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Levels of Polychlorinated Biphenyls in Foods from Catalonia, Spain: Estimated Dietary Intake

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ABSTRACT

From June to August 2000, food samples were randomly acquired in seven cities in Catalonia, Spain. Polychlorinated biphenyl (PCB) concentrations were determined for 108 samples of vegetables, fruits, pulses, cereals, fish and shellfish, meats and meat products, eggs, milk and dairy products, and oils and fats. Levels of 11 PCB congeners (IUPAC 28, 52, 77, 101, 105, 118, 126, 138, 153, 169, and 180) were determined by high-resolution gas chromatography–high-resolution mass spectrometry. For toxic equivalent (TEQ) calculations, World Health Organization (WHO) toxicity equivalent factors (WHO-TEFs) were used. The highest levels of most congeners were found in fish and shellfish (11,864.18 ng/kg [wet weight]), and the next highest levels, which were substantially lower, were found in milk and dairy products (674.50 ng/kg [wet weight]). For the general population of Catalonia, the total dietary intake of PCBs was found to be 150.13 pg WHO-TEQ/day. The largest contribution to this intake came from fish and shellfish (82.87 pg WHO-TEQ/day) and dairy products (29.38 pg WHO-TEQ per day). A relatively large contribution was also noted for cereals (11.36 pg WHO-TEQ/day). Among the PCB congeners determined in this study, PCB 126 showed the largest contribution to total TEQ intake (50.56%). The data obtained in this study should be useful in risk assessment with regard to human PCB exposure through food in Catalonia.

Polychlorinated biphenyls (PCBs) are complex mixtures of chlorinated aromatic hydrocarbons that have relatively low water solubility levels and prolonged environmental and biological half-lives. PCBs comprise a family of 209 possible congeners ranging from three monochlorinated isomers to the fully chlorinated decachlorobiphenyl isomer. These organic chemicals are ubiquitous in the environment. They can be found in the adipose tissue, blood, and milk of the general population. Humans are exposed to PCBs primarily through the ingestion of foods contaminated with commercial mixtures of these compounds *(12, 15, 18)*.

PCBs have been used as dielectric fluids in transformers and large capacitors, as pesticide extenders, and as heat exchange fluids and flame retardants and have also been used in many other different industrial applications *(21)*. The wide use of PCB mixtures over decades eventually led to significant levels of the most common congeners in a large number of environmental samples. Although PCB production was banned in industrialized countries during the 1970s and 1980s, these pollutants can still enter the environment through leaks from industrial facilities, through recycling processes, and through transboundary influx via major rivers and long-range atmospheric transport *(16, 21)*.

Commercial PCB mixtures elicit a broad spectrum of biochemical and toxic responses, with most of these adverse effects being similar to those caused by 2,3,7,8-tetrachlorodibenzo-*p*-dioxin *(10, 16)*. Health effects that have been associated with exposure to PCBs include acne-like skin conditions in adults and neurobehavioral and immunological changes in children *(1)*. In animals, exposure to PCBs has been linked to reproductive dysfunction, immune suppression, and behavioral and learning disturbances *(10, 14)*. In addition, PCBs are known to cause cancer in animals *(1)*.

Although human exposure to PCBs can occur by various routes, food is the primary source. Since PCBs are lipophilic, they tend to accumulate mainly in the fatty tissues of animals. Recently, the World Health Organization (WHO) identified 12 PCB congeners whose toxicity levels are similar to those of polychlorinated dibenzo-*p*-dioxins (PCDDs) and dibenzofurans (PCDFs) and assigned them toxicity equivalent factors (TEFs) for the calculation of toxic equivalents (TEQ) *(22, 24)*. Dioxin-like PCBs make an important contribution to total TEQ in many environmental media, especially fish and animal products, which are widely consumed by humans. PCBs seem to be even more important than PCDD-PCDF with regard to human health risks associated with exposure to dioxins and dioxin-like compounds *(7, 9, 11, 19)*. Episodes such as the Belgian dioxin and PCB contamination of feedstuffs in 1999 *(23)* or the Yusho (Japan) and Yu-Cheng (Taiwan) exposure to PCBs *(2)* from the ingestion of contaminated rice oil show that PCBs can pose an important risk of accidental contamination of the food chain.

Recently, we analyzed the levels of PCDD-PCDF in

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TABLE 1. *Polychlorinated biphenyl (PCB) concentrations in food samples from Catalonia, Spain*

Congener	PCB concn in food samples[a]							
	Vegetables and tubercles (n = 20)	Pulses and cereals (n = 12)	Fruits (n = 12)	Fish and shellfish (n = 16)	Meat (n = 30)	Eggs (n = 4)	Milk and dairy products (n = 8)	Fats and oils (n = 6)
PCB 28	2.61	33.31	0.77	102.89	48.66	36.17	52.10	93.17
PCB 52	2.98	13.99	0.74	275.43	24.10	11.19	69.10	123.00
PCB 77	0.27	0.65	0.13	18.62	1.55	1.32	3.24	5.16
PCB 101	3.49	17.22	0.74	593.99	13.66	7.57	41.18	65.17
PCB 105	0.52	<1.67	0.47	219.86	5.01	12.64	18.53	10.52
PCB 118	1.75	8.91	0.69	877.85	24.38	53.17	70.39	31.00
PCB 126	<0.05	<0.33	<0.07	7.32	<0.40	0.16	1.23	2.28
PCB 138	3.93	26.62	0.99	3,202.40	88.31	120.12	131.58	33.50
PCB 153	3.84	14.43	0.94	4,757.45	111.50	136.48	191.58	40.50
PCB 169	<0.09	<0.72	<0.14	5.70	<0.46	<0.23	1.02	2.73
PCB 180	2.13	13.44	0.72	1,802.79	56.13	96.75	94.70	45.33
All PCBs	21.09	125.90	4.46	11,864.18	373.55	475.18	674.50	451.53
WHO-TEQ	6.03	41.86	8.79	900.77	47.85	24.82	142.68	260.33

[a] Results for congeners are given in nanograms per kilogram (wet weight). WHO-TEQ values, calculated on the basis of levels of congeners PCB 77, PCB 105, PCB 118, PCB 126, and PCB 169, for which WHO-TEFs have been established, are given in picograms per kilogram (wet weight).

foods and estimated the dietary intake of these pollutants for the population of Catalonia, Spain *(3)*. Taking into account the importance of PCBs in establishing TEQ, the present study was undertaken to determine current PCB concentrations in food samples collected in various areas of Catalonia and to estimate exposure to PCBs through the diet. The results obtained in this study were compared with data from a number of recent international surveys.

MATERIALS AND METHODS

In the summer of 2000, food samples were randomly obtained from local markets, big supermarkets, and grocery stores in seven cities (Barcelona, Tarragona, Lleida, Girona, L'Hospitalet de Llobregat, Badalona, and Terrassa) in Catalonia, Spain, with populations of between 150,000 and 1,800,000 people. Samples were collected for two food groups. The first group included beef (steak and hamburger), pork (loins and sausage), and chicken (breasts); lamb (steak), white fish (hake), blue fish (sardines), and shellfish (mussel); vegetables (lettuce, tomatoes, potatoes, green beans, and cauliflower); fresh fruits (apples, oranges, and pears); and eggs. The second group included cow's milk (whole and semi-skimmed) and dairy products (yogurt and cheese), cereals (bread, pasta, and rice), pulses (lentils and beans), fats (margarine) and oils (olive and sunflower), tinned fish (tuna and sardines), and meat products (ham, hot dogs, and salami). Because products in the first group are usually retailed, their origins could be very diverse for the different cities. Therefore, for this group, four composite samples were analyzed for each food item. Each composite was made up of 10 individual samples of similar weights. These samples were collected at five different locations in the same city (two samples per location). In contrast, most food items in the second group were marketed under brands or trademarks that can be obtained in many different shops. Consequently, for this group, only two composite samples were analyzed for each food item. Each composite was made up of eight individual samples of similar weights that were collected at different locations in the same city (two samples per location). A total of 108 samples were analyzed for their PCB concentrations. In these samples, seven PCB markers (IUPAC 28, 52, 101, 118, 138, 153, and 180), coplanar congeners 77, 126, and 169, and mono-ortho congener 105 were determined.

Food samples were homogenized and blended with a domestic mixer. Composite samples were lyophilized for PCB analyses, which were performed in accordance with U.S. Environmental Protection Agency method 1625 (for semivolatile organic compounds, isotope dilution gas chromatography–mass spectrometry). Prior to extraction, dried samples were homogenized. Freeze-dried solid sample (5 to 10 g) was mixed with a small amount of Na_2SO_4 and spiked with a mixture of $^{13}C_{12}$-labeled

TABLE 2. *Concentrations of polychlorinated biphenyls (PCBs) in foodstuffs acquired at various locations in Catalonia, Spain*

Food group	PCB concn	
	ng WHO-TEQ/kg of fat	pg WHO-TEQ/kg (wet wt)
Vegetables	1.69	4.72
Tubercles	5.37	11.27
Pulses	0.96	15.30
Cereals	4.49	55.15
Fruits	5.39	8.79
White fish	13.70	246.56
Blue fish	26.09	2,452.86
Tinned fish	5.92	727.99
Shellfish	12.44	348.46
Pork and pork products	0.25	68.17
Chicken	0.43	17.65
Beef	0.17	24.75
Lamb	0.19	22.64
Eggs	0.21	24.82
Whole milk	0.23	8.76
Semiskimmed milk	0.48	7.65
Dairy products	1.49	277.17
Margarine	0.58	477.79
Oil	0.15	151.61

TABLE 3. *Estimated daily intake of polychlorinated biphenyls (PCBs) for 70-kg (body weight) adult males (20 to 65 years old) living in Catalonia, Spain*

Food group	Consumption rate (g/day)[a]	PCB intake (pg WHO-TEQ/day)
Vegetables	226 (15.7)	1.07
Tubercles	74 (5.1)	0.83
Pulses	24 (1.7)	0.37
Cereals	206 (14.3)	11.36
Fruits	239 (16.6)	2.10
Fish and shellfish	92 (6.4)	82.87
Meat	185 (12.8)	8.85
Eggs	34 (2.4)	0.84
Milk	217 (15.0)	1.78
Dairy products	106 (7.3)	29.38
Oils	41 (2.8)	10.67
Total	1,444 (100)	150.13

[a] The percentage of the total consumption is given in parentheses.

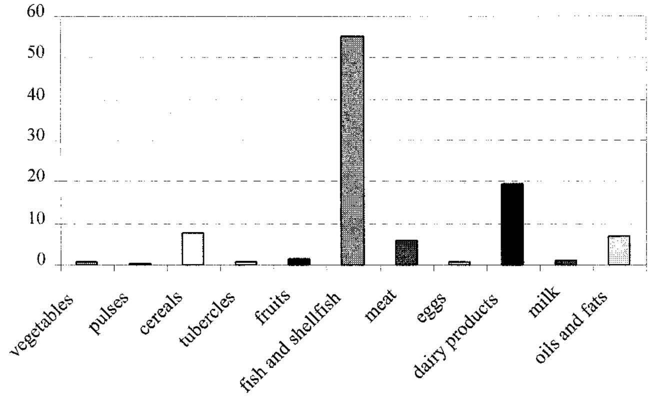

FIGURE 1. *Contributions (%) of food groups to the total dietary dioxin-like PCB intake of the general population of Catalonia, Spain.*

PCB standards (at least one PCB for each chlorination degree). Samples were extracted for 24 h with the following organic solvents (Soxhlet extraction): toluene for vegetables, fruits, cereals, eggs, and milk and milk-products; hexane/dichloromethane (1:1) for meat, fresh fish, and mussels; and petrolether for fish in oil. Oil and margarine were dissolved in hexane and immediately used for the cleanup procedure. Lipids were isolated and a portion of them was used for the cleanup procedure. The cleanup procedure and the fractionation of the sample aliquot were carried out as a multistep procedure involving adsorption chromatography, a multilayer silica column (from top to bottom: sodium sulfate, silica, silica–sulphuric acid, silica, silica–potassium hydroxide, silica), alumina columns, and gel permeation columns (BioBeads SX3). The final step involved the reduction of the PCB-containing fractions to the volume necessary for the analysis. Prior to PCB analysis, a ^{13}C-labeled PCB standard was added for the calculation of recovery ratios. The cleaned extract was analyzed by high-resolution gas chromatography–high-resolution mass spectrometry (a Fisons CE 8000 gas chromatograph coupled with a VG Autospec Ultima system with electronic impact and a multiple ion detection mode [with a resolution of $\geq$10,000]). A DB-XLB column was used. Internal standards were used for PCB quantification. For TEQ calculations, WHO-98 TEFs were used *(22, 24).*

Average daily food consumption data were obtained from a recent study carried out in the same area *(4).* PCB intake was estimated for each food group assuming that undetectable congener concentrations were equal to one-half of the limit of detection *(17, 20).*

RESULTS AND DISCUSSION

Table 1 shows the concentrations of PCB congeners in food samples obtained in Catalonia for eight food groups. Generally, the highest PCB levels were those for PCB 153 (4,757.45 ng/kg [wet weight] for fish and shellfish, 111.50 ng/kg [wet weight] for meat, 136.48 ng/kg [wet weight] for eggs, and 191.58 ng/kg [wet weight] for milk and dairy products). In contrast, PCB 169 was detected only in fish and shellfish (at 5.70 ng/kg [wet weight]), milk and dairy products (at 1.02 ng/kg [wet weight]), and fats and oils (at 2.73 ng/kg [wet weight]). In turn, PCB 126 levels were below the detection limit for vegetables, tubercles, pulses, cereals, fruits, and meat. The highest levels of most PCB congeners were found in fish and shellfish. Similar results

TABLE 4. *Estimated dietary intake (pg WHO-TEQ/day) of polychlorinated biphenyls (PCBs) for the general population of Catalonia according to sex and age*

Age group (yr)	Sex[a]	PCB intake for food group: Vegetables	Tubercles	Pulses	Cereals	Fruits	Fish/ shellfish	Meat	Eggs	Milk	Dairy products	Fats	All groups
4–9	M	0.61	0.78	0.40	11.03	1.75	48.64	6.70	0.84	2.67	31.3	8.59	113.3
	F	0.57	0.65	0.38	11.08	1.70	44.14	6.70	0.47	2.39	31.9	8.85	108.8
10–19	M	0.77	0.98	0.38	14.50	1.80	57.65	8.66	0.74	2.48	37.7	10.67	136.4
	F	0.76	0.74	0.35	9.87	1.75	54.05	7.32	0.52	1.89	30.2	8.07	115.5
20–34	M	0.89	0.89	0.35	13.07	1.85	67.56	10.77	0.94	1.88	34.4	11.19	143.8
	F	0.82	0.67	0.34	8.93	1.80	65.76	6.60	0.57	1.99	24.7	8.07	120.2
35–50	M	1.18	0.83	0.43	11.42	2.14	85.57	7.94	0.77	1.84	28.5	11.19	151.9
	F	1.02	0.62	0.40	7.44	1.84	69.36	6.08	0.62	1.98	24.1	8.33	121.8
51–65	M	1.13	0.78	0.32	9.65	2.29	95.48	7.80	0.84	1.62	25.2	9.63	154.8
	F	1.02	0.64	0.31	6.51	2.34	79.27	5.26	0.55	2.26	27.2	7.81	133.1
>65	M	0.90	1.01	0.38	9.82	2.62	70.3	5.79	0.72	1.80	19.1	7.29	119.7
	F	0.89	0.55	0.29	7.44	2.09	73.9	5.12	0.40	2.36	20.8	7.81	121.6

[a] M, male; F, females.

TABLE 5. *Comparison of results of a number of recent international surveys on dietary intake of polychlorinated biphenyls (PCBs)*

Region/country	Year of publication	Foods analyzed	Congeners analyzed	Main foods contributing to WHO-TEQ	Comments[a]	Reference
Aragón, Spain	1999	Legumes, potatoes, vegetables, fruits cereals, meat, egg-based foods, fish-based foods	28, 52, 101, 138, 153, 180	PCBs were found only in fish meals; mean level: 96.3 ng/g lipid	Congeners detected: 138, 153, and 180	*13*
Seven districts of Japan	1999	14 food group composites	Coplanar PCB 27, 126, 169	Fish and shellfish, (51.69 pg TEQ/day)	Total intake of PCBs: 72.7 pg TEQ/day	*19*
Italy	1999	Meats, dairy products, eggs, fish, oils and fats, vegetables, cereals, fruits, sweets, beverages	Most toxic and most abundant in the environment	Dairy products, meat, and fish	Exposure to the PCB with the highest TEF: vegetables; mean intake of total PCB: 3.72 ± 1.51 μg/person/day	*25*
Finland	2001	Rainbow trout, eggs, beef, cow's milk, pork, leafy vegetables, flour, potatoes, fruits, vegetables	77, 126, 169, 8 mono-ortho congeners, 25 other congeners	Rainbow trout, beef, and cow's milk	PCB intake: 53 pg TEQ/day	*11*
Five regions of the United States	2001	Pooled samples of beef, chicken, pork, sandwich meat, ocean fish, freshwater fish, butter, cheese, milk, ice cream, eggs, and a vegan diet	77, 126, 169, 105, 114, 118, 128, 138, 153, 180	Fish, meats, and dairy products	PCB intake: 0.68 pg I-TEQ/kg/day (adults); ND = ½LOD	*17*
Sixteen locations in Japan	2001	14 food group composites	4 nonortho congeners, 8 mono-ortho congeners	Fish and shellfish (56.6 pg TEQ/person/day)	PCB intake: 67.92 pg TEQ/person/day; ND = ½LOD	*20*
Catalonia, Spain	2002	Milk, eggs, meat, mussels, olive oil	77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, 189	Mussels	No data on PCB daily intake	*6*
Catalonia, Spain	2002	12 food group composites	28, 52, 77, 101, 105, 118, 126, 138, 153, 169, 180	Fish and shellfish, fats and oils	PCB intake: 150.13 pg TEQ/person/day; ND = ½LOD	This study

[a] ND, undetectable congener concentrations; LOD, limit of detection.

have been reported in recent international surveys *(8, 13, 19, 20)*. Total PCB levels were highest for fish and shellfish (11,864.18 ng/kg [wet weight]); the next highest levels, which were substantially lower, were those for milk and dairy products (674.50 ng/kg [wet weight]) and eggs (475.18 ng/kg [wet weight]). In terms of nanograms WHO-TEQ per kilogram (wet weight), the highest value was that for fish and shellfish (0.901 ng WHO-TEQ/kg [wet weight]), followed by those for fats and oils (0.260 ng WHO-TEQ/kg [wet weight]) and milk and dairy products (0.143 ng WHO-TEQ/kg [wet weight]).

The WHO-TEQ values for a number of foods obtained in seven cities in Catalonia are summarized in Table 2. Results are expressed both in terms of nanograms WHO-TEQ per kilogram of fat and in terms of picograms WHO-TEQ per kilogram (wet weight). For results expressed in terms of picograms WHO-TEQ per kilogram (wet weight), the highest value was that for blue fish (2,452.86 pg WHO-TEQ/kg [wet weight]), followed by those for tinned fish (727.99 pg WHO-TEQ/kg [wet weight]) and margarine (477.79 pg WHO-TEQ/kg [wet weight]). For results expressed in terms of nanograms WHO-TEQ per kilogram of fat, the highest value was that for blue fish (26.09 ng WHO-TEQ/kg of fat), followed by those for white fish (13.70 ng WHO-TEQ/kg of fat) and shellfish (12.44 ng WHO-TEQ/kg of fat).

Daily PCB intake estimates for the general population of Catalonia are presented in Table 3. The total dietary intake of PCBs was estimated to be 150.13 pg WHO-TEQ/day. The contributions of each food group to this intake are shown in Figure 1. In Figure 1, it can be seen that fish and shellfish contributed >55% to the total dietary intake. Another remarkable contribution was that of dairy products (excluding milk), at 19.57%. Cereals were found to contribute 7.58% of the total dietary PCB intake; the WHO-TEQ value for this group (including pulses) was not especially high (Table 2), but the notable consumption of cereals in the Mediterranean diet could explain their remarkable contribution. Similar conclusions were drawn when the dietary dioxin and furan intake of a single population was determined. In two recent studies, cereals were found to contribute 23.09% *(5)* and 13.74% *(3)* of the total PCDD-PCDF dietary intake.

The estimated PCB intake for the population of Catalonia according to sex and age is summarized in Table 4. Total daily intake levels ranged from 108.8 pg WHO-TEQ for girls under 10 years of age to 154.8 pg WHO-TEQ for men 51 to 65 years of age. For both males and females, the daily intake of PCB increased with age up to 65 years, after which a decrease could be noted. On the other hand, except for individuals over 65 years of age, the daily PCB intake level was always higher for males than for females, a finding that is doubtless due to the ingestion of less food by females. A similar trend was also observed for PCDD-PCDF intake *(3)*.

A comparison of the results of the present study with those of a recent report from Catalonia by Eljarrat et al. *(6)* shows that levels found in Eljarrat et al.'s study were generally higher than those found in the present survey, especially for mussels. Only chicken (0.43 ng WHO-TEQ/kg of fat), pork (0.25 ng WHO-TEQ/kg of fat), and oil (0.15 ng WHO-TEQ/kg of fat) samples showed higher PCB levels in the present survey. However, in the present study 108 samples were analyzed, whereas Eljarrat et al. analyzed only 29 samples.

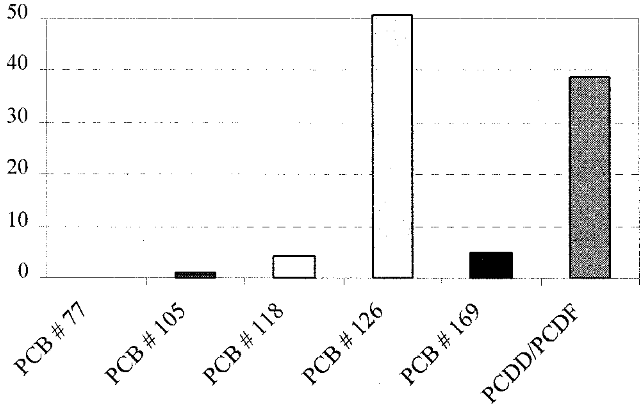

FIGURE 2. *Contributions (%) of PCB congeners and PCDD-PCDF to the total dietary intake (pg WHO-TEQ/day) of the general population of Catalonia, Spain.*

On the other hand, a comparison of the current PCB concentrations with data from a number of recent international studies is shown in Table 5. Because of the notable differences in the methods used, the specific PCB congeners determined in the different surveys, and the kinds and numbers of analyzed food samples, much care must be taken in comparing these results.

Recently, the dietary PCDD-PCDF intake of the population of Catalonia was determined *(3)*. The daily intake of these organic pollutants was 95.40 pg WHO-TEQ, while that of PCB was 150.13 pg WHO-TEQ. Thus, PCDD-PCDF and PCB contributed 38.85 and 61.15%, respectively, to the total dietary intake of both families of environmental contaminants. The specific contributions of the five PCB congeners with assigned WHO-TEFs *(22, 24)*, as well as the contribution of PCDD-PCDF, to the total dietary intake are depicted in Figure 2. It can be seen in Figure 2 that the largest contribution (even larger than that of PCDD-PCDF) was that of PCB 126. A similar trend for the contributions of PCDD-PCDF and PCBs to the total TEQ was noted by Toyoda et al. *(19)* and Kiviranta et al. *(11)*, who reported PCB contributions of 53.5 and 53%, respectively. In contrast, Schecter et al. *(17)* reported a larger contribution for PCDD-PCDF (72%) than for PCB (28%). In turn, Tsutsumi et al. *(20)* found similar contributions (about 50%) for PCBs and PCDD-PCDF (undetectable congener concentrations were assumed to be equal to one-half of the limit of detection). The results of the present study confirm the importance of PCBs in human risk assessment with regard to dioxins, furans, and dioxin-like PCBs in food.

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