DYNAMAC
CORPORATION

# TELONE

Final Report

# Task 2: Residue Chemistry Chapter

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# RESIDUE CHEMISTRY

# Task 2

## Table of Contents

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### INTRODUCTION

Telone (1,3-dichloropropene) is a nematicide/insecticide/fungicide/herbicide registered for use on a variety of agricultural crops such as vegetables, field crops, citrus fruit trees, deciduous fruit and nut trees, and bush and vine crops (refer to the EPA Index to Pesticide Chemicals, 1,3-dichloropropene, p. 3, dated 3-6-84 for specific use sites). The 92% ready-to-use (RTU) formulation, a single active ingredient product, is marketed under the trade name Telone II for which Dow Chemical Co. is the sole basic producer in the United States. Preplant applications (broadcast or row) are made by chisel injection into the soil. After application, excessive fumigant loss is prevented by compacting the soil surface or covering the treated area with tarps or heavy gauge plastic. No tolerances or exemptions from tolerances for residues of telone in or on food/feed commodities have been established. Heretofore, registration of telone formulations for preplant use has been categorized as a non-food agricultural use.

### NATURE OF THE RESIDUE IN PLANTS

#### Conclusions:

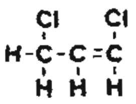
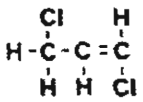
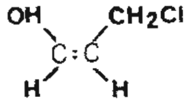
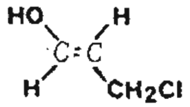
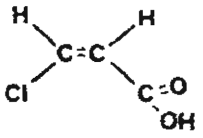
The metabolism of telone (1,3-dichloropropene) in plants is not adequately understood. The uptake and distribution of [$^{14}$C]1,3-dichloropropene (DCP) have been investigated in bush beans, carrots, tomatoes, and sugar beets; however, the major components of the terminal residues were identified only in sugar beets. Bauriedel et al (1973; MRID 00040721) showed that $^{14}$C-activity present in mature sugar beets grown in soil treated 2 weeks prior to planting with [$^{14}$C] 1,3-DCP was primarily incorporated into natural plant constituents; no residual [$^{14}$C]1,3-DCP was found. Another study (Berry, 1973; MRID 00040722) also demonstrated that a portion of hydroponically administered [$^{14}$C]1,3-DCP is incorporated into lipids, pigments, amino acids, and sugars in bush bean, carrot, and tomato plants; however, the extraction efficiency was extremely poor (5-33% of the plant-contained $^{14}$C-activity) and 33-37% of the extractable $^{14}$C-activity was unidentified. Residues of cis + trans 1,3-DCP (I) and cis + trans 3-CAA (II) were detected by GC in bush beans, tomatoes, and carrots following injection of unlabeled telone into the root zone of these plants; residues of trans 3-chloroacrylic acid (III) and 3-chloro-1-propanol (IV) were also detected by GC in the subject plants following injection of unlabeled 3-CAA. [Refer to Table 1 on p. 3 for a depiction of the molecular structures of these compounds.] We believe the available sugar beet metabolism data are nonrepresentative of all crop groups and that additional metabolism studies are essential in order to ascertain what residues of telone should be included in the tolerance definition. Since $^{14}$C-residues persist in the soil and may be taken up by plants , the present non-food determination is not appropriate. Accordingly we request:

- o Data depicting the distribution and metabolism of [$^{14}$C]1,3DCP in soybeans and lettuce harvested at regular intervals which encompass seedling and mature growth stages following a single preplant soil injection application at 6-8 inches below the soil surface with the 92% RTU formulation at a rate sufficiently high to permit complete characterization of $^{14}$C-residues. If a significant proportion of the terminal residues are unextractable by the solvent(s) used, then analyses must include hydrolysis and reextraction of plant residues to determine the nature of bound or conjugated residues.

Presently, no tolerances or exemptions from tolerances for residues of telone in or on food/feed commodities exist; upon receipt of the above-requested data an appropriate tolerance definition (parent compound plus all metabolites of toxicological concern) must be established along with tolerances for residues in or on all food crops on which use of telone is registered.

4

Table 1. Telone, its metabolites and postulated metabolites in plants.

| Code | Structure | Chemical Name | Abbreviations |
|---|---|---|---|
| I |  $\mathrm{H{-}\overset{Cl}{\underset{H}{C}}{-}\underset{H}{C}{=}\overset{Cl}{\underset{H}{C}}}$ | 1,3-dichloropropene (cis) | SD-949, 1,3-DCP (Z) |
| I[a] |  $\mathrm{H{-}\overset{Cl}{\underset{H}{C}}{-}\underset{H}{C}{=}\overset{H}{\underset{Cl}{C}}}$ | 1,3-dichloropropene (trans) | SD-950, 1,3-DCP (E) |
| II |  | 3-chloroallylalcohol (cis) | SD-42311, 3-CAA (Z) |
| II[a] |  | 3-chloroallylalcohol (trans) | SD-42312, 3-CAA (E) |
| III |  | 3-chloroacrylic acid | -- |
| IV | $Cl{-}CH_2CH_2CH_2OH$ | 3-chloro-1-propanol | -- |

[a] 1,3-DCP and 3-CAA exist as cis and trans isomers.

References (used):

00040721. Bauriedel, W.R.; Craig, L.F. 1973. A Study of the Residue Present in Sugar Beets Grown in Soil Treated with $^{14}$C-Labeled Cis- and Trans-1,3-Dichloropropene. (Unpublished study received July 22, 1980 under 464-EX-63; submitted by Dow Chemical U.S.A.; CDL:099516-L.)

00040722. Berry, D.L. 1973. Absorption, Translocation and Metabolism of 1,3-Dichloropropene in Selected Plants. Doctoral dissertation, Utah State Univ. (Unpublished study received July 22, 1980 under 464-EX-64; submitted by Dow Chemical U.S.A.; CDL:099516-M.)

Discussion of the data:

Dow Chemical Co. (MRID 00040722) submitted a study authored by Berry (1973) pertaining to the uptake, translocation, and metabolism of telone in bush beans, carrots, and tomatoes. Bush beans at the third trifoliate stage and tomatoes 7-8 inches tall received [$^{14}$C]1,3-dichloropropene (DCP) or [$^{14}$C] 3-chloroallyl alcohol (CAA) separately via hydroponic solution; 10-12 week old carrots were exposed to these compounds in moist vermiculite. Absorption of [$^{14}$C]1,3-DCP by the excised shoots of bean plants and by direct topical exposure of bean leaves was also investigated. Uptake of [$^{14}$C]1,3-DCP by roots from hydroponic solution occurred within 4 hours after exposure. After 9, 24, and 48 hours, 9%, 68%, and 63%, respectively, of the administered $^{14}$C-activity was present in whole bush bean plants. Autoradiography of whole bean plants from the 24-hour sample interval showed that $^{14}$C-activity is distributed throughout the entire plant. Translocation of $^{14}$C-activity from topically treated leaf tissue (bush bean) to other plant parts was limited; 2% and 15% of the applied $^{14}$C-activity was present in root and stem samples, respectively, following 24 hours of exposure. The authors report that $^{14}$C-activity present in plant tissues and extracted from the hydroponic culture solution with hexane was generally 80% of that initially administered; the remainder was apparently lost as $CO_2$ or volatilized from the culture solution. A $^{14}$C-balance study to support this contention was not performed. For identification of $^{14}C$-activity, bean, tomato, and carrot plants obtained from the 24-hour treatment interval were frozen, homogenized, and extracted nonsequentially with 80% ethanol and neutral, basic, and acidic ether. Assuming 68% of the administered $^{14}C$-activity was present in whole bean plants, the extraction efficiencies of these solvents was poor: 5% using neutral ether (pH 7), 20% using acidic ether (pH 1), 9% using basic ether (pH 12), and 33% using 80% ethanol. Insoluble $^{14}C$-residues of bush beans, carrot, and tomato following 80% ethanol or ether extraction were shown to contain less than 1% of the administered $^{14}C$-activity. The apparent loss of >50% of the $^{14}C$-residues present in the plant during extraction was not explained. The soluble fractions were concentrated and partitioned against various solvents prior to separation by TLC, paper chromatography, or ion exchange resins; quantification of $^{14}C$-activity was achieved by scintillation counting. A portion of the $^{14}C$-activity extractable from the subject plants with neutral ether was located in plant pigments and lipids (24-37%); however, most of the extractable labeled material was unidentified (63-76%). Chromatographic separation of the acidic ether extracts showed that 11-15% of the $^{14}C$-activity was present in plant pigments and lipids, 48-66% was present in anionic acids, and 23-36% was unidentified. A similar distribution of $^{14}C$-activity was exhibited following chromatographic separation of the alkaline ether fractions. Distribution of labeled material ([$^{14}C$]1,3-DCP) extracted from the subject plants with 80% ethanol is presented below.

| | % of extractable $^{14}C$-residues | | |
|---|---|---|---|
| Constituent | Bush beans | Tomato | Carrot |
| Anionic acids[a] | 13 | 16 | 16 |
| Cationic acids[b] | 8 | 10 | 8 |
| Neutral compounds[c] | 46 | 41 | 40 |
| Unidentified | 34 | 33 | 37 |

[a] Citric, succinic, fumaric, malic, α-ketoglutaric acids.

[b] Alanine, aspartic, glutamic, glycine, serine acids.

[c] Glucose and sucrose.

Distribution of labeled material extracted with 80% ethanol from bean, tomato, and carrot plants following 24 hours of hydroponic exposure to $^{14}$C-CAA was similar. We find that these data show that a portion of the administered $^{14}$C-DCP was incorporated into natural constituents (lipids, pigments, amino acids, and sugars) of the subject plants but they fail to indicate the magnitude or distribution of intact or conjugated parent and metabolite residues of $^{14}$C-DCP or $^{14}$C-CAA. Apparently, cochromatography of the ether or ethanol fractions from plants treated with $^{14}$C-DCP or $^{14}$C-CAA against known standards of these compounds was not attempted.

In a separate set of tests, also presented in MRID 00040722, beans, tomatoes, and carrots growing in vermiculite and described as at a "desired stage of maturity" received unlabeled cis + trans 1,3-DCP or 3-CAA by injection into the root zone. Plants were harvested at regular intervals from 0.5 to 120 hours posttreatment and frozen until extraction. Samples were homogenized with 2,2,4-trimethyl pentane or hexane and methanol and filtered. The filtrate was partitioned with water and the resulting organic fraction was purified on a Florosil column prior to GC analysis using a $^{63}$Ni electron capture detector and a glass column packed with 10% Carbowax 20 M on Chromosorb W (HP). Detection of 3-CAA and 3-chloro-1-propanol (IV) was accomplished by derivatization with trifloroacetic anhydride (TFAA); 3chloroacrylic acid and 3-chloropropionic acid were detected as trimethylsilyl derivatives. Residues of cis +trans 1,3-DCP and cis + trans 3-CAA were detected in the 1,3-DCPtreated subject plants within 1/2 to 1 hour of treatment; no detectable residues of 1,3-DCP or 3-CAA were found after the 48-hour sample interval. Plants treated with 3-CAA and sampled 1-72 hours posttreatment contained residues of cis + trans 3-CAA, 3-chloroacrylic acid, and 3-chloro-1-propanol; no detectable levels of any of these compounds were found after the 72-hour sample interval. No information regarding the specific limit of detection in these tests was provided. The presence of 1,3-DCP, 3-CAA, and 3-chloro-1-propanol in bush bean plants harvested 4 and 8 hours after treatment was confirmed by mass spectrometry; low concentrations of 3-chloroacrylic acid prevented mass spectral identification of this compound.

Another Dow Chemical Co. submission (MRID 00040721; 1973) contains a study in which sugar beets were planted in an outdoor field plot 2 weeks following soil treatment with the equivalent of 25 gal of product/A $^{14}$C-DCP at a

8

depth of 10 inches. Sugar beet tops and roots (six samples each) were harvested 159 days after treatment. The samples were frozen and stored for an unspecified time prior to analysis. Total $^{14}C$-activity determined by wet combustion and expressed as ppm DCP equivalents (calculated on a wet-weight basis) was 1.14-7.61 ppm in sugar beet leaves, 0.94-2.31 ppm in stems, and 0.27-0.98 ppm in roots. The authors report that treated plants contained only 0.017% of the $^{14}C$-activity administered to the soil. Sugar beets (roots) grown in untreated soil adjacent to the treated area also contained $^{14}C$-activity at 0.04-0.46 ppm, representing 10 samples. The nature of the $^{14}C$-activity in sugar beets was investigated by a yeast fermentation test. Juice from diced sugar beet roots was steam-sterilized and inoculated with <u>Saccromyces</u> <u>cerevisseae</u>. Evolved $^{14}CO_2$ was collected in a 1 N NaOH trap. The labeled carbon evolved from sugar fermentation was reported to be 6.3 ppm calculated as µg $^{14}C$-DCP per g-carbon. These data indicate that a portion of the $^{14}C$-activity present in sugar beets was incorporated into naturally occurring plant sugars. Another sugar beet sample was freeze-dried, ground, and subjected to Soxhlet extraction with diethyl ether for 8 hours. The solid material was blended with hot 70% ethanol, filtered, and the filtrate was subjected to ion exchange resins in series: elution of amino acids from Dowex 50 was accomplished with 3 N HCl, whereas organic acids were separated by elution from Dowex 1 with 3 M formic acid. Material insoluble in 70% ethanol was treated with 1 N NaOH; the filtrate was hydrolyzed with 6 N HCl to yield an amino acid fraction whereas the remaining solids were sequentially treated with 1 N HCl and 80% ethanol to separate cellulose, pectin, and hemicellulose. All of the various fractions contained $^{14}C$-activity and the organic acid fraction exhibited a relatively high level of $^{14}C$-activity thus indicating the possible presence of a metabolite of DCP. Distribution of $^{14}C$-activity in the various fractions is tabulated on the following page:

| | |
|---|---|
| Diethyl ether extract residue | <1 |
| 70% ETOH insoluble | |
| cellulose | 13 |
| pectin/hemicellulose | 2 |
| protein | 4 |
| unidentified | 13 |
| 70% ETOH soluble | |
| amino acids | 1 |
| organic acids | 10 |
| crude sucrose | 32 |
| Absorbed by Dowex 50 W | 9 |
| Absorbed by Dowex 1 | 13 |
| | 97% |

Another sugar beet root sample was subjected to a series of fractionations similar to those described above except that following the initial elution series, the Dowex 50 and Dowex 1 resins were further extracted (sequentially) in bulk at 70 C with 3 N HCl and 3 N HCl in methanol. This vigorous treatment recovered most of the $^{14}$C-activity from the ion exchange resins: amino acids composed ~7%, organic acids composed ~20%, and the ion exchange resins retained ~3% of the initial activity. Organic acid-containing eluates from the Dowex-1 column suspected of containing a metabolite of 1,3-DCP were combined, basified, and evaporated to dryness. Residues were taken up in 1 N HCl, extracted with diethyl ether (continuous liquid-liquid extraction for 4 days), and the ether extract was concentrated by distillation. Sublimation of the distillate under vacuum yielded white crystals identified as oxalic acid by IR analysis. Residues remaining after sublimation were taken up in ether, methylated with diazomethane and subjected to GC analysis. The retention time of the major $^{14}$C-containing moiety did not correspond to the methyl esters of the normal organic acids found in sugar beets (citric, acetic, oxalic, malic, and tartaric acids) or to the methyl ester of 3-chloroacrylic acid (III), a possible metabolite of DCP.

These tests indicate that the unknown must be rendered soluble by hydrolysis; it is apparently acidic, relatively non-volatile, and can be methylated with diazomethane to form a chromatographable reaction product. Attempts to isolate a sufficient quantity of the compound for GS-MS identification were unsuccessful. The authors estimate that the unknown compound constitutes 2.7-5.3% of the total $^{14}C$-residue.

We conclude that the plant metabolism of telone is not adequately understood. The major components of the terminal residues were identified only in sugar beets. In other tests using bush bean, carrot, and tomato plants, the extraction efficiency was poor (5-33%) and 33-37% of the extractable $^{14}C$-activity was unidentified. The available metabolism data do show that $^{14}C$-activity is present in the shoot portions of sugar beets and bush beans, following root exposure to $^{14}C$-DCP in hydroponic solution or soil and that most of the $^{14}C$-activity present in sugar beet roots is incorporated into natural plant compounds. Although these data do not define the complete metabolic route of 1,3-DCP, Barry (1973; MRID 0004-0722) suggested that 1,3-DCP and its 3-CAA metabolite may be metabolized to an intermediate (possible pyruvate) because of the occurrence of $^{14}C$-activity in natural plant constituents such as pigments and lipids. We believe additional metabolism studies with soybeans and lettuce must be conducted in order to determine what residues must be included in the tolerance definition for telone.

## RESIDUE ANALYTICAL METHODS

### Conclusions:

Adequate GC methods are available for the analysis of the cis and trans forms of both 1,3-DCP and its chloroallyl alcohol metabolite, 3-CAA in or on plant commodities. Adequate GC methods are also available for the analysis of 1,2-dichloropropene (an impurity in telone formulations). The methods which detect 1,3-DCP include: WAMS 112-1, WAMS 222-1, MMS-R-480-1, MMS-R-272-3, ACR 80.9, ACR 79.15, and MMS-R-505-2. The methods which detect 3-CAA include: WAMS 158-1, WAMS 222-1, MMS-R-481-1, ACR

80.1, and MMS-R-506-2. The formulation impurity, 1,2-DPE, is detected by methods WAMS 222-1, MMS-R-480-1, and MMS-R-272-3.

References (used):

00030385. Glas, R.D. 1979. Determination of Residues of Cis- and Trans-1,3-Dichloropropene in Plant Materials: ACR 79.15. Method dated Nov. 30, 1979. (Unpublished study received Feb. 7, 1980 under 464-511; submitted by Dow Chemical U.S.A.; CDL:241761-B.)

00033255. McKinney, W.J.; Wendt, M.B.; Abbott, R.; et al. 1978. Residues in Sugarbeets: TIR-24-355-76. (Unpublished study including TIR-24-355-76-B, received June 25, 1980 under 464-511; prepared by Shell Development Co., submitted by Dow Chemical U.S.A.; CDL:242726-A.)

00033256. McKinney, W.J.; Wendt, M.B.; Fries, F.A.; et al. 1978. Residues in Cabbage: TIR-24-160-78-A. (Unpublished study received June 25, 1980 under 464-511; prepared by Shell Development Co., submitted by Dow Chemical U.S.A.; CDL:242726-B.)

00033257. McKinney, W.J.; Wendt, M.B. 1978. Residues in Potatoes: TIR-24-172-78-A. (Unpublished study including TIR-24-172-78-B, received June 25, 1980 under 464-511; prepared by Shell Development Co., submitted by Dow Chemical U.S.A.; CDL:242726-C.)

00033258. McKinney, W.J.; Fries, F.A.; Wendt, M.B.; et al. 1978. Residues in Cauliflower: TIR-24-180-78. (Unpublished study received June 25, 1980 under 464-511; prepared by Shell Development Co., submitted by Dow Chemical U.S.A.; CDL:242726-D.)

00033259. McKinney, W.J.; Fries, F.A.; Wendt, M.B.; et al. 1978. Residues in Lettuce: TIR-24-191-78A. (Unpublished study received June 25, 1980 under 464-511; prepared by Shell Development Co., submitted by Dow Chemical U.S.A.; CDL:242726-E.)

00033260. McKinney, W.J.; Fries, F.A.; Bierman, B.; et al. 1979. Residues in Watermelon: TIR-24-227-788. (Unpublished study received June 25, 1980 under 464-511; prepared by Shell Development Co. and others, submitted by Dow Chemical U.S.A.; CDL:242726-F.)

00033262. Dutson, N.J.; Seager, S.V.; Wallace, B.G.; et al. 1977. Residues of the Major Components of D-D and Primary Metabolites in Lettuce from Germany: Group Research Report BLGR.0024.77. (Unpublished study received June 25, 1980 under 464-511; prepared by Shell Research, Ltd., submitted by Dow Chemical U.S.A.; CDL:242726-H.)

00033263. Bosio, P.G.; Granier, R. 1977. Residues of D-D in Potatoes from France--1976/77 Trials: Group Research Report BEGR.0086.77. (Unpublished study received June 25, 1980 under 464-511; prepared by Shell Chimie, submitted by Dow Chemical U.S.A.; CDL:242726-I.)

00033264. Sherren, A.J.; Murray, S.M.; Wallace, B.G.; et al. 1978. Residues of the Major Components of D-D and Primary Metabolites in Pineapples from South Africa: Group Research Report BLGR.0071.78. (Unpublished study received June 25, 1980 under 464-511; prepared by Shell Research, Ltd., submitted by Dow Chemical U.S.A.; CDL:242726-J.)

00039694. Glas, R.D. 1980. Determination of Residues of Cis- and Trans-1,3-Dichloropropene in Fruit: ACR 80.9. Method dated July 9, 1980. (Unpublished study received July 22, 1980 under 464-EX-63; submitted by Dow Chemical U.S.A.; CDL:099515-X.)

00039695. Glas, R.D. 1980. Determination of Residues of Cis- and Trans-Chloroallyl Alcohols in Fruit by Gas Chromatography Using and Electrolytic Conductivity Detector: ACR 80.10. Method dated July 9, 1980. (Unpublished study received July 22, 1980 under 464-EX-63; submitted by Dow Chemical U.S.A.; CDL:099515-Y.)

00109672. Shell Chemical Co. 1978. D-D Crop Residue and Analytic Methods: Volume III. (Compilation; unpublished study received Aug. 30, 1978 under 201-119; CDL:235253-A.)

00109291. Shell Development Co. 1981. Residue Determination of the Z and E Isomers of 3-chloroallyl Alcohol (CAA) in Agricultural Commodities, Soils, and Water: Capillary GLC/Hall Electrolytic Conductivity Detector Method: MMS-R-506-2. (Unpublished study received Jan. 15, 1982 under 201-253; CDL:246671-A.)

00109420. Shell Development Co. 1981. Residue Determination of 1,2-dichloropropane and the Z and E Isomers of 1,3-dichloropropene in Agricultural Commodities, Soil and Water: Capillary GLC/Hall Electrolytic Conductivity Detector Method: MMS-R-505-2. (Unpublished study received Jan. 15, 1982 under 201-253; CDL:246672-A.)

00115214. Dow Chemical U.S.A. 1982. Residue Data, Where Pertinent, on (a) Food or Feed Commodities; (b) Non-food Crops Such as Tobacco; and (c) Foliage or Other Sites which May Related to Worker Hazard or Adverse Effects on the Environment. Include a Description of the Analytical Method(s) Used and a Summary of the Data. (Compilation; unpublished study received Sept. 22, 1982 under 464-EX-63; CDL:248406-B.)

00117045. Dow Chemical U.S.A. 1982. Telone II chemistry data. (Compilation; unpublished study received Sept. 22, 1982 under 464-EX-63; CDL:248416-A.)

Discussion of the data:

An undesignated GLC method, submitted by Shell Chemical Co. (1978; MRID 00109672), prescribes a codistillation procedure in which the crop sample is refluxed for 30 min. at 100 C in a distilled water/petroleum spirit mixture. The petroleum spirit distillate fraction is dried with sodium sulfate, cleaned up by shaking with "Fuller's earth," and chromatographed using a GLC equipped with a glass column (10% OV-1 on 80-100 mesh Gas Chrom Q) and an electron capture detector. This method detects the cis and trans isomers of 1,3-DCP. Method recovery data obtained from crop residue data submissions are tabulated on the following page:

| Commodity | Fortification level (ppm) | Recovery range (%) cis and trans 1,3-DCP | MRID |
|---|---|---|---|
| Chinese cabbage | 0.07 | 73-83 | 00109672 |
| Cucumbers | 0.07 | 82-86 | 00109672 |
| Rice | 0.07 | 67-84 | 00109672 |
| Soybeans, green plant | 0.07 | 64 | 00109672 |
| Soybeans, nature bean | 0.07 | 57-64 | 00109672 |
| Carrots | 0.07 | 72-76 | 00109672 |
| Sugar beets | 0.02-0.07 | 71-85 | 00109672 |

A modification, referred to as WAMS 112-1, of the above-described, undesignated method was submitted by Shell Chemical Co. (1978; MRID 00109672). The procedural changes in WAMS 112-1 include (i) use of hexane in the co-distillation step, and (ii) a different column packing: 30% w/w Carbowax 20 M on 80-100 Gas Chrom Q. A companion method designated WAMS 158-1 for the determination of 3-CAA (cis and trans isomers) was also included in this submission. The procedural steps for the derivatization and determination of 3-CAA prescribed in WAMS 158-1 are essentially identical to those discussed below in method WAMS 222-1. Method recovery data obtained with WAMS 112-1 and WAMS 158-1 and presented in crop residue data submissions are tabulated on the following page:

| | | Residue Range (%) | | |
|---|---|---|---|---|
| Commodity | Fortification level (ppm) | cis and trans 1,3-DCP | cis and trans 3-CAA | MRID(s) |
| Potatoes | 0.1 | 70-80 | 120-150 | 00033263, 00109672 |
| Carrots | 0.1-0.2 | 70-90 | 85 | 00109672 |
| Radishes | 0.1 | 100 | 80 | 00109672 |
| Grapes | 0.1 | 100-120 | 60-90 | 00109672 |
| Tobaccco | 0.1-0.5 | 100-150 | 80 | 00109672 |

Shell Chemical Co. (1978; MRID 00109672) submitted a method designated as WAMS 222-1 (or in some residue data submissions, SAMS 222-1) for the determination of the cis and trans isomers of both 1,3-DCP and 3-CAA, and for the determination of 1,2-DPE (a telone formulation impurity) in crops by GC. In this method, frozen tissue samples are quickly macerated (a delay in the maceration step may result in loss of residues by volatilization), steam distilled from acidified (0.2 M sulfuric acid) water, treated with 20% (w/v) NaOH, and further steam-distilled into ethyl ether. For extraction, the distillate is partitioned with ethyl ether in the presence of NaCl and a portion of the ethyl ether extract is eluted with additional ethyl ether from an acidic alumina chromatography column. Analysis of the purified extract for 1,3-DCP is accomplished by GC equipped with a 10% w/w OV-1 (on 80-100 mesh Gas Chrom Q) silicone fluid column and an electron capture detector. Extraction and cleanup procedures for the determination of 1,3-DPE are essentially identical; determination is by GC equipped with a 20% w/w tricresyl phosphate (on 80-100 mesh Gas Chrom Q) glass column and a flame ionization detector. A second portion of the ethyl ether extract is reacted with 3,5-dinitrobenzoyl chloride and pyridine, thus forming the 3-CAA derivative, 3,5-dinitrobenzoate. Purification of this solution is achieved by column chromatography prior to final determination by GC equipped with a 2% w/w OV-225 (on 100-120 mesh Gas Chrom Q) silicone fluid column and an electron capture detector. Residues are calculated and reported as 1,3-DCP or 3-CAA; a correction factor is required to adjust for the molecular weight difference between CAA and its derivative. Method recovery data presented in crop residue data submissions are summarized below:

| | | Residue Range (%) | | |
|---|---|---|---|---|
| Commodity | Fortification level (ppm) | cis and trans 1,3-DCP | cis and trans 3-CAA | MRID |
| Carrots | 0.2 | 85 | 65-110 | 00109672 |
| Sweet potato | 0.05 | -- | 79-88 | 00109672 |
| Radish | 0.2-0.5 | 70-75 | 70-100 | 00109672 |
| Lettuce | 0.1-0.4 | 70 | 60-80 | 00033262 |
| Spinach | 0.1-0.5 | 75 | 40-60 | 00109672 |
| Pineapple | 0.05-0.1 | 75-80 | 75 | 00033264 |
| Potato | 0.1 | 65-75 | 65-70 | 00109672 |

Recoveries of 1,2-DPE were 65-100% from the crops tabulated above fortified with 1,2-DPE at 0.1-2 ppm.

Modifications of method WAMS 222-1, designated MMS-R-480-1 and MMS-R-481-1, were also submitted by Shell Chemical Co. (1978; MRID 00109672) for the determination of 1,3-DCP and 3-CAA. Method MMS-R-480-1 may also be used for the determination of 1,2-DPE. In method MMS-R-480-1 frozen, macerated crop samples are codistilled from a mixture of water and ethyl acetate. For determination of 1,3-DCP, an aliquot of the ethyl acetate distillate is dried with sodium sulfate and chromatographed using a GC equipped with a glass capillary column (front 70' OV-1 followed by 110'

FFAP w/4:1 inlet splitter) and a $^{63}$Ni electron capture detector. Determination of 3-CAA (MMS-R-481-1) is accomplished by steam distillation for 20 min. The resulting distillate is partitioned with hexane, the aqueous fraction is extracted with ethyl ether and then transferred to ethyl acetate. This fraction is concentrated by evaporation and derivatized by reaction with Trifluoroacetic acid(TFAA) for 2 hrs. at room temperature. The reaction mixture is chromatographed as described above for 1,3-DCP. A summary of the method recovery data obtained from crop residue data submissions is presented in the table below:

| | | Recovery Range (%) | | |
|---|---|---|---|---|
| Commodity | Fortification level (ppm) | cis and trans 1,3-DCP | cis and trans-3-CAA | MRID |
| Potatoes | 0.05-0.5 | 112-116 | 79-101 | 00033257 |
| Lettuce | 0.05-0.52 | 89-101 | 78- 90 | 00033259 |
| Cabbage | 0.05-0.25 | 86-115 | 78-104 | 00033256 |
| Cauliflower | 0.05-0.25 | 84- 93 | 106 | 00033258 |
| Watermelon | 0.05-0.25 | 100-108 | 82- 94 | 00033260 |

Recoveries of 1,2-DPE were 88-114% from the above listed crops fortified with 1,2-DPE at 0.5-1 ppm.

Shell Chemical Co. (1978; MRID 00109672) submitted information regarding a GC method entitled "Residue determination of dibromochloropropane in crops, soil, and water, GLC/EC method" and designated MMS-R-272-3, which was used for analysis of 1,3-DCP and 1,2-DPE in or on sugar beets and sweet potatoes. Frozen plant samples are codistilled in water and ethyl acetate and the ethyl acetate distillate is chromatographed without further cleanup using a GC equipped with a 20% SP-2100/0.1% Carbowax 1500 (on 100/120 mesh Supelcoport) glass column and a $^{63}$Ni electron capture detector.

Determination of 3-CAA as its TFAA derivative was accomplished using a method essentially identical to the previously described method MMS-R-481-1. Recoveries of 1,3-DCP were 93-125% from sugar beet roots and 73-103% from sugar beet tops fortified with 1,3-DCP at 0.025-0.05 ppm; recoveries of 1,3-DCP were 110% from sweet potatoes fortified at the same levels. Recoveries of 3-CAA were 79-99% from sugar beet roots and 82-98% from sugar beet tops fortified with 3-CAA at 0.1-0.5 ppm.

Dow Chemical Co. (1980, MRIDs 00039694 and 00039695) submitted methods for the determination of the cis and trans isomers of 1,3-DCP (ACR 80.9) and 3-CAA (ACR 80.1) in fruit; the procedures described in ACR 80.9 are essentially the same as those presented in an earlier Dow method (ACR 79.15; MRID 00030385) for the determination of 1,3-DCP (cis and trans) in plant materials. Dichloropropenes are steam distilled into hexane and an aliquot of the hexane extract is chromatographed by GC equipped with a 20% SP-2100 plus 0.1% Carbowax 1500 (on 100/120 mesh Supelcoport) glass column and a $^{63}$Ni electron capture detector. Chloroallyl alcohol metabolites are also steam distilled. The distillate is partitioned with hexane and the aqueous fraction is extracted with ethyl ether in the presence of NaCl. The ethyl ether is removed by evaporation and the residue (diluted with hexane) is quantified by GC with electrolytic conductivity detection equipped with a 10% SP-1000 (on 80/100 mesh Gas Chrom Q) column. Method recovery data included with the subject methods and obtained from crop residue data submissions are tabulated on the following page:

| | | Recovery Range (%) | | |
|---|---|---|---|---|
| Commodity | Fortification level (ppm) | cis and trans 1,3-DPE | cis and trans 3-CAA | MRID(s) |
| Almonds (nuts, shells, hulls) | 0.01-0.05 | 70-102 | 76-110 | 00115214 |
| Broccoli | 0.01-0.05 | 86-88 | -- | 00030385 |
| Cantaloupe | 0.01-0.05 | 85-88 | -- | 00030385 |
| Carrots | 0.01-0.05 | 85-86 | -- | 00030385 |
| Beans, green | 0.01-0.05 | 90-93 | -- | 00030385 |
| Bean vines, green | 0.01-0.05 | 82-88 | -- | 00030385 |
| Beans, dry | 0.01-0.05 | 78-81 | -- | 00030385 |
| Bean vines, dry | 0.01-0.05 | 91-95 | -- | 00030385 |
| Grapes | 0.01-0.05 | 74-100 | 78-100 | 00039695, 00115214, 00039694 |
| Lettuce | 0.01-0.05 | 89 | -- | 00030385 |
| Onions | 0.01-0.05 | 81-85 | -- | 00030385 |
| Oranges (peel, pulp) | 0.01-0.05 | 70-100 | 80-100 | 00115214, 00039699, 00039694, 00039695 |
| Peaches | 0.01-0.05 | 80-110 | 70-100 | 00115214, 00039699, 00039694, 00039695 |
| Plums | 0.01-0.05 | 80-100 | 70-90 | 00115214 |
| Potatoes | 0.01-0.1 | 91-92 | -- | 00030385 |
| Pineapples | 0.01-0.1 | 88 | -- | 00030385 |

Table continued.

| Commodity | Fortification level (ppm) | Recovery Range (%)<br>cis and trans 1,3-DPE | Recovery Range (%)<br>cis and trans 3-CAA | MRID(s) |
|---|---|---|---|---|
| Pineapple tops | 0.01-0.1 | 93-94 | -- | 00030385 |
| Soybeans | 0.01-0.05 | 77-78 | -- | 00030385 |
| Soybean forage | 0.01-0.05 | 84-94 | -- | 00030385 |
| Soybean straw | 0.01-0.05 | 89-91 | -- | 00030385 |
| Sugar beets | 0.01-0.1 | 90-91 | -- | 00030385 |
| Sugar beet tops | 0.01-0.1 | 88-89 | -- | 00030385 |
| Tomatoes | 0.01-0.05 | 86-95 | -- | 00030385 |
| Walnuts | 0.01-0.05 | 80-102 | 72-100 | 00115214 |
| Almonds, green | 0.01-0.05 | 90-91 | 85-92 | 00039694, 00039695 |

Table concluded.

The Shell Development Co. (1981; MRID 00109420 and 00109291) submissions contain additional methods for the determination of 1,3-DCP designated MMS-R-505-2 and 3-CAA designated MMS-R-506-2 in agricultural commodities. These methods differ from the previously-described steam distillation procedures in that (i) 1 N $H_2SO_4$ is added to the distillation flask to release 3-CAA and (ii) determination is achieved by GC using a Hall electrolytic conductivity detector. Average recoveries of cis and trans 1,3-DCP from a variety of agricultural crops were 88% and 91%, respectively, at an unspecified fortification level. Average recoveries of cis and trans 3-CAA were 86% and 84%, respectively, at a 0.05 ppm fortification level. These methods are adequate for data collection and are reported here for informational purposes only; these methods were not used for residue analysis in any data submission.

Dow Chemical U.S.A. (1982; MRID 00117045) also submitted information regarding the specificity of the residue determination method (refer to method ACR 80.9 described previously) used for 1,3-DCP analysis. Analytical standards of nemacur (ethyl 4-[methylthio]-m-tolyl isopropyl phosphoramidate), aldicarb, carbofuran, and oxamyl gave no GC response; 1,2-dibromo-3-chloropropane resulted in a retention time of ~40 min which would not interfere with the ~ 3 min retention time exhibited by 1,3-DCP.

## STORAGE STABILITY DATA

### Conclusions:

The available data show that the storage stability of 1,3-DCP in or on frozen plant samples is poor; levels of 1,3-DCP in potato and lettuce samples were 27-40% and 57-66%, respectively, of the initial fortification level after 75 days in frozen storage. The following data are required:

- o The storage conditions and interval from harvest to analysis of all plant samples used to obtain all residue data requested in this standard must be submitted. The data must be accompanied by stability data depicting the percent decline of all residues of concern in plant samples stored under the conditions and intervals specified.

### Reference (used):

00033261. McKinney, W.J.; Brown, L.J.; Doern, B.L.; et al. 1979. Residues in Various Crops: TIR-24-642-78. (Unpublished study including TIR-24-614-79, received June 25, 1980 under 464-511; prepared by Shell Development Co., submitted by Dow Chemical U.S.A.; CDL:242726-G.)

Discussion of the data:

Dow Chemical Co. (1979; MRID 00033261) submitted a storage stability study, TIR-24-642-78, prepared by Shell Development Co. in which macerated potato and lettuce samples were fortified with 1 ppm 1,3-DCP (cis and trans isomers) and stored frozen at -23.3 C. Samples were removed from storage at various intervals and levels of 1,3-DCP were determined by GC using an EC $^{63}$Ni detector (MMS-R-480-1). The results are summarized in the table on the following page:

| | | % of initial 1 ppm fortification level | |
|---|---|---|---|
| Sample | Storage interval (days) | 1,3-DCP (cis) | 1,3-DCP (trans) |
| Potato | 1 | 50 | 57 |
| | 12 | 52 | 62 |
| | 47 | 37 | 47 |
| | 75 | 27 | 40 |
| Lettuce | 1 | 73 | 77 |
| | 12 | 81 | 88 |
| | 47 | 58 | 60 |
| | 75 | 57 | 66 |

These data were not corrected for method recovery which was 82-97% for potatoes and 72-92% for lettuce fortified with 1,3-DCP (cis and trans isomers) at 0.05 ppm. Since method recovery was shown to be fairly good, it appears that substantial loss of residues from plant samples occurs during frozen storage.

In our opinion, the storage stability of telone in or on plant samples is poor. No storage stability data were presented for the chloroallyl alcohol metabolite of telone, 3-CAA, although considerable residue data were presented regarding the presence of this moiety in or on plant commodities. Additional data are required.

## MAGNITUDE OF THE RESIDUE IN PLANTS

It should be noted that the nature of the residue in plants has not been adequately described. It should also be noted that the stability of 1,3DCP in or on frozen plant samples is poor; loss of 1,3-DCP from samples stored >30 days may be as much as ~ 60%. Thus, the nondetectable residues (<0.02-<0.04 ppm) of 1,3-DCP reported in this section for samples stored >30 days are considered of limited value.

### Tolerances:

No tolerances or exemptions from tolerances for residues of telone in or on food/feed commodities have been established. Heretofore, registration of telone formulations for preplant use has been categorized as a non-food agricultural use.

### Use directions and limitations:

The 92% (9.2 lb/gal) RTU formulation is registered for use as a preplant soil fumigant on all agricultural crops. Broadcast (overall) applications are made by plowsole or chisel injection; row applications are made with 1-2 chisels per row. Injection into the soil at a depth of 10-12 inches is accomplished with chisels mounted on 12- to 30-inch centers. Shallow rooted plants (field crops, small fruits, and vegetables) may be treated at 36.8-331.2 lb ai/A, whereas citrus fruit trees, deciduous fruit trees, nut trees, and grapes may be treated at 138-938.4 lb ai/A; rates vary with soil type and desired depth of penetration. After application, excessive fumigant loss is prevented by compacting the soil surface or covering the treated area with tarps or heavy gauge plastic. The minimum exposure time is 7-14 days. Before planting, the treated soil is allowed to aerate for 1 week for each 10 gallons of the 92% RTU applied per acre.

24

Conclusions:

The available data are not adequate to determine the magnitude of terminal residues of telone in or on food and feed commodities because: (i) dosage information was unclear; (ii) storage stability is poor; and (iii) most data were generated on foreign sites. Therefore the following data are required:

- Representative commodities from each crop group listed in 40 CFR 180.34 (with the exception of those in the Herbs and Spices group) and asparagus, okra, cotton, flax, hops, peanuts, safflower, sugarcane, olives, persimmons, pineapple and pomegranates must be treated preplant at representative geographic locations with the 92% RTU according to the label directions at the maximum registered rate. Samples of all raw agricultural commodities from each crop must be collected at the shortest interval after planting in which they could be used for food or feed purposes. Since frozen storage stability is poor, samples should be analyzed as soon as possible following collection. Analyses should be made for all residues of toxicological concern (refer to the section entitled "Nature of the Residue in Plants"). Tolerances reflecting maximum expected residues levels or, if no measurable residues are detected, the limit of detection must be proposed.

References (used):

00033255. McKinney, W.J.; Wendt, M.B.; Abbott, R.; et al. 1978. Residues in Sugarbeets: TIR-24-355-76. (Unpublished study received June 25, 1980 under 464-511; prepared by Shell Development Co., submitted by Dow Chemical U.S.A.; CDL:242726-A.)

00033256. McKinney, W.J.; Wendt, M.B.; Fries, F.A.; et al. 1978. Residues in Cabbage: TIR-24-160-78-A. (Unpublished study received June 25, 1980 under 464-511; prepared by Shell Development Co., submitted by Dow Chemical U.S.A.; CDL:242726-B.)

00033257. McKinney, W.J.; Wendt, M.B. 1978. Residues in Potatoes: TIR-24-172-78-A. (Unpublished study including TIR-24-172-78-B, received June 25, 1980 under 464-511; prepared by Shell Development Co., submitted by Dow Chemical U.S.A.; CDL:242726-C.)

00033258. McKinney, W.J.; Fries, F.A.; Wendt, M.B.; et al. 1978. Residues in Cauliflower: TIR-24-180-78. (Unpublished study including TIR-24-180-78-B, received June 25, 1980 under 464-511; prepared by Shell Development Co., submitted by Dow Chemical U.S.A.; CDL:242726-D.)

00033259. McKinney, W.J.; Fries, F.A.; Wendt, M.B.; et al. 1978. Residues in Lettuce: TIR-24-191-78A. (Unpublished study including TIR-24-191-78-B, TIR-24-192-78A and TIR-24-192-78-B, received June 25, 1980 under 464-511; prepared by Shell Development Co., submitted by Dow Chemical U.S.A.; CDL:242726-E.)

00033260. McKinney, W.J.; Fries, F.A.; Bierman, B.; et al. 1979. Residues in Watermelon: TIR-24-227-78B. (Unpublished study received June 25, 1980 under 464-511; prepared by Shell Development Co. and others, submitted by Dow Chemical U.S.A.; CDL:242726-F.)

00033262. Dutson, N.J.; Seager, S.V.; Wallace, B.G.; et al. 1977. Residues of the Major Components of D-D and Primary Metabolites in Lettuce from Germany: Group Research Report BLGR.0024.77. (Unpublished study received June 25, 1980 under 464-511; prepared by Shell Research, Ltd., submitted by Dow Chemical U.S.A.; CDL:242726-H.)

00033263. Bosio, P.G.; Granier, R. 1977. Residues of D-D in Potatoes from France--1976/77 Trials: Group Research Report BEGR.0086.77. (Unpublished study received June 25, 1980 under 464-511; prepared by Shell Chimie, submitted by Dow Chemical U.S.A.; CDL:242726-I.)

00033264. Sherren, A.J.; Murray, S.M.; Wallace, B.G.; et al. 1978. Residues of the Major Components of D-D and Primary metabolites in Pineapples from South Africa: Group Research Report BLGR.0071.78. (Unpublished study received June 25, 1980 under 464-511; prepared by Shell Research, Ltd., submitted by Dow Chemical U.S.A.; CDL:242726-J.)

00039693. Dow Chemical U.S.A. 19??. Summary of Residue Analyses of 1,3-Dichloropropenes in Crops Treated Post-plant with Telone II. (Unpublished study received July 22, 1980 under 464-EX-63; CDL:099515-W.)

00039696. Lembright, H.W.; Hart, W.; Rough, D. 1980. Residues of 1,3-Dichloropropenes and Chloroallyl Alcohols in Green Almonds Grown in Soil Fumigated with Telone II Soil Fumigant. (Unpublished study received July 22, 1980 under 464-EX-63; submitted by Dow Chemical U.S.A.; CDL:099515-Z.)

00109672. Shell Chemical Co. 1978. D-D Crop Residue and Analytic Methods: Volume III. (Compilation; unpublished study received Aug. 30, 1978 under 201-119; CDL:235253-A.)

00115214. Dow Chemical U.S.A. 1982. Residue Data, Where Pertinent, on (a) Food or Feed Commodities; (b) Non-food Crops Such as Tobacco; and (c) Foliage or Other Sites Which May Relate to Worker Hazard or Adverse Effects on the Environment, Include a Description of the Analytical Method(s) Used and a Summary of the Data. (Compilation; unpublished study received Sep. 22, 1982 under 464-EX-63; CDL:248406-B.)

References (not used):

[Data presented in the following references appeared in duplicate in previously-cited references.]

00039697. Glas, R.D.; Turner, G.D. 1980. Analysis of Oranges and Peaches for Residues of Dichloropropenes and Chloroallyl Alcohols after Postplant Application of Telone II Soil Fumigant: GH-C 1312. (Unpublished study received July 22, 1980 under 464-EX-63; submitted by Dow Chemical U.S.A.; CDL:099515-AA.)

00039698. Glas, R.D.; VanGundy, S. 1980. Analysis of Peel, Pulp, Leaves, and Soil from an Orange Grove for Residues of Dichloropropenes and Chloroallyl Alcohols after Post Plant Injection of Telone II Soil Fumigant: GH-C 1308. (Unpublished study received July 22, 1980 under 464-EX-63; prepared in cooperation with Univ. of California--Riverside, submitted by Dow Chemical U.S.A.; CDL:099515-AB.)

00039699. Glas, P.D.; Turner, G.D. 1980. Analysis of Grapes for Residues of Dichloropropenes and Chloroallyl Alcohols after Postplant Application of Telone II Soil Fumigant: GH-C 1281. (Unpublished study received July 22, 1980 under 464-EX-63; submitted by Dow Chemical U.S.A.; CDL:099515-AC.)

27

00039700. Lembright, H. 1980. Residues of 1,3-Dichloropropenes and Chloroallyl Alcohols in Grapes Grown in Soil Fumigated with Telone II Soil Fumigant. (Unpublished study received July 22, 1980 under 464-EX-63; submitted by Dow Chemical U.S.A.; CDL:099515-AD.)

00040718. Dow Chemical U.S.A. 19??. Residue Data, Where Pertinent, on (a) Food or Feed Commodities; (b) Non-food Crops Such As Tobacco; and (c) Foliage or Other Sites Which May Relate to Worker Hazard or Adverse Effects on the Environment: Telone II. (Unpublished study received July 22, 1980 under 464-EX-63; CDL:099516-I.)

Discussion of the data:

Shell Chemical Co. (MRID 00109672) and Dow Chemical Co. (MRIDs 00033255, 00038256, 00033257, 00033258, 00033259, 00033260, 00033262, 00033263, and 00033264) submitted data pertaining to residues of 1,3-DCP, 3-CAA, and 1,2-DPE in or on various commodities from tests conducted in Europe, Japan, South Africa, and the United States. Data depicting residues of 1,2-DPE are presented for informational purposes only. 1,2-DPE is not a metabolite of 1,3-DCP; it is an impurity of Telone II, Dow's tradename for an ~1:1 mixture of the cis and trans isomers of technical 1,3-DCP. These data, tabulated below according to the agricultural crop categories which appear on the label (EPA Reg. No. 464-511), reflect single preplant applications of telone by soil injection. The formulations used in these tests were described as D-D, D-D technical, Shell D-D, and Shell D-D 75% EC, but information regarding the percentage of 1,3-DCP present in the designated products was unspecified; thus we have reported the maximum dosages in gal/A. In a few tests, the formulation used was described as "unformulated D-D, >95% 1,3-DCP" for which ai/A rates were estimated based on 95% 1,3-DCP and a density of ~10 lb/gal.

| Commodity | Test Location | Maximum Dose gal/A (lb ai/A) | Formulation | Interval after treatment[e] | Residues (ppm) | | | Analytical method[d] | MRID |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | 1,3-DCP[a] | 3-CAA[b] | 1,2-DPE[c] | | |
| Vegetable Crops | | | | | | | | | |
| Carrots | Germany, Holland | 32 (304) | Unformulated D-D >95% 1,3-DCP | 20-37 | <0.02 | <0.08-<0.1 | <0.1 | C | 00109672 |
| Carrots | Japan, Germany | 64 | D-D technical | 17-24 | <0.02 | <0.02 | <0.01 | A,B | 00109672 |
| Cabbage | CA, FL | 25 | D-D | 81-134 | <0.02 | <0.05* | <0.1 | D | 00033256 |
| Chinese cabbage | Japan | 43 | D-D technical | 14-16 | <0.02 | -- | -- | A | 00109672 |
| Cauliflower | CA | 10 | D-D | 20 | <0.02 | <0.02 | <0.1 | D | 00033258 |
| Cucumbers | Japan | 43 | Technical | 8-14 | <0.02 | -- | -- | A | 00109672 |
| Lettuce | Germany | 32 (304) | Unformulated D-D >95% 1,3-DCP | ~ 12 | <0.02 | <0.1 | <0.1 | C | 00033262 |
| Lettuce, leaf | CA | 14 | D-D | ~ 6 | <0.02 | <0.04 | <0.1 | D | 00033259 |
| Potatoes | AL, Europe | 96 | Shell D-D | 3.5-11 months | <0.02 | <0.02-<0.1 | -- | B,C,D | 00033263,<br>00033257,<br>00019672 |
| Radishes | Germany | 43 | Shell D-D, 75% EC | 9-12.5 | <0.02 | <0.02-<0.08 | <0.01-<0.25 | B,C | 00109672 |
| Sweet potatoes | NC | 10 | Shell D-D | ~ 18 | <0.02 | <0.03 | <0.1 | C,E | 00109672 |
| Spinach | Germany | 32 (304) | Unformulated D-D >95% 1,3 DCP | 11-12 | <0.02 | <0.02 | | C | 00109672 |
| Watermelon | TX, GA | 20 | D-D | ~ 13-15 | <0.02 | <0.02 | | D | 00033260 |

Table continued.

| Commodity | Test Location | Maximum Dose gal/A (lb ai/A) | Formulation | Interval after treatment$^e$ | Residues (ppm) |  |  | Analytical method | MRID |
|---|---|---|---|---|---|---|---|---|---|
|  |  |  |  |  | 1,3-DCP$^a$ | 3-CAA$^b$ | 1,2-DPE$^c$ |  |  |
| Field Crops |  |  |  |  |  |  |  |  |  |
| Rice | Japan | 43 | D-D technical | 27 | <0.02 | -- | -- | A | 0010967 |
| Soybeans, green plant | France | 43 | D-D technical | 10-14 | <0.02 | -- | -- | A | 0010967 |
| Soybeans, mature bean | France | 43 | D-D technical | 10-14 | <0.02 | -- | -- | A | 0010967 |
| Sugar beet, roots | ID, CO | 27 | D-D | 11-12 months | <0.02 | <0.02-<0.04 | <0.1-<0.3 | E | 0003325 |
| Sugar beet, tops | ID, CO | 27 | D-D | 11-12 months | <0.02 | <0.02-<0.04 | <0.1-<0.3 | E | 0003325 |
| Sugar beet, roots | Holland | 43 | Shell D-D | ~ 37 | <0.02 | <0.1 | <0.1 | C | 0010967 |
| Tobacco, cured leaves | S. Africa | 21 | Shell D-D, 75% EC | 12.5 | <0.04 | <0.08 | <0.1 | B | 0010967 |
| *Deciduous Fruit and Nut Tree Planting Sites* |  |  |  |  |  |  |  |  |  |
| Pineapple | S. Africa | 43 (408) | Unformulated D-D >95% 1,3-DCP | 27 months | <0.02 | <0.04 | <0.1 | C | 0003326 |
| Bush and Vine Planting Sites |  |  |  |  |  |  |  |  |  |
| Grapes | France | 107 | Shell D-D | 3 years | <0.02 | <0.02 | <0.01 | B | 0010967 |

Table continued.

[a] Combined residues of the cis and trans isomers of 1,3-DCP.

[b] Combined residues of the cis and trans isomers of 3-CAA.

[c] 1,2-DPE is an impurity of Telone II, Dow's tradename for a ~1:1 mixture of the cis and trans isomers of technical 1,3-DCP.

[d] A - undesignated GCEC;
B - WAMS 112-1, 122-1, 158-1;
C - WAMS 222-1;
D - MMS-R-480/481-1;
E - MMS-R-272-3.

[e] Interval after treatment is reported in weeks, except as noted.

* Detectable (0.04 ppm) residues of cis 3-CAA were attributed to interferring coextractives also present in control samples at 0.03-0.04 ppm.

Samples were reportedly stored at -18 to -26 C but the interval from harvest to analysis was reported only for a limited number of crops: cabbage, 88-232 days; cauliflower, 81 days; potatoes, 25 days; sweet potatoes, 196-210 days; and watermelon 83-190 days. Adequate analytical methods (specific method designation numbers are presented on the previous page) were used for data collection.

Dow Chemical Co. (MRIDs 00115214, 00039693, and 00039696) also submitted data pertaining to residues of 1,3-DCP and 3-CAA in or on oranges, grapes, walnuts, almonds, peaches, plums, and cherries harvested at various intervals following single, postplant soil injection applications (chisel or shank) with the 92% RTU formulation at 73.6-276 lb ai/A. No residues of 1,3-DCP or 3-CAA were detected using analytical methods ACR 79.15 and ACR 80.4; the combined detection limits for the cis/trans isomers of both 1,3-DCP and 3-CAA is <0.02 ppm. These data were submitted in support of temporary tolerances (PP#0G2392) for the combined residues of 1,3-DCP and 3-CAA in or on almonds, cherries, figs, grapefruit, grapes, lemons, oranges (except Valencia), peaches, plums, fresh prunes, and walnuts which expired 10/1/82 and the extension of an EUP (#464-EUP-63; PP#0G2392, amendment of 9/21/82) for postplant use of the 92% RTU on the same crops. These data are presented for informational purposes only.

We note three points regarding the available residue data which warrant consideration: (i) determination of the maximum dosage (lb ai/A) used in most of the residue trials was not made because information regarding the formulation was lacking; (ii) the nondetectable residues (<0.02-<0.04 ppm) of 1,3-DCP reported for samples stored >30 days are considered of limited value because loss of 1,3-DCP during frozen storage may be significant; (iii) no information regarding the stability of 3-CAA during frozen storage were submitted; and (iv) most residue data reflect foreign,rather than domestic use.

In addition, if real residues are found in feed-commodities, data depicting the metabolism of Telone in ruminants and poultry is required. If transfer of residues of Telone to ruminant- and poultry tissues occurs then appropriate feeding studies with these animals are required.

13544

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| | |
|---|---|
| **Chemical:** | **1,3-Dichloropropene** |
| **PC Code:** | **029001** |
| **HED File Code** | **11000 Chemistry Reviews** |
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