TERBUFOS (167)

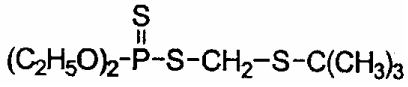
First draft prepared by Dr Salwa Dogheim, Central Laboratory of Residue Analysis of Pesticides and Heavy Metals in Food, Agriculture Research Centre, Ministry of Agriculture, Cairo, Egypt.

EXPLANATION

Terbufos was evaluated for the first time by JMPR in 1989. A further residue review was undertaken in 1990. At the 36th Session of the CCPR, the compound was scheduled for residues periodic review in 2005. The toxicological review was conducted in 2003, which established an ADI of 0.0006 mg/kg bw/day and an acute RfD of 0.002 mg/kg bw/day.

The manufacturer provided information on the latest GAP, residue data on a number of crops: including banana, coffee beans, sugar beets, maize, sorghum, and sweet corn. Metabolism, analytical methods, and relevant storage stability studies were also provided. Australia submitted information on the use pattern, national MRLs and residue definition.

IDENTITY

ISO common name:	Terbufos
IUPAC Name	*S-tert*-butylthiomethyl *0,0*-diethyl phosphorodithioate
CAS No.	13071-79-9
Synonyms and trade names	Counter, CL 92100, AC 92100, Hunter
Structural formula	
Molecular Formula	$C_9H_{21}O_2PS_3$
Molecular Weight	288.43 g/mole

Physical and Chemical Properties

The physical and chemical properties of terbufos are summarized in Table 1.

Table 1. Physical and chemical properties of terbufos

Property	Characteristics	Test Substance	Reference
Physical state	Liquid at ambient temperature	TGAI	TE-301-007
Colour	Colourless to pale yellow	TGAI	TE-301-007
Odour	Mercaptan-like	TGAI	TE-301-007
Purity	86% - 89%	TGAI	TE-301-007
Melting point	Product is liquid at room temperature	TGAI	TE-301-007
Boiling Point	55 ^oC at 0.02 mm Hg	TGAI	TE-301-007
Relative density	1.11 g/mL at 20 ^oC	TGAI	TE-301-007
pH	4.12 average in H_2O/dioxane mixture	TGAI	TE-301-007
Storage stability	Stable for more than two years at room temperature. Decomposes upon prolonged heating at temperatures above 120°C. Subject to alkaline hydrolysis in presence of strong bases.	TGAI	TE-301-007
Solubility in organic solvents, g/100 g at 20^oC	Solubility was ≥100 g/100mL solvent for each of the following solvents at 20 ºC: Acetone, acetonitrile, benzene, chloroform, dichloroethane, ethanol, n-heptane, dichloromethane, and toluene	TGAI	TE-301-007

Property	Characteristics	Test Substance	Reference
Solubility in water	5.4 mg/L water at 25°C; pH 4, 7, 10 buffers: 5.6, 4.9, 4.5 mg/L, respectively, at 25°C	PAI	TE-301-007
	Solubility of two important metabolites: terbufos sulfoxide, 2936 mg/L; terbufos sulfone, 240 mg/L (each in water at 27°C)	PAI	TE-311-003
Vapour pressure	3.16×10^{-4} mm Hg at 25°C 6.98×10^{-4} mm Hg at 35°C 12.4×10^{-4} mm Hg at 45°C	PAI	TE-301-007
Dissociation constant	Not applicable; compound does not dissociate	---	---
Octanol/water partition coefficient	Log Kow = 4.71	PAI	TE-301-007
Hydrolysis	At pH 5 and 20-25°C, half-life 4.5 days At pH 7 and 25°C, half-life 5.5 days At pH 9 and 25°C, half-life 8.5 days At the conclusion of a four-week study, 75.1, 72.4, and 68.3% of the radioactivity at pH 5, 7, and 9, respectively, was hydrophilic, with formaldehyde constituting the principal degradation product. Organophilic products consisted of the phosphorylated series of oxidative metabolites.	PAI QUES	TE-630-001
Photolysis	Less than 1% of the applied dose (4 ppm) of terbufos remained after 1-day exposure to natural sunlight in pond water, with the sulfoxide CL 94301 accounting for 45.2%. Formaldehyde appeared to be the principal water-soluble reaction product. Organophilic radioactivity was due mainly to the phosphorylated oxidative metabolites and a minor amount of the methylated mercaptan series.	PAI	TE-630-001

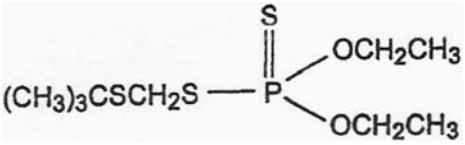
TGAI= Technical grade active ingredient.
PAI = Pure active ingredient.

Formulations

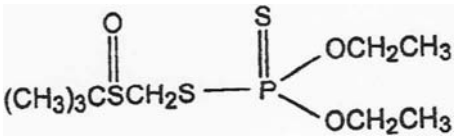
Terbufos is available in granular formulations with active ingredient content of 5%, 10%, 15%, or 20%.

METABOLISM AND ENVIRONMENTAL FATE

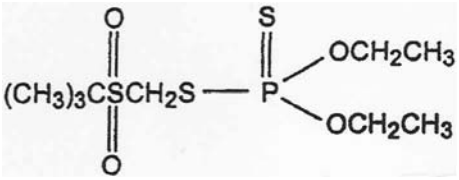
The metabolism of terbufos was investigated in animals (goat and poultry) and plants (soybean, sugar beets, sweet corn, cabbage and rape) using [methylene-^{14}C]terbufos. In some studies, [^{13}C]-terbufos labelled at the same methylene carbon was used as mass marker to facilitate the identification of metabolites by mass spectrometry. The list of metabolites and major breakdown products of terbufos, together with the code names and common names and chemical structures are presented in Figure 1. The chemical structure of terbufos, showing the positions of the carbon-13 and carbon-14 label is shown in Figure 2.



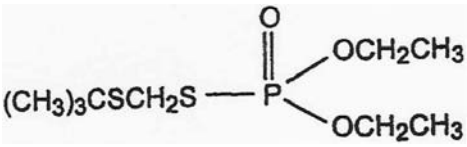
Terbufos (CL 92,100)
Phosphorodithioic Acid, *S*-(*tert*-butylthio) methyl *O*,*O*-diethyl ester



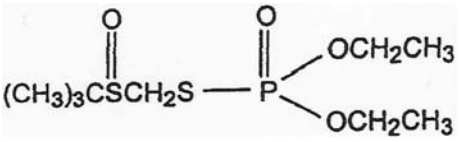
Terbufos sulfoxide (CL 94,301)
Phosphorodithioic acid. *S*-(*tert*-butylsulfinyl) methyl *O,O*-diethyl ester



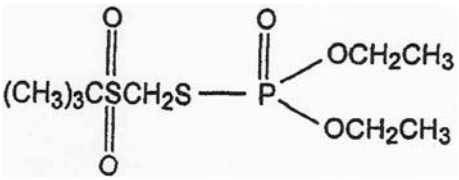
Terbufos sulfone (CL 94,320)
Phosphorodithioic acid, *S*-(*tert* -butylsulfonyl) methyl *O,O*-diethyl ester



Terbufoxon (CL 94,221)
Phosphorothioic acid, *S*-(*tert*-butylthio) methyl, *O,O* -diethyl ester



Terbufoxon sulfoxide (CL 94,365)
Phosphorothioic acid, .*S*-(*tert* -butylsulfinyl) methyl,*O,O* -diethyl ester



Terbufoxon sulfone (CL 94,302)
Phosphorothioic acid, *S-(tert*-butylsulfonyl) methyl, *O,O*-diethyl ester

Figure 1. Terbufos and its metabolites.

Figure 2. Chemical structure of terbufos showing positions of 13C and 14C-labels.

Animal Metabolism

Data was submitted to the meeting by the manufacturer on a study conducted to determine the absorption, distribution, metabolism and elimination of terbufos in rats, study no. (TE-440-004) by Cheng, T. (1992).

Metabolism in goat

In goat metabolism studies (TE 440-002 and TE-440-005) by Zulalian J. (1990) and Zulalian J. (1992) respectively, [^{14}C]terbufos was administered via capsule to two lactating goats. Each goat was dosed, once daily for seven consecutive days at dosages equivalent to 0.281 and 2.53 mg/kg in the diet calculated on the basis of dose administered and actual feed consumption. A third goat served as a control. The three experimental goats were females with average weight (45-65 kg) and aged over two years.

The total recoveries of radioactivity in the urine, faeces, cage wipes, cage washes, milk and tissues from the low and high dosed animals were 98.9 and 98.0%, respectively. The major route of excretion was via the urine, which accounted for 96.0 and 86.9% of the administered radioactivity, respectively.

The total radioactive residue (TRR) in daily milk samples was < 0.01 mg/kg (low dose) and 0.02-0.03 mg/kg (high dose, day 7). Residues in the liver, kidney, muscle, fat and blood of the low dose animal were all < 0.01 mg/kg. In the high dose animal, residues were 0.08, 0.04, < 0.01, < 0.01 and 0.03 mg/kg, respectively. The radioassay was validated at a detection limit equal to 0.01 mg/kg equivalents of [^{14}C]terbufos. The percentage of administered radioactivity and TRR in goat milk, blood and tissues are summarized in Tables 2 and 3.

Table 2. Radioactivity in milk of goats dosed with [^{14}C]terbufos for 7 consecutive days.

Collection Time	Group A (Control)	Group B (0.28 mg/kg in diet)	Group C (2.53 mg/kg in diet)
Percent Total Radioactivity			
Pre-dose	NA	NA	NA
Day 1	NA	ND	0.12
Day 2	NA	0.06	0.12
Day 3	NA	0.08	0.13
Day 4	NA	0.08	0.12
Day 5	NA	0.09	0.11
Day 6	NA	0.09	0.13
Day 7	NA	0.09	0.14
Total	NA	0.49	0.87
µg Equivalents ^{14}C-labeled CL 92100/g			
Pre-dose	NA	NA	NA
Day 1	NA	ND	0.02
Day 2	NA	< 0.01	0.02
Day 3	NA	< 0.01	0.02
Day 4	NA	< 0.01	0.02
Day 5	NA	< 0.01	0.02
Day 6	NA	< 0.01	0.02
Day 7	NA	< 0.01	0.03

NA = not applicable, ND = not detectable (< 0.01 mg/kg)

Table 3. Radioactivity in blood and tissues of goats dosed with [^{14}C]terbufos for 7 consecutive days.

Tissue	Group A (Control)	Group B (0.28 mg/kg in diet)	Group C (2.53 mg/kg in diet)
Percent of Radioactivity			
Blood	NA	< 0.01	< 0.01
Fat (omental)	NA	0.02	0.02
Kidneys	NA	0.02	0.03
Liver	NA	0.20	0.35
Muscle (leg and loin)	NA	0.08	0.02
Total	NA	0.32	0.42
µg Equivalents ^{14}C-Labeled CL 92100/g			
Blood	NA	< 0.01	0.03
Fat (omental)	NA	< 0.01	< 0.01
Kidneys	NA	< 0.01	0.04
Liver	NA	< 0.01	0.08
Muscle (leg and loin)	NA	< 0.01	< 0.01

Of the TRR in milk (high dose), 32.7% (< 0.01 mg/kg) was organosoluble, 34.8% (0.01 mg/kg) was water soluble and 29.3% (< 0.01 mg/kg) remained in the post-extracted solid (PES). HPLC and two-dimensional TLC analysis of the organosoluble milk residue showed two unknowns each in concentrations of < 0.01 mg/kg and accounting for 25.5% and 3.6% of the TRR. Three metabolites of the non-phosphorylated series, CL 202474, CL 99843, and CL 99875, were detected by TLC. The concentration of each of these components was < 0.01 mg/kg. Significantly, terbufos or its phosphorylated metabolites were not detected in milk.

Of the TRR in liver (high dose), 13.9% (0.01 mg/kg) was organosoluble, 27.5% (0.02 mg/kg) was water soluble, and 55.6% (0.04 mg/kg) remained in the PES. HPLC and TLC analyses of the organosoluble liver residue showed several unknowns accounting for 1 to 5% of the TRR (all < 0.01 mg/kg) and trace amounts of terbufos (2.1%, < 0.01 mg/kg) and the non-phosphorylated metabolite CL 99875 (1.9%, < 0.01 mg/kg). HPLC analysis of the water soluble residue showed several components each accounting for < 0.01 mg/kg of the TRR. Characterization of the PES liver residue (0.04 mg/kg) by enzyme hydrolysis with protease released 5.9% (0.01 mg/kg) of the radioactivity as organosoluble and 27.6% (< 0.01 mg/kg) as water soluble. Chemical (base) hydrolysis (6N NaOH) released 5.7% (< 0.01 mg/kg) as organosoluble, 67.9% (0.03 mg/kg) as water soluble and 5.1% (< 0.01 mg/kg) remained in the PES.

Of the TRR in kidney (high dose), 11.9% (< 0.01 mg/kg) was organosoluble, 46.5% (0.02 mg/kg) was water soluble, and 32.3% (0.01 mg/kg) remained in the PES. HPLC analysis of the water soluble radioactivity showed multiple components, each accounting for < 0.01 ppm. Terbufos was also observed (< 0.01 mg/kg). The concentration of metabolites found in goat tissues, milk and urine are summarized in Table 4.

Table 4. Metabolites detected in tissues, milk, and urine from goats treated with [^{14}C]terbufos.

Compound	Kidney[a]	Liver[b]	Milk[a]	Urine
Percent of TRR (mg/kg) HPLC Method I				
Unknown A[c]	30.3 (0.01)	16.9 (0.01)	24.5 (< 0.01)	79.1 (1.21)
CL 99843	ND	ND	2.9 (< 0.01)	11.3 (0.17)
CL 99875	ND	1.90 (< 0.01)	ND	ND
Terbufos (CL 92100)	9.50 (< 0.01)	2.10 (< 0.01)	ND	ND
Percent of TRR (mg/kg) HPLC Method II				
Unknown B	3.1 (< 0.01)	ND	ND	17.48 (0.22)
Unknown C	18.7 (< 0.01)	2.36 (< 0.01)	25.5 (< 0.01)	58.4 (0.75)
Unknown D	ND	17.3 (0.014)[d]	ND	14 (0.20)
Unknown E	ND	5.39 (< 0.01)	3.6 (< 0.01)	4.66 (0.07)
Unknown F	ND	0.88	ND	

Data are from a goat treated with [^{14}C]terbufos at 3.41 mg/day (2.53 ppm in the diet Group C) for 7 consecutive Days

[a] From organic extract.

[b] From aqueous extracts.

[c] Unknown A contained several components based on HPLC Method II.

[d] More than one peak in this area.

The metabolism study indicated that when terbufos is administered to lactating goats, it is absorbed, detoxified and eliminated (see 2003 JMPR Toxicological Evaluation on rat metabolism) predominantly in the urine. Neither terbufos nor any of the phosphorylated oxidative metabolites were observed in milk. None of the phosphorylated oxidative metabolites were detected in tissues, however, terbufos was observed at low concentrations in liver (< 0.01 mg/kg) and in kidney (< 0.01 mg/kg).

Small amounts (< 0.01 mg/kg) of unchanged terbufos, and the non-phosphorylated metabolites (CL 202474, CL 99843, CL 99875), were detected. The presence of terbufos, and possibly free and/or protein-bound non-phosphorylated metabolites (CL 99843, CL 99875, CL 202474), indicated terbufos was extensively metabolized. Hydrolysis of the thiolophosphorus bond (P-S) followed by enzymatic S-methylation, desulfuration, and sulfoxidation occurred in the metabolism of terbufos in goats. The proposed metabolic pathway of terbufos in goats is presented in Figure 3.

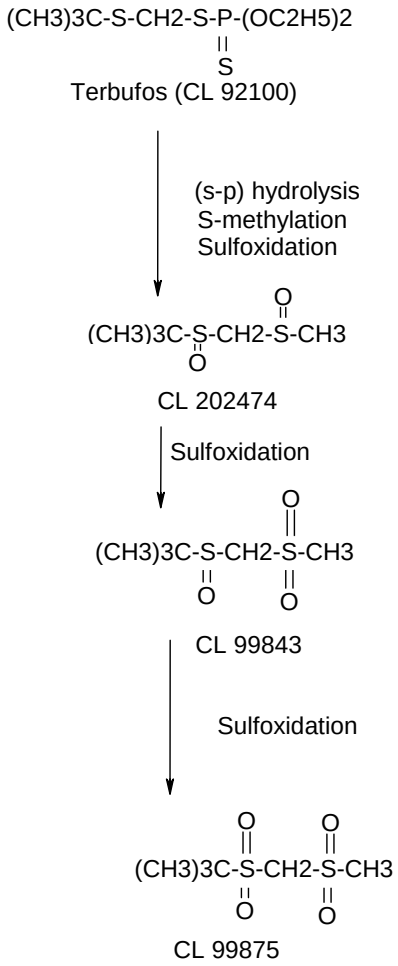


Figure 3. Proposed metabolic pathway of terbufos in lactating goat.

Metabolism in hens

A hen metabolism study (TE-440-003) was conducted by Brindle, P. (1990) to determine the residue levels as well as the nature of terbufos (CL 92100)-derived residues in the eggs, blood and edible tissues of laying hens following administration of highly exaggerated levels of [^{14}C]terbufos.

Five groups of DeKalb XL White Leghorn hens, each hen about 46 weeks old and weighing approximately 1.5 kg, were used as test animals. Three groups of five hens, A, B and C, were dosed via capsule for five consecutive days with the feed equivalent of 0 mg/kg (control), 0.35 mg/kg, and 1.05 mg/kg [^{14}C]terbufos, respectively. Two groups of ten hens, D and E, both dosed at 1.05 mg/kg with high specific activity of [^{14}C]terbufos were included in the study for use in metabolite identification and residue method validation work, in the event that the total residues exceeded the validated detection limit of the radioassay. The specific activity of the test substance used for the preparation of capsules for Groups B and C was 0.9 μCi/mg, and 30.2 μCi/mg (containing the ^{13}C-mass marker) for Groups D and E, respectively.

Recovery of [^{14}C]residues in excreta over the 5-day treatment period averaged 91.4% of the total administered dose for Group B, and 88.9% for Group C, respectively. For both dose levels, residues in eggs (days 1 through 5, both white and yolk), blood, skin with adhering fat, muscle, liver or kidney tissues were all less than the validated detection limit of the radioassay (< 0.05 mg/kg). Since the number of eggs collected from Groups B and C on the last day of the study were limited, the eggs produced on days 4 and 5 of treatment from Groups D and E were analyzed and the results provided additional evidence that total residues were < 0.05 mg/kg in both egg white and yolk.

Plant Metabolism

The metabolic fate of [^{14}C]terbufos in plants was studied in soybeans, sugar beet, sweet corn, cabbage and rape.

Metabolism in soybeans

In the soybean metabolism study (TE-640-001), by Chiu, T. (1981), plants were grown under field conditions from seed treated in the furrow at a rate of 1.1 kg ai/ha with [methylene-^{14}C]terbufos (specific activity of 18.4 μCi/mg, radiopurity of 98.6%). The [^{14}C]terbufos was diluted two-fold with non-radioactive standard compound (99% purity) resulting in a specific activity of 9.2 μCi/mg. This material was then formulated as 15G by dissolving 103 mg of the compound in 1 mL of dichloromethane and mixing with 585 mg of the formulation reagent consisting of 27.5 mg of Deactivator A mixed with 557.5 mg of Creek-O-Nite granules.

A 1.22 x 1.22 metres plot fenced with the chicken wire and set up with two furrows, each 1.22 metres in length and spaced 0.76 metres apart, was used for planting soybeans. The furrows were about 1.3 to 2.6 cm. in depth and were evenly treated with the formulated material. The seeds were planted about 1 inch apart in the furrow and covered with the top soil. The soybean plants of Adelphi variety were grown in Princeton sandy loam soil under the natural field conditions at the Agricultural Centre in Princeton, New Jersey.

The total radioactive residues (TRR) found in the plant, expressed as mg/kg equivalents of terbufos, were 13.3 mg/kg and 1.5 mg/kg in plants at one and two months after the treatment, respectively. At harvest, residue levels were 1.8 mg/kg in fodder, 1.6 mg/kg in hulls and 1.3 mg/kg in seeds (Table 5).

Table 5. TRR in soybeans grown in soil treated with [^{14}C]terbufos at 1.1 kg ai/ha.

Sample	Post treatment Interval (Months)	Residue (mg/kg)[a]
Control Plant	1	< 0.01
Treated Plant		13.3
Control Plant	2	< 0.01
Treated Plant		1.5

Sample	Post treatment Interval (Months)	Residue (mg/kg)[a]
Control Fodder	4[b]	< 0.01
Treated Fodder		1.8
Control Hull		< 0.01
Treated Hull		1.6
Control Seeds		< 0.01
Treated Seeds		1.3

[a] Expressed as terbufos equivalents.
[b]Harvest (normal maturity).

In the early stages of growth, 75% of the radioactivity was extractable from the plants. At the one-month sampling, 43% of the total extractable residue was identified as the phosphorylated metabolites CL 94,301 (sulfoxide), CL 94,302 (oxygen analog sulfone), CL 94,320 (sulfone) and CL 94,365 (oxygen analog sulfoxide).

Accounting for 11% of the residue were the non-phosphorylated metabolites CL 99,875 and CL 9,843. The remaining residue was comprised of five unknown metabolites (4%) and origin-bound compounds (17%). At harvest the only identifiable metabolite was CL 99,875, which was found in all three commodities, hulls (5%), fodder (2%) and seed (7%). The remaining residue was either shown to be very polar extractable materials or proven to have the carbon-14 incorporated into the cellulose and lignin of the hulls and fodder, and into the protein and oil of the seed.

The extractable radioactive residues from soybean fodder, hull and seed treated with [^{14}C]terbufos at 1.1 kg ai/ha and collected at 4-months post-treatment are summarized in Table 6 and Table 7. At harvest, the only identifiable metabolite in soybean seeds was CL 99,875 (Table 8).

Table 6. Extractability of radioactivity from soybean fodder and hull treated with [^{14}C]terbufos.

Fractions From Sequential Extraction	Percent Radioactivity	
	Fodder	Hull
1. Methanol	21.0	25.9
2. 2% Conc. HCl in 80% Methanol:H2O	13.3	32.5
3. 10% NaOH Reflux	37.7	30.9
a. Acid-Soluble at pH 1.0 [(a)]	(22.9)	(24.6)
b. Crude Lignin [(b)]	(7.8)	(6.3)
4. Crude Cellulose [(c)]	28.0	10.7
Total	100.0	100.0

[a] The filtrates of PES refluxed with 10% NaOH for 24 hours, followed by adjusting the filtrates to pH 1.0.
[b] The pellets were centrifuged after adjusting the 10% NaOH-soluble fractions of PES to pH 1.0.
[c] The filtered cake obtained from the 10% NaOH refluxed mixture, followed by washing with l0% NaOH solution and water.

Table 7. Extractability of radioactivity from soybean seed treated with [^{14}C]terbufos.

Fractions of Isolation	Percent Distribution in Various Fractions
1. Methanol: Dichloromethane (10:90; 300 mL x 2)	35.1
2. Methanol: Acetone (1:1; 300 mL x 2)	5.2
3. Concentrated HCl: Methanol (2:98; 300 mL x 2)	20.4
4. Soybean Seed PES [(a)]	39.3
Total	100.0

[a] The filtered cake obtained from the extraction with 2% concentrated HC1 in methanol.

Table 8. Metabolites identified in soybean seed treated with [^{14}C]terbufos.

Components	Fraction Analyzed[a,b]	
	Acetonitrile (0.17 mg/kg)	Hexane (0.36 mg/kg)
Oil	2.5 (0.03)	8.8 (0.12)
CL 99,875	4.2 (0.05)	2.7 (0.4)
Origin-Bound	6.1 (0.08)	12.5 (0.16)
Other Unknown	0.3 (<0.01)	3.2 (0.04)

[a] The results of TLC analysis of the combined extracts of solvent mixtures 1 and 2 (40.3%, Table 9), after partition into acetonitrile (13.1%) and hexane fractions (27.2%) accounting for 40.3% TRR (0.53 mg/kg) in seed.
[b] The total extractable residue expressed as [^{14}C]terbufos was 40% (0.53 mg/kg) of total residue of 1.32 mg/kg in soybean seeds. It was partitioned between acetonitrile and hexane. The calculated residue levels in mg/kg are shown in parenthesis.

Metabolism in sugar beet

In a sugar beet metabolism studies (TE-640-003 and TE-640-010) by, Caballa, S. (1973 and 1974), plants were grown from seed in soil treated with [^{14}C]terbufos at a rate of 6.8 kg ai/ha. The levels of radioactivity in both foliage and roots were determined at 4.5, 8, l6, and 32 weeks after treatment and were found to decline rapidly with time.

The total radioactive residues (TRR) found in the various samples declined with time from 6.27 mg/kg to 1.07 mg/kg in foliage and from 7.44 mg/kg to 0.284 mg/kg in roots (Table 9). This decline was primarily due to dilution from plant growth. The increase in the absolute amount of radioactivity recovered in the 32-week roots was due to the fact that the roots were mature and very big at this stage. The radioactivity recovered in all the plants represented a total of only 2.3% of the applied dose.

Table 9. TRR in sugar beets grown in soil treated with [^{14}C]terbufos at 6.8 kg ai/ha.

Sugar beet matrix	Time Post-Treatment (Weeks)	TRR (mg/kg)
Foliage	4.5	6.27
	8	3.38
	16	2.22
	32	1.07
Roots	4.5	7.44
	8	1.92
	16	0.295
	32	0.284

Extraction and partitioning data showed that metabolism of CL 92100 occurred at a faster rate in the roots. Chromatographic data obtained at different stages of plant growth indicated that terbufos is degraded mainly by way of oxidation, hydrolysis and methylation followed by subsequent oxidation to yield principally non-toxic metabolite CL 99875.

The concentration of various metabolites found is summarized in Table 10. In terms of mg/kg the values for the phosphorylated metabolites at 4.5 weeks were 4.2 mg/kg in the foliage and 1.2 mg/kg in the roots. At the end of 32 weeks, the major metabolite found was CL 99875, a non-phosphate compound. It is believed to be formed from the hydrolysis, subsequent methylation, and further oxidation of the analogs of terbufos. This compound accounted for nearly 95% (0.596 mg/kg) of the organosoluble radioactivity in foliage.

In the roots, the low levels of organosoluble radioactivity (0.007 mg/kg) were predominantly due to metabolite CL 99875 (0.0044 mg/kg). The decline in the amount of metabolites belonging to the phosphorylated series was accompanied by an increase of the metabolites in the methylated mercaptan series, mainly metabolites CL 99843 and 99875. These compounds have the same basic structure but different oxidation states, and are of very low toxicity. The LD_{50} in the mouse, for example, of metabolite CL 99875 is 4660 mg/kg compared with 3.5 mg/kg, for technical CL 92100. At 32 weeks, the concentration of phosphorylated compounds in foliage and roots was 0.004 mg/kg and 0.001 mg/kg, respectively.

There is evidence of incorporation of terbufos-derived radioactivity into the sucrose fraction of sugar beets. In the 16-week roots, 4.8% (0.014 mg/kg) of the total radioactivity was found in the purified sucrose fraction. Most of the aqueous-soluble activity (0.233 mg/kg) is believed to be due to soluble and insoluble natural products together with 13.2% (0.038 mg/kg) of unidentified water-soluble metabolites.

Table 10. Metabolites identified in sugar beets treated with [^{14}C]terbufos at 6.8 kg ai/ha.

Metabolite	Foliage (mg/kg)				Roots (mg/kg)			
	Time Interval (weeks)				Time Interval (Weeks)			
	4.5	8	16	32	4.5	8	16	32
CL 202474	0.0187	0.0189	0.011	< 0.001	0.0169	0.0014	< 0.001	< 0.001
CL 99844	0.0174	0.0194	< 0.001	< 0.001	0.0093	0.0015	< 0.001	< 0.001
CL 99843	0.0497	0.1714	0.0650	0.0136	0.0260	0.0093	< 0.001	< 0.001
CL 99875	0.0240	0.3754	0.7024	0.5962	0.0080	0.0256	0.0047	0.0044
CL 94365	0.2742	0.0930	< 0.001	< 0.001	0.0404	< 0.001	< 0.001	< 0.001
CL 94302	0.1129	0.0215	0.0014	0.0023	0.01490	0.0089	< 0.001	< 0.001
CL 94301	2.6014	0.0279	< 0.001	0.0014	0.6666	0.0162	< 0.001	0.0013
CL 94221	< 0.001	< 0.001	< 0.001	< 0.001	0.0180	< 0.001	< 0.001	< 0.001
CL 94320	1.1957	0.0343	< 0.001	< 0.001	0.3236	0.0339	< 0.001	< 0.001
CL 92100	0.0083	0.009	< 0.001	< 0.001	0.0081	< 0.001	< 0.001	< 0.001

Metabolism in sweet corn

In sweet corn metabolism studies (TE-640-005 and TE-640-006) by North H. *et al.* (1972) and Barringer, D. F. (1973), corn was grown in a Wisconsin greenhouse, in soil contained in metal cylinders and treated with [thiomethylene-^{14}C]terbufos at 1.1 kg ai/ha. Two high-dose tests were also conducted at 6.7 kg ai/ha, one in metal cylinders and the other one in plastic pots, to facilitate isolation and identification of metabolites. Plants from the low-dose tests were harvested at post-treatment intervals of 2, 4, 7 and 10 weeks by cutting the stem at the soil level. Sweet corn grown in soil treated with [^{14}C]terbufos at 1.1 kg ai/ha contained 0.34, 2.64, 4.70 and 6.85% of the applied dose at 2, 4, 7 and 10 weeks of growth, respectively, as shown in Table 11 .

Table 11. Recovery of ^{14}C-activity from sweet corn plants treated with [^{14}C]terbufos at 1.1 kg ai/ha.

Sampling Interval (week)	% Dose Recovered		Total % of Dose Accounted For
	Corn Plants	Soil	
2	0.336	-	-
4	2.64	50.4	53
7	4.70	41.5	46
10	6.85	33.2	40

Sweet corn was grown in a Wisconsin greenhouse soil treated with [thiomethylene-^{14}C]terbufos at 1.1 kg ai/ha.

Radioactivity extracted from plants separated into at least 19 radioactive metabolites on TLC. The metabolites found in sweet corn plants are shown in Table 12. The expected oxidation products of terbufos, i.e., the sulfoxide (CL 94,301), the sulfone (CL 94,320), the oxygen analog sulfoxide (CL 94,365) and sulfone (CL 94,302), were confirmed by two-dimensional co-TLC in the corn plant. In the 10-week corn plant, these phosphorylated metabolites accounted for 34 percent of the chloroform-soluble extractable radioactivity.

Table 12. Metabolites found in sweet corn plants grown in soil treated with [^{14}C]terbufos at 1.1 kg ai/ha.

Compound	Residues [%] of chloroform-soluble radioactivity			
	2 Weeks 0.88 mg/kg	4 Weeks 2.70 mg/kg	7 Weeks 2.87 mg/kg	10 Weeks 5.51 mg/kg
CL 94,302	2.5	3.9	6.3	5.6
CL 94,301	28	29.9	14.6	8.1
CL 94,221	0	0	0.52	0.3
CL 94,320	5.4	7.9	5.1	2.8
CL 94,365	12.7	13.7	19.2	16.9
Terbufos (CL 92,100)	0.5	0.4	0.18	0.073
Non-phosphorylated metabolites	52	44	53	66

The corn plants were extracted with methanol-acetone and methanol, and these extracts were subsequently partitioned into chloroform and water fractions. Results in this table are expressed as mg/kg equivalent of CL 92100 based on fresh plant

tissue weight, and in terms of % chloroform-soluble radioactivity, which accounted to 57%, 65%, 61 and 55% of the total radioactivity in corn plants harvested at 2, 4, 7 and 10 weeks.

The major metabolites in the 10-week old sweet corn plants grown in soil treated with [^{14}C]terbufos at 1.1 kg ai/ha (TE-640-006, Barringer, D. F.,(1973) are shown below in Table 13.

Table 13. Metabolites identified in corn plants grown in soil treated with [^{14}C]terbufos at 1.1 kg ai/ha.

Compound	Concentration (mg/kg)	% Recovered Radioactivity
Corn Plants (10 weeks post-treatment)		
CL 94365	0.515	26
CL 94301	0.260	13
CL 94302	0.179	9
CL 94320	0.095	5
CL 94221	0.009	0.4
CL 99875	0.666	34
CL 99843	0.173	9
CL 99844	0.043	2
CL 202474	0.007	4
CL 92100	0.002	0.1

Metabolism in cabbage

In the cabbage metabolism study (TE-640-004) by Peterson, R. (1976), cabbage plants were grown in the greenhouse and outside from seed in soil treated with [^{14}C]terbufos at a rate of 2.2 kg ai/ha, using both a 15G granular formulation and a liquid concentrate.

The concentration of radioactive residues found in the cabbage plants, expressed as mg/kg equivalent of terbufos, decreased with time (4 to 16 weeks) from 3.93 mg/kg to 0.09 mg/kg for outside granular treatment, from 1.48 mg/kg to 0.04 mg/kg for outside liquid treatment, and from 1.71 to 0.07 mg/kg for the greenhouse liquid treatment. This decline is primarily due to dilution from plant growth. The absolute amounts of radioactivity (in μCi) recovered in plants did not vary much with time.

The recovered radioactivity represented a maximum of 1.5% of the total applied dose. Terbufos is readily metabolized by cabbage plants by way of oxidation, s-p hydrolysis, methylation followed by subsequent oxidation to yield the non-toxic metabolites CL 202474, CL 99843 and CL 99875.

At the end of 12 weeks, 92% (0.07 to 0.22 mg/kg) of the total radioactivity consisted of unidentified water-soluble metabolites and the total amount of phosphate compounds were less than 0.01 mg/kg. There was no apparent metabolic difference between granular (15G) or liquid-treated soil in growing cabbage. The only notable difference in the total metabolic pattern of the entire study was the early appearance of metabolites CL 99843 and CL 99875 in the greenhouse cabbage at 4 weeks while none were evident in the outside grown plants at this early time.

The metabolism of terbufos in cabbage is similar to that reported in sugar beet (TE-640-003) by Caballa, S. (1974). The proposed metabolic pathway is presented in Figure 4.

Metabolism in rape seed

In a rape metabolism study (TE-640-007 by Chiu, T. Y. (1980)), rape was grown in soil treated with [^{14}C]terbufos in the furrow at 0.28 kg ai/ha. The total residual radioactivity in rape plants expressed in terms of parent was 0.63 mg/kg and 0.68 mg/kg for 1 and 2 month samples respectively. Residues were 0.42 mg/kg in the 2 month pods sample. At harvest (3 months post-treatment), the residue levels in fodder, hull and seed were 3.21, 3.63 and 1.11 mg/kg, respectively. The TRR found in various matrices of rape are summarized in Table 14.

Table14. TRR in rape after in-furrow treatment with [^{14}C]terbufos at 0.28 kg ai/ha.

Sample	Time Interval (Month)	TRR (mg/kg)
Plants	1	0.63
Plants	2	0.68
Pods		0.42
Fodder	3[a]	3.21
Hull		3.63
Seed		1.11

[a] Harvest (normal maturity).

The extractable radioactivity from the 1-month rape plant was 90%, of which 48% was organosoluble and 42% was aqueous soluble. By two-dimensional TLC analysis, about 16.3% of the radioactive organosolubles migrated away from the plate origin and the remaining 31.7% of the radioactivity stayed at the origin. Among the migrating radiocomponents, CL 99875 predominated with 4.9%, CL 94365 accounted for 4.0% and, CL 99843 and CL 94301 contributed 1.7 and 1.3% of the resolved organoextractables, respectively. The remaining 4.4% of the migrating radioactivity was composed of at least 6 minor components.

The extractability of rapeseed residues with dichloromethane followed by methanol was about 52%. The PES was further extracted with a methanol-water solvent system with mild heating, providing an additional 22.4% of extractable radioactivity. The acid-methanol extraction produced only 1.2% of the radioactivity, thus leaving 24.3% of the radioactivity in the seed PES.

The combined extractable radioactivity of seeds from dichloromethane and methanol extractions (52%) was partitioned and separated into hexane, acetonitrile and methanol fractions containing 22.0, 11.6 and 18.4% of the radioactivity, respectively. In the hexane fraction the chromatographic pattern indicated the majority of radioactivity migrating along with the oil, suggesting the incorporation of radioactivity into natural lipids. The TLC pattern from the acetonitrile fraction demonstrated that CL 99,875 (1.3%) and the oil-incorporated radio spots (5.5%) were the major components in this fraction. Several other radio-metabolites, such as CL 943,20, CL 94,301, CL 94,302, CL 99,843 and CL 94,365 were found in trace amounts each in this fraction.

The identified components found in the organosoluble fractions of plant and seed extracts are summarized in Table 15 and Table 16.

Table 15. Metabolites identified in rape forage treated with [^{14}C]terbufos at 0.28 kg ai/ha.

Identified Component	% of Organosoluble Radioactivity	% Total Radioactivity[a]	mg/kg[b]
CL 94320	1.7	0.816	0.005
CL 94301	2.7	1.296	0.008
CL 94302	0.6	0.288	0.002
CL 99875	10.2	4.896	0.031
CL 99843	3.5	1.68	0.011
-	2.4	1.152	0.007
CL 94365	8.4	4.032	0.025
-	0.5	0.24	0.001
-	2.3	1.104	0.007
-	2.0	0.96	0.006
Origin	65.7	31.68	0.2
Total	**100**	**48**	**0.303**

[a] Identified components from TLC of organosolubles of 1-month plant extract (48% of Total Radioactivity).

[b] Calculated level based on 48% (0.303 mg/kg) of total residue of 0.63 mg/kg in 1-month plant expressed as parent equivalents.

Table 16. Metabolites identified in rapeseed treated with [^{14}C]terbufos at 0.28 kg ai/ha

Components	Distribution of organosoluble metabolites in various solvents[a]		
	Hexane (22%)	Acetonitrile (11.6%)	Methanol (18.4%)
Oil	22	5.48	2.04
Origin-Bound	Trace	1.53	16.36
CL 99875	ND[b]	1.3	ND
CL 94301	ND	Trace	ND
CL 94302	ND	Trace	ND
CL 94320	ND	Trace	ND
CL 99843	ND	Trace	ND

[a] Radioactivity (52% of TRR) in the combined dichloromethane and methanol extracts of rapeseed (3-months post-treatment) after partition and separation into hexane, acetonitrile and methanol fractions.

[b] ND = Not Detected

Rape plants can readily absorb terbufos and closely related metabolites from the soil. The absorbed compounds are then initially metabolized in plant tissues by oxidation to phosphorylated metabolites, such as CL 94365 and CL 94301. These oxidized products degrade further through hydrolysis, methylation and subsequent oxidation thus leading to the formation of certain non-phosphorylated metabolites, such as CL 99843 and CL 99875.

In the present study, the total radioactivity in one-month old plants was composed of components (about 16%) which migrated on the TLC plate, components (74%) which were too polar to migrate, and 10% radioactivity which could not be extracted from the plant marc. The principal metabolites were identified as CL 99875 and CL 94365, accounting for 4.9% (0.031 mg/kg) and 4.0% (0.025 mg/kg), respectively.

In rape seeds, the hexane fraction comprised 22% of the radioactivity, which was probably associated with fatty acids or lipid-type compounds. The acetonitrile fraction, accounting for about 12%, mainly consisted of the oil-related compounds and CL 99,875 along with trace amounts of several other minor components. The proposed metabolic pathway of terbufos in rape is shown in Figure 4.

The hydrolysis study (TE-630-001 by Miller, P. (1973)), indicated that [methylene-^{14}C]terbufos was hydrolyzed to formaldehyde, whereas soil studies (TE-620-006 by North, H. (1973)) demonstrated the degradation of [^{14}C]terbufos to $^{14}CO_2$. Thus, in rape plants, it is likely that incorporation of carbon14-formaldehyde or $^{14}CO_2$ derived from [^{14}C]terbufos into natural products of various rape tissues accounts for a very large fraction of the radioactivity present in the plants or seeds.

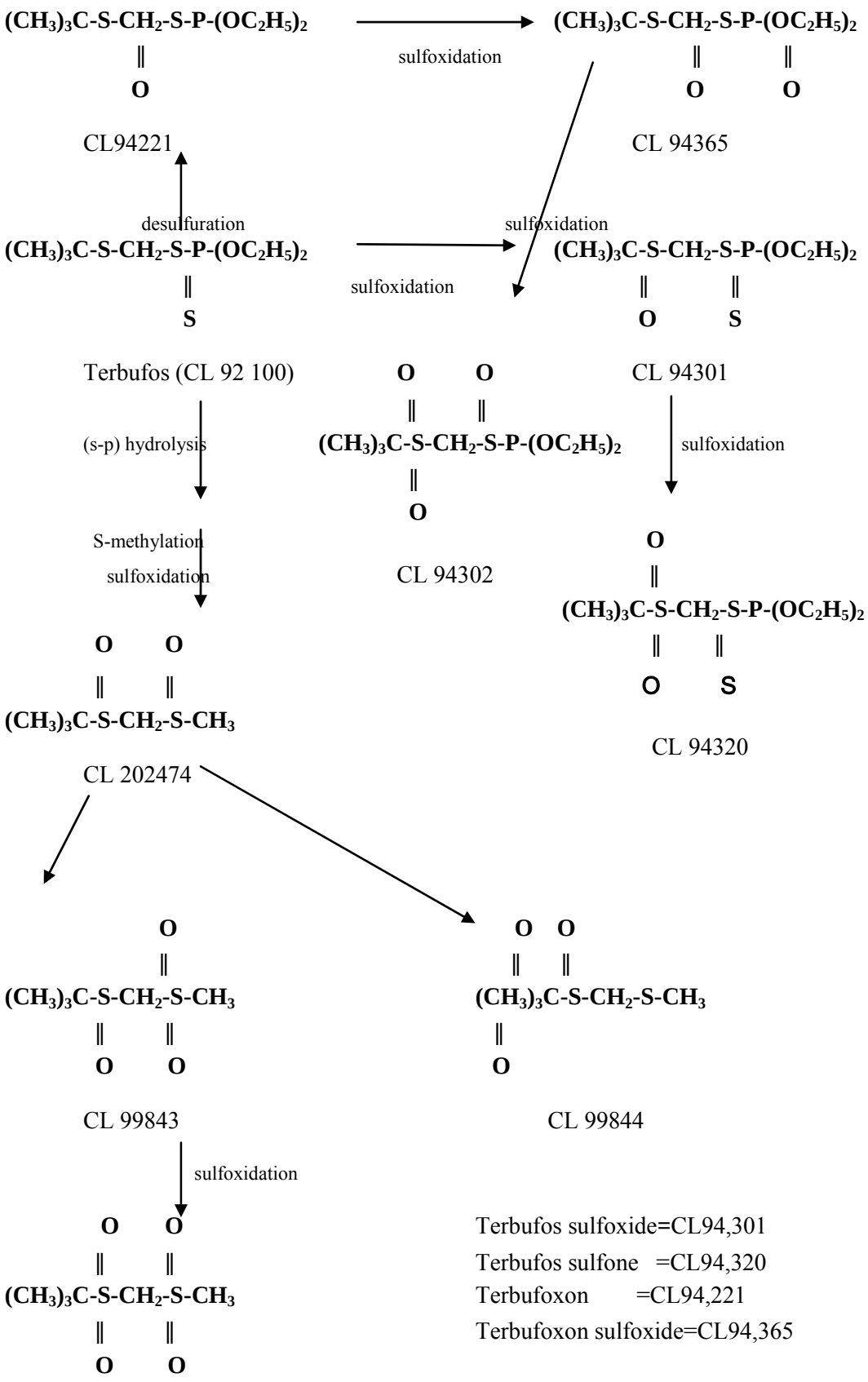


Figure 4 . Proposed metabolic pathway in plants.

Environmental Fate

Hydrolysis and biodegradation are the primary dissipation processes for terbufos in the environment when terbufos is incorporated into soil (US EPA, 1999). Under conditions favourable to microbial growth, the linear metabolic half-life in aerobic soil is approximately 27 days (5.6 days for nonlinear) and in anaerobic soil is 67 days (21 days for non-linear). Under abiotic conditions, the hydrolysis half-life is 12.3-13.7 days in the typical range of environmental pH values (pHs 5, 7, and 9).

The important metabolites terbufos sulfoxide and terbufos sulfone are more mobile and persistent than parent terbufos. The sulfoxide and sulfone half-lives are 116 and 96 days, respectively. These metabolites are also mobile in all tested soils and may reach ground water when terbufos is used in a location where irrigation or rain water moves through the soil profile to groundwater. In addition, terbufos and its metabolites may enter surface water as a result of run-off events.

Volatilization may be a major dissipation route for the portion of parent terbufos that remains on the surface of soil after incorporation. The relatively high vapour pressure (3.16×10^{-4} mm Hg) and observed Henry's Law Constant (6.58×10^{-3}) suggest that some of the parent compound will dissipate by diffusion into the atmosphere, but the amount that may volatilize will vary depending on the use site conditions and the mode of application.

Degradation in soil (aerobic)

The degradation route of methylene-^{14}C-Terbufos was investigated in silt loam soil in Wisconsin incubated under aerobic conditions for a period of 12 months (TE-620-004 by Peterson, R., 1983), ^{14}-C-Terbufos (specific activity 18.4 mcCi/mg and purity of 96.7%) in acetone was applied to 200 g of soil (wet weight basis) at a nominal rate of 5 mg/kg in the soil. Treated samples and controls were incubated in environmental control chambers maintained at an average temperature of 19 ± 2°C in the dark under continuous ventilation with moistened air. Systems were connected to gas washing bottles, containing ethylene glycol and monoethanolamine for collection of carbon dioxide and to trap radioactive volatiles from the reaction vessels.

Soil was sampled on days 0, 4, 7, 14, 30, 60, 120, 180, 270, and 365 post-treatment. Contents of the volatile traps and the carbon dioxide trap were also sampled at this time. To ensure efficient trapping of the carbon dioxide, a fresh portion of monoethanolamine was added to all remaining carbon dioxide traps at the 120-day sampling interval. The total of the 6, 9, and 12-month intervals was the sum of the two traps. All soil samples were either extracted immediately or stored at approximately 0°C until analysis.

Samples from all time points were extracted three times, first with aqueous methanol, next with methanol, and the third with 0.5% hydrochloric acid in methanol. After each extraction, the sample was vacuum filtered and rinsed with methanol and radioactivity was quantified in each filtrate by liquid scintillation counting (LSC).

The first two extracts were combined because they contained the majority of the radioactivity and after dilution with water, were partitioned into dichloromethane. As the sampling progressed, the amount of radioactivity increased in the acid extracts. Those extracts were partitioned separately with dichloromethane. All dichloromethane fractions were dried, concentrated, and analysed by thin layer chromatography (TLC).

Thin layer chromatography was performed on the various extracts, which were compared to the Rf values obtained from a series of TLC plates of the terbufos and metabolite standards. The standards were located by exposure to iodine vapours. The standard compounds appeared as brownish-yellow spots. The location of radioactive metabolites was by radioautography.

Based on the ^{14}C radioactivity present in the soil extracts, bound radioactivity and the volatiles, the mass balance for the applied radioactivity was determined to be in the range of 93 to 108

% of the applied dose of 5 mg/kg over the 365-day test period (Table 17). ^{14}C-carbon dioxide levels rose from 1.8% of the applied dose at the 4-day interval to 46% at 365 days, indicating extensive degradation of the applied ^{14}C-terbufos. Volatile organic components were found only at trace levels with a maximum of 0.5% of the applied dose found in the last sampling interval.

The distribution of dichloromethane-soluble ^{14}-C components of the combined aqueous/methanol and methanol soil extracts is presented in Table 18. Degradation of terbufos is quite rapid with a half-life of approximately 5 days. After 30 days, 4% of the applied dose remained as terbufos (CL 92,100) and after 365 days only 0.4% remained. The two major oxidative metabolites found were terbufos sulfoxide (CL 93, 201) and terbufos sulfone (CL 94,320). Concentrations of these metabolites decreased steadily until 365 days, when 0.3 g/kg of the sulfoxide and 0.1 mg/kg of the sulfone remained. The half-life of total CL 92,100-related compounds was approximately 100 days with an equivalent of about 8% of the applied dose remaining after one year.

TLC analyses of the dichloromethane partitioning phases of 60 and 120 day acid-methanol extracts showed only trace levels of terbufos sulfoxide and sulfone. These compounds were probably incompletely extracted with the first two extractions or slightly bound by the soil. No other unexpected metabolites were found in this fraction.

The presence of terbufoxon (oxygen analogue of terbufos) and terbufoxon sulfoxide were detected only at several early sampling intervals and never exceeded 0.04 mg/kg. Terbufoxon sulfone was not detected at all.

Table 17. Percent of applied dose of 5 mg/kg ^{14}C-CL 92,100 in aerobic soil at indicated time intervals.

Fraction	0-Day	14-Day	30-Day	120-Day	180-Day	270-Day	365-Day
Extract 1 (aqueous/methanol)	107.4	79.2	70.6	53.8	46.6	38.5	18.0
Extract 2 (methanol)	0.1	0.9	0.9	0.8	2.9	0.9	0.7
Extract 3 HCL/methanol	0.1	3.4	5.6	7.0	7.0	10.2	6.5
Soil Marc (air dried)	0.1	3.5	4.6	10.0	11.1	12.2	21.4
Volatiles (traps)	-	0.1	0.1	< 0.1	< 0.1	0.1	0.5
Carbon dioxide (trap)	-	9.0	13.5	22.0	25.3	33.6	45.9
TOTAL	**107.7**	**96.1**	**95.3**	**93.6**	**92.9**	**95.5**	**93.0**

Table 18. Nature and distribution of CL 92,100 and its metabolites in dichloromethane rextracts[1] of aerobic soil at various times.

Compound	0-Day		14-Day		30-Day		120-Day		180-Day		270-Day		365-Day	
	mg/kg[2]	%[3]	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%
CL 92,100	4.31	86.2	0.77	15.4	0.21	4.1	0.03	0.6	0.04	0.8			0.02	0.38
CL 94,301	0.31	6.2	2.55	50.9	2.62	52.3	1.47	29.4	1.24	24.7	0.88	17.7	0.29	5.9
CL 94,320			0.40	8.1	0.53	10.5	0.85	17.0	0.71	14.3	0.60	12.0	0.11	2.3
CL 94,221	0.04	0.8												
CL 94,365			0.02	0.4										
CL 94,302														
TLC origin	0.02	0.4	0.01	0.2	0.01	0.1	0.02	0.4	0.01	1.2	0.01	0.1	0.01	0.1
Unknowns[4]			0.03	0.6	0.03	0.6	0.08	1.6	0.08	1.5	0.06	1.2	0.07	1.4
TOTAL	**4.68**	**93.6**	**3.78**	**75.6**	**3.4**	**67.6**	**2.45**	**49.0**	**2.08**	**42.5**	**1.55**	**30.9**	**0.49**	**10.1**

[1] Soil extracts 1 and 2 were combined, diluted with water and extracted with dichloromethane.
[2] mg/kg of chemical in soil as calculated from the amount found in dichloromethane phase.
[3] Percent of applied dose based on mg/kg found divided by 5 mg/kg (applied dose).
[4] No individual compound was greater than 0.1% of applied dose.

Hydrolysis Rate and Products

The hydrolysis of ^{14}C-terbufos in sterile water at a pH of 5, 7, and 9 demonstrated that greatest stability occurs at pH 9, with a half-life for terbufos of 8.5 days (TE-630-001 by Miller, P., 1973). At a pH of 7 and 5, the half-life is 5.5 and 4.5 days respectively.

At the conclusion of a four-week study, 75.1, 72.4, and 68.3% of the radioactivity at pH 5, 7, and 9, respectively, was hydrophilic, with formaldehyde constituting the principal degradation product. Organophilic products consisted of the phosphorylated series of oxidative metabolites.

The existence of the principal hydrolysis products was demonstrated. Tert-butyl mercaptan and 0,0-diethylphosphorodithioic acid were converted to their benzyl derivatives with benzyl chloride and identified by mass spectrometry. Formaldehyde was converted to its 2,4-dinitrophenylhydrazone and identified by coincident thin-layer chromatography with an authentic standard.

The hydrolysis of ^{14}C-terbufos (CL 92,100), ^{14}C-terbufos sulfoxide (CL 94,301), and ^{14}C-terbufos sulfone (CL 94,320) in sterile buffer systems was studied under laboratory conditions (TE-630-005 by Marin, C. and Heim, D., 1999). Three buffers (pH 5, 7, and 9) and three temperature regimes (varying for each test substance and pH) were used to study the hydrolysis behaviour.

Hydrolysis of terbufos was strongly temperature dependent, and its rate of hydrolysis also increased slightly as the pH increased. Formaldehyde was the primary degradation product formed. Hydrolysis of terbufos sulfoxide and terbufos sulfone was strongly temperature and pH dependent. At pH 5 and pH 7, major degradates were des-ethyl terbufos sulfoxide, des-ethyl terbufos sulfone, and formaldehyde. Formaldehyde was the primary degradate at pH 9 for terbufos sulfoxide and terbufos sulfone systems.

The degradation pathways of terbufos sulfoxide and terbufos sulfone were pH dependent at elevated temperatures with de-esterification being the predominant reaction at pH 5 and 7, but only a minor reaction at pH 9.

Terbufos hydrolyzes rapidly under abiotic conditions at environmentally relevant temperatures and will not be expected to persist in aquatic systems. Hydrolysis of terbufos sulfoxide and terbufos sulfone occurs more slowly, but the des-ethyl derivatives that formed are not expected to be of toxicological concern.

The Arrhenius equation was used to estimate the DT_{50} values at 25° C. For terbufos, the hydrolysis half-lives at 25° C were estimated to be 1.20, 1.07, and 1.01 days for pH 5, 7, and 9 respectively. For terbufos sulfoxide, the hydrolysis half-lives at 25° C were estimated to be 239, 153, and 8.83 days at pH 5, 7, and 9 respectively. For terbufos sulfone, the hydrolysis half-lives at 25° C were estimated to be 127, 93.5, and 7.00 days at pH 5, 7, and 9, respectively.

Confined Rotational Crop Study

Confined rotational crop studies (TE-790-030 and TE-790-031) were conducted in Wisconsin and Nebraska by Lee, T and Belcher, D. (1984, 1986 a and b). Residues of CL 92,100-related compounds were determined in soil and rotational crops (cabbage, red beets, and wheat) from a treated corn field. In the study in Wisconsin, corn was planted in a silt loam soil and treated at planting with 2.24 kg ai/ha. After 30 days, the corn was destroyed and wheat, cabbage, and red beets were planted in the treated and untreated areas of the field.

At 127 days after treatment, cabbage was harvested, quartered, and stored within 2 hours at 0° C until shipment to the laboratory in about 4 months, where samples were frozen at -10° C to about -20° C until analysis about 14 months after sampling. Red beets and spring wheat were also harvested 127 days after treatment. Red beet tops were separated from roots and spring wheat heads and straw

were separated. Red beet and wheat samples were handled in the same manner as described for cabbage.

Soil samples were taken at 0-7.5 cm and 0-15 cm deep on the day of application, 33 and 127 days after treatment. The samples were stored at 0º C within 2 hours of sampling and handled as described for the plant samples.

The samples were analyzed using procedures described in Method M-1061 for soil, Method M-503 for cabbage, Method M-395 for red beets, and Method M-1592 for wheat straw and grain. Residues of terbufos-related compounds were analyzed using gas chromatography with flame photometric detection. Residues of CL-92,100 related compounds were less than the validated sensitivity of the method (0.05 mg/kg) in all cabbage, red beet and wheat grain samples. Wheat straw showed residues of 0.1 mg/kg. The soil half-life of CL-92,100-related compounds was calculated to be 30 days. Concurrent recovery tests were run with each group of samples to show that the analyses were performing properly throughout the study. For soils, they averaged 109% with a range of 88% to 140%. For cabbage, red beets roots, red beet tops, and spring wheat straw and heads, the recovery rates were 114%, 92%, 112%, and 102%, respectively. The results are summarized in Table 19 for soil and Table 20 for rotational crops.

In another study conducted in Nebraska, corn, planted in silt loam soil was treated at planting by soil incorporation with terbufos at the rate of 2.24 kg ai/ha (TE-790-031 by Lee, T and Belcher, D., 1986b). After 30 days, one area (Area I) of the corn was destroyed and planted with cabbage, sugar beets and wheat. At 130 days after treatment, cabbage and sugar beet samples were harvested and the samples frozen at -10°C within 2 hours of collection. The samples were shipped in dry ice and maintained frozen at -10 to -20°C until analysis about 14 months after sampling. Wheat green forage was harvested 69 days after treatment and wheat plants were harvested 95 days after treatment. The wheat samples were handled as described for cabbage and sugar beets.

One area of the originally planted corn (Area II) was harvested and planted with winter wheat. The wheat was allowed to mature and harvested 439 days after treatment. The samples were frozen and handled as described above for cabbage and sugar beets.

Soil samples were taken at 0-3 inches and 3-6 inches deep on the day of application, 30 and 125 days after treatment in the sugar beet and cabbage plots, and at day of treatment, 30, 69, and 97 days after treatment in wheat plots. Samples were handled as described for plant samples.

The samples were analyzed using procedures described in Method M-1061 for soil, Method M-503 for cabbage, Method M-395 for red beets, and Method M-1592 for wheat straw and grain. Residues of terbufos-related compounds were analyzed using gas chromatography with flame photometric detection. Residues of CL 92,100-related compounds were less than the validated sensitivity of the method (0.05 mg/kg) in all cabbage, sugar beet and wheat grain samples. Spring wheat forage showed residues of 0.15 mg/kg. No residues were detected in winter wheat straw and grain. The soil half-life of CL-92,100-related compounds was calculated to be 17 days in beet plots, 16 days in cabbage plots, and 10 days in wheat plots.

Concurrent recovery tests were run with each group of samples to show that the analyses were performed properly throughout the study. The average recoveries were 108% for soils, 80% for cabbage, 92% for beet roots and tops, 77% for spring wheat forage and heads, and 77% for winter wheat and grain. Results for both studies are summarized in Table 19 for soil and Table 20 for rotational crops.

Table 19. Summary of CL 92,100-related residues in soil.

Plot	Application rate (kg ai/ha)	Interval (days)	CL 92,100-Related Residues (mg/kg)			
			0 – 3”	3 – 6”	Average	Reference
	2.24	0	26.60	3.43	15.02	TE-790-030
	2.24	33	1.41	5.19	3.30	
	2.24	127	0.32	1.13	0.73	
Beet	2.24	0	7.70	4.02	5.86	TE-790-031
Beet	2.24	30	7.08	0.94	4.01	
Beet	2.24	125	0.06	< 0.05	0.06	
Cabbage	2.24	0	28.00	3.98	15.99	
Cabbage	2.24	30	15.70	0.38	8.04	
Cabbage	2.24	125	0.14	< 0.05	0.09	
Wheat	2.24	0	30.40	3.41	16.91	
Wheat	2.24	30	12.80	1.08	6.94	
Wheat	2.24	69	0.11	< 0.05	0.08	
Wheat	2.24	97	< 0.05	< 0.05	< 0.05	

Table 20. Summary of CL-92,100-related residues in rotational crops.

Commodity	Application rate (kg ai/ha)	Interval (days)	Residues (mg/kg)	Reference
Cabbage	2.24	127	< 0.05	TE-790-030
Red beet tops	2.24	127	< 0.05	
Red beet roots	2.24	127	< 0.05	
Spring wheat grain	2.24	127	< 0.05	
Spring wheat straw	2.24	127	0.10	
Cabbage	2.24	130	< 0.05	TE-790-031
Beet root	2.24	130	< 0.05	
Beet tops	2.24	130	< 0.05	
Spring wheat forage	2.24	69	0.15	
Spring wheat heads	2.24	95	< 0.05	
Winter wheat grain	2.24	439	< 0.05	
Winter wheat straw	2.24	439	< 0.05	

RESIDUE ANALYSIS

Analytical Methods

Plant Matrices

Several analytical methods have been developed for the determination of terbufos in plant commodities, which are suitable for data collection and some for enforcement (Table 21). Validation data are shown in (Table 22).

Similar data collection methods were used in the analysis of terbufos residues in or on crop commodities. All analytical methods for terbufos residues are designed to extract parent terbufos and its oxygenated metabolites: terbufos sulfoxide (CL 94301), terbufos sulfone (CL 94320), terbufoxon (CL 94221) and terbufoxon sulfoxide (CL 94365). Terbufos and its metabolites are oxidized to the common moiety metabolite of terbufoxon sulfone (CL 94302) using m-chlorobenzoic acid, which is then analyzed by gas chromatography. The methods vary slightly, usually in the extraction solvent used.

Residues of terbufos are extracted by blending, typically with methanol:chloroform (10:90, v/v), filtered, and then an aliquot of the filtrate is evaporated to dryness. The residue is reconstituted, or if necessary, is taken up in hexane and further cleaned by partitioning into acetonitrile before reconstitution. The residues are taken up in acetone, the solution treated with activated charcoal, filtered, and the solvent evaporated. For some substrates like maize, a charcoal/benzene treatment is used.

Residues are oxidized to terbufoxon sulfone using m-chloroperbenzoic acid. Excess reagent is destroyed with sodium sulfite. If further cleanup is necessary, the residue is dissolved in acetone and mixed with precipitating solution (aqueous 1.25 g/L ammonium chloride/2.1 g/L phosphoric acid). The filtrate from this solution is partitioned into the chloroform, which is evaporated and reconstituted in acetone. Final determination of terbufoxon sulfone is carried out on a gas chromatograph equipped with a phosphorus-selective detector, either flame ionization detector (FID) or a flame-photometric detector (FPD) in the phosphorus mode.

Table 21. Methods for the determination of terbufos in or on plant commodities

Method No.	Crop	Extraction	Detection Method	LOQ (mg/kg)	Reference
M-1340	Banana	10% Methanol in dichloromethane	GC/FPD	0.01	TE-244-005
M-3072	Banana	10% methanol in dichloromethane	GC/FPD	0.002	TE-244-025; TE 244-057
M-1360	Coffee	dichloromethane	GC/FPD	0.05	TE-244-015
M-1754	Maize	10% methanol in chloroform	GC/FPD	0.01 grain, 0.05 others	TE-244-049
M-336	Maize	10% methanol in chloroform	GC/FID ??	0.05	TE-244-054
M-1754	Sorghum	10% methanol in chloroform	GC/FPD	0.01 grain, 0.05 others	TE-244-049
M-995	Sorghum	10% methanol in chloroform	GC/FPD	0.05	TE-244-056
M 2457	Sugar beets[b]	10% methanol in chloroform	GC/FPD	0.01	TE-244-023
RLA10333V	Sugar beets[b]	10% methanol in chloroform	GC/FPD	0.05	TE-244-009
RLA-10156	Sugar beets[b]	10% methanol in chloroform	GC/FPD	0.05	TE-244-063
M-395	Sugar beets[b]	10% methanol in chloroform	GC/FID	0.05	TE-244-004, TE-244-064
M-1747	Sugar beets[a]	10% methanol in chloroform	GC/FPD	0.01	TE-244-007
M-336	Sweet corn	10% methanol in chloroform	GC/FPD	0.01 grain, 0.05 others	TE-244-054
M-1754	Sweet corn	10% methanol in chloroform	GC/FID	0.05	TE-244-049

[a] Methods for sugar beets (roots).
[b] Methods for sugar beets roots and tops.

The limit of determination for most of the reported trials was 0.05 mg/kg, but limits for some methods/substrates were 0.01 or 0.005 mg/kg. Recoveries of terbufos and its related metabolites were tested on all the sample types reported in the trials over the concentration range 0.01 – 1.0 mg/kg. Method validation data are summarized in Table 22.

Table 22. Validation of analytical methods for the determination of terbufos in plant products.

Crop	Method (Ref.)	Matrix [a]	Fort. Level (mg/kg)	% Recovery (Avg ± SD, Range, Number of Samples)[b]						
				All[c] Analytes	CL 92100	CL 94301	CL 94320	CL 94221	CL 94365	CL 94302
Banana	M 1340 (TE-244-005)	Whole banana	0.01, 0.5	93 ± 21 70-133 (n=8)					109 ± 19 79-140 (n=12)	
	M 1340 (PGD-183)	Whole banana	0.01	95 80-116 (%RSD= 14, n=5)						
			3.0	85 74-101 (%RSD= 13, n=5)						
		Banana pulp	0.01	125 117-135 (%RSD= 125,n=5)						

Crop	Method (Ref.)	Matrix [a]	Fort. Level (mg/kg)	% Recovery (Avg ± SD, Range, Number of Samples)[b]						
				All[c] Analytes	CL 92100	CL 94301	CL 94320	CL 94221	CL 94365	CL 94302
			3.0	80 73-92 (%RSD= 8, n=5)						
	M-3072 (TE-244-057)	Whole banana	0.002-0.02	84± 5.7 76 –89 n=6						
		Pulp	0.002-0.02	90 ± 5.5 83- 98 n=6						
Coffee	M 1360 (TE-244 -015)	Beans	0.05-0.5		114 ± 17 101-143 (n=6)				124 ±14 103-140 (n=8)	
Maize	M 1754 (TE-244 -049)	Dry plant	0.05-1.0	111 ± 22 86-137 (n=6)						
		Green plant	0.05-1.0	96 ± 4 90-102 (n=6)						
		Cannery waste	0.05-1.0	102 ± 17 85-133 (n=6)						
		Grain	0.01-0.2	97 ± 14 76-111 (n=6)						
	M-336 (TE-244-054)[e]	Silage	0.05-1.0		97 ± 20 71-130 (n=9)					110 ± 20 88-136 (n=6)
		Fodder (stover)	0.05-1.0		99 ± 32 68-134[f] (n=4)					90 ± 3 84-91 (n=5)
		Grain	0.05-1.0		101 ± 11 83-115 (n=9)					101 ± 14 83-118 (n=7)
Sorghum	M-995 (TE-244-056)	Grain	0.05-0.2		112± 22 90 – 136 n=4	88 ± 13 88 – 118 n = 4	109 ± 7 111 – 116 n = 4	102 ± 5 99 – 109 n = 4	105 ± 20 78 – 121 n=4	102 ± 11 92 – 114 n = 4
		Fodder	0.05-0.2		112 ± 10 97 – 118 n=4				110 ± 15 94 – 130 n=4	
		Silage	0.05-0.5		95 ± 19 73 –111 n=4				113 ± 14 93 – 125 n = 4	
Sorghum	M-1754 (TE-730-052)	Grain	0.01-0.4	105 ± 18 86 – 123 n=4						
		Forage	0.05-0.5	91 ± 5.0 86 – 96 n= 3						
		Fodder	0.05-0.50	80 ± 14 64 – 103 n=6						
Sugar Beet	M2457 (TE-244 -023)	Tops	0.01-0.2	76 ± 9 62-85 (n=8)						
		Roots	0.01-0.2	91 ± 21 56-128 (n=11)						

Crop	Method (Ref.)	Matrix [a]	Fort. Level (mg/kg)	% Recovery (Avg ± SD, Range, Number of Samples)[b]						
				All[c] Analytes	CL 92100	CL 94301	CL 94320	CL 94221	CL 94365	CL 94302
	M1747 (TE-244 -007)	Roots	0.01-1.0	84 ± 13 63-100 (n=12)						
Sugar Beets	M1747 (TE-244 -009)	Tops	0.05-1.0		89 ± 10 79-104 (n=5)					93 ± 10 83-106 (n=5)
	M-395 (TE-244 -004)	Roots	0.05-1.0		107 ± 15 84-133 (n=10)					84 ± 12 71-110 (n=11)
		Tops	0.05-1.0		100 ± 24 71-137 (n=11)					98 ± 19 62-130 (n=11)
Sweet corn	M1754 (TE-244 -049)	S. Corn K+C [d]	0.01-0.2	91 ± 17 69-116 (n=6)						
	M-336 (TE-244 -054)[e]	S. Corn (K+C)[d]	0.05-0.5		113 ± 11 101-120 (n=3)					100 ± 17 86-119 (n=3)

[a] Interferences in control samples were insignificant to none.
[b] All analytes fortified were converted by the method to the common moiety and detected as CL94301.
[c] Samples were fortified with terbufos, CL94301, CL94320, CL94221, CL94365, and CL94302, except for two validation studies on corn and sugar beets (TE-244-049 and -007) in which samples were fortified with a 1:1:1 mixture of terbufos, CL94301 and CL94365.
[d] K+C = Sweet corn kernel plus cob.
[e] Includes additional method validation recoveries provided in a field trial study report (TE-723-002).

Animal Matrices

All analytical methods for terbufos residues in animal tissues, milk, and egg are designed to extract parent, terbufos, and its oxygenated metabolites: terbufos sulfoxide, terbufos sulfone, terbufoxon, and terbufoxon sulfoxide. Terbufos and its metabolites are oxidized to the common moiety metabolite of terbufoxon sulfone using m-chlorobenzoic acid that is analyzed by GC. The methods vary slightly usually in the extraction solvent used. A brief description of the key points of the methods follows.

After extraction, the sample is filtered, and an aliquot of the filtrate is evaporated to dryness. Residues are oxidized to terbufoxon sulfone using m-chloroperbenzoic acid. The excess oxidizing agent is neutralized with sodium sulfite. The chloroform solution is evaporated and reconstituted in acetone. Final determination of terbufoxon sulfone is carried out on a gas chromatograph equipped with a phosphorus-selective detector, either flame ionization detector (FID) or a flame-photometric detector (FPD) in the phosphorus mode. Methods reported for the determination of terbufos in domestic animal commodities are shown in Table 23, with validation data in Table 24.

Table 23. Methods for the determination of terbufos and its metabolites in animal tissues.

Method Number (Reference Number)	Matrix	Extraction	Detection Method	LOQ (mg/kg)
M-372 (TE-705-002)	Cattle tissues	Acetonitrile	GC/FID	0.05
M-353 (TE-705-003)	Milk	Dichloromethane	GC/FID	0.01
M-1829 (TE-245-001)	Milk	CH_2Cl_2/acetone	GC/FPD	0.005
M-401 (TE-245-002)	Chicken tissues	Acetonitrile	GC/FID	0.05
M-396 (TE-705-001)	Egg	Acetonitrile	GC/FID	0.01

The LOQ for the milk method is 0.005 or 0.01 mg/kg, for the tissue methods, 0.05 mg/kg, and for the egg, 0.01 mg/kg. Recoveries of terbufos and its related metabolites were tested on the samples over the concentration range 0.005 – 1.0 mg/kg. Method validation data are summarized in Table 24.

Table 24. Validation of analytical methods for terbufos in animal products.

Matrix	Method (Ref.)[a]	Fort. Level (mg/kg)	Terbufos CL 92100	Terbufos Sulfoxide CL 94301	Terbufos Sulfone CL 94320	Terbufoxon CL 94221	Terbufoxon Sulfoxide CL 94365	Terbufoxon Sulfone CL 94302
Cattle								
Milk	M-1829 (TE-245-001)	0.005-1.0	88±12 67-110 (n=17)	95±6 87-101 (n=4)	---	---	---	---
	M-353 (TE-705-003)	0.01-0.50	79±13 58-97 (n=7)	---	---	---	---	88 ± 19 73-131 (n=7)
		0.10	71	74	98	100	78	73
Fat	M-372 (TE-705-002)	0.05-1.0	85±16 72-120 (n=7)	---	---	---	---	91 ± 19 71-126 (n=7)
		0.20	76	71	83	94	86	103
Kidney	M-372 (TE-705-002)	0.05-1.0	90±10 65-93 (n=7)	---	---	---	---	93 ± 16 69-110 (n=7)
		0.20	71	72	65	69	52	78
Muscle	M-372 (TE-705-002)	0.05-1.0	74±12 54-92 (n=8)	---	---	---	---	81 ± 11 65-96 (n=8)
		0.20	84	81	71	75	81	94
Liver	M-372 (TE-705-002)	0.05-1.0	70±10 60-81 (n=3)	---	---	---	---	95 ± 13 68-102 (n=5)
Poultry								
Fat	M-401 (TE-245-002)	0.05-1.0	80±19 58-100 (n=7)	---	---	---	---	101 ± 21 82-136 (n=7)
		0.2	94	86	95	105	104	86
Muscle	M-401 (TE-245-002)	0.05-1.0	84±10 67-94 (n=7)	---	---	---	---	93 ± 17 66-114 (n=7)
		0.2	67	74	101	98	93	85
Liver	M-401 (TE-245-002)	0.05-1.0	70±9 67-94 (n=7)	---	---	---	---	79 ± 18 48-109 (n=8)
		0.2	59	68	88	92	60	64
Skin	M-401 (TE-245-002)	0.05-1.0	85±10 72-98 (n=7)	---	---	---	---	93 ± 9 84-108 (n=7)
		0.2	85	92	103	93	97	84
Kidney	M-401 (TE-245-002)	0.05-1.0	64±10 52-86 (n=11)	---	---	---	---	78 ± 6 71-86 (n=7)
		0.2	58	60	74	73	70	71
Egg	M-396 (TE-705-001)	0.1	71	70	67	69	74	73
		0.01- 0.5	82±17 60-111 (n=7)					93 ± 20 65-119 (n=7)
		0.1	71	70	67	69	74	73

[a] The results reflect the validation data generated/reported with the feeding studies with the exception of recovery data for methods M-1829 and M-401, which appeared in separate method validation studies.

Environmental Samples

Methods were also reported for the determination of terbufos in environmental samples. The methods for soil and water are shown in Table 25, with method validation data in Table 26.

The methods that have used in the analysis of terbufos residues in soil and water are designed to extract parent terbufos and its oxygenated metabolites, terbufos sulfoxide and terbufos sulfone.

One soil method, M-1638, also determines terbufoxon, terbufoxon sulfoxide and terbufoxon sulfone. The limit of determination for total residues or individual analytes is 0.01 or 0.05 mg/kg in soil and 0.001 or 0.0001 mg/kg in water. A brief description of the key points of all of the methods follows.

Residues in soil are extracted with 10% aqueous methanol and partitioned into dichloromethane (CH_2Cl_2). For the water method, residues are extracted with CH_2Cl_2. The CH_2Cl_2 is evaporated and the residue reconstituted in acetone. If further clean up is necessary, an aliquot is passed through a silica solid phase extraction cartridge. Final determination is carried out using GC/FPD in the phosphorus mode or, in the case of water method M-2623, GC equipped with a mass selective detector (MSD). Two water methods (M-1615 and M-1144) convert all terbufos residues to the common moiety, terbufoxon sulfone, using m-chloroperbenzoic acid.

Table 25. Methods for the determination of terbufos in soil and water.

Method No. (Reference No.)	Matrix	Extraction[a]	Detection Method	Analytes determined (LOQ)
M-1912 (TE-242-006)	Soil	10% MeOH:H_2O	GC/FPD	Terbufos, CL94301 and CL94320 (each at 0.05 mg/kg)
M-1784 (TE-242-003)		10% MeOH:H_2O	GC/FPD	Terbufos, CL94221, CL94301 and CL94320 (each at 0.05 mg/kg)
M-1638 (TE-242-001)		10% MeOH:H_2O	GC/FPD	The LOQ is 0.010 mg/kg for terbufos and terbufoxon (CL 94221), and 0.050 mg/kg for terbufos sulfoxide (CL94301) and sulfone (CL94320) and terbufoxon sulfoxide (CL94365) and sulfone (CL94302).
M-1644 (TE-243-007)	Water	CH_2Cl_2	GC/FPD	Terbufos, CL94301 and Cl94320 (each at 0.001 mg/kg)
M-2623 (TE-243-004)		CH_2Cl_2	GC/MSD	
M-1149 (TE-243-002)		CH_2Cl_2	GC/FPD	Terbufos, CL94301 and Cl94320 (each at 0.001 mg/kg)
M-1615 (TE-243-003)		CH_2Cl_2	GC/FPD	Total terbufos-related residues (0.0001 mg/kg)
M-1144 (TE-243-001)		CH_2Cl_2	GC/FPD	

[a] MeOH = methanol; CH_2Cl_2 = dichloromethane.

Table 26. Validation of analytical methods for the determination of terbufos in soil and water.

Matrix	Method	Fort. Levels (mg/kg)	All	Terbufos CL92100	Terbufos Sulfoxide CL94301	Terbufos Sulfone CL94320	Terbufoxon CL94221	Terbufoxon Sulfoxide CL94365	Terbufoxon Sulfone CL94302
Soil	M-1912 (TE-242-006)	0.05-10		90 ± 6 78-100 (n=20)	101 ± 4 96-108 (n=20)	98 ± 3 95-104 (n=20)			
	M-1784 (TE-242-003)	0.05-20		97 ± 3 92-101 (n=9)	102 ±7 92-109 (n=7)	99 ± 5 91-105 (n=7)	97 ± 3 91-101 (n=7)		
	M-1638 (TE-242-001)	0.01-15[a] 0.05-5[b]		90 ± 7 81-101 (n=10)	93 ± 7 79-101 (n=10)	102 ± 11 87-115 (n=8)	93 ± 10 83-106 (n=8)	90 ± 10 80-108 (n=8)	91 ± 9 81-110 (n=8)
Water	M-1644 (TE-243-007)	0.0001-0.1		109 ± 9 100-125 (n=8)	106 ± 7 100-118 (n=6)	103 ± 8 97-113 (n=6)			
	M2623 (TE-243-004)	0.0001-0.1		87±5 78-91 (n=6)	98±6 91-106 (n=6)	96±4 90-100 (n=6)			
	M-1149 (TE-243-002)	0.0001-0.1		91 ± 4 85-96 (n=6)	96 ± 20 62-119 (n=6)	98 ± 15 72-114 (n=6)			
	M-1615	0.0001-	100 ±						

Matrix	Method	Fort. Levels (mg/kg)	All	Terbufos CL92100	Terbufos Sulfoxide CL94301	Terbufos Sulfone CL94320	Terbufoxon CL94221	Terbufoxon Sulfoxide CL94365	Terbufoxon Sulfone CL94302
	(TE-243-003)	00.010	15 77-121 (n=12)						

[a] Range of fortification levels for terbufos and CL94221.
[b] Range of fortification levels for terbufos, CL94301, CL94320, CL94302 and CL94365.

Enforcement methods

An adequate method is available for enforcement of terbufos MRLs in or on plant commodities. The GC-flame ionization detection method for determining terbufos and its phosphorylated metabolites is described in PAM (Pesticide Analytical Manual), Vol.II as Method I. The hazardous reagent, benzene, is specified in this method. Method M-1754, a modification of Method I in PAM, substitutes acetone for benzene and dichloromethane for chloroform. This method underwent successful method validation trial by the Residue Analytical Laboratory and was forwarded by the US EPA to FDA (Food & Drug Administration) for revision of PAM Vol II.

Multiresidue method

Terbufos and its metabolites were taken through the US FDA Multiresidue Method protocols described in PAM Volume 1 with some success (TE-244-059 by Gross, J., 1990). Terbufos and its metabolites were not tested through Protocols A and B since these protocols do not pertain to organophosphates. Terbufos sulfoxide and terbufoxon sulfoxide did not pass Protocol C.

Since terbufos sulfoxide and terbufoxon sulfoxide did not chromatograph adequately with any of the four columns, they were not tested by Protocol D and Protocol E. The other metabolites terbufos sulfone, terbufoxon sulfone, and terbufoxon did perform well through Protocols D and E and could be determined by the GC multi residue method.

Table 27 summarizes recovery data under Protocol D for terbufos sulfone, terbufoxon, and terbufoxon sulfone using sugar beet roots as non-fat food representative. Recoveries were run in duplicate with 0.05 mg/kg and 0.10 mg/kg of each compound.

Under Protocol E, Florisil had shown good recoveries and correct elution for standards of heptachlor epoxide and endrin. Only Terbufos sulfone was found to elute with one of the Florisil elution systems (PAM I,252.12b-dichloromethane solvents) and even then, the compound split between the second and third elution fractions.

Corn grain was used as a fatty food and sugar beet roots as a non-fat food. In three out of four recoveries, terbufos sulfone split between the second and third eluates. Table 28 summarizes the recovery data.

Table 27. Summary of recovery data with sugar beet roots using Protocol D

Fortification level (mg/kg)	Terbufos sulfone CL 94320	Terbufoxon CL 94221	Terbufoxon sulfone CL 94302
0.05	110	102	137
0.05	101	100	123
Average	105	101	130
0.10	117	109	122
0.10	121	115	132
Average	119	112	127

Table 28. Summary of recovery data for Terbufos Sulfone using Protocol E.

Sample	Fortification level (mg/kg)	Sugar beet roots (% recovery)
Eluant 2	0.05	38.6
Eluant 3	0.05	47.4
Eluant 3	0.05	77.0
	Average	54.3
Sample	**Fortification level (mg/kg)**	**Maize grain(% recovery)**
Eluant 2	0.05	46.8
Eluant 3	0.05	42.8
Eluant 2	0.05	48.9
Eluant 3	0.05	40.7
	Average	44.8

Stability of pesticide residues in stored analytical samples

Frozen storage stability studies were conducted in a variety of substrates including corn (grain, forage, fodder), sugar beets (tops and roots), banana (whole and pulp), milk, soil, and water samples. Control samples were fortified with known concentrations of terbufos and then placed in frozen storage at approximately -10 C or less. The fortified samples were analyzed periodically for total terbufos-related residues using the same analytical method as that used for the residue field trial or processing samples.

Plant commodities

The stability of terbufos residues has been determined in freezer storage stability studies in the representative plant commodities of corn (grain, plants, straw); sugar beet (tops and roots); peanut nutmeat, and banana (unpeeled and pulp).

Control samples were fortified with a mixed standard of terbufos (CL 92100), terbufoxon sulfoxide (CL 94365), and terbufoxon sulfone (CL 94302), at a concentration of either 0.1 or 0.5 mg/kg. Banana pulp and whole fruit were fortified with a mixture of all six analytes identified as residues of concern: terbufos, terbufos sulfoxide (CL 94301), terbufos sulfone (CL 94320), terbufoxon (CL 94221), terbufoxon sulfoxide, terbufoxon sulfone. In addition, a recently conducted study on sugar beets used samples fortified with a mixed standard containing CL 94301, CL 94320, and CL 94221.

The samples were stored frozen (< 0–10° C) and then removed from storage at various intervals and analyzed for total residues of terbufos. The methods of analysis were the same as those used for data collection. The storage intervals investigated were selected to follow the same intervals and conditions as the field crop trials.

The storage stability results for the representative commodities are summarized in Table 29. Terbufos residues fortified in representative crop samples (root, grain, watery and oily commodities) were shown to be stable in frozen storage for at least 18 months.

Table 29. Terbufos storage stability in various frozen plant commodities.

Commodity	Fortification level (mg/kg)	Storage (°C)	Interval (Months)	Concurrent Recovery	% Survived	Survived, %, corrected for recovery[a]	Reference (Method)
Banana (whole)[b]	0.1	≤-10	0	79	74, 73	94, 92	RES-99-070 TE-326-015 (M 3072)
			3	84	71, 81	85, 96	
			6	108	108, 109	100, 101	
			12	101	91, 90	90, 89	
			18	79; 107	40, 103; 84, 101	51, 130; 79, 94	
Banana Pulp[b]			0	75	79, 81	105, 108	
			3	93	76, 72	82, 77	

Commodity	Fortification level (mg/kg)	Storage (°C)	Interval (Months)	Concurrent Recovery	% Survived	Survived, %, corrected for recovery[a]	Reference (Method)
			6	100	67, 79	67, 79	
			12	74	48, 61	65, 82	
			18	93; 102	51, 28 40,78	55, 30; 39, 76	
Corn Plants[c] (Forage)	0.5	approx. -10	0	96	96	100	C-3299 TE-326-014 (M-336)
			6	137	83	83	
			8	76	108	142	
			12	30	97	97	
			23	111	72	65	
			25	96	78	81	
Corn Fodder/ Straw[c]	0.5		0	90	90	100	C-3299 TE-326-014 (M-336)
			6	98	106	108	
			8	70	89	127	
			12	136	70	70	
			23	114	82	72	
			25	118	122	103	
Corn Grain[c]	0.1		0	130	130	100	
			5	60	68	68	
			8	61	95	95	
			12	74	69	93	
			21	109	119	109	
			25	74	99	134	
Sugar Beet Roots[c]	0.1	approx. -10	0	85	85	100	C-3298 TE-326-012 (M-395)
			5	78	77	99	
			8	99	107	108	
			13	78	92	118	
			22	95	97	102	
			24	123	87	87	
Sugar Beet Tops[c]	0.5		0	70	71	101	
			5	82	102	124	
			8	94	89	95	
			13	111	71	64	
			22	69	74	74	
			24	84	92	110	
Sugar Beet Roots[d,e]	0.2	< 0	0.6	89	89	100	RES-97-017 TE-326-007 (M-2457)
			1	87	78	90	
			3	96	91	95	
			6	88	70	80	
			12	93	77	83	
			18	102	67	66	
			24	90	61	68	
Sugar Beet Tops[d,e]	0.5	< 0	0.6	109	88	81	RES-97-017 TE-326-007 (M-2457)
			1	80	74	93	
			3	92	79	86	
			6	87	70	80	
			12	82	51	62	
			18	90	66	73	
			24	83	51	61	

[a] Corrected percent recovery determined by dividing the percent recovered by the respective concurrent recovery and multiplying by 100. (No corrections were made when concurrent recoveries were not in the acceptable range of 70-120%.)

[b] Samples were fortified with a mixed standard containing terbufos (CL 92100) and its metabolites, terbufos sulfoxide (CL 94301), terbufos sulfone (CL 94320), terbufoxon (CL 94221), terbufoxon sulfoxide (CL 94365), terbufoxon sulfone.

[c] Samples were fortified with a mixed standard of terbufos, CL 94365, and CL 94302.

[d] Samples were fortified with a mixed standard containing CL 94301, CL 94320, and CL 94221.

[e] The samples were not successfully analyzed until the 0.6 month interval due to a low concurrent recovery on the initial analytical run. Also note that the "% recovery" is the average of duplicate analyses of the same sample, except at 12-months when two additional (contingency) tops stability samples were analyzed

Animal commodities

The stability of terbufos residues in cold storage has been determined for milk. Control samples were fortified with a representative mixed standard of terbufos and terbufos sulfoxide (CL 94301) at a concentration of 0.05 mg/kg, stored in 1.7 – 3.3 °C and samples removed and analyzed at various intervals. The results are summarized in Table 30.

Table 30. Terbufos frozen storage stability in milk

Commodity	Fortification level (mg/kg)	Storage (°C)	Interval (Days)	Survived residue %	Study Reference
Milk	0.05	1.7 - 3.3	0 7 14	94 84 79	TE-245-001

Soil and Water

The stability of terbufos residues in frozen storage has been determined for soil and water. Control samples were fortified with a representative mixed standard of terbufos, terbufos sulfoxide (CL 94301), and terbufos sulfone (CL94320), at a concentration of 0.250 mg/kg for soil and 0.010 mg/kg for water. The samples were stored at 0°C, and the samples were later removed and analyzed at various intervals. Soil samples were stored in glass while water was stored in plastic. The storage stability results (Table 31) indicate that terbufos residues are stable in frozen water and soil for at least 4 and 24 months, respectively.

Table 31. Terbufos Storage Stability in Soil and Water.

Fortification level (mg/kg)	Storage (°C)	Storage Interval	Residue remained in Stored Sample (%)[a]			Reference Number
			Terbufos	CL 94301	CL 94320	
Soil						
0.25 (each analyte)	0	0 months	90 (96) 92 (98)	104 (100) 107 (103)	99 (98) 100 (99)	TE-326-006
		3 months	85 (93) 86 (95)	112 (109) 118 (115)	100 (100) 102 (102)	
0.25 (each analyte)	0	6 months	91 (82) 90 (81)	126 (107) 128 (108)	112 (94) 113 (95)	
		12 months	72 (80) 72 (80)	134 (126) 134 (126)	100 (100) 100 (100)	
		14 months	71 (78) 68 (75)	128 (128) 125 (125)	98 (99) 96 (97)	
		18 months	65 (71) 66 (72)	134 (128) 133 (127)	102 (98) 101 (97)	
		24 months	62 (65) 64 (67)	136 (121) 136 (121)	99 (93) 101 (94)	
Water						
0.01 (each analyte)	-23 to -29	0 weeks	102 [100][b] 107 [99]	106 108	103 106	TE-326-010
		1 week	93	107	103	
		2 weeks	69 [98] 67 [102]	NA[c] NA	NA NA	
		3 weeks	69 63	111 110	109 100	
		4 weeks	77 [100]	100	99	
		5 weeks	76	104	104	
		8 weeks	55 [96] 52 [96]	108 103	103 102	
		16 weeks	52 [83] 59	109 106	109 109	

[a] Soil and water samples were analyzed by GC methods M-1912 and M-1644, respectively.

[b] After losses of parent were observed (probably due to adsorption to the plastic) another set of water samples was fortified with terbufos alone and analyzed to determine whether water could be stored in glass containers and also to determine if terbufos was being oxidized to CL94301 or CL94320. The results for terbufos fortified in water and stored in glass bottles

are shown in brackets; all other results are for distilled water samples fortified with each analyte and stored frozen in plastic bottles.

[c] NA = Not analyzed.

USE PATTERN

Terbufos is a systemic and contact organophosphorous insecticide/nematicide. It is formulated as a granule for application to crops and soil. It is usually applied once at planting or as a subsequent side-dressing.

Terbufos is registered in a number of countries and the registered uses are listed in Table 32.

Table 32. Registered uses of terbufos.

Crop	Country	Form type	Conc.	Application method[a]	Rate kg ai/ha	Number of application.	PHI (days)
Banana	Australia	G	150 g/kg	In established plantations, granules are spread in soil around follower plant; In new plantations, granules are spread in soil around the plant at sowing	3 g ai/plant	4	--
Banana	Brazil	G	50 g/kg 150 g/kg		3-4 g ai/plant	3	3
Banana	Philippines	G	100 g/kg		2 g ai/plant	4	--
Banana	Mexico	G	50 g/kg; 150 g/kg		3 g ai/plant	2-3	--
Banana	Guatemala Belize Honduras Nicaragua Costa Rica Panama	G	100 g/kg 150 g/kg		3-4 g ai/mat	2 – 3	--
Banana	Chile	G	100 g/kg		15-20	-	60
Coffee	Brazil	G	50 g/kg; 150 g/kg	In 10 cm under soil at planting or in crown projection	1.5-3.0 g ai/plant up to 7.5 kg ai/ha	1	90
Coffee	Guatemala Belize Honduras El Salvador Costa Rica Panama	G	100 g/kg; 150 g/kg	Broadcast in small radius around plant	0.75-1.05 g ai/tree	2	60
Coffee Seedbeds[b]	Guatemala Belize Honduras El Salvador Costa Rica Panama	G	100 g/kg 150 g/kg	At transplanting	1.2 – 1.5 g ai/m^2	1	--
Maize	Australia	G	150 g/kg	In-furrow at planting	0.26-0.3	1	--
Maize	Brazil	G	50 g/kg; 150 g/kg	In-furrow at planting	1.95-2	1	--
Maize	Chile Nicaragua	G	100 g/kg	In-furrow or banded at planting	1.5-2	1	60
Maize	Guatemala Belize Honduras El Salvador	G	100 g/kg	In-furrow at planting	1-1.5	1	

Crop	Country	Form type	Conc.	Application method[a]	Rate kg ai/ha	Number of application.	PHI (days)
	Costa Rica Panama						
Maize	Mexico	G	50 g/kg; 150 g/kg	In-furrow at planting	1	1	60
Maize	USA	G	150 g/kg; 200 g/kg	Banded or in-furrow, or knifed-in	1.5	1	30 (forage)
Sorghum	Australia	G	150 g/kg	Ground at sowing	0.255 – 0.3	1	--
Sorghum	Guatemala Belize Honduras El Salvador Costa Rica Panama	G	100 g/kg	In-furrow at planting	1-1.5	1	
Sorghum	South Africa	G	150 g/kg	In-furrow at plating	4.95 g ai/100 m plant row	1	80
Sorghum	USA	G	200 g/kg	Knife-in, banded, at bedding or at planting	2.0	1	50 (forage) 100 (grain, fodder)
Sugar beet	Chile	G	100 g/kg	Banded; in-furrow/ground	1.5-2	1	60
Sugar beet	USA	G	150 g/kg; 200 g/kg	Banded, in-furrow, knifed-in at planting or post-emergence	2.2	1	110 (banded application) 150 (knifed-in application
Sweet corn	USA	G	150 g/kg; 200 g/kg	Banded or in-furrow, or knife-in	1.5	1	60 (for post-emergence use)

[a] All treatments are at-planting (at seeding or transplanting), except for established banana and coffee crops, and reflect outdoor or field use, with application to the soil.

[b] For seeder coffee (*almacigo* coffee in Costa Rica, Honduras, and Panama) the label also allows 1-1.5 g ai/m2, incorporated 8 days before watering or immediately after transplanting.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Supervised field trials were reported for numerous commodities. Where multiple samples were taken from a single plot or multiple analyses conducted on a single sample, the average value is reported. Where results from separate plots with distinguishing characteristics such as different formulations, varieties or treatment schedules were reported, results are listed for each plot. Results from trials at ± 30% of application rate or ± 30% of PHI were considered as complying with the GAP.

Most of the trials were conducted in the USA, where application rates were expressed in ounces active ingredient per 1000 ft. row (oz ai//1000 ft row). The labels provide limited tables to convert these to equivalent lbs ai/acre. When not specified in the study, or when the row width was not indicated, the rate in lbs/acre was estimated assuming 20-inch row spacing and applying the equation below. The resulting lbs/A is converted to kg/ha by multiplying by 1.12.

$$\text{Lbs/A} = [(\text{oz}/1000\ \text{ft row}) \times (43560\ \text{ft}^2/\text{acre})] \div [(1000\ \text{ft row}) \times (16\ \text{oz/lb}) \times (\text{row width (feet)})]$$

The following tables summarize information on residues resulting from supervised trials, where underlined residues are from trials according to GAP and were used to estimate maximum residue levels. Results have not been corrected for concurrent method recoveries unless indicated.

Classification	Table	Commodity	CCN
Assorted tropical and sub-tropical fruits, inedible peel	Table 33	Banana	FI 0327
Root and tuber vegetables	Table 34	Sugar beets	VR 0596
Fruiting vegetables other than cucurbits	Table 35	Corn-on-the cob; Corn kernels	VO 0447 VO 1275
Cereal grains	Table 36	Maize	GC 0645
	Table 37	Sorghum	GC 0651
Seeds for beverages and sweets	Table 38	Coffee beans	SB 0716
Fodder and forage of cereal grains	Table 39	Maize forage and fodder	AF 0645 AS 0645
	Table 40	Sorghum forage and fodder	AF 0651 AS 0651
Miscellaneous forage and fodder crops	Table 41	Sugar beet tops	AV 0596

Assorted Tropical and Subtropical Fruit – Inedible Peel

Banana (FI 0327)

Field residue trials were conducted on bananas in Australia, Costa Rica, Ecuador, Honduras, Panama, Philippines, and Mexico, the main banana producing areas of the world, between 1984 and 1997. For trials conducted in 1984-1990, terbufos (10% G or 15% G) was applied to the soil around the base of daughter or follower banana plants at a rate of 3–4 g ai/plant/ application. A total of 2-3 treatments with a maximum of 12 g ai/ plant were applied per year. Some trials exceeded these total number of treatments and/or maximum amount applied. In one trial, up to 20 g ai/plant/application was used. One sample from treated plots was collected at each sampling interval, stored frozen for a maximum of 11 months, and analyzed for total terbufos residues by GC/FPD method M-1340. The method has been successfully validated on whole bananas to a lower limit of 0.01 mg/kg (see Table 22).

In banana field trials from Mexico and Ecuador in 1997, a single broadcast soil application of terbufos (15% G) was directed to the base of the daughter plants at 4 or 8 g ai/mat (2× rate). Duplicate samples were collected at selected intervals, stored frozen (<-10°C) for a maximum of 11 months, and analyzed by method M-3072. Method M-3072 is essentially the same method as M-1340; however, it was validated at 0.002 mg/kg. The method has also undergone a successful independent laboratory validation (Study no. TE-244-057 by Zheng, S. and Gross, J. 1998, Table 22). Recoveries of terbufos-related residues fortified simultaneously in whole banana and pulp at 0.002 and 0.02 mg/kg were 66-121% (92 ± 11%, n=72), with only three recoveries outside the range of 70 to 120%. Details of the trials are presented in Table 33, where results of trials according to GAP are underlined

Table 33. Total terbufos residues in or on bananas.

Crop Report no. Location/Year/var.	Application			PHI	Residues (mg/kg)			Reference Number
	Form.	Max no. of Appl. or g ai/yr	Rate g ai/plant	days	Whole	Peel	Pulp	
Central American GAP (Guatemala, Belize, Dominican Rep, Honduras, Nicaragua, Costa Rica, Panama): 10G or 15G, at a rate of 3-4 g ai/plant around follower plant at max of 2-3 applications or 12 g ai/ plant /year								
C-2793 Limon, Costa Rica, 1986 Grand Naime	10G	1	9	1 2 3	<u>< 0.01</u> < 0.01 < 0.01			TE-714-001
C-2789 Coyoles, Honduras,1986 Giant Cavendish	10G	1	2	1 2 4 8 14 28	<u>< 0.01</u> < 0.01 < 0.01 < 0.01 < 0.01 < 0.01			TE-714-002
		1	6	1 2 4	<u>< 0.01</u> < 0.01 < 0.01			

Crop Report no. Location/Year/var.	Application			PHI	Residues (mg/kg)			Reference Number
	Form.	Max no. of Appl. or g ai/yr	Rate g ai/plant	days	Whole	Peel	Pulp	
				8	< 0.01			
				14	< 0.01			
				28	< 0.01			
C-2706 Coyoles, Honduras, 1986 Giant Cavendish	10G	1	10	14	0.02	0.04	0.02	TE-714-003
				33	0.02	0.03	0.01	
				47	0.02	0.02	0.02	
				89	0.01	0.01	< 0.01	
C-2705 Limon, Costa Rica, 1986 Giant Cavendish	10G	12 (41 g ai/yr)	2-4	20	< 0.01			TE-714-004
				32	< 0.01			
				43	< 0.01			
				46	< 0.01			
				88	< 0.01			
				90	< 0.01			
C-2704 Rio Frio, Costa Rica, 1985 Giant Cavendish	10G	1	20	27	0.02	0.03	< 0.01	TE-714-005
				35	0.03	0.05	0.02	
				48	0.02	0.05	0.01	
				95	0.01	0.02	< 0.01	
C-2674 Limon, Costa Rica, 1984 Giant Cavendish	10G	8 (32 g ai/yr)	4	18	0.02	0.02	< 0.01	TE-714-006
				44	0.01	0.02	0.01	
				104	< 0.01			
		9 (36 g ai/yr)	4	57	< 0.01			
C-2622.1 Limon, Costa Rica, 1984 Giant Cavendish	10G	6 (24 g ai/yr)	4	18	0.02	0.02	< 0.01	TE-714-007
				44	0.01	0.02	0.01	
		7 (28 g ai/yr	4	60	< 0.01			
		7 (28 g ai/yr)	4	59	< 0.01			
		4 (16 g ai/yr)	4	43	< 0.01			
C-2621 Limon, Costa Rica, 1984 Giant Cavendish	10G	1	3	89	0.02	0.02	0.02	TE-714-008
					0.01	0.01	< 0.01	
				114	< 0.01			
C-2493 Limon, Costa Rica, 1984 Giant Cavendish	10G	4 (16 g ai/yr)	4	4	< 0.01			TE-714-009
				43	< 0.01			
C-2438 Limon, Costa Rica, 1983 Valery	10G	1	3	14	0.02			TE-714-010
				28	0.01			
				60	< 0.01			
		1	6	14	0.02			
				28	< 0.01			
				60	< 0.01			
C-2792 Davila, Panama, 1986 Grand Naime	10G	1	9	1	< 0.01			TE-714-011
				2	< 0.01			
				4	< 0.01			
				7	< 0.01			
				13	< 0.01			
C-2494 Guapiles, Costa Rica, 1984 Grande Naime	10G	4 (14 g ai/yr)	4 (2x)+ 3 (2x)	95	< 0.01			TE-714-012
C-2622 Limon, Costa Rica, 1985 Giant Cavendish	10G	6 (24 g ai/yr)	4	18	0.02	0.02	< 0.01	TE-714-013
				44	0.01	0.02	0.01	
		6 (24 g ai/yr)		105	< 0.01			
		7	4	59	< 0.01			

Crop Report no. Location/Year/var.	Application			PHI	Residues (mg/kg)			Reference Number
	Form.	Max no. of Appl. or g ai/yr	Rate g ai/plant	days	Whole	Peel	Pulp	
		(28 g ai/yr)						
C-3136 Guapiles, Costa Rica, 1987 Giant Cavendish	10G	1	2.5	33 68 89	< 0.01 < 0.01 < 0.01			TE-714-015
C-3614 Coyoles, Honduras, 1989 Giant Cavendish	10G	1	8	7 14 21 28 42 56 70 84	0.01 0.02 0.03 0.02 < 0.01 < 0.01 < 0.01 < 0.01			TE-714-017
Australian GAP: 150 g /kg G, at a rate of 2-3 g ai/plant applied around follower plant at max of 4 applications or 12 g ai/year								
TRR-93-005, 006, 007, 008 Palmerston, Queensland, Australia,1992 Giant Cavendish	15G	3 (9 g ai/yr)	3	1 3 7 14 21 28 56		< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	TE-714-018, 019, 020, 021
TRR-93-005, 006, 007, 008 Palmerston, Queensland, Australia,1992 Giant Cavendish (cont'd)		3 (18 g ai/yr)	6	1 3 7 14 21 28 56		< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	TE-714-018, 019, 020, 021
Philippines GAP: 100 g/kg G at the rate of 2 g ai/plant and a maximum of 4 total application or 8 g ai/year								
TTR-86-012 Philippines 1986 Giant Cavendish	10G	1	2	16 30 58 86	< 0.01 < 0.01 < 0.01 < 0.01			TE-714-014
	10G	2 (4 g ai/yr)	2	1 3 5 7	< 0.01 < 0.01 < 0.01 < 0.01			
TTR-87-025 Philippines, 1987 Giant Cavendish	10G	1	3	9 16 23 30 58 87 113 156 176	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	TE-714-022
		2 (6 g ai/yr)	3	23 58 87 114 163 191	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	

Crop Report no. Location/Year/var.	Application			PHI	Residues (mg/kg)			Reference Number
	Form.	Max no. of Appl. or g ai/yr	Rate g ai/plant	days	Whole	Peel	Pulp	
		3 (9 g ai/yr)	3	25 74 102	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	
TTR-86-011 Twin Rivers Philippines, 1986 Giant Cavendish	10G	1	3	16 30 58 86	< 0.01 < 0.01 < 0.01 < 0.01			TE-714-023
		1	6	16 30 58 86	< 0.01 < 0.01 < 0.01 < 0.01			
Ecuador (Central America GAP): 10G or 15G, at a rate of 3-4 g ai/plant around follower plant at max of 2-3 applications or 12 g ai/ plant /year								
RES 97-062 La Peana Ecuador, 1997 Valery	15G	1	4	2 4 7 14 28 45	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002		< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	TE-714-024
		1	8	2 4 7 14 28 45	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002		< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	
RES 97-063 Rio Verde, Ecuador, 1997 Cavendish	15G	1	4	2 4 7 14 28 45	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002		< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	TE-714-025
		1	8	2 4 7 14 28 45	< 0.002 < 0.002 < 0.002 0.003 < 0.002 < 0.002		< 0.002 < 0.002 < 0.002 0.003 < 0.002 < 0.002	
Mexican GAP: 150 g/kg G at the rate of 3 g ai/plant and a maximum of 2-3 applications or 9 g ai/year								
RES 97-064 Teapa, Mexico, 1997 Enano Gigante	15G	1	4	2 4 7 14 30 45	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002		< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	TE-714-026
		1	8	2 4 7 14 30 45	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002		< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	

Root and Tuber Vegetables

Sugar beets (VR 0596)

Field trials were conducted in the USA and Canada during 1972-1975 in which terbufos (15% G) was applied in-furrow or banded at 1.1–2.5 kg ai/ha (approximating GAP) and also at exaggerated rates

(4.0–12.3 kg ai/ha). Several tests also depicted sequential at-planting and post-emergence banded applications, typically reflecting exaggerated rates. Treated samples were collected at each sampling interval, stored frozen for a maximum of 22 months, and analyzed for total terbufos residues by method M-395. This method was successfully validated on roots at 0.05 mg/kg (Table 22).

Seven additional sugar beet trials were conducted in the USA during the 1986 and 1989 growing seasons. In 1986, terbufos (15% G) was applied at planting (banded, knifed-in, or in-furrow) at 2.2 kg ai/ha. A single treated root sample was collected at each interval, stored frozen for a maximum of 10 months and analyzed for total terbufos residues by method M-1747. This method has been validated on roots at 0.01 mg/kg (Table 22). Concurrent recoveries of terbufos-related residues fortified in roots at 0.01 mg/kg were 82–108% (92 ± 14%, n=3).

In the trials conducted in 1989, terbufos (15%G) was knifed in as a band at planting at 4.9 kg ai/ha, in excess of the current GAP. A single treated sample was harvested by hand at maturity, 150–180 days after treatment. The samples were placed in frozen storage for a maximum of 6 months at about -17°C, and were analyzed for total residues of terbufos using method M-395. As noted above, method M-395 has been validated on roots at 0.05 mg/kg (Table 22). Concurrent recoveries of terbufos-related residues (parent and CL 94302) fortified in roots at 0.1 mg/kg were 64–126% (92 ± 24%, n=6).

In more recent field trials from the USA (1994), terbufos (15%G) was applied as a band over the row to sugar beets 15–58 cm tall at 2.2 to 2.4 or 4.4–4.9 kg ai/ha. The lower rate reflects the maximum GAP rate. Sugar beet root samples were collected at each sampling interval, stored frozen (about -8°C) for a maximum of 8 months, and analyzed by method M-2457. The validated sensitivity for the method was 0.01 mg/kg for roots. Concurrent recoveries of terbufos-related residues (parent, CL94221, CL94301, CL94302, CL94320, CL94365) fortified simultaneously in roots at 0.01 to 0.60 mg/kg were 78–130% (93 ± 12%, n=18). Additional data from a method validation study on method M-2457 are presented in Table 22. The residue results for sugar beet roots are presented in Table 34.

Table 34. Terbufos residues in sugar beet roots

Report No. Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type	no. Appl.	Rate kg/ai/ha	Rate oz ai/1000 ft			
GAP, USA: 15G (150g/kg) at the rate of 0.6 – 1.2 oz ai/1000 ft row or an annual maximum of 2.2 kg ai/ha. PHI is 110 days, except for 150 days for knifed-in applications.								
C-3064 TE-724-064 Colorado, USA, 1986 Mono-Hy A4	15G	In furrow	1	2.2	1.2	162	< 0.01	TE-724-064
		Knifed-in	1	4.9	3	162	< 0.01	
		Banded	1	2.2	1.2	162	< 0.01	
		Banded Post	1	2.2	1.2	134	< 0.01	
C-3065 TE-724-065 ND, USA, 1986 Bush Johnson 19	15G	In furrow	1	2.2	1.2	141	< 0.01	TE-724-065
		Knifed in	1	4.9	3	141	0.02	
		Banded	1	2.2	1.2	141	< 0.01	
		Banded-Post	1	2.2	1.2	91	< 0.01	
C-3066 TE-724-066 Idaho, USA, 1986 Betaseed 8654	15G	In furrow	1	2.2	1.2	148	< 0.01	TE-724-066
		Knifed in	1	4.9	3	148	0.01	
		Banded	1	2.2	1.2	148	< 0.01	
		Banded-Post	1	2.2	1.2	115	< 0.01	
C-3067 Minnesota USA, 1986 Bush Johnson 19	15G	In furrow	1	2.2	1.2	139	0.01	TE-724-067
		Knifed in	1	4.9	3	139	0.03	
		Banded	1	2.2	1.2	139	0.01	
		Banded-Post	1	2.2	1.2	120	< 0.01	
C-3366 ND, USA, 1989 UltraMono	15G	Knifed-in	1	5.0		150	< 0.05	TE-724-068
C-3367 Nebraska, USA,1989 ACH 164	15G	Knifed-in	1	5.0		180	< 0.05	TE-724-069

Report No. Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type	no. Appl.	Rate kg/ai/ha	Rate oz ai/1000 ft			
GAP, USA: 15G (150g/kg) at the rate of 0.6 – 1.2 oz ai/1000 ft row or an annual maximum of 2.2 kg ai/ha. PHI is 110 days, except for 150 days for knifed-in applications.								
C-3368 Idaho, USA,1989 WS88	15G	Knifed-in	1	5.0		153	< 0.05	TE-724-070
C-3369 California, USA 1989, Z-1	15G	Knifed-in	1	5.0		170	< 0.05	TE-724-071
C-667 Colorado, USA 1974, Mono-HY-1	15G	In-furrow Banded	1 1	2 4		190 190	< 0.05 < 0.05	TE-724-004
C-666 Wyoming, USA 1975, Mono-HY	15G	In-furrow	1 1 1	1 2 12		142 142 142	< 0.05 < 0.05 < 0.05	TE-724-005
		Post	1 1 1	1 2 12		116 116 116	< 0.05 < 0.05 < 0.05	
		Banded	1 1 1	1 2 12		142 142 142	< 0.05 < 0.05 < 0.05	
		Post	1 1 1	1 2 12		116 116 116	< 0.05 < 0.05 < 0.05	
C-665 Idaho, USA 1973	15G	Banded	1	2.2	1.3	152	< 0.05	TE-724-006
C-664 Idaho, USA, 1974 AH-A1	15G	Banded	1 1	1.1 2.2		168 168	< 0.05 < 0.05	TE-724-007
C-964 Manitoba, Canada 1975	15G	In-furrow	1	1.12		117	< 0.05	TE-724-012
C-916 Manitoba, Canada 1974	15G	In-furrow	1	1.12		117	< 0.05	TE-724-013
C-694 North Dakota, USA 1973/ American Crystal Hybrid #1	15G	Banded	1 1 1	2.2 4.5 9.0	1.35 2.7 5.4	155 155 155	< 0.05 < 0.05 < 0.05	TE-724-014
		In-furrow	1 1	2.2 4.5	1.35 2.7	155 155	< 0.05 < 0.05	
		Post	1 1 1	2.2 4.5 9.0	1.35 2.7 5.4	124 124 124	< 0.05 < 0.05 < 0.05	
		Banded + Post	2 2	4.4+2.2 4.4+4.4		124 124	< 0.05 < 0.05	
C-695 North Dakota, USA, 1974/ American . Crystal Hybrid #2B	15G	Banded	1 1 1	2.5 5.0 12.3	1.35 2.7 6.75	129 129 129	< 0.05 < 0.05 < 0.05	TE-724-016
		In-furrow	1 1 1	2.5 5.0 12.3	1.35 2.7 6.75	129 129 129	< 0.05 < 0.05 < 0.05	
		Banded + Post	2 2 2	2.5+2.5 2.5+4.9 2.5+12.3		118 118 118	< 0.05 < 0.05 < 0.05	
C-693 Michigan, USA, 1974/	15G	In-furrow	1 1	1.8 3.6	1.35 2.7	174 174	< 0.05 0.11	TE-724-017

Report No. Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type	no. Appl.	Rate kg/ai/ha	Rate oz ai/1000 ft			
GAP, USA: 15G (150g/kg) at the rate of 0.6 – 1.2 oz ai/1000 ft row or an annual maximum of 2.2 kg ai/ha. PHI is 110 days, except for 150 days for knifed-in applications.								
Monitor common			1	8.5	5.4	174	< 0.05	
		Banded	1	1.8	1.35	174	< 0.05	
			1	3.6	2.7	174	< 0.05	
			1	8.5	5.4	174	< 0.05	
C-668	15G	Banded	1	2.2	1.36	157	< 0.05	TE-724-018
Colorado, USA, 1973/			1	4.5	2.7	157	< 0.05	
Mono-Hy-1		Post	1	2.2	1.36	126	≤ 0.05	
			1	4.5	2.7	126	< 0.05	
			1	9.0	5.4	126	< 0.05	
		Banded + Post	2	4.5+2.2		126	< 0.05	
			2	4.5+4.5		126	< 0.05	
C-914	15G	Banded	1	1.1		114	< 0.05	TE-724-029
Manitoba, Canada, 1973								
C-917	15G	In-Furrow	1	1.1		126	< 0.05	TE-724-030
Manitoba, Canada			1	1.1		133	< 0.05	
1971								
C-656	15G	Banded	1	1.1		135	< 0.05	TE-724-048
North Dakota,			1	2.2		135	≤ 0.05	
USA, 1972		Post	1	1.1		119	< 0.05	
			1	2.2		119	≤ 0.05	
C-657	15G	Banded	1	0.56		156	< 0.05	TE-724-049
Wyoming, USA, 1972			1	1.1		156	< 0.05	
Mono-Hi			1	6.7		156	< 0.05	
C-696	15G	Banded	1	2.2		102	≤ 0.05	TE-724-050
Minnesota, USA, 1973			1	4.4		102	< 0.05	
American Crystal			1	8.9		102	< 0.05	
Hybrid #13			1	2.2		138	≤ 0.05	
			1	4.4		138	< 0.05	
			1	8.9		138	< 0.05	
		Post	1	2.2		73	≤ 0.05	
			1	4.4		73	0.11	
			1	8.9		73	0.28	
			1	2.2		109	≤ 0.05	
			1	4.4		109	< 0.05	
			1	8.9		109	0.06	
		Banded+Post	2	4.5+2.2		109	< 0.05	
			2	4.5+4.5		109	< 0.05	
RES-95-046	15G	Banded,	1	2.24	1.2	50	< 0.01	TE-724-035
Idaho, USA, 1994		Post				70	< 0.01	
HM-WS91						90	≤ 0.01	
		Banded,	1	4.9	2.7	50	0.04	
		Post				70	0.02	
						90	0.02	
RES-95-039	15G	Banded,	1	2.2	1.2	50	0.02	TE-724-036
Michigan, USA, 1993		Post				70	< 0.01	
H23						91	≤ 0.01	
		Banded,	1	4.4	2.4	50	0.06	
		Post				70	0.02	
						91	< 0.01	
RES-95-040	15G	Banded,	1	2.2	1.2	50	0.02	TE-724-037
Nebraska, USA, 1994		Post				70	< 0.01	
HM 1605						90	≤ 0.01	
				4.4	2.4	50	0.01	
						70	0.01	
						90	< 0.01	

Report No. Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type	no. Appl.	Rate kg/ai/ha	Rate oz ai/1000 ft			
GAP, USA: 15G (150g/kg) at the rate of 0.6 – 1.2 oz ai/1000 ft row or an annual maximum of 2.2 kg ai/ha. PHI is 110 days, except for 150 days for knifed-in applications.								
RES-95-045 North Dakota, USA, 1994 ACH 192	15G	Banded, Post	1	2.4	1.3	50 70 90	< 0.01 < 0.01 < 0.01	TE-724-038
				4.6	2.5	50 70 90	0.03 0.01 < 0.01	
GAP, USA: 150 g/kg at the rate of 0.6 – 1.2 oz ai/1000 ft row or an annual maximum of 2.2 kg ai/ha. PHI is 110 days, except for 150 days for knifed-in applications.								
RES-95-047 Minnesota, USA, 1994 ACH 192	15G	Banded, Post	1	2.2	1.2	50 70 90	0.03 < 0.01 < 0.01	TE-724-039
				4.4	2.4	50 70 90	0.04 0.01 < 0.01	

Fruiting vegetables other than cucurbits

Sweet Corn, Corn-on-the-Cob (VO 0447) and Kernels (VO 1275)

In trials in the USA in 1972 - 1974, terbufos granules were applied in the furrow or in a band at the time of planting at rates of 1.1 to 9.0 kg ai/ha. In 1986 terbufos granules were applied to the soil at planting in furrow or in a band, at post-emergence or at cultivation at a combined rate of about 6 kg ai/ha. One sample was collected at each sampling interval, stored frozen for a maximum of 9 months, and analyzed by methods M-336 (1973-1974) and M-1754 (1986). The methods have been validated on sweet corn (kernels + cob) at 0.05 and 0.01 mg/kg, respectively (Table 22).

The results of the field trials on sweet corn are shown in Table 35. Total terbufos residues in sweet corn from trials according to the GAP (grain/ kernels, ears, or kernels + cob) were non-detectable (< 0.01 or < 0.05 mg/kg,). Residues from trials at higher application rates were also non-detectable, except in three samples of kernel + cob in which residues were detected at a level of 0.01 mg/kg.

Table 35. Terbufos residues in sweet corn (corn-on the-cob).

Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type	no. Appl.	Rate kg/ai/ha	Rate oz ai/1000 ft			
GAP USA: 15G formulation at the rate of 1.2 oz ai/1000 ft row or a maximum of 1.5 kg ai/ha applied once at planting, post-emergent, or at cultivation. For post-emergent applications, the PHI is 30 days for forage, and 60 days for corn-on-the cob								
C-417 Illinois, USA, 1972	15G	Banded	1 1 1	1.1 2.2 4.5		88 88 88	< 0.05 < 0.05 < 0.05	TE-723-002
California, USA, 1972	15G	Banded	1 1	1.1 2.2		90 90	< 0.05 < 0.05	
Illinois, USA, 1973	15G	In-furrow	1 1	1.1 1.1		78 76	< 0.05 < 0.05	
Oregon, USA, 1973	15G	In-furrow	1 1	2.2 4.5		111 111	< 0.05 < 0.05	
	15G	Banded	1	2.2 4.5		111 111	< 0.05 < 0.05	
Wisconsin, USA, 1973	15G	In-furrow Banded	1 1	1.1 1.1		71 71	< 0.05 < 0.05	

Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type	no. Appl.	Rate kg/ai/ha	Rate oz ai/1000 ft			
GAP USA: 15G formulation at the rate of 1.2 oz ai/1000 ft row or a maximum of 1.5 kg ai/ha applied once at planting, post-emergent, or at cultivation. For post-emergent applications, the PHI is 30 days for forage, and 60 days for corn-on-the cob								
C--642 Illinois, USA, 1974 Super sweet	15G	In-furrow	1	1.2	1.2	105	< 0.05	TE-723-003
C--635 Iowa, USA, 1974 Silver Queen	15G	Banded	1	1.5		95	< 0.05	TE-723-005
			1	2.9		95	< 0.05	
			1	5.8		95	< 0.05	
	15G	In-furrow	1	1.5		95	< 0.05	
			1	2.9		95	< 0.05	
			1	5.8		95	< 0.05	
C-632 Florida, USA, 1974 Tobelle	15G	In-furrow	1	2.2		75	< 0.05	TE-723-014
				4.5		75	< 0.05	
				9.0		75	< 0.05	
C-425 Colorado, USA, 1973	15G	Banded	1	1.1		113	< 0.05	TE-730-002
C-639 Minnesota, USA, 1974	15G	In-furrow	1	1.1	1.2	69	< 0.05	TE-730-003
			1	2.2	2.4	69	< 0.05	
			1	4.5	4.8	69	< 0.05	
	15G	Banded	1	1.1	1.2	69	< 0.05	
			1	2.2	2.4	69	< 0.05	
			1	4.5	4.8	69	< 0.05	
C-638 Virginia, USA, 1974 Silver Queen	15G	In-furrow	1	1.1	1.2	85	< 0.05	TE-730-005
			1	2.2	2.4	85	< 0.05	
	15G	Banded	1	1.1	1.2	85	< 0.05	
			1	2.2	2.4	85	< 0.05	
C-3096 Wisconsin, USA, 1986 Commander	15G	In-furrow	2	4 + 2	2.4 + 1.2	68	< 0.01	TE-723-004
			2	2 + 4	1.2 + 2.4	68	< 0.01	
C--3109 Florida, USA, 1986 Silver Queen	15G	In-furrow +	2	4 + 2	2.4 + 1.2	41	< 0.01	TE-723-006
	15G	POST	2	2 + 4	1.2 + 2.4	41	0.01	
						41	0.01	
		In-furrow +	2	4 +2	2.4 + 1.2	41	0.01	
		Post	2	2 + 4	1.2 + 2.4	41	< 0.01	
						41	< 0.01	
C-3093 New York, USA, 1986, Jubilee	15G	In-furrow	2	4 + 2	2.4 + 1.2	72	< 0.01	TE-723-036
	15G	In-furrow	2	2 + 4	1.2 + 2.4	72	< 0.01	

Cereal Grains

Maize (GC 0645)

A number of supervised trials on maize were conducted from 1972–1996 in the USA. In trials conducted from 1972–1974 and 1981–1986, terbufos granules were applied to the soil at planting, either in furrow or as a band, at the rate of 1.1 to 1.8 kg ai/ha. In some trials, additional plots were treated with terbufos at rates up to 5 times the recommended label rates. In one trial, a treatment at 11 kg ai/ha was applied. In trials conducted from 1990–1996 terbufos granules were applied post-emergent at the recommended rate of 1.5 kg ai/ha as well as at higher rates up to five times the recommended application rates. Treated samples of maize grain were collected at intervals, and the samples were stored frozen (-10°C), for a maximum of 8 months, prior to analysis.

Residues of terbufos-related compounds were determined either by method M-336 or M-1754, which have been validated to lower limits of 0.05 and 0.01 mg/kg, respectively (see Table 22). Additional method recoveries of terbufos residues from studies conducted during 1990-1996 further demonstrated the suitability of method M-1754. In the trials performed in 1995-1996, concurrent recoveries of terbufos-related residues in maize grain fortified at 0.01-0.4 mg/kg were 65–114% (94 ± 12%, n=22). Additionally, concurrent recoveries of parent, CL94301, CL94302, CL94320, CL94221, and CL94365 fortified in grain at 0.01 or 0.4 mg/kg were 82–92% (88 ± 4%, n=4).

Table 36 summarize results of trials on maize grain, with residues according to GAP, underlined. Treatments according to the GAP resulted in residues below the limit of determination (< 0.01 or < 0.05 mg/kg, depending on the method used).

Table 36. Terbufos residues in maize grain.

Location/Year/ Variety	APPLICATION					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type[a]	no. Appl.	Rate kg/ai/ha	Rate oz ai/1000 ft			
GAP USA: 15G or 20G formulation at the rate of 1.2 oz ai/1000 ft row or a maximum of 1.5 kg ai/ha applied once at planting, post-emergent, or at cultivation. A PHI of 30 days is required for forage if applied post-emergent.								
C-2082 North Carolina, USA 1981 Pioneer 3184	15G 20G 20G 20G	Banded Banded In-furrow In-furrow	1 1 1 1	2.2 2.2 2.2 2.2		148 148 92 148	< 0.05 < 0.05 < 0.05 < 0.05	TE-723-026
C-2215 Nebraska, USA, 1982 Stauffer 6595	15G 15G	Infurrow/ Banded	2 2	2.2 + 2.2 4.4 + 2.2		105 105	< 0.05 < 0.05	TE-730-018
C-3038 Illinois, USA, 1986 Hybrid Funks G4507	15G 20G	Banded Banded	1 1	2.24 2.24	2.4 2.4	181 181	< 0.01 < 0.01	TE-730-010
C-3037 Nebraska, USA, 1986 Pioneer 337	15G 20G	Banded Banded	1 1	1.12 1.12	1.2 1.2	147 147	<u>< 0.01</u> <u>< 0.01</u>	TE-730-011
C-3095 Minnesota, USA, 1986/ Cargill 809	15G 15G	Banded+POST Banded+POST	2 2	4.4+ 2.2 2.2 + 1.2	2.2 + 1.2 4.4 + 2.2	120 120	< 0.01 < 0.01	TE-730-012
RES-95-059 Wisconsin, USA, 1994 Pioneer Hybrid 3861	15G	Banded, POST	1	1.5	1.2	60 80	<u>< 0.01</u> < 0.01	TE-730-028
	15G	Banded, POST	1	3	2.4	60 80	< 0.01 < 0.01	
RES-95-058 Michigan, USA, 1994 Pioneer 3921	15G	Banded, POST	1	1.5	1.2	59 80	<u>< 0.01</u> < 0.01	TE-730-029
	15G	Banded, POST	1	3	2.5	60 80	< 0.01 < 0.01	
RES-96-021 Iowa, USA, 1995 434 VARIETY	15G	Banded, POST	1	1.5	1.2	56 80	<u>< 0.01</u> < 0.01	TE-730-030
	15G	Banded, POST	1	3	2.4	60	< 0.01	

Location/Year/ Variety	APPLICATION					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type[a]	no. Appl.	Rate kg/ai/ha	Rate oz ai/1000 ft			
GAP USA: 15G or 20G formulation at the rate of 1.2 oz ai/1000 ft row or a maximum of 1.5 kg ai/ha applied once at planting, post-emergent, or at cultivation. A PHI of 30 days is required for forage if applied post-emergent.								
						80	< 0.01	
RES-96-022 Nebraska, USA, 1995	15G	Banded, POST	1	1.5	1.2	64 79	≤ 0.01 < 0.01	TE-730-031
Northrup King N3803	15G	Banded, POST	1	3	2.4	64 79	< 0.01 < 0.01	
C-3566 Iowa, USA, 1990 Circle seed #7111	15G 15G	Banded, POST Banded, POST	1 1	1.5 7.3	1.2 5.9	132 113	≤ 0.01 < 0.01	TE-723-032
C-3567 Iowa, USA, 1990 DeKalb DK535	15G 15G	Banded, POST Banded, POST	1 1	1.5 7.3	1.2 5.9	119 119	≤ 0.01 < 0.01	TE-723-033
C-3568 Illinois, USA, 1990 Dockendorf 7670	15G 15G	Banded, POST Banded, POST	1 1	1.5 7.3	1.2 5.9	109 109	≤ 0.01 < 0.01	TE-723-034
C-3569 Illinois, USA, 1990 Pioneer 3615	15G 15G	Banded, POST Banded, POST	1 1	1.5 7.3	1.2 5.9	109 109	≤ 0.01 < 0.01	TE-723-035
RES-96-084 Illinois, USA, 1996	15G	Banded, POST	1	1.5	1.2	60 80	≤ 0.01 < 0.01	TE-730-051
Pioneer 3751	15G	Banded, POST	1	3	2.7	60 80	< 0.01 < 0.01	
C-1129 Maryland, USA, 1974 DeKalb 264	15G	In-furrow	1 1 1	1.3 2.6 5.2		134 134 134	≤ 0.05 < 0.05 < 0.05	TE-730-008
C-416: Illinois, USA, 1973	15G	In-furrow	1	1.12		124	≤ 0.05	TE-730-016
Nebraska, USA,1973	15G	In-furrow	1 1 1	1.12 2.24 5.6		168 168 168	≤ 0.05 < 0.05 < 0.05	
ND, USA, 1973	15G	In-furrow	1 1 1	1.12 1.7 2.2		159 159 159	< 0.05 ≤ 0.05 < 0.05	
Nebraska, USA, 1973	15G	In-furrow	1 1 1	1.12 2.24 5.6		162 162 162	≤ 0.05 < 0.05 < 0.05	
Iowa, USA, 1973	15G	In-furrow	1 1 1 1	1.12 2.24 5.6 11.2		135 135 135 135	≤ 0.05 < 0.05 < 0.05 < 0.05	
C-415 Indiana, USA, 1972	15G	In-furrow	1	1.12 1.7		146 146	≤ 0.05 ≤ 0.05	TE-730-017
South Dakota, USA, 1972	15G	In-furrow	1	1.1		131	≤ 0.05	
Missouri, USA, 1972	15G	In-furrow	1	1.7		184	≤ 0.05	
Nebraska, USA, 1972	15G	In-furrow	1	1.12		168	≤ 0.05	
NC, USA, 1972	15G	In-furrow	1 1	1.12 1.12		143 148	≤ 0.05 ≤ 0.05	
ND, USA, 1972	15G	In-furrow	1 1	1.12 2.24		159 159	≤ 0.05 < 0.05	
C-631 Minnesota, USA, 1974	15G	In-furrow	1	1.12		115	≤ 0.05	TE-723-013
C-636	15G	In-furrow	1	1.1	1.2	143	≤ 0.05	TE-723-016

Location/Year/ Variety	APPLICATION					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type[a]	no. Appl.	Rate kg/ai/ha	Rate oz ai/1000 ft			
GAP USA: 15G or 20G formulation at the rate of 1.2 oz ai/1000 ft row or a maximum of 1.5 kg ai/ha applied once at planting, post-emergent, or at cultivation. A PHI of 30 days is required for forage if applied post-emergent.								
Minnesota, USA, 1974			1 1	2.2 4.5	2.4 4.8	143 143	< 0.05 < 0.05	
C-637 ND, USA, 1974 NK 420	15G	In-furrow	1 1 1 1	1.2 1.8 2.5 4.9	1.2 1.8 2.4 4.8	134 134 134 134	< 0.05 <u>< 0.05</u> < 0.05 < 0.05	TE-723-017
C-640 Michigan, USA, 1974	15G	In-furrow	1 1 1	1.1 2.2 4.5	1.2 2.4 4.8	151 151 151	<u>< 0.05</u> < 0.05 < 0.05	TE-723-018
C-641 Maryland, USA, 1974 DeKalb 264	15G	In-furrow	1 1 1	1.5 2.9 5.8	1.2 2.4 4.8	130 130 130	<u>< 0.05</u> < 0.05 < 0.05	TE-723-019
C-644 Colorado, USA, 1974 NC +	15G	In-furrow	1 1 1 1	1.1 1.5 2.9 5.8	0.9 1.2 2.4 4.8	179 179 179 179	< 0.05 <u>< 0.05</u> < 0.05 < 0.05	TE-723-021
C-647 Kentucky, USA, 1973 Pioneer 3369A	15G	In-furrow	1 1	0.84 1.1	0.9 1.2	142 142	< 0.05 <u>< 0.05</u>	TE-723-022

Sorghum (GC 0651)

Supervised trials were conducted from 1978–1996 in major sorghum-growing areas in the USA. In trials conducted in 1996, terbufos granules were applied to the soil in a band during the vegetative stages of the plant, at the rate of 2.0 – 2.2 kg ai/ha. Grain samples (approximately 1 kg) were collected 88 to 90 days after the last application, frozen (-10°C) and stored up to a maximum of 8 months until analysis. A freezer storage stability study on the related crop, corn or maize grain, showed that residues of terbufos-related compounds are stable in corn grain up to at least 24 months when stored at approximately -10°C (Table 29, study no. TE-326-014 by Dixon, C 1990).

Terbufos residues were determined by Method M-1754, using a gas chromatograph equipped with a flame photometric detector. The validated sensitivity of the method was 0.01 mg/kg for sorghum grain (see Table 22). The average concurrent recovery from field samples was 105%.

In trials conducted in 1978-1979 and 1986-1991, terbufos granules were applied to the soil at planting, either in-furrow, knifed-in, or in a band, at rates ranging from 2.0 to 4.3 kg ai/ha. Samples of sorghum grain were harvested from 95 to 150 days after the application, immediately frozen (-10°C), and stored until analysis. Total terbufos-related residues were determined by Method 995, which had previously been validated (Table 22). The validated sensitivity for the method is 0.05 mg/kg for sorghum grain.

Results for all the trials are summarized in Table 37, with residue values from trials according to the GAP, underlined. Treatments according to the GAP resulted in residues below the limit of determination (< 0.01 or < 0.05 mg/kg, depending on the method used).

Table 37. Terbufos residues in sorghum grain

Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type	no. Appl.	Rate kg/ai/ha	Rate oz ai/1000 ft			
GAP USA: 15G formulation at the rate of 1.1 oz ai/1000 ft row or a maximum of 2.0 kg ai/ha applied once and a PHI of 50 days for forage, and 100 days for grain and fodder.								
RES-96-082 Missouri, USA, 1996 KS 714Y	15G	Banded (POST)	1	2.2	1.2	72 90	< 0.01 (2) < 0.01 (2)	TE-730-057
RES-96-078 Texas, USA, 1996 F-200	15G	Banded (POST)	1	2.2	1.2	59 76 90	< 0.01 (2) < 0.01 (2) < 0.01 (2)	TE-730-053
RES-96-077 Texas, USA 1996 F 200	15G	Banded (POST)	1	2.2	1.2	58 73 88	< 0.01 (2) < 0.01 (2) < 0.01 (2)	TE-730-052
RES-96-081 Illinois, USA, 1996 Northrup King 1210	15G	Banded (POST)	1	2.2	1.2	60 75 90	< 0.01 (2) < 0.01 (2) < 0.01 (2)	TE-730-056
RES-96-079 Louisiana, USA, 1996 DeKalb 37	15G	Banded (POST)	1	2.0	1.2	60 75 90	< 0.01 (2) < 0.01 (2) < 0.01 (2)	TE-730-054
RES-96-080 Kansas, USA, 1996 6R55E	15G	Banded	1	2.1	1.2	62 76 90	< 0.01 (2) < 0.01 (2) < 0.01 (2)	TE-730-055
C-1626 Kansas, USA, 1978	15G	Banded Banded In-furrow In-furrow	1 1 1 1	2.0 4.0 2.0 4.0	1.2 2.4 1.2 2.4	95 95 95 95	< 0.05 < 0.05 < 0.05 < 0.05	TE-730-039
C-1742 Texas, USA, 1978 Mitchell Standking Y	15G	Banded	1	4.0	2.4	150	< 0.05	TE-730-040
C-1752 Texas, USA, 1978 Harpool 8409	15G	In-furrow Banded	1 1	4.0 4.0	2.4 2.4	104 104	< 0.05 < 0.05	TE-730-041
C-1773 Oklahoma, USA 1979 Rawhide	15G	In-furrow Banded	1 1	4.0 4.0	2.4 2.4	117 117	< 0.05 < 0.05	TE---730-042
C-1776 Colorado, USA, 1978 DeKalb A28+	15G	Banded In-furrow	1 1	4.0 4.0	2.4 2.4	175 175	< 0.05 < 0.05	TE-730-043
C-3374 Kansas, USA, 1989 FSIA +	15G	Knifed-in	1	4.3		132	< 0.05	TE-730-046
C-3375 Nebraska, USA, 1989 NC + 271	15G	Knifed-in	1	4.3		133	< 0.05	TE-730-047
C-3376 Missouri, USA, 1989 5511	15G	Knifed-in	1	4.3		139	< 0.05	TE-730-048
C-3851 Texas, USA,1991 F-270G	15G	Knifed-in	1	4.3		100 114 131	< 0.05 < 0.05 < 0.05	TE-730-049

Seed for beverages and sweets

Coffee beans (SB 0716)

Residue trials were conducted during 1982-1988 in the major coffee producing areas of the world: Costa Rica, Guatemala, and El Salvador.

In field trials in Costa Rica conducted in 1982-1983, a 10% granular formulation of terbufos was applied to the soil around the base of established coffee plants at the rate of 0.75–7.5 g ai/plant. Treated samples of berries were collected at intervals, field dried according to common practice, and the outer shell or pericarp removed, leaving the dried beans.

In the trials in El Salvador and Guatemala (1988), terbufos (10% G) was applied in band to plants after flowering but before bean formation, at the rate of 1 or 5 g ai/plant. Treated samples of berries were collected at each sampling interval, field dried (38–56 days for El Salvador; 163–197 days for Guatemala), and the outer shell removed.

The coffee bean samples from each trial were shipped ambient and stored frozen prior to analysis for total terbufos residues by method M-1360. The method (GC/FPD) has been successfully validated on coffee beans at 0.05 mg/kg (Table 22). Freezer storage stability studies in corn and sugar beets have shown that residues of terbufos are stable up to two years in frozen storage. Coffee bean samples in these studies were stored frozen for 12-16 months. In the studies performed in 1988, procedural recovery data were provided. Recoveries of terbufos in beans fortified at 0.05 mg/kg were 84-112% (99 ± 12%, n=4).

The results of the residue trials on coffee are presented in Table 38. Residue levels were below the LOQ (< 0.05 mg/kg) for all treated coffee bean samples collected 58–120 days after treatment with terbufos at 0.75-7.75 g ai/plant rate. At one site (TE-790-002a) where application rates of 5 and 10 times the label rate, i.e., 3.75 and 7.5 g ai/plant, were used maximum residues of 0.12 and 0.17 mg/kg, were found in coffee beans collected at 47 or 35 days after treatment, which represented a shorter PHI than the GAP of 60 days. For both these treatments residues declined to < 0.05 mg/kg at the next sampling interval, i.e., 124 or 53 days post-treatment, respectively.

Table 38. Terbufos residues in coffee beans

Location/Year/ Variety	Application				PHI days	Residues (mg/kg)	BASF Reference Number
	Form	Type Application	no. Appl.	Rate g ai/plant			
GAP Central America (Guatemala, Belize, Dominican Rep, Honduras, El Salvador, Costa Rica, Panama): 10G formulation applied up to 2 times per year, at the rate of 0.75 to 1.1 g ai/plant for a total of 1.5 to 2.2 g ai/year and a PHI of 60 days for established plantations							
C-2351 Costa Rica, 1982 Caturra	10G	Broadcast in soil around trunk	1	0.75	60 90	< 0.05 < 0.05	TE-790-001
			1	1.5	60 90	< 0.05 < 0.05	
C-2459 Costa Rica, 1983 Caturra	10G	Broadcast in soil around trunk	1	0.75	47 63 97	< 0.05 < 0.05 < 0.05	TE-790-002a
			1	2.25	47 124	< 0.05 < 0.05	
			1	3.75	47 124	0.12 < 0.05	
			2	7.5	35 53 114	0.17 < 0.05 < 0.05	
	10G	Broadcast in soil around trunk	1	0.75	24 58	< 0.05 < 0.05	

Location/Year/ Variety	Application				PHI days	Residues (mg/kg)	BASF Reference Number
	Form	Type Application	no. Appl.	Rate g ai/plant			
GAP Central America (Guatemala, Belize, Dominican Rep, Honduras, El Salvador, Costa Rica, Panama): 10G formulation applied up to 2 times per year, at the rate of 0.75 to 1.1 g ai/plant for a total of 1.5 to 2.2 g ai/year and a PHI of 60 days for established plantations							
					90	< 0.05	
			1	2.25	90	< 0.05	
			1	3.75	90	< 0.05	
			2	7.5	24	< 0.05	
					58	< 0.05	
					90	< 0.05	
C-3380 Guatemala, 1988 Catuai	10G	Broadcast in soil around trunk	1	1	60	< 0.05	TE-790-004
					94	< 0.05	
			1	5	60	< 0.05	
					94	< 0.05	
C-3379 El Salvador, 1988 Pacas	10G	Broadcast in soil around trunk	1	1	60	< 0.05	TE-790-005
					90	< 0.05	
					120	< 0.05	
			1	5	60	< 0.05	
					90	< 0.05	
					120	< 0.05	

[a] Samples of coffee beans from two nearby growers were composited as the quantity of each individual sample was not large enough for analysis.

Fodder and forage of cereal grains

Maize forage (AF 0645) and fodder (AS 0645)

The same GAP applies to both maize and sweet corn. Data from trials on maize and sweet corn for residues in fodder and forage were conducted in the USA during 1972-1990. Terbufos granules were applied to the soil either in-furrow or in a band during planting at the rate of 1.1 – 5.8 kg ai/ha. In a trial on maize in 1973 and another on sweet corn in 1974 terbufos was applied at rates ranging from 9– 11kg ai/ha. In a few trials, tests were performed where two applications were made to maize, one at planting and a second treatment 5–6 weeks after planting.

At each sampling interval, approximately 2.3 kg each of treated forage and fodder samples were collected using either a machete or pruning shears. The samples were frozen at -10°C and stored for a maximum of 8 months prior to analysis. In one trial (TE-723-014 by Higham, J. and Alvarez, C.G. 1975) forage samples were stored frozen for up to 24 months prior to analysis. A freezer storage stability study on corn or maize forage and fodder showed that residues of terbufos-related compounds are stable in these matrices up to at least 24 months when stored at approximately -10°C (Table 29, study no. TE-326-014 by Dixon, C. 1990).

The samples were analyzed by methods M-336 or M-1754, which have both been validated at 0.05 mg/kg for terbufos-derived residues in forage and fodder (Table 22). Additional method recoveries provided in studies conducted during 1990 further demonstrated the suitability of method M-1754. Concurrent recoveries of parent fortified in forage and fodder at 0.05 mg/kg, were 82–103% (97 ± 12%, n=8).

The residue data on maize and sweet corn forage and fodder are summarized in Table 39, with residue levels from trials within the GAP, underlined.

Table 39. Terbufos residues in maize forage and fodder.

Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	**Appl. Type**	**no. Appl.**	Rate kg/ai/ha	Commodity			
GAP USA: 15G or 20G formulation at the rate of 6 oz/1000 ft row or a maximum of 1.5 kg ai/ha applied once at planting, post-emergent, or at cultivation. A PHI of 30 days is required for forage when applied post-emergent.								
Data from trials on maize								
C-2082 North Carolina, USA 1981 Pioneer 3184	15G 20G 15G	Banded Banded In-furrow	1 1 1	2.2 2.2 2.2	Forage Forage Forage	92 92 92	< 0.05 < 0.05 < 0.05	TE-723-026
C-2215 Nebraska, USA, 1982 Stauffer 6595	15G	In-furrow/ Banded	2	2.2 + 2.2	Forage Forage Fodder	30 60 105	0.25 < 0.05 0.06	TE-730-018
		In-furrow/ Banded	2	4.4 + 2.2	Forage Forage Fodder	30 60 105	< 0.05 < 0.05 < 0.05	
C-3038 Illinois, USA, 1986 Hybrid Funks G4507	15G	Banded	1	2.24	Forage Forage Fodder	31 63 181	0.07 < 0.05 < 0.05	TE-730-010
	20G	Banded	1	2.24	Forage Forage Fodder	31 63 181	< 0.05 < 0.05 < 0.05	
C-3037 Nebraska, USA, 1986 Pioneer 337	15G	Banded	1	1.12	Forage Forage Fodder	31 62 147	0.07 < 0.05 < 0.05	TE-730-011
	15G	Banded	1	1.12	Forage Forage	31 62	< 0.05 < 0.05	
	20G	Banded	1	1.12	Forage Forage Fodder	31 62 147	< 0.05 < 0.05 < 0.05	
C-3566 Iowa, USA, 1990 Circle seed #7111	15G	Banded, POST	1	1.5	Forage Forage Fodder	31 46 132	< 0.05 < 0.05 < 0.05	TE-723-032
C-3567 Iowa, USA, 1990 DeKalb DK535	15G	Banded, POST	1	1.5	Forage Forage Fodder	31 45 119	< 0.05 < 0.05 < 0.05	TE-723-033
C-3568 Illinois, USA, 1990 Dockendorf 7670	15G	Banded, POST	1	1.5	Forage Forage Fodder	30 45 109	0.17 0.08 < 0.05	TE-723-034
C-3569 Illinois, USA, 1990 Pioneer 3615	15G	Banded, POST	1	1.5	Forage Forage Fodder	30 47 109	0.07 < 0.05 < 0.05	TE-723-035
C-1129 Maryland, USA DeKalb 264 1974	15G	In-furrow	1	1.3	Forage Forage Fodder	61 106 134	< 0.05 < 0.05 < 0.05	TE-730-008
	15G	In-furrow	1	2.6	Forage Forage Fodder	61 106 134	< 0.05 < 0.05 < 0.05	
	15G	In-furrow	1	5.2	Forage Forage Fodder	61 106 134	0.24 < 0.05 < 0.05	
C-645 Colorado, USA, 1972 PAG 5X53B	15G	Banded + POST	2	1.12+ 1.12	Forage Forage Forage Forage Fodder	10 30 40 60 121	0.31 < 0.05 < 0.05 < 0.05 < 0.05	TE-730-015
	15G	Banded + POST		1.12 + 2.24	Forage Forage	10 30	8.9 0.21	

Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	**Appl. Type**	**no. Appl.**	Rate kg/ai/ha	Commodity			
GAP USA: 15G or 20G formulation at the rate of 6 oz/1000 ft row or a maximum of 1.5 kg ai/ha applied once at planting, post-emergent, or at cultivation. A PHI of 30 days is required for forage when applied post-emergent.								
					Forage	40	< 0.05	
					Forage	60	< 0.05	
					Fodder	121	< 0.05	
C-645	15G	Banded +		2.24 +	Forage	10	8.0	TE-730-015
Colorado, USA, 1972		POST		1.12	Forage	30	< 0.05	
PAG 5X53B					Forage	40	< 0.05	
(cont'd)					Forage	60	< 0.05	
					Fodder	121	< 0.05	
	15G	Banded +		2.24 +	Forage	10	6.1	
		POST		2.24	Forage	30	< 0.05	
					Forage	40	< 0.05	
					Forage	60	0.11	
					Fodder	121	< 0.05	
	15G	Banded +		1.12 +	Fodder	121	< 0.05	
		POST		5.6				
	15G	Banded +		2.24+ 5.6	Fodder	121	0.07	
		POST						
C-416:								TE-730-016
Illinois, USA, 1973	15G	In-furrow	1	1.12	Fodder	124	< 0.05	
Nebraska, USA,1973	15G	In-furrow	1	1.12	Fodder	168	< 0.05	
			1	2.24	Fodder	168	< 0.05	
			1	5.6	Fodder	168	< 0.05	
ND, USA, 1973	15G	In-furrow	1	1.12	Fodder	159	< 0.05	
			1	1.7	Fodder	159	< 0.05	
			1	2.2	Fodder	159	< 0.05	
Nebraska, USA, 1973	15G	In-furrow	1	1.12	Fodder	162	< 0.05	
			1	2.24	Fodder	162	< 0.05	
			1	5.6	Fodder	152	0.10	
Iowa, USA, 1973	15G	In-furrow	1	1.12	Fodder	135	< 0.05	
			1	2.24	Fodder	135	< 0.05	
			1	5.6	Fodder	135	< 0.05	
			1	11.2	Fodder	135	0.09	
C-415								
Indiana, USA, 1972	15G	In-furrow	1	1.12	Fodder	146	< 0.05	TE-730-017
				1.7	Fodder	146	< 0.05	
Missouri, USA, 1972	15G	In-furrow	1	1.12	Fodder	131	< 0.05	
		In-furrow	1	1.7	Fodder	184	< 0.05	
Nebraska, USA, 1972	15G	In-furrow	1	1.12	Fodder	162	< 0.05	
			1	1.12	Fodder	168	< 0.05	
NC, USA, 1972	15G	In-furrow	1	1.12	Fodder	148	< 0.05	
			1	1.12	Fodder	143	< 0.05	
			1	2.24	Fodder	105	< 0.05	
			1	5.6	Fodder	105	0.24	
ND, USA, 1972	15G	In-furrow	1	1.12	Fodder	159	< 0.05	
			1	2.24	Fodder	159	< 0.05	
Iowa, USA, 1973	15 G	In-furrow	1	1.12	Fodder	135	< 0.05	
C-631	15G	In-furrow	1	1.12	Forage	45	< 0.05	TE-723-013
Minnesota, USA, 1974					Forage	65	< 0.05	
					Fodder	115	< 0.05	
C-636	15G	In-furrow	1	1.2	Forage	71	0.23	TE-723-016

Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	**Appl. Type**	**no. Appl.**	Rate kg/ai/ha	Commodity			
GAP USA: 15G or 20G formulation at the rate of 6 oz/1000 ft row or a maximum of 1.5 kg ai/ha applied once at planting, post-emergent, or at cultivation. A PHI of 30 days is required for forage when applied post-emergent.								
Minnesota, USA, 1974					Forage	88	< 0.05	
					Forage	111	< 0.05	
					Fodder	143	< 0.05	
	15G	In-furrow	1	2.2	Forage	71	0.16	
					Forage	88	< 0.05	
					Forage	111	< 0.05	
					Fodder	143	< 0.05	
	15G	In-furrow	1	4.5	Forage	71	0.24	
					Forage	88	0.06	
					Forage	111	< 0.05	
					Fodder	143	< 0.05	
C-637 ND, USA, 1974 NK 420	15G	In-furrow	1	1.2	Forage	40	< 0.05	TE-723-017
					Forage	57	< 0.05	
					Forage	90	< 0.05	
					Forage	106	< 0.05	
					Fodder	134	< 0.05	
	15G	In-furrow	1	1.8	Forage	40	0.45	
					Forage	57	0.07	
					Forage	90	< 0.05	
					Forage	106	< 0.05	
					Fodder	134	< 0.05	
	15G	In-furrow	1	2.5	Forage	40	0.23	
					Forage	57	0.09	
					Forage	90	< 0.05	
					Forage	106	< 0.05	
					Fodder	134	< 0.05	
	15G	In-furrow	1	4.9	Forage	40	0.484	
					Forage	57	0.13	
					Forage	90	< 0.05	
					Forage	106	< 0.05	
					Fodder	134	< 0.05	
C-640 Michigan, USA, 1974	15G	In-furrow	1	1.1	Fodder	151	< 0.05	TE-723-018
			1	2.2	Fodder	151	< 0.05	
			1	4.5	Fodder	151	0.33	
C-641 Maryland, USA, 1974 DeKalb 264	15G	In-furrow	1	1.5	Forage	60	< 0.05	TE-723-019
					Forage	103	< 0.05	
					Fodder	130	< 0.05	
	15G	In-furrow	1	2.9	Forage	60	< 0.05	
					Forage	103	< 0.05	
					Fodder	130	< 0.05	
	15G	In-furrow	1	5.8	Forage	60	0.24	
					Forage	103	< 0.05	
					Fodder	130	< 0.05	
C-643 New York, USA, 1974 DeKalb XL 12	15G	In-furrow	1	1.5	Forage	44	0.14	TE-723-020
					Forage	62	0.06	
					Forage	92	< 0.05	
					Forage	110	< 0.05	
					Fodder	134	< 0.05	
	15G	In-furrow	1	2.9	Forage	44	0.17	
					Forage	62	0.06	
					Forage	92	< 0.05	
					Forage	110	< 0.05	
					Fodder	134	0.05	
	15G	In-furrow	1	5.8	Forage	44	0.48	
					Forage	62	0.25	

Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	**Appl. Type**	**no. Appl.**	Rate kg/ai/ha	Commodity			
GAP USA: 15G or 20G formulation at the rate of 6 oz/1000 ft row or a maximum of 1.5 kg ai/ha applied once at planting, post-emergent, or at cultivation. A PHI of 30 days is required for forage when applied post-emergent.								
					Forage	92	0.05	
					Forage	110	< 0.05	
					Fodder	134	0.12	
					Fodder	134	0.10	
C-644	15G	In-furrow	1	1.1	Forage	40	0.88	TE-723-021
Colorado, USA, 1974					Forage	60	0.32	
NC +					Forage	90	0.41	
					Forage	110	0.10	
					Fodder	179	< 0.05	
	15G	In-furrow	1	1.5	Forage	40	0.91	
					Forage	60	0.56	
					Forage	90	0.96	
					Forage	110	0.65	
					Fodder	179	0.08	
	15G	In-furrow	1	2.9	Forage	40	0.90	
					Forage	60	0.64	
					Forage	90	1.08	
					Forage	110	1.1	
					Fodder	179	< 0.05	
	15G	In-furrow	1	5.8	Forage	40	1.28	
					Forage	60	0.56	
					Forage	90	1.56	
					Forage	110	0.70	
					Fodder	179	0.56	
					Fodder	179	0.60	
C-647	15G	In-furrow	1	0.84	Fodder	142	< 0.05	TE-723-022
Kentucky, USA, 1973			1	1.1	Fodder	142	< 0.05	
Pioneer 3369A								
Data from trials on sweet corn								
C--642	15G	In-furrow	1	1.1	Fodder	105	< 0.05	TE-723-003
Illinois, USA, 1974								
Super sweet								
C--635	15G	Banded	1	1.5	Forage	51	< 0.05	TE-723-005
Iowa, USA, 1974					Forage	60	< 0.05	
Silver Queen					Fodder	95	< 0.05	
				2.9	Forage	51	< 0.05	
					Forage	60	< 0.05	
					Fodder	95	< 0.05	
				5.8	Forage	51	< 0.05	
					Forage	60	< 0.05	
					Fodder	95	< 0.05	
	15G	In-furrow	1	1.5	Forage	51	< 0.05	
					Forage	60	< 0.05	
					Fodder	95	< 0.05	
				2.9	Forage	51	< 0.05	
					Forage	60	0.057	
					Fodder	95	< 0.05	
				5.8	Forage	51	0.091	
					Forage	60	0.1	
					Fodder	95	< 0.05	
C-632	15G	In-furrow	1	2.2	Forage	30	0.06	TE-723-014
Florida, USA, 1974					Forage	44	0.06	
Tobelle					Forage	60	< 0.05	

Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	**Appl. Type**	**no. Appl.**	Rate kg/ai/ha	Commodity			
GAP USA: 15G or 20G formulation at the rate of 6 oz/1000 ft row or a maximum of 1.5 kg ai/ha applied once at planting, post-emergent, or at cultivation. A PHI of 30 days is required for forage when applied post-emergent.								
					Forage	75	< 0.05	
	15G	In-furrow	1	4.5	Forage Forage Forage Forage	30 44 60 75	0.05 0.07 < 0.05 0.07	
	15G	In-furrow	1	9.0	Forage Forage Forage Forage	30 44 60 75	0.08 0.30 0.09 0.14	
C-639 Minnesota, USA, 1974	15G	In-furrow	1	1.1	Forage Fodder	40 69	< 0.05 < 0.05	TE-730-003
	15G	In-furrow	1	2.2	Forage Fodder	40 69	< 0.05 < 0.05	
	15G	In-furrow	1	4.5	Forage Forage Fodder	40 40 69	0.07 0.11 0.05	
C-639 Minnesota, USA, 1974	15G	Banded	1	1.1	Forage Fodder	40 69	< 0.05 < 0.05	TE-730-003
	15G	Banded	1	2.2	Forage Fodder	40 69	< 0.05 < 0.05	
	15G	Banded	1	4.5	Forage Forage Fodder	40 40 69	< 0.05 < 0.05 < 0.05	

Sorghum forage (AF 0651) and fodder (AS 0651)

Supervised trials on sorghum were conducted during 1978-1996. In the 1996 trials, terbufos granules were applied post-emergent, at the rate of 2.1 or 2.2 kg ai/ha. Forage samples were harvested 48 to 72 days after treatment while fodder samples were taken at normal grain harvest time, 88 to 90 days after treatment. In the rest of the trials (1978-1991), terbufos granules were applied at planting, at the GAP rate (2 kg ai/ha) and at twice that rate (4 – 4.3 kg ai/ka).

At each sampling interval, about 12 plants (forage) weighing approximately 1.4 kg were collected. Approximately 1.4 kg of dried stalks remaining after removal of the grain heads were collected for fodder/stover samples. All samples were immediately frozen (-10°) and stored up to a maximum of about 8 months until analysis. A freezer storage stability study on the related crop, maize, showed that residues of terbufos-related compounds are stable in corn forage and fodder/stover up to at least 24 months when stored at approximately -10°C (Table 29, study no. TE-326-014 by Dixon, C. 1990).

Terbufos residues from the 1996 trials were determined by Method M-1754, using a gas chromatograph equipped with a flame photometric detector. The validated sensitivity of the method was 0.05 mg/kg for sorghum forage and fodder (see Table 22). For the other trials, residues of

terbufos were determined by Method M-995, which had a validated sensitivity of 0.05 mg/kg for sorghum forage and fodder samples (Table 22).

Results of all trials are summarized in Table 40, with residue levels from trials according to the GAP, underlined.

Table 40. Terbufos residues in sorghum forage and fodder

Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type	no. Appl.	Rate kg/ai/ha	Commodity			
GAP USA: 15G or 20G formulation at the rate of 1.0 oz ai/1000 ft row or a maximum of 2.0 kg ai/ha applied once and a PHI of 50 days for forage, and 100 days for grain and fodder.								
RES-96-082 Missouri, USA, 1996 KS 714Y	15G	Banded (POST)	1	2.2	Forage Forage Forage Fodder	50 61 72 90	< 0.05 (2) < 0.05 (2) < 0.05 (2) < 0.05 (2)	TE-730-057
RES-96-081 Illinois, USA, 1996 Northrup King 1210	15G	Banded (POST)	1	2.2	Forage Forage Fodder	50 60 90	< 0.05 (2) < 0.05 (2) < 0.05 (2)	TE-730-056
RES-96-080 Kansas, USA, 1996 6R55E	15G	Banded (POST)	1	2.1	Forage Forage Forage Fodder	52 52 62 90	0.05 < 0.05 < 0.05 (2) < 0.05 (2)	TE-730-055
RES-96-079 Louisiana, USA, 1996 DeKalb 37	15G	Banded (POST)		2.0	Forage Forage Forage Fodder	50 50 60 90	< 0.05 0.07 < 0.05 (2) < 0.05 (2)	TE-730-054
RES-96-078 Texas, USA, 1996 F-200	15G	Banded (POST)	1	2.2	Forage Forage Fodder	50 59 90	< 0.05 (2) < 0.05 (2) < 0.05 (2)	TE-730-053
RES-96-077 Texas, USA 1996 F-200	15G	Banded (POST)	1	2.2	Forage Forage Fodder Fodder	48 58 88 88	< 0.05 (2) < 0.05 (2) 0.19 0.12	TE-730-052
C-3374 Kansas, USA, 1989 FSIA +	15G	Knifed-in	1	4.3	Forage Fodder	60 132	< 0.05 < 0.05	TE-730-046
C-3375 Nebraska, USA, 1989 NC + 271	15G	Knifed-in	1	4.3	Forage Fodder	60 133	0.10 < 0.05	TE-730-047

Table 40. Terbufos residues in sorghum forage and fodder, cont'd

Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type	no. Appl.	Rate kg/ai/ha	Commodity			
GAP USA: 15G or 20G formulation at the rate of 1.0 oz ai/1000 ft row or a maximum of 2.0 kg ai/ha applied once and a PHI of 50 days for forage, and 100 days for grain and fodder.								
C-3376 Missouri, USA, 1989 5511	15G	Knifed-in	1	4.3	Forage Fodder	69 139	0.05 0.05	TE-730-048

C-3851 Texas, USA,1991 F-270G	15G	Knifed-in		4.3	Forage Fodder Fodder Fodder	50 100 114 131	0.88 0.49 0.13 0.18	TE-730-049
C-1626 Kansas, USA, 1978	15G 15G	Banded Banded	1 1	2.0 4.0	Fodder Fodder	95 95	< 0.05 < 0.05	TE-730-039
	15G 15G	In-furrow In-furrow	1 1	2.0 4.0	Fodder Fodder	95 95	< 0.05 0.14	
C-1742 Texas, USA, 1978 Standking Y	15G	In-furrow	1	4.0	Forage	150	< 0.05	TE-730-040
C-1752 Texas, USA, 1978, Harpool 8409	15G 15G	In-furrow Banded	1 1	4.0 4.0	Forage Forage	104 104	< 0.05 < 0.05	TE-730-041
C-1773 Oklahoma, USA, 1979; Rawhide	15G 15G	In-furrow Banded	1 1	4.0 4.0	Fodder Fodder	117 117	< 0.05 < 0.05	TE---730-042
C-1776 Colorado, USA, 1978 DeKalb A 28+	15G	Banded	1	4.0	Forage Forage	64 103	0.1 < 0.05	TE-730-043
	15G	In-furrow	1	4.0	Forage Forage	64 103	0.80 0.08	

Miscellaneous forage and fodder crops

Sugar beet tops (AV 0596)

Field trials were conducted in the USA and Canada during 1971-1975 in which terbufos (15%G) was applied in-furrow or banded at 1.0 to 2.5 kg ai/ha and also at exaggerated rates (4.0-12.3 kg ai/ha). Several trials were also conducted which consisted of sequential at-planting and post-emergence banded applications, typically reflecting exaggerated rates. Treated samples were collected at each sampling interval, stored frozen for a maximum of 22 months, and analyzed for total terbufos residues by method M-395. The freezer storage stability study conducted using this analytical method for sugar beet tops showed that samples are stable up to 24 months when stored frozen at -10°C (Table 29). The method was successfully validated for sugar beet tops at 0.05 mg/kg (Table 22).

Several trials were also conducted in the USA during the 1989 growing season in which terbufos (15%G) was knifed in at planting at 4.9 kg ai/ha. Treated samples were harvested by hand at maturity, 150–180 days after treatment. The samples were placed in frozen storage (< -17°C) for a maximum of 6 months, and were analyzed for total terbufos residues using method M-395. The only deviation from the method was the substitution of dichloromethane for chloroform. As noted above, the method has been validated for tops at 0.05 mg/kg. Residues were < 0.05 mg/kg in or on all tops control samples. Concurrent recoveries of terbufos-related residues (parent and CL94302) in tops at 0.1 mg/kg were 81-137% (101 ± 26%, n=4).

In more recent field trials in the USA in 1994, terbufos (15%G) was applied as a band over the row of sugar beets 6-23 inches tall at 2.2 – 2.4 or 4.4 – 4.9 kg ai/ha. The lower rate reflects the maximum GAP rate. Treated samples were collected at each sampling interval, stored frozen (< -8°C) for a maximum of 8 months, and analyzed by method M-2457, which had a validated sensitivity of 0.01 mg/kg for sugar beet tops. Concurrent recoveries of terbufos-related residues (parent, CL94221, CL94301, CL94302, CL94320, CL94365) fortified simultaneously in sugar beet tops at 0.01-0.60 mg/kg were 76-110% (88 ± 8%, n=26). Additional data from a method validation study on method M-2457 is presented in Table 22. A freezer storage stability study on sugar beet tops was conducted

using this method and results showed that residues in samples are stable up to at least 24 months (Table 29).

Results for all trials on sugar beet tops are summarized in Table 41, where residues from trials according to the GAP are underlined.

Table 41. Terbufos residues in sugar beet tops.

Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type	no. Appl.	Rate kg/ai/ha	Rate oz ai/1000 ft			
GAP, USA: 15G formulation at the rate of 0.6 to 1.2 oz ai/1000 ft row or an annual maximum of 2.2 kg ai/ha. PHI is 110 days, except for knifed-in applications, where the PHI is 150 days.								
RES-95-046 Idaho, USA, 1994 HM-WS91	15G	Banded, POST	1	2.2	1.2	50 70 90	0.16 0.13 <u>0.04</u>	TE-724-035
		Banded, POST	1	4.9	2.7	50 70 90	0.37 0.20 0.18	
RES-95-039 Michigan, USA, 1994 H23	15G	Banded, POST	1	2.2	1.2	50 70 91	0.05 < 0.01 <u>< 0.01</u>	TE-724-036
		Banded, POST	1	4.4	2.4	50 70 91	0.16 0.02 < 0.01	
RES-95-040 Nebraska, USA, 1994 HM 1605	15G	Banded, POST	1	2.2	1.2	50 70 90	0.04 0.01 <u>< 0.01</u>	TE-724-037
				4.4	2.4	50 70 90	0.11 0.03 0.01	
RES-95-045 North Dakota, USA, 1994	15G	Banded, POST	1	2.4	1.3	50 70 90	0.13 0.05 <u>0.01</u>	TE-724-038
ACH 192				4.6	2.5	50 70 90	0.24 0.16 0.02	
RES-95-047 Minnesota, USA, 1994 ACH 192	15G	Banded, POST	1	2.2	1.2	50 70 90	0.02 0.02 <u>< 0.01</u>	TE-724-039
		Banded, POST	1	4.4	2.4	50 70 90	0.13 0.11 0.04	
C-3366 ND, USA, 1989 UltraMono	15G	Knifed-in	1	4.9		150	< 0.05	TE-724-068
C-3367 Nebraska, USA,1989 ACH 164	15G	Knifed-in	1	4.9		180	0.08	TE-724-069
C-3368 Idaho, USA,1989 WS88	15G	Knifed-in	1	4.9		153	0.06	TE-724-070
C-3369 California, USA 1989, Z-1	15G	Knifed-in	1	4.9		170	< 0.05	TE-724-071
C-667 Colorado, USA 1974, Mono Hy	15G	In-furrow Banded	1 1	2 4		190 190	< 0.05 < 0.05	TE-724-004
C-666 Wyoming, USA	15	In-furrow	1 1	1 2		142 142	< 0.05 <u>< 0.05</u>	TE-724-005

Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type	no. Appl.	Rate kg/ai/ha	Rate oz ai/1000 ft			
GAP, USA: 15G formulation at the rate of 0.6 to 1.2 oz ai/1000 ft row or an annual maximum of 2.2 kg ai/ha. PHI is 110 days, except for knifed-in applications, where the PHI is 150 days.								
1975, Mono-HY			1	12		142	< 0.05	
		POST	1	1		116	< 0.05	
			1	2		116	< 0.05	
			1	12		116	< 0.05	
C-665 Idaho, USA 1973	15G	Banded	1	2.2	1.3	152	< 0.05	TE-724-006
C-664 Idaho, 1974 AH-A1	15G	Banded	1	1.1		60	0.05	TE-724-007
						90	< 0.05	
						168	< 0.05	
			1	2.2		60	0.21	
						90	0.15	
						168	< 0.05	
C-964 Manitoba, Canada 1975	15G	In-furrow	1	1.12		117	< 0.05	TE-724-012
C-916 Manitoba, Canada 1974	15G	In-furrow	1	1.12		117	< 0.05	TE-724-013
C-694 North Dakota, USA 1973	15G	Banded	1	2.2	1.35	121	< 0.05	TE-724-014
			1	4.5	2.7	121	< 0.05	
			1	9.0	5.4	121	< 0.05	
			1	2.2	1.35	155	< 0.05	
			1	4.5	2.7	155	< 0.05	
			1	9.0	5.4	155	< 0.05	
		In-furrow	1	2.2	1.35	121	< 0.05	
			1	4.5	2.7	121	< 0.05	
			1	2.2	1.35	155	< 0.05	
			1	4.5	2.7	155	< 0.05	
		POST	1	2.2	1.35	124	< 0.05	
			1	4.5	2.7	124	< 0.05	
			1	9.0	5.4	124	< 0.05	
		Banded + POST	1	4.4+2.2		124	< 0.05	
			1	4.4+4.4		124	< 0.05	
C-695 North Dakota, USA, 1974/ American . Crystal Hybrid #2B	15G	Banded	1	2.5	1.35	31	< 0.05	TE-724-016
			1	4.9	2.7	31	< 0.05	
			1	12.3	6.75	31	< 0.05	
			1	2.5	1.35	62	< 0.05	
			1	4.9	2.7	62	< 0.05	
			1	12.3	6.75	62	< 0.05	
			1	2.5	1.35	94	< 0.05	
			1	4.9	2.7	94	< 0.05	
			1	12.3	6.75	94	< 0.05	
			1	2.5	1.35	129	< 0.05	
			1	4.9	2.7	129	< 0.05	
			1	12.3	6.75	129	< 0.05	
		In-furrow	1	2.5	1.35	31	< 0.05	
			1	4.9	2.7	31	< 0.05	
			1	12.3	6.75	31	< 0.05	

Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type	no. Appl.	Rate kg/ai/ha	Rate oz ai/1000 ft			
GAP, USA: 15G formulation at the rate of 0.6 to 1.2 oz ai/1000 ft row or an annual maximum of 2.2 kg ai/ha. PHI is 110 days, except for knifed-in applications, where the PHI is 150 days.								
			1	2.5	1.35	62	< 0.05	
			1	4.9	2.7	62	< 0.05	
			1	12.3	6.75	62	< 0.05	
			1	2.5	1.35	94	< 0.05	
			1	4.9	2.7	94	< 0.05	
			1	12.3	6.75	94	< 0.05	
			1	2.5	1.35	129	< 0.05	
			1	4.9	2.7	129	< 0.05	
			1	12.3	6.75	129	< 0.05	
		Banded + POST	2	2.5+2.5		51	< 0.05	
			2	2.5+4.9		51	< 0.05	
			2	2.5+12.3		51	< 0.05	
			2	2.5+2.5		83	< 0.05	
			2	2.5+4.9		83	< 0.05	
			2	2.5+12.3		83	< 0.05	
			2	2.5+2.5		118	< 0.05	
			2	2.5+4.9		118	< 0.05	
			2	2.5+12.3		118	< 0.05	
C-693 Michigan, USA, 1974 Monitor common	15G	In-furrow	1	1.8	1.35	40	2.78	TE-724-017
			1	3.6	2.7	40	3.75	
			1	1.8	1.35	60	1.36	
			1	3.6	2.7	60	1.04	
			1	1.8	1.35	91	0.12	
			1	3.6	2.7	91	0.09	
			1	1.8	1.35	174	< 0.05	
			1	3.6	2.7	174	< 0.05	
			1	7.2	5.4	174	0.06	
C-693 Michigan, USA, 1974 Monitor common		Banded	1	1.8	1.35	40	2.81	TE-724-017
			1	3.6	2.7	40	3.86	
			1	1.8	1.35	60	1.12	
			1	3.6	2.7	60	2.18	
			1	1.8	1.35	91	0.82	
			1	3.6	2.7	91	0.21	
			1	1.8	1.35	174	< 0.05	
			1	3.6	2.7	174	< 0.05	
			1	7.2	5.4	174	< 0.05	
C-668 Colorado, USA, 1973/ Mono-Hy-1	15G	Banded	1	2.2	1.36	60	0.06	TE-724-018
			1	4.5	2.7	60	0.16	
			1	9.0	5.4	60	0.58	
			1	2.2	1.36	90	< 0.05	
			1	4.5	2.7	90	< 0.05	
			1	9.0	5.4	90	< 0.05	
			1	2.2	1.36	119	< 0.05	
			1	4.5	2.7	119	< 0.05	
			1	9.0	5.4	119	< 0.05	
			1	2.2	1.36	157	< 0.05	
			1	4.5	2.7	157	< 0.05	
			1	8.96	5.4	157	< 0.05	
		POST	1	2.2	1.36	29	0.06	
			1	4.5	2.7	29	0.21	
			1	9.0	5.4	29	0.31	
			1	2.2	1.36	59	< 0.05	
			1	4.5	2.7	59	< 0.05	
			1	9.0	5.4	59	< 0.05	
			1	2.2	1.36	88	< 0.05	
			1	4.5	2.7	88	< 0.05	
			1	9.0	5.4	88	< 0.05	

Location/Year/ Variety	Application					PHI	Residues (mg/kg)	Reference Number
	Form.	Appl. Type	no. Appl.	Rate kg/ai/ha	Rate oz ai/1000 ft			
GAP, USA: 15G formulation at the rate of 0.6 to 1.2 oz ai/1000 ft row or an annual maximum of 2.2 kg ai/ha. PHI is 110 days, except for knifed-in applications, where the PHI is 150 days.								
			1	2.2	1.36	126	< 0.05	
			1	4.5	2.7	126	< 0.05	
			1	8.96	5.4	126	< 0.05	
		Banded + POST	2	4.5+2.2		29	0.12	
			2	4.5+4.5		29	0.30	
			2	4.5+2.2		59	< 0.05	
			2	4.5+4.5		59	< 0.05	
			2	4.5+2.2		88	< 0.05	
			2	4.5+4.5		88	< 0.05	
			2	4.5+2.2		126	< 0.05	
			2	4.5+4.5		126	< 0.05	
C-914 Manitoba, Canada, 1973	15G	Banded	1	1.12		114	< 0.05	TE-724-029
C-917 Manitoba, Canada 1971	15G	In-Furrow	1	1.1		126	< 0.05	TE-724-030
			1	1.1		133	< 0.05	
C-656 North Dakota, USA, 1972	15G	Banded	1	1.1		135	< 0.05	TE-724-048
			1	2.2		135	< 0.05	
		POST	1	1.1		119	< 0.05	
			1	2.2		119	< 0.05	
C-657 Wyoming, USA, 1972 Mono-Hi	15G	Banded	1	0.56		156	< 0.05	TE-724-049
			1	1.1		156	< 0.05	
			1	6.7		156	0.08	
C-696 Minnesota, USA, 1973 American Crystal Hybrid #13	15G	Banded	1	2.2		102	< 0.05	TE-724-050
			1	4.4		102	0.06	
			1	8.9		102	0.11	
			1	2.2		138	< 0.05	
			1	4.4		138	< 0.05	
			1	8.9		138	< 0.05	
		POST	1	2.2		73	0.09	
			1	4.4		73	0.78	
			1	8.9		73	1.23	
			1	2.2		109	< 0.05	
			1	4.4		109	< 0.05	
			1	8.9		109	0.20	
		Banded+POST	2	4.5 + 2.2		73	0.26	
			2	4.5 + 4.5		73	0.46	

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

Terbufos is not registered for use in stored products.

In Processing

Since terbufos residues in maize grain, sweet corn kernels, corn-on-the-cob, coffee beans, sugar beets (roots) and sorghum grain were at non-detectable levels, processing studies were not relevant.

Residues in the Edible Portion of Food Commodities

For commodities belonging to the group with inedible peel, residue data on both the whole commodity and the edible portion are submitted to the Meeting. Although terbufos is systemic in nature, it is expected that some of the residues remain in the peel and are removed by peeling. In order

to determine the amount of terbufos residues in the edible portion (pulp), in some supervised trials conducted on bananas (Table 33), terbufos residues were determined in both whole bananas and pulp. In general, residue levels even in whole bananas were relatively low (< 0.01 – 0.03 mg/kg). Therefore, from trials according to the GAP, the level of residue actually remaining in the pulp was difficult to ascertain since the residues in the pulp were equal or only slightly less than those in whole bananas. However, in trials where exaggerated rates were used (study no. TE-714-003, 005, 006, 007, 008 and 013, respectively by Khunachak, A., 1986b, d; Khunachak, A., 1997, Bohn, W., 1985a, and Bohn, W. and Behm, J., 1985) and residues in the pulp and whole bananas were determined, results showed that an average of 79% of the residues of terbufos remained in the pulp. Twenty one percent was removed by peeling. The details are presented in Table 42.

Table 42. Estimated terbufos-related residues in banana pulp.

Terbufos-related residues, mg/kg		% Residues in pulp	Reference
Whole banana	**Pulp**		
0.02	0.02	100	TE-714-003
0.02	0.01	50	
0.02	0.02	100	
0.01	< 0.01	100	
0.02	< 0.01	50	TE-714-005
0.03	0.02	67	
0.02	0.01	50	
0.01	< 0.01	100	
0.02	< 0.01	50	TE-714-006
0.01	0.01	100	
0.02	< 0.01	50	TE-714-007
0.01	0.01	100	
0.02	0.02	100	TE-714-008
0.01	< 0.01	100	
0.02	< 0.01	50	TE-714-013
0.01	0.01	100	
Average		**79%**	

RESIDUES IN ANIMAL COMMODITIES

Metabolism studies on ruminants and poultry showed that terbufos was rapidly metabolized leaving no detectable residues in tissues, milk, and eggs. All residues in tissues, milk, and eggs were below the limit of determination for the corresponding analytical methods used. Based on these, feeding studies could have been waived. However, ruminant and poultry feeding studies were conducted and made available to the Meeting, the results of which confirm the findings from the metabolism studies.

Ruminant feeding studies

Beef cattle feeding study

In a beef cattle feeding study conducted in the USA, a mixture of terbufos and its phosphorus-containing metabolites was administered to two groups of three steers each for 21 days at a dose equivalent to 0.05 ppm body weight/day or 2 ppm dry weight in the diet (study no. TE-705-002 by Manuel, A., 1972b). The mixture was representative of the relative metabolite concentrations found in 10-week-old maize plants resulting from a terbufos soil treatment at planting. Tissues of fat, muscle, liver and kidney from treated animals sacrificed 24 hours after the last dose showed no detectable residues of terbufos and related metabolites (Table 43). The tissues were analyzed by method M-372, using a gas chromatograph equipped with an alkali flame ionization The method was validated for cattle tissues with a sensitivity of 0.05 mg/kg (Table 24).

Table 43. Summary of total terbufos-related residues in beef cattle edible tissues from cattle fed a nominal dose equivalent to 2 ppm for 21 days

Cattle No.	Dose (mg/kg in diet)	Total terbufos-related residues (mg/kg)			
		Muscle	Fat	Kidney	Liver
A-56	2	< 0.05	< 0.05	< 0.05	< 0.05
A-61	2	< 0.05	< 0.05	< 0.05	< 0.05
A-62	2	< 0.05	< 0.05	< 0.05	< 0.05

Dairy cattle feeding studies

In a dairy cattle feeding study the same mixture of terbufos and metabolites at the same dose level (2 ppm in the feed) was administered daily to two groups of three dairy cows for 21 days (Manuel, A., 1972c). Milk samples were taken on days 0 (pre-treatment), 7, 14, and 21 and analyzed by method M-353, using a gas chromatograph equipped with an alkali flame ionization detector. The method was validated for milk samples with a sensitivity of 0.01 mg/kg (Table 24). Results from whole milk analyses indicated that cows from both groups showed total apparent terbufos-related residues below the limit of determination (< 0.01 mg/kg) at days 7, 14 and 21 during dosing (Table 44).

Table 44. Summary of total terbufos-related residues in milk from dairy cattle fed a nominal dose equivalent to 2 ppm for 21 days

Cattle No.	Dose (mg/kg in diet)	Total terbufos-related residues (mg/kg)		
		7 DAY	14 DAY	21 DAY
A-1	2	< 0.01	< 0.01	< 0.01
A-3	2	< 0.01	< 0.01	< 0.01
A-4	2	< 0.01	< 0.01	< 0.01

A complementary study was conducted in 1987 where terbufos was administered to three dairy cows at a dose level of 50 ppm in the diet (study no. TE-705-004 by Peterson, R., 1989). The protocol called for 7 consecutive days of treatment followed by a withdrawal period of 7 days where no terbufos was fed. Milk samples were scheduled to be collected in the morning and in the after noon for 14 days. However, cow mortality prevented the study from being completed as proposed. Instead, milk samples were collected only for 3.5 days, at 0.5 day sampling intervals. Milk samples were analyzed by method M-1829, which had a validated sensitivity of 0.005 mg/kg. The method validation data is summarized in Table 24.

Residues of terbufos-related compounds in milk from this study are summarized in Table 45. Residues in milk samples were all below the limit of determination, except for two samples where residues were detected at levels of 0.011 mg/kg and 0.005 mg/kg.

Table 45. Terbufos-related residues in milk after dosing with terbufos at 50 ppm in the diet for a period of 3.5 days

Sampling interval from 1st treatment, days)	Dose (mg/kg in diet)	Total terbufos-related residues (mg/kg)		
		Cow # 365	Cow # 220	Cow # 367
0	50	< 0.005		
0.5	50	0.011	< 0.005	< 0.005
1	50	< 0.005	< 0.005	
1.5	50	< 0.005	0.005	< 0.005 (2)
2	50	< 0.005		
2.5	50	< 0.005		
3.5	50	< 0.005		

Poultry feeding study

A mixture of terbufos and its phosphorus-containing metabolites was administered by gavage to nine chickens for 21 days at a dose equivalent to 0.1 mg/kg body weight or 2 ppm in the diet on a dry weight basis (study no. TE-705-001 by Manuel, A., 1972a). The mixture was the same as that used in the cattle feeding studies. Egg samples were taken for analysis at 7, 14, and 21 days, and analyzed for terbufos-related residues by method M-396, using a gas chromatograph equipped with an alkali flame ionization detector. The method had been validated with a sensitivity of 0.01 mg/kg (Table 24). Results showed that all residues of terbufos-related compounds in eggs were below the limit of determination (< 0.01 mg/kg). Table 46 summarize the results for eggs.

Table 46. Summary of total terbufos-related residues in eggs from poultry fed a nominal dose equivalent to 2 ppm for 21 days

Sample No.	Dose (mg/kg in diet)	Total terbufos-related residues (mg/kg)		
		7 DAY	14 DAY	21 DAY
B-4	2	< 0.01	< 0.01	< 0.01
B-5	2	< 0.01	< 0.01	< 0.01
B-6	2	< 0.01	< 0.01	< 0.01

The chickens were sacrificed at the end of the feeding period and samples of fat, muscle, liver, kidney, and skin were taken and analyzed for total terbufos residues using method M-401, which had been validated at a sensitivity level of 0.05 mg/kg (Table 24). Results showed that residues of terbufos-related compounds are below the limit of determination (< 0.05 mg/kg) in poultry tissue samples taken after 21 days of continuous feeding with treated rations. Table 47 summarizes the results on poultry tissues.

Table 47. Summary of total terbufos-related residues in poultry edible tissues from chickens fed a nominal dose equivalent to 2 ppm for 21 days

Sample No.	Dose (mg/kg in diet)	Total terbufos-related residues (mg/kg)			
		Muscle	Fat	Kidney	Liver
B-7	2	< 0.05	< 0.05	< 0.05	< 0.05
B-8	2	< 0.05	< 0.05	< 0.05	< 0.05
B-9	2	< 0.05	< 0.05	< 0.05	< 0.05
B-10	2	< 0.05	< 0.05	< 0.05	< 0.05
B-11	2	< 0.05	< 0.05	< 0.05	< 0.05
B-12	2	< 0.05	< 0.05	< 0.05	< 0.05

RESIDUES IN FOOD IN COMMERCE AND AT CONSUMPTION

The Pesticide Data Program (PDP) of the US Department of Agriculture (USDA) collects data on pesticide residues in food (USDA, 2002). Table 48 summarizes the data for terbufos and its metabolites in the PDP database. The data represent the results of monitoring pesticide residues in food up to the year 2002.

Table 48. Monitoring data for terbufos from the US Pesticide Data Program (PDP)

Commodity	Total Samples Screened	Samples with Detection	% Samples with Detection	Range of values detected (mg/kg)	Range of LODs (mg/kg)
TERBUFOS					
Apples	556	0			0.006-0.014
Asparagus	622	0			0.004-0.006
Banana	727	0			0.006-0.014
Broccoli	125	0			0.006
Carrots	536	0			0.001-0.006
Celery	170	0			0.001-0.006
Cucumbers	129	0			0.006

Commodity	Total Samples Screened	Samples with Detection	% Samples with Detection	Range of values detected (mg/kg)	Range of LODs (mg/kg)
Mushrooms	642	0			0.001-0.06
Peaches	563	0			0.001-0.002
Pineapples	106	0			0.006
Potatoes	370	0			0.006-0.015
Spinach	363	0			0.015
Sweet bell peppers	186	0			0.002
Sweet corn, caned/frozen	727	0			0.006-0.015
Sweet peas. Canned/frozen	643	0			0.004-0.006
TOTAL	**6,465**	**0**			
TERBUFOS SULFONE					
Apples	556	0			0.004-0.007
Asparagus	623	0			0.004
Banana	727	0			0.004-0.007
Broccoli	125	0			0.004
Carrots	536	0			0.001-0.004
Celery	170	0			0.002-0.004
Cucumbers	129	0			0.004
Mushrooms	642	0			0.001-0.004
Peaches	563	0			0.001-0.002
Pineapples	106	0			0.004
Potatoes	370	0			0.004-0.048
Spinach	363	0			0.048
Sweet bell peppers	186	0			0.002
Sweet corn, caned/frozen	727	0			0.004-0.018
Sweet peas. Canned/frozen	643	0			0.004
TOTAL	**6,466**	**0**			

NATIONAL MAXIMUM RESIDUE LIMITS

The manufacturer reported MRLs for the following countries: Australia, Brazil, Chile, Japan, Korea and USA. Terbufos is not authorized for use on agricultural crops in the Netherlands.

APPRAISAL

Terbufos, a systemic nematicide and soil insecticide, was evaluated for the first time by JMPR in 1989. A further residue review was undertaken in 1990. At the 36th Session of the CCPR the compound was scheduled for a residue evaluation within the periodic review program for 2005. The toxicological review was conducted in 2003, which established an ADI of 0.0006 mg/kg bw/day and an ARfD of 0.002 mg/kg bw/day.

The Meeting received information on identity; metabolism and environmental fate; analytical methods; relevant storage stability studies; use pattern; residues resulting from supervised trials on a number of crops including bananas, coffee beans, sugar beets, maize, sorghum, and sweet corn; residues in food in commerce and at consumption and national maximum residue limits.

List of terbufos and related metabolites:

Terbufos	*S-tert*-butylthiomethyl *O,O*-diethyl phosphorodithioate
Terbufos sulfoxide	*S-tert*-butylsulfinylmethyl *O,O*-diethyl phosphorodithioate
Terbufos sulfone	*S-tert* -butylsulfonylmethyl *O,O*-diethyl phosphorodithioate
Terbufoxon	*S-tert*-butylthiomethyl, *O,O*-diethyl phosphorodithioate
Terbufoxon sulfoxide	*S-tert* -butylsufinylmethyl, *O,O*-diethyl phosphorodithioate
Terbufoxon sulfone	*S-tert*-butylsulfonylmethyl, *O,O*-diethyl phosphorodithioate

Animals Metabolism

The Meeting received information on the fate of [methylene-^{14}C]terbufos in rats, lactating goats and laying hens dosed orally.

Studies on metabolism in rats were evaluated by the WHO Expert Group of the 2003 JMPR, which concluded that absorption of single doses of ^{14}C-terbufos was rapid and fairly complete. Most of the radiolabel was excreted within 24 – 48 h. Excretion was primarily by the urinary route (about 70 – 80% of the administered dose). Terbufos was extensively metabolized and little radioactivity was found in the tissues. Sulfoxidation and desulfuration of terbufos is followed by hydrolysis of the thiolophosphorus bond, enzymatic S-methylation and then additional S-oxidation. On the basis of a 14-day study of repeated doses, terbufos showed little potential for accumulation.

[Methyllene-^{14}C]terbufos at doses equivalent to 0.281 and 2.53 mg/kg body weight, were administered via capsule to two lactating goats separately, i.e., one dose regime per goat. Each goat was dosed once daily for seven consecutive days. The major route of excretion was via the urine, which accounted for 96.0 and 86.9% of the administered radioactivity respectively. The main metabolic pathway in lactating goats and rats is qualitatively similar, thus suggesting a common metabolic pathway. Neither terbufos nor any of the phosphorylated oxidative metabolites - sulfoxide, sulfone, oxygen analog and its sulfoxide and sulfone - were observed in milk. None of the phosphorylated oxidative metabolites were detected in tissues. However, terbufos (parent) was observed at low concentrations in liver (< 0.01 mg/kg eq) and in kidney (< 0.01 mg/kg eq).

The total radioactive residue (TRR) in daily milk samples were < 0.01 mg/kg eq (low dose, 0.28 mg/kg eq in diet, day 7) and 0.02-0.03 mg/kg eq (high dose, 2.53 mg/kg eq in diet, day 7). Residues in the liver, kidney, muscle and fat of the low dose animal were all < 0.01 mg/kg eq. In the high dose animal, residues were 0.08, 0.04, < 0.01 and < 0.01 mg/kg eq, respectively.

Two groups of laying hens were dosed via capsules with [methyllene-^{14}C]terbufos for five consecutive days with the feed equivalent of 0.35 ppm for one group (Group B) and an exaggerated level of 1.05 ppm equivalent for the second group (Group C). Recovery of [^{14}C] residues in excreta over the 5-day treatment period averaged 91.4% of the total administered dose for the 1st group, and 88.9% for the 2nd group. For both dose levels, residues in eggs (days 1 through 5, both white and yolk), skin with adhering fat, muscle, liver or kidney tissues were all less than the LOQ of the radioassay (< 0.05 mg/kg eq).

The results of the hen study showed that terbufos when orally ingested at highly-exaggerated levels does not give rise to residues in the eggs or edible tissues of the laying hen.

Plant metabolism

The Meeting received information on the metabolic fate of ^{14}C-terbufos in soybeans, sugar beet, sweet corn, cabbage and rape seed.

Soybean plants were grown under field conditions from seed treated in the furrow at a rate of 1.1 kg ai/ha with [methylene-^{14}C]terbufos. The TRR levels found in the plant, expressed as terbufos equivalent, were 13.3 and 1.5 mg/kg in plants at one and two months after treatment, respectively. At harvest, residue levels were 1.8 mg/kg in fodder, 1.6 mg/kg in hulls and 1.3 mg/kg in the seed.

At the one-month sampling, 43% of the total extractable residue was identified as the phosphorylated metabolites: sulfoxide, sulfone, oxygen analog sulfone, and oxygen analog sulfoxide. The non-phosphorylated metabolites accounted for 11% of the residue. The remaining residue was comprised of five unknown metabolites (4%) and origin-bound compounds (17%). At harvest only non-phosphorylated metabolites were identifiable at low (< 10%) levels in all three commodities, i.e., hulls, fodder and seed. The remaining residue was shown to be very polar extractable materials or to have the ^{14}C incorporated into the cellulose and lignin of the hulls, fodder and protein and oil of the seed.

In conclusion, soybean seedlings can readily take up terbufos applied to the soil. The absorbed compound is then translocated and metabolized by oxidation, hydrolysis, methylation and subsequent oxidation to eventually yield principally non-phosphorylated, non-toxic metabolites.

In sugar beet metabolism studies, plants were grown from seed in soil treated with [methyllene-^{14}C]terbufos at a rate of 6.8 kg ai/ha. The levels of radioactivity in both foliage and roots were determined at 4.5, 8, l6, and 32 weeks after treatment. The TRR levels found in the various samples declined with time from 6.27 to 1.07 mg/kg eq in foliage and from 7.44 to 0.284 mg/kg eq in roots. The levels of ^{14}C recovered in all plants represented a total of only 2.3% of the applied dose. The data showed that metabolism of terbufos occurred at a faster rate in the roots. Chromatographic data obtained at different stages of plant growth indicated that terbufos is degraded mainly by way of oxidation, hydrolysis and methylation followed by subsequent oxidation to yield principally non-phosphorylated, non-toxic metabolites.

There is also evidence of incorporation of terbufos-derived radioactivity into the sucrose fraction of sugar beets.

In sweet corn metabolism studies, corn was grown in metal cylinders contained in greenhouses and treated with [methylene-^{14}C]terbufos at 1.1 kg ai/ha. Sweet corn contained 0.34, 2.64, 4.70 and 6.85% of the applied dose at 2, 4, 7 and 10 weeks of growth. The identified phosphate esters found as metabolites in sweet corn accounted for about 89% of the radioactivity. Levels of ^{14}C extracted from plants were separated into at least 19 radioactive metabolites using thin layer chromatography (TLC). The expected oxidation products of terbufos, i.e., the sulfoxide, the sulfone, the oxygen analog of sulfoxide and sulfone, were confirmed to be present as residues in the corn plants. In the corn plants sampled at 10 weeks the phosphorylated metabolites, terbufos sulfoxide (8.1 mg/kg eq), terbufos sulfone (2.8 mg/kg eq), terbufoxon (0.3 mg/kg eq), terbufoxon sulfoxide (16.9 mg/kg eq) and terbufoxon sulfone (5.6 mg/kg eq) accounted for 34% of the chloroform-soluble extractable radioactivity. A significant amount of the total hydrophilic radioactivity could be in the form of natural products.

In the cabbage metabolism study, plants were grown in a greenhouse and externally from seed in soil treated with [methyllene-^{14}C]terbufos at a rate of 2.2 kg ai/ha, using both a 15-G granular formulation and a liquid concentrate. The levels of radioactivity found in the cabbage plants, expressed as mg/kg equivalent of terbufos, declined with time (4 to 16 weeks) from 3.93 to 0.09 mg/kg eq for external granular treatment, from 1.48 to 0.04 mg/kg eq for the external liquid treatment and from 1.71 to 0.07 mg/kg eq for the greenhouse liquid treatment. The absolute amounts of radioactivity (in μCi) recovered in plants did not vary much with time. The recovered radioactivity represents a maximum of 1.5% of the total applied dose. At the end of 12 weeks, 92% (0.07 to 0.22 mg/kg eq) of the total radioactivity consisted of unidentified water-soluble metabolites and the total amount of phosphate compounds were less than 0.01 mg/kg eq. There was no apparent metabolic difference between granular (15-G) or liquid-treated soil in developing cabbage. The metabolism of terbufos in cabbage is similar to that reported for sugar beet.

In a rape metabolism study, rape seed was grown in soil treated with [methylene^{14}C]terbufos in the furrow at 0.28 kg ai/ha. The total residual radioactivity in rape plants expressed as parent was 0.63 and 0.68 mg/kg eq for 1 and 2 month post-treatment samples respectively. Residues were 0.42 mg/kg eq in the 2 month hulls sample. At harvest (3-months post treatment), the residue levels in fodder, hull and seed were 3.21, 3.63 and 1.11 mg/kg eq, respectively. The extractable radioactivity from the 1-month old rape plant was 90%, of which 48% was organosoluble and 42% was aqueous soluble. By two-dimensional TLC analysis, about 16.3% of the radioactive organosolubles migrated away from the plate origin and the remaining 31.7% of the radioactivity stayed at the origin. Among the migrating radiocomponents, non-phosphorylated compounds predominated with 4.9%, terbufoxon sulfoxide accounted for 4.0% and non-phosphorylated compounds and terbufos sulfoxide contributed to 1.7 and 1.3% of the resolved organoextractables respectively. The remaining 4.4% of the migrating radioactivity was made up of at least 6 minor components.

Rape plants can readily take up terbufos and closely related metabolites from the soil. The absorbed compounds are then initially metabolized in plant tissues by way of oxidation to phosphorylated metabolites such as terbufos sulfoxide and terbufoxon sulfoxide. These oxidized products degrade further through hydrolysis, methylation and subsequent oxidation thus leading to the formation of certain non-phosphorylated metabolites. In rape seeds, the hexane fraction comprised of 22% of the radioactivity which was probably associated with fatty acids or lipid-type compounds. The acetonitrile fraction, accounting for about 12%, mainly consisted of oil-related compounds and a non-phosphorylated compound along with trace amounts of several other minor components. The hydrolysis study indicated that incorporation of ^{14}C-formaldehyde or $^{14}CO_2$ derived from [^{14}C]terbufos, into natural products of various rape tissues accounts for a very large fraction of the radioactivity present in the plants or seeds.

In conclusion, the metabolic pathway for the formation of observed metabolites arises from sulfoxidation and desulfuration of terbufos, hydrolysis of the thiol-phosphorous bond (S=P), enzymatic S-methylation and finally S-oxidation. The studies evaluated show that the same oxidative phosphorylated metabolites of terbufos occur in plants and in animals. In addition, terbufos has been shown to be taken up by the roots, with the residues and metabolites translocated to all parts of the plants examined.

Environmental fate

The Meeting received information on aerobic degradation in soil, hydrolysis rates and products and a confined rotational crop study.

Degradation in soil (aerobic)

The metabolic fate of terbufos in soil was investigated in silt loam soil under aerobic conditions using [methylene-^{14}C]terbufos. The half-life of terbufos was approximately 5 days and of the total terbufos related residues was approximately 100 days. Major degradation products were carbon dioxide and the oxidative metabolites terbufos sulfoxide and terbufos sulfone. The concentration of terbufos sulfoxide in soil increased rapidly to a maximum of 2.6 mg/kg eq (52% of the applied dose) after 30 days and then declined to 0.3 mg/kg eq (6% of dose) after one year. Terbufos sulfone residues increased slowly to a maximum level of 1.0 mg/kg eq. (20% of applied dose) at 60 days and then decreased to 0.1 mg/kg eq (2.3% of dose) after one year.

Hydrolysis Rate and Products

Terbufos hydrolyses rapidly under abiotic conditions at environmentally relevant temperatures and would not be expected to persist in aquatic systems. Hydrolysis of terbufos sulfoxide and terbufos sulfone occurs more slowly, but the des-ethyl derivatives that formed are not expected to be of toxicological concern.

Confined Rotational Crop study

Residues of terbufos and related compounds were determined in soil and rotational crops (cabbage, red beets, and wheat) from a treated corn field. In the study in Wisconsin, corn was planted in a silt loam soil and treated at planting with 2.24 kg ai/ha. Residues of terbufos and related compounds were less than the LOQ of the method (0.05 mg/kg) in all cabbage, red beet and wheat grain samples. Wheat straw contained residues of 0.1 mg/kg. The soil half-life of terbufos and related compounds was calculated to be 30 days.

In another study conducted in Nebraska, corn planted in silt loam soil was treated at planting by soil incorporation with terbufos at the rate of 2.24 kg ai/ha. Residues of terbufos and related compounds were less than the LOQ of the method (0.05 mg/kg) in all cabbage, sugar beet and wheat grain samples. Spring wheat forage contained residues of 0.15 mg/kg. No residues were detected in winter wheat straw and grain. The soil half-life of terbufos and related compounds was calculated to be 17 days in beet plots, 16 days in cabbage plots, and 10 days in wheat plots.

Methods of analysis

The Meeting received information on validated methods of analysis of terbufos in plant matrices, animal matrices and environmental samples that were used in supervised trials, rotational crops studies and storage stability studies. Enforcement methods and multiresidue methods of analysis were also submitted to the Meeting.

Several analytical methods have been developed for the determination of terbufos in plant commodities and animal tissues, suitable for data collection and enforcement. All analytical methods for terbufos residues are designed to extract parent terbufos and its oxygenated metabolites: terbufos sulfoxide, terbufos sulfone, terbufoxon and terbufoxon sulfoxide. Terbufos and its metabolites are oxidized to the common moiety terbufoxon sulfone using m-chlorobenzoic acid, which is then analysed by gas chromatograph equipped with a phosphorus-selective detector. The methods vary slightly, usually in the extraction solvent used.

In plant samples, the LOQ for most of the reported trials was 0.05 mg/kg, but limits for some methods/substrates were 0.01 or 0.005 mg/kg. Recoveries of terbufos and its related metabolites were tested over the concentration range of 0.01 – 1.0 mg/kg on samples from all plant commodities reported in the trials.

In animal tissue samples, the LOQ for the milk is 0.005 or 0.01 mg/kg, for the tissue, 0.05 mg/kg, and for eggs, 0.01 mg/kg. Recoveries of terbufos and its related metabolites were tested on the samples over the concentration range of 0.005 – 1.0 mg/kg.

An adequate method is available for enforcement of terbufos MRLs in or on plant commodities. The GC method for determining terbufos and its phosphorylated metabolites is described in the Pesticide Analytical Manual (PAM), Vol.II as Method I modified by Method M-1754 substituting acetone for benzene and dichloromethane for chloroform.

Terbufos and its metabolites were taken through the US FDA Multiresidue Method with limited success.

Stability of pesticide residues in stored analytical samples

The stability of terbufos residues has been determined in freezer storage stability studies (from < 0 to -10ºC or -17ºC) in the representative plant commodities of corn (grain, plants and straw); sugar beet (tops and roots); and banana (unpeeled and pulp). Terbufos residues fortified in representative crop samples (root, grain, watery and oily commodities) were shown to be stable in frozen storage for approximately 18 months.

The stability of terbufos residues in milk (1.7–3.3ºC) has been determined and 79% of the residues were recovered after 14 days.

No stability studies were submitted to the Meeting on other animal matrices.

Definition of the residue

Metabolic studies on animals and plants have demonstrated that terbufos is metabolized in much the same way in all the biological systems studied. The decrease in the parent compound is accompanied by a short-term build-up of the sulfoxide and sulfone metabolites. The corresponding oxygen analogues are also formed, but at a much slower rate. Cleavage of the P=S bond yields, after methylation of the resulting thiol, a series of methylated metabolites differing in the oxidation state of the sulfur atoms.

Terbufos and all oxidation products are considered potent anticholinesterase agents.

Terbufos is readily metabolized in both plant and animal tissues by way of oxidation, hydrolysis and methylation which is then followed by further oxidation to principally non-toxic metabolites.

All analytical methods used to determine terbufos residues are designed to extract parent terbufos, and its oxygenated metabolites terbufos sulfoxide, terbufos sulfone, terbufoxon, and terbufoxon sulfoxide.

The Meeting confirmed the previous (JMPR 1989) residue definition for terbufos, both for enforcement and for risk assessment and for both animal and plant commodities as follows:

The sum of terbufos, its oxygen analogue and their sulfoxides and sulfones expressed as terbufos.

Although terbufos has a log k_{ow} of 4.71 based on the parent terbufos, the total residue of terbufos and related metabolites are not considered fat soluble.

Results of supervised trials on crops

Supervised residue trials were available for bananas, sugar beets, sweet corn, cereal grains (maize and sorghum); coffee beans, fodder and forage of cereal grains (maize and sorghum); and miscellaneous forage and fodder crops (sugar beet tops). A large number of trials were submitted from the 1970s based on analytical methods with an LOQ of 0.05 mg/kg. More recent trials were provided which had an improved LOQ of 0.01 and were used in estimating residues and establishing MRLs. In cases of finite residues, then relevant data from trials with an LOQ of 0.05 were considered acceptable to include in the data set. Supervised trials on the remaining commodities that currently have CXLs were not provided. The Meeting decided to withdraw the current recommendations for broccoli, cabbages (head), mustard seed, onion (bulb), peanut, peanut fodder, peanut forage (green), popcorn, rape seed, rapeseed oil (crude), soy beans (dry); straw and fodder of cereal grains, sugar beet fodder and wheat.

In situations where residues from supervised trials from GAP show nil residues, the MRL was chosen to reflect a level of sensitivity that is compatible with enforcement activities. Where analytical methods applied had different LOQs, the lowest value was chosen only if the nil residue could be expected. In this case, the High Residue value would be recommended at the highest LOQ used in the study unless a majority of the observations were derived from the more sensitive LOQ.

In situations where supervised trials from GAP showed nil residues, even at exaggerated rates, the MRL was chosen to reflect an LOQ that is compatible with enforcement activities. However, both the STMR and high residue values were recommended at zero.

Banana

Thirty six field trials were submitted to the Meeting from banana producing areas of the world including Australia, Costa Rica, Ecuador, Honduras, Panama, Philippines and Mexico. In the trials 100 g ai/kg (10G) or 150 g ai/kg (15G) granule (G) terbufos was applied to the soil at the base of daughter banana plants at 1-9 g ai/plant/application. Application rates varied with a maximum rate of application per plant per year at of 41 g ai. GAP application rates ranged from 2-4 g ai/plant with a maximum of 12 g ai/year in Australia and Central America, 2 g ai/plant with a maximum of 8 g ai/year in Philippines and 3g ai/plant to the maximum of 9 g ai/year in Mexico. No PHI was specified in the various national GAPs.

Residue levels ranged from <LOQ (< 0.01 or < 0.002) to 0.03 mg/kg for those trials where substantially exaggerated rates (2-3 times GAP) were applied. However, the majority of the trials did not conform to GAP. The residues from trials that were conducted according to GAP were $\underline{< 0.01}$(6) and 0.02(2) mg/kg.

The meeting estimated a maximum residue level for bananas of 0.05 mg/kg, and STMR of 0.01 mg/kg and a HR of 0.02 mg/kg.

Sugar beets (roots)

Field trials involving at-planting and post-emergence treatments with terbufos were made available to the Meeting from the USA. The trials were conducted during the 1986, 1989 and 1994 growing seasons. In 1986, terbufos (15G) was applied at planting (banded, knifed-in, or in-furrow) at 2.2 kg ai/ha. In the trials conducted in 1989, terbufos (15G) was knifed in as a band at planting at 4.9 kg ai/ha, in excess of the current US GAP. Residues reflecting GAP were < 0.01(6) and 0.01(2) mg/kg where the PHI was considered equivalent to GAP, i.e., from 91-141 days.

In more recent field trials (1994), terbufos (15G) was applied as a band over the row to sugar beets at 2.2 to 2.4 or 4.4-4.9 kg ai/ha. The lower rate reflects the maximum GAP rate. Again, residues reflecting GAP were < 0.01(5) mg/kg. The PHI was considered equivalent to GAP at 90 days.

For knifed-in applications data was available at only 2 times the GAP rate where low finite residues could be found in some cases (< 0.01, 0.01, 0.02 and 0.03). Another knifed-in application trial had residues at < 0.01. The PHI for these trials ranged from 139-180 days (GAP is 150 days).

For all trials conducted according to GAP, total terbufos-related residues were: < 0.01(11) and 0.01(2) mg/kg.

The meeting withdrew its previous recommendation of 0.1 mg/kg and estimated a maximum residue level for sugar beets of 0.02 mg/kg, an STMR of 0.01 mg/kg and a highest residue of 0. 01 mg/kg.

Sweet corn Kernels and Corn-on-the Cob

Field trials involving at-planting and post-emergence treatments with terbufos were made available to the Meeting from the USA. In trials from 1972–1974, terbufos granules were applied in the furrow or in a band at the time of planting at rates of 1.1 to 9.0 kg ai/ha. In 1986 terbufos granules were applied to the soil at planting (in furrow or in a band), at post-emergence or at cultivation at a combined rate of about 6.0 kg ai/ha. GAP in the USA for 15G or 20G (200 g ai/kg) terbufos formulations is at a maximum rate of 1.5 kg ai/ha applied once at planting, post-emergent, or at cultivation. For post-emergent applications, the PHI is 30 days for forage, and 60 days for corn-on-the cob.

For post-emergent use, samples were analysed only where the PHI was less than that for GAP. Residue values from the majority of trials (7) were lower than the LOQ (0.01 mg/kg). For two trials, where the equivalent of three times the GAP rate was applied in two applications, residues found were 0.01 mg/kg (2).

The meeting withdrew its previous recommendation of 0.01 (*) mg/kg and estimated a maximum residue level for sweet corn of 0.01(*) mg/kg, an STMR of 0.01 mg/kg and a HR of 0.01 mg/kg.

Cereal Grains

Maize grain

Field trials involving at-planting and post-emergence treatments with terbufos were made available to the Meeting from the USA. GAP in the USA for terbufos 15G or 20G formulations is at the maximum rate of 1.5 kg ai/ha applied once at planting, post-emergent, or at cultivation. A PHI of 30 days is required for forage if applied post-emergent. In trials conducted from 1981 to 1986, terbufos granules were applied to the soil at planting, either in furrow or as a band, at the rate of 1.1 to 1.8 kg ai/ha. In some trials, additional plots were treated with terbufos at rates up to five times the recommended label rates. In trials conducted from 1990 to 1996 terbufos granules were applied post-emergent at the

recommended rate of 1.5 kg ai/ha as well as at higher rates up to five times the recommended application rates.

In all the trials conducted on maize grain according to GAP, total terbufos-related residues were below the LOQ of the analytical method: < 0.01 mg/kg (13). In trials where higher rates of application or more than one application was made, the residue levels were also below the LOQ. Since there were finite residues found in the trials for sweet corn at exaggerated rates, the use pattern for maize grain is not considered a nil residue situation and relevant values for STMR and HR have been proposed.

The meeting confirmed its previous recommendation for a maximum residue level of 0.01(*) mg/kg and estimated an STMR of 0.01 mg/kg and a highest residue of 0.01 mg/kg for maize.

Sorghum grain

Field trials involving at-planting and post-emergence treatments with terbufos were made available to the Meeting from the USA. GAP in the USA for terbufos 15G or 20G formulation consists of a maximum rate of 2.0 kg ai/ha applied once with a PHI of 50 days for forage, and 100 days for grain and fodder.

Results of all trials conducted according to the GAP for sorghum grain, including post-emergent applications, showed total terbufos-related residues below the LOQ: < 0.01 mg/kg (5). Residues were at non-detectable levels even in trials where higher rates of application or shorter PHI 58-76 days (6 trials) were used.

The meeting estimated a maximum residue level for sorghum grain of 0.01(*) mg/kg, an STMR of 0.

Coffee beans

Residue trials were conducted during 1982–1988 in Costa Rica, Guatemala, and El Salvador.

In field trials in Costa Rica conducted in 1982–1983, a 10G granular formulation of terbufos was applied to the soil at the base of established coffee plants at the rate of 0.75–7.5 g ai/plant. Berries were collected from treated plants at various intervals, field dried according to common practice, and the outer shell removed from the dried beans.

In the trials in El Salvador and Guatemala (1988), terbufos (10G) was band applied to plants after flowering but before bean formation, at the rate of 1 or 5 g ai/plant. From treated plants field dried berries, with outer shell removed, were collected at 38–56 days in El Salvador and at 163-197 days in Guatemala.

GAP in coffee bean plantations permits the application of terbufos at a maximum rate of 1.1g ai/plant for up to 2 applications with a PHI of 60 days. No trials were conducted at the maximum GAP. However, residue levels were below the LOQ (< 0.05 mg/kg) in all coffee bean samples (10) collected 58–120 days after treatment with terbufos at 0.75–7.75 g ai/plant rate. At one site, where coffee beans had been treated with 3.75 and 7.5 g ai/plant and shorter than GAP PHI of 60 days (47 or 35 days after treatment), maximum residues of 0.12 and 0.17 mg/kg respectively, were found. Residues declined to < 0.05 mg/kg at the next sampling interval, 124 or 53 days post-treatment.

The meeting confirmed its previous recommendation for a maximum residue level of 0.05 (*) mg/kg and estimated an STMR of 0.05 mg/kg for coffee beans.

Animal feed commodities

Fodder and forage of cereal grains

As maize forage, sorghum forage and sugar beet tops are not moving in international trade the Meeting made no recommendations regarding maximum residue levels for these commodities.

Maize forage and fodder

The GAP for terbufos 15G or 20G formulation in the USA allows for a maximum application rate of 1.5 kg ai/ha applied once either at-planting, early post-emergence, or at cultivation. A PHI of 30 days is required for forage when applied post-emergent. The same GAP applies to both maize and sweet corn. Trials on maize and sweet corn for residues in fodder and forage were conducted in the USA during 1972–1990. Terbufos granules were applied to the soil either in-furrow or in a band during planting at the rate of 1.1 – 5.8 kg ai/ha. In a few trials, tests were performed where two applications were made to maize one at planting and a second treatment 5–6 weeks after planting.

The residues deriving from trials conducted in sweet corn and maize were found to represent similar populations which could be combined (Mann-Whitney U-test). Residues, on a fresh weight basis, from trials conducted according to GAP were, with median underlined, $\underline{\leq 0.05}$ (11), 0.07(2), 0.14, 0.16, 0.17, 0.23, 0.32 and 0.96 mg/kg. The highest residue value (HR) was 0.96 mg/kg from trials in Colorado, USA from forage samples taken 90 days after treatment at planting at a rate of 1.5 kg ai/ha. Applying the default percent dry matter content (average between %DM of sweet corn forage and field corn forage, as listed in the *FAO Manual* (FAO, 2002) for maize forage (44%)), the highest residue on dry weight basis is estimated as 2.2 mg/kg.

The Meeting withdrew its previous recommendation of 1 mg/kg and estimated an STMR of 0.10 mg/kg and a highest residue of 2.2 mg/kg for maize forage.

Residue levels from trials according to GAP for maize fodder were: < LOQ i.e., < 0.05(38) and 0.08 mg/kg (from one trial in Colorado, USA, sampled at harvest after treatment at the rate of 1.5 kg ai/ha at planting). Applying the default percent dry matter value of 83% for corn fodder, as listed in the *FAO Manual* (FAO, 2002), the highest residue on dry weight basis was calculated as 0.10 mg/kg.

The meeting withdrew its previous recommendation of 0.1 mg/kg and estimated, on a dry weight basis, a maximum residue level of 0.2 mg/kg, an STMR of 0.06 mg/kg and a highest residue of 0.10 mg/kg for maize fodder.

Sorghum forage and fodder

Supervised trials on sorghum were conducted during 1978–1996. In the 1996 trials, terbufos granules were applied post-emergent, at the rate of 2.1 or 2.2 kg ai/ha. Forage samples were harvested 48 to 72 days after treatment while fodder samples were taken at normal grain harvest time, 88 to 90 days after treatment. In the rest of the trials (1978–1991), terbufos granules were applied at planting, at the GAP rate (2 kg ai/ha) and at twice that rate (4 – 4.3 kg ai/ka).

All trials according to the GAP resulted in residues below the LOQ for sorghum forage ($\underline{\leq 0.05}$ mg/kg), except one trial (Louisiana, USA) where a level of 0.07 mg/kg was recorded. This highest residue value was from forage samples taken 50 days after treatment with terbufos at a rate of 2.0 kg ai/ha at the vegetative stage. The moisture content of samples was only determined from some trials with the results showing wide variations. The Meeting therefore decided to use the default percent dry matter for sorghum forage (35%), as listed in the *FAO Manual* (FAO, 2002) to estimate the highest residue value.

The Meeting estimated an STMR for sorghum forage, on a dry weight basis, of 0.14 mg/kg and a highest residue of 0.20 mg/kg.

Residue levels in sorghum fodder ranged from < 0.05 to 0.19 mg/kg. Residues from trials conducted according to GAP were < 0.05 (12), 0.12 and 0.19 mg/kg. The highest residue value was 0.19 mg/kg from trials where fodder samples were taken 88 days after a post-emergent treatment at a rate of 2.2 kg ai/ha. Applying the default percent dry matter for sorghum fodder/stover of 88%, as listed in the *FAO Manual* (FAO, 2002), the highest residue on dry weight basis was estimated as 0.22 mg/kg.

The Meeting estimated, on a dry weight basis, a maximum residue level of 0.3 mg/kg, an STMR of 0.057mg/kg and a highest residue of 0.22 mg/kg for sorghum fodder.

Sugar beet tops

Field trials were conducted in the USA and Canada during 1971–1975 in which terbufos (15G) was either applied in-furrow or banded at 1.0 to 2.5 kg ai/ha or at exaggerated rates of 4.0–12.3 kg ai/ha. Several trials were also conducted which consisted of sequential at-planting and post-emergence banded applications, typically utilizing exaggerated rates.

Several trials were also conducted in the USA during the 1989 growing season in which terbufos (15G) was knifed in at-planting at 4.9 kg ai/ha. Samples were harvested by hand at maturity, 150–180 days after treatment. Residues found in all control samples of tops were < 0.05 mg/kg.

In US field trials in 1994, terbufos (15G) was applied as a band over the row to sugar beets at the maximum GAP rate of 2.2 to 2.4 kg ai/ha and at 2× GAP rates of 4.4 to 4.9 kg ai/ha. Residue levels ranged from < LOQ (0.01 or < 0.05) to 0.82 mg/kg for sugar beet tops samples. Residues found from trials conducted according to GAP were < 0.01(3), 0.01, 0.04, ≤ 0.05 (18), 0.12, 0.15 and 0.82 mg/kg. The highest residue value (HR) found was 0.82 mg/kg, from samples taken 91 days following an at-planting treatment of 1.8 kg ai/ha Applying the default percent dry matter for sugar beet tops (23%), as listed in the *FAO Manual* (FAO, 2002), the highest residue on dry weight basis was estimated as 3.57 mg/kg.

The Meeting withdrew its previous recommendation for a maximum residue level of 1 mg/kg for fodder beet leaves or tops and estimated, on a dry weight basis, an STMR of 0.22 mg/kg for sugar beet tops and a highest residue of 3.6 mg/kg.

Dietary burden in farm animals

The Meeting estimated the dietary burden of terbufos residues in farm animals on the basis of the diets listed in Appendix IX of the *FAO Manual* (FAO, 2002). One feed commodity from each Codex Commodity Group was used. Calculation from the HR values provides the concentrations in feed suitable for estimating MRLs for animal commodities, while calculation based on STMR values for feed is suitable for estimating the STMR values for animal commodities.

Table 49. Estimated maximum dietary burden of farm animals.

Commodity	Codex group	Residue (mg/kg)	Basis	DM (%)	Residue, dry wt. (mg/kg)	*Diet content (%)*			Residue Contribution (mg/kg)		
						Beef cattle	*Dairy cows*	*Poultry*	Beef cattle	Dairy cows	Poultry
Maize forage	AF	0.96	highest residue	44%	2.2	*40%*	*50%*	*NU*	0.88	1.1	
Maize fodder	AS	0.08	highest residue	83%	0.10			*NU*			
Maize grain	GC	0.01	highest residue	88%	0.011	*40%*	*40%*	*80%*	0.004	0.004	0.009
Sorghum	GC	0.0	highest residue	86%	0			*20%*			0
Sorghum forage	AF	0.07	highest residue	35%	0.20			*NU*			
Sorghum fodder	AS		highest	88%	0.22			*NU*			

Commodity	Codex group	Residue (mg/kg)	Basis	DM (%)	Residue, dry wt. (mg/kg)	*Diet content (%)*			Residue Contribution (mg/kg)		
						Beef cattle	*Dairy cows*	*Poultry*	Beef cattle	Dairy cows	Poultry
Sugar beet tops	AV	0.19 0.82	residue highest residue	23%	3.60	*20%*	*10%*	*NU*	0.72	0.36	
TOTAL						*100%*	*100%*	*100%*	1.60	1.47	0.009

Table 50. Estimated median dietary burden of farm animals.

Commodity	Codex group	Residue (mg/kg)	Basis	DM (%)	Residue, dry wt. (mg/kg)	Diet content (%)			Residue Contribution (mg/kg)		
						Beef cattle	Dairy cows	Poultry	Beef cattle	Dairy cows	Poultry
Maize forage	AF	0.05	STMR	44%	0.10	40%	50%	NU	0.04	0.05	
Maize fodder	AS	0.05	STMR	83%	0.06			NU			
Maize grain	GC	0.01	STMR	88%	0.011	40%	40%	80%	0.004	0.004	0.009
Sorghum	GC	0.0	STMR	86%	0.0			20%			0
Sorghum forage	AF	0.05	STMR	35%	0.14			NU			
Sorghum fodder	AS	0.05	STMR	88%	0.057			NU			
Sugar beet tops	AV	0.05	STMR	23%	0.22	20%	10%	NU	0.044	0.022	
TOTAL						100%	100%	100%	0.088	0.076	0.009

Estimation of Dietary Burdens

The highest residues or STMR values for feed commodities were used in calculating the worst-case dietary burden for dairy cows, beef cattle and poultry while the STMR values were used in the estimation of the median dietary burdens. The respective dietary burdens were then compared with the results of the feeding studies at various dose levels (mg/kg in diet) to estimate the maximum residue levels and STMR in animal commodities.

The dietary burdens of terbufos for estimates of STMR and highest residue level values in animal commodities (residue levels in animal feeds expressed as dry weight) are 0.088 mg/kg and 1.60 mg/kg for beef cattle, 0.076 mg/kg and 1.47 mg/kg for dairy cows and 0.009 mg/kg and 0.009 mg/kg for poultry.

Farm animal feeding studies

Feeding studies indicated that at a dose (2 ppm for 21 days) approximately equivalent to the calculated animal diets, no residues (< 0.05 mg/kg) of terbufos or its metabolites were detectable in cattle tissues and milk. In another study, done at an exaggerated rate (50 ppm), only one milk sample had a finite residue (0.011 mg/kg) while one sample had residue at the LOQ (0.005 mg/kg) and the rest were below the LOQ.

The Meeting received a feeding study in poultry. Hens were fed at 2 ppm terbufos for 21 days and residues were determined in poultry tissues and eggs. The LOQ was 0.05 and 0.01 mg/kg for tissues and eggs, respectively. All tissues and eggs samples contained residues below the LOQ value.

MAXIMUM RESIDUE LEVELS

The estimated maximum dietary burdens for beef cattle (1.60 mg/kg) and for dairy cows (1.47 mg/kg) matched the feeding level from the respective cattle feeding studies (2 mg/kg). As a result the Meeting decided to use the residue levels from the feeding studies as estimates of the maximum residue levels for cattle tissues and milk. Residues in cattle tissues and milk in the feeding studies were all below the LOQ (< 0.05 mg/kg for cattle fat, muscle, liver, and kidney, and < 0.01 mg/kg for milk). The

calculated median dietary burdens were lower than the actual feeding level in both transfer studies, 0.088 mg/kg for beef cattle and 0.076 mg/kg in dairy cattle therefore the calculated median residues would also be expected to be lower.

The actual feeding level of laying hens was (2 ppm for 21 days), the calculated maximum and median dietary burdens (0.009 ppm) were lower than the residue levels in both tissue and eggs. Consequently, no detectable residues are expected in both tissues and eggs. Therefore, residues are expected to be well below the LOQ for the method used (< 0.05 mg/kg for poultry tissues and < 0.01 mg/kg for eggs).

The calculations confirmed the findings of the animal metabolism studies as well as the results of the feeding studies, that showed no residues of terbufos or its metabolites were detectable in cattle tissues, poultry tissues, milk, and eggs. The MRL and STMR for residues of terbufos in animal commodities are proposed at the limit of quantification of the analytical method.

The Meeting withdrew its previous recommendation of 0.05 (*) mg/kg for cattle meat, cattle edible offal, chicken meat and chicken edible offal and 0.01 (*) mg/kg for cattle milk. The Meeting confirmed its previous recommendation of 0.01 (*) mg/kg for eggs and estimated a maximum residue level of 0.05 (*) mg/kg for meat from mammals other than marine mammals and mammalian edible offal, and 0.01(*) mg/kg for milks. The Meeting recommended an STMR of 0.05 mg/kg for mammalian meat and edible offal and poultry tissues and 0.01 mg/kg for milk and eggs. The estimated high residues are 0.05 mg/kg for mammalian meat, edible mammalian offal, chicken meat and edible chicken offal and 0.01 mg/kg for milks and eggs.

RECOMMENDATIONS

Definition of residue is for compliance with MRLs and for estimation of dietary intake for plant and animal commodities: the sum of terbufos, its oxygen analogue, and their sulfoxides and sulfones, expressed as terbufos.

Table 51. Summary of recommendations.

Commodity		MRL mg/kg		STMR ,	HR ,
CCN	Name	New	Previous	mg/kg	mg/kg
FI 0327	Banana	0.05	0.05	0.01	0.02
VB 0400	Broccoli	W	0.05(*)		
VB 0041	Cabbages, head	W	0.05(*)		
	Cattle meat	W	0.05 (*)		
	Cattle milk	W	0.01 (*)		
	Cattle, Edible offal of	W	0.05 (*)		
	Chicken meat	W	0.05 (*)		
	Chicken, Edible offal of	W	0.05 (*)		
SB 0716	Coffee beans	0.05(*)	0.05(*)	0.05	
PE 0112	Eggs	0.01(*)	0.01 (*)	0.01	0.01
AV 1051	Fodder beet leaves or tops	W	1		
GC 0645	Maize	0.01(*)	0.01(*)	0.01	
AF 0645	Maize forage,	W	1		
AS 0645	Maize fodder,(dry)	0.2	0.1		
MO 0105	Edible offal(mammalian)	0.05(*)	0.05(*)	0.05	0.05
MM 0095	Meat (from mammals other marine mammals)	0.05(*)	0.05 (*)	0.05	0.05
ML 0106	Milks	0.01(*)	0.01 (*)	0.01	
SO 0485	Mustard seed	W	0.05(*)		
VA 385	Onion, bulb	W	0.05(*)		
SO 0697	Peanut	W	0.05(*)		

Commodity		MRL mg/kg		STMR ,	HR ,
CCN	Name	New	Previous	mg/kg	mg/kg
AL 0697	Peanut fodder	W	1		
AL 1270	Peanut forage (green)	W	1		
	Popcorn	W	0.01		
PM 0110	Poultry meat	0.05(*)		0.05	0.05
PO 0111	Poultry edible offal of	0.05(*)		0.05	0.05
PE 0112	Eggs	0.01(*)		0.01	0.01
SO 0495	Rape seed	W	0.05(*)		
	Rape seed	W	0.05		
	Rape seed oil, Crude	W	0.05		
VD 0541	Soybeans (dry)	W	0.05(*)		
GC 0651	Sorghum	0.01(*)		0	
AS 0651	Sorghum straw and fodder,(dry)[1]	0.3			
AS 0081	Straw and fodder of cereal grains	W	1		
VR 0596	Sugar beet	0.02	0.1	0.01	0.01
VO 0447	Sweet corn (corn-on-the-cob)	0.01	0.01(*)	0.01	0.01
GC 0654	Wheat	W	0.01(*)		

1Expressed on dry weight basis

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Dietary Intakes (IEDIs) were calculated for the five GEMS/Food regional diets using the STMR for banana, coffee beans, edible offal (mammalian), eggs, maize (fresh, flour), meat from mammals other than marine mammals, milks, poultry meat, poultry edible offal, sorghum, sugar beet and sweet corn (corn on the cob) estimated by the current Meeting (Annex 3 of the 2005 JMPR Report). The ADI is 0–0.0006 mg/kg and the calculated IEDIs were 9–40% of the maximum ADI. The Meeting concluded that the intake of residues of terbufos resulting from the uses considered by the current JMPR were unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intakes (IESTIs) of terbufos by the general population and by children were calculated for commodities by the current Meeting (Annex 4 of the 2005 JMPR Report). This was based on HRs estimated by the Meeting from available information on consumption. The ARfD is 0.002mg/kg and the calculated IESTIs for children up to 6 years range from 0–60% and those for general population from 0–30% of the ARfD. The Meeting concluded that the short-term intake of residues of terbufos resulting from the uses considered by the current Meeting were unlikely to present a public health concern.

REFERENCES

Anonymous. (1973). Gas Chromatographic Procedure for the Determination of CL 92,100-Related Compounds in Sugar Beet Tops (Greens) and Roots. [TE-244-009]

Anonymous. (1986). Gas Chromatographic Determination of AC 92100 Related Compounds in Sugar beet Tops and Roots. [TE-244-063]

Babbitt, B. (1986). Counter Terbufos (CL 92,100): Validation of GC Method M-1615 for the Determination of Total CL 92,100 Related Residues in Drinking Water. Report C-2678. [TE-243-003]

Barringer, D. F. (1973). COUNTER ® Insecticide: The Identification of the Major Metabolites Found in Mature Sweet Corn Plants and in Soil, American Cyanamid Company, PD-M Volume 10, (Princeton). [TE-640-006]

Bohn, R. (1981). Counter Terbufos (CL 92,100): Validation of GC Method M-1144 for the Determination of Total CL 92,100 Related Residues in Water. Report C-1833. [TE-243-001]

Bohn, R. (1981). Counter Terbufos (CL 92,100): Validation of GC Method M-1149 for the Determination of CL 92,100, CL 94,301 and CL 94,320 Residues in Water. Report C-1892. [TE-243-002]

Bohn, W. (1979). COUNTER Terbufos CL 92,100: Solubility of CL 94, 301 (terbufos sulfoxide) and CL 94,320 (terbufos sulfone) in Distilled Water. Report C-1600. [TE-311-003]

Bohn, W. (1984). Counter (CL 92,100/10 G): Residues of Total CL 92,100-Related Compounds in Green Coffee Beans (Costa Rica, 1983). ARD Princeton, NJ. USA. Report C-2459. Unpublished. [TE-790-002]

Bohn, W. (1985). Counter Terbufos (CL 92,100/10-G): Residues of Total CL 92,100-Related Compounds in Whole Bananas (Costa Rica; 1984). ARD Princeton, NJ. USA. Report C-2622. Unpublished. [TE-714-007]

Bohn, W. (1985). Counter Terbufos (CL 92,100/10-G): Residues of Total CL 92,100-Related Compounds in Whole Bananas (Costa Rica; 1984). ARD Princeton, NJ. USA. Report C-2621. Unpublished. [TE-714-008]

Bohn, W. (1984). Counter Terbufos (CL 92,100/10-G): Residues of Total CL 92,100-Related Compounds in Whole Bananas (Costa Rica; 1984). ARD Princeton, NJ. USA. Report C-2493. Unpublished. [TE-714-009]

Bohn, W. (1984). Counter Terbufos (CL 92,100/10-G): Residues of Total CL 92,100-Related Compounds in Whole Bananas (GND; Costa Rica; 1984). ARD Princeton, NJ. USA. Report C-2494. Unpublished. [TE-714-012]

Bohn, W. and Behm, J. (1985). Counter Terbufos (CL 92,100/10G): Residues of Total CL 92,100-Related Compounds in Bananas (Costa Rica, 1984). ARD Princeton, NJ. USA. Report C-2622. Unpublished. [TE-714-013]

Bohn, W. (1986). Counter Terbufos (CL 92,100/10-G): Residues of Total CL 92,100-Related Compounds in Whole Bananas (Costa Rica, 1986). ARD Princeton, NJ. USA. Report No. C-2793. Unpublished. [TE-714-001]

Bohn, W. (1986). Counter Terbufos (CL 92,100/10-G): Residues of Total CL 92,100-Related Compounds in Whole Bananas (Costa Rica; 1984). ARD Princeton, NJ. USA. Report C-2674. Unpublished. [TE-714-006]

Bohn, W. (1986). Counter Terbufos (CL 92,100/10-G): Residues of Total CL 92,100-Related Compounds in Whole Bananas (Costa Rica; 1986). ARD Princeton, NJ. USA. Report C-2792. Unpublished. [TE-714-011]

Bohn, W. (1987). Counter Terbufos (CL 92,100): Validation of GC Method M-1747 For The Determination of Total CL 92,100-Related Residues in Sugar Beet Roots (0.01 ppm). Report C-2973. [TE-244-007]

Bohn, W. and Zummo, G. (1988). Terbufos 15-G: Residues of Total CL 92,100-Related Compounds in Sugar Beet Roots (Colorado). . ARD Princeton, NJ. USA. Report C-3064. Unpublished. [TE-724-064]

Bohn, W., and Zummo, G. (1988). Terbufos 15-G: Residues of Total CL 92,100-Related Compounds in Sugar Beet Roots (North Dakota). . ARD Princeton, NJ. USA. Report C-3065. Unpublished. [TE-724-065]

Bohn, W., and Zummo, G. (1988). Terbufos 15-G: Residues of Total CL 92,100-Related Compounds in Sugar Beet Roots Idaho). . ARD Princeton, NJ. USA. Report C-3066. Unpublished. [TE-724-066]

Bohn, W., and Zummo, G. (1988). Terbufos 15-G: Residues of Total CL 92,100-Related Compounds in Sugar Beet Roots (Minnesota). ARD Princeton, NJ. USA. Report C-3067. Unpublished. [TE-724-067]

Brindle, P. (1990). Terbufos (CL 92,100): Metabolic Fate of Carbon-14 CL 92,100 in Tissues, Blood and Eggs of the Laying Hen, PD-M Volume 27-13, (Princeton). [TE-440-003]

Brindle, C. (1990). Terbufos 10G: Residues of Total CL 92,100-Related Compounds in Dry Processed Coffee (Guatemala, 1988). ARD Princeton, NJ. USA. Report C 3380. Unpublished. [TE-790-004]

Brindle, C. (1990). Terbufos 10G: Residues of Total CL 92,100-Related Compounds in Dry Processed Coffee (El Salvador, 1988.). ARD Princeton, NJ. USA. Report C 3379. Unpublished. [TE-790-005]

Caballa, S. (1973). COUNTER® Insecticide (15-G): Metabolism of Carbon-14 Labeled CL 92,100 in Sugar Beets. American Cyanamid Company. Report C-405. [TE-640-010]

Caballa, S. (1974). COUNTER® Insecticide (15-G): Metabolism of Carbon-14 Labeled CL 92,100 in Sugar Beets. PD-M 11: 418-440 (Princeton). [TE-640-003]

Cheng, T., (1992). Metabolism of 14C-Terbufos (CL 92,100) in Rats (Preliminary and Definitive Phases), Princeton, NJ. [TE-440-004]

Chiu, T. Y. (1980). COUNTER® Insecticide (CL 92,100): Metabolism of Carbon-14 CL 92,100 in Rape, PD-M Volume 17-8 (Princeton). [TE-640-007]

Chiu, T. (1981). COUNTER® Terbufos (CL 92,100): Metabolism of Carbon-14 Labeled CL 92,100 in Soybean. PD-M 18-19; 1-65 (Princeton). [TE-640-001]

Dixon, C. (1989). Freezer Stability of Total CL 92,100-Related Residues in Sugar Beet Tops and Roots. Report C-3298. [TE-326-012]

Dixon, C. (1990). Freezer Stability of Total CL 92,100 Related Residues in Corn Grain Plants and Straw. Report C-3299. [TE-326-014]

Dixon, C. (1990). Terbufos (CL 92,100): Analysis of Total CL 92,100-Related Residues in Bananas Treated with Counter 10G Insecticide. ARD Princeton, NJ. USA. Report C-3509. Unpublished. [TE-714-016]

Elenewski, C. and Walgenbach, P. (1982). Counter Terbufos (CL 92,100/15-G, 20G): Residues of Total CL 92,100-Related Residues in Corn Plants and Grain (North Carolina). American Cyanamid Company. Report C-2082. Unpublished. [TE-723-026]

Elenewski,C. (1983). Counter Terbufos (CL 92,100): Validation of GC Method M-1340 for the Determination of CL 92,100-Related Residues in Whole Bananas. Report C-2260. [TE-244-005]

Elenewski,C. (1983). Counter Terbufos (CL 92,100): Validation of GC Method M-1360 for the Determination of CL 92,100-Related Residues in Coffee Beans. Report C-2305. [TE-244-015]

Elenewski, C. and Walgenbach, P. (1983). Counter terbufos (CL 92,100/15-G): Residues of Total CL 92,100-Related Compounds in Field Corn Plants, Fodder and Grain (Nebraska). Report C-2215. American Cyanamid Company. Unpublished. [TE-730-018]

Fletcher, P. (1998). CL 92,100 (Terbufos): Total CL 92,100-Related Residues in Corn Grain after Treatment at Cultivation with COUNTER 15G Systemic Insecticide-Nematicide in Illinois. Report RES 96-084. American Cyanamid Company. Unpublished. [TE-730-051]

Fletcher, P. (1997). CL 92100 Terbufos: Total CL 92100-Related Residues in Sorghum Forage, Grain, and Stover after Treatment at Cultivation with Counter 15G Systemic Insecticide-Nematicide in Texas. American Cyanamid Company, NJ, USA. Report RES-96-077. [TE-730-052]

Fletcher, P. (1997). CL 92100 Terbufos: Total CL 92100-Related Residues in Sorghum Forage, Grain, and Stover after Treatment at Cultivation with Counter 15G Systemic Insecticide-Nematicide in Texas. American Cyanamid Company, NJ, USA. Report RES-96-078. [TE-730-053]

Fletcher, P. (1997). CL 92100 Terbufos: Total CL 92100-Related Residues in Sorghum Forage, Grain, and Stover after Treatment at Cultivation with Counter 15G Systemic Insecticide-Nematicide in Louisiana. American Cyanamid Company, NJ, USA. Report RES-96-079. [TE-730-054]

Fletcher, P. (1997). CL 92100 Terbufos: Total CL 92100-Related Residues in Sorghum Forage, Grain, and Stover after Treatment at Cultivation with Counter 15G Systemic Insecticide-Nematicide in Kansas. American Cyanamid Company, NJ, USA. Report RES-96-080. [TE-730-055]

Fletcher, P. (1997). CL 92100 Terbufos: Total CL 92100-Related Residues in Sorghum Forage, Grain, and Stover after Treatment at Cultivation with Counter 15G Systemic Insecticide-Nematicide in Illinois. American Cyanamid Company, NJ, USA. Report RES-96-081. [TE-730-056]

Fletcher, P. (1997). CL 92100 Terbufos: Total CL 92100-Related Residues in Sorghum Forage, Grain, and Stover after Treatment at Cultivation with Counter 15G Systemic Insecticide-Nematicide in Missouri. American Cyanamid Company, NJ, USA. Report RES-96-082. [TE-730-057]

Gross, J. (1990). Terbufos (CL 92,100): Characteristics of Terbufos CL 92,100 and Its Phosphorylated Metabolites through FDA Multiresidue Methods. Report C-3363. [TE-244-059]

Higham, J.W. (1973). Counter 15G: Total Counter (CL 92,100)-Related Residues in Sweet Corn (California, Illinois, Oregon and Wisconsin). Report C-417. American Cyanamid Company. Unpublished. [TE-723-002]

Higham, J. (1973). Counter (CL 92,100)-Related Residues in Field Corn Treated In-Furrow (Illinois, Iowa, Nebraska, New York, and North Dakota). American Cyanamid Company, NJ, USA. Report No. C-416. [TE-730-016]

Higham, J. (1973). Counter 15G: Total Counter (CL92100) Related Residues in Field Corn Treated "in a Band" (Indiana, Iowa, Missouri, Nebraska, New York, North Carolina, North Dakota and South Dakota). Report C-415. American Cyanamid Company. Unpublished. [TE-730-017]

Higham, J. (1974). Counter 15G: Total Counter (CL 92100) Related Residues in Irrigated Field Corn, Sweet corn and Popcorn (Colorado). Report C-425. American Cyanamid Company. Unpublished. [TE-730-002]

Higham, J. (1974). Counter (CL 92,100): The Gas Chromatographic Determination of Total CL 92,100 Related Residues in Fortified Sugar Beet Tops (Greens) and Roots. ARD Princeton NJ USA. Report C-532. [TE-244-004]

Higham, J. (1975). Counter (CL 92,100): The Gas Chromatographic Determination of CL 92,100 Related Residues in Fortified Corn Silage Corn Fodder Field Corn Grain Popcorn Grain and Sweet Corn Kernels Plus Cob With Husk Removed. ARD Princeton NJ USA. Report C-607. [TE-244-054]

Higham, J. (1975). Counter: Determination Of Total CL 92,100 Related Residues In Sugar Beet (Tops And Roots) (North Dakota). ARD Princeton, NJ. USA. Report C-694. [TE-724-014]

Higham, J. (1975). Counter: Determination Of Total CL 92,100 Related Residues In Sugar Beet Tops And Roots Following Band, In-Furrow And Post Emergent Applications (ND). ARD Princeton, NJ. USA. Report C-695. [TE-724-016]

Higham, J. and Alvarez, C.G. (1975). Counter (CL 92,100): Determination of CL 92,100(S-[[(1,1-dimethylethyl) thio]methyl] O,O-diethyl phosphorodithioate) Related Residues in Sweet Corn Plant and Ears following "In-furrow" Treatment at Planting (Florida). Report C-632. American Cyanamid Company. Unpublished. [TE-723-014]

Higham, J., and Alvarez, C. (1975). Counter CL92100 Determination Of Total CL92100 Related Residues In Sugar Beet Tops And Roots (North Dakota). ARD Princeton, NJ. USA. Report C-656. [TE-724-048]

Higham, J. and Alvarez, C. (1975). Counter CL92100 Determination Of Total CL92100 Related Residues In Sugar Beet Tops And Roots (Wyoming). ARD Princeton, NJ. USA. Report C-657. [TE-724-049]

Higham, J. and Nau, H. (1975). Counter CL92100 Determination Of Total CL92100 Related Residues In Sugar Beet Tops And Roots (Minnesota). ARD Princeton, NJ. USA. Report C-696. [TE-724-050]

Higham, J. and Nau, H. (1975). Counter (CL92100): Determination of CL92100 (S-[[(1,1-dimethylethyl) thio]methyl] O,O-diethyl phosphorodithioate) Related Residues In Field Corn Following "In-Furrow" Application (Minnesota). Report C-631. American Cyanamid Company. Unpublished. [TE-723-013]

Higham, J. and Nau, H. (1975). Counter (CL92100): Determination of CL92100(S-[[(1,1-dimethylethyl) thio]methyl] O,O-diethyl phosphorodithioate) "Terbufos" Residues in Corn. (North Dakota). Report C-637. American Cyanamid Company. Unpublished. [TE-723-017]

Higham, J. and Nau, H. (1975). Counter (CL92100): Determination of CL92100 (S-[[(1,1-dimethylethyl) thio]methyl] O,O-diethyl phosphorodithioate) Related Residues in "No-Tillage" Field Corn Following "In-Furrow" Treatment at Planting (Kentucky). Report C-647. American Cyanamid Company. Unpublished. [TE-723-022]

Higham, J. and Nau, H. (1975). Counter (CL 92,100): Determination of CL 92,100 (S-[[(1,1-dimethylethyl)thio] methyl] 0,0-diethyl phosphorodithioate) Related Residues in Field Corn Following Treatments at Planting and at Cultivation Time (Colorado). American Cyanamid company, NJ, USA. Report No. C-645. [TE-730-015]

Higham, J. (1984). Counter Terbufos (CL 92,100/10-G): Residues of Total CL 92,100-Related Compounds in Whole Bananas (Costa Rica; 1983). ARD Princeton, NJ. USA. Report C-2438. Unpublished. [TE-714-010]

Higham, J. (1987). Counter Terbufos (CL 92,100): Validation of GC Method M-1754 for the Determination of Total CL 92,100 Related Residues in Corn Commodities (Field Corn Grain Sweet Corn Kernels Plus Cob Cannery Waste and Green and Dry Corn Plants) ABC Lab. Report C-2961. [TE-244-049]

Higham, J. (1988). Terbufos (CL 92,100): Validation of GC Method M-1912 for the Determination of CL 92,100-Related Residues in Soil. Report C-3544. [TE-242-006]

Higham, J. and Fisher, R. (1988). CL 92,100 (Terbufos/15-G): Residues of Total CL 92,100-Related Compounds in Sweet Corn Kernels plus Cob (Wisconsin). Report C-3096. American Cyanamid Company. Unpublished. [TE-723-004]

Higham, J. and Fisher, R. (1988). CL92100 (terbufos/15-G): Residues of Total CL92100 Related Residues In Sweet Corn Kernels Plus Cob (BAND + POST; FL, 1986 (C-2961). Report C-3109. American Cyanamid Company. Unpublished. [TE-723-006]

Higham, J. and Fisher, R. (1988). CL 92,100 (Terbufos/15-G): Residues of Total CL 92,100-Related Compounds in Field Corn Grain (Minnesota). Report C-3095. American Cyanamid Company. Unpublished. [TE-730-012]

Higham, J. and R. Fisher. 1988. CL92100 (terbufos/15-G): Residues of Total CL92100 – Related Compounds In Sweet Corn Kernels Plus Cob (BAND + POST; NY, 1986) C-2961 [TE-723-036]

Higham, J. and Zummo, G. (1988). CL 92,100 Terbufos (15-G, 20-PG): Residues of Total CL 92,100-Related Compounds in Green Corn Plants, Dry Corn Plants and grain (Illinois). Report C-3038. American Cyanamid Company. Unpublished. [TE-730-010]

Higham, J. and Zummo, G. (1988). CL 92,100 Terbufos (15-G, 15-PG, 20-PG): Residues of Total CL 92,100-Related Compounds in Green Corn Plants, Dry Corn Plants and Grain (Nebraska). (C-2961). Report C-3037. American Cyanamid Company. Unpublished. [TE-730-011]

Higham, J. (1991). Terbufos (CL 92,100): Validation of GLC Method M-1912 for the Determination of Terbufos (CL 92,100) and Related Residues (CL 93,401 and CL 94,320) in Soil. . American Cyanamid Company, NJ, USA. Report No. C-3544. [TE-242-006]

Higham, J. (1996). Terbufos Validation of Method M 2457 for the Determination of Total CL 92,100 Related Residues in Sugar Beet Tops and Roots. RES 95-118. [TE-244-023]

Higham, J. (1997). Freezer Storage Stability of Residues of Terbufos-Related Compounds (CL 94,301, CL 94,320, and CL 94,221) in Sugar Beet Tops and Roots. American Cyanamid Company. USA. Unpublished. Report RES 97-017. [TE-326-007]

Horton, W., and Kleiner, A. (1998). Total CL 92,100-Related Residues in Bananas after Treatment with Counter 15G Systemic Insecticide-Nematicide in Ecuador. Central Analytical Labs State College, PA. USA. Report RES 97-062. Unpublished. [TE-714-024]

Horton, W., and Kleiner, A. (1998). Total CL 92,100-Related Residues in Bananas after Treatment with Counter 15G Systemic Insecticide-Nematicide in Ecuador. Central Analytical Labs State College, PA. USA. Report RES 97-063. Unpublished. [TE-714-025]

Horton, W. and Kleiner, A. (1998). Total CL 92,100-Related Residues in Bananas after Treatment with Counter 15G Systemic Insecticide-Nematicide in Mexico. Central Analytical Labs State College, PA. USA. Report RES 97-064. Unpublished. [TE-714-026]

Hui, T. 1973. COUNTER Soil Insecticide: Soil-Leaching Studies of CL 92,100, ARD Princeton NJ USA Project #2-402 [TE-620-005]

Jones, A.(2005). Validation of Analytical Methodology to Determine Terbufos and Metabolites in Whole Banana and Banana Pulp. Central Science Laboratory, UK. Exponent International. Report No. PGD-183. [PGD-183]

Khunachak, A. (1986). Counter Terbufos (CL 92,100/10-G): Residues of Total CL 92,100-Related Compounds in Whole Bananas (Honduras; 1986). ARD Princeton, NJ. USA. Report C-2789. Unpublished. [TE-714-002]

Khunachak, A. (1986). Counter Terbufos (CL 92,100/10-G): Residues of Total CL 92,100-Related Compounds in Whole Bananas, Peel and Pulp (Honduras; 1985). ARD Princeton, NJ. USA. Report C-2706. Unpublished. [TE-714-003]

Khunachak, A. (1986). Counter Terbufos (CL 92,100/10-G): Residues of Total CL 92,100-Related Compounds in Whole Bananas (Costa Rica; 1986). ARD Princeton, NJ. USA. Report C-2705. Unpublished. [TE-714-004]

Khunachak, A. (1986). Counter Terbufos (CL 92,100/10-G): Residues of Total CL 92,100-Related Compounds in Whole Bananas, Peel and Pulp (Costa Rica; 1985). ARD Princeton, NJ. USA. Report C-2704. Unpublished. [TE-714-005]

Khunachak, A. (1998). CL 92,100/CL 35024: Validation of GC/MS Method M-2623 for the Determination of Residues of CL 35,024 and CL 92,100 and their Sulfoxides (CL 18,177 and CL 94,301) and Sulfones (CL 18161 and CL 94,320) in Water. American Cyanamid Company, NJ, USA. Report No. RES 97-074. [TE-243-004]

Khunachak, A. and Behm, J.(1988). Terbufos (CL 92,100/10-G): Residues of Total CL 92,100-Related Compounds in Whole Bananas (GND; Costa Rica; 1987). ARD Princeton, NJ. USA. Report C-3136. Unpublished. [TE-714-015]

Khunachak, A. (1997). Freezer Storage Stability of Residues of Terbufos CL 92,100 and Its Related Compounds CL 94,301 and CL 94,320 in Soil. Report RES 96-073.01. [TE-326-006]

Kleiner, A. (1990). Terbufos 15-G: Residues of Total CL 92,100-Related Compounds in Sugar beet Roots and Foliage (North Dakota). ARD Princeton, NJ. USA. Report C-3366. Unpublished. [TE-724-068]

Kleiner, A. (1990). Terbufos 15-G: Residues of Total CL 92,100-Related Compounds in Sugar beet Roots and Foliage (Nebraska). ARD Princeton, NJ. USA. Report C-3367. Unpublished. [TE-724-069]

Kleiner, A. (1990). Terbufos 15-G: Residues of Total CL 92,100-Related Compounds in Sugar beet Roots and Foliage (Idaho). ARD Princeton, NJ. USA. Report C-3368. Unpublished. [TE-724-070]

Kleiner, A. (1990). Terbufos 15-G: Residues of Total CL 92,100-Related Compounds in Sugar beet Roots and Foliage (California). ARD Princeton, NJ. USA. Report C3369. Unpublished. [TE-724-071]

Kleiner, A. (1990). CL 92,100 (terbufos/15G): Residues of Total CL 92,100-Related Compounds in Dual Purpose Sorghum. American Cyanamid, NJ, USA. Report No. C-3374. [TE-730-046]

Kleiner, A. (1990). CL 92,100 (terbufos/15G): Residues of Total CL 92,100-Related Compounds in Grain Sorghum. American Cyanamid, NJ, USA. Report No. C-3375. [TE-730-047]

Kleiner, A. (1990). CL 92,100 (terbufos/15G): Residues of Total CL 92,100-Related Compounds in Grain Sorghum. American Cyanamid, NJ, USA. Report No. C-3376. [TE-730-048]

Kleiner, A. (1992). CL 92,100 (terbufos/15G): Residues of Total CL 92,100-Related Compounds in Dual Purpose Sorghum. American Cyanamid, NJ, USA. Report No. C-3851. [TE-730-049]

Lee, T and Belcher, D (1986). COUNTER Terbufos (CL 92,100): Residues of CL 92,100-Related Compounds in Soil and Rotational Crops (cabbage, Red Beets, and Wheat) from a Treated Corn Field (Waunakee, WI, 1984). American Cyanamid Company, Princeton, NJ, USA. Report No. C-2658. [TE-790-030]

Lee, T and Belcher, D (1986). COUNTER Terbufos (CL 92,100): Residues of CL 92,100-Related Compounds in Soil and Rotational Crops (cabbage, Sugar Beets, and Wheat) from a Treated Corn Field (York, NE, 1984). American Cyanamid Company, Princeton, NJ, USA. Report No. C-2721. [TE-790-031]

Leonard, R. (1991). CL 92,100 (Terbufos/15G): Residues of Total CL 92,100-Related Residues in Field Corn after Application at the Maximum Labelled Use Rate and at 5X the Maximum Labelled Use Rate (Iowa). Report C-3566. American Cyanamid Company. Unpublished. [TE-723-032]

Leonard, R. (1991). CL 92,100 (Terbufos/15G): Residues of Total CL 92,100-Related Residues in Field Corn after Application at the Maximum Labelled Use Rate and at 5X the Maximum Labelled Use Rate (At Cultivation; IA, 1990). Report C-3567. American Cyanamid Company. Unpublished. [TE-723-033]

Leonard, R. (1991). CL 92,100 (Terbufos/15G): Residues of Total CL 92,100-Related Residues in Field Corn after Application at the Maximum Labeled Use Rate and at 5X the Maximum Labeled Use Rate (Illinois). Report C-3569. American Cyanamid Company. Unpublished. [TE-723-035]

Leonard, R. (1991). CL 92,100 (Terbufos/15G): Residues of Total CL 92,100-Related Residues in Field Corn after Application at the Maximum Labelled Use Rate and at 5X the Maximum Labelled Use Rate (At Cultivation; IL, 1990). American Cyanamid Company. Report C-3568. Unpublished. [TE-723-034]

Luckhowec, J. (1989). Pesticide Assessment Guidelines Subdivision D: Product Chemistry Requirements for the Technical Grade Active Ingredient (TGAI) and Manufacturing Use Product (MP) COUNTER terbufos: Section 63-2 to 63-21, Physical and Chemical Characteristics. American Cyanamid Company. [TE-301-007]

Manuel, A. (1972). CL 92,100 and its Metabolites in Chicken Fat, Muscle, Liver, Kidney, Skin, and Eggs. American Cyanamid Company, NJ, USA. Report No. C-372. [TE-705-001]

Manuel, A. (1972). CL 92,100 and its Metabolites in Cattle Fat, Muscle, Liver and Kidney. American Cyanamid Company, NJ, USA. Report No. C-337. [TE-705-002]

Manuel, A. (1972). CL 92,100 and its Metabolites in Milk; American Cyanamid Company, NJ, USA. Report No. C-336. [TE-705-003]

Manuel, A. (1973). Determination of Total CL 92,100 and Oxidative Metabolites in Chicken Fat, Muscle, Skin, Liver and Kidney by Gas Liquid Chromatography: Recommended Method of Analysis. ARD Princeton NJ USA. Report M-401. [TE-245-002]

Manuel, A. and Nau, H. (1975). Counter (CL92100): Determination of CL92100(S-[[(1,1-dimethylethyl) thio]methyl] O,O-diethyl phosphorodithioate "terbufos" residue in corn (Minnesota). Report C-639. American Cyanamid Company. Unpublished. [TE-730-003]

Manuel, A. and Nau, H. (1975). Counter (CL92100): Determination of CL92100 (S-[[(1,1-dimethylethyl) thio]methyl] O,O-diethyl phosphorodithioate "terbufos" residue in corn (Virginia). Report C-638. American Cyanamid Company. Unpublished. [TE-730-005]

Manuel, A., Peterson, R. and Nau, H. (1975). Counter (CL 92,100): Determination of CL 92,100 (S-[[(1,1-dimethylethyl) thio]methyl] O,O-diethyl phosphorodithioate "Terbufos" Residue in Sweet Corn and Popcorn(Iowa). Report C-642. American Cyanamid Company. Unpublished. [TE-723-003]

Manuel, A.J and Nau, H. (1975). Counter (CL 92,100): Determination of CL 92,100(S-[[(1,1-dimethylethyl) thio]methyl] O,O-diethyl phosphorodithioate "Terbufos" Residue in Corn (Minnesota). Report C-636. American Cyanamid Company. Unpublished. [TE-723-016]

Manuel, A. and Nau, H. (1975). Counter (CL92100): Determination of CL92100 (S-[[(1,1-dimethylethyl) thio]methyl] O,O-diethyl phosphorodithioate "terbufos" Residue in Corn (New York). Report C-641. American Cyanamid Company. Unpublished. [TE-723-019]

Manuel, A.J. and Nau, H.H. 1975. Counter (CL92100): Determination of CL92100 (S-[[(1,1-dimethylethyl) thio]methyl] O,O-diethyl phosphorodithioate "terbufos" residue in corn (Maryland). Report C-643. American Cyanamid Company. Unpublished, [TE-723-020]

Manuel, A. and Nau, H. (1975). Counter (CL92100): Determination of CL92100 (S-[[(1,1-dimethylethyl) thio]methyl] O,O-diethyl phosphorodithioate "Terbufos" Residue in Corn (Colorado). Report C-644. American Cyanamid Company. Unpublished. [TE-723-021]

Manuel, A. (1976). Counter 15G: Determination Of Total CL 92,100- Related Residues In Sugar Beet (Root Pulp And Tops) Manitoba, Canada, 1975. ARD Princeton, NJ. USA. Report C-964. [TE-724-012]

Miller, P. (1973). CL 92,100 COUNTER Insecticide: Metabolic Studies of 14-C-Labeled CL 92,100 in Hydrolytic and Photolytic Environments, ARD Princeton NJ USA PD-M Volume 10 P 959-1007. [TE-630-001]

Marin, C. and Heim, D. (1999). 14C-Terbufos (CL 82,100) and its Sulfoxide (CL 94,301) and Sulfone (CL 94,320) Metabolites: Hydrolysis in Sterile pH 5, 7, and 9 Buffers. American Cyanamid Company. Report No. ENV 97-017. [TE-630-005]

Minoura, M. (1986).). Counter Terbufos (CL 92,100): Residues of Total CL 92,100-Related Compounds in Whole Banana Fruits in the Philippines. Cyanamid Japan, Ltd. Report TTR-86-012. Unpublished. [TE-714-014]

Minoura, M. (1986). Counter Terbufos (CL 92,100): Residues of Total CL 92,100-Related Compounds in Whole Banana Fruits in The Philippines. Cyanamid Japan Ltd., Tahara, Japan. Report Ttr-86-011. Unpublished. [TE-714-023]

Minoura, M. (1987). Counter Terbufos (CL 92,100/10G): Residues of Total CL 92,100-Related Compounds in Banana Fruits in the Philippines. Cyanamid Japan Ltd., Tahara, Japan. Report TTR-87-025. Unpublished. [TE-714-022]

North H., Barringer, D., and Gatterdam, P. (1972). Fate of Carbon-14 Labeled CL 92,100 in Sweet Corn Plants. PD-M 9: 249-301 (Princeton). [TE-640-005]

North, H. (1973). COUNTER Soil Insecticide: Metabolism of CL 92,100 in Soils, ARD Princeton NJ USA Project #2-402 Oct 1973. BASF Ref. No. 1973/7000281. [TE-620-006]

Ohba, K. (1993). Terbufos (CL 92,100): Analysis of Total CL 92,100-Related Residues in Banana Pulp Treated With Counter 15G Insecticide/ Palmerston, Queensland, Australia (3g Ai Per Stool). Tahara Agricultural Center, Tahara Japan. Report TTR-93-005. Unpublished. [TE-714-018]

Ohba, K. (1993). Terbufos (CL 92,100): Analysis of Total CL 92,100-Related Residues in Banana Skin Treated With Counter 15G Insecticide/ Palmerston, Queensland, Australia (3g Ai Per Stool). Tahara Agricultural Center, Tahara Japan, Report TTR-93-006. Unpublished. [TE-714-019]

Ohba, K. (1993). Terbufos (CL 92,100): Analysis of Total CL 92,100-Related Residues in Banana Pulp Treated with Counter 15G Insecticide/ Palmerston, Queensland, Australia (6g ai/stool). Tahara Agricultural Center, Tahara, Japan. Report TTR-93-007. Unpublished. [TE-714-020]

Ohba, K. (1993). Terbufos (CL 92,100): Analysis of Total CL 92,100-Related Residues in Banana Skin Treated With Counter 15G Insecticide/ Palmerston, Queensland, Australia. Tahara Agricultural Center, Tahara, Japan. Report TTR-93-008. Unpublished. [TE-714-021]

Peterson, R. (1973). Gas Chromatographic Procedure for the Determination of CL 92,100-Related Compounds in Sugar Beet Tops Greens and Roots. Recommended Method of Analysis M-395. [TE-244-064]

Peterson, R. (1974). Counter: Determination Of Total CL 92,100 Related Residues In Sugar Beet Tops And Roots (Colorado). ARD Princeton, NJ. USA. Report C-667. [TE-724-004]

Peterson, R. (1975). Counter: Determination Of Total CL 92,100 Related Residues In Sugar Beet Tops And Roots (Wyoming). ARD Princeton, NJ. USA. Report C-666. [TE-724-005]

Peterson, R. (1975). Counter: Determination Of Total CL 92,100 Related Residues In Sugar Beet Tops And Roots (Idaho). ARD Princeton, NJ. USA. Report C-665. [TE-724-006]

Peterson, R. (1975). Counter: Determination Of Total CL 92,100 Related Residues In Sugar Beet Tops And Roots (Idaho). ARD Princeton, NJ. USA. Report C-664. [TE-724-007]

Peterson, R. (1975). Counter: (CL 92,100) Determination Of Total CL 92,100 Related Residues In Sugar Beet Tops And Roots (Michigan). ARD Princeton, NJ. USA. Report C-693. [TE-724-017]

Peterson, R. (1975). Counter: Determination Of Total CL 92,100 Related Residues In Sugar Beet Tops And Roots (Colorado). ARD Princeton, NJ. USA. Report C-668. [TE-724-018]

Peterson, R. and Nau, H. (1975). Counter (CL92100): Determination of total CL92100 (S-[[(1,1-dimethylethyl) thio)methyl] O, O-diethylphosphorodithioate) "terbufos" Related Residues in Corn (Michigan). Report C-640. American Cyanamid Company. Unpublished. [TE-723-018]

Peterson, R. (1976). COUNTER Insecticide 15 G and Liquid (CL 92,100): Metabolism of 14C-Labeled CL 92,100 in Cabbage Plants, American Cyanamid Company, PD-M Volume 13-1 (Princeton). [TE-640-004]

Peterson, R. (1976). Counter: Determination Of Total CL 92,100 Related Residues In Sugar Beets (Tops And Roots) (Winnipeg, Manitoba, Canada). ARD Princeton, NJ. USA. Report C-914. [TE-724-029]

Peterson, R. (1976). Counter: Determination Of Total CL 92,100 Related Residues In Sugar Beets (Tops And Roots) (Plum Coulee And Altona, Manitoba, Canada). ARD Princeton, NJ. USA. Report C-917. [TE-724-030]

Peterson, R. (1976). Counter: Determination Of Total CL 92,100 Related Residues In Sugar Beet (Root And Tops) (Gnadenthal, Manitoba, Canada). ARD Princeton, NJ. USA. Report C-916. [TE-724-013]

Peterson, R. and Byrne, D. (1977). Counter 15-G (CIr92100): Determination of total CL92100 Related Residues in no-Till Field Corn Tissues (Maryland, 1974). Report C-1129. American Cyanamid Company. Unpublished. [TE-730-008]

Peterson, R. and Baugher, D., (1979). Counter Terbufos (CL 92100/15G): Residues of Total CL 92,100-Related Compounds in Sorghum Silage, Fodder, and Grain . American Cyanamid Company. Report No. C-1626. [TE-730-039]

Peterson, R. (1979). Counter Terbufos (CL 92,100): Validation of GC Method M-0995 for the Determination of Total CL 92,100 Related Residues in Sorghum Tissues (Silage, Fodder, and Grain). American Cyanamid Company, NJ, USA. Report C-1592. [TE-244-056]

Peterson, R. (1981). COUNTER Terbufos (CL 92,100): Aerobic Radiobalance and Metabolism Study with Carbon-14 Labeled CL 92,100 in a Silt Loam Soil. ARD Princeton NJ USA. Report C-1857. [TE-620-002]

Peterson, R. 1983. COUNTER, terbufos (CL 92,100): Aerobic and Anaerobic Metabolism of CL 92,100 in a Silt Loam Soil, ARD Princeton NJ USA PD-M Volume 20-4. [TE-620-004]

Peterson, R. (1988). Validation of GC Method M-1829 For The Determination of Total CL 92,100 Related Residues in Cow's Milk. American Cyanamid Co., USA. Report C-3140. [TE-245-001]

Peterson, R. (1989). Terbufos/15G (CL 92,100): Residues of Total CL 92,100-Related Compounds in Cow's Milk (Feed; NJ, 1987). American Cyanamid Company, NJ, USA. Report No. C-3247. [TE-705-004]

Picard, G. and Kim, D. 1997. Terbufos GC/FPD Determinative Method and LC/MS/MS Confirmatory Method for Total Terbufos CL 92,100-Related Residues in Whole Banana and Banana Pulp. [TE-244-025]

Poepell, M. and Nau, H. (1975). Counter (CL 92,100): Determination of Total CL 92,100 (S-[[(1,1-dimethylethyl) thio)methyl] O, O-diethylphosphorodithioate) "Terbufos"-Related Residues in Sweet Corn Ears and Fodder (Iowa). Report C-635. American Cyanamid Company. Unpublished. [TE-723-005]

Potts, C. (1984). Counter (CL 92,100/10 G): Residues of Total CL 92,100-Related Compounds in Green Coffee Beans (Costa Rica, 1982) (C-2305). ARD Princeton, NJ. USA. Report C-2351. Unpublished. [TE-790-001]

Roman, M. and Lublinkhof, J. (1980). Counter Terbufos (CL 92100/15G): Residues of Total CL 92,100-Related Compounds in Sorghum Plant and Grain . American Cyanamid Company. Report No. C-1742. [TE-730-040]

Roman, M. and Lublinkhof, J. (1980). Counter Terbufos (CL 92100/15G): Residues of Total CL 92,100-Related Compounds in Sorghum Plant and Grain. American Cyanamid Company. Report No. C-1776. [TE-730-043]

Roman, M. and Lublinkhof, J. (1980). Counter Terbufos (CL 92100/15G): Residues of Total CL 92,100-Related Compounds in Sorghum Plant and Grain . American Cyanamid Company. Report No. C-1752. [TE-730-041]

Roman, M. and Lublinkhof, J. (1980). Counter Terbufos (CL 92100/15G, 15G/C): Residues of Total CL 92,100-Related Compounds in Sorghum. American Cyanamid Company. Report No. C-1773. [TE-730-042]

Roman, M. (1986). Counter Terbufos (CL 92,100): Validation of GC Method M-1644 for the Separate Determination of CL 92,100, CL 94,301, CL 94,320 Residues in Water. Report. C-2769. [TE-243-007]

Roman, M. (1987). Counter Terbufos (CL 92,100): Validation of GC Method M-1784 for the Separate Determination of CL 92,100 and Metabolites (CL 94,221, CL 94,301, and CL 94,320) in Soil. American Cyanamid Company, NJ. Report No. C-3020. [TE-242-003]

Roman, M. (1987). Freezer Stability of CL 92,100, CL 94,301 CL 94,320 Residues in Water. Report C-3032. [TE-326-010]

Skorczynski, S. (1991). Terbufos (CL 92,100/10G): Residues of Total CL 92,100-Related Compounds in Whole Bananas. ARD Princeton, NJ. USA. Report C-3614. Unpublished. [TE-714-017]

Shimel, K. (1986). Counter Terbufos (CL 92,100): Validation of GC Method M-1784 for the Separate Determination of Residues of CL 92,100 and Its Metabolites (CL 94,221, CL 94,301, CL 94,365, CL 94,302 and CL 94,320) in Soil. American Cyanamid Company, NJ. Report No. C-2766. [TE-242-001]

United State Department of Agriculture. Pesticide Data Program Annual Summary Calendar Years 2002 , page 40. Agricultural Marketing Service.

United States Environmental Protection Agency. (1999). Revised Environmental Fate and Effects Assessment (Section C: Environmental Assessment).

York, C. (1995). Total CL 92,100-Related Residues in Sugar Beets after Treatment with Counter 15 G Applied Post emergence (Michigan). CARC Princeton, NJ. USA. Report RES 95-039. Unpublished. [TE-724-036]

York, C. (1995). Total CL 92,100-Related Residues in Sugar Beets after Treatment with Counter 15 G Applied Post emergence (North Dakota). CARC Princeton, NJ. USA. Report RES 95-045. Unpublished. [TE-724-038]

York, C. (1995). Total CL 92,100-Related Residues in Sugar Beets after Treatment with Counter 15 G Applied Post Emergence (Minnesota). CARC Princeton, NJ. USA. Report RES 95-047. Unpublished. [TE-724-039]

York, C. (1996). Total CL 92,100-Related Residues in Sugar Beets after Treatment With Counter 15 G Applied Post emergence (Nebraska). CARC Princeton, NJ. USA. Report RES 95-040. Unpublished. [TE-724-037]

York, C. (1996). CL 92,100 (Terbufos/15G): Total CL 92,100-Related Residues in Corn Grain after Treatment with COUNTER 15G Insecticide Applied Post emergence (Wisconsin). Report RES 95-059. American Cyanamid Company. Unpublished. [TE-730-028]

York, C. (1996). CL 92,100 (Terbufos/15G): Total CL 92,100-Related Residues in Corn Grain after Treatment with COUNTER 15G Insecticide Applied Post emergence (MI, 1994). Report RES 95-058. American Cyanamid Company. Unpublished. [TE-730-029]

York, C. (1996). CL 92,100 (Terbufos/15G): Total CL 92,199-Related Residues in Sugar Beets after Treatment with Counter 15G Insecticide Applied Post-emergence (ID; 1994). American Cyanamid Company, NJ, USA. Report Norse 95-046. [TE-724-035]

York, C. (1997). CL 92,100 (Terbufos/15G): Total CL 92,100-Related Residues in Corn Grain after Treatment with COUNTER 15G Insecticide Applied Post emergence (IA, 1995). Report RES-96-021. American Cyanamid Company. Unpublished. [TE-730-030]

York, C. (1997). CL 92,100 (Terbufos 15G): Total CL 92,100-Related Residues in Corn Grain after Treatment with COUNTER 15G Insecticide Applied Post emergence (Nebraska). Report RES 96-022. American Cyanamid Company. Unpublished. [TE-730-031]

Zheng, S. Gross, J. (1989). Freezer Storage Stabilities of Total CL 92,100-Related Residues in Whole Banana and Banana Pulp. Report RES 99-070. [TE-326-015]

Zheng, S and Gross, J. (1998). Terbufos (CL 92,100): Independent Laboratory Validation of GC Method M3072 for the Determination of Total Terbufos –Related Residues in Whole Banana and Banana Pulp. American Cyanamid. [TE-244-057]

Zulalian J. (1990), Terbufos (CL 92,100): Metabolic Fate of Carbon-14 CL 92,100 in Milk and Tissues of Lactating Goats, (Princeton). [TE-440-002]

Zulalian J. (1992). Terbufos (CL 92,100): Metabolic Fate of Carbon-14 CL 92,100 in Milk