	75	28	325.9	255.9	75	28
		<i><sup>13</sup>C-PCB 153</i>	<b>34.50</b>	<b>371.9</b>	<b>301.9</b>	<b>25</b>	<b>28</b>	<b>369.9</b>	<b>299.9</b>	<b>25</b>	<b>28</b>
		<b>PCB 153</b>	<b>34.51</b>	<b>359.8</b>	<b>289.9</b>	<b>75</b>	<b>28</b>	<b>357.8</b>	<b>287.9</b>	<b>75</b>	<b>28</b>
		<sup>13</sup> C-PCB 105	35.15	335.9	266.0	25	28	337.9	268.0	25	28
		PCB 105	35.16	323.9	253.9	75	28	325.9	255.9	75	28
		<i><sup>13</sup>C-PCB 138</i>	<b>35.88</b>	<b>371.9</b>	<b>301.9</b>	<b>25</b>	<b>28</b>	<b>369.9</b>	<b>299.9</b>	<b>25</b>	<b>28</b>
		<b>PCB 138</b>	<b>35.89</b>	<b>359.8</b>	<b>289.9</b>	<b>75</b>	<b>28</b>	<b>357.8</b>	<b>287.9</b>	<b>75</b>	<b>28</b>
		<sup>13</sup> C-PCB 167	37.64	371.9	301.9	25	28	369.9	299.9	25	28
PCB 167	37.65	359.8	289.9	75	28	357.8	287.9	75	28		
3	38.5	<sup>13</sup> C-PCB 156	38.78	371.9	301.9	25	28	369.9	299.9	25	28
		PCB 156	38.79	359.8	289.9	75	28	357.8	287.9	75	28
		<sup>13</sup> C-PCB 157	39.06	371.9	301.9	25	28	369.9	299.9	25	28
		PCB 157	39.07	359.8	289.9	75	28	357.8	287.9	75	28
		<i><sup>13</sup>C-PCB 180</i>	<b>39.17</b>	<b>407.8</b>	<b>337.9</b>	<b>25</b>	<b>30</b>	<b>405.8</b>	<b>335.9</b>	<b>25</b>	<b>30</b>
		<b>PCB 180</b>	<b>39.18</b>	<b>393.8</b>	<b>323.9</b>	<b>75</b>	<b>30</b>	<b>395.8</b>	<b>325.9</b>	<b>75</b>	<b>30</b>
		<sup>13</sup> C-PCB 189	42.43	407.8	337.9	25	30	405.8	335.9	25	30
		PCB 189	42.44	393.8	323.9	75	30	395.8	325.9	75	30

Table 9. MS/MS Settings for Native Non-Ortho PCB Congeners and <sup>13</sup>C-Internal Standards

TS	Segment start time (min)	Analyte	RT (min)	Quant pre-cursor	Product	Dwell (ms)	CE (V)	Qual pre-cursor	Product	Dwell (ms)	CE (V)
1	19.0	<sup>13</sup> C-PCB 81	20.74	301.9	232.0	25	28	303.9	234.0	25	28
		PCB 81	20.75	289.9	220.0	125	28	291.9	222.0	125	28
		<sup>13</sup> C-PCB 77	21.12	301.9	232.0	25	28	303.9	234.0	25	28
		PCB 77	21.13	289.9	220.0	125	28	291.9	222.0	125	28
2	22.0	<sup>13</sup> C-PCB 126	23.55	335.9	265.9	25	28	337.9	267.9	25	28
		PCB 126	23.56	323.9	253.9	125	28	325.9	255.9	125	28
3	25.0	<sup>13</sup> C-PCB 169	26.26	371.9	301.9	25	28	369.9	299.9	25	28
		PCB 169	26.27	359.9	289.9	125	28	357.8	287.9	125	28

## Results and Discussion

### Chromatography

The multiple reaction monitoring (MRM) chromatograms for the native mono-*ortho* and ndl-PCB congeners, with an analysis time of 50 minutes, are shown in Figure 4. The multiple reaction monitoring (MRM) chromatograms for the native non-*ortho* PCB congeners, with an analysis time of 33 minutes, are shown in Figure 5.

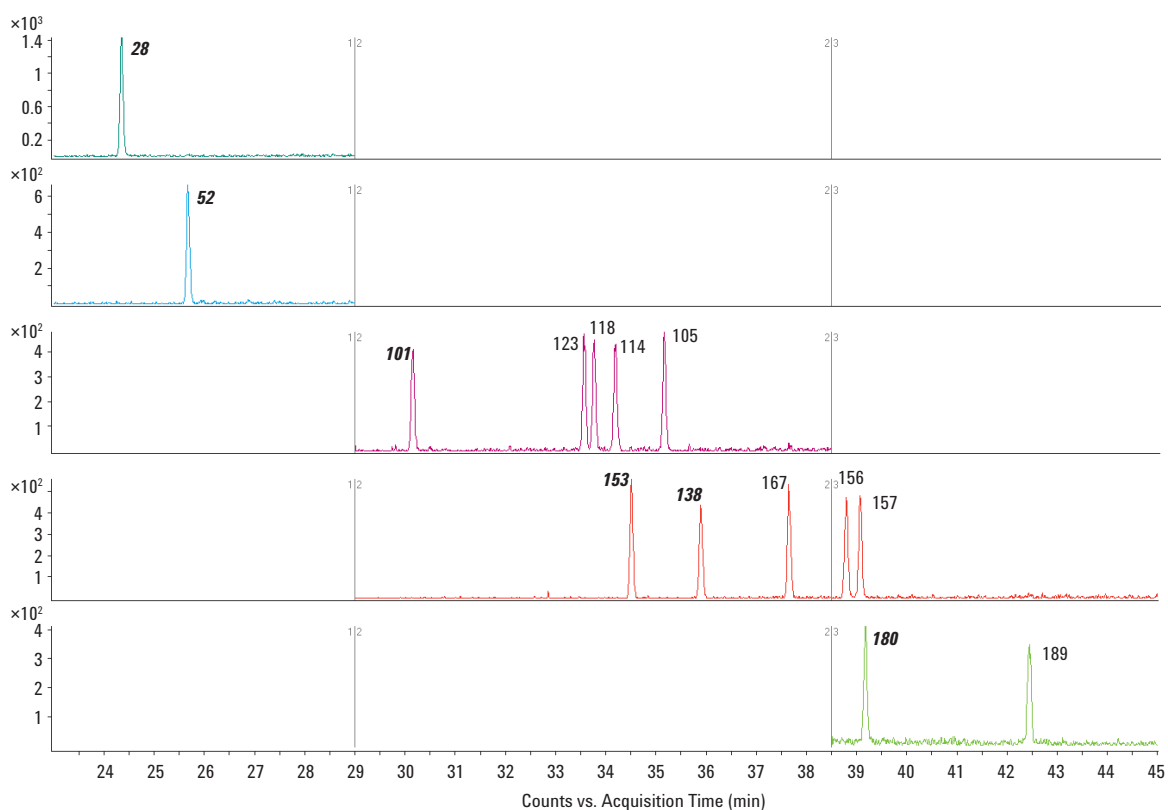


Figure 4. MRM chromatograms of native Mono-*ortho* and ndl-PCB congeners (ndl-PCB congeners labelled in bold italics).

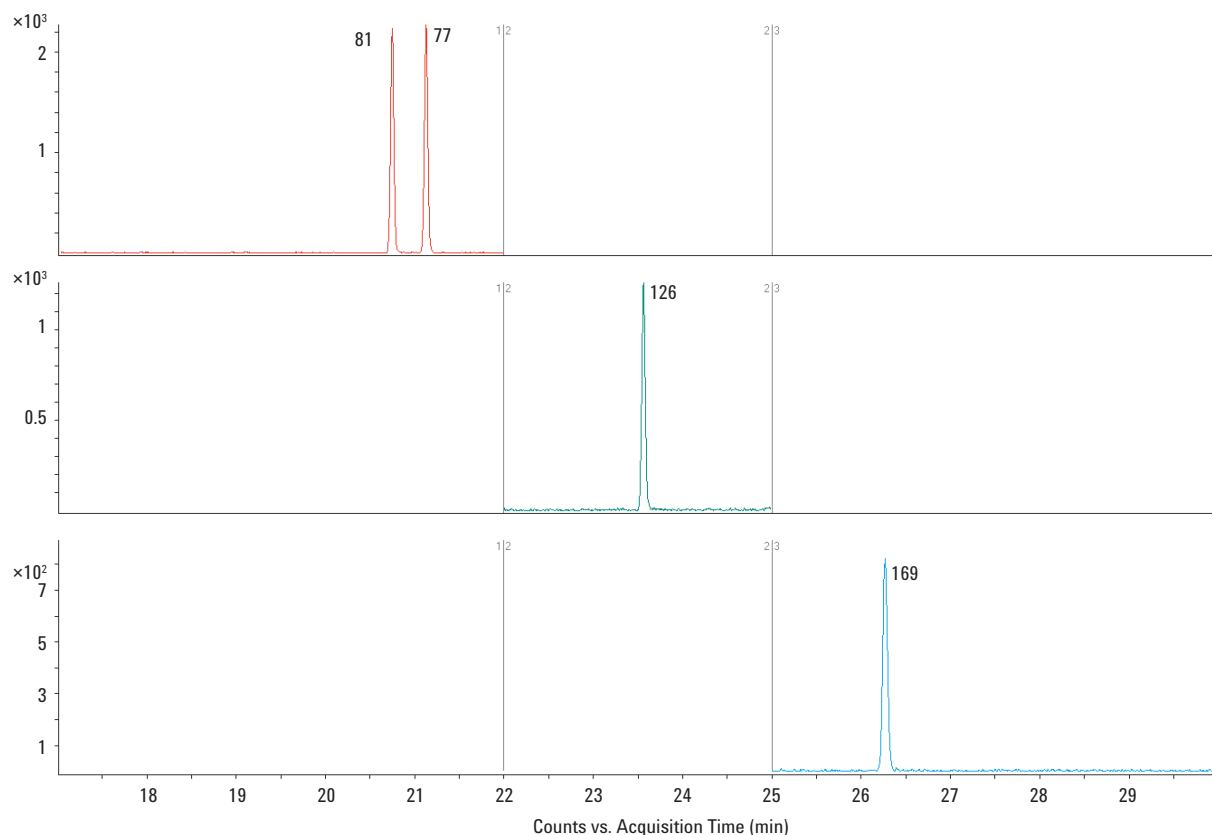


Figure 5. MRM chromatograms of native Non-ortho PCB congeners.

## Linearity of Response

All PCB congeners were measured using  $^{13}\text{C}$ -labelled internal standard (ISTD) calibration. Seven-point ISTD calibration curves were created using calibration standard solutions at the concentrations given in Tables 10 and 11, respectively.

Table 10. Concentration of Native Mono-Ortho and ndI-PCB Congeners and their  $^{13}\text{C}$ -ISTD Calibration Standards

Mono-ortho PCB	Natives pg/ $\mu\text{L}$	$^{13}\text{C}$ pg/ $\mu\text{L}$	$^{13}\text{C}$ (PCB 180, 153, 138 141 = recovery) pg/ $\mu\text{L}$
M1	0.05	5.00	50.0
M2	0.15	5.00	50.0
M3	0.50	5.00	50.0
M4	1.50	5.00	50.0
M5	5.00	5.00	50.0
M6	15.00	5.00	50.0
M7	50.00	5.00	50.0

Table 11. Concentration of Native Non-Ortho PCB Congeners and their  $^{13}\text{C}$ -ISTD Calibration Standards

Non-Ortho PCB	Natives pg/ $\mu\text{L}$	$^{13}\text{C}$ pg/ $\mu\text{L}$
C1	0.10	2.50
C2	0.25	2.50
C3	0.50	2.50
C4	1.00	2.50
C5	2.50	2.50
C6	5.00	2.50
C7	10.00	2.50

Excellent linearity was obtained over the required concentration range for all the PCB congeners and example calibration curves for PCB 126 and PCB 169 are shown in Figures 6 and 7, respectively.



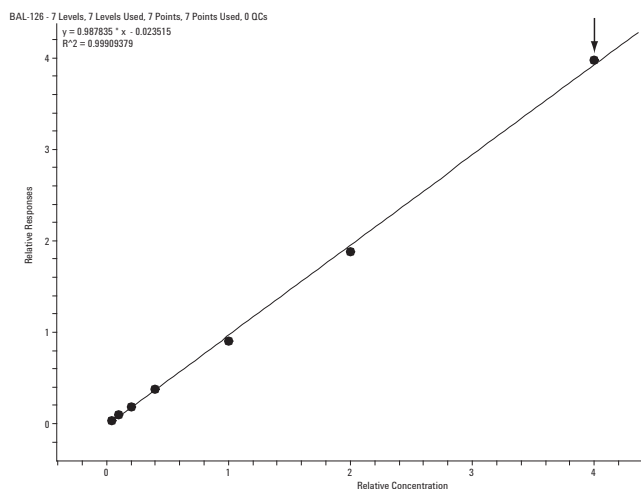


Figure 6. 7-point ISTD calibration curve for PCB 126 with linear fit.

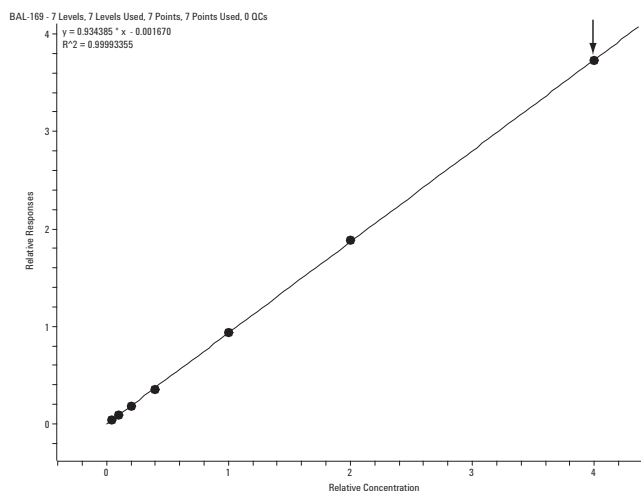


Figure 7. Seven-point ISTD calibration curve for PCB 169 with linear fit.

The linear calibration curve fits for all the PCB congeners are shown in Table 12. All analytes gave linear curve fit coefficients ( $R^2$ ) greater than 0.998.

## Sample Analysis

Eighty samples of four different foodstuffs and animal feed: animal feed ( $n = 45$ ), cows' milk ( $n = 11$ ), meat ( $n = 19$ ) and liver ( $n = 5$ ) were extracted and analyzed using a GC-High Resolution Mass Spectrometer (GC-HRMS) at a resolution of

Table 12. Linear Correlation Coefficients for Seven-Point ISTD Calibration Curves Over the Range  $0.05 \text{ pg}/\mu\text{L} - 50 \text{ pg}/\mu\text{L}$  for Mono-Ortho and ndl-PCB Congeners and  $0.1 \text{ pg}/\mu\text{L} - 10 \text{ pg}/\mu\text{L}$  for Non-Ortho PCB Congeners, Injection Volume =  $2 \mu\text{L}$

Mono-ortho PCB	$R^2$	Non-ortho PCB	$R^2$
PCB 28	0.9999	PCB 81	0.9992
PCB 52	0.9993	PCB 77	0.9991
PCB 101	0.9991	PCB 126	0.9991
PCB 123	0.9997	PCB 169	0.9999
PCB 118	0.9994		
PCB 114	0.9998		
PCB 153	0.9997		
PCB 105	0.9999		
PCB 138	0.9993		
PCB 167	0.9988		
PCB 156	0.9985		
PCB 157	0.9987		
PCB 180	0.9995		
PCB 189	0.9990		

$R = 10,000$ . The same sample vials were then transferred to the Agilent 7000 Triple Quadrupole GC /MS system and reanalyzed.

Figure 8 shows the comparative sample results (total TEQ-dl-PCB, upperbound values) of the two sets of measurements expressed as the percentage difference between the results obtained by the GC-HRMS and GC/MS/MS analyses.

The agreement between the results obtained for the total of the 12 dl-PCB congeners on the GC-HRMS and the GC/MS/MS system for foodstuffs and animal feed samples at levels above  $1 \text{ pg TEQ}/\text{g}$  were within the range of  $\pm 10\%$ .

The comparative results for the 68 foodstuffs and animal feed samples that gave total dl-PCB results less than  $1.2 \text{ TEQ pg}/\text{g}$  are shown in Figure 9.

The agreement between the results obtained for the sum of the 12 dl-PCB congeners on the GC-HRMS and the GC/MS/MS system for foodstuffs and animal feed samples at levels between  $0.1$  and  $1 \text{ pg TEQ}/\text{g}$  was within the range of  $\pm 15\%$ . Only those animal feed samples with total dl-PCB congener concentrations below  $0.1 \text{ TEQ pg}/\text{g}$  gave some results with percentage differences greater than  $15\%$ .

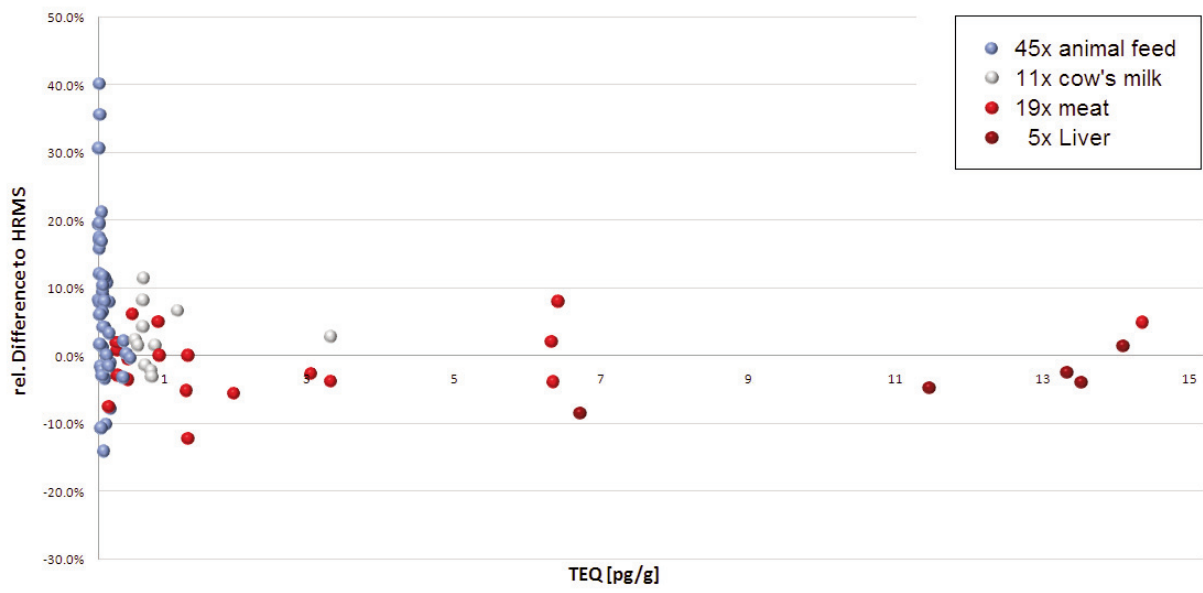


Figure 8. Comparative results for the sum of the 12 dl-PCB congeners (TEQ<sub>WHO98</sub> upperbound values) for 80 foodstuffs and animal feed samples analyzed by GC-HRMS and GC/MS/MS.

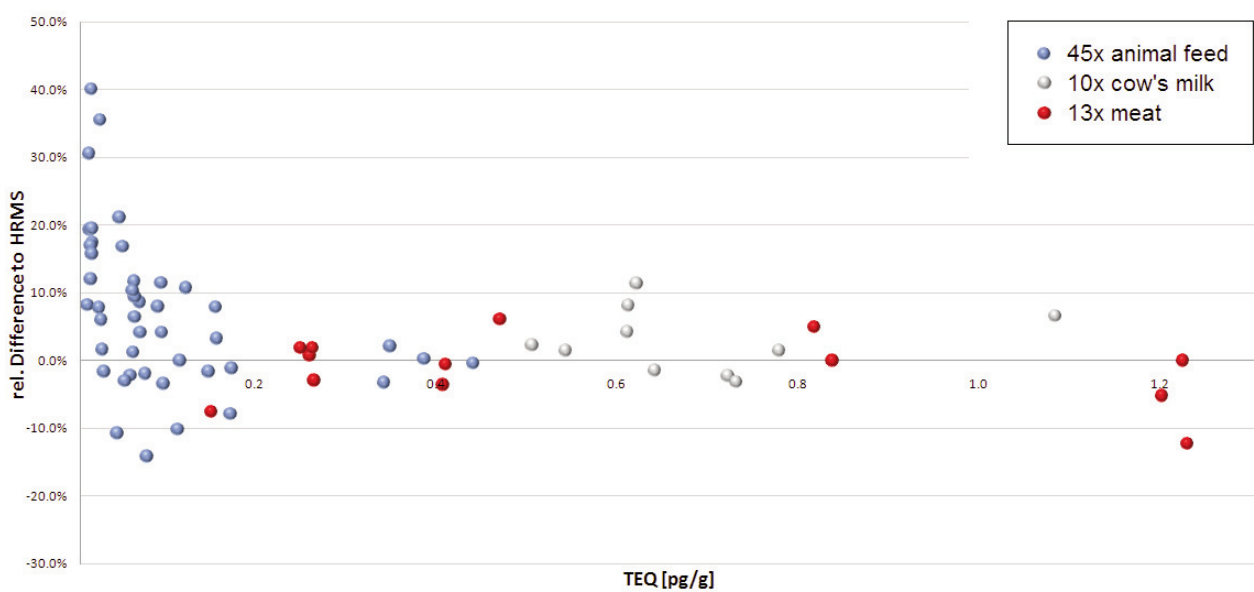


Figure 9. Comparative results for the sum of the 12 dl-PCB congeners (TEQ<sub>WHO98</sub> upperbound values) for 68 foodstuffs and animal feed samples analyzed by GC-HRMS and GC/MS/MS that gave values less than ~1.2 pg TEQ/g product.

Figure 10 shows the comparative sample results (total ndl-PCB congeners, upperbound values) of the two sets of measurements expressed as the percentage difference between the results obtained by the GC-HRMS and GC/MS/MS analyses.

The agreement between the sum of the results obtained for the six ndl-PCB congeners on the GC-HRMS and the GC/MS/MS for foodstuffs and animal feed samples at levels between 0.5 and 10 ng/g was within the range of  $\pm 10\%$ . Some animal feed samples with total ndl-PCB congener concentrations below 0.5 ng/g gave results with percentage differences greater than + 10%.

## Conclusion

The Agilent 7000 Triple Quadrupole GC/MS system provides linear, reproducible and sensitive detection of dl-PCB

congeners in foodstuffs and animal feed samples down to low pg TEQ/g values. Comparison of analytical results for foodstuffs and animal feed samples by GC-HRMS and GC/MS/MS indicates the suitability of the Agilent 7000 Triple Quadrupole GC/MS system for the routine screening of dl-PCB congeners in foodstuffs and animal feed that meets the requirements of European Union legislation.

Additionally, the Agilent 7000 Triple Quadrupole GC/MS system has been shown to determine total ndl-PCB congeners in foodstuffs and animal feed samples at concentration levels of 1 ng/g product and below, which is also in good agreement with results obtained by GC-HRMS.

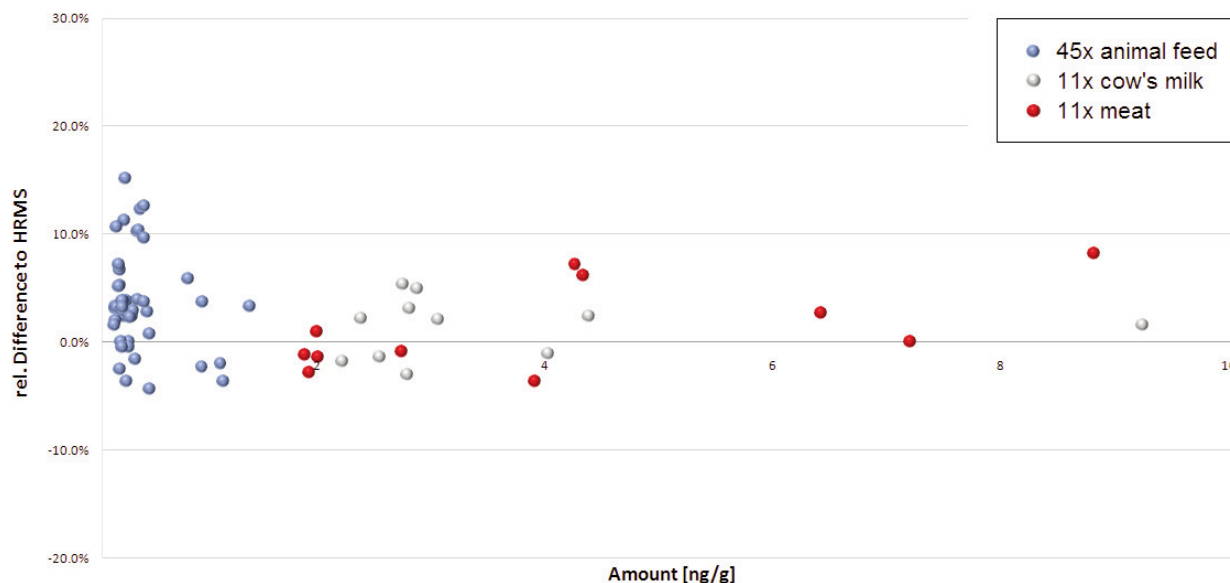


Figure 10. Comparative results for the sum of ndl-PCB congeners (upperbound values) for 67 foodstuffs and animal feed samples analyzed by GC-HRMS and GC/MS/MS that gave values less than 10 ng/g product.

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4. Commission Regulation (EC) No 1881/2006 of December 19, 2006 Setting maximum levels for certain contaminants in foodstuffs.
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6. Directive 2002/32/EC of the European Parliament and of the Council of 7 May 2002, on undesirable substances in animal feed.

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