## **QUINTOZENE (064)**

## **EXPLANATION**

Quintozene was evaluated as a periodic review by the 1995 JMPR. That Meeting estimated a number of maximum residue levels but agreed not to recommend their use as MRLs, and recommended the withdrawal of existing MRLs, because of the lack of critical supporting data on environmental fate. Some of the processing studies could not be evaluated because of their uncertain validity.

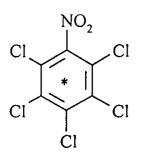
The CCPR in 1996 was informed that environmental fate studies would be made available to the JMPR (ALINORM 97/24, para 48). The compound was scheduled for evaluation by the JMPR in 1997, but rescheduled to 1998 at the request of the company.

The 1997 CCPR recommended the deletion of the CXL for banana as the use was not supported, and retained the other CXLs for 4 years according to the Periodic Review procedure. The Committee noted that data from supervised trials on head lettuce and potatoes would be submitted for evaluation by the 1998 JMPR (ALINORM 97/24A, para 54).

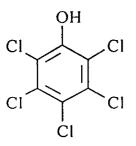
Data on environmental fate, residues in rotational crops, supervised trials on lettuce and the effects of processing on residues in cotton seed, peanuts and potatoes have been made available to the present Meeting by the manufacturer but no new information from supervised residue trials on potatoes was received. Information on residue analytical methods and national MRLs was provided by The Netherlands and on national MRLs by Germany and Poland.

## METABOLISM AND ENVIRONMENTAL FATE

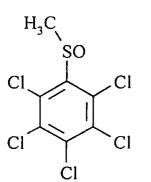
Figure 1. Structures, chemical names and abbreviation codes of quintozene and degradation products found in environmental fate studies.



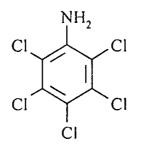
pentachloronitrobenzene quintozene (PCNB)



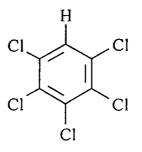
pentachlorophenol (PCP)



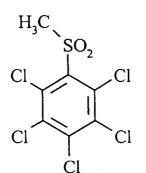
methyl pentachlorophenyl sulfoxide pentachlorothioanisole sulfoxide (PCTASO)



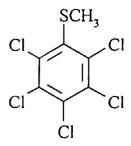
pentachloroaniline (PCA)



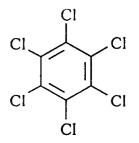
pentachlorobenzene (PB)



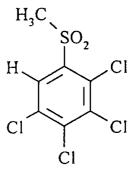
methyl pentachlorophenyl sulfone pentachlorothioanisole sulfone (PCTASOO)



methyl pentachlorophenyl sulfide pentachlorothioanisole (PCTA)



hexachlorobenzene (HCB)



methyl-2,3,4,5-tetrachlorophenyl sulfone 2,3,4,5-tetrachlorothioanisole sulfone TCTASOO)

#### **Environmental fate in soil**

#### **Photolysis**

Bowman (1988a) exposed sandy loam soil treated with  $[{}^{14}C]$ quintozene at 10.5 :g/g to a xenon arc lamp for 30 days. Radio-analysis demonstrated a decrease in the extractable  ${}^{14}C$  residues and a corresponding increase in the  ${}^{14}C$  bound to the soil. The levels of  $[{}^{14}C]$ quintozene in the sample extracts indicated a rate constant of 0.031/day and a half-life of 22 days for the exposed samples and a rate constant of 0.002/day and half-life of 367 days for the dark control samples. It was later discovered that these samples had been exposed to the xenon source for 25 hours so the dark control

study was repeated. The repeat showed that <sup>14</sup>C was increasingly bound to the soil with time. The extractable activity was essentially all due to quintozene and no degradation products were detected. A rate constant based on the binding of <sup>14</sup>C to the soil was calculated to be 0.007 (half-life 100 days).

A corrected photolysis rate constant ( $k_{exposed} - k_{dark}$ ) was calculated to be 0.024/day (half-life 28.5 days based on 30 days continuous exposure of the samples). The mean total radioactivity recovered was 97.5% of that applied. After 30 days of exposure PCA accounted for 35% of the extracted <sup>14</sup>C. No other photoproducts were detected.

Misra (1993a). A 30-day photolysis study with 12 hour light/12 hour dark cycles, of uniformly ring-labelled [<sup>14</sup>C]quintozene in a sandy loam soil at or near 75% field moisture capacity (FMC) was conducted at 25°C. Both PCA and PCTA were found in the irradiated and control samples but the rate of formation of PCA under irradiation was about twice that in the dark control (Table 1). The soil-bound activity in the presence of light was about twice that in the control. The half-life values in irradiated and control samples were 13 and 62 days respectively (Table 2).

Table 1. Concentrations of quintozene and its major degradation products in soil extracts (Misra, 1993a).

	<sup>14</sup> C residues as quintozene, mg/kg soil						
Irradiated				Cont	rol		
Time, hours	Quintozene	PCA	PCTA	Time, days	Quintozene	PCA	РСТА
0	9.6	ND	ND	0	9.4	NA	NA
13	8.1	0.38	ND	1	9.2	ND	ND
25	6.3	0.41	0.04	2	8.2	0.16	0.04
49	4.8	0.52	ND	4	8.0	ND	ND
99	5.2	0.68	0.05	8	7.4	0.04	0.05
186	3.4	0.83	0.32	15	7.7	0.37	0.22
261	2.6	0.88	0.31	22	6.9	0.44	0.36
359	1.6	0.70	0.21	30	6.4	0.42	0.30

NA: not analysed ND: below LOD

Table 2. Results of regression analysis (Misra, 1993a).

Parameters	Irradiated	Control
Constant	2.05	2.17
$\mathbb{R}^2$	0.942	0.826
No of observations	8	8
Degrees of freedom	6	6
X coefficient	-0.0044	-0.0009
Estimated half-life values	13 days	62 days

## Aerobic and anaerobic degradation

Daly (1989) treated sandy loam soil with uniformly ring-labelled [ $^{14}$ C]quintozene at 25 mg/kg in the dark at a temperature of 25°C. The soils were first incubated aerobically for 30 days, then the systems were flooded with 30 ml deionized water and 0.1 g of glucose was added to each tube to facilitate microbial activity. The air supplies were changed to nitrogen to further ensure anaerobic conditions. Samples were extracted with methanol on days 0 and 1 and subsequent samples were extracted with methanol/1N acetic acid (80:20). The supernatants were analysed by LSC and TLC.

The mean balance of radioactivity was 97.8%. TLC analysis of the soil extracts and water samples revealed the presence of quintozene and three known degradation products (Tables 3 and 4). Quintozene was detected in all the soil extracts and the day 90 flood water. PCA was the main degradation product and was found in all extracts and water samples after day 1. The aerobic and anaerobic half-lives of quintozene, based on first order degradation kinetics, were calculated to be 37 days and 15 days respectively.

Table 3. Distribution of quintozene and its degradation products in soil extracts determined by TLC (Daly, 1989).

Time, days	Quintozene, % of TRR	PCTA, % of TRR	PCA, % of TRR	PCP, % of TRR
0	78.8	9.1	ND	ND
1	85.9	5.6	ND	ND
3	63.8	4.5	23.8	ND
7	72.7	6.0	6.9	ND
14	63.6	3.5	15.5	ND
30	54.5	3.2	23.4	1.6
60	4.2	ND	86.6	1.8
90	3.8	ND	84.1	2.0

ND: below LOD

Table 4. Distribution of quintozene and its degradation products in flood water determined by TLC (Daly, 1989).

Times, days	Quintozene, % of TRR	PCA, % of TRR	PCP, % of TRR
60	ND	25.2	7.5
90	4.9	28.3	6.6

Misra (1993b) conducted a year-long study of the aerobic degradation of uniformly ringlabelled [<sup>14</sup>C]quintozene on a sandy loam at or near 75% FMC at 25°C in darkness. Test vessels containing 20 g dry weight equivalent of sandy loam soil were treated with a solution of [<sup>14</sup>C]quintozene in acetonitrile at 10 mg/kg and placed in a stainless steel container at 25°C. At intervals, the test container was purged with carbon dioxide-free humidified air to collect volatile compounds from the container and to maintain aerobic conditions in the test systems. The soil moisture was adjusted to 75% field moisture capacity at dosing and sampling. The soil samples were extracted with 10 to 20% 1N phosphoric acid in acetonitrile. The trapping solutions, test system wipe extracts, soil extracts and test vessel washes were analysed by LSC and HPLC. GC-MSD was used as a confirmatory method of identification. The TRR recovered at each sampling was from soil extracts, soil-bound <sup>14</sup>C residues (determined by combustion and LSC), and accumulated volatile compounds collected from the test system and trapping solutions (Table 5).

Incubation time, days	Extractable <sup>14</sup> C, %	Soil bound residues, %	Volatile compounds, %	Total recovery, %
0	99.4	2.7	ND	102.1
1	98.6	4.2	0.17	103.0
3	89.7	6.1	1.4	97.2
7	85.8	8.9	3.5	98.2
14	80.0	10.8	6.2	97.0
30	74.7	11.1	9.5	95.3
60	69.4	10.8	12.6	92.8
90	67.1	13.5	15.7	96.3
120	69.5	10.1	19.3	98.9
180	64.1	12.2	23.0	99.3
270	56.6	14.2	28.3	98.9
365	50.5	17.1	33.7	101.2
			mean	98.4
			SD	2.9

Table 5. <sup>14</sup>C balance (Misra, 1993b).

ND: below LOD

Most of the volatile <sup>14</sup>C during the aerobic incubation was due to [<sup>14</sup>C]quintozene. The other identified volatile compounds were PCA, PB and PCTA. Soil-bound <sup>14</sup>C residues increased to a maximum of 17% on day 365. The mean <sup>14</sup>C balance calculated for the duration of the study was  $98.6\pm5.7\%$ . The distribution of quintozene and its major degradation products in soil extracts are shown in Table 6.

Table 6. Concentration of quintozene and its major degradation products in soil extracts (Misra, 1993b).

Time,		Residue, mg/kg						
days	Quintozene	PCA	PCTA	PCTASO	PCTASO <sub>2</sub>			
0	9.96	ND	ND	ND	ND			
1	9.8	ND	ND	ND	ND			
3	8.4	0.36	0.14	ND	ND			
7	7.3	0.64	0.37	0.09	ND			
14	6.2	0.67	0.41	0.23	0.13			
30	5.0	0.71	0.49	0.33	0.28			
60	4.4	0.78	0.54	0.29	0.30			
90	3.9	0.76	0.64	0.28	0.34			
120	3.9	0.91	0.81	0.36	0.41			
180	3.2	0.91	0.73	0.34	0.49			
270	2.7	0.85	0.63	0.32	0.52			
365	2.3	0.80	0.53	0.37	0.61			

ND: below LOD

The half-life was calculated to be 189 days with a coefficient of determination of 0.79 using a first-order approximation and all the results. Independent first-order regression analysis of the first five and the last eight results yields half-lives of 20 and 278 days respectively with associated correlation coefficients of 0.95 and 0.92. This illustrates the non-linearity of quintozene degradation (Table 7).

Table 7. First order regression analysis, days 0 to 14 and 14 to 365, (Misra, 1993b).

Parameters	Day 0 to day 14	Day 14 to day 365
Constant	2.28	1.69
$\mathbb{R}^2$	0.955	0.923
Number of observations	5	8
Degree of freedom	3	6
X coefficient(s)	-0.034	-0.0025

Figure 2 shows the proposed pathways for the aerobic degradation of quintozene in soil. PCTA was apparently formed from PCA by deamination and nucleophilic displacement at the aromatic ring. PCTASO and PCTASO<sub>2</sub> were formed as the incubation increased by oxidation, with a corresponding decrease in the concentration of their precursor PCTA. PCA reached a maximum concentration of about 9% of the applied <sup>14</sup>C and remained almost constant from day 14 to the end of the study. PCA was the major primary degradation product of quintozene.

## Mobility (adsorption/desorption)

Esposito (1977) carried out laboratory leaching studies on sandy loam, silt loam, Smithdale sandy soil, and Carnasaw silt loam from the cotton-growing region of Arkansas, USA. [<sup>14</sup>C]quintozene was added uniformly to the top 5 cm of a 20 cm column of untreated soil. Three column volumes of aqueous 0.01 M CaCl<sub>2</sub> were then passed through the column. <sup>14</sup>C was measured in the CaCl<sub>2</sub> effluents and in 5 cm soil sections. Leaching amounted to 2 to 17% in the 4 soils and the <sup>14</sup>C only reached the adjacent untreated soil zone. Less than 0.1% of the added <sup>14</sup>C was found in the CaCl<sub>2</sub> effluent from all the soils. Leaching experiments with two soils aged for 30 days gave similar results.

Bowman (1987a, b) equilibrated aqueous solutions of  $[^{14}C]$ quintozene and  $[^{14}C]$ PCA with four soils and determined the adsorption and desorption coefficients. Liquid scintillation counting was used to measure the  $^{14}C$  concentrations in the aqueous phases (Tables 8 and 9).

Soil	% Organic carbon	Adsorption K <sub>d</sub>	Adsorption K <sub>OC</sub>	Desorption K <sub>d</sub>	Desorption K <sub>OC</sub>
Silt loam	0.4	24	6030	38	9584
Sandy loam	0.8	46	5807	47	5929
Sand	1.05	31	2966	34.5	3285
Clay loam	1.3	89	6852	110	8480

Table 8. Adsorption/desorption of [<sup>14</sup>C]quintozene by soil (Bowman, 1987a).

The radiochemical purity of the applied [ $^{14}$ C]quintozene was 99%, and that at the end of the adsorption period approximately 95% as determined by TLC. The recovery of  $^{14}$ C was 95.8%, 104%, 96.2% and 98.6% from the silt loam, sandy loam, sand and clay loam respectively (Bowman, 1987a).

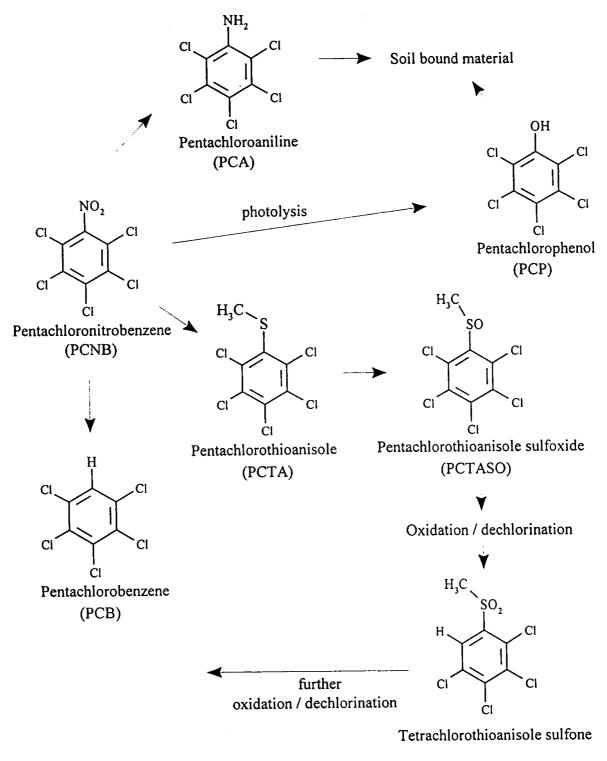
Table 9. Adsorption/desorption of [<sup>14</sup>C]PCA by soil (Bowman, 1987b).

Soil	% Organic carbon	Adsorption K <sub>d</sub>	Adsorption K <sub>OC</sub>	Desorption K <sub>d</sub>	Desorption K <sub>OC</sub>
Silt loam	0.4	39	9695	33	8273
Sandy loam	0.8	80	9991	67	8367
Sand	1.05	150	14256	40	3837
Clay loam	1.3	267	20543	237	18256

The radiochemical purity of the applied [ $^{14}$ C]PCA was 98%, and that at the end of the adsorption period was approximately 91% as determined by TLC. The recovery of  $^{14}$ C was 103%,

105%, 105% and 101% for the silt loam, sandy loam, sand and clay loam respectively (Bowman, 1987b).

Figure 2. Proposed degradation pathways of quintozene in soil (Misra, 1993b).



(TCTASOO)

Quintozene is immobile to slightly mobile in soil (the Freundlich adsorption constants in silt loam, sandy loam, clay loam and sand with about 1% organic matter were 24, 46, 89 and 31 respectively).

The Freundlich constants of PCA in these soils were even higher (39, 80, 267 and 150 respectively) indicating lower mobility than the parent compound.

# Terrestrial field dissipation

Several field dissipation studies (Irissarri,1988; Lengen, 1989; Rice, 1989a,b; Noon, 1990; Harned, 1993, 1997a) showed that quintozene applied to the soil surface disappeared more quickly than when applied by incorporation. When Lengen (1989) applied it to a turf surface in California the half-life was 35 days, but incorporation in soil in California and Minnesota (Rice, 1989) gave half-lives of 128-193 days. When quintozene was applied to a soil surface in Georgia (USA) at 11.2 kg ai/ha (Harned, 1993) the half-life was 13 days but when incorporated in a Texas soil before planting cotton at 2.2 kg ai/ha the half-life was 135 days (Harned, 1997a). In the recent dissipation studies by Harned the level of quintozene decreased from 17.6 mg/kg to 0.8 mg/kg by day 545, the end of the study. The main product was PCA which was present at a terminal level of 3.2 mg/kg. PB, PCTA, PCTASO and TCTASO<sub>2</sub> were formed and declined, the highest residue being 0.47 mg/kg (PCTA). The highest PCP residue was only 0.018 mg/kg. The impurity HCB ranged randomly from undetectable to 0.017 mg/kg.

It can be concluded that quintozene would not be a source of groundwater contamination and would dissipate significantly before subsequent annual applications.

# Rotational crops

The uptake of [*phenyl*-U-<sup>14</sup>C]quintozene by rotational crops was investigated in 1990, 1993 and 1997.

In a confined rotational crop study [<sup>14</sup>C]quintozene was applied at about 34 kg ai/ha (Halls, 1990). The three most common rotational crop scenarios, replanting after crop failure (30 days), immediate recropping (120 days) and following-year recropping (365 days) were reproduced. Quintozene was applied on 11 May, 1988. Lettuce, turnips and wheat were planted after the appropriate ageing period. The results are shown in Table 10.

	_			
Sample	Days after treatment	Crop age, days	Soil residues, mg/kg	Crop mg/kg
Immature lettuce				

Table 10. <sup>14</sup>C in rotational crops, expressed as quintozene (Halls, 1990).

Sample	Days after	Crop age,	Soil residues,	Crop residues,
	treatment	days	mg/kg	mg/kg
Immature lettuce				
30 day plot	75	45	84	3.3
120 day plot	154	34	7.2	1.4
365 day plot	404	39	5.9	5.7
Mature lettuce				
30 day plot	91	61	10	1.6
120 day plot	187	67	8.7	0.15
365 day plot	426	61	5.4	0.61
Immature turnips				
30 day plot	63	33	6.3	4.6
120 day plot	154	34	5.7	2.4
365 day plot	404	39	6.4	1.6

Sample	Days after	Crop age,	Soil residues,	Crop residues,
-	treatment	days	mg/kg	mg/kg
Mature turnip tops				
30 day plot	104	74	7.1	3.6
120 day plot	187	67	5.5	1.7
365 day plot	426	61	5.6	0.73
Mature turnip roots				
30 day plot	104	74	7.1	20
120 day plot	187	67	5.5	4.8
365 day plot	426	61	5.6	1.5
Immature wheat			·	
30 day plot	75	45	8.7	2.6
120 day plot	154	34	5.9	5.0
365 day plot	420	53	5.4	5.1
Mature wheat straw	·		·	
30 day plot	107	77	7.6	23
120 day plot	212	92	6.6	22
365 day plot	472	107	4.8	26
Mature wheat hulls	·		·	
30 day plot	107	77	7.6	11
120 day plot	212	92	6.6	6.1
365 day plot	472	107	4.8	8.0
Mature wheat grain	·	•		
30 day plot	107	77	7.6	0.33
120 day plot	212	92	6.6	0.71
365 day plot	472	107	4.8	0.38

Murty (1993) reported the uptake of quintozene by rotational wheat planted in soil that had been treated 30, 120 or 365 days previously with [ $^{14}$ C]quintozene at about 34 kg ai/ha (Table 11).

Table 11	Distribution	of ${}^{14}C$ r	esidues	in rotational	wheat	(Murty	1993)
	Distribution		coluces.	in iotational	wheat	(munty,	1995).

Planting interval	<sup>14</sup> C, mg/kg as quintozene							
	Straw	Hull	Grain					
30 days	22	9.6	0.38					
120 days	27	7.5	0.54					
365 days	24	7.3	0.42					

In the previous studies (Halls, 1990; Murty, 1993) the components of the residue were not identified. Harned (1997b) used more modern analytical techniques to isolate and identify the low levels of the residue constituents.

[<sup>14</sup>C]quintozene was applied broadcast to soil and then manually incorporated. The treated soil was allowed to age for periods of 30, 120 or 365 days after which turnips, lettuce and wheat were planted. A rate of 2.2 kg ai/ha was applied to match US GAP for cotton and 11 kg ai/ha to match US GAP for peanuts. A rate of 34 kg ai/ha was applied only for rotational wheat. The results are shown in Table 12.

Sample	Days after	Treatment,	<sup>14</sup> C, 1	mg/kg as quintozene
_	treatment	kg ai/ha	Total	Parent
Turnip top	365	2.2	0.05	ND
	365	11.2	0.12	ND
Turnip root	365	2.2	0.05	ND
	365	11.2	0.21	ND
Lettuce, mature	30	11.2	0.15	0.023
	120	11.2	0.10	0.001
	365	11.2	0.43	0.006
Lettuce, immature	30	11.2	0.18	0.032
	120	11.2	0.15	0.004
	365	11.2	0.56	0.005
Wheat forage	30	2.2	0.38	NA
C	30	11.2	3.0	NA
	30	33.6	4.1	ND
	120	2.2	0.53	NA
	120	11.2	3.4	NA
	365	2.2	0.17	NA
	365	11.2	0.64	NA
Wheat grain	30	2.2	0.09	NA
	30	11.2	0.79	NA
	30	33.6	3.7	NA
	120	2.2	0.12	ND
	120	11.2	0.94	NA
	365	2.2	0.03	NA
	365	11.2	0.14	NA
Wheat straw	30	2.2	2.2	NA
	30	11.2	11	NA
	30	33.6	24	ND
	120	2.2	3.5	0.012
	120	11.2	17	NA
	365	2.2	1.3	NA
	365	11.2	4.6	NA

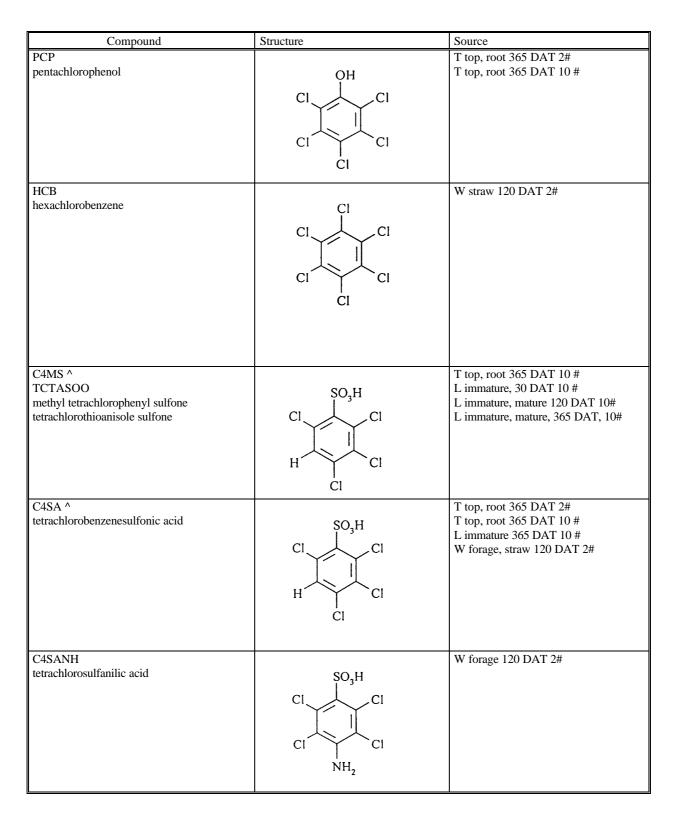
Table 12. Results of rotational crop studies (Harned, 1997b).

NA: not analysed ND: not detected

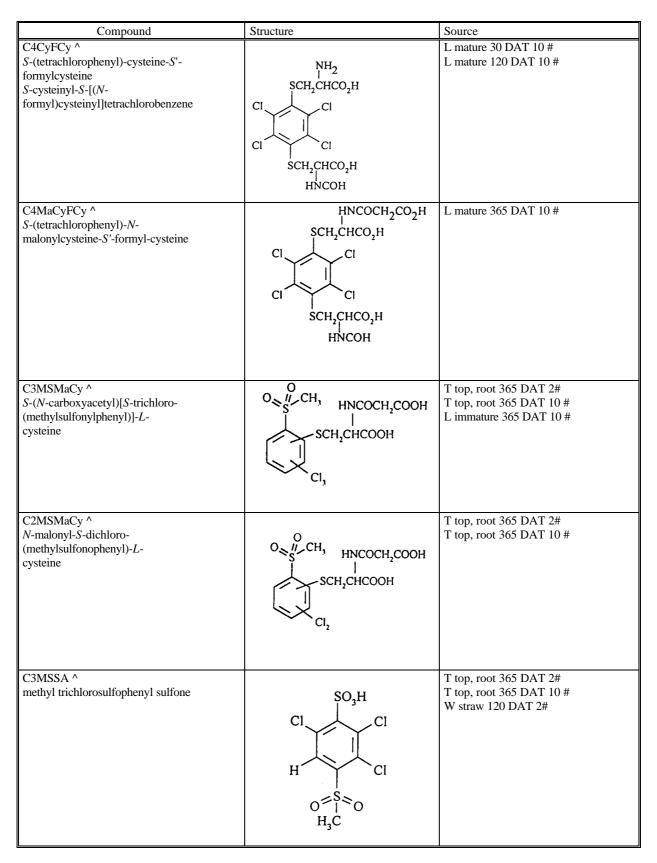
The compounds were extracted from the plant tissues and isolated by HPLC, TLC and solidphase extraction (SPE). Identification was mainly by mass spectrometry with GLC and LC sample introduction combined with electrospray ionization and tandem quadruple MS-MS where required. Table 13 shows the compounds identified in rotational crops with their structures and abbreviation codes.

Compound	Structure	Source
Quintozene PCNB pentachloronitrobenzene	$CI \xrightarrow{NO_2} CI$ $CI \xrightarrow{*   CI}$ $CI \xrightarrow{CI}$	L immature, mature 30 DAT 10 # L immature, mature 120 DAT 10 # L immature, mature 365 DAT 10 # W straw, 120 DAT 2#
PB pentachlorobenzene		L mature 30 DAT 10 #
PCA pentachloroaniline	$CI \rightarrow CI \rightarrow CI$ $CI \rightarrow CI$ $CI \rightarrow CI$	T top, root 365 DAT 2# T root, 365 DAT 10 # L immature, mature 30 DAT 10 # L immature, mature 120 DAT 10 # L immature, mature 365 DAT, 10 # W forage, straw 30 DAT 30 # W straw 120 DAT 2#
PCTA methyl pentachlorophenyl sulfide pentachlorothioanisole	CI CI CI CI CI CI	T root, 365 DAT 2# T root, 365 DAT 10 # L immature, 30 DAT 10 # L immature, mature 120 DAT 10 # L immature, mature 365 DAT, 10 # W forage, straw 30 DAT 30 # W straw 120 DAT 2#
PCTASO methyl pentachlorophenyl sulfoxide pentachlorothioanisole sulfoxide	H <sub>3</sub> C SO Cl Cl Cl Cl Cl	T top, root365 DAT 2# T top, root 365 DAT 10 #
C5SA pentachlorobenzenesulfonic acid	CI CI CI CI CI	T top, root 365 DAT 2# T top, root 365 DAT 10 # L mature 120 DAT 10 # L immature 365 DAT 10 # W forage, straw 120 DAT 2#

# Table 13. Compounds identified in rotational turnip, lettuce and wheat (Harned, 1997b).



Compound	Structure	Source
C3MS ^ methyl trichlorophenyl sulfone trichlorothioanisole sulfone	$H_{3}C$ $SO_{2}$ $H$ $Cl$ $Cl$ $H$ $Cl$ $H$	T top, root 365 DAT 2# T top, root 365 DAT 10 # L immature 30 DAT 10 # L immature, mature 120 DAT 10 # L immature, mature 365 DAT 10 # W straw 30 DAT 30 # W straw 120 DAT 2#
C3SA ^ trichlorobenzenesulfonic acid	H CI H CI H	T top, root 365 DAT 2# T top, root 365 DAT 10 # W straw 120 DAT 2#
C3MSOH ^ hydroxytrichlorophenyl methyl sulfone hydroxytrichlorothioanisole sulfone)	$H_3C$ $SO_2$ H HO CI CI	W straw 120 DAT 2#
C2SA ^ dichlorobenzenesulfonic acid	H CI H H	T top, root 365 DAT 2# T top, root 365 DAT 10 #
C5MaCy <i>N</i> -malonyl- <i>S</i> -pentachlorophenyl- <i>L</i> - cysteine	$CI \rightarrow CI \rightarrow$	L mature 30 DAT 10 #



^position of ring substituents indeterminate; more than one isomer likely

 $\begin{array}{ll} T = turnip, \ L = lettuce, \ W = wheat, \ DAT = days \ after \ treatment. \ 2\# = 2.2 \ kg \ ai/ha, \ 10\# = 11.2 \ kg \ ai/ha, \ 30\# = 33.6 \ kg \ ai/ha \\ Code \ key: \\ Code \ key: \\ Cn = phenyl \ ring \ with \ n \ chlorine \ atoms; \ MX = methyl \ sulfoxide; \ MS = methyl \ sulfone; \\ SA = sulfonic \ acid; \ Ma = malonyl; \ Cy = cysteinyl; \ OH = hydroxy; \ NH = amino \\ F = formyl \end{array}$ 

Tables 14-17 show the quantitative results from the application of 11.2 kg ai/ha before planting lettuce. An average of 49% of the TRR was identified and quantified; the remainder of the radioactivity was either bound to plant material (about 31% of the TRR) and hydrolysed with enzymes and KOH, or was present in polar extracts (Tables 15, 16) as a complex mixture of metabolites, typically <0.03 mg/kg each. An average of 96% of the nonpolar compounds in lettuce extracts (29-50% of the TRR) were identified and quantified (Table 14). Non-proteolytic enzymes at pH 5 and pH 7 did not release significant amounts of bound material, and what was released was a complex mixture at trace levels. Most of the bound residues were associated with proteinaceous material and lignin, as evidenced by significant amounts of residues released by proteases and KOH (about 25% and 36% of the bound residues respectively). These bound residues appear to be related to PCA, which was the only compound identified in these fractions (Table 17).

Sample	Non-polar ex	tract		%	
-	% of TRR	:g/kg as quintozene	Compound <sup>1</sup>	:g/kg, as quintozene	identified
30 DAT	34	53	PCNB	32	112
immature			C3MS	3.9	
(trial 570)			PCA	15	
			PCTA	3.1	
			C4MS	2.5	
			Conjugate 330 <sup>2</sup>	2.8	
30 DAT	29	31	PB	0.3	106
mature			PCNB	23	
(trial 596)			PCA	6.9	
~ /			Conjugate 330	2.8	
120 DAT	44	60	PCNB	3.9	79
immature			C3MS	5.0	
(trial 571)			PCA	7.1	
()			PCTA	2.5	
			C4MS	19	
			Conjugate 330	3.9	
			Unknowns-		
			(3 metabolites)	6.2	
120 DAT	50	48	PCNB	0.8	91
mature			C3MS	5.6	
(trial 597)			PCA	11	
( ,			PCTA	0.4	
			C4MS <sup>3</sup>	8.4	
			C4MS <sup>3</sup>	6.5	
			Conjugate 330	11	
365 DAT	36	206	PCNB	4.9	95
immature			C3MS	63	
(trial 572)			PCA	48	
()			PCTA	4.6	
			C4MS	46	
			C4MS	15	
			Conjugate 330	15	
365 DAT	36	131	PCNB	5.6	90
mature	20		C3MS	28	20
(trial 598)			PCA	39	
(			PCTA	5.0	
			C4MS	20	
			C4MS	6.9	
			Conjugate 330	13	

Table 14	Quantification	of non n	olar com	nounds in	lattuca (	Uarnad	1007h)
1 abic 14.	Quantineation	or non-p	olai com	pounds m	Iculue (	I lameu,	19970).

<sup>1</sup> See Table 13 for structures <sup>2</sup> Conjugate 330: cysteinyl conjugate, exact structure not confirmed

<sup>3</sup>Two methyl tetrachlorophenyl sulfone isomers were found, substitution positions not known because insufficient material for NMR

Peak RT, min	Metabolite <sup>1</sup>	Total peak, mg/kg as quintozene	Metabolite, mg/kg as quintozene	Total peak, % of TRR	Metabolite, mg/kg, % of TRR
37.0	C3MSMaCy	0.029	0.012	6.9	2.9
37.0	C4MaCyFCy	0.029	0.007	6.9	1.7
41.0	C4SA	0.023	0.012	5.5	2.9
55.0	C5SA	0.028	0.024	6.7	5.7

Table 15. Polar metabolite concentrations in lettuce, trial 598, 365 DAT, mature (Harned, 1997b).

<sup>1</sup>See table 13 for structures RT: retention time

Table 16. Summary of polar metabolite concentrations in lettuce, trials 596 and 597, (Harned, 1997b).

Sample	Metabolite	Fraction	mg/kg as quintozene	% of TRR
596 (30 DAT, mature)	C4CyFCy	ether	0.0019	1.8
596	C5MaCy	ether	0.0029	2.6
(30 DAT, mature)				
596 (30 DAT, mature)	C5MaCy	acidic water	0.0044	4.1
597 (120 DAT, mature)	C4CyFCy	ether	0.0027	2.8
597 (120 DAT, mature)	C5SA	acidic water	0.0021	2.2

Table 17. Pentachloroaniline (PCA) concentrations in lettuce digests and KOH hydrolysates (Harned, 1997b).

Sample	PCA, mg/kg	% of TRR
trial 570, protease digest	0.002	1.4
(30 DAT, immature)		
trial 572, protease digest	0.004	0.66
(365 DAT, immature)		
trial 598, protease digest	0.003	0.59
(365 DAT, mature)		
trial 570, KOH hydrolysate	0.008	5.3
(30 DAT, immature)		
trial 572, KOH hydrolysate	0.013	2.3
(365 DAT, immature)		
trial 596, KOH hydrolysate	0.009	8.5
(30 DAT, mature)		
trial 598, KOH hydrolysate	0.007	1.7
(365 DAT, mature)		

Fourteen metabolites were isolated from turnips and identified. Sulfonic acids and *N*-malonylcysteinyl conjugates were the major metabolites in the MeOH fractions of both tops and roots. The phenyl sulfoxides and sulfones were mainly in the CHCl<sub>3</sub> fractions of the tops, whereas pentachloroaniline was predominant in the CHCl<sub>3</sub> fractions of the roots. Reduction to pentachloroaniline and formation of PCTA through glutathionyl conjugation are two major metabolic pathways, followed by oxidation of the PCTA to the sulfonic acid or sulfoxide and sulfone. Tables 18 and 19 show the metabolite distribution in turnip tops and roots respectively. Table 18. Distribution of metabolites in turnip tops (Harned, 1997b).

Fraction	HPLC	Metabolite			Sam	ple			
	RT, min			L-tops			H-tops		
			% of <sup>14</sup> C in fraction	% of TRR in tops	mg/kg as quintozene	% of <sup>14</sup> C in fraction	% of TRR in tops	mg/kg as quintozene	
Me0H	4.0-5.0	Unknown	4.05	1.96	0.001	9.02	5.87	0.008	
	17.5-18.5	C3MSSA & C2MSMaCy	18.5	8.91	0.004	25.1	16.3	0.024	
	18.5-19.5	C2SA & C3MSMaCy	47.9	23.1	0.011	27.8	18.1	0.026	
	21.0	C3SA	12.9	6.24	0.003	-	-	-	
	23.0	C4SA	6.83	3.30	0.002	13.2	8.60	0.012	
	24.5-25.0	C5SA	6.61	3.19	0.002	8.27	5.38	0.008	
	27.0	Unknown	3.26	1.57	0.001	4.16	2.71	0.004	
	29.5	Unknown	-	-	-	5.88	3.82	0.006	
	32.0	Unknown	-	-	-	6.54	4.25	0.006	
Subtotal			100	48.3	0.024	100	65	0.094	
CHCl <sub>3</sub>	17.0	C3MSSA & C2MSMaCy	12.2	3.46	0.002	-	-	-	
	20.0	C3SA	7.30	2.07	0.001	-	-	-	
	22.5-23.0	C4SA	13.2	3.75	0.002	8.48	1.10	0.002	
	26.0-27.0	C5SA	5.47	1.55	0.001	4.90	0.64	0.001	
	28.5	Unknown	-	-	-	3.94	0.51	0.001	
	30.0-31.0	Unknown	4.47	1.27	0.001	5.85	0.76	0.001	
	32.5	Unknown	-	-	-	6.69	0.87	0.001	
	34.0-34.5	Unknown	8.85	2.51	0.001	4.66	0.60	0.001	
	37.5-38.0	C3MS	28.5	8.06	0.004	39.7	5.15	0.007	
	39.5-40.0	C3MS & PCTASO	14.0	3.95	0.002	16.5	2.14	0.003	
	41.5	C4MS	-	-	-	5.97	0.77	0.001	
	44.0	PCP	2.01	0.57	< 0.001	3.35	0.43	0.001	
	52.0	PCA	4.01	1.14	0.001	-	-	-	
Subtotal			100.5	28.3	0.015	100	13	0.019	
Water	4.5-5.0	Unknown	28.4	2.72	0.001	11.3	1.08	0.001	
	18.0-19.0	C3MSSA & C2MSMaCy	32.6	3.11	0.002	39.9	3.79	0.005	
	21.0	C3SA	21.6	2.06	0.001	12.6	1.19	0.002	
	23.0-24.0	C4SA	10.9	1.04	0.001	17.4	1.65	0.002	
	25.0	C5SA	6.58	0.63	< 0.001	-	-	-	
	30.0	Unknown	-	-	-	8.06	0.77	0.001	
	33.0	Unknown	-	-	-	10.7	1.02	0.001	
Subtotal			100.1	9.56	0.005	100	9.5	0.012	
Hexane	NA			4.63	0.002		3.50	0.005	
PES	NA			9.18	0.004		8.98	0.013	
Total				100	0.050		99.9	0.143	

NA: not analysed L-tops: treated with 2.2 kg ai/ha, H-tops: treated with 11.2 kg ai/ha PES: post-extraction solid fraction

Fraction	HPLC	Metabolite			Sar	nple	nple			
	RT,			L-roots		H-roots				
	min		% of <sup>14</sup> C in fraction	% of TRR in roots	mg/kg as quintozene	% of <sup>14</sup> C in fraction	% of TRR in roots	mg/kg as quintozene		
Me0H	3.5-4.5	Unknown	9.92	3.14	0.002	11.4	2.98	0.007		
	17.5-19.0	C3MSSA & C2MSMaCy	22.0	6.96	0.004	9.35	2.45	0.006		
	19.5-20.0	C2SA & C3MSMaCy	19.3	6.11	0.003	61.6	16.2	0.039		
	20.5-21.5	C3SA	9.40	2.97	0.002	11.8	3.1	0.008		
	22.0-23.5	C4SA	28.3	8.96	0.005	2.98	0.78	0.002		
	25.0-26.5	C5SA	5.97	1.89	0.001	2.87	0.75	0.002		
	31.5	Unknown	5.07	1.6	0.001		-	-		
Subtotal			99.96	31.63	0.018	100	26.26	0.064		
CHCl <sub>3</sub>	17.0	C3MSSA & C2MSMaCy	-	-	-	3.57	1.1	0.003		
	18.0	C3SA	-	-	-	3.46	1.07	0.003		
	37.5	C3MS	7.93	2.72	0.002	2.79	0.86	0.002		
	40.0	C3MS & PCTASO	9.02	3.09	0.002	5.8	1.79	0.004		
	41.0	C4MS	-	-	-	4.35	1.34	0.003		
	43.0-43.5	PCP	4.12	1.41	0.001	4.85	1.49	0.004		
	52.0	PCA	76.4	26.2	0.015	71.5	22.0	0.054		
	54.5	PCTA	2.48	0.85	< 0.001	3.68	1.13	0.0031		
Subtotal			99.95	34.25	0.02	100	30.78	0.076		
Hexane- EtOAc	48.0	Unknown	-	-	-	5.35	1.36	0.003		
	51.5	PCA	100	20	0.01	87.4	22.2	0.053		
	54.5	PCTA	-	-	-	7.24	1.84	0.004		
Subtotal			100	20	0.01	100	25.4	0.06		
AQ-2	NA			1.68	0.001		5.32	0.013		
AQ-3	NA			1.92	0.001		2.63	0.006		
CH <sub>2</sub> Cl <sub>2</sub>	NA			2.32	0.001		2.34	0.006		
PES	NA			8.24	0.004		7.3	0.018		
Total				100.04	0.055		100.03	0.243		

Table 19. Distribution of metabolites in turning	p roots (Harned, 1997b).
--	--------------------------

NA: not analysed

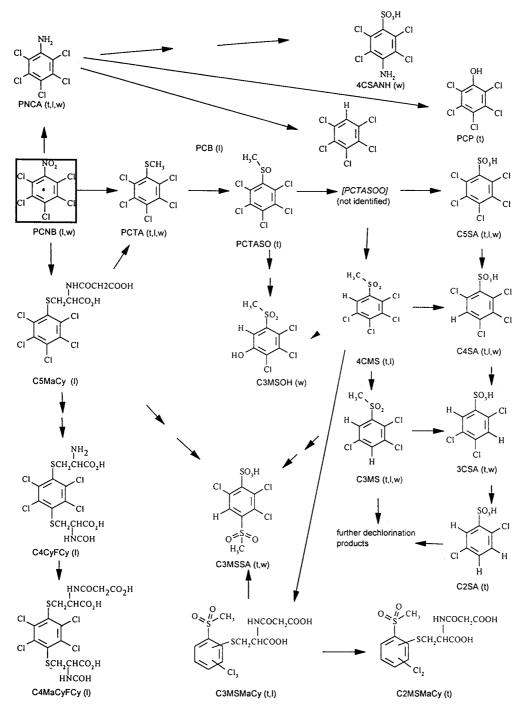
L-roots: treated with 2.2 kg ai/ha, H-roots: treated with 11.2 kg ai/ha PES: post-extraction solid fraction

Twelve compounds were isolated from wheat: pentachlorobenzenesulfonic acid, 0.14 mg/kg in straw; three isomers of tetrachlorobenzenesulfonic acid, 0.08, 0.33 and 0.06 mg/kg in straw, 0.22 mg/kg in forage; an isomer of trichlorobenzenesulfonic acid, 0.17 mg/kg in straw; methyl trichlorosulfophenyl sulfone, 0.11 mg/kg in straw; hydroxytrichlorophenyl methyl sulfone, 0.009 mg/kg in grain; pentachloroaniline, 0.013 mg/kg in straw; pentachlorothioanisole, 0.019 mg/kg in straw, and quintozene 0.023 mg/kg in straw from the 2.2 kg ai/ha treatment planted at 120 days; tetrachlorosulfanilic acid, 0.15 mg/kg in forage from 11.2 kg/ha, 30-day planting; methyl trichlorophenyl sulfone, 0.15 mg/kg in forage from 33.6 kg ai/ha, 30-day planting.

In summary, the identification of terminal metabolites indicated that reduction and conjugation as well as dechlorination are the main metabolic pathways of quintozene in wheat. The reduction product PCA was detected in straw and forage. Oxidation of PCTA, derived from glutathione conjugates produced by plant metabolism or possibly taken up by the roots from the soil,

yielded other major metabolites, the sulfonic acids and sulfones. Proposed metabolic pathways of quintozene in rotational crops are shown in Figure 3.

Figure 3. Proposed metabolic pathways of quintozene in rotational crops (Harned, 1997b). t: turnip, l: lettuce, w: wheat



Gaydosh (1996a,b) applied unlabelled quintozene to bare soil at the maximum GAP rate for peanuts (11 kg ai/ha), allowed the soil to age for 30, 120 or 365 days and then planted wheat, lettuce and turnips (365 days only) in two field trials in the typical peanut growing regions of Georgia and Texas. At both locations the test substance was applied in bands using common granular application equipment at a typical pegging time for peanuts and 15 or 45 days pre-harvest. The rotational crops were planted within the treated band for maximum effect. The results are shown in Tables 20 and 21.

Table 20. Residues in rotational crops planted 365 days after quintozene application (Gaydosh, 1996a).

Sample				Residu	e, mg/kg			
	PCNB	PCB	HCB	PCTA	PCA	TCA	TCTASOO	PCTASO
Immature wheat	<0.005 (8)	< 0.005 (8)	< 0.005 (8)	< 0.005 (8)	< 0.005 (8)	<0.005 (8)	<0.005 (8)	<0.005 (8)
Mature grain	<0.005 (8)	<0.005 (8)	<0.005 (8)	<0.005 (8)	<0.005 (6) 0.0155 0.0074	<0.005 (8)	<0.005 (8)	<0.005 (8)
Mature straw	<0.005 (8)	<0.005 (8)	< 0.005 (8)	<0.005 (8)	<0.005 (8)	<0.005 (8)	<0.005 (8)	<0.005 (8)
Lettuce	< 0.005 (8)	< 0.005 (8)	< 0.005 (8)	< 0.005 (8)	< 0.005 (8)	< 0.005 (8)	< 0.005 (8)	< 0.005 (8)
Turnip tops	<0.005 (8)	< 0.005 (8)	< 0.005 (8)	< 0.005 (8)	< 0.005 (8)	<0.005 (8)	< 0.005 (8)	<0.005 (8)
Turnip roots	<0.005 (8)	<0.005 (8)	<0.005 (8)	<0.005 (8)	<0.005 (4) 0.014 0.014 0.009 0.012	<0.005 (5) 0.01 0.006 0.007	<0.005 (8)	<0.005 (8)

TCA: tetrachloroaniline

Table 21. Residues in rotational crops planted 30 and 120 days after quintozene application (Gaydosh, 1996 b).

Sample				Residue	, mg/kg			
	PCNB	PCB	HCB	PCTA	PCA	TCA	TCTASOO	PCTASO
30 day planting								
Immature wheat	< 0.005 (8)	< 0.005 (8)	<0.005 (8)	< 0.005 (8)	< 0.005 (7)	<0.005 (8) 0.007	<0.005 (8)	< 0.005 (8)
Mature grain	<0.005 (8)	< 0.005 (8)	< 0.005 (8)	<0.005 (8)	< 0.005 (8)	<0.005 (8)	<0.005 (8)	<0.005 (8)
Mature straw	< 0.005 (8)	<0.005 (8)	< 0.005 (8)	< 0.005 (8)	< 0.005 (8)	< 0.005 (8)	< 0.005 (8)	<0.005 (8)
Lettuce	<0.005 (4) 0.010 0.011 0.013 0.012	<0.005 (8)	<0.005 (8)	<0.005 (8)	<0.005 (8)	<0.005 (8)	<0.005 (8)	<0.005 (8)
120 day pla	nting <sup>1</sup>							
Immature wheat	< 0.005 (6)	< 0.005 (6)	< 0.005 (6)	< 0.005 (6)	< 0.005 (6)	< 0.005 (6)	< 0.005 (6)	<0.005 (6)
Mature grain	< 0.005 (6)	< 0.005 (6)	< 0.005 (6)	< 0.005 (6)	< 0.005 (6)	< 0.005 (6)	< 0.005 (6)	<0.005 (6)
Mature straw	< 0.005 (6)	< 0.005 (6)	< 0.005 (6)	< 0.005 (6)	<0.005(6)	< 0.005 (6)	< 0.005 (6)	<0.005 (6)

Sample		Residue, mg/kg								
	PCNB	PCB	HCB	PCTA	PCA	TCA	TCTASOO	PCTASO		
Lettuce	<0.005 (3) 0.008 0.005 0.008	<0.005 (6)	<0.005 (6)	<0.005 (6)	<0.005 (6)	<0.005 (6)	<0.005 (6)	<0.005 (6)		

<sup>1</sup>The loss of one 120-day plot in Texas reduced the number of samples to 6 TCA: tetrachloroaniline

## **Environmental fate in water**

No data were submitted on natural aquatic or water/sediment systems. Both the following studies relate to the stability of the purified active ingredient in buffered water.

Bowman (1988b) incubated [<sup>14</sup>C]quintozene in sterile aqueous buffer containing less than 1% acetonitrile as co-solvent at 25°C and pH 5, 7 and 9 for 30 days. The half-life could not be determined experimentally as there was too little degradation. It was estimated to be more than 180 days. No hydrolysis products were detected at any pH. 96-106% of the initial radioactivity was recovered.

Horree (1992) determined the photodegradation of [ $^{14}$ C]quintozene at 25°C and pH 5 after 32 hours continuous exposure to a xenon arc lamp. The half-life was 13.4 hours (R<sup>2</sup>=0.99). Polar photodegradation products, accounting for 50% of the applied radioactivity after 32 hours, were identified as a mixture of 8 to 10 isomeric chlorophenols and/or chloronitrophenols formed by dechlorination of the benzene ring and subsequent hydroxylation at the same position(s). Identification was by derivatization with chloroacetic anhydride and MS. Volatile photodegradation products accounted for 20% of the applied radioactivity after 32 hours, none individually exceeding 10% of the original quintozene. The recovered  $^{14}$ C accounted for 88-96% of the applied radioactivity.

#### METHODS OF RESIDUE ANALYSIS

#### **Analytical methods**

The Meeting received details of analytical methods for determining quintozene, the impurities HCB and PB and the metabolites PCA and PCTA in meat, milk and eggs (Griffith, 1975) and soil (Griffith, 1970) and a reference to the latest edition of the official methods of analysis in The Netherlands (Anon., 1996).

Griffith (1975) extracted egg whites, egg yolks, fat, liver and bile with acetonitrile, white meat and faeces with hexane, and blood with a mixture of acetone and hexane (1:10). After the addition of water, liquid-liquid partition with hexane and further clean-up the compounds were determined by GLC with an ECD. An LOD of 0.005 mg/kg with a recovery of 60% was claimed but no validation data were submitted.

Griffith (1970) in a similar method extracted soil with acetone, adding water and subsequently partitioning the compounds into hexane. The hexane extract was analysed by GLC with an ECD. An LOD of 0.005 mg/kg with 60% recovery was claimed but with no validation data.

The latest version of the official analytical method of The Netherlands for quintozene, PCA and PCTA, reported by the 1995 JMPR, was reported (Anon., 1996).

# **Definition of the residue**

The 1995 JMPR considered that the residue definition for risk assessment purposes for plant and animal commodities should be the sum of quintozene, pentachloroaniline and methyl pentachlorophenyl sulfide, expressed as quintozene. This definition is also suitable for animal commodities for enforcement purposes since the parent quintozene is not an appropriate indicator compound for such commodities. Quintozene alone is a suitable definition of the residue for enforcement purposes for crops. The residue is fat-soluble.

The impurity hexachlorobenzene (HCB) should also taken into account for risk assessment. Before 1988, the HCB content of the technical material was approximately 0.5%. In 1988 modifications to the manufacturing process reduced the level of HCB to 0.1% or less. The ADI of 0.01 mg/kg bw was established by the 1995 JMPR for quintozene containing less than 0.1% HCB.

## **USE PATTERN**

The Meeting received new information on use patterns in Greece, Japan and Spain (Tables 22 and 23). Registered used in other countries are listed in the 1995 evaluation.

## Correction to 1995 evaluation:

In Table 9 (page 638) the rate of application for seed treatment should be shown as kg ai/100 kg seed, not ka ai/ha.

Сгор	Country	Form.	Application rate, kg ai/100 kg seed
Barley	Spain	ES	0.036-0.19
		WS	0.04-0.2
Cereals	Greece	WP	0.075-0.11
Cotton	Greece	WP	0.23
	Spain	ES	0.036-0.19
		WS	0.04-0.2
Maize	Spain	ES	0.036-0.19
		WS	0.04-0.2
Sugar beet	Japan	WP	0.38-0.75

Table 22. Registered uses of quintozene for seed treatments.

$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	Crop	Country	Form.	appl	lication			PHI,
AlfalfaGreeceWPlinear soil application surface banded pre-plant incorporation at sowing0.75-2.3 1.9-3121 YBeanGreeceWPlinear soil application surface banded pre-plant ncorporation at sowing0.75-2.3 1.9-31F.SpainWPirrigation water and/or soil incorporation pre-plant row0.1-2.1.6 0.23-0.26121 21CabbageJapanDust18-4011Chinese cabbageJapanDust18-401Chinese cabbageJapanDust40 -501Chinese cabbageJapanDust1.0-3121 21CottonGreeceWPlinear soil application sori incorporation pre-plant incorporation pre-plant row banded, surface banded, 0.1 0.23-0.26121 21CucumberGreeceGRsoil incorporation pre-plant incorporation pre-plant incorporation pre-plant rigation water and/or soil 0.38(tinear)1.1-2.31FCucumberGreeceGRsoil incorporation pre-plant incorporation at sowing1.2-1.60.12-0.361-2 2.22.1 F, CEggplantGreeceGRsoil incorporation pre-plant surface banded, uncorporation at sowing1.2-1.60.12-0.361-2 2.22.1 F, CLettuceJapanDustpre-transplanting pre-transplanting sowing sowing0.32-0.2612.1 F, CMelonsGreeceWPdrip irrigation and soil drenching1.	_			method		Spray conc.,	No.	days
					÷	.g ai/hl		F/G
Bean         Greece         WP         linear soil application         0.75-2.3         1         2           Spain         WP         infgaton water and/or soil         1.1-2.3         1         F.           GR         Soil incorporation at sowing         1.1-2.3         1         F.           Cabbage         Japan         Dust         1.2-1.6         1         2.1           Cabbage         Japan         Dust         irrigation water and/or soil         4.5         1         F.           Chinese         Japan         Dust         irrigation water and/or soil         40         -0         -0           Cotton         Greece         WP         incorporation at sowing         0.1         1         21           Cotton         Greece         WP         incorporation pre-plant         1.2-1.6         1         21           surface banded         now banded,         0.1         0.23-0.26         2.3         1         21           surface banded         now pre-plant         1.2-1.6         0.12-0.36         1-2         21           surface banded         now propation at sowing         0.23-0.26         2.3         1         F.           Cusumber         Greece         <	Alfalfa	Greece	WP				1	
	Bean	Greece	WP				1	
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	-	с ·	WD				1	
		Spain	WP		1.1-2.3		1	F, G
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	-		GR		12-16		1	21
			OK				1	
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$								-
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Chinese cabbage         Japan         Dust         40 -50         1           Cotton         Greece         WP         linear soil application surface banded pre-plant         1.9-3         1         21           GR         soil incorporation pre-plant         1.2-1.6         1         21           row banded, surface banded, through sowing         0.31         1         21           Spain         WP         incorporation at sowing         0.324.0.26         F           Cucumber         Greece         WP         drip irrigation water and/or soil         1.1-2.3         1         F           Cucumber         Greece         GR         soil incorporation at sowing         0.38(linear)         1         F, C           Lettuce         Japan         Dust         pre-transplanting pre-transplanting         5-24         1         21           Maize         Greece         GR         row banded sowing         0.12-0.36         1         21         F, C           Maize         Greece         GR         row banded sowing         0.12-0.36         1         21         F, C           Maize         Greece         GR         row banded sowing         0.12-0.36         1         21         21		Spain	WP	irrigation water and/or soil	4.5		1	F, G
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row banded 0.1 surface banded 0.23-0.26				linear application pre-plant		1.4-4.5		
surface banded 0.23-0.26			GR				1	21
1 = 1 broad application at planting $1 = 23$ (broad) $1 = 1$								
				broad application at planting				
Image: SquashGreeceWPdrip irrigation and soil drenching0.98(linear)1.2-1.60.12-0.361-221	Squash	Graace	WD			0.12.0.26	10	21
	squasn	Greece	WP		1.2-1.0	0.12-0.30	1-2	21 F, G

Table 23. Registered uses of quintozene for soil and plant treatment.

Crop	Country	Form.	applic	ation			PHI,
			method	Rate, kg ai/ha	Spray conc., .g ai/hl	No.	days F/G
Sugar beet	Greece	GR	soil incorporation pre-plant row banded surface banded through sowing equipment during sowing	1.2 -1.6 0.1 0.23-0.26 2.3 (broad) 0.98(linear)		1	21 F
	Japan	WP		0.34-0.75			
Tobacco	Greece	GR	soil incorporation pre-plant row banded surface banded through sowing equipment during sowing	2.8 0.1 0.23-0.26 2.3 (broad) 0.98(linear)		1	21 F
		WP	soil drenching (seedling) soil drenching (grown plants)	2.8-3.3 1.2-1.6	0.01-0.015 0.012-0.036	1-4 1-2	21 F
Tomatoes	Greece	GR	soil incorporation pre-plant row banded surface banded	2.8 0.1 0.23-0.26		1	21 F
		WP	drip irrigation and soil drenching (grown plants)	1.2-1.6	0.12-0.36	1-2	21 F, G
	Spain	WP	irrigation water and/or soil incorporation at sowing	2.3		1	F, G
Vegetables	Greece	WP	linear soil application pre-plant surface banded pre-plant drip irrigation and soil drenching (group plant)	0.75-2.3 1.9–3 2.8–3.3	0.01-0.015	1 1-4	21 21 E.C
			(grown plants) application before sowing or planting	(broad) (linear)	1.9-4.5 1.4-4.5	$1^2$ $1^2$	F, G
		GR	soil incorporation pre-plant row banded surface banded application pre-sowing or pre- planting	2.8 0.1 0.23-0.26 2.3 (broad) 0.98(linear)		1	21 F

<sup>1</sup> kg ai/m

<sup>2</sup> Not on cucurbits

F: field, G: glasshouse

## **RESIDUES RESULTING FROM SUPERVISED TRIALS**

The Meeting received new supervised residue trials data only on lettuce.

<u>Lettuce</u>. The 1995 JMPR recommended withdrawal of the previous recommendation of 3 mg/kg because no residue data were submitted. The present Meeting received reports of two Japanese trials (1 x 60 kg ai/ha pre-transplanting, PHI 49 or 67 days) resulting in quintozene residues of 0.007 and 0.01 mg/kg (Table 24). No metabolites or impurities were determined and it was not clear whether the varieties were head or leaf lettuce.

Two further pre-planting trials in 1970 in Germany (Anon., 1997) showed quintozene residues of 0.2 and 0.27 mg/kg, but no information on rates of treatment or the analytical methods used was provided.

Table 24. Residue of quintozene in lettuce, Japan, 1989.

Variety	Applicatio	n	PHI, days	Residues, mg/kg	Reference
	method k	g ai/ha			
Universe	pre-transplanting	60	49	0.01	Mitsui Toatsu Chem., 1990
Sacrament	pre-transplanting 60		67	0.007	Mitsui Toatsu Chem., 1990

The Meeting estimated STMRs for the sum of quintozene, PCA and PCTA and for HCB of the commodities evaluated in 1995.

## Animal feeding studies

No new work was reported, but the Meeting was informed that the studies on cows (Griffith *et al.*, 1969) and chickens (Griffith, 1969) evaluated by 1995 JMPR were with quintozene which contained 1.4% HCB.

## FATE OF RESIDUES IN STORAGE AND PROCESSING

#### In storage

No information.

## In processing

Ball (1990) determined residues of quintozene, PCA, PCTA, HCB and PB in unpeeled and peeled potatoes and the peels after one broadcast treatment at 28 kg ai/ha in Minnesota (MN) and North Dakota (ND), USA. The mean ratio of wet to dry peel weights was 7.5. The results are shown in Tables 25 and 26.

Table 25.	Weights	of potatoes	and peels	(Ball, 1990).
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Location/Sample	Whole tubers, kg	Peeled tubers, kg	Peel, % of whole
MN - untreated	26.6	23.7	10.9
MN - treated	27	24.1	10.7
ND - untreated	28.3	25.3	10.6
ND - treated	27.9	25	10.4
		Average	10.65
	Wet peel, kg	Dry peel, kg	Ratio wet/dry peel
MN - untreated	2.7	0.37	7.3
MN - treated	2.7	0.36	7.5
ND - untreated	3.2	0.42	7.6
ND - treated	2.8	0.37	7.6

Location	Application	PHI	Sample			Residues,	mg/kg	
	kg ai/ha	days		Quintozene	HCB	PCA	PB	PCTA
Hollondale, MN	28	123	whole tuber, unpeeled	0.13	0.0012	0.02	0.012	0.03
			wet peel	1.0	0.002	0.066	0.024	0.12
			dried peel	2.1	0.0028	0.27	0.15	0.20
			whole tuber, peeled	0.014	0.0007	0.0076	0.008	0.009
Northwood, ND	28	122	whole tuber, unpeeled	0.012	0.0007	0.008	0.013	0.01
			wet peel	0.067	0.0009	0.021	0.012	0.023
			dried peel	0.17	0.0015	0.091	0.031	0.051
			whole tuber, peeled	0.002	< 0.0005	0.003	0.005	0.003

Table 26. Quintozene residues in potatoes and potato peels, USA, 1988 (Ball, 1990).

Gaydosh and Smudin (1996a) treated cotton at 6.7 kg ai/ha, about 3 times the US GAP rate. No residues of quintozene, PCA, PCTA or HCB above 0.0005 mg/kg, or of PB above 0.1 mg/kg were in the seed (RAC) after treatment. No residues of quintozene or its metabolites or impurities above the LOD were detected in hulls or crude or refined oil. In soapstock no residues of quintozene, PCTA or PB were detected above 0.1 mg/kg and no residues of HCB above 0.0005 mg/kg, but PCA was detected at a mean level of 064 mg/kg. From the results it cannot be concluded whether residues of quintozene are concentrated in processed products of cotton seed.

Gaydosh and Smudin (1996b) treated peanuts at 112 kg ai/ha, 10 times the US GAP rate. The maximum residues in whole peanuts of quintozene, PCA, PCTA, PB and HCB were 6.9, 1.8, 0.56, 0.34 and 0.083 mg/kg respectively. Quintozene was concentrated by a mean factor of 2.5 in the hull but not in any other fractions. PB showed slight concentration in the hulls (1.3 times), crude oil (1.3 times) and refined oil (1.7 times). HCB was concentrated in the kernels (1.7 times), soapstock (1.2 times), crude oil (3 times), and refined oil (3.9 times). PCTA was concentrated in the kernels (1.2 times), crude oil (1.6 times) and refined oil (2.1 times), and PCA in the hulls (1.2 times) and refined oil (1.2 times). The results are shown in Table 27.

Application,	PHI,	Sample	Sample Residues, mg/kg				
kg ai/ha	days		Quintozene	HCB	PCA	PB	PCTA
112	45	whole peanuts	1.6, 6.9 UC: 0.012	0.045, 0.083	1.5, 1.8 UC:0.015	0.26, 0.34	0.4, 0.56
		kernels	1.2, 1.3	0.1, 0.12	0.87, 1.1	0.21, 0.27	0.49, 0.61
		meal	0.045, 0.019	< 0.01 (2)	<0.01, 0.01	< 0.01 (2)	< 0.01 (2)
		soapstock	0.58, 0.72	0.069, 0.077	0.99, 1.03	0.3, 0.36	0.32, 0.34
		crude oil	1.95, 1.7	0.18, 0.195	1.7, 1.8	0.39, 0.4	0.77, 0.79
		refined oil	2.6, 2.7	0.24, 0.26	1.8, 2.1	0.49, 0.51	0.98, 1.06
		hulls	12, 9.2 UC: 0.012	0.042, 0.037	2.2, 1.7	0.46, 0.32	0.43, 0.33

Table 27. Quintozene residues in peanuts and their processed fractions, Hawkinsville, GA, USA, 1992 (Gaydosh and Smudin, 1996b).

UC: residues in untreated control, validated LOD = 0.01 mg/kg for each commodity

## **RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION**

Table 28 shows the results of monitoring in The Netherlands during 1994-1996 (Olthof, 1998).

Table 28. Residues (expressed as quintozene) in food in commerce in The Netherlands 1994-1996 (Olthof, 1998).

Commodity	No. of samples	No. of samples	No of samples with	No of samples	MRL, mg/kg
	analysed	<lod< td=""><td>residues <mrl< td=""><td>with residues</td><td></td></mrl<></td></lod<>	residues <mrl< td=""><td>with residues</td><td></td></mrl<>	with residues	
		(0.01 mg/kg)		> MRL	
Oranges	982	982	-	1	0.01*
Carrots	500	494	-	6	0.01*
Melons	455	454	-	1	0.01*
Lettuce	3834	3833	-	1	0.01*
Endive	1297	1296	-	1	0.01*
Spinach	532	532	-	-	0.01*
Parsley	390	389	-	1	0.01*
Other herbs	224	223	-	1	0.01*
Beans, fresh (with pods)	690	689	-	1	0.01*
Other arable products	375	374	-	1	0.01*

\* LOD

# NATIONAL MAXIMUM RESIDUE LIMITS

New information on national MRLs in Germany (Anon., 1997), The Netherlands (Olthof, 1998) and Poland (Lzdebski, 1997) is shown below.

Country	Commodity	MRL (mg/kg)	
Germany	Bananas	1	
	Lettuce and similar, chicory	0.3	
	Tealike products	0.1	
	Oilseeds	0.03	
	Brassica vegetables	0.02	
	Other foods of plant origin	0.01	
Netherlands	Bananas	1	
	Tomatoes	0.1	
	Head lettuce	3	
	Broccoli	0.02	
	Head cabbage	0.02	
	Peanuts	2	
	Cotton seed	0.03	
	Beans (dry)	0.2	
	Potatoes	0.2	
	Other food commodities	0.01*	
Poland	Banana	1	
	Other food commodities of plant origin	0.01	

\*at LOD

## APPRAISAL

Quintozene was evaluated as a periodic review by the 1995 JMPR. That Meeting estimated a number of maximum residue levels but agreed not to recommend their use as MRLs, and recommended the withdrawal of existing MRLs, because of the lack of critical supporting data on environmental fate. Some of the processing studies could not be evaluated because of their uncertain validity.

Data on environmental fate, residues in rotational crops, supervised trials on lettuce and the effects of processing on residues in cotton seed, peanuts and potatoes have been made available to the present Meeting.

## **Environmental fate in soil**

Quintozene is unstable in soil under photolytic conditions. In sandy loam soil exposed to simulated sunlight quintozene had a half-life of 28.5 days. Pentachloroaniline (PCA) was the only significant degradation product.

Quintozene in sandy loam soil under aerobic conditions had a half-life of 189 days. The major degradation product was pentachloroaniline (PCA). A secondary product, methyl pentachlorophenyl sulfide (pentachlorothioanisole, PCTA), was formed from PCA by deamination via nucleophilic displacement at the aromatic ring. Other degradation products were methyl pentachlorophenyl sulfone (PCTASO<sub>2</sub>), methyl pentachlorophenyl sulfoxide (PCTASO) and pentachlorobenzene (PB). Soil-bound products and CO<sub>2</sub> were formed to a limited extent.

When quintozene was subjected to anaerobic conditions in the same soil after a period of 30 days under aerobic conditions, it was degraded significantly more quickly, with a half-life of 15 days. PCA was the major residue. Minor products such as PCTA and PB were also observed.

Quintozene is immobile to slightly mobile in soil (Freundlich adsorption constants in silt loam, sandy loam, clay loam and sand with about 1% organic matter were 24, 46, 89 and 31 respectively). The mobility of the major metabolite PCA was also investigated in the same soils: its Freundlich constants were even higher, indicating less mobility than the parent compound.

Field dissipation studies showed that the half-life of quintozene applied on soil surfaces was significantly shorter than when applied by incorporation.

The uptake of [*phenyl-*<sup>14</sup>C]quintozene by rotational crops was investigated. Treated soil was allowed to age for periods of 30, 120 or 365 days (DAT) before planting turnips, lettuce and wheat. The soil treatments were at a low rate of 2.2 kg ai/ha (the US GAP rate for cotton), 11 kg ai/ha (the US rate for peanuts) and 34 kg ai/ha, which was used only for rotational wheat.

After the 11 kg ai/ha treatment the total radioactive residue (TRR) expressed as quintozene in turnip tops and roots was 1.12 and 0.21 mg/kg respectively at 365 DAT, in mature lettuce 0.15, 0.10 and 0.43 mg/kg at 30, 120 and 365 DAT, in wheat forage, grain and straw 3.0, 0.79 and 11 mg/kg respectively at 30 DAT, 3.4, 0.94 and 17 mg/kg at 120 DAT, and 0.64, 0.14 and 4.6 mg/kg at 365 DAT.

Residues of the parent compound were found only in lettuce (max. 0.032 mg/kg) and wheat straw (2.2 kg ai/ha, 120 DAT, 0.012 mg/kg). PCA was found in all three crops, at the highest levels of 0.11 mg/kg in turnip roots (365 DAT, 11 kg ai/ha), 0.065 mg/kg in immature lettuce (365 DAT, 11 kg ai/ha), 0.85 mg/kg in wheat straw (30 DAT, 34 kg ai/ha) and 0.11 mg/kg in wheat forage (30 DAT, 34 kg ai/ha). PB was found only in lettuce (30 DAT, 11 kg ai/ha) at less than 0.001 mg/kg.

In another field rotational crop study unlabelled quintozene was applied to bare soil at the maximum GAP rate for peanuts (11 kg ai/ha per season) at typical times of 15 or 45 days before peanut harvest. No residues above the LOD of 0.005 mg/kg of quintozene, PB, HCB, PCTA or PCTASO were found in the leaves, roots or grain of lettuce, turnips or wheat planted 365 days after the treatment. Residues of PCA and tetrachloroaniline (TCA) were found in the turnip roots at levels

not exceeding 0.014 mg/kg, and PCA residues in two of eight mature wheat grain samples (0.007, 0.016 mg/kg).

## **Environmental fate in water**

Quintozene is stable to hydrolysis. In buffered solutions at pH 5, 7 and 9, the half-life was longer than 180 days. However, aqueous photodegradation occurred in simulated natural sunlight (with a xenon arc lamp), with a half-life of less than two days in buffered water at pH 5.

## **Definition of the residue**

The 1995 JMPR considered that for risk assessment purposes the residue in plant and animal commodities should be defined as the sum of quintozene, pentachloroaniline and methyl pentachlorophenyl sulfide, expressed as quintozene. This definition is also suitable for animal commodities for enforcement purposes since the parent quintozene is not an adequate indicator compound in such commodities. Quintozene alone is a suitable definition of the residue in crops for enforcement purposes. The residue is fat-soluble.

Residues of the impurity hexachlorobenzene (HCB) arising from the use of quintozene should also taken into account for risk assessment. Before 1988, the HCB content of the technical material was approximately 0.5%. In 1988 modifications to the manufacturing process reduced the level of HCB to 0.1% or less. An ADI of 0-0.01 mg/kg bw was established by the 1995 JMPR for quintozene containing less than 0.1% HCB.

## New supervised residue trials

<u>Lettuce</u>. The 1995 JMPR recommended withdrawal of the existing CXL of 3 mg/kg because no residue data were submitted. The current Meeting received reports of two Japanese trials (1 x 60 kg ai/ha pre-transplanting, PHI 49 or 67 days) resulting in quintozene residues of 0.007 and 0.01 mg/kg. No metabolites or impurities were determined. Two German trials could not be evaluated as the application rates were not reported. The Meeting agreed that the data were insufficient to estimate a maximum residue level and confirmed the recommendation of the 1995 JMPR to withdraw the CXL of 3 mg/kg.

## New processing studies

<u>Potatoes</u>. Potatoes treated with 1 x 28 kg ai/ha were analysed for quintozene, PCA, PCTA, HCB and PB. Analyses of whole tubers, peeled potatoes, and wet and dry peel showed that most of the residue is on the peel.

The residues of HCB were 0.0012 mg/kg in the unpeeled whole potatoes, 0.0007 mg/kg in the peeled tubers, 0.002 mg/kg in wet peel and 0.0028 mg/kg in dry peel.

<u>Peanuts</u>. After treatment at 112 kg ai/ha (10 times the US GAP rate) the maximum residues in whole peanuts of quintozene, PCA, PCTA, PB and HCB were 6.9, 1.8, 0.56, 0.34 and 0.083 mg/kg respectively. The mean quintozene residue was concentrated 2.5 times in the hulls but not in any other fractions. PB was concentrated 1.3 times in the hulls and crude oil and 1.7 times in the refined oil. HCB was concentrated in the kernels (x 1.7), soapstock (x 1.1), crude oil (x 3), and refined oil (x 3.9), and PCTA in the kernels (x 1.1), crude oil (x 1.6) and refined oil (x 2.1). PCA was concentrated slightly in the hulls (x 1.2) and refined oil (x 1.2).

<u>Cotton seed</u>. No residues of quintozene, PCA, PCTA or HCB above 0.0005 mg/kg or PB above 0.1 mg/kg were found in cotton seed after treatment at 6.7 kg ai/ha. No residues of quintozene or its metabolites or impurities were detected in crude or refined oil, or in the hulls above the LOD (0.0005 mg/kg). No residues of quintozene, PCTA or PB were detected above 0.1 mg/kg in soapstock, and no residues of HCB above 0.0005 mg/kg. Residues of PCA were detected in soapstock however (mean 0.0064 mg/kg). It cannot be concluded from the results whether quintozene or its metabolites or impurities become concentrated in the processed products of cotton seed. As quintozene is fat-soluble, it would be expected that residues in the seeds would be transferred to the oil. Since the processing procedure for the preparation of cotton seed oil is comparable to that for the preparation of peanut oil the results of the peanut processing study should be applicable to cotton seed and other oilseeds.

# Recommendations for MRLs and estimation of STMR levels for commodities for which maximum residue levels were estimated by the 1995 JMPR

The evaluation of the residue data was described in the 1995 monograph and appraisal. If there were different populations of residues, the STMRs are derived from the highest population. In some commodities (sweet peppers, soya beans, soya bean forage and fodder, sugar beet, sugar beet leaves, barley straw and fodder) the residues of quintozene, PCTA and PCA were below the LOD in all the samples. The results of a rotational crop metabolism study indicated that residues could occur in succeeding crops. The main part of the total residue consisted of quintozene (about 60%), with about 30% of PCA and 10% of PCTA. The Meeting therefore estimated the STMRs for commodities with no detectable residues of quintozene, PCTA or PCA on the basis of the LOD for quintozene only.

The present Meeting recommended the maximum residue levels estimated in 1995 for use as MRLs.

No HCB residues above the LOD were found in broccoli, head cabbages, sweet peppers, tomatoes, common beans (dry), peas (dry), soya beans (dry), sugar beet (roots and leaves), barley, maize, wheat, cotton seed, pea hay (dry), maize forage, soya bean fodder or soya bean forage, and rotational crop studies did not show residues of HCB in succeeding crops. The Meeting therefore estimated an STMR of 0 for HCB in these commodities.

Broccoli. On the basis of the 1995 evaluation, the Meeting recommended an MRL of 0.05 mg/kg.

The critical use was treatment of the soil after planting the seedlings and this resulted in total residues of 0.04, 0.046, 0.057, 0.06, 0.075 and 0.098 mg/kg. The Meeting estimated an STMR of 0.0585 mg/kg for broccoli, based on the sum of quintozene, PCTA and PCA expressed as quintozene.

<u>Head cabbages</u>. The Meeting recommended an MRL of 0.1 mg/kg, based on the residues in samples with wrapper leaves after transplant solution treatment (the soil was treated after the seedlings were planted). The STMR estimate was based on the residues of quintozene and PCA in the samples without wrapper leaves as the PCTA residues in all the samples without wrapper leaves were below the LOD (<0.002 mg/kg). The highest residue population of the samples without wrapper leaves resulted from GR broadcast treatment, with residues of 0.0042 (6), 0.0052, 0.0053, 0.0108, 0.0119, 0.0122, 0.0152 and 0.0182 mg/kg. The Meeting estimated an STMR of 0.0052 mg/kg for head cabbages, based on the sum of quintozene and PCA expressed as quintozene.

<u>Sweet peppers</u>. The Meeting recommended an MRL of  $0.05^*$  mg/kg as a practical limit of determination.

The residues of quintozene, PCTA and PCA in all the samples were below the LOD. The Meeting estimated an STMR of 0.05 mg/kg.

<u>Tomatoes</u>. The Meeting recommended an MRL of 0.02 mg/kg. The STMR was estimated on the basis of the quintozene residues (from the WP in-furrow treatment) as the PCA and PCTA residues were all below the LOD (<0.002 mg/kg). The quintozene residues in rank order were <0.002 (7), 0.003, 0.006 and 0.012 mg/kg. The Meeting estimated an STMR of 0.002 mg/kg.

<u>Common beans and common beans (dry)</u>. The Meeting recommended an MRL of 0.02 mg/kg for common bean (dry). The EC treatment (1 x 1.7 kg ai/ha, 1993) was identified as giving rise to the population with the highest residues.

The residues in fresh beans, as the sum of quintozene, PCA and PCTA, in rank order were <0.002 (2), <u>0.0327</u>, <u>0.0358</u>, 0.0795 and 0.0925 mg/kg, expressed as quintozene. The Meeting estimated an STMR of 0.0342 mg/kg for common bean (pods and/or immature seeds).

The residues in dry beans (quintozene, PCA and PCTA) were < 0.002 (4), 0.0168 and 0.0297 mg/kg, expressed as quintozene. The Meeting estimated an STMR of 0.002 mg/kg for common bean (dry).

<u>Peas (dry)</u>. The Meeting recommended an MRL of 0.01 mg/kg. The STMR was estimated on the basis of the quintozene residues as no PCA or PCTA residues exceeded the LOD (0.005 mg/kg) in any of the samples. The quintozene residues were <0.005 (6), 0.005 and 0.007 mg/kg. The Meeting estimated an STMR of 0.005 mg/kg.

<u>Soya beans (dry)</u>. The Meeting recommended an MRL of  $0.01^*$  mg/kg as a practical limit of determination. The residues of quintozene, PCTA and PCA in all the samples were below the LOD. The Meeting estimated an STMR of 0.005 mg/kg.

<u>Potatoes</u>. The 1995 Meeting could not estimate a maximum residue level and recommended withdrawal of the CXL as no information on GAP was available. The present Meeting was informed that there is only a conditional time-limited registration for the use of quintozene on potatoes in the USA. The Meeting therefore confirmed the recommendation of the 1995 Meeting.

<u>Sugar beet</u>. The Meeting recommended an MRL of  $0.01^*$  mg/kg as a practical limit of determination. The residues of quintozene, PCTA and PCA in all the samples were below the LOD. The Meeting estimated an STMR of 0.005 mg/kg.

<u>Barley, maize</u>. The Meeting recommended an MRL of 0.01\* mg/kg as a practical limit of determination for both commodities. The residues of quintozene, PCTA and PCA in all the samples were below the LOD. The Meeting estimated STMRs of 0.005 mg/kg for barley and maize.

<u>Wheat</u>. The Meeting recommended an MRL of 0.01 mg/kg. The STMR was estimated on the basis of the quintozene residues as the PCA and PCTA residues were all below the LOD (<0.005 mg/kg). The quintozene residues were <0.005 (19) and 0.0061 mg/kg. The Meeting estimated an STMR of 0.005 mg/kg.

<u>Cotton seed</u>. The Meeting recommended an MRL of 0.01 mg/kg. The STMR was estimated on the basis of the 1988 trials with EC and FL treatments (1995 monograph, Table 19), which gave residues of < 0.016 (24), 0.0188 (2), 0.0249 and 0.0284 mg/kg for the sum of quintozene, PCTA and PCA, expressed as quintozene. The Meeting estimated an STMR of 0.016 mg/kg.

<u>Peanuts</u>. The Meeting recommended an MRL of 0.5 mg/kg. The STMR was estimated on the basis of the 1994 trials (EC treatment). The residues were 0.099, 0.106, <u>0.324</u>, <u>0.382</u>, 0.436 and 0.446 mg/kg

for the sum of quintozene, PCTA and PCA, expressed as quintozene. The Meeting estimated an STMR of 0.353 mg/kg.

The processing study showed that only PCTA moved into the oily fractions, with concentration factors exceeding 1.5 (1.6 for crude and 2.1 for refined oil). Because PCTA residues are only a third of the total, and no concentration of quintozene or PCA would be expected, no STMR was estimated for residues of quintozene or its metabolites.

On the basis of HCB residues of 0.0022, 0.0025, <u>0.0064</u>, <u>0.008</u>, 0.016 and 0.017 mg/kg in the 1994 trials, the Meeting estimated an STMR of 0.0072 mg/kg for HCB in peanuts.

Multiplication of the STMR by the processing factors for HCB gave derived STMRs of 0.0216 mg/kg in crude oil and 0.0281 mg/kg in refined oil.

<u>Maize forage</u>. The Meeting recommended an MRL of  $0.01^*$  mg/kg as a practical limit of determination. The residues of quintozene, PCTA and PCA in all the samples were below the LOD. The Meeting estimated an STMR of 0.005 mg/kg.

<u>Cereal straws and fodders</u>. The Meeting recommended MRLs of  $0.01^*$  mg/kg (a practical limit of determination) for barley straw and fodder, dry, 0.01 mg/kg for maize fodder and 0.03 mg/kg for wheat straw and fodder, dry.

In barley straw, the residues of quintozene, PCTA and PCA in all the samples were below the LOD. The Meeting estimated an STMR of 0.005 mg/kg for barley straw and fodder, dry.

The STMRs for maize fodder and wheat straw were calculated on the basis of the quintozene residues as the PCA and PCTA residues were all below the LOD (<0.005 mg/kg). The quintozene residues were <0.005 (12) and 0.006 mg/kg in maize fodder and <0.005 (17), 0.006, 0.007 and 0.023 mg/kg in wheat straw. The Meeting estimated STMRs of 0.005 mg/kg for maize fodder and for wheat straw and fodder, dry.

In one of 20 wheat straw samples, the HCB residue was higher than the LOD (population: <0.005 (19) and 0.014 mg/kg). Because the supervised residue trials were carried out in 1987, the HCB content of the formulation could be as high as 0.5% and the trials were therefore not applicable to the current situation.

In view of the high persistence of HCB in the soil and the results of a rotational crop metabolism study indicating that it could be taken up from the soil by cereals, the Meeting could not estimate an STMR for HCB in cereal straws and fodders, dry.

<u>Pea hay</u>. The Meeting recommended an MRL of 0.05 mg/kg and an STMR of 0.021 mg/kg, based on residues of 0.0156 (3), <u>0.0166</u>, <u>0.0255</u>, 0.0268, 0.0305 and 0.0415 mg/kg, for the sum of quintozene, PCTA and PCA expressed as quintozene, in pea hay (dry).

<u>Soya bean fodder and forage</u>. The Meeting recommended an MRL of 0.01\* mg/kg as a practical limit of determination for both commodities. In whole green plants, the residues of quintozene, PCTA and PCA in all the samples were below the LOD. The Meeting estimated an STMR of 0.005 mg/kg for soya bean forage.

No residues of quintozene, PCTA or PCA were found above the LOD in the hay or the whole plant at harvest except one PCA residue of 0.0154 mg/kg (expressed as quintozene). The residues in rank order were < 0.0055 (29) and 0.0154 mg/kg. The Meeting estimated an STMR of 0.0055 mg/kg for soya bean fodder, based on the residues of PCA expressed as quintozene.

<u>Sugar beet leaves</u>. No maximum residue level was estimated for sugar beet leaves in 1995 but they could be used as a feed item. The residues of quintozene, PCTA and PCA in all the samples were below the LOD. The Meeting estimated an STMR of 0.005 mg/kg for sugar beet leaves.

## **Animal products**

<u>Chickens</u>. The Meeting recommended MRLs of  $0.03^*$  mg/kg for eggs and  $0.1^*$  mg/kg for chicken meat (in the fat) and edible offal as practical limits of determination.

The residues of quintozene, PCTA and PCA in all the samples from feeding levels up to the equivalent of 5 ppm in the diet were below the LODs.

Since (1) the main part of the total residue in plants is quintozene, (2) the residues in feed items for poultry will be below 1 mg/kg, (3) no quintozene occurred in meat, liver or eggs at 1 ppm in the diet or in fat at 15 ppm, and (4) the residue is fat-soluble, the Meeting estimated STMRs of 0.01 mg/kg for eggs, 0.04 mg/kg for chicken meat (in the fat) and 0.03 mg/kg for edible offal, based on the LODs for quintozene.

No STMR was estimated for HCB as the feeding study was carried out with quintozene which contained 1.4% HCB.

## RECOMMENDATIONS

On the basis of data from supervised trials the Meeting estimated the maximum residue levels and STMRs listed below. The estimates of maximum residue levels confirm those of the 1995 JMPR, which were not recommended as MRLs at that time owing to the lack of critical supporting studies. The maximum residue levels are now recommended for use as MRLs.

Definition of the residue

- (1) For compliance with MRLs for plant commodities: quintozene.
- (2) For compliance with MRLs for animal commodities: sum of quintozene, pentachloroaniline and methyl pentachlorophenyl sulfide, expressed as quintozene.
- (3) For estimation of dietary intake for plant and animal commodities: sum of quintozene, pentachloroaniline and methyl pentachlorophenyl sulfide, expressed as quintozene.

The compounds are fat-soluble.

Commodity		Recommendation			
-		MRL mg/kg		STMR, STMR-P mg/kg	
CCN	Name	New	Previous <sup>1</sup>	quintozene <sup>1</sup>	HCB
GC 0640	Barley	0.01*	-	0.005	0
AS 0640	Barley straw and fodder, dry	0.01*	-	0.005	
VB 0400	Broccoli	0.05	0.02	0.0585	0
VB 0041	Cabbages, Head	0.1	0.02	0.0052	0
PO 0840	Chicken, Edible offal of	0.1*	-	0.03	
PM 0840	Chicken meat	0.1* (in the fat)	-	0.04 (in the fat)	
VD 0526	Common bean (dry)	0.02	0.2	0.002	0
VP 0526	Common bean (pods and/or immature seeds)	0.1	0.01	0.0342	0
SO 0691	Cotton seed	0.01	0.03	0.016	0
PE 0112	Eggs	0.03*	-	0.01	
VL 0482	Lettuce, Head	$W^2$	3		
GC 0645	Maize	0.01*	-	0.005	0
AS 0645	Maize fodder	0.01	-	0.005	
AF 0645	Maize forage	0.01*	-	0.005	0
SO 0697	Peanut	0.5	2	0.353	0.0072
OC 0697	Peanut oil, crude				0.0216
OR 0697	Peanut oil, edible				0.0281
VD 0072	Peas (dry)	0.01		0.005	0
AL 0072	Pea hay or Pea fodder (dry)	0.05		0.021	0
VO 0445	Peppers, Sweet	0.05*	0.01	0.05	0
VR 0589	Potato	$W^2$	0.2		
VD 0541	Soya bean (dry)	0.01*		0.005	0
AL 0541	Soya bean fodder	0.01*		0.0055	0
AL 1265	Soya bean forage (green)	0.01*		0.005	0
VR 0596	Sugar beet	0.01*		0.005	0
AV 0596	Sugar beet leaves or tops			0.005	0
VO 0448	Tomato	0.02	0.1	0.002	0
GC 0654	Wheat	0.01		0.005	0
AS 0654	Wheat straw and fodder, dry	0.03		0.005	

<sup>1</sup>Withdrawal of all previous MRLs (now CXLs) was recommended by the 1995 JMPR owing to lack of supporting studies. The 29th (1997) CCPR agreed to retain them for 4 years according to the periodic review procedure

<sup>2</sup>This confirms the recommendation of the 1995 Meeting to withdraw the CXL.

# FURTHER WORK OR INFORMATION

Desirable

Animal transfer studies on ruminants.

## DIETARY RISK ASSESSMENT

The International Estimated Daily Intakes of quintozene for the five GEMS/Food regional diets were in the range of 0 to 1% of the ADI. The Meeting concluded that the intake of residues of quintozene resulting from its uses that have been considered by the JMPR is unlikely to present a public health concern.

The IEDIs of hexachlorobenzene arising from the use of quintozene were estimated on the basis of the STMRs for peanuts and refined peanut oil for comparison with a Tolerable Daily Intake (TDI) of 0.00016 mg/kg bw established by WHO<sup>1</sup>.

The IEDIs for hexachlorobenzene arising from the use of quintozene were in the range 0 to 1% of the TDI. The Meeting concluded that the intake of residues of hexachlorobenzene resulting from uses of quintozene that have been considered by the JMPR is unlikely to present a public health concern.

<sup>1</sup>"Hexachlorobenzene - Environmental Health Criteria 195", WHO, Geneva (1997)

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