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Persistence of Captan on Apples, Grapes, and Pears in Ontario, Canada, 1981–1983

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Blocks of apples, grapes, and pears were treated with captan at 1.7, 2.8, or 3.4 kg ha^{-1} for 1–15 applications. Captan residues were measured over a 14-day period following the last application. Residues declined significantly in seven of nine experiments. Correlations between rainfall and captan residues were observed in five experiments. No correlations were observed in four experiments in which little or no rain fell in the first 7 days. In four of the trials the captan residues immediately following the last application did not exceed the 5 mg kg^{-1} maximum residue limit permitted under the Canadian Food and Drug Act. The longest periods required for residues to decline below the 5 mg kg^{-1} tolerance were 5 days for grapes, 3 days for pears, and 7 days for apples.

INTRODUCTION

The fungicide captan, *N*-[(trichloromethyl)thio]-4-cyclohexene-1,2-dicarboximide is widely used for the prevention of fungal diseases of pome fruits and grapes around the world. During 1980 22 000 ha of apples, grapes, and pears were grown in Ontario (Ontario Ministry of Agriculture and Food, 1980) and treated with 130 000 kg of captan to prevent fungal diseases on foliage and fruit. The weather conditions during the growing season and especially prior to harvest are generally conducive to heavy fungal infection and serious crop losses. In Ontario, the major diseases of apple, pear, and grape against which captan is effective are respectively apple scab (*Venturia inaequalis* (Cke) Wint.), pear scab (*V. pirina* Aderh.), downy mildew (*Plasmopara viticola* (Berk. and Curt.) Berl. and de Toni), and Botrytis bunch rot (*Botrytis cinerea* Pers.). Captan has been recommended (Ontario Ministry of Agriculture and Food, 1983) and extensively used for over 30 years and its importance has increased recently with the loss of efficacy of benomyl, dodine, and iprodione as the result of development of fungicide-resistant pathogens. Applications of captan prior to harvest are often necessary for the protection of fruit during the preharvest period, and this could result in residues that exceed the established residue tolerance.

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Table I. Official Maximum Residue Limits for Captan on Pome Fruit and Grapes in 18 Countries[a]

country	maximum residue limits of captan, mg/kg^{-1}		
	apples	pears	grapes
Japan	5		
Canada	5	5	5
Switzerland	3	3	15
New Zealand	10	10	10
Belgium, France, Italy, Netherlands, Singapore, South Africa, Spain, Sweden, West Germany, Yugoslavia	15	15	15
Kenya	40	30	
Israel	40	30	15
Mexico, U.S.A.	25	25	50

[a] Health and Welfare Canada, 1981.

On a global scale, official maximum residue limits for captan range from 3 to 50 mg kg^{-1} (Table I). Canadian maximum residue limits were reduced from 25 to 5 mg kg^{-1} on 24 March, 1983 (Health and Welfare, Canada, 1983). These studies were undertaken to measure the rate of disappearance of captan and to determine preharvest intervals adequate to meet the new residue tolerance and were part of a larger study on several fruit crops jointly reported with Northover et al.

MATERIALS AND METHODS

Nine field experiments were undertaken and all except two (2 and 6) were conducted on large blocks of mature grapes, apples, and pears located on the Agriculture Canada Research Station, Jordan, Ontario. The soil at this

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Table II. Details on Nine Experiments Conducted on Grapes, Apples, and Pears to Study the Disappearance of Captan, 1981–1983

year	expt no.[a]	fruit and cultivar	application of captan[b] dates	rate, kg ha^{-1}	water, L ha^{-1}
1981	1	grape–Chardonnay	19 June; 10, 30 July; 7, 26 Aug	2.8	1500
	2	grape–De Chaunac	14 Sept	2.8	1500
1982	3	grape–De Chaunac	13 Sept; wine–29 Sept	2.8 (1.4)	1500
	4	pears–Bartlett	14, 30 June; 14 July; 13 Sept	3.4	630
	5	apples–McIntosh	14, 20 June; 14, 27 July; 11, 24 Aug; 13 Sept	3.4	630
	6	apples–Delicious	14, 20 June; 14, 27 July; 11, 24 Aug; 13 Sept	3.4	630
	7	apples–McIntosh	8, 25 Aug; 9 Sept	1.7	225
		apples–McIntosh	8, 25 Aug; 9 Sept	3.4	225
1983	8	pears–Bartlett	23 Aug; 1, 12 Sept	3.4	630
	9	apples–McIntosh	29 April; 10, 19, 31 May; 9, 16, 24 June; 7, 14, 21 July; 2, 12, 23 Aug; 1, 12 Sept	3.4	630
		apples–McIntosh	same as above except 23 Aug; 1, 12 Sept at reduced rate	1.7	630

[a] Experiment 6 was conducted in an orchard at the University of Guelph; experiment 1 was conducted on a private farm near St. Catharines; the remainder were conducted on Agriculture Canada Research Station, Jordan Station, Ontario. [b] Stauffer Captan 50WP was used in experiments 1, 2, 3, 8, and 9, Uniroyal Captan 50WP was used in experiments 4, 5, and 6, and Stauffer Captan 80WP was used in experiment 7.

location was a well drained Vineland sandy loam. Experiment 2 was conducted on a commerical vineyard near St. Catharines also on a Vineland sandy loam. Experiment 6 was located on a 40 year old apple orchard belonging to the University of Guelph and located on a Guelph sandy loam. Daily measurements of rainfall and temperature (maxima and minima) were recorded during the spray period.

Grapes. Experiments 1 and 3. Field experiments were conducted in 1981 and 1982 on *Vitis vinifera* cultivar Chardonnay and on a *V. vinifera* hybrid cultivar De Chaunac grape (Seibel 9549) (Table II). The Chardonnary vines were 6 years old and planted in rows 2.6 m apart while the De Chaunac vines were 9 years old and growing in rows 3.3 m apart. Captan was applied with an hydraulic over-the-row boom sprayer operating at a pressure of 2800 kPa (kilopascals). Spray dates, rates of captan applied, and amount of water used appear in Table II.

Mature grapes were harvested immediately before and at several intervals following the final application. In 1981 400 g of destemmed berries were sampled from 12 bunches from each of three randomized plots. In 1982 De Chaunac grapes were collected and processed into wine. For this purpose a second application of captan at half rate (1.4 kg ha^{-1}) was made on 29 September and eight days later grapes were picked, destemmed, and crushed for fermentation. Samples of grapes or wine were collected for analyses.

Pear and Apple Orchards. Experiments 4, 5, 6, 8, and 9. Bartlett pears were 20 years old and spaced 6.7 m apart. Both McIntosh and Delicious apples were from 20 year old trees which had been grafted on Mąlling VII rootstock and spaced 8.2 m apart. Captan was applied to apples and pears with a Swanson 530 air blast sprayer delivering 630 L ha^{-1} of spray suspension at a pressure of 2100 kPa. Spray dates and rates of captan applied appear in Table II. Four replicates of ten fruit each were collected from randomly selected trees within the sprayed blocks. Samples were collected for residue analysis just prior to and at several intervals up to 14 days following the last captan application.

Apple Orchard. Experiment 7. The McIntosh apples treated in this experiment were large 40 year old standard trees belonging to the University of Guelph, Guelph, Ontario. Full and half rates of captan were applied on the dates 8 and 25 August and 9 September with a Kinkelder air blast sprayer delivering 225 L ha^{-1} of spray suspension at a pressure of 1060 kPa. Fruit was collected at varying intervals after treatment by random selection to give four replications representing different rows, each sample weighing 5 kg.

Analytical Procedure. Fruit samples were macerated and a 50-g subsample was blended with an acetonitrile–water (2:1) mixture according to the multiresidue procedure described in the Pesticide Analytical Manual (1973). The extracts were filtered and an aliquot equivalent to 25 g of sample was diluted with four volumes of 2% sodium chloride in water and partitioned by shaking three times for 60 s with 50-mL portions of dichloromethane. The dichloromethane extracts were dried, combined by percolation through anhydrous sodium sulfate, evaporated to dryness with rotary vacuum at 45 °C, and redissolved in 5 mL of hexane. Samples were cleaned up on activated Florisil (Floridin Company, Berkley Springs, WV) according to the procedure of Mills et al. (1972); only the third elution fraction was collected and analyzed. Analysis was carried out by gas–liquid chromatography with ^{63}Ni-electron capture detection and a 1.8 m × 2 mm id column packed with 1.5% OV-17/2% OV-210 on 100–120 mesh Gas Chrom Q at an isothermal column temperature of 190 °C. Under the described GLC conditions, no evidence of chemical decomposition of captan on the GLC column was observed; lack of decomposition was further substantiated by the fact that good linearity between detector response and amount of captan was obtained in the range of 2.0–10 ng. Recoveries were determined at fortification levels of 0.1, 1.0, and 10 mg kg^{-1} in apples, grapes, and pears and ranged from 84% mean recovery at the 0.1 mg kg^{-1} level to 97% mean recovery at the 10 mg kg^{-1}. The detection limit of captan was 0.01 mg kg^{-1}.

RESULTS

Grapes. In experiment 1 the final application of captan raised the residue on grapes by 4.6 mg kg^{-1} to 8.3 mg kg^{-1} (Table III). Residues declined slowly over the next 38 days to 2.9 mg kg^{-1}. Rainfall records were maintained and regression analysis revealed that the first-order equation $\log y = 0.818 - 0.014x$ was highly significant ($F = 47.7$, $P = 0.05$) where y was the captan residue (mg kg^{-1}) and x the rainfall (mm). The calculated half residue disappearance was given by 21 mm of rain. Residues declined below the 5 mg kg^{-1} maximum residue limit (MRL) regulated under the Canadian Food and Drug Act (Health and Welfare Canada 1983) by day 5.

In experiments 2 and 3 the single application of captan added 3.2 and 1.7 mg kg^{-1} to the fruit and over the next 14 days, the residue did not decline significantly in experiment 2 and declined significantly only by day 15 in

Table III. Captan Residues on Grapes (Chardonnay) Treated with 2.8 kg ha^{-1} (Experiment 1)

time of harvest	captan residues, mg kg^{-1}	rainfall, mm[a] periodic	rainfall, mm[a] accumulated
prespray	3.7bc[b]		
postspray–day 0	8.3a	0	0
day 2	7.3a	2	2
day 5	4.9b	6	8
day 7	4.1bc	0	8
day 16	3.4bc	54	62
day 21	2.4c	0	62
day 38	2.9bc	47	109
mean temp, °C	16.7		

[a] The first order regression equation was $\log y = 0.818 - 0.014x$ ($r^2 = 0.91$) where y = captan residue (mg kg^{-1}) and x = rainfall (mm). [b] Values followed by the same letter are not significantly different at $P = 0.05$ (Duncan, 1951).

experiment 3 (Table IV). In 1981, 48 mm of rain fell during the 14 day test period but this failed to reduce captan residues on grapes and hence there was no significant correlation with rainfall ($r^2 = 0.07$). In 1982, 89 mm fell in the 15-day period following the application of captan and it was only in the last 5 days that a significant decline in residues occurred during a 40-mm rain. Earlier rains of 37 mm failed to reduce residues. Regression analysis revealed the first-order equation $\log y = 0.337 - 0.005x$ was significant ($F = 6.8$, $P = 0.05$, $r^2 = 0.58$) where y = captan residues and x = rainfall. Half residue disappearance required 63 mm of rain. In both experiments residues of captan were below the 5 mg kg^{-1} MRL following the final application.

The De Chaunac grapes which were processed into wine revealed that captan residues present at destemming and crushing had disappeared from the fermented juice following pressing after the skins and seeds were removed (Table V). Analysis of the liquid after the second fermentation and then when the wine was finished failed to detect captan to a limit of <0.01 mg kg^{-1}.

Pears (Experiments 4 and 8). Applications of 3.4 kg ha^{-1} of captan to pears increased surface deposits by 3.0 and 3.4 mg kg^{-1} in 1982 and 1983, respectively (Table IV). Residues of captan declined significantly over the next 15 or 14 days, respectively. In 1982 the regression analysis revealed a significant negative correlation ($F = 13.0$, $P = 0.05$, $r^2 = 0.72$) for the equation $\log y = 0.280 - 0.185 \log x$ where y = residue of captan and x = rainfall. A calculated half residue disappearance occurred with a rainfall of 65 mm. In 1983 the decline of captan residues associated with rainfall gave a significant first-order equation $\log \gamma = 0.818 - 0.014x$ ($F = 47.7$, $r^2 = 0.90$). The calculated half residue decline was equivalent to 21 mm of rain. In 1982, when the interval between the penultimate and ultimate applications was 61 days, the residue following the final application was less than the tolerance of 5.0 mg kg^{-1}. However, in 1983 when two applications were made within 20 days prior to the final application, the initial postapplication residue was 7.8 mg kg^{-1}, and this residue declined below 5 mg kg^{-1} only after 7 days and an associated 18 mm of rainfall.

Apples (Experiments 5 and 6). The final application of captan to apples added 1.5 and 3.3 mg kg^{-1} to the residues remaining from earlier treatments. Residues on McIntosh (experiment 5) failed to decline significantly over the 15 days following the final application despite an accumulated rainfall of 89 mm (Table VI). In the case of Delicious apples (experiment 6) residues did decline significantly but this occurred between day 0 and day 1 and was not accompanied by a rain. No further decline of captan residues was observed between day 1 and day 15 despite an 89–mm rainfall. Regression analyses between captan residue and rainfall did not show a significant correlation ($r^2 = 0.17$–0.39). Captan residues were below 5 mg kg^{-1} except for the postspray sample collected on day 0.

Apples (Experiment 7). The final application of captan added 9.0 and 12.2 mg kg^{-1} respectively after applying 1.7 and 3.4 kg ha^{-1}, i.e., Table VI. Apples in this

Table IV. Captan Residues in Grapes and Pears Treated with Captan at 2.8 and 3.4 kg ha^{-1}, Respectively, 1981–1983 (Experiments 2, 3, 4, and 8)

time of harvest	captan residue, mg kg^{-1}: DeChaunac grapes 1981 (expt 2)	DeChaunac grapes 1982 (expt 3)	Bartlett pears 1982 (expt 4)	Bartlett pears 1983 (expt 8)	accumulated rainfall, mm: 1981 (expt 2)	1982 (expt 3, 4)	1983 (expt 8)
prespray	0.11b[a]	<0.01c	0.5d	4.4bcd			
postspray–day 0	3.3a	1.7b	3.5ab	7.8a	0	0	0
day 1	4.5a	1.5b	3.7a	5.5ab	0	0	2
day 2	3.1a				0		
day 3		2.7a	2.2bc	4.7b		0	2
day 5	2.5a	2.1ab	1.1cd	5.9ab	18	12	10
day 7	3.4a	2.1ab	2.3bc	3.8bcd	19	12	18
day 10	4.6a	2.1ab	1.1cd	2.1cd	42	49	36
day 14	3.5a			1.7d	48		38
day 15		0.6c	1.2cd			89	
mean temp, °C	14.1	14.6	14.6	14.0			

[a] Values followed by the same letter are not significantly different at $P = 0.05$ (Duncan, 1951).

Table V. Captan Residues at Various Stages of Processing Grapes into Wine (Experiment 2)

treatment	data	days after treatment	captan residues, mg kg^{-1}: treated, mean ± SD	captan residues, mg kg^{-1}: untreated
destemmed grapes	7 Oct 82	8	2.2 ± 0.7	<0.01
stems	7 Oct 82	8	16 ± 3	<0.01
crushed,[a] destemmed grapes	7 Oct 82	8	1.9 ± 0.1	<0.01
pressed, fermented juice,[a] skins and seeds removed	14 Oct 82	14	<0.01	<0.01
fermented–end secondary	10 Nov 82	42	<0.01	<0.01
finished wine	14 June 83	258	<0.01	<0.01

[a] ca. pH 3.

Table VI. Captan Residues on Apples Treated with 1.7 and 3.4 kg ha^{-1} 1982–1983 (Experiments 5, 6, 7, and 9)

time of harvest	captan residues, mg kg^{-1}						accumulated rainfall, mm		
	McIntosh expt. 5	Delicious expt. 6	McIntosh[a] expt. 7		McIntosh expt. 9		expt. 5, 6	expt. 7	expt. 9
rate, kg ha^{-1}	3.4	3.4	1.7	3.4	1.7	3.4			
prespray	3.8a[b]	1.8b	3.0ef	3.5ef	3.2d	4.1cd			
postspray–day 0	5.3a	5.1a	12.0b	15.7a	4.2cd	9.4a	0	0	0
day 1	3.9a	2.8b	3.7e	8.5c	3.3d	4.2cd	0	0	2
day 3	4.4a	2.6b	4.3def	5.1de	4.7cd	4.1cd	0	0	2
day 5	3.8a	3.0b	4.3def	7.3cd	5.4bcd	9.4a	12	18	10
day 7	4.7a	3.5ab	3.4ef	3.9ef	3.9cd	6.9abc	12	19	18
day 10	3.3a	2.3b	3.0ef	3.2ef	3.2d	7.1abc	49	33	36
day 14					4.2cd	8.1ab			38
day 15	4.4a	2.2b					89		
day 18			1.9f	3.5ef				94	
mean temp, °C	14.6	14.6	13	13	14	14			

[a] Apples from experiment 7 were small ranging in size from 80 to 100 g while those from other experiments were of normal size and weight, i.e. 130–150 g. [b] Values followed by the same letter are not significantly different at $P = 0.05$ (Duncan, 1951).

trial were unusually small (80–100 g) which might have accounted for the large captan deposits. Residues declined significantly over the next 18 days with most of the disappearance occurring in the first 24 h after application. The linear regression for captan residue decline (y) and rainfall (x) produced the significant equation of $\log y = 0.721 - 0.005x$ ($F = 5.2$, $P = 0.05$, $r^2 = 0.51$) for the half rate (1.7 kg ha^{-1}) and $\log y = 0.763 - 0.094 \log \log x$ ($F = 5.1$, $r^2 = 0.50$) for the full rate (3.4 kg ha^{-1}). The half residue disappearance required 58 mm of rain for the half rate and 67 mm for a full rate. After half rate (1.7 kg ha^{-1}) applications residues of captan fell below the legal tolerance of 5 mg kg^{-1} within 24 h, but 7 days were required for residues from the full rate (3.4 kg ha^{-1}) to decline below mg kg^{-1} (Table VI).

Apples (Experiment 9). The application of 1.7 and 3.4 kg ha^{-1} of captan to McIntosh apples added only 1.0 and 5.3 mg kg^{-1} to the residue deposits already on the fruit from previous applications (Table IV). Residues of captan did not decline significantly between days 0 and 14. Rainfall during this period was low (38 mm) and had no effect on residues (Table VI). Regression analysis failed to reveal any correlation between residue and rainfall ($r^2 = 0.09$ and 0.11). Residues from 3.4 kg ha^{-1} were above the legal tolerance of 5 mg kg^{-1} over the entire 14 days of the test while residues on fruit treated with 1.7 kg ha^{-1} were at or below the MRL following application and for the 14 days thereafter.

DISCUSSION

The persistence of captan on fruit in the field was surprisingly longer than would have been predicted based on its instability in water. Wolfe et al. (1976) reported a pH dependent pseudo-first-order rate reaction constant for captan in water of $1.8 \pm 0.1 \times 10^{-5}$ s^{-1} for a pH range between 2 and 6 and for alkaline hydrolysis between pH 7–9 an average second-order rate constant of $5.7 \pm 0.4 \times 10^2$ M^{-1} s^{-1}. Frank et al. (1983) reported a half life of 1 h for captan in water at pH 8.5 and a temperature of 22 °C, 13 h at pH 8.5 and 5 °C and at pH 5.5 and 22 °C, and 13 days at pH 5.5 and 5 °C. The pseudo-first-order hydrolysis constant at 5 °C and pH 8.5 and at 22 °C and pH 5.5 was calculated at 1.5×10^{-6} s^{-1}. The average field temperatures for the nine trials were between 14 and 16 °C and rainfall varied from 8 mm to 77 mm per week. The pH of summer rainfall approximates 5.5 and hence the half residue disappearance period would have been predicted to be between 1 and 4 days in the event of rainfall. Of the nine experiments a correlation between rainfall and residue decline was demonstrated in five (experiments 1, 3, 4, 7, and 8) and no correlation in four. Of the five where a correlation was demonstrated half residue declines required 21 mm of rain in two and between 58 and 67 mm in three. In the four experiments (no. 2, 5, 6, and 9) where no correlation between rainfall and captan disappearance occurred, very little rain fell in the first seven days after treatment, i.e., 12 and 18 mm. In three trials rain between day 7 and 15 amounted to 77 mm, however, this failed to reduce residues. In the fourth trial only 20 mm fell in the second week, probably insufficient to effect captan removal.

When grapes were processed into wine, captan was rapidly hydrolyzed and disappeared in 7 days after crushing even at a pH of 3. Koivistoinen et al. (1965) demonstrated that captan declined in storage by as much as 39% in 7 days at 20 °C and 69% in 28 days at 10 °C. Koivistoinen et al. (1965) and Frank et al. (1983) demonstrated that processing and culinary procedures involving either the release of moisture or use of water resulted in substantial declines in residues.

Captan is an essential fungicide for the protection against fruit rot in graphes and pome fruit (Dunnet, 1982). This is especially the case with the development of resistance in *Venturia inaequalis* to dodine and benzimidazole fungicides and in *Botrytis cinerea* to benzimidazole and dicarboximide fungicides (Northover, 1984; OMAF, 1983). A surface deposit of 0.63 μg of captan cm^{-2} provided approximately 90% protection of susceptible apple foliage against infection by *Venturia inaequalis* under laboratory conditions (Northover, unpublished). For an apple of 7.0-cm diameter, of approximately spherical shape, and specific gravity of 1.0, a uniform deposit of 0.63 μg cm^{-2} is equivalent to a whole fruit residue of 0.54 mg kg^{-1}. For small fruits such as grape berries the fruit residue is higher in relation to surface deposits. For the 90% protection of sweet cherry fruits (2.0-cm diameter) against infection by *Monilinia fructicola* the calculated surface deposit was 0.9 μg cm^{-2}, equivalent to a whole fruit residue of 2.7 mg kg^{-1} (Northover, unpublished). A residue of 5 mg kg^{-1} of captan is therefore adequate for the protection of apple, pear, and grape fruits. In the nine experiments, residues declined to less than the 5 mg kg^{-1}, Canadian legal tolerance by day 5 on grapes, day 3 on pears, and day 7 on apples, except for the 3.4 kg ha^{-1} application rate in one experiment. Under field conditions a spray program of captan will normally control apple scab sufficiently to allow a half rate application (i.e., 1.7 kg ha^{-1}) to be made close to harvest and this is recommended by the authors.

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Laboratory and Field Studies on the Fate of 1,3,6,8-Tetrachlorodibenzo-*p*-dioxin in Soil and Sediments

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The fate of ^{14}C-ring-labeled 1,3,6,8-tetrachlorodibenzo-*p*-dioxin (TCDD) was studied in sandy loam soil under field conditions and in silty-clay pond and lake sediments under laboratory conditions. Dissipation of 1,3,6,8-TCDD from small field plots was relatively rapid with 44% of the applied radioactivity lost after 131 days posttreatment. In sediment, 80% of the radioactivity could still be accounted for as intact chemical after 675 days under static aerobic conditions (10 and 25 °C) or after 310 days under a nitrogen or air purge. Transformation of 1,3,6,8-TCDD to degradation products and unextractable radioactivity in soils and sediments was very slow. Unidentified polar products represented a maximum of 2.5% of extractable ^{14}C in field soils and 7.0% in sediments. DDT incubated in sediments under the same conditions had half-lives of <310 days.

INTRODUCTION

The fate of the 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD) isomer in soils and sediments has been studied extensively in laboratory and field experiments (Kearney et al., 1972; Ward and Matsumura, 1978; Tsushimoto et al., 1982; Nash and Beall, 1980). There has been much less study of the persistence and degradation of other dioxin isomers in soils and sediments. The 1,3,6,8-TCDD isomer is the principal tetrachlorodioxin contaminant in 2,4-D ester formulations (Cochrane et al., 1982) and diphenyl ether herbicides (Tamagishi et al., 1981). This isomer has also been reported to be a major component of total TCDD congeners in fly ash from a municipal incinerator (Buser et al., 1978) and in particulates from wood combustion (Nestrick and Lamparski, 1982).

The 1,3,6,8-isomer is reported to be much less toxic than the 2,3,7,8-isomer based on its ability to induce aryl hydrocarbon hydroxylase activity and on an LD_{50} (rat) of >100 mg/kg (Esposito et al., 1980). The fate of this compound is nevertheless of interest because of its possible widespread introduction to terrestrial and aquatic ecosystems as a herbicide and fly ash microcontaminant. 1,3,6,8-TCDD has been identified in fish from agricultural areas of Japan where diphenyl ether herbicides were used (Tamagishi et al, 1981) and in fish from Lake Michigan (Stalling et al., 1983).

The purpose of this study was to examine the fate of 1,3,6,8-TCDD by using ^{14}C-labeled compound in soils and sediments from the Canadian prairies, a region in which 2,4-D esters contaminated with this isomer were used in large quantities until recently, and to compare results with published reports on the 2,3,7,8-isomer.

MATERIALS AND METHODS

Chemicals. [^{14}C]1,3,6,8-TCDD (U-ring-labeled) was obtained from New England Nuclear, (Boston, MA) and purified before use by reverse-phase TLC by using a solvent system of acetone–water (95:5). The final product (>99.5% radiochemically pure, sp act 1701 Bq/μg) was dissolved in hexane–ethyl acetate (1:1) for addition to soils and in acetone for addition to sediment incubations. [^{14}C]*p,p'*-DDT (Amersham Radiochemicals, Oakville, Ont.)

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