



Levels of Dioxins, Dibenzofurans, PCB and DDE Congeners in Pooled Food Samples Collected in 1995 at Supermarkets Across the United States

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ABSTRACT

Food, particularly dairy products, meat, and fish, has been identified as the primary immediate source of intake of polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), and polychlorinated biphenyls (PCBs) for the general population. We previously reported PCDD/Fs in individual analyses of food samples from a number of countries, including the U.S., the former Soviet Union, and Vietnam. We also previously estimated daily intake of dioxins and related chemicals in Americans at various ages in these reports. In this paper, the levels of dioxins, dibenzofurans, dioxin toxic equivalents (TEQs), selected dioxin-like PCBs, and DDE (a persistent metabolite of DDT) were measured in 12 pooled food samples from over 90 individual specimens collected from supermarkets throughout the United States during 1995. Samples were pooled by food groups and then analyzed. Food samples were collected in Binghamton, New York; Atlanta, Georgia; Chicago, Illinois; San Diego, California; and Louisville, Kentucky. In addition to the meat, dairy, and fish samples, a vegan (all vegetable, fruit and grain, no animal product) diet, was simulated; this showed the lowest level of dioxins. © 1997 Elsevier Science Ltd. All rights reserved

Keywords: Food, dioxins, dibenzofurans, polychlorinated biphenyls, dioxins in U.S. food

INTRODUCTION

In previous work, we described polychlorinated dibenzo-p-dioxin (PCDD), polychlorinated dibenzofuran (PCDF), and dioxin toxic equivalent (TEQ) levels in over 22 individual analyses of American food samples as well as food from other countries.¹⁻⁵ The food was obtained from one supermarket in Binghamton, New York. We began our survey of dioxins in U.S. food with one

supermarket because regional differences in human tissue dioxin levels in the U.S. general population have not been observed and food from supermarkets generally represent a wide geographical sampling with respect to origins.⁶

In this study, in order to better characterize U.S. food, we collected approximately 100 specimens of meat (including poultry); dairy products; and fish; as well as vegetables and fruit from major regions of the U.S. These include the northeast (Binghamton, New York), the midsouth (Louisville, Kentucky), the south (Atlanta, Georgia), the midwest (Chicago, Illinois) and the west (San Diego, California). Food was obtained from one large supermarket in each city.

Previous food studies from a number of countries found very low dioxin content in fruit and vegetables, compared to meat, dairy and fish.⁷⁻¹⁴ For this reason, and in order to be parsimonious with scarce and costly resources, we elected to also pool our fruit and vegetable specimens in a simulated vegetarian diet (fruits, vegetables, and grains, but no animal products) which is sometimes referred to as a "vegan" diet, and performed one analysis on this food mixture.

High resolution gas chromatography-mass spectroscopy was performed on each of the 12 pooled samples in the now usual fashion by a laboratory (Midwest Research Institute) certified by the World Health Organization for analysis of dioxins, dibenzofurans and dioxin like PCBs in biological material.⁵

METHODS

A. Collection

Food was collected in original packaging from supermarkets in Binghamton, New York; Chicago, Illinois; Louisville, Kentucky; Atlanta, Georgia; and San Diego, California and then immediately frozen. The samples were kept separate and frozen until delivered to the dioxin laboratory where they were thawed, weighed and mixed by weight into pooled samples for analyses. Most geographical regions were included in each analysis.

B. Chemistry

Samples were prepared by grinding samples of the same types, after removal of bone if indicated, (eg. meat = beef, or chicken, or pork) together using an auger-fed Hobart® grinder. Each sample composite was ground and homogenized three times. Equal amounts of each type of individual food were taken for a composite sample. One hundred grams of each sample type were taken and mixed with approximately 300-g of pre-cleaned sodium sulfate, then the sample was split and placed into two soxhlet extractors. The sample was fortified with ¹³C-labeled

analogs consistent with EPA Method 8290 and extracted for 16 hours with 1:1 dichloromethane:hexane. After extraction the solvent was removed and the percent extractable lipid was gravimetrically determined. The lipid was re-dissolved in 200 mL of hexane and slurried for 2 hours in 100-g of acidified silica gel. The extract was then eluted through an acidified silica gel column, a neutral alumina column, and an AX-21/Celite 545 carbon column. The final toluene extract was reduced in volume to 10 μ L with ¹³C-labeled recovery standards added. Ten percent of the extract was taken after the acid silica gel step and analyzed for mono- and di-ortho PCBs.

The extract was analyzed on a VG70-250S at a resolution of 10,000. A 60-m DB-5MS column was used to provide chromatographic resolution between 2,3,7,8-TCDD and all other TCDD isomers. Method 8290 analysis criteria were met. The ions monitored were changed to accommodate the monitoring of coplanar PCBs in the same analytical run. Results were calculated on a wet weight and on an extractable lipid weight.

RESULTS

Table I lists the various foods pooled for the 12 analyses. Food categories included: Fish (fresh-water and ocean fish); Meat (beef, chicken, pork, hot dog and bologna); Dairy (cheese, butter, ice cream, milk); Eggs; and Vegetarian, or Vegan type mixture. Table II describes the simulated vegan or non-animal diet, and lists its dioxin toxic equivalent (I-TEQ) value, 0.07 pg/g or parts per trillion (ppt) on a wet weight basis.

Table III presents detailed results from the 12 analyses of the pooled food samples, reported on a wet weight basis. Measured dioxin, dibenzofuran, PCB congeners, and DDE values are shown in Table III. Also, the current estimates of dioxin-like toxicity using "international" dioxin toxic equivalency factors or I-TEF for PCDD/Fs and current PCB estimates are given.¹⁵⁻¹⁸ At the bottom of the table, total dioxin toxicity from PCDD; PCDFs; PCDD/Fs; and PCDDs /PCDFs /PCBs are presented. Measured total dioxin levels range from 0.4 pg/g for the vegan diet to 63.3 pg/g for the butter sample. Measured total dibenzofurans range from 0.93 pg/g for the vegan sample to 7.9 pg/g for the fresh-water fish sample. Total PCDD/F/PCB TEQ values range from 0.07 ppt TEQ on a wet or whole weight basis for the vegan sample to 1.43 ppt TEQ for the fresh-water fish sample. The highest DDE level was measured in the butter sample, 20,700 pg/g. The sample of fresh-water fish has higher values of the organochlorine DDE, 9,950 ppt, compared to ocean fish, 978 ppt. The vegan diet value, 453, is not lowest in DDE content, unlike the findings for the PCDD/F/PCBs.

Figure I illustrates the data in bar graph format, with dioxin toxic equivalent values shown for dioxins, dibenzofurans, PCBs and the total values. Fresh-water fish has the highest TEQ,

**Table I. Pooled Food Samples for Dioxin Analyses Collected in 1995 from
Different Geographical Areas in the United States**

Food Category	Food Homogenized for Analysis	City of Purchase
FISH		
Fresh-water fish	Catfish steak, rainbow trout Rainbow trout, lemon pepper catfish Rainbow trout, catfish fillets Perch fillets, Lake Superior white fish Boneless rainbow trout, fresh salmon steak	San Diego, CA Louisville, KY Atlanta, GA Chicago, IL Binghamton, NY
Ocean fish	True cod fillets, fresh salmon steaks, king salmon steak Tiger prawns, sea bass Ocean perch fillets Halibut, ocean perch, salmon steak Ocean perch fillets, fresh scrod/cod, fresh/frozen cod fillet	San Diego, CA San Diego, CA Louisville, KY Atlanta, GA Binghamton, NY
DAIRY		
Cheese	American cheese slices, brie, hot pepper cheese food, American cheese slices, swiss cheese slices, cracker barrel, sharp American cheese food Processed cheese food, natural muenster, pasteurized cheese food Cheese variety pack, processed cheese food	San Diego, CA San Diego, CA Louisville, KY Atlanta, GA Binghamton, NY
Butter	Butter, unsalted butter Sweet cream butter (salted)	San Diego, CA Atlanta, GA
Ice cream	Chocolate-chocolate chip Chocolate-chocolate chip Chocolate-chocolate chip Chocolate-chocolate chip Chocolate-chocolate chip	San Diego, CA Louisville, KY Atlanta, GA Chicago, IL Binghamton, NY
Milk	Whole milk Vitamin D milk Homogenized milk Whole milk Homogenized whole milk	San Diego, CA Louisville, KY Atlanta, GA Chicago, IL Binghamton, NY
MEAT		
Beef	Lean ground beef, ground beef 75% lean Ground beef Cubed beefsteak, ground beef Bone in hind beefsteak, ground beef (choice), round stew beef Beef rib steak (USDA choice)	San Diego, CA Louisville, KY Atlanta, GA Binghamton, NY Binghamton, NY
Poultry	Boneless chicken thigh, boneless chicken breast Chicken drumstick Chicken thigh Split chicken breast, chicken thighs, chicken fryer split	San Diego, CA Louisville, KY Atlanta, GA Binghamton, NY
Pork	Boneless pork loin chops, pork link sausage Pork loin thin sliced Pork loin Thin boneless pork chop Pork sausage, boneless center cut pork chop	San Diego, CA Louisville, KY Atlanta, GA Chicago, IL Binghamton, NY
Hot dog/bologna	Sliced bologna, hot dogs Oscar Meyer bologna	San Diego, CA unknown
EGGS		
	Hard boiled large eggs Hard boiled eggs Hard boiled eggs	San Diego, CA Atlanta, GA Binghamton, NY
VEGAN*	See Table 2.	Binghamton, NY

* Only vegetables, grains, and fruit included in analysis (no animal products)

Table II. Composition of Simulated Vegan Diet Sample			
Food	Weight	% of Composite	% of Overall Sample
Fruit Composite			
Apple	41	8	2
Banana	171	32	8
Orange	75	14	4
Canned Fruit	142	27	7
Nectarine	104	19	5
Total	533	100	25
Vegetable Composite			
Frozen mixed vegetables	99	31	8
Green beans	121	38	38
Salad	102	32	9
Total	322	100	25
Protein Composite			
Kidney beans	130	29	7
Lentils	199	44	11
Tofu	92	20	5
Peanut Butter	33	7	2
Total	454	100	25
Grain Composite			
Graham crackers	67	27	7
Bread	53	20	5
Cereal	42	16	4
Macaroni	52	20	5
Rice	45	17	4
Total	261	100	25
Total %			100
% Lipid in sample			2.72%
I-TEQ (ppt, wet weight)			0.07

Table IIIA. Dioxins, Dibenzofurans, Coplanar, Mono-Ortho and Di-Ortho PCBs, and Dioxin TEQs in

Congener	TEF	Beef	Chicken	Pork	Hot Dog/Bologna	Eggs
DIOXINS						
2378 TCDD	1	ND (1) (0.03)	ND (0.03)	ND (0.03)	ND (0.03)	ND (0.02)
12378 PeCDD	0.5	ND (0.13)	ND (0.12)	ND (0.13)	ND (0.13)	ND (0.12)
123478 HxCDD	0.1	ND (0.18)	ND (0.12)	ND (0.13)	ND (0.13)	ND (0.12)
123678 HxCDD	0.1	0.65	ND (0.12)	0.17	0.36	ND (0.16)
123789 HxCDD	0.1	0.14	ND (0.12)	ND (0.13)	ND (0.13)	ND (0.12)
1234678 HpCDD	0.01	2.74	0.43	1.57	3.99	2.74
12346789 OCDD	0.001	4.29	1.07	8.00	25.70	33.10
TCDD (2)		ND (0.03)	ND (0.03)	ND (0.03)	ND (0.03)	ND (0.03)
PeCDD (2)		ND (0.13)	ND (0.12)	ND (0.13)	ND (0.13)	ND (0.12)
HxCDD (2)		0.82	0.13	0.18	0.54	0.17
HpCDD (2)		2.74	0.43	1.57	--	5.04
Total Dioxins		11.6	2.4	11.8	30.9	41.4
DIBENZOFURANS						
2378 TCDF	0.1	0.06	ND (0.10)	0.18	ND (0.05)	0.03
12378 PeCDF	0.05	ND (0.13)	ND (0.12)	ND (0.13)	ND (0.13)	ND (0.12)
23478 PeCDF	0.5	ND (0.13)	0.14	ND (0.13)	ND (0.13)	ND (0.12)
123478 HxCDF	0.1	ND (0.16)	ND (0.12)	ND (0.14)	ND (0.35)	ND (0.67)
123678 HxCDF	0.1	ND (0.28)	ND (0.15)	ND (0.59)	ND (0.71)	ND (0.77)
234678 HxCDF	0.1	ND (0.13)	ND (0.12)	ND (0.13)	ND (0.25)	ND (0.56)
123789 HxCDF	0.1	ND (0.13)	ND (0.16)	ND (0.13)	ND (0.36)	ND (0.83)
1234678 HpCDF	0.01	ND (1.42)	ND (0.29)	ND (1.85)	0.89	0.28
1234789 HpCDF	0.01	ND (0.13)	ND (0.12)	ND (0.13)	ND (0.13)	ND (0.12)
12346789 OCDF	0.001	ND (0.39)	ND (0.25)	ND (0.30)	0.56	1.34
TCDF (2)		0.13	2.25	0.43	0.03	0.11
PeCDF (2)		ND (0.13)	1.64	ND (0.13)	0.20	ND (0.12)
HxCDF (2)		0.15	ND (0.12)	ND (0.13)	ND (0.28)	ND (0.62)
HpCDF (2)		ND (0.13)	ND (0.12)	ND (0.13)	0.98	1.1
Total Dibenzofurans		1.9	4.9	2.6	3.9	4.9
COPLANAR PCBs						
77 3344 Tetra PCB	0.0005	3.93	10.7	10.6	--	--
78 33'45 Tetra PCB		ND (0.10)	ND (0.10)	ND (0.10)	ND (0.10)	ND (0.10)
79 33'45' Tetra PCB		ND (0.10)	ND (0.10)	0.26	0.20	ND (0.10)
80 33'55' Tetra PCB		38	120	160	154	--
81 344'5 Tetra PCB		0.25	0.59	ND (0.67)	0.24	0.10
126 33445 Penta PCB	0.1	0.39	0.38	0.10	0.71	0.29
127 33'455' Penta PCB		ND (0.10)	ND (0.10)	10.6	ND (0.10)	ND (0.10)
169 334455 Hexa PCB	0.01	0.12	ND (0.10)	0.14	ND (0.10)	ND (0.10)
Total Coplanar PCBs		42.8	131.9	182.1	155.5	0.6
MONO-ORTHO PCBs						
105 233'44' Penta PCB	0.0001	ND (250)	78	ND (250)	400	ND
114 2344'5 Penta PCB	0.0005	ND (250)	ND (250)	ND (250)	ND	ND
118 23'44'5 Penta PCB	0.0001	94	200	ND (250)	1100	64
Total Mono-Ortho PCBs		344	403	375	1500	64
DI-ORTHO PCBs						
128 22'33'44' Hexa PCB	0.00002	ND (250)	ND (250)	ND (250)	310	ND
138 22'344'5' Hexa PCB	0.00002	ND (250)	40	0.10	860	62
153 22'44'55' Hexa PCB	0.00002	83	110	72	560	89
180 22'344'55' Hepta PCB	0.00001	260	230	ND (250)	140	16
Total Di-Ortho PCBs		593	505	322	1871	147
DDE		433	149	206	1090	674
% LIPID		13.13	5.33	9.18	29	14.68
PCDD TEQ		0.16	0.07	0.10	0.16	0.12
PCDF TEQ		0.08	0.11	0.11	0.13	0.18
PCDD/F TEQ		0.25	0.18	0.21	0.29	0.31
PCB TEQ		0.14	0.14	0.11	0.25	0.04
PCDD/F/PCB TEQ		0.38	0.32	0.32	0.54	0.35

(1) ND = not detected with one-half the detection limit in () used in calculation of TEQ.

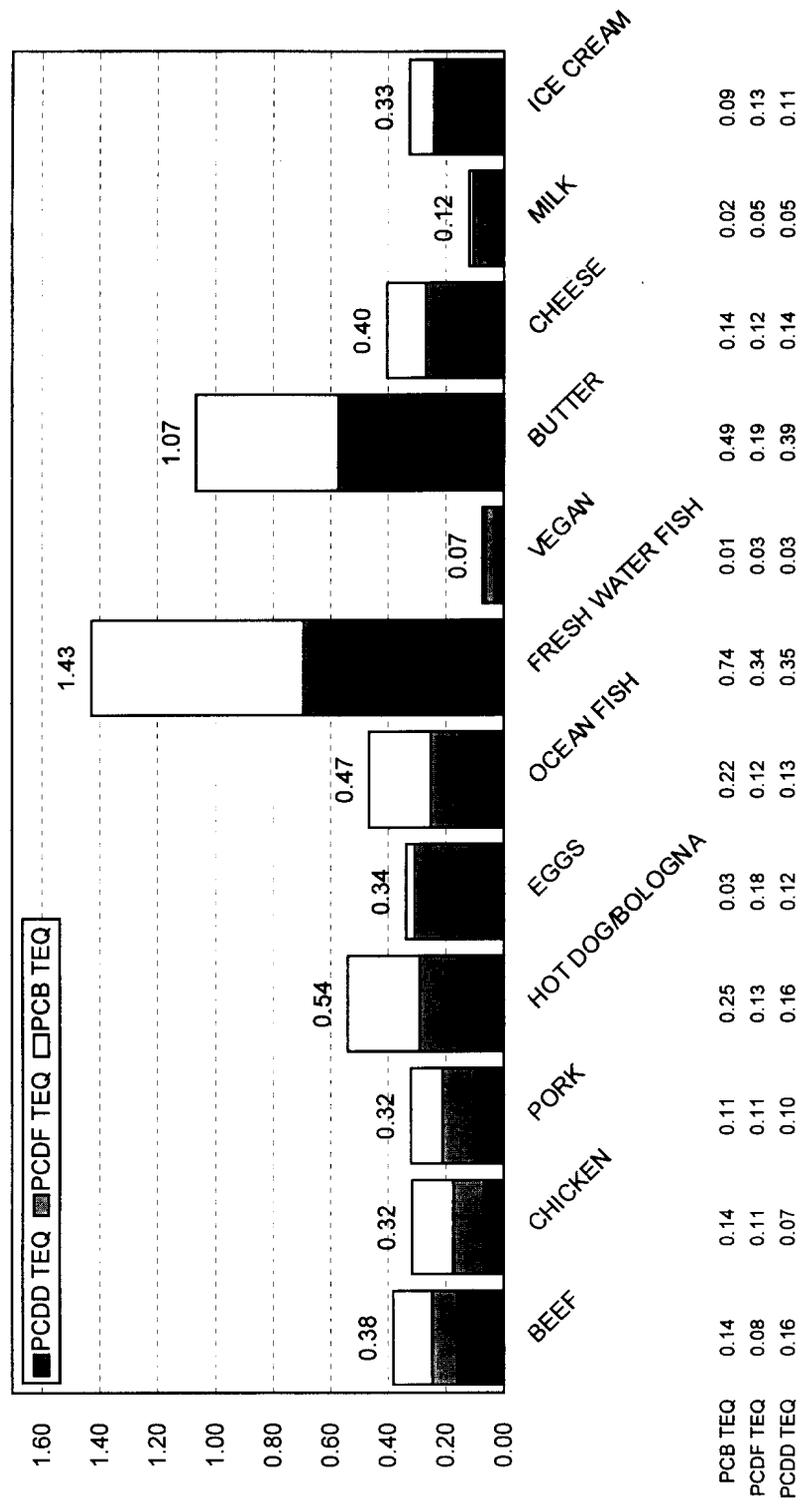
(2) = non 2,3,7,8 chlorine substituted

Table IIIB. Foods Collected Across the United States [$\mu\text{g/g}$ (ppt) wet weight]

Ocean Fish	Fresh Fish	Vegan Diet	Butter	Cheese	Milk	Ice Cream
0.03	0.15	ND (0.01)	0.10	ND (0.05)	ND (0.02)	ND (0.04)
ND (0.13)	0.25	ND (0.06)	ND (0.03)	ND (0.25)	ND (0.10)	ND (0.20)
ND (0.13)	0.15	ND (0.06)	ND (0.25)	ND (0.25)	ND (0.10)	ND (0.20)
ND (0.13)	0.26	ND (0.06)	1.00	0.31	ND (0.10)	0.28
ND (0.13)	0.20	ND (0.06)	0.39	ND (0.25)	ND (0.10)	ND (0.19)
0.17	1.14	--	--	--	--	--
0.45	5.88	--	59.2	--	--	--
0.06	0.21	ND (0.01)	0.10	ND (0.05)	ND (0.02)	ND (0.04)
ND (0.13)	0.25	ND (0.06)	ND (0.25)	ND (0.25)	ND (0.10)	ND (0.19)
ND (0.13)	0.61	ND (0.06)	1.7	--	ND (0.10)	0.30
0.17	1.14	--	--	--	--	--
1.3	10.2	0.2	62.8	0.9	0.3	1.0
ND (0.17)	0.69	ND (0.06)	ND (0.05)	ND (0.05)	ND (0.20)	ND (0.04)
ND (0.13)	ND (0.37)	ND (0.06)	ND (0.25)	ND (0.25)	ND (0.10)	ND (0.20)
0.13	0.37	ND (0.06)	ND (0.25)	ND (0.25)	ND (0.10)	ND (0.20)
0.15	ND (0.16)	ND (0.06)	ND (0.47)	ND (0.25)	ND (0.10)	ND (0.30)
ND (0.24)	ND (0.96)	ND (0.06)	ND (0.82)	ND (0.25)	ND (0.10)	ND (0.33)
0.14	ND (0.13)	ND (0.06)	ND (0.75)	ND (0.25)	ND (0.10)	ND (0.41)
0.21	ND (0.13)	ND (0.06)	ND (0.25)	ND (0.25)	ND (0.13)	ND (0.39)
ND (0.69)	ND (1.50)	ND (0.06)	--	ND (0.25)	ND (0.10)	ND (0.19)
0.13	ND (0.13)	ND (0.06)	ND (0.25)	ND (0.25)	ND (0.10)	ND (0.19)
0.25	ND (0.25)	ND (0.12)	--	ND (0.5)	ND (0.21)	ND (0.38)
0.25	1.95	0.07	0.16	ND (0.05)	ND (0.02)	ND (0.04)
0.13	1.05	0.10	ND (0.25)	ND (0.25)	ND (0.10)	ND (0.20)
0.16	ND (0.13)	ND (0.06)	ND (0.25)	ND (0.25)	ND (0.10)	ND (0.29)
0.13	ND (0.13)	ND (0.06)	--	ND (0.25)	ND (0.10)	ND (0.19)
2.3	6.0	0.6	1.9	1.7	0.8	1.7
6.19	ND (29.5)	--	--	--	--	--
ND (0.10)	0.10	ND (0.05)	ND (0.2)	ND (0.2)	ND (0.08)	ND (0.15)
0.17	2.03	ND (0.05)	ND (0.2)	ND (0.2)	ND (0.08)	ND (0.15)
85.3	509	--	--	--	--	--
0.33	1.5	ND (0.05)	ND (0.2)	0.24	ND (0.08)	ND (0.15)
0.83	ND (6.07)	(0.07)	3.36	1.04	0.16	0.86
ND (0.10)	0.10	ND (0.05)	ND (0.2)	ND (0.2)	ND (0.08)	ND (0.15)
0.20	0.94	ND (0.05)	0.39	ND (0.2)	ND (0.08)	ND (0.15)
93.0	531.4	0.2	4.2	1.7	0.4	1.2
120	ND (600)	ND	220	ND	NQ	NQ
ND (250)	250	ND	ND	ND	NQ	NQ
320	1800	15	930	240	NQ	NQ
565	2350	15	1150	240	NQ	NQ
ND (250)	270	14	350	ND	NQ	NQ
430	1800	87	790	150	NQ	NQ
390	1900	36	690	160	NQ	NQ
170	610	7	250	32	NQ	NQ
1100	4600	144	2080	342	NQ	NQ
978	9950	453	20700	2780	NQ	2780
1.43	4.83	2.72	89.1	21.75	2.15	22.03
0.13	0.35	0.03	0.39	0.14	0.05	0.11
0.12	0.34	0.03	0.19	0.12	0.05	0.13
0.25	0.69	0.06	0.52	0.27	0.10	0.24
0.22	0.74	0.01	0.49	0.14	0.02	0.09
0.47	1.43	0.07	1.07	0.40	0.12	0.33

NQ = Not quantifiable due to interference

Figure I. Dioxin, Dibenzofuran and PCB TEQs in Collected Foods, pg/g (ppt) Wet Weight



1.43 ppt, wet weight, followed by butter, with 1.07 ppt. Chicken and pork are similar with a TEQ value of 0.32 ppt each. Eggs and beef are similar in levels, at 0.34 and 0.38 ppt, respectively. Ocean fish has a higher TEQ at 0.47 ppt. The largest PCB contribution to the total TEQ value is found in fresh-water fish, followed by butter.

DISCUSSIONS AND CONCLUSIONS

These 12 dioxin and dibenzofuran analyses present the first data on U.S. food samples pooled by food type from all major U.S. geographical regions. For the first time also, dioxin-like PCBs are reported. In addition, current (1995) DDE values are presented. Also, illustrated is the persistence of DDE, the metabolite of DDT, in U.S. food, despite the ban on DDT in U.S. agriculture since the late 1970's. It should be noted that the specific country or countries of origin of the food samples is not known, only the sites where distributed.

Conversion of measured dioxin, dibenzofuran and PCB congeners to currently accepted dioxin toxic equivalents suggests PCBs may be adding significantly to dioxin toxicity in some instances. This is most striking in the case of fresh-water fish. PCBs also significantly contribute to the total TEQ value in the butter, beef, chicken, pork, hot dog, bologna, ocean fish, and cheese samples. Unlike dioxin and dibenzofuran levels in human tissues, the dioxin TEQs are not consistently found at higher levels than dibenzofuran TEQs in various food products.

The PCB dioxin toxic equivalency factors cited and used in this study are the relatively new WHO factors for the PCB congeners.¹⁸ These are, in general, lower than those formerly used to estimate dioxin toxic equivalency for PCBs.¹⁹ For some of the PCBs measured, there are no current estimates within the WHO recommendations. For these, a weighting of zero was used for those congeners.

It is possible that, with further research, numerical values will be assigned to some or all of these PCB congeners. If so, the estimates of total TEQ stated in this paper would underestimate total dioxin-like toxicity. These toxic equivalents values will require recalculation should new TEFs become available in the future, as expected. For dioxin and dibenzofuran TEFs, we used current U.S. EPA or "international" dioxin toxic equivalency estimates, which are also subject to change over time.¹⁵⁻¹⁷

In addition, toxic equivalency factors may be available in the future for specific toxic endpoints, so again recalculations based upon the measured values may be indicated. These factors may differ for carcinogenicity, immune deficiency, reproductive or developmental outcome, as well as for endocrine or neurological effects. There are also non 'dioxin-like' effects from PCBs and also DDE which are not accounted for in the calculations for dioxin toxic equivalents and are not considered in this paper.²⁰

Additional sampling and analysis of individual and pooled food samples to further characterize U.S. food dioxin levels is indicated from this work. There are suggestions of rapid decrease over the past few years in the level of dioxins and dibenzofurans in human tissue in Germany and in the Netherlands which would suggest a drop in food PCDD/F levels.^{21,22} Levels of PCDD/Fs in food in England have also decreased rapidly during the past decade.²³ At present, there is no data available to document whether or not such decreases are occurring in human tissue or food from the U.S. In addition, the effect of cooking food on dioxin levels has not yet been characterized. Further research is clearly indicated to clarify these issues.

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