

PCB and DDE Residues in Milk Supplies of Ontario, Canada 1985–1986

R. Frank and H. E. Braun

Agricultural Laboratory Services Branch, Ontario Ministry of Agriculture and Food, University of Guelph, Guelph, Ontario, Canada N1G 2W1

Concerns on the presence of PCB's in milk have arisen because of spills of these industrial chemicals on roads, around storage sites, and because of their presence in waste oils used to treat gravel roads with up to 25 ppm being allowed up to 1970 and 5 ppm up to 1984. The possibility of the spread of PCB into water supplies of rural areas of the province has been a constant concern and the presence of PCB in bovine milk serves as a reliable indicator of the degree of contamination.

Milk supplies in Southern Ontario have been monitored for PCB and DDE since 1970-71 (Frank et al. 1970; 1975; 1979; 1985). In 1985-86 the monitoring program was extended to include all regions of the province where milk is produced in order to further assess the incidence, levels, and trends of PCB in the provincial milk supply.

MATERIALS AND METHODS

Single milk samples were collected from each of 1184 bulk transporters hauling milk from all regions of Ontario. Sampling commenced in May 1985 and was completed in August, 1986. The milk in each transporter represented the accumulation of 2-days of milking from 5 to 20 dairy farms; the average load approximated 12,000 L and ranging from 1,300 to 24,000 L.

Collections of approximately 240-mL milk samples were made by personnel of the Dairy Inspection Branch, Ontario Ministry of Agriculture and Food, and were delivered within 24 hr to Agricultural Laboratory Services Branch for analysis. Subsamples were relayed to the Central Milk Testing Laboratory, Ontario Ministry of Agriculture and Food, where butterfat determinations were made according to the official AOAC infrared procedure (AOAC 1980).

Samples were extracted, partitioned, cleaned up and fractionated according to the procedure described by Frank et al. (1979).

Send reprint requests to R. Frank at the above address.

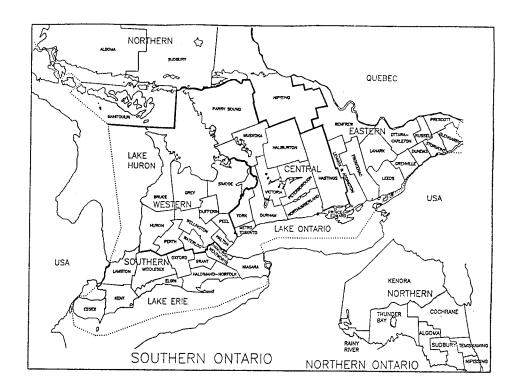


Figure 1. Map of the province of Ontario, Canada

PCB and DDE determinations were made by electron capture gas chromatography with detector operation in the constant current mode; the analytes were chromatographed on a 1.8 m x 2 mm i.d. glass column packed with 1.5% OV-17/2.0% OV-210 on 100/120 mesh Gas Chrom Q, a nitrogen carrier flow of 60 mL/min and isothermal column temperature of 190° . PCB were identified and quantitated by comparisons with mixtures of Aroclors 1254 and 1260 and measurement of peaks VII, VIII, and X according to the system of Reynolds (1971).

Recoveries were determined by fortification of whole milk with acetone solutions of PCB and DDE; fortified samples were stored under refrigerated conditions for 24 hr with occasional shaking and then analyzed according to the described procedure. Recoveries averaged 91% for DDE and 87% for PCB. Detection limits approximated 0.05 ug/L for DDE and 0.2 ug/L for PCB. Limits of detection are expressed as ug/L whole milk since the method was based on whole milk analysis; detection limits on a fat basis, therefore, are dependent on and vary directly as the variation in milkfat content.

Residue data in this report are expressed as ug/kg of milkfat and were derived by mathematical conversion from residues in whole milk. Standard deviations for samples collected from each of five regions of the province (Figure 1) were determined and regression analyses were applied to the present and all previous

surveys for PCB and DDE in Southern Ontario.

RESULTS AND DISCUSSION

A total of 1184 bulk tankers were analysed for PCB and 920 for DDE. The residue levels by region appear in Table 1. Regional levels varied from a mean of 12 to 19 ug/kg for PCB and 1.4 to 16.5 ug/kg for DDE. The frequency distribution of PCB in bulk tankers with respect to concentration appears in Table 2. The highest residues of PCB and DDE occurred in the southern region.

Table 1. Residue levels of PCB and DDE by region in Ontario milk supplies, 1985-86.

	No. of Samples	% milkfat		Residue - ug/kg	
Region		Mean	(S.D.)	Mean	(S.D.)
РСВ					
Southern Western Central Eastern Northern	316 328 153 331 56	3.75 3.77 3.81 3.67 3.92	(0.52) (0.45) (0.29) (0.51) (0.91)	19 12 15 13 14	(15) (5) (8) (8) (10)
Total, Means	1184	3.75	(0.40)	15	(10)
Southern Western Central Eastern Northern	246 328 153 156 37	3.74 3.77 3.81 3.70 3.91	(0.51) (0.45) (0.29) (0.50) (0.90)	16.5 5.9 9.1 2.9 1.4	(17.1) (3.2) (9.0) (2.8) (0.8)
Total, Means	920	3.76	(0.47)	8.6	(11.1)

Table 2. Frequency distribution of PCB residue levels in 1184 bulk tankers

Residue	PCB in Milkfat (ug/kg)						
	<5	5-50	51-100	Highest			
Southern	2	300	14	99			
Western	8	319	1	57			
Central	0	151	2	57			
Eastern	25	304	2	64			
Northern	1	54	1	69			
Totals	36	1128	20	99			

Changes in PCB and DDE levels since 1970 are shown in Table 3. Disappearances of PCB since the peak year of 1973 followed a first order regression equation of log $y=2.03-0.057 \times (F=21.9)$ where y equals the PCB residue in ug/kg and x equals time in years; r^2 was calculated at 0.88 and a half-life disappearance of 5.9 years is indicated for PCB. Disappearance of DDE since 1970 when DDT use was restricted followed a regression of $y=76.3-58.1 \log x \ (F=46.5)$ where y=DDE concentration in ug/kg and x equals years; r^2 was calculated at 0.94 and the half-life disappearance at 5.8 years.

Table 3. PCB and DDE levels in milkfat from Southern region of Ontario, 1970 to 1986.

Year	No. of	PCB - ug/kg		DDE - ug/kg	
	Tankers	Mean (S.D.)		Mean (S.D.)	
1970-71	337	85	(74)	96	(68)
1973	350	115	(64)	51	(26)
1977	308	35	(30)	12	(11)
1983	253	23	(175)	13	(11)
1985-86	316	19	(15)	16	(17)

REFERENCES

AOAC (1984) Official Methods of Analysis, 14th ed. Association of Official Analytical Chemists, Arlington VA

Frank R, Braun HE, McWade JW (1970) Chlorinated hydrocarbon residues in the milk supply of Ontario, Canada. Pestic Monit J 4:31-41

Frank R, Smith EH, Braun HE, Holdrinet M, McWade JW (1975) Organochlorine insecticides and industrial pollutants in the milk supply of the southern region of Ontario, Canada, 975. J. Milk Food Technol 38:65-72

Frank R, Braun HE, Holdrinet M, Sirons GJ, Smith EH, Dixon DW (1979) Organochlorine insecticides and industrial pollutants in the milk supply of Southern Ontario, Canada - 1977. J Food Prot 42:31-37

Frank R, Braun HE, Sirons GH, Rasper J, Ward GG (1985)
Organochlorine and organophosphorus insecticides and
industrial pollutants in the milk supplies of Ontario - 1983.
J Food Prot 48:499-504

Reynolds LM (1969) Polyclorobiphenyls (PCB's) and their interference with pesticide residue analysis. Bull Environ Contam Toxicol 4:128-143

Received February 9, 1988; accepted July 20, 1988.