US EPA ARCHIVE DOCUMENT

DYNAMAC
CORPORATION

CAPTAN ADDENDUM

Task 1: Review and Evaluation of Individual Studies

August 3, 1988

Final Report

Contract No. 68-02-4250

Submitted to:
Environmental Protection Agency
Arlington, VA 22202

Submitted by:
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Rockville, MD 20852

CAPTAN

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STUDY 1

CHEM 081301 Captan §161-3

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40658010
Ruzo, L.O., A.L. Kesterson, S.B. Jackson, and L.J. Lawrence. 1988a. Soil surface photolysis of [^{14}C]captan in natural sunlight. Laboratory Project ID ORTHO:MEF-0068. PTRL Report No. 1142. PTRL Project No. 232. Prepared by Pharmacology and Toxicology Research Laboratory (PTRL), Lexington, KY, and submitted by Chevron Chemical Company, Richmond, CA.

REVIEWED BY: K. Patten TITLE: Staff Scientist

EDITED BY: J. Harlin TITLE: Staff Scientist

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Laurie C. Lewis

AUG 4 1988

SIGNATURE:

CONCLUSIONS:

Degradation - Photodegradation on Soil

This study is unacceptable because the text does not adequately and clearly explain the experimental design and the source of some data. If the study is resubmitted with additional explanation, it may fulfill EPA Data Requirements for Registering Pesticides by providing information on the half-life of tetrahydrophthalamine-labeled [^{14}C]captan and the formation of [^{14}C]degradates on sunlight-irradiated soil.

SUMMARY OF DATA BY REVIEWER:

Tetrahydrophthalamide-labeled [^{14}C]captan (labeled in the C1 and C2 positions of the cyclohexene ring; radiochemical purity 97.1%), at 44.13 $\mu g/cm^2$, degraded with an observed half-life of 3-5 days (54-89 W·minute/cm^2) on moist sandy loam soil irradiated outdoors in Lexington, Kentucky, at 22.5-28.6°C in January, 1988. After 5 days of irradiation, an average 48.3% of the recovered radioactivity was captan, 21.3% was

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dicarboximide (THPI), 9.4% was cyclohex-4-ene-2-cyano-1-carboxylic acid (THCY), 3.2% was cyclohex-4,5-epoxy-1,2-dicarboximide (THPI epoxide), 2.1% was cyclohex-4-ene-2-amido-1-carboxylic acid (THPAM), 0.8% was cyclohex-4-ene-1,2-dicarboxylic acid (THPAL), and 3.2% was unextractable. There were three major and several minor unknowns totaling 11.5% of the recovered (each was ≤2.8% of the recovered). Volatilized [^{14}C]-residues were <0.01% of the applied. In the dark control at 5 days posttreatment, an average of 62.2% of the recovered was captan; the relative concentration of degradates was similar to that in the irradiated soil. The registrant-calculated half-lives for captan were 129.5 hours (≈5.4 days) in the irradiated soil and 236.1 hours (≈9.8 days) for the dark control.

DISCUSSION:

1. It was unclear how the "total dpm applied" to each dish of soil was determined. Because untreated soil was distributed to individual sample dishes and treated, rather than the entire mass of soil being treated, then distributed to the dishes, the initial concentration of [^{14}C]residues in each soil was different. This difference was greater than what would be attributed to normal variability because 3-21% of the 400 µL captan stock solution remained in the transfer pipette. The study authors reported that [^{14}C]residues remaining in the transfer pipettes were eluted, counted by LSC, and subtracted from the "dose" in order to obtain the applied dpm values. It was assumed that dose referred to theoretical dose of 0.87 mg/dish, rather than a measured value.

   However, footnotes to Table I (Extraction and recovery of radiocarbon) give reason to question this assumption. In that table, the study authors stated that the individual plate designations for day 1 and 2 samples were lost because of "experimental conditions", and the values used for "total dpm applied" were the average of total dpm applied to all 24 dishes (4 plates/day x 6 days). The dpm values for day 1 and 2 are 17819387 and 17508774, respectively; the values logically should be identical. Either the dpm applied to the four dishes sampled at each interval were averaged or "dose" did not refer to 0.87 mg/dish but to measured values.

2. The material balance, referred to as "total recovery", is variable. "Total recovery" values, which ranged from 83.1 to 128.0%, are defined as a comparison of "total dpm applied" (Discussion #1) and the sum of residues in each extract, unextractable residues in the extracted soil, and volatiles. If "total dpm applied" is a calculated value, it is probable that the variability primarily reflects differences between the calculated and actual application (which was not measured) to each sample, rather than material lost from the system. The researchers appear to have taken adequate measures to account for all radioactivity applied to the soil.

3. Total dpm in the soil after incubation and prior to extraction was not determined.

4. The concentration of captan in the day 4 samples is 14.8 and 16.3% of the recovered, which is considerably lower than the day 3 and 5 concentrations. The study authors stated that the methylene chloride sonication was longer than the prescribed 5 minutes, and that this resulted in partial evaporation of the methylene chloride and a change in the relative proportions of specific [^{14}C]residues. Data from day 4 were not used by the study authors to calculated the half-lives.

5. The study authors stated that in order to test the homogeneity of application, the test plates were each sectioned into three equal parts, which were separately extracted with 60 mL of methylene chloride; two aliquots of each extract (total of 12) were analyzed by LSC to determine total radioactivity. Homogeneity was >90%. It is unclear whether twelve refers to aliquots (2 aliquots x 3 sections x 2 dishes) or extracts (3 sections x 4 dishes). The study protocol states that four samples would be assayed from two dishes.

6. Table III states that data are expressed in terms of "percent of the applied". Elsewhere in the original document it is stated that these data were normalized to 100%; therefore the data in Table III are actually expressed in terms of "percent of the recovered".

7. The methylene chloride extracts were quantified by TLC rather than HPLC because of "serious problem encountered in reverse-phase HPLC on injection" of methylene chloride. HPLC was used to identify [^{14}C]residues in the methylene chloride extracts.

8. TLC detection limits and recovery values from fortified soil samples were not reported. Extraction efficiencies ranged from 79.5 to 125.7% of the applied. LSC counting efficiencies were reported to be >95%.

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MATERIALS AND METHODS

MATERIALS AND METHODS:

[^{14}C]Captan (labeled at the C1 and C2 positions of the cyclohexene ring; radiochemical purity 97.1%, specific activity 16.6 mCi/mM, Chevron Chemical Company) plus unlabeled captan (purity 100%) were applied at 44.13 $\mu g/cm^2$ (0.234:1 mix ratio) to moist (75% of field capacity) nonsterile sandy loam soil (70% sand, 17% silt, 13% clay, 1.0% organic matter, pH 7.1, CEC 7.5 meq/100 g) in petri dishes (10 g/dish, 19.6 cm^2 surface area). The dishes were incubated in quartz-covered stainless steel chambers outdoors in Lexington, Kentucky (latitude 38.05°N, longitude 84.30°W), between January 7 and 12, 1988. The chambers were surrounded by a jacket through which polyethylene glycol was circulated, which maintained the soil surface temperature at 22.5 to 28.6°C. Air was drawn through the chambers containing the soil dishes, then sequentially through one tube of ethylene glycol and three tubes of 10% sodium hydroxide trapping solutions. The chambers were "occasionally" flushed with water-saturated air (continuous use of humidified air was difficult because the water froze and impeded air movement). For the dark control, half of the chambers were covered with a "dark material" (unspecified). Sunlight intensity and cumulative sunlight energy were monitored constantly throughout the study. Duplicate soil and trapping solution samples were taken daily.

Portions of each soil sample were air-dried and analyzed for total radioactivity by LSC following combustion. On the same day as sampling, additional portions of each soil were extracted sequentially three times with methylene chloride (using sonication or stirring), three times with water (stirring), and twice with acetone (stirring). The separate extracts were analyzed for total radioactivity by LSC. The methylene chloride extracts were combined, and aliquots were analyzed for total radioactivity by LSC and for specific [^{14}C]residues by TLC. The methylene chloride extracts were cochromatographed with captan, THPAL, THPAM, AND THCY reference standard on silica gel TLC plates developed in acetic acid:chloroform (1:20, v:v). Radioactive areas were visualized by autoradiography; relevant regions were scraped from the plates, extracted with methanol, and analyzed by LSC. Unlabeled standards were visualized by exposure to iodine vapors. The water and acetone extracts were combined, and aliquots were analyzed for total radioactivity by LSC and for specific [^{14}C]residues by TLC as described. Also, aliquots of the methylene chloride (dried and redissolved in acetone) and water/acetone extracts were analyzed using reverse-phase HPLC (Supelco Reverse Phase LC-18 column equipped with a Micromeritics variable wavelength UV detector and a Radiomatic Beta flow-one detector). The extracted soil was analyzed for nonextractable [^{14}C]residues by LSC following combustion. The LSC background was 40 dpm; it was reported that the HPLC equipment could detect signals as low as 150 dpm.

The trapping solutions were analyzed for total radioactivity by LSC.

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The material not included contains the following type of information:

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_____ Description of the product manufacturing process.

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_____ Identity of the source of product ingredients.

_____ Sales or other commercial/financial information.

_____ A draft product label.

_____ The product confidential statement of formula.

_____ Information about a pending registration action

__X__ FIFRA registration data.

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The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

DATA EVALUATION RECORD

STUDY 2

CHEM 081301 Captan §161-3

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40658009
Ruzo, L.O., A.L. Kesterson, S.B. Jackson, and L.J. Lawrence. 1988b. Soil surface photolysis of [^{14}C-trichloromethyl]captan in natural sunlight. Laboratory Project ID ORIHO:MEF-0075. PIRL Report No. 231-2. PIRL Project No. 231. Prepared by Pharmacology and Toxicology Research Laboratory (PIRL), Lexington, KY, and submitted by Chevron Chemical Company, Richmond, CA.

REVIEWED BY: K. Patten TITLE: Staff Scientist

EDITED BY: J. Harlin TITLE: Staff Scientist

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Laurie C. Lewis

AUG 4 1988

SIGNATURE:

CONCLUSIONS:

Degradation - Photodegradation on Soil

This study is unacceptable because the text does not adequately and clearly explain the experimental design and the source of some data. If the study is resubmitted with additional explanation, it may fulfill EPA Data Requirements for Registering Pesticides by providing information on the formation of trichloromethyl-labeled [^{14}C]degradates on sunlight-irradiated soil.

SUMMARY OF DATA BY REVIEWER:

Trichloromethyl-labeled [^{14}C]captan (radiochemical purity 96.9%), at 22.2-28.4 $\mu g/cm^2$, photodegraded with an observed half-life of 10-16 days (238-366 W·minute/cm^2) on moist sandy loam soil irradiated outdoors in Lexington, Kentucky, at -0.9 to 36.7°C in March-April, 1988. After 16 days of irradiation, 44.0% of the recovered radioactivity (41.7% of the applied) was captan, 0.8% was acetone/water-extractable, 6.7% was acid-

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extractable, 41.8% had been evolved as CO_2, and 6.7% was unextractable. In the dark control at 16 days posttreatment, an average of 49.8% of the recovered (47.7% of the applied) was captan. The registrant-calculated half-lives were 359.1 hours (≈15 days) for the irradiated samples and 496.5 hours (≈21 days) for the dark control.

DISCUSSION:

1. This study consisted of two independent experiments, which were conducted using different stock solutions, different application rates, and different study dates. Experiment A was sampled at 0 through 10 days posttreatment, and Experiment B was sampled at 10, 16, and 21 days posttreatment. Experiment B was not sampled at day 0 to confirm the application rate, and data from day 10 of Experiments 1 and 2 are not similar; degradation occurred more rapidly during Experiment 2. However, the study authors have combined the two experiments to calculate a half-life for trichloromethyl-labeled [^{14}C]captan.

2. It was unclear how the "total dpm applied" to each dish of soil was determined. This study should be comparable to Study 1, since it was conducted by the same research group using approximately the same methods. However, the "total dpm applied" values reported in this study do not appear to have been obtained using the same procedures as in Study 1. The "total dpm applied" values for each sample are identical, which is improbable. As was done in Study 1, untreated soil was distributed to each dish and then treated. Unlike Study 1, the study authors do not state that a portion of the captan stock solution remained in the transfer pipette; since no apparent changes in procedure were made, it is probable that a similar problem occurred. The soils should not have contained identical amounts of [^{14}C]residues; either the "total dpm applied" is the theoretical application rate or it is an averaged value.

3. The material balance, referred to as "total recovery", ranges from 61.5 to 112.6% of the "total dpm applied". Since it is uncertain how "total dpm applied" was obtained (Discussion #1), it is uncertain what comparison is being made. If "total dpm applied" is the theoretical application rate, it is probable that the variability primarily reflects differences between the theoretical and actual application (which was not measured) to each sample, rather than material lost from the system. The researchers appear to have taken adequate measures to account for all radioactivity applied to the soil.

4. The soil surface temperature ranged from -0.9 to 36.7°C (Table IVA). The lowest temperature, which occurred during the 10-day study, was ascribed to a circulator malfunction. Temperatures as low as 11.2°C were recorded when the circulator was apparently functioning properly. This may explain why the half-life of captan in Experiment B was shorter than in Experiment A, and why the half-life determined in Study 2 is longer than the half-life determined in Study 1, in which the samples were kept at 22.5 to 28.6°C.

5. Total dpm in the soil after incubation and prior to extraction was not determined.

6. It was stated that the diameter of the petri dish was 60 mm and the surface area of the dish was 19.6 cm^2. However, the diameter of a dish with a surface area of 19.6 cm^2 would be 50 mm. Since in Study 1 the diameter of the dish was reported to be 50 mm, and since Studies 1 and 2 were conducted by the same laboratory, the 60 mm figure is probably an error.

7. The study authors stated that in order to test the homogeneity of application, two test plates were each sectioned into three equal parts, which were separately extracted with 30 mL of methylene chloride; three aliquots of each extract were analyzed by LSC to determine total radioactivity. Homogeneity was >85%.

8. Data in Table 3 are apparently expressed in terms of "percent of the recovered", since the data sum to 100.0%. No units are reported on the table.

9. TLC detection limits and recovery values from fortified soil samples were not reported. Extraction efficiencies ranged from 31.7 to 99.7% of the applied (except for one sample which was partially lost during analysis). LSC counting efficiencies were reported to be >95%.

10. The study authors stated that standards containing the S-trichloromethyl group, other than captan, were not available.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Trichloromethyl-labeled [^{14}C]captan (radiochemical purity 96.9%, specific activity 40.4 mCi/mM, Chevron Chemical Company) plus unlabeled captan (purity 100%) were applied at 28.4 $\mu g/cm^2$ (0.017:1 mix ratio) to moist (75% of field capacity) nonsterile sandy loam soil (70% sand, 17% silt, 13% clay, 1.0% organic matter, pH 7.1, CEC 7.5 meq/100 g) in petri dishes (10 g/dish, 19.6 cm^2 surface area). The dishes were incubated in quartz-covered stainless steel chambers outdoors in Lexington, Kentucky (latitude 38.05°N, longitude 84.30°W), between March 1 and 10, 1988. The chambers were surrounded by a jacket through which polyethylene glycol was circulated, which maintained the soil surface temperature at -0.9 to 33.0°C. Air was drawn into the chambers containing the soil dishes, then sequentially through one tube of ethylene glycol and three tubes of 10% sodium hydroxide trapping solutions. The chambers were "occasionally" flushed with water-saturated air (continuous use of humidified air was difficult because the water froze and impeded air movement). For the dark control, half of the chambers were covered with a "dark material" (unspecified). Sunlight intensity and cumulative sunlight energy were monitored constantly throughout the study. Duplicate soil and trapping solution samples were taken at 0, 1, 3, 5, 7, and 10 days posttreatment.

On the same day as sampling, additional portions of each soil were extracted three times with acetone. The acetone extracts were combined, and aliquots were analyzed for total radioactivity by LSC and for specific [^{14}C]residues by both TLC and HPLC. The acetone extracts were cochromatographed with a captan reference standard on silica gel TLC plates developed in acetic acid:chloroform (1:20, v:v). Radioactive areas were visualized by autoradiography; relevant regions were scraped from the plates, extracted with methanol, and analyzed by LSC. Also, aliquots of the acetone extracts were analyzed using reverse-phase HPLC (Supelco Reverse Phase LC-18 column equipped with a Micromeritics variable wavelength UV detector and a Radiomatic Beta flow-one detector). The acetone-extracted soil was analyzed for nonextractable [^{14}C]-residues by LSC following combustion. The LSC background was 40 dpm; it was reported that the HPLC equipment could detect signals as low as 150 dpm.

The trapping solutions were analyzed for total radioactivity by LSC. The sodium hydroxide trapping solution was saturated with barium chloride, and the precipitate was analyzed by LSC to determine total CO_2 evolved.

Since the half-life had not occurred by the last sampling interval (10 days), the study was repeated. Trichloromethyl-labeled [^{14}C]captan plus unlabeled captan were applied at 22.2 $\mu g/cm^2$ (0.027:1 mix ratio) to sandy loam soil in petri dishes. The dishes were incubated as previously described between March 28 and April 18, 1988; soil surface temperatures ranged from 12.3 to 36.7°C. Duplicate soil samples were taken at 10, 16, and 21 days posttreatment; trapping solutions were sampled more frequently.

On the same day as sampling, additional portions of each soil sample was extracted three times with acetone and once with water; the extracts were combined and analyzed by LSC, TLC, and HPLC as described. Acetone/water-extracted soil that contained >10% of the applied radioactivity was further

extracted with 1 N HCl at 70°C for 30 minutes. The resulting extract and the acid-extracted soil were analyzed by LSC. The trapping solutions were analyzed as previously described.

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_____ A draft product label.

_____ The product confidential statement of formula.

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DATA EVALUATION RECORD

STUDY 3

CHEM 081301 Captan 162-1

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40658007

Pack, D.E. and I.S. Verrips. 1988a. Aerobic soil metabolism of [trichloromethyl-^{14}C]captan. Laboratory Project ID MEF-0060/8809887. Prepared and submitted by Chevron Chemical Company, Richmond, CA.

REVIEWED BY: K. Patten TITLE: Staff Scientist

EDITED BY: J. Harlin TITLE: Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

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APPROVED BY: L. Lewis

TITLE: Environmental Scientist

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Laurie C. Lewis

AUG 4 1988

SIGNATURE:

CONCLUSIONS:

Metabolism - Aerobic Soil

This study is acceptable and partially fulfills EPA Data Requirements for Registering Pesticides by providing information on the metabolism of trichloromethyl-labeled [^{14}C]captan in aerobic soil.

SUMMARY OF DATA BY REVIEWER:

Trichloromethyl-labeled [^{14}C]captan (radiochemical purity >99.6%), at 4.9 and 6.1 ppm, degraded with a half-life of <1 day in aerobic sandy loam soil incubated in the dark at 25 + 0.1 C and 80% of field capacity. At 1 day posttreatment, an average of 79.4% of the applied radioactivity was undegraded captan, 46.0% had been evolved as CO_2, 0.6% had been evolved as organic compounds (not further characterized), and 16.7% was unextractable [^{14}C]residues. No nonvolatile degradates were detected. At 30 days posttreatment, an average of 7.6% of the applied radioactivity was undegraded captan, 84.5% had been evolved as CO_2, 1.0% had been evolved as organic compounds (not further characterized), and 14.6% was unextractable [^{14}C]residues. The material balances ranged from 82.1-114.2% of

the applied during the 30-day study (values <90% were from days 1 and 3 only).

DISCUSSION:

1. The study authors stated that the quantity of organic volatiles trapped was too small to identify.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Trichloromethyl-labeled [^{14}C]captan (radiochemical purity >99.6%, specific activity 40.4 mCi/mmol, Amersham) was applied at 4.9 and 6.1 ppm to flasks containing moist (80% of field capacity) sandy loam soil (58% sand, 30% silt, 12% clay, 1.2% organic matter, pH 7.2, CEC 7.7 meq/100 g) using a direct-displacement digital microdispenser. The application rate was determined by measuring the amount of stock solution dispersed from the microdispenser, rather than by analyzing treated soil. Individual flasks were attached to independent continuous air-flow systems; humidified air was drawn through the sample flask and then sequentially through traps containing ND-103 (an aromatic solvent) and 2-methoxyethanol:ethanolamine (6:4, v:v). The treated soils were incubated in the dark at 25°C. One hour prior to sampling, the soil to be sampled was acidified with 1 M $NaHSO_4$ to remove residual $^{14}CO_2$ and volatile collection continued until sampling. Quadruplicate samples (two each from the 4.9 and 6.1 ppm treatments) were taken at 0 and 7 days posttreatment; duplicate samples (from the 6.1 ppm treatment only) were taken at 1, 3, 14, and 30 days posttreatment.

Soil samples were extracted three times by blending the soil with ethyl acetate on a mixer for 10 minutes. The three extracts were combined, and aliquots were analyzed for total radioactivity by LSC. Additional aliquots were evaporated to dryness in a vacuum rotary evaporator at ambient temperature, the residues were redissolved in acetonitrile, and aliquots of the acetonitrile solution were analyzed by LSC. The acetonitrile solutions were analyzed by ion-pairing HPLC with radioactive flow detection and by TLC. The solutions were cochromatographed with unlabeled captan, thiazolidine-2-thione-4-carboxylic acid, and dithiobis(methane sulfonic acid) reference standards on silica gel TLC plates in two dimensions, using chloroform:acetic acid (40:1, v:v) and n-propanol:water (7:3, v:v) solvent systems. Radioactive areas on the plates were located using autoradiography; the unlabeled standards were visualized by quenching of UV fluorescence. The ethyl acetate-extracted soil was analyzed for unextractable radioactivity by LSC following combustion.

The trapping solutions were analyzed by LSC. The trapping efficiency of the 2-methoxyethanol:ethanolamine solution was 108%.

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS

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DATA EVALUATION RECORD

STUDY 4

CHEM 081301 Captan 162-2

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40658008

Pack, D.E. and I.S. Verrips. 1988b. Anaerobic soil metabolism of [trichloromethyl-^{14}C]captan. Laboratory Project ID MEF-0061/8809887. Prepared and submitted by Chevron Chemical Company, Richmond, CA.

REVIEWED BY: K. Patten TITLE: Staff Scientist

EDITED BY: J. Harlin TITLE: Staff Scientist

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APPROVED BY: L. Lewis
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Laurie C. Lewis

AUG 4 1988

SIGNATURE:

CONCLUSIONS:

Metabolism - Anaerobic Soil

This study is acceptable and partially fulfills EPA Data Requirements for Registering Pesticides by providing information on the anaerobic soil metabolism of trichloromethyl-labeled [^{14}C]captan.

SUMMARY OF DATA BY REVIEWER:

Trichloromethyl-labeled [^{14}C]captan (radiochemical purity >99.6%), at 6.1 ppm, degraded with a half-life of <1 day in aerobic sandy loam soil incubated in the dark at 25 ± 0.1 C. After 1 day of aerobic incubation, an average of 19.4% of the applied radioactivity was undegraded captan, 46.0% had been evolved as CO_2, 0.6% had been evolved as organic compounds (not further characterized), and 16.7% was unextractable [^{14}C]residues. No nonvolatile degradates were detected. After 1 day of aerobic incubation plus 29 days of anaerobic (N_2 gas) incubation, an average of 4.0% of the applied radioactivity was undegraded captan, 85.6% had been evolved as CO_2, 0.8% had been evolved as organic compounds (not further characterized), and 16.6% was unextractable [^{14}C]residues. The material

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balances ranged from 82.1-108.8% of the applied during the 30-day study (values <99% were from day 1 only).

DISCUSSION:

1. The captan-treated soils were aged for longer than one half-life; only 19.4% of the applied radioactivity was captan when the samples were converted to anaerobic conditions.

2. The study was terminated after only 30 days, although EPA guidelines specify a 60-day study. However, at 30 days posttreatment,80.9% of the applied residues had been volatilized and only 4% of the applied captan remained undegraded.

3. The same day-1 samples were used for this study and the aerobic metabolism study (Study 3). Two of the four day-0 samples (those treated at 6.1 ppm) were used for this study and the aerobic metabolism study.

4. The study authors stated that the quantity of organic volatiles trapped was too small to identify.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Trichloromethyl-labeled [^{14}C]captan (radiochemical purity >99.6%, specific activity 40.4 mCi/mmol, Amersham) was applied at 4.6 and 6.1 ppm to flasks containing moist (80% of field capacity) sandy loam soil (58% sand, 30% silt, 12% clay, 1.2% organic matter, pH 7.2, CEC 7.7 meq/100 g) using a direct-displacement digital microdispenser. The application rate was determined by measuring the amount of stock solution dispersed from the microdispenser, rather than by analyzing treated soil. Individual flasks were attached to independent continuous air-flow systems; humidified air was drawn through the sample flask and then sequentially through traps containing ND-103 (an aromatic solvent) and 2-methoxyethanol:ethanolamine (6:4, v:v). The treated soils were incubated in the dark at 25°C. At 1 day posttreatment, the air flow was switched to nitrogen gas to convert the system to anaerobic conditions. One hour prior to sampling, the soil to be sampled was acidified with 1 M $NaHSO_4$ to remove residual $^{14}CO_2$ and volatile collection continued until sampling. Quadruplicate samples (two each from the 4.6 and 6.1 ppm treatments) were taken at 0 days posttreatment; duplicate samples were taken at 1 day (6.1 ppm treatment) and 30 days (4.6 ppm treatment) posttreatment.

Soil samples were extracted three times by blending the soil with ethyl acetate on a mixer for 10 minutes. The three extracts were combined, and aliquots were analyzed for total radioactivity by LSC. Additional aliquots were evaporated to dryness in a vacuum rotary evaporator at ambient temperature, the residues were redissolved in acetonitrile, and aliquots of the acetonitrile solution were analyzed by LSC. The acetonitrile solutions were analyzed by ion-pairing HPLC with radioactive flow detection and by TLC. The solutions were cochromatographed with unlabeled captan, thiazolidine-2-thione-4-carboxylic acid, and dithiobis(methane sulfonic acid) reference standards on silica gel TLC plates in two dimensions, using chloroform:acetic acid (40:1, v:v) and n-propanol:water (7:3, v:v) solvent systems. Radioactive areas on the plates were located using autoradiography; the unlabeled standards were visualized by quenching of UV fluorescence. The ethyl acetate-extracted soil was analyzed for unextractable radioactivity by LSC following combustion.

The trapping solutions were analyzed by LSC. The trapping efficiency of the 2-methoxyethanol:ethanolamine solution was 108%.

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_____ Description of product quality control procedures.

_____ Identity of the source of product ingredients.

_____ Sales or other commercial/financial information.

_____ A draft product label.

_____ The product confidential statement of formula.

_____ Information about a pending registration action

__X__ FIFRA registration data.

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DATA EVALUATION RECORD

STUDY 5

CHEM 081301 Captan §163-1

FORMULATION--90--FORMULATION NOT IDENTIFIED

FICHE/MASTER ID 40658011
Pack, D.E. 1977. Soil mobility of captan, folpet, and captafol as determined by soil thin-layer chromatography. File No:722.0. Prepared and submitted by Chevron Chemical Company, Richmond, CA.

FICHE/MASTER ID 40658011
Pack, D.E. 1987. Estimation of soil adsorption coefficient of captan from TLC data. Laboratory Project ID MEF-0073/8726836. Prepared and submitted by Chevron Chemical Company, Richmond, CA.

REVIEWED BY: K. Patten TITLE: Staff Scientist

EDITED BY: J. Harlin TITLE: Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD
TEL: 468-2500

APPROVED BY: L. Lewis
TITLE: Environmental Scientist
ORG: EAB/HED/OPP
TEL: 557-7442

Laurie C. Lewis

AUG 4 1988

SIGNATURE:

CONCLUSIONS:

Mobility - Leaching and Adsorption/Desorption

This study is acceptable and partially fulfills EPA Data Requirements for Registering Pesticides by providing information on the mobility (soil TLC) of unaged [^{14}C]captan in sandy loam, silty clay loam, two clay loam, and clay soils. An additional study is needed to determine the mobility of captan degradates in one soil, preferably a sandy loam.

SUMMARY OF DATA BY REVIEWER:

Using soil TLC methods, [^{14}C]captan (test substance not further characterized) was determined to be slightly mobile (Helling's mobility class 2) in Crowley clay loam soil (R_f 0.21), Macksbury silty clay loam soil (R_f 0.15), and Nicollet clay loam soil (R_f 0.14), and immobile (Helling's

71

mobility class 1) in Oakley sandy loam (R_f 0.09) and Stockton adobe clay (R_f 0.08) sieved through a 500 µm sieve.

DISCUSSION:

1. The soils were ground with a mortar and pestle and sieved through a #35 (500 µm mesh) sieve, which would remove the coarse sand fraction and possibly cause the pesticide to appear less mobile. However, it is unlikely that repeating the study with properly sieved soil would significantly alter the mobility classification, since the pesticide is of very low mobility and the soils were primarily fine-textured.

2. The study was not conducted using a soil with ≤1% organic matter. Also, four of the five soils contained 34-38% clay.

3. The [^{14}C]captan was not characterized; label position, radiochemical purity, and specific activity were not reported.

4. Using the observed R_f values, soil K_d values of 3 to 8 were calculated.

5. Mobility of aged captan residues was not addressed.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Sandy loam, silty clay loam, clay loam, and clay soils were ground with a mortar and pestle, sieved through a #35 (500 μm mesh) sieve, and mixed with water. The slurries were spread on glass plates to a 500 μm thickness, and the plates were allowed to dry. Each plate was spotted with 40,000 dpm each of [^{14}C]captan, [^{14}C]folpet, [^{14}C]captafol, [^{14}C]acephate, [^{14}C]thiobencarb, [^{14}C]BUX, and [^{14}C]paraquat. The plates were developed in water for 3-4 hours, air-dried, and visualized with autoradiography. R_f values were determined from the center of each spot.

Soil K_d values (adsorption coefficients) were calculated from the R_f values.

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REFERENCES

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Pack, D.E. 1987. Estimation of soil adsorption coefficient of captan from TLC data. Laboratory Project ID MEF-0073/8726836. Prepared and submitted by Chevron Chemical Company, Richmond, CA. (40658011)

Pack, D.E. and I.S. Verrips. 1988a. Aerobic soil metabolism of [trichloromethyl-^{14}C]captan. Laboratory Project ID MEF-0060/8809887. Prepared and submitted by Chevron Chemical Company, Richmond, CA. (40658007)

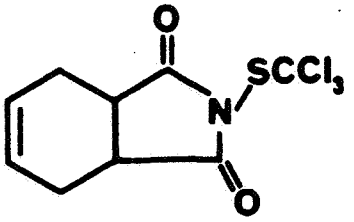
Pack, D.E. and I.S. Verrips. 1988b. Anaerobic soil metabolism of [trichloromethyl-^{14}C]captan. Laboratory Project ID MEF-0061/8809887. Prepared and submitted by Chevron Chemical Company, Richmond, CA. (40658008)

Ruzo, L.O., A.L. Kesterson, S.B. Jackson, and L.J. Lawrence. 1988a. Soil surface photolysis of [^{14}C]captan in natural sunlight. Laboratory Project ID ORTHO:MEF-0068. PTRL Report No. 1142. PTRL Project No. 232. Prepared by Pharmacology and Toxicology Research Laboratory (PTRL), Lexington, KY, and submitted by Chevron Chemical Company, Richmond, CA. (40658010)

Ruzo, L.O., A.L. Kesterson, S.B. Jackson, and L.J. Lawrence. 1988b. Soil surface photolysis of [^{14}C-trichloromethyl]captan in natural sunlight. Laboratory Project ID ORTHO:MEF-0075. PTRL Report No. 231-2. PTRL Project No. 231. Prepared by Pharmacology and Toxicology Research Laboratory (PTRL), Lexington, KY, and submitted by Chevron Chemical Company, Richmond, CA. (40658009)

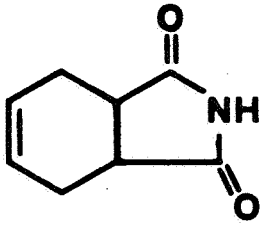
APPENDIX

CAPTAN AND ITS DEGRADATES



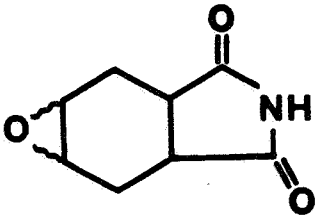
Captan

N-[Trichloromethylthio]cyclohex-4-ene-1,2-dicarboximide



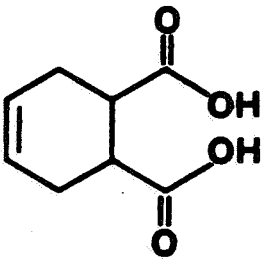
TPHI

Cyclohex-4-ene-1,2-dicarboximide



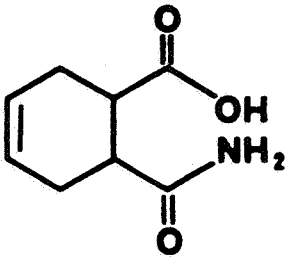
THPI Epoxide

Cyclohex-4,5-epoxy-1,2-dicarboximide



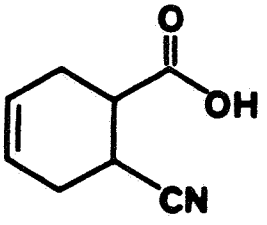
THPAL

Cyclohex-4-ene-1,2-dicarboxylic acid



THPAM

Cyclohex-4-ene-2-amido-1-carboxylic acid



THCY

Cyclohex-4-ene-2-cyano-1-carboxylic acid

Shaughnessy #: 081701

Date out of EAB: _____

Signature: _____

To: H. Jacoby
Product Manager # 21
Registration Division (TS-767)

From: John Jordan, Ph.D., Acting Chief
Registration Standards, Section #3
Exposure Assessment Branch
Hazard Evaluation Division (TS-769)

Attached please find the EAB review of:

Reg./File No.: 239-2230

Chemical: Captafol

Type Product: Fungicide

Product Name: _____

Company Name: Chevron

Submission Purpose: Response to RS data gaps

Action Code: 625

Date In: 4/25/85

EAB # 5564

Date Completed: _____

TAIS (level II) Days

15

Deferrals To:

_____ Ecological Effects Branch

_____ Residue Chemistry Branch

_____ Toxicology Branch

27

CONCLUSION

Mobility - Adsorption/Desorption

1) Study is scientifically valid

2) Study in part fullfills EPA Guidelines for Registration of Pesticides 1983 section 163-1

3) Study documents the adsorption/desorption properties of captafol and the ring degradation products; however, the fate of the side chain, 1,1,2,2 tetrachlorethane thiol, shown to be the remaining hydrolysis product, is not discussed.

4) Captafol is strongly sorbed whereas THPI and THPAM are weakly sorbed. The fate of the 1,1,2,2, tetrachlorethane thiol is underscribed.

Materials and Methods

Table 1 charcterizes the physical properties of the soils used this study. The soils were seived through a 100 mesh seive. The percent dry matter was determined by drying a sample overnight at 100°C.

The test materials were [ethyl-1-^{14}C-] captafol specific activity 113,600 dpm/ug, [carbonyl-^{14}C] THPI specific activity 52,800 dpm/ug and carbonyl-^{14}C] THPAM at 47,200 dpm/ug. The [carbonyl-^{14}C] THPAM was prepared by controlled hydrolysis overnight in dilute NaOH.

Prior to use, the compounds were purified by preparatory TLC on 0.25 mm Silca gel 60 F-254. Labelled captafol was located by quenching of UV florescence. THPI and THPAM was located by autoradiography. All compounds were eluted from the silca gel with acetone.

HPLC analysis showed these compounds to be 99% radiochemically pure.

Counting was done on a Beckman Model 9800 liquid scintillation counter with an external H# method. Background was 35 cpm with a 95% counting efficiency. All samples were counted in 10 ml Hydrocount (J.T. Baker, Cat No. 7635-2).

A stock solution of labelled captafol was prepared in acetone and a second stock solution of labelled THPI and THPAM was prepared in methanol. Since captafol is hydrolyzed rapidly at pH's above 5,

the adsorption tests were done at pH 5. Soils (1g dry weight) were mixed with 5 volumes of 0.01 M $CaSO_4$ and the necessary amount of dilute HCl or NaOH needed to bring the soil to pH 5 . After an hour equilibration, aliquots THPI - THPAM or captafol stock solutions were added. A magnetic stirring bar added, the vials capped and placed in thermostated (25°C) water bath on a magnetic stirrer and the stirrer started. Samples were removed after 30 minutes stirring. The soil water mixture was immediately centrifuged and the supernatant filtered through a centrix centrifugal microfilter. Two 50 uL aliquots were removed for counting; the remainder was used for HPLC analysis. Analysis was run using ion pairing. The aqueous phase consisted 0.005M Q7 (heptytheithylammonium phosphate) and 0.001M KH_2PO_4 and 0.005M NaN_3 which was adjusted to pH 5.0 with H_3PO_4. The organic modifier was methanol.

The adsorption data were fitted to the Freundlich adsorption equation $\frac{X}{M} = KC^{1/n}$

X = weight of adsorbed chemical

M = weight of solid adsorbent

C = concentration in equilibrium solution

k and n are constants which can be found by taking logarithms of both sides of the equation.

$$\log \frac{X}{M} = \log K + 1/n \log C$$

These values were calculated by linear regression analysis of log transformed data.

Reported Results:

The K value described above is a measure of the amount absorbed which is in equilibrium with a solution of unit concentration. The higher the K value, the stronger the adsorption.

Tables 2-4 and figures 1-3 described in adsorption of captafol THPI and THPAM to the various soils used in this study. The data indicate that captafol is more strongly adsorbed than THPI or THPAM.

Discussion

1) The study is scientifically valid and partially acceptable under EPA Guidelines for the Registration of Pesticide 1983 section 163-1.

9 9

2) Based on the uses of this chemical, sufficient soil types and classes were used to described the adsorption/desorption reactions of the ring portion of captafol.

3) The fate and adsorption/desorption reactions of the 1,1,2,2. Tetrachloroethane thiol sidechain is not described in this document and must be.

Captan Science Reviews

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CONCLUSIONS

Mobility - Volatility

Captafol has a reported vapor pressure of 1.1×10^{-6}mm of Hg. Although this is considered to be low, all data are required. Requirements for field volatility data are contingent on the results of the laboratory volatility. Of particular interest will be the results of the fate, half-life and volatility of the 1,1,2,2 tetrachloroethane thiol sidechain.

EXPOSURE ASSESSMENT BRANCH
PESTICIDE ENVIRONMENTAL FATE ONE-LINER

CAPTAN

File No.: 081301 CAS No.: 133-06-2
Type Pesticide: Fungicide
Chemical Name: N-(trichloromethylthio)-4-cyclohexene-1,2-dicarboximide

Empirical Form.: C9H8Cl3NO2S

Uses: Variety of fruit, vegetable, nut, and ornamental crops; seed treatment; postharvest dip; nonfood uses; incorporated into plastics, paints/pastes, textiles, paper, and cosmetics.

Form.Type: WP, SP, D, Seed treatment

Mole Wt.	Sol.@20C (ppm)	Vap.Pres (torr)	Log KOW
300.61	Practically insoluble in water	$<10^{-6}$ mm Hg @25C	

Hydrolysis (161-1)
pH 5:
pH 7: 0.25 days#
pH 9: 3.6 minutes#

Photolysis (161-2, -3, -4)
Air:
Soil:
Water: 10 hours (pH 5, not photodegraded)

Mobility Studies (163-1)

Soil Partition (Kd)	Rf Factors
1	0.08-0.21#
2	
3	
4	
5	

Soil Metabolism Studies - Terrestrial

Aerobic (162-1)	Anaerobic (162-2)
1 14-21 days (volcanic ash)#	<7 days (loamy sand)#
2 1-2 days	
3 2-3 days (loam)#	
4 99% degraded within 7 days (sandy loam)#	
5 3 days (sandy loam)	
6 <1 day (sandy loam)	
7	

Soil Metabolism Studies - Aquatic

Aerobic (162-4)	Anaerobic (162-3)
1	
2	
3	
4	

Field Dissipation Studies

Terrestrial (164-1)	Aquatic (164-2)
1	
2	
3	
4	
5	
6	

Field Dissipation Studies

Forest (164-3)	Other (164-5)
1	
2	

Ground Water Findings
1
2
3

Rotational Crop Restrictions (165-1, -2)
1
2

Fish Accumulation Studies (165-4)
1
2

Degradation Products
1 delta^4-Tetrahydrophthalimide
2 delta^4-Tetrahydrophthalmic acid
3 N-(trichloromethylthio)-4,5-epoxyhexahydrophthalimide
4 4,5-epoxyhexahydrophthalimide
5 3-Hydroxy-delta^4-tetrahydrophthalimide
6 5-Hydroxy-delta^4-tetrahydrophthalimide
7 delta^4-Tetrahydrophthalic acid
8 4,5-Dihydroxyhexahydrophthalimide
9 Phthalimide
10 3-Hydroxy-delta^4-tetrahydrophthalamic acid

Notes

References: EAB Files
Writer: L. Lewis

Shaughnessy No.: 081301-4

Date Out of EAB: 8/11/88

TO: Richard F. Mountfort
Product Manager #23
Registration Division (TS-767C)

FROM: Frank L. Davido, Chief Frank Davido
Field Studies and Special Projects Section #5
Exposure Assessment Branch/HED (TS-769C)

THRU: Paul F. Schuda, Chief
Exposure Assessment Branch/HED (TS-769C) Paul F. Schuda

Attached, please find the EAB review of...

Reg./File # : 239-1246

Chemical Name: Captan

Type Product : Fungicide

Product Name :

Company Name : CAPTAN Task Force (I.C.I. Americas Inc.)

Purpose : Submission of nine protocols for reentry studies being conducted with Captan

Action Code: 352

EAB #(s) : 80911

Date Received: 8/2/1988

TAIS Code: 49

Date Completed: 8/11/88

Total Reviewing Time: 8 hours

Monitoring study requested: NO

Monitoring study voluntarily: NO

Deferrals to:

NO Ecological Effects Branch

NO Residue Chemistry Branch

NO Toxicology Branch

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REVIEW OF REENTRY DATA

1. CHEMICAL:

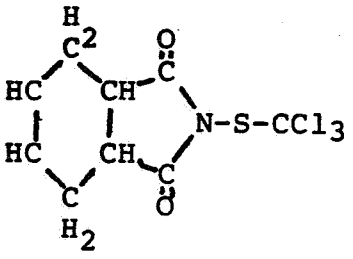
Common name: Captan

Product name: Captan 50 WP.

Chemical name:

N-(trichloromethylthio)-cis-cyclohex-4-ene-1,2-dicarboximide

Structure: $C_9H_8Cl_3NO_2S$ MWt 300.59



Other names: Captane, Merpan, Pillarcap, Vondcaptan, Vancide 89, CAS # 133-06-2, RTECS # GW5075100.

2. TEST MATERIAL:

Foliar dislodgeable residues, fine soil residues, and dermal and inhalation exposure samples gathered at intervals after maximum allowed application of Captan 50 WP.

3. STUDY/ACTION TYPE:

Review of protocols for 7 studies.

4. STUDY IDENTIFICATION:

Reg. File Nos. 239-1246
Accession No. NONE
Record Nos. 228,369
Letter dated 7/20/88 from Anne Mueller, Chairman CAPTAN Task Force, to Richard Mountfort, RD.

5. REVIEWED BY:

James D. Adams, PhD
Chemist
Field Studies and Special Projects Section #5 8/11/1988

6. APPROVED BY:

Frank Davido, Chief
Field Studies and Special Projects Section #5
Exposure Assessment Branch, HED (TS-769) 8/11/1988

7. CONCLUSIONS:

Not all of the data being gathered are required by Subdivision K and 40 CFR § 158.390 (formerly § 158.140). The protocols are not completely in accord with the suggested protocols of Subdivision K, but data gathered according to the protocols are expected to be useful for assessment of fieldworker exposure.

8. RECOMMENDATIONS:

No action is necessary. The testing is underway and nearing completion.

9. BACKGROUND:

On October 16, 1987, Karen E. Warkentien, Special Review Section, EAB, reviewed a protocol for captan reentry exposure studies. On December 3, 1987, J. C. White, Chevron Chemical Co. (and in his role as Chairman of the Captan Task Force) submitted a 4 page protocol for reentry exposure studies in 5 crops. There were no dislodgeable residue studies proposed.

10. DISCUSSION OF INDIVIDUAL TESTS OR STUDIES:

This submission is to provide information on the current activities of the Captan Task Force. It consists of 7 protocols for reentry studies presently being conducted as follows:

1. Soil and foliar dislodgeable residues with apple trees in New York State;
2. Soil and foliar dislodgeable residues with peach trees in the San Joaquin Valley;
3. Soil and foliar dislodgeable residues with grape vines in the San Joaquin Valley;
4. Soil and foliar dislodgeable residues with tomato crop in Madera, California;
5. Harvester exposure study after application of Captan on Peach trees in the San Joaquin Valley;
6. Soil and foliar dislodgeable residues with strawberry crop in Salinas, California; and
7. Harvester exposure study after application of Captan on strawberry crop in Salinas, California.

All studies are being conducted after maximum allowed application of Captan 50 WP by normal application techniques.

Registrants are not required, but are encouraged to submit protocols for reentry studies. When such protocols are received, they are reviewed and suggestions are made as appropriate. Since the studies are already underway and nearing completion, any further advice on the protocols to the Task Force from EAB is redundant.

RD is asking if the data being gathered will satisfy the requirements of 40 CFR § 158.390 and Subdivision K. The protocols are largely acceptable, but two things should be brought out. First; the soil residue data that is being gathered is not required for crops such as grapes and tree fruit. Such data are only required if crop practices, "... will involve exposure of workers to large amounts of soil..." [Subdivision K, § 132-2(c)(7)(ii)(A)]. Soil residue may be of interest for strawberry and tomato harvester exposures because there may be some exposure to soil in those crops, but that issue is not resolved.

Second; the exposure studies proposed above in peach and strawberry crops are not required by Subdivision K. Exposure studies may be submitted to support a proposed reentry interval at the Registrant's option, but reentry data could be limited to the dislodgeable residue data required by Subdivision K.

11. COMPLETION OF ONE-LINER:

NOT APPLICABLE

12. CBI APPENDIX:

NOT APPLICABLE