

Chemical Code: **106401**

Date Out:

~~56 DEC 1993~~
56 DEC 1993

ENVIRONMENTAL FATE AND GROUND WATER BRANCH

Review Action

To: Walt Waldrop, PMT #71
Reregistration Division (H7508W)

From: Paul J. Mastradone, Chief *PJM*
Environmental Chemistry Review Section #1
Environmental Fate & Ground Water Branch/EFED (H7507C)

Thru: Henry Jacoby, Chief *Henry Jacoby 12/1/93*
Environmental Fate & Ground Water Branch/EFED (H7507C)

Attached, please find the EFGWB review of...

DP Barcode:	D159474, D165114, D179060, D186692, D192906		
Common Name:	Difenzoquat methyl sulfate	Trade name:	AVENGE
Company Name:	American Cyanamid Company		
ID #:	106401		
Purpose:	List A RED: Review environmental fate data.		

Type Product:	Action Code:	EFGWB #(s):	Review Time:
Herbicide	627	91-0273, 91-0655, 92-1007, 93-0328, 93-0887	20 days

STATUS OF STUDIES IN THIS PACKAGE:

Guideline #	MRID	Status ¹
161-1	41325403	A
161-2	41325404	A
161-3	41325405	A
162-1	41903701	A
162-2	41903702	A
163-1	41703401	A

STATUS OF DATA REQUIREMENTS:

	Status ²
161-1	S
161-2	S
161-3	S
162-1	S
162-2	S
163-1	S

¹Study Status Codes: A=Acceptable U=Upgradeable C=Ancillary I=Invalid.
²Data Requirement Status Codes: S=Satisfied P=Partially satisfied N=Not satisfied R=Reserved.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460



OFFICE OF PREVENTION,
PESTICIDES AND TOXIC
SUBSTANCES

- 6 DEC 1993

Chemical Barcode #: 106401
DP Barcode #: D159474, D165114, D179060,
D186692, D192906
EFGWB #: 91-0273, 91-0655, 92-1007,
93-0328, 93-0887

MEMORANDUM

SUBJECT: Difenzoquat - RED Candidate

FROM: Henry M. Jacoby, Chief
Environmental Fate and Ground Water Branch
Environmental Fate and Effects Division (H7507C)

TO: Lois Rossi, Chief
Reregistration Branch
Special Review and Reregistration Division (H7508W)

Evert Byington, Chief
Science Analysis and Coordination Staff
Environmental Fate and Effects Division (H7507C)

Attached is the environmental fate and ground water package for the difenzoquat List A RED. The package contains an environmental fate assessment and reviews of environmental fate studies received by EFGWB. Except for field dissipation studies, all of the required environmental fate data requirements are fulfilled at this time (hydrolysis [161-1], photolysis in water [161-2], photolysis on soil [161-3], aerobic [162-1] and anaerobic [162-2] soil metabolism and leaching and adsorption/desorption [163-1]).

A preliminary assessment of the environmental fate of difenzoquat indicates that soil binding appears to be the principal route of dissipation. This assessment is supported by laboratory data which shows a high degree of adsorption to soil but no degradation of the parent material. Nevertheless, the field dissipation studies contrast sharply with laboratory data and indicate that difenzoquat residues decline with time. However, because different methods were used in the laboratory and field studies to extract difenzoquat from soil, EFGWB can not conclude at this time that difenzoquat is bound in the field soils. Because the laboratory and field data differ, EFGWB is unable to determine the principal route of dissipation of difenzoquat with a high degree of confidence at this time.

EFGWB notes that when the six field dissipation studies were reviewed in 1975 they were judged supplemental but did not fulfill EPA Data Requirements for Registering Pesticides primarily because the soil freezer storage stability data was not provided. However, under present day review criteria these studies are not acceptable primarily because the soils were not sampled deep enough to define the extent of leaching and many details needed to judge the validity of the studies are lacking. Furthermore, the registrant should describe a route of dissipation of difenzoquat and its residues. In these field studies, half-lives varied from 49-75 days in California to 254-354 days in Oregon.

If SRRD feels that a more thorough environmental fate assessment is needed to make its decision on the reregistration of difenzoquat, EFGWB recommends that the registrant perform a non-guideline laboratory study comparing the recovery of ¹⁴C-difenzoquat between the methods used in laboratory and field studies. This added information is needed to assist EFGWB in determining if the major route of difenzoquat dissipation is soil binding. The soils in these new studies should be the same as used in the field studies.

Furthermore, since the field dissipation studies are so seriously flawed, EFGWB recommends that at least two additional field dissipation studies be conducted. These studies may confirm the laboratory results and should address the route of dissipation of difenzoquat and any major degradates in actual use conditions. The information gained from these studies may enable EFGWB to determine the persistence, potential mobility and route of dissipation of difenzoquat under actual use conditions.

Although most of the data requirements are fulfilled, and indicate that difenzoquat is persistent in the environment and immobile in the soil, a sound quantitative assessment of the fate of difenzoquat in the environment cannot be made at the present time because of the lack of key data. EFGWB cannot bridge the studies together into a coherent description of the environmental fate of difenzoquat nor explain the discrepancies between laboratory (which indicate persistence) and field (which indicate slow to moderate dissipation) data. Nevertheless, it appears from the laboratory data that difenzoquat is immobile in the soil and the potential for ground water contamination is minimal. Furthermore, since the chemical binds tightly to soil, runoff of soil-bound residues to near bodies of water may occur. Whereas the available laboratory and field data have not demonstrated significant leaching of residues of difenzoquat or its degradates, the field data are extremely limited due to lack of residue analysis of soil below the 6- to 9-inch depth and the variability of the data.

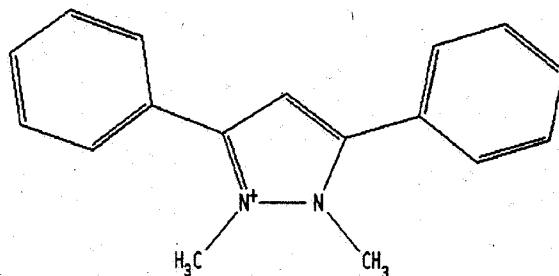
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1.0 CHEMICAL:

Common Name: Difenzoquat

Chemical Name: 1, 2-dimethyl-3-5-diphenyl-1H-pyrazolium methyl sulfate

Chemical Structure:



Formulations: Soluble concentrate/liquid and soluble concentrate/solid

Physical/Chemical properties:

Molecular Formula:	C ₁₈ H ₂₀ N ₂ O ₄ S
Molecular Weight:	360.4
Vapor Pressure:	<9.06 x 10 ⁻⁸ mm Hg at 35°C
Melting Point:	156.5-158°C
Solubility:	765,000 ppm in water @23°C; 9.75 mg/ml in acetone; 71.2 mg/ml in ethylene glycol; 588 mg/ml in methanol; n- <10 mg/ml in n-heptane and xylene.
Log K _{ow} :	-0.320 to 0.648 depending on pH

2.0 TEST MATERIAL: Discussed in individual DERs.

3.0 STUDY/ACTION TYPE:

Prepare EFGWB Science Chapter for Difenzoquat List A RED.

Review hydrolysis (161-1), photolysis in water (161-2) and soil (161-3), aerobic (162-1) and anaerobic metabolism (162-2), leaching/adsorption/desorption (163-1) and storage stability studies.

4.0 STUDY IDENTIFICATION:

Chudwudebe, A. 1991. Difenzoquat methyl sulfate (CL 84,777): Freezer stability of residues of CL 84,777 in soil at the 12-month interval. Laboratory Report No. C-3528. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41903703)

Gross, J. 1992. Difenzoquat methyl sulfate (CL 84,777): Freezer stability of residues of CL 84,777 in soil after twenty-four (24) months. Laboratory Report No. C-3781. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (42327501)

Mangels, G. 1989a. Difenzoquat (AC 84,777): Hydrolysis. Laboratory Project No. E-89-3. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41325403)

Mangels, G. 1989b. Difenzoquat (AC 84,777): Photodegradation in water. Laboratory Project No. E-89-6. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41325404)

Mangels, G. 1989c. Difenzoquat (AC 84,777): Photolysis on soil. Laboratory Project No. E-89-24. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41325405)

Mangels, G.D. 1988a. Difenzoquat (AC 84,777): Aerobic soil metabolism. Laboratory Report No. PD-M 25-58. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41903701)

Mangels, G.D. 1988b. Difenzoquat (AC 84,777): Anaerobic soil metabolism. Laboratory Report No. PD-M 25-50. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41903702)

Mangels, G. 1987. Difenzoquat (AC 84,777): Adsorption/desorption on soil. Laboratory Report No. PD-M 24-1. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41703401)

5.0 REVIEWED BY: Richard J. Mahler, Hydrologist
Environmental Chemistry Review Section 1
EFGWB/EFED

SIGNATURE:

Richard J. Mahler

DATE:

6 DEC 1993

4.0 APPROVED BY: Paul J. Mastradone, Chief
Environmental Chemistry Review Section 1
EFGWB/EFED

SIGNATURE: Paul J. Mastradone

DATE: 6 DEC 1993

7.0 CONCLUSION:

7.1 Although most of the data requirements are fulfilled, and indicate that difenzoquat is persistent in the environment and immobile in the soil, a sound quantitative assessment of the fate of difenzoquat in the environment cannot be made at the present time. EFGWB can not connect the studies together into a coherent description of the environmental fate of difenzoquat and can not explain the discrepancies between laboratory (which indicate persistence) and field data (which indicate persistence to moderately rapid dissipation). Until EFGWB has a better understanding of the route of dissipation of difenzoquat, a complete environmental fate, ground water leaching and surface runoff assessment cannot be made. Although the available laboratory and field data have not demonstrated significant leaching of residues of difenzoquat or its degradates, the field data are extremely limited due to lack of residue analysis of soil below the 6- to 9-inch depth. However, it appears from the laboratory data that difenzoquat is immobile in the soil and the potential for ground water contamination is minimal. Since the chemical binds tightly to soil, surface runoff of soil-bound residues may occur.

A preliminary assessment of the environmental fate of difenzoquat indicates that soil binding appears to be the principal route of dissipation. This assessment is supported by laboratory data which shows a high degree of adsorption to soil but no degradation of the parent material. Nevertheless, the field dissipation studies contrast sharply with laboratory data and indicate that difenzoquat residues decline with time. However, because different methods were used in the laboratory and field studies to extract difenzoquat from soil, EFGWB can not conclude at this time that difenzoquat is bound in the field soils. Because the laboratory and field data differ, EFGWB is unable to determine the principal route of dissipation of difenzoquat with a high degree of confidence at this time.

7.2 The following environmental fate studies submitted in support of reregistration have been reviewed and are included in the RED:

Hydrolysis (161-1)

Difenzoquat (1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate) did not hydrolyze in sterile aqueous buffered solutions (pH 5, 7, and 9) that were incubated in the dark at 25 C for 28 days. Using three different TLC systems, [¹⁴C]difenzoquat was the only compound detected in the treated solutions at 28

days posttreatment. During the study, material balances ranged from 98.2 to 101.1% of the applied with no discernable pattern of decline.

Photolysis in water (161-2)

Difenzoquat (1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate) did not degrade in sterile aqueous buffered (pH 7) solutions that were continuously irradiated with a xenon arc lamp at 25 C for 28 days. Using three different TLC systems, [¹⁴C]difenzoquat was the only compound detected in the irradiated and dark control solutions at 28 days posttreatment. During the study, material balances ranged from 98.6 to 101.6% of the applied with no discernable pattern of decline.

Photolysis on soil (161-3)

Difenzoquat (1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate) did not degrade on sandy loam soil that was continuously irradiated with a xenon arc lamp for 28 days at 25 C. Based on data from three different TLC systems, [¹⁴C]difenzoquat comprised 98.0-99.8% of the applied radioactivity in the irradiated and dark control solutions at 28 days posttreatment. Two unidentified [¹⁴C]compounds were each $\leq 1.4\%$ of the applied in the irradiated and dark control solutions; unidentified "1" was detected at all sampling intervals, and unidentified "2" was detected only at 3 and 4 weeks posttreatment. During the study, the material balances ranged from 99.07 to 106.23% of the applied with no discernable pattern of decline.

Aerobic soil metabolism (162-1)

Difenzoquat (1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate) did not degrade in aerobic sandy loam soil that was incubated for 1 year in the dark at 20 C and 75% of 0.33 bar moisture. During the study, extractable [¹⁴C]residues totaled 96.9-98.9% of the applied; [¹⁴C]difenzoquat was the only compound detected in the soil extracts at 12 months posttreatment. Unextracted [¹⁴C]residues in the soil were 1.1-3.1% of the applied at all sampling intervals, and [¹⁴C]volatiles (organic and ¹⁴CO₂) totaled $<1\%$ at 12 months posttreatment. During the study, the material balances ranged from 97.10 to 107.68% of the applied with no discernable pattern of decline.

Anaerobic soil metabolism (162-2)

Difenzoquat (1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate) did not degrade in anaerobic (flooded plus oxygen-free atmosphere) sandy loam soil that was incubated in the dark at 20 C for 2 months. During the study, extractable [¹⁴C]residues associated with the soil totaled 96.5-102.2% of the applied; during anaerobic incubation, only 0.14-0.21% was associated with the floodwater. [¹⁴C]Difenzoquat was the only compound detected in the soil extracts. Unextracted [¹⁴C]residues in the soil were 1.18-2.48% of the applied at all sampling intervals. [¹⁴C]Volatiles (organic and ¹⁴CO₂) were not detected during the aerobic portion of the experiment, and were not measured during the anaerobic portion of the experiment. During the study, the material balances ranged from 99.06 to 104.23% of the applied with no discernable pattern of decline.

Leaching and adsorption/desorption (163-1)

Based on batch equilibrium experiments, difenzoquat (1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate) was immobile in sandy loam, sandy clay loam, silt loam, and clay loam soils, with Freundlich K_{ads} values of 124-685 in calcium chloride solutions and 181-2680 in water. The study author suggested that the difference between the calcium chloride and water solutions may result from the competitive interaction of the calcium chloride and difenzoquat methyl sulfate for binding sites due to the charged nature of difenzoquat methyl sulfate.

7.4 Environmental Fate Assessment:

Based upon a review of the submitted studies, most of which were found to be acceptable, difenzoquat is persistent (the chemical did not degrade in any of the laboratory studies performed: hydrolysis, aqueous and soil photolysis, and aerobic and anaerobic soil metabolism).

Difenzoquat is relatively immobile (K_{ds} ranged from 124 to 685, K_{ocs} ranged from 23,071 to 36,231). In aged and unaged soil column leaching studies with sand, sandy loam and silt loam soils, 90.88 and 96.7% of the applied radioactivity remained in the top 3.5 inches of the columns after 20 inches of water was applied, respectively, and 0.47-2.59 % was recovered in the leachates.

Data from the field dissipation studies indicate that the difenzoquat appears to be moderately persistent to very persistent, since half-lives varied from 49-75 (CA), 84-97 (SD), 105-116 (MT), 104-160 (MN, uncropped), 129-210 (MN, cropped) and 254-354 (OR) days under normal use conditions. These studies are not acceptable because the depth of leaching is not determinable since in most cases the soils were only sampled to 3-6 inch soil depth. Where the soil was sampled to 6-9 inch depth (at the end of the study), the amount of difenzoquat was always <0.1 ppm. Results from the field dissipation study along with data from the other environmental fate studies are used to determine the leaching potential of difenzoquat and whether groundwater monitoring studies are needed. However, since it can not be determined how far through the soil difenzoquat or its degradates have moved by leaching, because of insufficient soil sampling with depth, the potential for contamination of groundwater cannot be assessed.

7.5 Environmental Fate Summary of Previously Reviewed Studies:

Hydrolysis--161-1: (Ku, et al., 1974, MRID 00036788) Ring-labeled [^{14}C]difenzoquat methyl sulfate (radiochemical purity unspecified), at 20 ppm, was stable to hydrolysis during 4 weeks of incubation in the dark at 20-25°C in pH 5, 7, and 9 aqueous buffered sterile solutions. At 4 weeks posttreatment, $\geq 93.2\%$ of the applied [^{14}C]difenzoquat methyl sulfate remained undegraded. No degradates were detected (detection limit unspecified) in solution at any sampling interval. This portion of the study is scientifically sound and provides supplemental information towards the registration of difenzoquat methyl sulfate. This portion of the study does

not fulfill EPA Data Requirements for Registering Pesticides because the test substance and the buffer solutions were incompletely characterized.

Photolysis--161-3: (Ku, et al., 1974, MRID 00036788) Ring- and methyl-labeled [¹⁴C]difenzoquat methyl sulfate (radiochemical purity unspecified), at 5.5 ppm, did not degrade on silt loam soil (TLC plates) irradiated with natural sunlight (200-11,000 foot candles) at 22-42°C for 8 weeks. At 8 weeks posttreatment, difenzoquat methyl sulfate comprised $\geq 97.8\%$ of the applied on the irradiated soil and $\geq 101\%$ of the applied on the dark control. This portion of the study is scientifically sound and provides supplemental information towards the registration of difenzoquat methyl sulfate. This portion of the study does not fulfill EPA Data Requirements for Registering Pesticides because the natural sunlight was inadequately characterized; the test substance and test soil were incompletely characterized; and the incubation temperature ranged from 22-42°C.

Leaching/adsorption/desorption--163-1: (Hui, 1974, MRID 00043775) [¹⁴C]Difenzoquat methyl sulfate was immobile in columns (14-inch height) of sand, sandy loam, silt loam, and clay loam soils treated with difenzoquat methyl sulfate (test substance not further characterized) at 5.07×10^6 or 2.939×10^6 dpm and leached with 20 inches of deionized water. In the sand, sandy loam, and silt loam soil columns, 96.70% of the applied radioactivity remained in the top 3.5 inches of the columns and 1.45-2.59% was recovered in the leachate; total radioactivity in the soil columns was 81.88-104.57% of the applied. In the clay loam soil column, of the 55.29% total radioactivity recovered after leaching, 52.56% of the applied remained in the top 3.5 inches of soil and 0.732% was in the leachate.

Aged (30 days) [¹⁴C]difenzoquat methyl sulfate residues (uncharacterized) were immobile in a column (14 inches) of sandy loam soil treated with [¹⁴C]difenzoquat methyl sulfate (test substance not further characterized) at 13.027×10^6 dpm that had been aged in sandy loam soil under unspecified greenhouse conditions. The columns were leached with 0.5 inches of water per day for 45 days. Following leaching, 90.88% of the applied radioactivity remained in the top 3.5 inches of the column and 0.47% of the applied residues were recovered in the leachate; total radioactivity in the soil column was 91.67% of the applied. This study is scientifically sound and provides supplemental information towards the registration of difenzoquat methyl sulfate. This study does not fulfill EPA Data Requirements for Registering Pesticides because: unaged - the test substance was not fully characterized; and aged - the test substance was not fully characterized, the description of incubation conditions during the aging period was inadequate, and [¹⁴C]residues in the soil before and after leaching were not characterized.

Field dissipation--164-1: (Kim et al., 1975, MRID 00045626) Difenzoquat methyl sulfate (Avenge 2A-S, 2 lb/gallon EC), at 0.62 or 1.00 lb ai/A, dissipated with a half-life of 49-75 days in the 0- to 3-inch depth of field plots of sandy loam soil planted to wheat that were located in Corcoran, California.

Field dissipation--164-1: (Kim et al., 1974, MRID 00045627) Difenzoquat methyl sulfate (Avenge 2A-S, 2 lb/gallon EC), at 0.5 or 1.0 lb ai/A, dis-

sipated with a half-life of 104-160 days in the 0- to 3-inch depth of bare ground field plots of sandy loam soil that were located in Crookston, Minnesota.

Field dissipation--164-1: (Kim et al., 1974, MRID 00045628) Difenzoquat methyl sulfate (Avenge 2A-S, 2 lb/gallon EC), at 0.5 or 1.0 lb ai/A, dissipated with a half-life of 129-210 days in the 0- to 3-inch depth of field plots of sandy loam soil planted to wheat that were located in Crookston, Minnesota.

Field dissipation--164-1: (Kim et al., 1974, MRID 00045629) Difenzoquat methyl sulfate (Avenge 2A-S, 2 lb/gallon EC), at 0.5 or 1.0 lb ai/A, dissipated with half-lives of 105-116 days, respectively, in the 0- to 3-inch depth of field plots of clay loam soil planted to barley that were located in Huntley, Montana.

Field dissipation--164-1: (Kim et al., 1974, MRID 00045631) Difenzoquat methyl sulfate (Avenge 2A-S, 2 lb/gallon EC), at 1.00 or 2.00 lb ai/A, dissipated with half-lives of 254-354 days, respectively, in the 0- to 3-inch depth of field plots of sandy clay loam soil planted to wheat that were located in Corvallis, Oregon.

Field dissipation--164-1: (Kim et al., 1974, MRID 00045632) Difenzoquat methyl sulfate (Avenge 2A-S, 2 lb/gallon EC), at 0.5 or 1.0 lb ai/A, dissipated with a half-life of 84-97 days in the 0- to 3-inch depth of field plots of silt loam soil planted to wheat that were located in Redfield, South Dakota.

EFGWB notes that when these six field dissipation studies were reviewed in 1975 they were judged supplemental but did not fulfill EPA Data Requirements for Registering Pesticides primarily because the soil freezer storage stability data was not provided. However, under present day review criteria these studies are not acceptable primarily because the soils were not sampled deep enough to define the extent of leaching and many details needed to judge the validity of the studies are lacking. If difenzoquat degrades in the field as these studies imply, there should be analyses for degradates so that dissipation of the compound in actual use conditions can be assessed. Conversely, in the laboratory studies, the compound is stable to hydrolysis, aqueous and soil photolysis and aerobic and anaerobic soil metabolism. The registrant needs to explain the discrepancy.

8.0 RECOMMENDATIONS:

8.1 Although most of the data requirements are completely fulfilled, there is still insufficient information available from the acceptable and supplemental studies to make a sound quantitative assessment of the fate of difenzoquat in the environment. Until EFGWB has a better understanding of the route of dissipation of difenzoquat, complete environmental fate, ground water leaching and surface runoff assessments cannot be made. Moreover, the available data on field dissipation have not demonstrated significant leaching of residues of difenzoquat or its degradates. However, these data are extremely limited due to lack of

sufficient soil sampling depth (in most studies the soils were sampled only to the 3-6 inch depth). Nevertheless, it appears from the laboratory data that difenzoquat is immobile in the soil and the potential for ground water contamination appears to be minimal. Since laboratory data indicate that the chemical binds to soil, runoff of soil-bound residues to near bodies of water may occur.

If SRRD feels that a more thorough environmental fate assessment is needed to make its decision on the reregistration of difenzoquat, EFGWB recommends that the registrant perform a non-guideline laboratory study comparing the recovery of ¹⁴C-difenzoquat between the methods used in laboratory and field studies. This added information is needed to assist EFGWB in determining if the major route of difenzoquat dissipation is soil binding. The soils in these new studies should be the same as used in the field studies.

Furthermore, since the field dissipation studies are so seriously flawed, EFGWB recommends that at least two additional field dissipation studies be conducted. These studies may confirm the laboratory results and should address the route of dissipation of difenzoquat and any major degradates in actual use conditions. The information gained from these studies may enable EFGWB to determine the persistence, potential mobility and route of dissipation of difenzoquat under actual use conditions.

If the registrant decides to perform new terrestrial field dissipation studies, EFGWB recommends that the registrant follow the directions in the Standard Evaluation Procedure guidance document for terrestrial field dissipation studies (§ 164-1), 40 CFR Section 158.290 Subdivision N Guidelines, as well as PR Notice 86-5. These documents generally describe reporting and evaluation requirements which apply to studies conducted and submitted to support environmental fate studies.

- 8.2. The status of the Environmental Fate Data Requirements for reregistration of difenzoquat methyl sulfate for what is classified as a terrestrial food use pattern is as follows:

<u>Environmental Fate Data Requirements</u>	<u>Status of Data Requirement</u>	<u>MRID No.</u>
<u>Degradation</u>		
161-1 Hydrolysis	Fulfilled (RJM;11/XX/93)	41325403
162-1 Photodegradation in water	Fulfilled (RJM;11/XX/93)	41325404
162-3 Photodegradation on soil	Fulfilled (RJM;11/XX/93)	41325405

Metabolism

162-1 Aerobic (Soil)	Fulfilled (RJM;11/XX/93)	41903701
162-2 Anaerobic (Soil)	Fulfilled (RJM;11/XX/93)	41903702

Mobility

163-1 Leaching, Adsorption/ Desorption	Fulfilled (RJM;11/XX/93)	41703401
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Dissipation

164-1 Soil	Not fulfilled ¹	00045626, 00045627 00045628, 00045629 00045631, 00045632
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Accumulation

165-4 In Fish	Waived ²	
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Spray Drift

201-1 Drift Field Evaluation ³	Not Submitted	
202-1 Drift Size Spectrum ³	Not Submitted	

9.0 BACKGROUND:

Difenzoquat (methyl sulfate) or AVENGE is a selective postemergence herbicide registered for use to control wild oats (*Avena fatua*) in barley, wheat, rye, maize, ryegrass, alfalfa (seed crop in CA) and flax crops. Wild oats is one of the most serious annual weeds in the hard red spring wheat growing areas of Montana, North Dakota and Minnesota.

¹Although satisfactory freezer storage stability data have been submitted, under present day review criteria these studies are not acceptable primarily because the soils were not sampled deep enough to define the extent of leaching. Furthermore, the registrant should describe a route of dissipation of difenzoquat and its residues.

² Waived as per the Graybeard decision (HMJ/SCT, 4/6/93)

³ Spray drift data are required by 40 CFR §158.142 when aerial application and/or mist blower or other ground application are proposed and it is expected that the detrimental effect level of nontarget organisms (humans, domestic animals, fish, wildlife, and nontarget plants) expected to be present are exceeded. Furthermore, these data are required for all herbicides applied by air.

Difenzoquat is readily absorbed by plants and is not significantly metabolized or further degraded. The exact herbicidal mode of action of difenzoquat is not understood.

Difenzoquat is applied postemergence broadcast by ground or aerial equipment when wild oats are in the three to five leaf stage (tillering). Single active ingredient formulations include soluble concentrate/liquid and soluble concentrate/solid. The rates of application range from 0.6 to 1.0 lbs/A. This herbicide may be used on all varieties of barley and certain varieties of wheat. Since difenzoquat is highly selective in its weed control, it is usually tank mixed with broadleaf herbicides such as the amine salts or esters of MCPA (2-methyl-4-chlorophenoxyacetic acid) or 2,4-D (2,4-dichlorophenoxyacetic acid), metsulfuron methyl, chlorsulfuron, cereal fungicides and the growth regulator chlormequat chloride.

- 10.0 DISCUSSION OF INDIVIDUAL STUDIES: See attached DERs for review of individual studies.
- 11.0 COMPLETION OF ONE-LINER: Updated one-liner attached.
- 12.0 CBI APPENDIX: No claim of confidentiality was made by the registrant for any information contained in these studies on the basis of them falling within the scope of FIFRA 10(d)(1)(A), (B) or (C).

DIFENZOQUAT METHYL SULFATE

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INTRODUCTION

Difenzoquat (methyl sulfate) or AVENGE is a selective postemergence herbicide registered for use to control wild oats in barley, wheat, rye, maize, ryegrass, alfalfa (seed crop in CA) and flax crops. It is readily absorbed by plants and is not significantly metabolized or further degraded. The exact herbicidal mode of action of difenzoquat is not understood.

The only pest claim on the end-use products is for control of wild oats (Avena fatua) which is one of the most serious annual weeds in the hard red spring wheat growing areas of Montana, North Dakota and Minnesota. Wild oats is an annual grassy weed with growth habits that out-compete wheat and barley and create serious yield losses.

Difenzoquat is applied postemergence broadcast by ground or aerial equipment when wild oats are in the three to five leaf stage (tillering). Single active ingredient formulations include soluble concentrate/liquid and soluble concentrate/solid. The rates of application range from 0.6 to 1.0 lbs/A. This herbicide may be used on all varieties of barley and certain varieties of wheat. Since difenzoquat is highly selective in its weed control, it is usually tank mixed with broadleaf herbicides such as the amine salts or esters of MCPA (2-methyl-4-chlorophenoxyacetic acid) or 2,4-D (2,4-dichlorophenoxyacetic acid), metsulfuron methyl, chlorsulfuron, cereal fungicides and the growth regulator chlormequat chloride.

DATA EVALUATION RECORD

STUDY 1

CHEM 106401 Difenzoquat methyl sulfate §161-1

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41325403

Mangels, G. 1989a. Difenzoquat (AC 84,777): Hydrolysis. Laboratory Project No. E-89-3. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ.

DIRECT REVIEW TIME = 14

REVIEWED BY: L. Parsons TITLE: Staff Scientist

EDITED BY: K. Ferguson TITLE: Task Leader
 J. Harlin Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

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APPROVED BY: Richard J. Mahler
TITLE: Hydrologist
ORG: EFGWB/EFED/OPP
TEL: 703-305-7991

SIGNATURE: *Richard J. Mahler*

DEC 6 1993

CONCLUSIONS:

Degradation - Hydrolysis

1. This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information that shows pyrazole ring-labeled [3-¹⁴C]difenzoquat (1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate) did not hydrolyze in sterile aqueous buffered solutions (pH 5, 7, and 9) that were incubated in the dark at 25 °C for 28 days.
2. No additional information on the hydrolysis of difenzoquat is required at this time.

METHODOLOGY:

Pyrazole ring-labeled [3-¹⁴C]difenzoquat (CL 84,777; 1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate; radiochemical purity >98%, specific activity 13.48 uCi/mg, American Cyanamid) was added at 20 ppm to aqueous 0.05 M buffer solutions that had been adjusted to pH 5 (phthalate), 7 (phosphate), or 9 (borate). The treated buffered solutions were filter-sterilized (0.22 um); then aliquots (15 mL) of the solutions were dispensed into sterile 20-mL bottles; three bottles were prepared for each pH. The bottles were sealed and wrapped in foil, and the samples were placed in a constant temperature chamber at 25 C. Aliquots of the treated solution in each bottle were removed for analysis at 0, 1, 2, 3, and 4 weeks posttreatment using a sterile syringe.

At each sampling interval, aliquots of each buffered solution were analyzed for total radioactivity by LSC. Additional aliquots were analyzed using one-dimensional TLC on normal-phase silica gel plates developed with ethyl acetate:n-propanol:5% sodium perchlorate:formic acid (6:10:3:1, v:v:v:v) or ethyl acetate:n-propanol:5% sodium perchlorate:ammonium hydroxide (6:10:3:1, v:v:v:v), and on reverse-phase C-18 TLC plates developed with methanol:0.1 N sodium chloride in water:tetrahydrofuran (60:30:20, v:v:v). An unlabeled reference standard was cochromatographed with the samples and was located by UV detection; radioactive areas were located by autoradiography.

DATA SUMMARY:

Pyrazole ring-labeled [3-¹⁴C]difenzoquat (CL 84,777; 1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate; radiochemical purity >98%), at approximately 20 ppm, did not degrade in sterile aqueous buffered solutions (pH 5, 7, and 9) that were incubated in the dark at 25 ± 1 C for 28 days (Table 1). Using three different TLC systems, [¹⁴C]difenzoquat was the only compound detected in the treated solutions at 28 days posttreatment (Figure 1); quantitative data were not provided. During the study, material balances ranged from 98.2 to 101.1% of the applied with no discernable pattern of decline (Table 2).

REVIEWER'S COMMENTS:

1. The study author stated that the pH at the initiation and termination of the study did not vary more than 0.1 pH unit; the pH was reported to be 5.00, 6.97, and 8.91.
2. The sterility of the test solutions was determined at the initiation and termination of the experiment using "Nalgene Nutrient Pad Kits...with standard media". No contamination was detected.
3. The solubility of difenzoquat in distilled water and pH 4, 7, and 10 buffer solutions was reported to be 800,000 mg/L by the study author in the photodegradation in water study (Study 2, MRID 41325404). The

solubility of difenzoquat methyl sulfate in water was reported to be 765 g/L at 25 C in The Agrochemicals Handbook (3rd Edition, 1992).

AVENGE (DIFENZOQUAT)

Page is not included in this copy.

Pages 20 through 26 are not included.

The material not included contains the following type of information:

- Identity of product inert ingredients.
 - Identity of product impurities.
 - Description of the product manufacturing process.
 - Description of 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.
 - FIFRA registration data.
 - The document is a duplicate of page(s) .
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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 106401 Difenzoquat methyl sulfate §161-2

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41325404

Mangels, G. 1989b. Difenzoquat (AC 84,777): Photodegradation in water. Laboratory Project No. E-89-6. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ.

DIRECT REVIEW TIME = 13

REVIEWED BY: L. Parsons TITLE: Staff Scientist

EDITED BY: K. Ferguson TITLE: Task Leader
 J. Harlin Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

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TEL: 703-305-7991

SIGNATURE: *Richard J. Mahler*

DEC 6 1993

CONCLUSIONS:

Degradation - Photodegradation in Water

1. This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information that shows pyrazole ring-labeled [3-¹⁴C]difenzoquat (1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate) did not degrade in sterile aqueous buffered (pH 7) solutions that were continuously irradiated with a xenon arc lamp at 25 °C for 28 days.
2. No additional information on the photodegradation of difenzoquat in water is required at this time.

METHODOLOGY:

Pyrazole ring-labeled [$3\text{-}^{14}\text{C}$]difenzoquat (CL 84,777; 1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate; radiochemical purity >98%, specific activity 13.48 uCi/mg, American Cyanamid) was added at 40 ppm to an aqueous 0.05 M phosphate solution that had been buffered to pH 7. The treated buffered solution was filter-sterilized (0.22 μm), then aliquots (20 mL) of the treated solution were dispensed into four sterile quartz test tubes. Two tubes were wrapped in foil, and the samples were incubated in a constant temperature chamber at 25 C to serve as dark controls. The remaining two tubes of treated solution were placed in a "custom-made photolysis chamber" (not further described) and the samples were continuously irradiated using a xenon arc lamp (Atlas Electric Devices Company) for 28 days at 25 ± 1 C. The lamp was equipped with borosilicate filters to filter out wavelengths ≤ 300 nm; the emission spectra of the lamp was measured immediately prior to the study with a Li-Cor LI-1800 spectroradiometer and is presented in Figure 1. It was reported that, according to the manufacturer's specifications, the "output" of the lamp was "comparable to noon summer sunlight in Chicago, IL". Aliquots of the treated solution in each tube were removed for analysis at 1, 2, 3, 10, 14, 21, and 28 days posttreatment using a sterile syringe.

At each sampling interval, aliquots of each solution were analyzed for total radioactivity by LSC. Additional aliquots were analyzed using one-dimensional TLC on normal-phase silica gel plates developed with ethyl acetate:n-propanol:5% sodium perchlorate:formic acid (6:10:3:1, v:v:v:v) or ethyl acetate:n-propanol:5% sodium perchlorate:ammonium hydroxide (6:10:3:1, v:v:v:v), and on reverse-phase C-18 TLC plates developed with methanol:0.1 N sodium chloride in water:tetrahydrofuran (60:30:20, v:v:v). An unlabeled reference standard was cochromatographed with the samples and was located by UV detection; radioactive areas were located by autoradiography.

DATA SUMMARY:

Pyrazole ring-labeled [$3\text{-}^{14}\text{C}$]difenzoquat (CL 84,777; 1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate; radiochemical purity >98%), at 40 ppm, did not degrade (Table 1) in a sterile pH 7 buffered solution that was continuously irradiated for 28 days using a xenon arc lamp at 25 ± 1 C. The "output" of the lamp was reported to be "comparable to noon summer sunlight in Chicago, IL" (Figure 1). Using three different TLC systems, [^{14}C]difenzoquat was the only compound detected in the irradiated and dark control solutions at 28 days posttreatment (Figure 2); quantitative data were not provided. During the study, material balances ranged from 98.6 to 101.6% of the applied with no discernable pattern of decline (Table 2).

REVIEWER'S COMMENTS:

1. Although a graph of the measured emission spectrum of the xenon lamp was provided, the intensity of individual wavelengths emitted by the xenon lamp was not compared to those of natural sunlight. The study author reported that the manufacturer's specifications state that the "output" of the lamp was "comparable to noon summer sunlight in Chicago, IL".
2. The absorption spectrum of difenzoquat in the buffered solution was not provided.
3. The only description provided for the photolysis chamber was that it was "custom-made".
4. EFGWB suggests that the registrant provide more detailed information related to the above three mentioned subjects in future submitted studies.
5. The study author stated that the pH of the solution at the initiation and termination was 6.99 and 7.00, respectively.
6. The sterility of the test solutions was determined at the initiation and termination of the experiment using "Nalgene Nutrient Pad Kits...with standard media". No contamination was detected.
7. Difenzoquat did not hydrolyze in sterile aqueous buffered solutions (pH 5, 7, and 9) that were incubated in the dark at 25 C for 28 days (Study 1, MRID 41325403).
8. The solubility of difenzoquat in distilled water and pH 4, 7, and 10 buffer solutions was reported to be 800,000 mg/L by the study author in this study. The solubility of difenzoquat methyl sulfate in water was reported to be 765 g/L at 25 C in The Agrochemicals Handbook (3rd Edition, 1992).

AVENGE (DIFENZOQUAT)

Page _____ is not included in this copy.

Pages 30 through 37 are not included.

The material not included contains the following type of information:

- Identity of product inert ingredients.
 - Identity of product impurities.
 - Description of the product manufacturing process.
 - Description of 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.
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DATA EVALUATION RECORD

STUDY 3

CHEM 106401 Difenzoquat methyl sulfate §161-3

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41325405

Mangels, G. 1989c. Difenzoquat (AC 84,777): Photolysis on soil. Laboratory Project No. E-89-24. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ.

DIRECT REVIEW TIME = 18

REVIEWED BY: L. Parsons

TITLE: Staff Scientist

EDITED BY: K. Ferguson
 J. Harlin

TITLE: Task Leader
 Staff Scientist

APPROVED BY: W. Spangler

TITLE: Project Manager

ORG: Dynamac Corporation
 Rockville, MD

TEL: 301-417-9800

APPROVED BY: Richard J. Mahler

TITLE: Hydrologist

ORG: EFGWB/EFED/OPP

TEL: 703-305-7991

SIGNATURE: *Richard J. Mahler*

DEC 6 1993

CONCLUSIONS:

Degradation - Photodegradation on Soil

1. This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information that shows pyrazole ring-labeled [3-¹⁴C]difenzoquat (1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate) did not degrade on sandy loam soil that was continuously irradiated with a xenon arc lamp for 28 days at 25°C. In both the irradiated and dark control samples, difenzoquat comprised 98.0-99.8% of the applied at the termination of the experiment.
2. No additional information on the photodegradation of difenzoquat on soil is required at this time.

METHODOLOGY:

Sieved (1 mm) Sassafras sandy loam soil (62.8% sand, 25.6% silt, 11.6% clay, 1.0% organic matter, pH 6.9, CEC 5.38 meq/100 g) was weighed (5 g) into 20 glass Petri dishes and treated with 200 ug of pyrazole ring-labeled [3-¹⁴C]difenzoquat (CL 84,777; 1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate; radiochemical purity >98%, specific activity 13.48 uCi/mg, American Cyanamid) in acetonitrile. The soil in the dishes was air-dried for 1 hour, then one-half of the samples were placed on a tray and covered with aluminum foil for the dark controls. Both the covered and unshielded Petri dishes were placed in a "custom-made photolysis chamber" (not further described) and continuously irradiated using a xenon arc lamp (Atlas Electric Devices Company) for 28 days at 25 ± 1 C. The lamp was equipped with borosilicate filters to filter out wavelengths ≤300 nm; the emission spectrum of the lamp was measured immediately prior to the study "at the point where the samples were irradiated" with a Li-Cor LI-1800 spectroradiometer and is presented in Figure 1. It was reported that, according to the manufacturer's specifications, the "output" of the lamp was "comparable to noon summer sunlight in Chicago, IL". Duplicate irradiated and dark control samples were collected for analysis at 0, 7, 14, 21, and 28 days posttreatment.

At each sampling interval, the soil was extracted three times by shaking for 30 minutes with 2% HCl in methanol; the mixture was filtered under vacuum after each extraction, and aliquots of each extract were analyzed for total radioactivity using LSC. The extracts from each sample were combined and concentrated, and the pH of the concentrate was adjusted to 7 with 50% NaOH. A precipitate that formed when the extracts were neutralized was separated from the solution by centrifuging, then the precipitate was extracted three times with methanol. The methanol extracts were combined and concentrated under a stream of nitrogen. Aliquots of the methanol concentrates were analyzed using one-dimensional TLC on normal-phase silica gel plates developed with ethyl acetate:n-propanol:5% sodium perchlorate:formic acid (6:10:3:1, v:v:v:v) or ethyl acetate:n-propanol:5% sodium perchlorate:ammonium hydroxide (6:10:3:1, v:v:v:v), and on reverse-phase C-18 TLC plates developed with methanol:0.1 N sodium chloride in water:tetrahydrofuran (60:30:20, v:v:v). An unlabeled reference standard was cochromatographed with the samples and was located by UV detection; radioactive areas were located by autoradiography. Subsamples of the extracted soil were analyzed by LSC following combustion.

DATA SUMMARY:

Pyrazole ring-labeled [3-¹⁴C]difenzoquat (CL 84,777; 1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate; radiochemical purity 98%) did not photodegrade on sandy loam soil that was continuously irradiated for 28 days using a xenon arc lamp at 25 ± 1 C. The "output" of the lamp was reported to be "comparable to noon summer sunlight in Chicago, IL" (Figure 1). Based on data from three different TLC

systems, [¹⁴C]difenzoquat comprised 98.0-99.8% of the applied radioactivity in the irradiated and dark control solutions at 28 days posttreatment (Table 4). Two unidentified [¹⁴C]compounds were each ≤1.4% of the applied in the irradiated and dark control solutions; unidentified "1" was detected at all sampling intervals, and unidentified "2" was detected only at 3 and 4 weeks posttreatment. During the study, the material balances ranged from 99.07 to 106.23% of the applied with no discernable pattern of decline (Table 3).

REVIEWER'S COMMENTS:

1. Although a graph of the measured emission spectrum of the xenon lamp was provided, the intensity of individual wavelengths emitted by the xenon lamp was not compared to those of natural sunlight. The study author reported that the manufacturer's specifications state that the "output" of the lamp was "comparable to noon summer sunlight in Chicago, IL".
2. The absorption spectrum of difenzoquat in soil was not provided.
3. The only description provided for the photolysis chamber was that it was "custom-made".
4. EFGWB suggests that the registrant provide more detailed information related to the above three mentioned subjects in future submitted studies.
5. The dosing solution was analyzed by LSC prior to soil application.
6. Difenzoquat did not degrade in sterile aqueous buffered (pH 7) solutions that were continuously irradiated with a xenon arc lamp at 25 C for 28 days (Study 2, MRID 41325404).

AVENGE (DIFENZOQUAT)

Page _____ is not included in this copy.

Pages 41 through 45 are not included.

The material not included contains the following type of information:

- Identity of product inert ingredients.
 - Identity of product impurities.
 - Description of the product manufacturing process.
 - Description of 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.
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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 4

CHEM 106401 Difenzoquat methyl sulfate §162-1

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41903701

Mangels, G.D. 1988a. Difenzoquat (AC 84,777): Aerobic soil metabolism. Laboratory Report No. PD-M 25-58. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ.

DIRECT REVIEW TIME = 17

REVIEWED BY: L. Parsons TITLE: Staff Scientist

EDITED BY: K. Ferguson TITLE: Task Leader
 J. Harlin Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
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TEL: 301-417-9800

APPROVED BY: Richard J. Mahler
TITLE: Hydrologist
ORG: EFGWB/EFED/OPP
TEL: 703-305-7991

SIGNATURE: *Richard J. Mahler*

DEC 6 1993

CONCLUSIONS:

Metabolism - Aerobic Soil

1. This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information that shows pyrazole ring-labeled [3,5-¹⁴C]difenzoquat (1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate) did not degrade in aerobic sandy loam soil that was incubated for 1 year in the dark at 20 C and 75% of 0.33 bar moisture.
2. No additional information on the aerobic soil metabolism of difenzoquat is required at this time.

METHODOLOGY:

Sieved (2 mm) Sassafras sandy loam soil (62.8% sand, 25.6% silt, 11.6% clay, 1.0% organic matter, pH 6.9, CEC 5.38 meq/100 g) was weighed (50 g) into 2-ounce jars and treated at 1 ppm with pyrazole ring-labeled [3,5-¹⁴C]difenzoquat (AC 84,777; 1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate; radiochemical purity 99%, specific activity 13.48 uCi/mg, American Cyanamid) in water. The volume of water in the dosing solution was sufficient to moisten the soil to 75% of 0.33 bar. The treated soils were mixed on a mechanical shaker for 30 minutes, then the open jars were placed inside foil-wrapped, wide-mouth 1-gallon jars that were sealed and connected to a flow-through volatile trapping system. CO₂-free air was pumped through the jars and then sequentially through ethylene glycol and NaOH trapping solutions. The samples were maintained at 20 C. Duplicate samples were collected for analysis immediately posttreatment and at 1, 2, 4, 6, 8, 10, and 12 months. Trapping solutions were sampled weekly and replaced monthly.

At each sampling interval, the soil was extracted three times with 4% HCl in methanol; the 6-month samples were extracted four times. Aliquots of each extract were analyzed for total radioactivity by LSC, then the extracts from each sample were combined and neutralized (pH 7) with 1 N NaOH. Aliquots of the neutral solutions were analyzed by LSC; the remainder was concentrated to dryness. The resulting residues were extracted twice with acetone, and the acetone extracts were combined and analyzed by LSC. The acetone-extracted solids were dissolved in water, and the aqueous solutions were partitioned with methylene chloride; aliquots of both the water and methylene chloride solutions were analyzed by LSC. The acetone and methylene chloride extracts were combined and concentrated. Aliquots of the concentrates were analyzed using two-dimensional TLC on normal-phase silica gel plates developed with ethyl acetate:n-propanol:5% sodium perchlorate:formic acid (6:10:3:1, v:v:v:v) and ethyl acetate:n-propanol:5% sodium perchlorate:ammonium hydroxide (6:10:3:1, v:v:v:v), and on reverse-phase C-18 TLC plates developed with methanol:0.1 N sodium chloride in water:tetrahydrofuran (60:30:20, v:v:v). An unlabeled reference standard was cochromatographed with the samples and was located by UV detection; radioactive areas were located by autoradiography. Subsamples of the extracted soil were analyzed by LSC following combustion.

The trapping solutions were analyzed for total radioactivity by LSC.

DATA SUMMARY:

Pyrazole ring-labeled [3,5-¹⁴C]difenzoquat (AC 84,777; 1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate; radiochemical purity 99%), at 1 ppm, did not degrade in aerobic sandy loam soil that was incubated for 1 year in the dark at 20 C and 75% of 0.33 bar moisture. During the study, extractable [¹⁴C]residues totaled 96.9-98.9% of the applied; [¹⁴C]difenzoquat was the only compound detected in the soil

extracts at 12 months posttreatment (Table 2, and Figures 2 and 3; quantitative data were not provided). Unextracted [^{14}C]residues in the soil were 1.1-3.1% of the applied at all sampling intervals, and [^{14}C]volatiles (organic and $^{14}\text{CO}_2$) totaled <1% at 12 months posttreatment (Table 2). During the study, the material balances ranged from 97.10 to 107.68% of the applied with no discernable pattern of decline.

REVIEWER'S COMMENTS:

1. Only autoradiographs of the one- and two-dimensional TLC analyses of the 12-month samples were provided to support the conclusion that difenzoquat was stable during the entire incubation period. No quantitative data were provided for difenzoquat.
2. It was not specified whether the soil was remoistened during the experiment.
3. The study author reported the incubation temperature as 20 C, which is 5 degrees lower than the recommended incubation temperature of 25 C.

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AVENGE (DIFENZOQUAT)

Page _____ is not included in this copy.

Pages 50 through 54 are not included.

The material not included contains the following type of information:

- Identity of product inert ingredients.
 - Identity of product impurities.
 - Description of the product manufacturing process.
 - Description of 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.
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DATA EVALUATION RECORD

STUDY 5

CHEM 106401 Difenzoquat methyl sulfate §162-2

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41903702

Mangels, G.D. 1988b. Difenzoquat (AC 84,777): Anaerobic soil metabolism. Laboratory Report No. PD-M 25-50. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ.

DIRECT REVIEW TIME = 17

REVIEWED BY: L. Parsons TITLE: Staff Scientist

EDITED BY: K. Ferguson TITLE: Task Leader
 J. Harlin Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
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TEL: 301-417-9800

APPROVED BY: Richard J. Mahler

TITLE: Hydrologist

ORG: EFGWB/EFED/OPP

TEL: 703-305-7991

SIGNATURE:

Richard J. Mahler

DEC 6 1993

CONCLUSIONS:

Metabolism - Anaerobic Soil

1. This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information that shows pyrazole ring-labeled [3,5-¹⁴C]difenzoquat (1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate) did not degrade in anaerobic (flooded plus oxygen-free atmosphere) sandy loam soil that was incubated in the dark at 20 °C for 2 months.
2. No additional information on the anaerobic soil metabolism of difenzoquat is required at this time.

METHODOLOGY:

Sieved (2 mm) Sassafras sandy loam soil (63% sand, 26% silt, 11% clay, 1.0% organic matter, pH 6.9, CEC 5.9 meq/100 g) was weighed (50 g) into 2-ounce jars and treated at 1.66 ppm with pyrazole ring-labeled [3,5-¹⁴C]difenzoquat (AC 84,777; 1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate; radiochemical purity 99%, specific activity 13.48 uCi/mg, American Cyanamid) in water. Additional water was added to bring the moisture level of the soil to 75% of 0.33 bar, and the treated soils were mixed on a mechanical shaker for 30 minutes. The open sample jars were then placed inside foil-wrapped, wide-mouth 1-gallon jars that were sealed and connected to a flow-through volatile trapping system. CO₂-free air was pumped through the jars and then sequentially through ethylene glycol and NaOH trapping solutions. The samples were maintained at 20 C. The trapping solutions were sampled weekly and analyzed using LSC.

After 1 month of aerobic incubation, the treated soils were flooded with 30 mL of water. The samples were mixed with a spatula to incorporate the water, then were placed on an N-EVAP evaporator. Nitrogen gas was bubbled through the system for 30 minutes; the samples were then placed in pairs into BBL flasks. Dry ice was added to each chamber "to displace any oxygen present"; and the flasks were sealed, wrapped in aluminum foil, and incubated in a darkened area at 20 C. Duplicate samples were collected for analysis immediately posttreatment, after 1 month of aerobic incubation, and after 1 and 2 months of anaerobic incubation (2 and 3 months posttreatment).

At each sampling interval, the water was removed (method not reported) and analyzed for total radioactivity by LSC. The soil was extracted three times with 4% HCl in methanol. Aliquots of each extract were analyzed for total radioactivity by LSC, then the extracts from each sample were combined and neutralized (pH 7) with 1 N NaOH. Aliquots of the neutral solutions were analyzed by LSC; the remainder was concentrated to dryness. The resulting residues were extracted twice with acetone, and the acetone extracts were combined and analyzed by LSC. The acetone-extracted solids were dissolved in water, and the aqueous solutions were partitioned with methylene chloride; aliquots of both the water and methylene chloride solutions were analyzed by LSC. The acetone and methylene chloride extracts were combined and concentrated. Aliquots of the concentrates were analyzed using two-dimensional TLC on normal-phase silica gel plates developed with ethyl acetate:n-propanol:5% sodium perchlorate:formic acid (6:10:3:1, v:v:v:v) and ethyl acetate:n-propanol:5% sodium perchlorate:ammonium hydroxide (6:10:3:1, v:v:v:v), and on reverse-phase C-18 TLC plates developed with methanol:0.1 N sodium chloride in water:tetrahydrofuran (60:30:20, v:v:v). An unlabeled reference standard was cochromatographed with the samples and was located by UV detection. [¹⁴C]Compounds on the plates were located by autoradiography, then scraped and analyzed by LSC. Subsamples of the extracted soil were analyzed by LSC following combustion.

DATA SUMMARY:

Pyrazole ring-labeled [3,5-¹⁴C]difenzoquat (AC 84,777; 1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate; radiochemical purity 99%), at 1 ppm, did not degrade in a sandy loam soil during 2 months of incubation under anaerobic conditions (flooded and oxygen-free, CO₂-enriched atmosphere) preceded by 1 month of incubation under aerobic conditions; the samples were maintained in the dark at 20 C for the entire 3-month study. During the study, extractable [¹⁴C]residues associated with the soil totaled 96.5-102.2% of the applied (summed from Table I); during anaerobic incubation, only 0.14-0.21% was associated with the floodwater. [¹⁴C]Difenzoquat was the only compound detected in the soil extracts (Figures 1-3; quantitative data were not provided). Unextracted [¹⁴C]residues in the soil were 1.18-2.48% of the applied at all sampling intervals (Table I). [¹⁴C]Volatiles (organic and ¹⁴CO₂) were not detected during the aerobic portion of the experiment, and were not measured during the anaerobic portion of the experiment. During the study, the material balances ranged from 99.06 to 104.23% of the applied with no discernable pattern of decline (Table I).

REVIEWER'S COMMENTS:

1. Only autoradiographs of the one- and two-dimensional TLC analyses of the 1-, 2-, and 3-month samples were provided to support the conclusion that difenzoquat was stable during the incubation period. No quantitative data were provided for difenzoquat.
2. The study author reported the incubation temperature as 20 C, which is 5 degrees lower than the recommended incubation temperature of 25 C.
3. The study author assumed that anaerobic conditions were established by flooding the soil, bubbling nitrogen through the samples, and adding dry ice to the sealed anaerobic chambers. No measurements were taken to confirm that these procedures were adequate.

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS
(INCLUDING PERTINENT TABLES AND FIGURES)

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DATA EVALUATION RECORD

STUDY 6

CHEM 106401 Difenzoquat methyl sulfate §163-1

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41703401

Mangels, G. 1987. Difenzoquat (AC 84,777): Adsorption/desorption on soil. Laboratory Report No. PD-M 24-1. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ.

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DEC 6 1993

CONCLUSIONS:

Mobility - Leaching and Adsorption/Desorption

1. This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information that shows difenzoquat (1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate) was immobile in sandy loam, sandy clay loam, silt loam, and clay loam soils.
2. Based on batch equilibrium experiments, Freundlich K_{ads} values of 124-685 in calcium chloride solutions and 181-2680 in water were determined. The study author suggested that the difference between the calcium chloride and water solutions may result from the competitive interaction of the calcium chloride and difenzoquat

methyl sulfate for binding sites due to the charged nature of difenzoquat methyl sulfate.

3. No additional information is needed on the mobility (batch equilibrium) of unaged difenzoquat at this time. Since difenzoquat did not degrade during 1 year of incubation in aerobic sandy loam soil, or during 2 months of incubation in anaerobic (flooded plus oxygen-free atmosphere) sandy loam soil, traditional mobility experiments using aged (30 days) difenzoquat residues would provide no additional information on the leaching potential of the compound and therefore are not required.

METHODOLOGY:

Sassafras sandy loam, Sharkey sandy clay loam, Tippeecanoe silt loam, and Beardon clay loam soils were air-dried and sieved (2 mm) prior to use (Table I). Based on preliminary batch equilibrium experiments, an equilibration time of 3 days was selected for use with all four soils in the definitive experiment.

For the definitive experiment, three subsamples of each soil type were weighed (5 g) into glass centrifuge tubes. Some samples were mixed with aliquots (25 mL) of 0.01 N CaCl₂ solutions containing pyrazole ring-labeled [3-¹⁴C]difenzoquat (CL 84,777; 1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate; radiochemical purity 98%, specific activity 13.4 uCi/mg, American Cyanamid) at 0.0968, 0.300, 0.605, or 1.07 ug/mL; other samples were mixed with aliquots of water containing [¹⁴C]difenzoquat at 0.105, 0.320, 0.645, or 1.08 ppm. There were three replicates for each treatment combination. The tubes were sealed, and the soil:solution slurries were shaken continuously on a shaker for 3 days at 23 C. Following equilibration, the samples were centrifuged and the supernatants decanted.

To measure desorption, pesticide-free 0.01 N CaCl₂ solution (25 mL) or water (25 mL) was added to the soil from the adsorption portion of the experiment. The soil:solution slurries were shaken for 3 days, then centrifuged, and the supernatants were decanted and analyzed by LSC as described. The procedure was repeated. Following the second desorption, the soil was extracted three times with 2% HCl in methanol.

Aliquots of the adsorption and desorption supernatants and of the acidic methanol extracts were analyzed for total radioactivity using LSC. The methanol-extracted soils were air-dried, pulverized, and analyzed for total radioactivity using LSC following combustion.

DATA SUMMARY:

Based on batch equilibrium experiments, [¹⁴C]difenzoquat was determined to be immobile in sandy loam, sandy clay loam, silt loam, and clay loam soils equilibrated with treated 0.01 N calcium chloride solutions. The soil:CaCl₂ slurries (5 g:25 mL) contained 0.0968, 0.300, 0.605, and 1.07 ug/mL of pyrazole ring-labeled [3-¹⁴C]difenzoquat (CL 84,777; 1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate; radiochemical purity 98%), and were equilibrated for 3 days at 23 C. Freundlich K_{ads} values were 124 for the sandy loam soil, 323 for the sandy clay loam soil, 471 for the silt loam soil, and 685 for the clay loam soil (Table II). Values for 1/n ranged from 0.960 to 1.236 for the adsorption phase. Following the second desorption step, average K_{des} values were 232 for the sandy loam soil, 198 for the sandy clay loam soil, 805 for the silt loam soil, and 4141 for the clay loam soil (Tables III, V, VII, and IX).

In soil:water slurries containing [¹⁴C]difenzoquat at 0.105, 0.320, 0.645, or 1.08 ug/mL, [¹⁴C]difenzoquat appeared to be immobile, with Freundlich K_{ads} values of 181 for the sandy loam soil, 636 for the sandy clay loam soil, 1093 for the silt loam soil, and 2680 for the clay loam soil (Tables II). Values for 1/n ranged from 0.983 to 1.257 for the adsorption phase. Following the second desorption step, average K_{des} values were 503 for the sandy loam soil, 893 for the sandy clay loam soil, 1106 for the silt loam soil, and 2353 for the clay loam soil (Tables IV, VI, VIII, and X). The study author suggested the observed differences in adsorption of difenzoquat between the calcium chloride and water slurries were due to the competitive interaction of the calcium chloride and difenzoquat methyl sulfate for binding sites due to the charged nature of difenzoquat methyl sulfate.

REVIEWER'S COMMENTS:

1. Material balances were not provided. The reviewer calculated material balances for all samples to be 90.6-100.3% of the applied, using the data provided in Tables III, V, VII, and IX.
2. Difenzoquat has been shown to be stable to hydrolysis, photodegradation, and aerobic and anaerobic microbial degradation. Difenzoquat did not degrade in sterile aqueous buffered solutions (pH 5, 7, and 9) that were incubated in the dark at 25 C for 28 days (Study 1, MRID 41325403), or in a sterile aqueous buffered (pH 7) solution or on soil that was continuously irradiated with a xenon arc lamp at 25 C for 28 days (Study 2, MRID 41325404; and Study 3, MRID 41325405). Difenzoquat did not degrade during 1 year of incubation in aerobic sandy loam soil, or during 2 months of incubation in anaerobic (flooded plus oxygen-free atmosphere) sandy loam soil (Study 4, MRID 41903701; and Study 5, MRID 41903702).
3. The solubility of difenzoquat in distilled water and pH 4, 7, and 10 buffer solutions was reported to be 800,000 mg/L by the study author

in the photodegradation in water study (Study 2, MRID 41325404). The solubility of difenzoquat methyl sulfate in water was reported to be 765 g/L at 25 C in The Agrochemicals Handbook (3rd Edition, 1992).

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3. This study is scientifically sound. Based on the information provided by this study, soil samples containing difenzoquat may be stored frozen for up to 24 months prior to analysis.
4. If samples are stored frozen for longer than 24 months prior to analysis, storage stability information for longer periods may be required.

METHODOLOGY:

Sandy loam soil (uncharacterized) was weighed (25 g) into 32 half-pint containers. Sixteen samples were fortified at 0.40 ppm with difenzoquat (CL 84,777; 1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate; purity 99.6%) dissolved in acetone; the remaining samples remained untreated to serve as controls. The soils were stored frozen at approximately -10 C. Duplicate soil samples were removed from the freezer for analysis after 0, 3, 6, 12, 18, and 24 months of storage. A portion of the control soils were fortified with difenzoquat prior to analysis to determine recovery efficiencies (no additional information provided).

The samples were analyzed using Method M-471, which was described in a previously-reviewed terrestrial field dissipation experiment (Study 4, MRID 00045637; Difenzoquat Methyl Sulfate Final Report dated July 11, 1988). The soil samples were air-dried and mixed with 75 g of sea sand. Glass wool was placed at the bottom of a chromatographic column, and the soil-sand mixture was poured into the column and topped with additional glass wool. The column was leached with two 250-mL volumes of 4% hydrochloric acid in methanol. The eluate resulting from the second leaching was transferred to a round-bottom flask and concentrated by flash evaporation. The concentrate was transferred to a separatory funnel, the round-bottom flask was rinsed with distilled water, and the rinsate was added to the funnel. The acidic methanol extract was partitioned with toluene twice; after each partitioning, the toluene fraction was discarded. The resulting aqueous fraction was partitioned twice with chloroform. The chloroform fractions were concentrated, then eluted through an alumina "cleanup" column using methanol:chloroform (1:99 and 1:9) as the eluting solvent. The resulting sample was analyzed for difenzoquat methyl sulfate using GC with nitrogen-phosphorus detection. The residue levels were not corrected for percent concurrent recoveries, which averaged $106 \pm 26\%$. The method detection limit was 0.05 ppm.

DATA SUMMARY:

Difenzoquat was stable in sandy loam soil that was treated with difenzoquat methyl sulfate (CL 84,777; 1,2-dimethyl-3,5-diphenyl pyrazolium methyl sulfate; purity 99.6%) at 0.40 ppm and stored frozen (-10 C) for 24 months. At 24 months posttreatment, difenzoquat comprised 103-116% of the applied radioactivity (Table

I). During the study, recovery of difenzoquat ranged from 100 to 136% of the applied.

REVIEWER'S COMMENTS:

1. Preparation of the soil prior to fortification (such as sieving or drying) was not reported. The soil was incompletely characterized; the percentages of sand, silt, clay, and organic matter, as well as the pH and CEC, were not reported.
2. The study author stated that the soil samples were analyzed according to American Cyanamid Method M-471 and that methylene chloride was used in place of chloroform (Gross, MRID 42327501). However, methylene chloride was used in the original method; therefore, it was assumed that the author meant that chloroform was used in place of methylene chloride. Other modifications to the original analytical method were the use of toluene in place of benzene and updated gas chromatographic conditions employing GC with nitrogen-phosphorus detection instead of GC with alkali salt-tip flame ionization detection.

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REFERENCES

The following studies were reviewed:

Chudwudebe, A. 1991. Difenzoquat methyl sulfate (CL 84,777): Freezer stability of residues of CL 84,777 in soil at the 12-month interval. Laboratory Report No. C-3528. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41903703)

Gross, J. 1992. Difenzoquat methyl sulfate (CL 84,777): Freezer stability of residues of CL 84,777 in soil after twenty-four (24) months. Laboratory Report No. C-3781. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (42327501)

Mangels, G. 1989a. Difenzoquat (AC 84,777): Hydrolysis. Laboratory Project No. E-89-3. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41325403)

Mangels, G. 1989b. Difenzoquat (AC 84,777): Photodegradation in water. Laboratory Project No. E-89-6. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41325404)

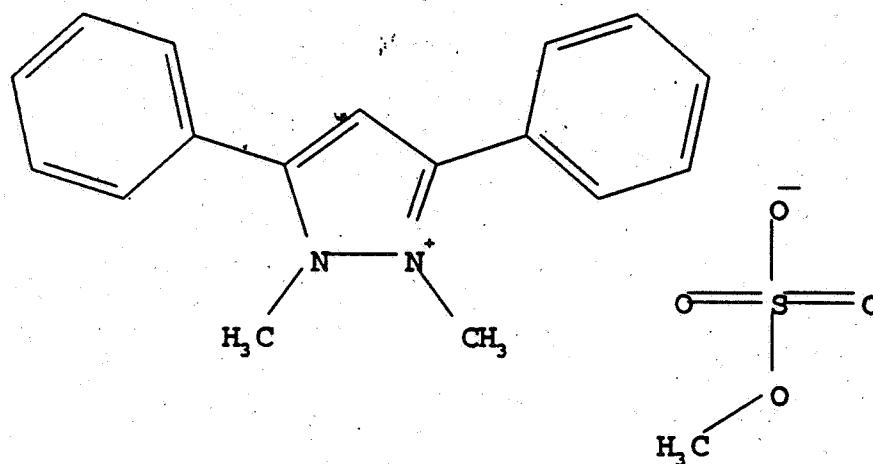
Mangels, G. 1989c. Difenzoquat (AC 84,777): Photolysis on soil. Laboratory Project No. E-89-24. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41325405)

Mangels, G.D. 1988a. Difenzoquat (AC 84,777): Aerobic soil metabolism. Laboratory Report No. PD-M 25-58. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41903701)

Mangels, G.D. 1988b. Difenzoquat (AC 84,777): Anaerobic soil metabolism. Laboratory Report No. PD-M 25-50. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41903702)

Mangels, G. 1987. Difenzoquat (AC 84,777): Adsorption/desorption on soil. Laboratory Report No. PD-M 24-1. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41703401)

APPENDIX
DIFENZOQUAT METHYL SULFATE



1,2-Dimethyl-3,5-diphenyl pyrazolium methyl sulfate
(Difenzoquat methyl sulfate)