DYNAMAC
CORPORATION

METSULFURON METHYL

TASK 1: REVIEW AND EVALUATION OF INDIVIDUAL STUDIES

TASK 2: ENVIRONMENTAL FATE AND EXPOSURE ASSESSMENT

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METSULFURON METHYL

Table of Contents

		Page
Scientific Studies		
1.	Aqueous photolysis of ^{14}C-DPX-T6376.	2
2.	Photodegradation of ^{14}C-phenyl-DPX-T6376.	5
3.	Anaerobic aquatic metabolism of [^{14}C-phenyl]-metsulfuron methyl.	7
4.	Soil column leaching studies with [^{14}C]-DPX-T6376.	14
5.	Adsorption of ^{14}C-DPX-T6376 on soil.	17
6.	Field dissipation study DPX-T6376 in Delaware, North Carolina, Florida and Mississippi.	20
7.	Crop rotation study with ^{14}C-DPX-T6376 in the greenhouse.	27
8.	Crop rotation study with metsulfuron methyl in the field.	30
Review information on studies included in the submission by reference.		33
Executive Summary		34
Recommendations		35
References		36
Appendix		38

This report is a scientific evaluation of environmental fate data submitted under Accession No. 072767. In addition to the eight studies reviewed herein, six studies reviewed previously are included by reference. The contribution of these studies to fulfillment of EPA Requirements for the Registration of Pesticides is considered under Recommendations. References for EAB reviews of these data are provided under Review Information On Studies Included In The Submission by Reference.

Diagrams of chemical structures included in this report have been redrawn by the reviewer. Other figures are photocopies of submitted materials. Tables have been retyped and in many instances reformated. Data not directly reported by the registrant (i.e., data calculated by the reviewer) are indicated as such either in tables or in the text.

STUDY 1

Friedman, P. 1984. Aqueous photolysis of ^{14}C-DPX-T6376. E.I. du Pont de Nemours and Company. Wilmington, DE. Document No. AMR-102-82. Acc. No. 072767. Reference G-2.

Procedure

Phenyl ring-labeled [^{14}C]metsulfuron methyl (specific activity 8.62 µCi/mg, 98% radiochemically pure) in acetone was added to distilled water (pH 5.6), standard reference water (pH 7.3 referenced but not described), clarified (centrifugation) Brandywine river water (pH 6.4), and uncentrifuged Brandywine river water. These test solutions (concentration 5 ppm) were placed in thermostatted (25°C) beakers with quartz lids. Two beakers were modified to allow head space vapor to pass over sodium hydroxide solution to trap evolved CO_2. Samples were continuously irradiated with an average UV light intensity (300-400 nm, peaking at 365 nm) of 1200 microwatts/cm^2 supplied by two sets of lamps. This was reportedly equal to ~half the solar light intensity on a clear, sunny June day in Wilmington, DE. Aluminum foil excluded light from a control (distilled water) solution.

Test-solutions were sampled at intervals of 0 and 6 hours, and 1, 2, 4, 7, and 14 days. The sodium hydroxide traps were sampled and replaced at the same intervals.

To facilitate degradate identification, a 20-ppm distilled-water solution of metsulfuron methyl (^{14}C properties unspecified) was irradiated for 14 days (under the above conditions) before analysis.

Methodology

Radioactivity in the gas traps was quantified by LSC. An aliquot from each trap was then mixed with saturated $BaCl_2$ followed by 2 M K_2CO_3. After centrifugation the supernatant was assayed for ^{14}C activity (LSC).

^{14}C activity in the test solutions was determined by LSC. Each sample was then acidified, (pH 4 to 5 with HNO_3), and extracted three times with methylene chloride. The combined, dried (Na_2SO_4) extracts and the aqueous phase were concentrated and examined by HPLC. ^{14}C activity in the extracts, the aqueous phase and the concentrates was quantified by LSC.

HPLC of the methylene chloride concentrates utilized a Zorbax SIL column (at 30°C) and a methylene chloride:acetic acid:water (1500:25:28) mobile phase. Reverse-phase HPLC of the aqueous concentrates (at 25°C) utilized an acidified (pH 2.2) acetonitrile:water (12:86) mobile phase. In both systems, eluant was monitored using a ^{14}C radioactivity detector. Fractions were collected and assayed for ^{14}C activity (LSC). One degradate in the aqueous concentrate was not retained by the reverse-phase system. The aqueous concentrate from the 20-ppm test solution was separated by HPLC using a Zorbax PSM-60 column (at 40°C) and a methanol:water:acetic acid (500:500:1) mobile phase. The eluate fraction containing the previously unretained unknown was characterized by chemical ionization mass spectrometry.

Results

Total ^{14}C recovery in the test solutions is summarized in Table 1. Recovery was high (91-108%) in the dark control over the full test period. For irradiated samples, non-volatile ^{14}C recovery was also high during the first 2 days. Recovery declined on further exposure. The registrant states that <1% of the ^{14}C applied to the dark control was detected in the gas trap. Over 40% of activity was lost as volatiles from irradiated clear solutions, and ~19% from the unclarified river water within 14 days. Characterization of gas trap contents indicated that the ^{14}C was evolved as $^{14}CO_2$.

The distribution of ^{14}C recovered from the test solutions is shown in Table 2. The proportion of ^{14}C recovered as parent declined with time in all solutions. Degradation was more rapid in irradiated than in control distilled water. Degradation rates also varied between irradiated samples. The major degradate in the control (distilled water) solution was methyl 2-(aminosulfonyl)-benzoate. This compound, and saccharin and 2-(aminosulfonyl)-benzoic acid were found in exposed distilled water.

Saccharin and 2-(aminosulfonyl)benzoic acid were reported in standard reference water and river water. Methyl 2-(aminosulfonyl)benzoate was reported in river water at the 7-day exposure interval only. The unknown polar compound was described as a "principal" degradate in reference and river water and was also, apparently, formed in irradiated distilled water. However, quantitative information was not provided.

Mass spectral analysis of the 20-ppm (distilled water) test solution indicated to the registrant that the unknown was probably an unsaturated aliphatic carboxylic acid.

Table 1. Recovery of non-volatile ^{14}C activity from phenyl-labeled [^{14}C]-metsulfuron methyl during photolysis study.[a]

Exposure interval (days)	Aqueous solutions				
	Dark control	Distilled	Standard reference	Brandywine river	River with sediment
0	100	100	100	100	100
0.25	100	108	105	106	104
1	106	101	105	99	96
2	100	92	102	101	104
4	91	74	96	89	93
7	102	60	78	71	95
14	108	58	56	58	81

[a] As a percent of ^{14}C applied.

Table 2. Distribution of ^{14}C from phenyl-labeled [^{14}C]metsulfuron methyl during photolysis study.

Sample	Exposure interval (days)	Percent of ^{14}C applied				
		Metsulfuron methyl	Methyl 2-(aminosulfonyl)-benzoate	Saccharin	2-(aminosulfonyl)-benzoic acid	Methyl 2-[N-(aminocarbonyl aminosulfonyl]benzoate
Control (distilled water)	0	99	1	<1	<1	<1
	0.25	99	1	<1	<1	<1
	1	98	1	<1	<1	<1
	2	95	3	<1	<1	1
	4	91	6	1	<1	1
	7	66	31	1	<1	2
	14	54	58	1	1	1
Distilled water	0	100	0	<1	<1	<1
	0.25	93	3	<1	<1	<1
	1	57	7	<1	<1	<1
	2	30	11	2	1	1
	4	8	13	9	2	<1
	7	0	5	21	4	<1
	14	0	9	23	3	<1
Standard reference water	0	100	<1	<1	<1	<1
	0.25	97	<1	<1	<1	<1
	1	83	<1	1	<1	<1
	2	85	<1	4	2	4
	4	50	<1	6	4	<1
	7	20	<1	8	9	<1
	14	<1	<1	7	7	<1
River water (clarified)	0	100	<1	<1	<1	<1
	0.25	98	<1	<1	<1	<1
	1	98	<1	3	<1	<1
	2	90	<1	1	2	<1
	4	69	<1	8	3	<1
	7	52	10	8	8	3
	14	23	<1	11	6	<1
River water (including sediment)	0	100	<1	<1	<1	<1
	0.25	99	<1	<1	<1	<1
	1	94	<1	1	<1	<1
	2	94	<1	2	<1	<1
	4	76	<1	5	1	2
	7	61	4	4	9	<1
	14	26	<1	8	14	2

Conclusions:

Metsulfuron methyl in distilled water (pH 5.6) exposed to artificial light degraded with a half-life of <2 days (reviewer calculated, assuming first order kinetics, $r^2 = 0.94$). A dark control [also in distilled water, (pH 5.6)] degraded with a half-life of ~14.5 days (reviewer calculated, assuming first order kinetics, $r^2 = 0.82$).

The residue in the dark control was dominantly methyl 2-(aminosulfonyl)-benzoate. In the exposed sample, metsulfuron methyl decomposed to CO_2, methyl 2-(aminosulfonyl)-benzoate, saccharin and 2-(aminosulfonyl)-benzoic acid. A polar compound (tentatively identified as an aliphatic unsaturated carboxylic acid) was also formed but quantitative information was not provided.

These results cannot fulfill EPA data requirements on photodegradation in water because metsulfuron methyl is not hydrolytically stable at the pH of the test solution. In a previous study (Friedman, 1982, Acc. No. 071434) metsulfuron methyl was hydrolysed at pH 5 (half-life ~3 weeks at 25°C) but was stable at pH 7 and 9. In addition the distilled water was unbuffered and it is not clear that the experiment was carried out under sterile conditions. Sample HPLC chromatograms and mass spectra were not provided for evaluation.

Dark controls were not provided for standard reference water or for river water. Information for these systems is therefore inadequate to characterize photodegradation of metsulfuron methyl.

This study provides no information on the fate or photolytic stability of the triazine moiety.

STUDY 2

Friedman, P. 1984. Photodegradation of ^{14}C-phenyl-DPX-T6376 on soil. E.I. du Pont de Nemours and Co., Inc. Wilmington, DE. Document No. AMR-77-82. Acc. No. 072767. Reference G-3.

Procedure

Samples of sieved (5.6 mm) Keyport silt loam soil (21% sand, 62% silt, 17% clay, 2.75% organic matter, pH 6.4, cation exchange capacity 8.2 meq/100 g) in jars were treated with an acetone:water (1:4) solution of phenyl-labeled [^{14}C]metsulfuron methyl (specific activity 8.6 µCi/mg, 98% radiochemically pure) at 5 ppm. Quartz lids were placed on the jars, which were irradiated continuously with an average UV light intensity (300 to 400 nm, peaking at 365 nm) of 1200 microwatts/cm^2 supplied by two sets of lamps. Selected samples were connected to gas traps (0.1 N NaOH) that were sampled and replaced weekly. Replicate samples were maintained in the dark as controls. Soil moisture levels were maintained at 70% of moisture holding capacity by adding water weekly as needed.

Samples were analyzed after 0, 7, 14, 21, and 30 days exposure. Controls were analyzed 0, 14, and 30 days posttreatment.

Methodology

Radioactivity in the gas traps was quantified by LSC and then characterized. An aliquot from each trap was mixed with saturated $BaCl_2$ then 2M K_2CO_3 and centrifuged. The supernatant was assayed for ^{14}C activity (LSC).

Soil samples were extracted three times with filtered methylene chloride: methanol:2M ammonium carbonate (3:4:1) by stirring for one hour. The extracted soil was air dried and ground prior to determination of residual ^{14}C (combustion and LSC). Filtered extracts were combined and concentrated by rotary evaporation. After acidification (pH 5) the concentrate was extracted three times with methylene chloride. Bulked extracts were dried ($MgSO_4$) and concentrated. The filtered aqueous phase was also concentrated. ^{14}C activity in all fractions and concentrates was quantified by LSC. The methylene chloride and aqueous concentrates were also examined by HPLC.

Aqueous concentrates were separated on a reverse phase-column using an acidified (pH 2) acetonitrile:water (12:88), mobile phase. The methylene chloride concentrates were purified using a Sep-Pak cartridge with a methylene chloride and methylene chloride:methanol (9:1) elution. The combined eluates were concentrated (1 ml), then analyzed by HPLC using a Zorbax SIL column and a methylene chloride:acetic acid:water (1500:25:28) mobile phase (detector not specified). Fractions containing the components of interest were collected and assayed for ^{14}C activity.

Results:

Results are reported in Table 3. Total ^{14}C recoveries ranged from 95-102% for controls. Sample ^{14}C recoveries declined somewhat with increased irradiation time (range 90 to 99%).

After thirty days irradiation 61% of applied ^{14}C remained as parent compared to 77% of that applied to dark controls.

Levels of non-extractable residues increased with time. In irradiated samples 2-(aminosulfonyl)-benzoic acid was a significant residue component, small amounts were found in controls. In both treatments, saccharin was a major degradate and methyl-2-(aminosulfonyl)-benzoate was a transient residue component.

Table 3. Distribution of ^{14}C in soil treated with phenyl-labeled [^{14}C]metsulfuron methyl.

		Percent of ^{14}C applied			
Treatment	Exposure interval (days)	Evolved as $^{14}CO_2$	Unextracted from soil	Metsulfuron methyl	Methyl 2-(aminosulfonyl)-benzoate
Exposed to light	0	<1	<1	98	<1
	7	<1	2	88	<1
	14	<1	5	81	1
	21	<1	7	72	<1
	30	<1	11	61	<1
Dark controls	0	<1	<1	98	<1
	14	<1	3	86	9
	30	<1	6	77	<1

Conclusions

Significant degradation (23% of applied) of phenyl-labeled [^{14}C]metsulfuron methyl occurred in a silt loam soil maintained in the dark. Exposure to artificial light increased the degradation to 39% during the same period. The same residue components were present in irradiated and control samples but their relative concentrations varied.

No definite conclusions can be drawn about the rate of photolysis of metsulfuron methyl on soil from this study. The temperature at which the experiment was run was not reported. No indication was given that exposed and control samples were maintained at the same constant temperature. In the absence of temperature control it is likely that the temperatures of exposed soil surfaces were substantially higher than those of dark controls.

No sample HPLC traces were provided for evaluation. The detection system was not specified.

This study provides no information on the fate of the triazine moiety.

STUDY 3

Friedman, P.L. 1984. Anaerobic aquatic metabolism of [^{14}C-phenyl]-metsulfuron methyl. E.I. du Pont de Nemours and Company, Inc. Wilmington, DE. Document No. AMR-134-83. Acc. No. 072767. Reference G-4.

Procedure

Pond waters and sediment were collected from Landenberg, PA; Pendleton, OR and Salina, KS. Characteristics of the water and sieved sediment (2 mm) are given in Table 4.

Metabolism Study: Aliquots of pond sediment (50 g) and the corresponding pond water (50 g) in glass bottles were treated at 1 ppm with an acetone solution (0.5 ml) of [^{14}C]metsulfuron methyl (uniformly phenyl-labeled, specific activity 0.025 µCi/µg, 98% radiochemically pure). The bottles were flushed with nitrogen, sealed and incubated in the dark at 25°C. Bottles were taken for analysis 0, 0.4, 1, 3, 6, 12, 24, 36, and 52 weeks posttreatment.

Sterile Samples: Autoclaved aliquots of pond sediment (50 g) and corresponding autoclaved pond water (100 g) with 1 g of sodium azide added were treated and incubated as described above (metabolism study). Bottles were taken for analysis 0, 1, 3, 12, 24 and 52 weeks posttreatment.

Cellulose Feeding Study: Aliquots of pond sediment (25 g) and the corresponding pond water (50 g) in glass bottles were treated at 1 ppm with an acetone solution (0.3 ml) of unlabeled metsulfuron methyl. ^{14}C-labeled cellulose (2.5 mg, specific activity 8 µCi/g) was then added. Sodium hydroxide (10 ml of 0.1 N) was placed in the side arm of each bottle to trap evolved CO_2. The bottles were then sealed. Incubation conditions were not specified. Control samples (containing no metsulfuron methyl) were also set up.

Methodology

Analysis of the ^{14}C metsulfuron methyl-treated samples was as follows. Samples were centrifuged. The aqueous phase was decanted, acidified (pH 4 with glacial acetic acid) then extracted three times with methylene chloride. The aqueous phase was concentrated (rotary evaporation) to 5 ml. Combined methylene chloride extracts were dried ($MgSO_4$), filtered, and concentrated (to 3 ml). After each procedural step ^{14}C activity was assayed by LSC. Concentrated aqueous and organic phases were also analyzed using TLC and HPLC, respectively. Sediment was extracted three times by stirring for 1 hour with methylene chloride:methanol:2M ammonium carbonate (3:4:1). The filtered extracts were combined, concentrated, acidified (pH 4 with glacial acetic acid), then extracted three times with methylene chloride. The combined methylene chloride extracts were dried ($MgSO_4$), filtered, and concentrated. The aqueous phase was filtered then concentrated. ^{14}C Activity in all fractions was quantified by LSC. The concentrated aqueous and organic phases were also examined by TLC and HPLC, respectively. Residual ^{14}C activity in extracted sediment was quantified by combustion and LSC.

TLC (of concentrated aqueous phases from sediment and water extractions) was performed on silica gel plates using a methylene chloride:methanol:ammonium hydroxide mobile phase (144:50:6). Radioactive areas were located by autoradiography and quantified by scraping and LSC.

HPLC was performed on methylene chloride extracts of sediment and water using a Zorbak SIL column and a methylene chloride:acetic acid:water (1500:25:2.8) mobile phase. Fractions containing the components of interest were collected. The radioactivity in each fraction was quantified by LSC.

^{14}C Levels in the gas traps (cellulose feeding study) were quantified by LSC. The activity in the traps was then characterized. Saturated $BaCl_2$ and 2M K_2CO_3 were added to aliquots from each trap. After centrifugation, ^{14}C activity in the supernatant was quantified by LSC.

Results

Metabolism Study and Sterile Samples: The total recovery of applied ^{14}C ranged from 85 to 116%. Distribution of ^{14}C (between water and sediment) was similar in the different pond samples. The results for the Landenberg samples are given in Table 5. The proportion of total activity recovered from the sediment increased with incubation time for both the sterile and non-sterile systems.

The residue components in the water and sediment at selected incubation intervals are given in Table 6. The proportion of applied ^{14}C that was not extracted from non-sterile sediment tended to increase with incubation time. The parent and three degradates were detected in water and sediment samples. At least four unidentified polar compounds were also detected. In all samples, levels of the parent decreased and levels of degradates increased with time. The amounts of the degradates varied between pond samples and also between non-sterile and sterile treatments for the same samples.

The degradation of metsulfuron methyl in the sediment and water systems is shown in Figure 1. The rate of degradation varied considerably between the samples from different sites. Degradation was slower in the sterile than in

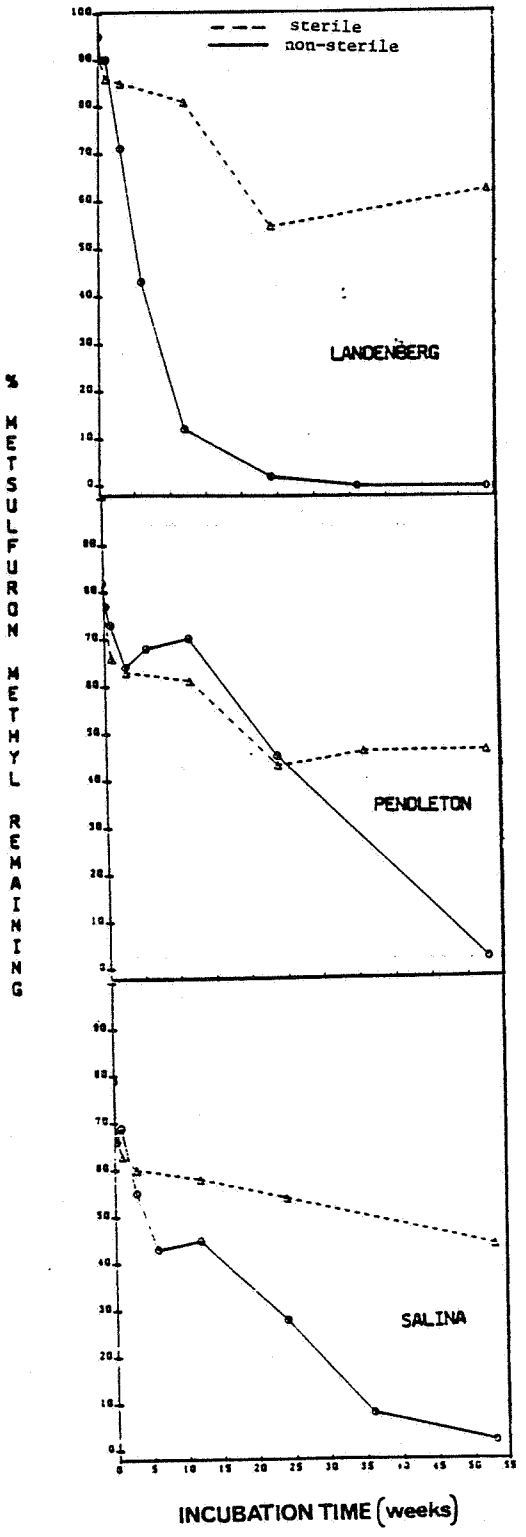


Figure 1. Anaerobic degradation of metsulfuron methyl in three pond systems (sediment plus water).

Table 4. Characteristics of pond sediments and waters.

Site	Textural class	Sand	Silt	Clay	OM[a]	pH Sediment	pH Water	CEC[b] (meq/100 g)
		%						
Landenberg, PA	Silt loam	25	74	1	3.7	5.6	5.8	11.0
Pendleton, OR	Silt loam	36	58	6	1.2	6.3	6.9	15.3
Salina, KS	Sandy loam	54.5	28.5	17	2.2	6.6	7.3	10.0

[a] Organic matter.

[b] Cation exchange capacity.

Table 5. Distribution of total radioactivity in Landenberg pond water and sediment.[a]

Incubation interval (weeks)	Non-sterile			Sterile		
	Water	Sediment	Total	Water	Sediment	Total
0	100	5	105	100	7	107
0.4	99	7	106	NS[b]	NS	NS
1	89	8	97	88	11	99
3	86	18	104	94	16	110
6	82	18	100	NS	NS	NS
12	85	20	105	90	18	108
24	85	17	102	92	18	110
36	68	25	93	NS	NS	NS
54[c]	67	28	95	87	21	108

[a] Expressed as percent of ^{14}C applied.

[b] Not sampled.

[c] Procedure reports last sampling at 52 weeks.

Table 6. Identification of ^{14}C residues in pond water and sediment treated with phenyl labeled [^{14}C]metsulfuron methyl.

Sample	Incubation interval (weeks)	^{14}C Residue distribution[a]					
		Metsulfuron methyl[b]	Methyl 2-(aminosulfonyl)-benzoate[b]	Saccharin[c]	2-(Aminosulfonyl)-benzoic acid[c]	Polar compounds[c]	Sediment bound[d]
Landenberg non-sterile	0	95	4	<1	<1	<1	<1
	3	71	4	2	2	9	2
	12	12	<1	5	16	47	5
	36	<1	<1	5	27	56	11
	54[e]	<1	<1	8	40	39	7
Landenberg sterile	0	91	4	1	1	<1	<1
	3	85	4	3	<1	<1	<1
	12	81	1	13	2	<1	<1
	24	55	8	26	6	<1	1
	54[e]	63	2	<1	1	26	2
Pendleton non-sterile	0	82	13	<1	<1	<1	<1
	3	64	4	6	<1	<1	<1
	12	70	5	14	<1	2	2
	24	45	<1	23	1	16	4
	53[e]	2	<1	12	3	21	9
Pendleton sterile	0	78	13	<1	<1	<1	<1
	3	63	2	16	1	<1	<1
	12	61	1	24	2	<1	<1
	24	43	1	33	9	<1	<1
	53	46	<1	6	13	18	1
Salina non-sterile	0	79	4	1	<1	<1	<1
	3	55	4	21	1	2	1
	12	45	<1	24	2	4	2
	36	8	<1	18	8	33	4
	53[e]	2	<1	22	17	37	3
Salina sterile	0	69	4	2	<1	<1	<1
	3	60	4	25	3	<1	<1
	12	58	1	27	7	<1	<1
	24	54	4	32	10	<1	<1
	53[e]	44	<1	5	5	36	1

[a] Expressed as a percent of ^{14}C applied.

[b] From HPLC analysis.

[c] From TLC analysis.

[d] By combustion.

[e] Procedure reports last sampling at 52 weeks.

Table 7. Cumulative $^{14}CO_2$ evolution from pond systems treated with ^{14}C-cellulose.[a]

Incubation interval (weeks)	Landenberg		Pendleton		Salina	
	Control	Treated[b]	Control	Treated[b]	Control	Treated[b]
0	<1	<1	<1	<1	<1	<1
1	43	22	73	58	10	8
3	60	48	76	61	28	29
6	69	57	87	76	40	43
12	78	66	87	77	43	46

[a] Expressed as a percent of ^{14}C applied.

[b] Treated with 1 ppm unlabeled metsulfuron methyl.

Table 8. Half-lives of phenyl-labeled [^{14}C]metsulfuron methyl in anaerobically incubated pond systems.[a]

Sample	r^2[b]	$t^{1/2}$[c]
Landenberg, non-sterile	0.87	7
Landenberg, sterile	0.53	103
Pendleton, non-sterile	0.79	37
Pendleton, sterile	0.69	89
Salina, non-sterile	0.96	11
Salina, sterile	0.95	115

[a] Reviewer calculated, assuming first-order kinetics.

[b] Correlation coefficient.

[c] Half-life, weeks.

the corresponding non-sterile samples for Landenberg and Salina throughout the incubation, and for Pendleton after 24 weeks incubation. For the first 24 weeks, degradation rates were similar in sterile and non-sterile Pendleton samples.

Cellulose Feeding Study: Results are given in Table 7. The evolution of $^{14}CO_2$ from [^{14}C]cellulose was similar from metsulfuron methyl treated and control samples.

Conclusions

The presence of metsulfuron methyl did not consistently reduce the rate of formation of $^{14}CO_2$ from [^{14}C]-cellulose in pond systems.

Results from non-sterile systems indicate that half-lives of metsulfuron methyl under anerobic aquatic conditions are variable (7-37 weeks, see Table 8). The parent was more persistent in the sterile systems. However, the accuracy of the half-life prediction (r^2) was inadequate in two systems for direct comparison.

Some rapid breakdown of metsulfuron methyl was observed in all systems. The distribution of residues changed with time, but some degradates (nonextractable residues, polar compounds, and 2-(aminosulfonyl)-benzoic acid) tended to accumulate in the sediment and water system. Only limited conclusions can be drawn about the nature of residue from this study because insufficient analytical details were provided and because the residue was inadequately characterized. No sample copies of TLC plates or HPLC traces were provided for examination. The registrant states that residue composition was similar in water and sediment fractions of the pond systems but does not present supporting data. At least four polar compounds were present. These were not characterized, reportedly because none was present in sufficient quantity for identification. However, up to 56% of recovered activity was present as unidentified polar material.

The decomposition products identified indicate that the triazine moiety is cleaved under anaerobic conditions. The fate of this group should be addressed.

Although the data were generally well presented, a number of discrepancies were either not addressed or were not adequately explained by the registrant. For example, total ^{14}C recovery values calculated from the residue distribution results (Table 6) do not correspond to those presented by the registrant. Some calculated values, for example those for the Salina pond, indicate low ^{14}C recoveries. The registrant's explanation of the aberrant decline pattern observed in the non-sterile Pendleton system is unpersuasive, and not adequately supported by the data. The maximum incubation time was reported variously as 52, 53, and 54 weeks throughout the report.

STUDY 4

Chrzanowski, R.L. 1984. Soil column leaching studies with [^{14}C]-DPX-T6376. E.I. du Pont de Nemours and Co., Inc., Wilmington, DE. Document No. AMR-82-82. Acc. No. 072767. Reference G-5.

Procedure

Direct Leaching Study: Four sieved (2 mm) soils, Fallsington sandy loam (DE), Flanagan silt loam (IL), Keyport silt loam (DE) and Myakka sand (FL), were packed into (2-in x 12-in) columns. Characteristics of the sieved soils are given in Table 9. The pre-wetted columns were treated with (934 µg, presumably in solution) metsulfuron methyl (phenyl-labeled, specific activity 8.62 µ Ci/mg, 99% radiochemically pure). The columns were then leached with water for 20 hours at a rate of 1 in/hr. Leachate was collected in (the equivalent of) 0.2-in increments. After leaching, the soils were removed from the columns in 2-in sections and air dried.

Aged Residue Leaching: Aliquots (100 g) of Fallsington and Flanagan soils were treated with [^{14}C]metsulfuron methyl (presumably of the same characteristics as that used in the Direct Leaching Study). Application rates were reported to be equivalent to 1.1 kg/ha. Samples were aged for 30 days in a greenhouse (temperature unspecified). Moisture was maintained at 75% of moisture holding capacity by adding water as necessary. The aged soils were transferred to the top of a (10-in x 2-in) column of the corresponding soil to produce a 12-in x 2-in column. Leaching and sample collection were then carried out as described above (Direct Leaching Study).

Methodology

Radioactivity in a subsample of each 0.2-in leachate increment was determined by LSC. ^{14}C activity in the leached soils was estimated by combustion.

Bulked column leachates from the aged-soil columns were concentrated by rotary evaporation. Concentrates were examined by TLC using cochromatography with authentic reference standards and a methylene chloride:methanol:ammonium hydroxide (144:53:3) mobile phase. Standards were visualized under UV light; ^{14}C activity was apparently quantified by radiochromatogram scanning.

The "breakthrough volume" (V_B, the column void volume) was determined using [^{36}Cl]. The method for this was referenced but was not described.

Results

Post-leaching ^{14}C distributions in each soil and ^{14}C quantity in leachate are summarized in Table 10. Total ^{14}C recoveries ranged from 87 to 108% of that applied.

Similar proportions (2.0 to 2.6%) of non-aged ^{14}C were retained by three of the soils; Keyport silt loam retained significantly more (~10%).

Column void volumes (V_B) are included in Table 9. Half of the non-aged ^{14}C applied was reportedly eluted at 1.05 V_B for Fallsington soil, 1.47 V_B for Flanagan soil, 1.45 V_B for Keyport soil and 1.14 V_B for Myakka sand.

Table 9. Soil characteristics.

Soil	Textural class	Sand	Silt	Clay	OM[a]	N[b]	pH	CEC[c] (meq/100 g)	V_B[d]
		%							
Fallsington	Sandy loam	56	29	15	1.4	0.085	5.6	4.8	6.5
Flanagan	Silty clay loam[e]	5	64	31	4.02	0.282	6.7	23.4	4.5
Keyport	Silt loam	21	62	17	2.75	0.097	6.4	8.2	6.9
Myakka	Sand	97	2	1	2.43	0.074	6.3	3.9	5.8

[a] Organic matter.

[b] Total nitrogen.

[c] Cation exchange capacity.

[d] Column void volume in inches of water.

[e] Reported as a silt loam.

Table 10. The distribution of ^{14}C residues in soil leaching columns and leachate.[a]

Depth (in)	Fallsington		Flanagan		Keyport	Myakka
	Non-aged	Aged	Non-aged	Aged	Non-aged	Non-aged
0-2	0.17	12	0.32	24	2.1	0.9
2-4	0.12	0.7	0.22	1.6	1.3	0.41
4-6	0.14	0.7	0.27	2.1	1.0	0.40
6-8	0.25	0.7	0.40	1.4	1.4	0.3
8-10	0.20	0.7	0.50	1.6	1.3	0.2
10-12	1.06	0.7	0.93	1.8	2.9	0.3
Total retained	2.0	16	2.6	33	10	2.5
Leachate	106	76	96	54	87	93
Total recovery	108	92	99	87	97	95

[a] Expressed as percent of applied ^{14}C.

Table 11. Identification of ^{14}C residues in eluate from soil columns treated with aged [^{14}C]metsulfuron methyl.[a]

Soil	^{14}C Residue Distribution[b]			
	Metsulfuron[c] methyl	Methyl-2(aminosulfonyl)-benzoate	Saccharin	Unidentified polar material
Fallsington	5	25	50	20
Flanagan	<0.1	5	85	10

[a] [^{14}C]metsulfuron methyl not characterized.

[b] Expressed as a percent of ^{14}C applied.

[c] See Appendix for structure of parent and all degradates.

In the same soils, aged residues were less mobile than unaged. In Fallsington ton and Flanagan soils, 16% and 33%, respectively, of aged ^{14}C were retained in the columns; the corresponding amounts for non-aged residues were 2.0% and 2.6%, respectively. Leachate components from the aged treatments are quantified in Table 11. There was some variability in component distribution between the two soils but very little parent was present in either case. Saccharin was the major residue component; methyl-2(aminosulfonyl)-benzoate and unidentified polar materials were also present.

Conclusions

The activity from phenyl-labeled [^{14}C]metsulfuron methyl was largely (>87%) eluted from 12-in columns of sandy loam, sand; silty clay loam, and silt loam soils by 20 in (50.8 cm) water. The application was equal to a total of 1029 ml (63 in^3) water (20 in x cross sectional area of column). After 30 days aerobic aging 76% and 54%, respectively, of the activity from phenyl-labeled [^{14}C]metsulfuron methyl were eluted from similar columns of the sandy loam and silty clay loam soils by the same treatment. Most activity in the eluate from the aged soils was present as saccharin along with methyl 2-(aminosulfonyl)-benzoate and unidentified polar materials.

This study suggests that freshly applied metsulfuron methyl is mobile in a range of soil types, and that aerobic aging reduces mobility. No information is provided on the mobility of the triazine moiety. Triazine herbicides have leached to groundwater and thus the fate of this group should be addressed. Furthermore, additional information is required before any definite conclusions can be made from the reported data. Column-packing techniques and pre-treatment conditions should be described. Columns were apparently pre-wetted but no details were provided. Inadequate details were provided of the application of non-aged metsulfuron methyl to columns; 934 µg were applied but it is not indicated if this was in solution, and if so, what solvent was used. No characterization (site of labeling, specific activity and radiochemical purity) of the [^{14}C]metsulfuron methyl used in the aged study was given; this should be provided. The aging procedure was inadequately described. Aged residues were not characterized prior to leaching and the aging temperature was not reported. The activity in the leached columns was not characterized. Insufficient information was provided on analytical methodology. No copies of TLC plates were provided. One autoradiogram was included but quantification of ^{14}C activity on the plates was not addressed in the text. K_d values were not reported and cannot be calculated from the data provided (because the weight of soil in the columns is not given).

A number of additional minor deficiencies are present. For instance the registrant states that unaged metsulfuron methyl is retained longer on soil columns with the higher percentages of organic matter. The total amount of ^{14}C retained on the columns (see Table 10) do not reflect this. The stated application rate (934 µg to 2-in diameter column for non-aged soil) is estimated by the reviewer to be equivalent to ~4.5 kg/ha not 1.1 kg/ha as stated in the study. Both treatments exceed the highest recommended treatment rate and are therefore satisfactory. (However, this matter should be clarified.) Finally, no reference list was provided although a number of citations were made in the text.

STUDY 5

Friedman, P.L. 1984. Adsorption of ^{14}C-DPX-T6376 on soil. E.I. du Pont de Nemours and Co., Inc., Wilmington, Delaware. Acc. No. 072767. Reference G-6.

Procedure

Four soils, a sandy loam (Fallsington), a silty clay loam (reported as a silt loam) (Flanagan), a silt loam (Keyport) and a sand (Cecil) were used in this study. Characteristics of the sieved (2 mm) soils are given in Table 12.

Batch Study: Aliquots (20 ml) of solutions of [^{14}C]metsulfuron methyl, [^{14}C] terbacil or [^{14}C]diuron (0.2, 0.5, 1.0, 2.0 or 6.0 ppm in 0.01 N aqueous calcium sulfate) were added to duplicate 5-g soil samples in polypropylene bottles, which were shaken (24 hours at 25°C) then centrifuged.

Structures of the test compounds are shown in Figure 2.

TLC Study: Subsamples (~140 g) of the soils were milled (400 microns), slurried with water, and applied to glass plates (20 cm x 20 cm) using a 450 micron TLC plate gauge. Duplicate plates of each soil were treated with duplicate spots of [^{14}C]metsulfuron methyl, [^{14}C]terbacil and [^{14}C]diuron and then developed (to 10 cm) in water.

Methodology

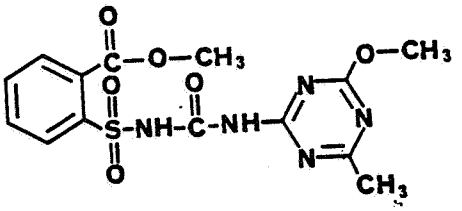
Batch Study: Initial test standard solutions and posttreatment supernatants were assayed for ^{14}C activity using LSC. The amount of each compound adsorbed by the different soils (C_s) was estimated as the difference in solution concentration before (C_1) and after (C_2) equilibration with soil. Values of C_s and C_2 were fitted graphically to the Freundlich isotherm equation. The Freundlich "K constant" was calculated at 1 ppm equilibrium solution concentration and used as the coefficient of adsorption per 5 g soil (K). Coefficients of adsorption per unit organic matter were calculated using K_{om} = K x 100/% organic matter.

TLC Study: Radioactive areas on the plates were located by autoradiography.

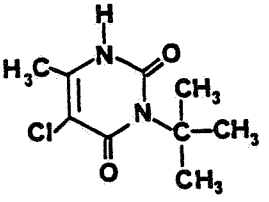
Results

Batch Study: Table 13 contains Freundlich K values (adsorption coefficients) on whole soil (K) and on organic matter content (K_{om}) bases, as well as slopes of the Freundlich plot (1/n). The adsorption coefficients indicate that sorption of metsulfuron methyl is related to organic matter content and soil textural class. Metsulfuron methyl was sorbed less extensively than terbacil by all soils except Cecil and was sorbed much less than diuron by all soils. The range of 1/n values (slope of Freundlich plots, reflecting the dependence of K on adsorbate concentration) was greater for metsulfuron methyl (0.57-1.14) than for the other two compounds. The variation was not obviously related to reported soil physical or chemical properties.

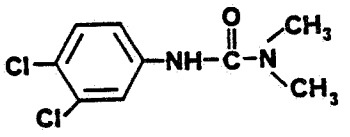
TLC Study: R_f values were of the order metsulfuron methyl>terbacil>>diuron for all soils tested (Table 14).



Metsulfuron methyl



Terbacil



Diuron

Figure 2. Structures of the test compounds.

Table 12. Characteristics of the test soils.

Soil	Textural class	Sand	Silt	Clay	OM[a]	pH	CEC[b] meq/100 g
		%					
Cecil	Sand	92	8	<1	0.3	6.1	4.9
Fallsington	Sandy loam	56	29	15	1.40	5.6	4.8
Keyport	Silt loam	21	62	17	2.75	6.4	8.2
Flanagan	Silty clay loam[c]	5	64	31	4.02	6.5	23.4

a Organic matter.

b Cation exchange capacity.

c Reported as silt loam.

Table 13. Adsorption of [^{14}C]metsulfuron methyl, [^{14}C]terbacil and [^{14}C]diuron on four soils.[a]

Soil	Metsulfuron methyl			Terbacil			Diuron		
	K[b]	K_{om}[c]	1/n[d]	K	K_{om}	1/n	K	K_{om}	1/n
Cecil sand	0.36	120	1.14	0.34	113	0.95	2.05	683	0.81
Fallsington	0.41	29	0.57	0.57	41	1.10	3.80	271	0.84
Keyport	0.84	31	0.85	1.30	47	0.92	8.50	309	0.78
Flanagan	1.40	35	0.97	2.15	53	0.91	14.7	366	0.87

a Parameters obtained using the Fruendlich equation.

b Freundlich K value.

c Coefficient of adsorption per unit organic matter.

d Slope of log-log plot.

Table 14. R_f values from soil TLC study.

Soil	R_f Values		
	Metsulfuron methyl	Terbacil	Diuron
Cecil sand	1.00	0.87	0.42
Fallsington	0.98	0.72	0.26
Keyport	0.90	0.62	0.20
Flanagan	0.64	0.40	0.14

Conclusions

This study provides information on the mobility of the parent compound only. Metsulfuron methyl was less extensively sorbed than terbacil and diuron by the test soils. Under the conditions of the TLC study, metsulfuron methyl was moderately to very mobile, and was more mobile than terbacil or diuron. Sorption and mobility appeared to depend on organic matter content and texture.

Insufficient information was provided to allow this study to fulfill registration requirements. The test substances were not characterized; purity, specific acitivity and site of labeling of all test compounds should be provided. Details of the LSC and autoradiography techniques were not given. Sample data plots were not included, so conformation to the adsorption model could not be evaluated.

STUDY 6

Anderson, J.J. and J. Harvey. 1984. Field dissipation study of DPX-T6376 in Delaware, North Carolina, Florida and Mississippi. E.I. du Pont de Nemours and Company, Inc., Wilmington, DE. Document No. AMR-117-83. Acc. No. 072767. Reference G-7.

Procedures

Field study: Between July and September 1981 columns of soils were isolated [by driving steel cylinders (10-cm i.d. x 38-cm length) into undisturbed ground] at four sites [Newark, DE; Stoneville, (also referred to as Scott in study) MS; Clayton, NC; Bradenton, FL] in the US. Soil characteristics are reported in Table 15. [^{14}C]Metsulfuron methyl (uniformly labeled in the phenyl ring, specific activity 8.62 µCi/mg, >99% radiochemically pure) in acetone solution was applied to the soil within the cylinders at a rate equivalent to 1 lb/acre. Treatment was followed by the application of 50 to 100 ml water. One cylinder per site was removed immediately; remaining cylinders (seven per site) were removed at intervals, up to 18 months posttreatment. On removal from the ground, cylinders were frozen (dry ice) immediately and were stored frozen (-20°C) until analysis.

Rainfall was measured at each experimental site, but soil and air temperatures were not reported.

Laboratory aerobic incubation: Fresh Keyport silt loam soil (200 g) was treated with (47 mg) of [^{14}C]metsulfuron methyl (specific activity 0.44 µCi/mg, presumably phenyl-labeled) and incubated at 25°C in a closed flask, that was flushed with oxygen weekly. After one month the soil was analyzed. The incubation was intended to generate sufficient quantities of the postulated major soil metabolite for mass spectral identification.

Methodology

Field Samples: Soil was extruded from each cylinder, divided into four (~8 cm) sections, air dried, then ground. Sub-samples were assayed for total ^{14}C activity by combustion followed by LSC. Soils sampled within 1 month of treatment were extracted with methylene chloride:methanol:2M ammonium carbonate (3:4:1) (with stirring) for 1 hour. After filtration, soils were washed with additional

extractant. Combined extracts and wash were concentrated (rotary evaporator), acidified, (pH 5 with glacial acetic acid) and extracted three times with methylene chloride. The combined methylene chloride extracts and the aqueous phase were both reduced to dryness and residues taken up in methylene chloride or methanol, respectively. The methanol solutions were then filtered. Soils sampled > 1 month after treatment were extracted as described above then reextracted with acetone:1 M ammonium carbonate (3:1) by refluxing for 1 hour. The filtered extract was combined with the first extract. Concentration and methylene chloride partitioning were then performed as described above.

All extract fractions were analyzed for ^{14}C activity by LSC. The polar (in methanol) and nonpolar (methylene chloride) fractions of the soil extracts were analyzed by TLC and by HPLC. Samples analyzed by TLC were cochromatographed with authentic reference standards (see Appendix) and developed in either methylene chloride:methanol:ammonium hydroxide (144:50:6, TLC solvent 1) or acetonitrile:ethyl acetate:formic acid (150:50:1.5, TLC solvent 2). ^{14}C-labeled compounds on the plates were located by autoradiography. HPLC analysis utilized cochromatography with authentic reference standards on a reversed phase column with one of three mobile phases (see Table 16). Prior to HPLC, samples were taken to dryness under N_2 and redissolved in the appropriate mobile phase.

Residue identification was reportedly by comparison of TLC R_f values and HPLC retention times with those of authentic reference standards.

Laboratory (aerobic incubation) samples: Samples were extracted and partitioned as described above (field samples). The pH of the polar fraction of soil extract was adjusted to 8.8 [$(NH_4)_2CO_3$], and applied to an anion exchange column that had been equilibrated with $(NH_4)_2CO_3$. Sequential elution with 0.1 M $(NH_4)_2CO_3$, acetonitrile and water removed pigmented, nonradiolabeled compounds; formic acid: acetonitrile (50:50) eluted radiolabeled compounds. This eluate was collected and taken to dryness (rotary evaporator). Residues, redissolved in methanol, were subjected to TLC (silica gel plate, TLC solvent 2). The band containing most (79%) ^{14}C was scraped and extracted with methanol. The extract was concentrated, then purified by HPLC, using a Zorbax NH_2 column and a phosphoric acid (0.1 M):acetonitrile (9:1) mobile phase. Material eluting at 7.4 minutes (reportedly constituting a sharp peak) was collected and taken to dryness. The residue, in methanol, was purified (using TLC solvent 1 on an ethyl acetate washed silica TLC plate). The radiolabeled band was scraped, extracted with methanol, filtered, then taken to dryness. Residues were derivatized [bis(trimethylsilyl)trifluoroacetamide) and analyzed by GC/MS.

Results

Field study: The change in total ^{14}C recovery over time at the various sites is shown in Table 17. Recovery was high initially but decreased rapidly, reaching 37 to 62% of that applied by 8 weeks after application. In this period some activity leached below 16 cm at all sites. Only minor amounts of ^{14}C were detected below 16 cm in the (Newport) silt loam and (Bradenton) sand soils. In (Scott) silt loam soil up to 13% of applied activity was detected in the 16 to 24 cm increment but only traces leached below 24 cm. In the Clayton sand a significant proportion of recovered activity was at the 16 to 32 cm depth 8 weeks after application. The residue composition is summarized in Table 18. Between

Table 15. Characteristics of the soils at the field sites.

Location	Textural class	Sand	Silt	Clay	OM[a]	pH	CEC[b] meq/100 g
		%					
Newark, DE	Silt loam	21	62	17	2.8	6.4	8.2
Clayton, NC	Sand	91	8	1	0.3	4.9	6.1
Scott, MS[c]	Silt loam	15	67	18	1.3	5.5	14.3
Bradenton, FL	Sand	97	2	1	2.4	6.3	3.9

[a] Organic matter.

[b] Cation exchange capacity.

[c] MS site also referred to as Stoneville in study.

Table 16. HPLC mobile phases.

Mobile phase no.	Solvent mixture
1	4% acetonitrile in pH 2 H_2O[a]
2	20% acetonitrile in pH 2.2 H_2O[a]
3	36% acetonitrile in pH 2.2 H_2O[a]

[a] pH adjusted with phosphoric acid.

Table 17. Recovery of ^{14}C activity at four sites treated with phenyl-labeled [^{14}C]metsulfuron methyl.[a]

Site	Soil depth (cm)	Sampling interval (weeks) 0	1	2	4	8	16	26	52
Newport, DE	0-8	96	95	88	57	53	38	31	NR[b]
	8-16	3	<0.5	<0.5	10	6	12	8	NR
	16-24	<0.5	<0.5	<0.5	5	1	4	2	NR
	24-32	<0.5	<0.5	<0.5	2	2	2	1	NR
	Total	99	95	88	74	62	56	42	--
Rainfall[c]		0.0	0.5	1.1	6.9	8.2	15.8	22.7	
Clayton, NC	0-8	98	NR	109	85	17	9	NR	NR
	8-16	<0.5	NR	<0.5	<0.5	5	4	NR	NR
	16-24	<0.5	NR	<0.5	<0.5	5	6	NR	NR
	24-32	<0.5	NR	<0.5	<0.5	10	7	NR	NR
	Total	98	--	109	85	37	26	--	--
Rainfall[c]		0.0	--	0.0	0.1	3.8	11.4	--	--
Scott, MS	0-8	100	NR	91	76	44	33	30	15
	8-16	<1	NR	<1	8	2	14	16	7
	16-24	<1	NR	<1	<1	13	5	6	2
	24-32	<1	NR	<1	2	<1	1	1	1
	Total	100	--	91	86	59	53	53	25
Rainfall[c]		0.0	--	0.0	2.5	3.6	10.3	18.0	39.
Bradenton, FL	0-8	101	NR	68	75	47	30	19	NR
	8-16	<1	NR	14	2	9	8	6	NR
	16-24	<1	NR	<1	<1	2	2	1	NR
	24-32	<1	NR	<1	<1	<1	1	2	NR
	Total	101	--	82	77	58	41	28	--
Rainfall[c]		0.0	--	0.8	0.8	3.3	6.7	17.8	--

[a] Expressed as percent of ^{14}C applied.

[b] Not reported.

[c] Cumulative rainfall at each site in inches.

Table 18. Distribution of ^{14}C activity at four sites treated with phenyl-labeled [^{14}C]metsulfuron methyl.[a]

Site		Sampling interval (weeks)							
		0	1	2	4	8	16	26	52
Newport, DE	Metsulfuron methyl	97	50	28	12	3	2	2	NS[b]
	Methyl 2-(aminosulfonyl)-benzoate	<1	<1	1	1	<1	<1	1	NS
	Nonpolar, unidentified	1	2	4	7	<1	1	1	NS
	Saccharin	NR[c]	NR	23	11	9	2	2	NS
	2-(aminosulfonyl)-benzoic acid	NR	NR	17	21	29	13	14	NS
	Polar, unidentified	2[d]	34[d]	7	3	4	1	2	NS
	Bound	<1	9	7	17	16	36	20	NS
	Total	100	95	87	72	61	55	40	NS
Clayton, NC	Metsulfuron methyl	96	NS	78	48	8	3	NS	NS
	Methyl 2-(amino sulfonyl)-benzoate	NR	NS	5	2	2	1	NS	NS
	Nonpolar, unidentified	NR	NS	6	5	4	3	NS	NS
	Saccharin	NR	NS	12	16	9	6	NS	NS
	2-(aminosulfonyl)-benzoic acid	NR	NS	3	5	8	1	NS	NS
	Polar, unidentified	2	NS	3	5	2	8	NS	NS
	Bound	<1	NS	3	5	4	5	NS	NS
	Total	98	--	110	86	37	27	--	--
Scott, MS	Metsulfuron methyl	98	NS	66	41	16	12	10	<1
	Methyl 2-(aminosulfonyl)-benzoate	1	NS	9	2	2	1	<1	<1
	Nonpolar, unidentified	<1	NS	1	3	1	3	<1	<1
	Saccharin	1	NS	11	22	12	8	3	<1
	2-(aminosulfonyl)-benzoic acid	<1	NS	2	6	3	13	16	14
	Polar, unidentified	<1	NS	2	4	9	4	10	6
	Bound	<1	NS	<1	7	14	7	14	1
	Total	100	--	91	85	57	48	53	21
Bradenton, FL	Metsulfuron methyl	88	NS	28	10	5	2	1	NS
	Methyl 2-(amino sulfonyl)-benzoate	<1	NS	4	3	3	<1	<1	NS
	Nonpolar, unidentified	<1	NS	<1	4	1	1	<1	NS
	Saccharin	4	NS	14	32	17	3	2	NS
	2-(aminosulfonyl)-benzoic acid	<1	NS	1	10	5	9	1	NS
	Polar, Unidentifed	2	NS	7	7	9	4	7	NS
	Bound	7	NS	29	9	13	17	12	NS
	Total	101	--	83	75	53	36	23	--

[a] Expressed as a percent of ^{14}C applied.

[b] NS, not reported presumably not sampled.

[c] NR, not reported presumably not detected.

[d] Aqueous fractions of these samples were not analyzed.

Table 19. Distribution of ^{14}C residues at Clayton (NC) site 8 and 16 weeks after treatment with [^{14}C]metsulfuron methyl.

Sampling interval (weeks)	Sampling depth (cm)	Distrubution of residues[a]				
		Metsulfuron methyl	Methyl 2-(amino-sulfonyl)-benzoate	Saccharin	2-(aminosul-fonyl)-benzoic acid	Non-extractable
8	0-8	1.3	0.6	4.6	2.9	2.7
	8-16	2.1	0.5	1.6	1.7	0.8
	16-24	1.5	0.3	1.3	1.4	0.4
	24-32	2.4	0.3	1.5	1.6	0.4
16	0-8	2.4	0.8	1.0	0.2	3.2
	8-16	<0.1	<0.1	1.2	0.3	0.7
	16-24	<0.1	<0.1	1.9	0.3	0.6
	24-32	0.3	<0.1	1.5	0.3	0.4

[a] Expressed as percent of ^{14}C applied.

2 and 4 weeks posttreatment, levels of the degradates generally increased as levels of the parent decreased. The proportions of the degradates varied with site. After ~8 weeks levels of the degradates began to decline; overall none showed a strong tendency to accumulate.

The variation in degradate distribution with depth was reported at two of the sampling intervals (8 and 16 weeks) for the Clayton site only. The data are reported in Table 19. Eight weeks posttreatment the parent and the three metabolites are found distributed throughout the profile. After a further 8 weeks, the only trace quantities of parent and methyl 2-(aminosulfonyl)-benzoate were present below 8 cm. Saccharin, however, appeared to degrade at the shallow depth only; levels of 2-(aminosulfonyl)-benzoic acid declined throughout the profile. At both dates bound residues were concentrated at the 0-8 cm depth.

Aerobic Incubation: The HPLC retention time of the unknown compound extracted from aerobically aged, [^{14}C]metsulfuron methyl treated Keyport silt loam soil was 7.4 minutes; the elution time for saccharin was similar (7.2 minutes). TLC and GC/MS data also reportedly indicated that the unknown was saccharin.

Conclusions

[^{14}C]metsulfuron methyl, applied at 1 lb ai/A to soil confined in 10-cm x 38-cm steel cylinders, dissipated rapidly at four field sites. Less than 50% of applied activity was recovered as parent within 2 to 4 weeks of treatment. Leaching of ^{14}C residues below 8 cm occurred at all sites. Leaching depths and quantity varied with site. Three major degradation products were detected. The parent, polar, nonpolar unidentified degradates and bound materials comprised the remainder of the residue.

This study does not fulfill EPA Data Requirements for Registering Pesticides because the test substance was not a typical end use product and pretreatment soil samples were not collected from the intended application site. Climatic data were incomplete; soil or air temperatures were not provided.

The dissipation of metsulfuron methyl in soil confined in 10-cm diameter cylinders may not represent behavior under actual use conditions.

Variation in degradate distribution with depth was not adequately described in the report. Data for only one site (two sampling dates) are presented and total ^{14}C recoveries calculated from these data do not correspond to those given in Table 17. Unidentified polar and nonpolar compounds (reported in Table 18) are not quantified.

No copies of TLC plates, HPLC or mass spectra were provided for evaluation. The HPLC detection system was not indicated.

STUDY 7

Harvey, J. 1984. Crop rotation study with ^{14}C-DPX-T6376 in the greenhouse. E.I. du Pont de Nemours and Company, Inc., Wilmington, Delaware. Document No. AMR-120-83. Acc. No. 072767. Reference G-8.

Procedure

Aliquots of Woodstown sandy loam soil (58% sand, 26% silt and 6% clay, 1.4% organic matter, pH 4.9, CEC 5.2 meq/100 g) were placed in 16-inch diameter clay pots. Soil surface area was 0.113 m^2, the volume in each pot was 0.021 m^3. An aliquot of [^{14}C]metsulfuron methyl (uniformly labeled in the phenyl ring, specific activity 24.8 µCi/mg, 98.4% radiochemically pure) in acetone was applied to the soil surface. Each pot received 0.177 mg (9.54×10^6 dpm); equivalent to 15.6 g/ha (0.22 oz/A). Treated soils were kept moist and aged (greenhouse).

Sugarbeet (Monogerm Hybrid), rape (Altex), and oats (Noble) were sown in separate pots, 120 days after treatment. A fourth pot was sown with soybeans (Amsoy) 20 days later. Soil surfaces were lightly cultivated prior to planting. Control pots containing untreated fresh Woodstown soil were seeded concurrently. Plants were grown under ambient greenhouse conditions with a 14-hour day length maintained with fluorescent lighting.

Soils in representative pots were sampled 0, 70 and 172 days after planting. Plant samples were taken 35 and 70 days after planting and at maturity.

Methodology

Total ^{14}C levels in air-dried, ground soils and in freeze-dried plant tissues were estimated by combustion.

Subsamples of air-dried ground soil were extracted four times with methylene chloride:methanol:2M aqueous ammonium carbonate (3:4:1). The mixture was sonicated for 10 minutes then centrifuged. Combined supernatants were concentrated (rotary evaporator). The soil was refluxed with acetone:2M aqueous ammonium carbonate (3:1) for 1 hour. Filtered extracts from the refluxing procedure were combined with concentrate from the initial extractions then concentrated (rotary evaporator). The concentrate was diluted with water, acidified with glacial acetic acid (pH 4-5) and partitioned three times with methylene chloride. The aqueous phase was concentrated. Combined organic phases were dried ($MgSO_4$), filtered, and concentrated.

Reverse phase HPLC was performed on all concentrates. The mobile phase was 36% or 40% acetonitrile (for methylene chloride and aqueous concentrates, respectively), in pH 2.2 water (H_3PO_4).

Extracted soils were air dried. Residual ^{14}C activity was determined by combustion.

Analysis of plant tissues was not fully described. The registrant reports that "selected" mature plant tissues were extracted three times with acetone:water (4:1) using a tissue homogenizer. The acetone was removed from the combined extracts, which were then acidified. Resulting concentrates were partitioned three times with methanol then three times with water-saturated 1-butanol. Insufficient details of the further treatment of the resultant fractions were provided.

Table 20. Metsulfuron methyl residues during greenhouse study in soils treated at 16 g/ha with [^{14}C]metsulfuron methyl.

Residue[a]	Posttreatment interval (days)		
	120[b]	190	292
Total ^{14}C residue[c]	1.2	1.3	0.9
Metsulfuron methyl	0.12	NA[d]	<0.02
Methyl 2-(aminosulfonyl)-benzoate	0.06	NA	NR[e]
Saccharin	0.05	NA	0.10
2-(aminosulfonyl)-benzoic acid	0.10	NA	0.05
Unextracted residue	0.48	NA	NR

[a] All residue concentrations expressed in ppb.

[b] Crop planting.

[c] Calculated as (ppb) metsulfuron methyl.

[d] NA, not analyzed.

[e] NR, not reported.

Table 21. ^{14}C Residues in greenhouse crops grown in soil treated at 16 g/ha with [^{14}C]metsulfuron methyl.

Crop	Postplanting interval (days)		
	35	70	Maturity
Sugarbeet (foliage)	<1	2	2
Sugarbeet (root)	NR[b]	NR	1
Rape (foliage)	<1	2	4
Rape (root)	NR	NR	3
Oat (foliage)	10	8[c]	--
Oat (grain)	NR	2[c]	--
Soybean (foliage)	10[d]	3[e]	41
Soybean (bean)	NR	NR	2

[a] All results are expressed as ppb on a fresh weight basis except 70 day (mature) oats (ppb, dry weight basis).

[b] NR, not reported.

[c] Oats harvested (mature) 70 days after planting.

[d] 22 days after planting.

[e] 50 days after planting.

Table 22. Characterization of ^{14}C residues in soybean foliage and seeds from mature plants grown in [^{14}C]metsulfuron methyl-treated soil.[a]

Residue fraction	Foliage	Seeds
Total ^{14}C	41	2
Methylene chloride soluble	27[b]	0.5[b]
Butanol soluble	6	1.1
Water soluble	5	0.3
Unextracted	3	0.1

[a] Residues expressed as ppb metsulfuron methyl, expressed on a fresh weight basis.

[b] Metsulfuron methyl present at <0.03 ppb in these fractions (from HPLC).

The aqueous fraction and the (presumably combined) methylene chloride extracts were presumably subjected to HPLC as described above. Analysis of the butanol fraction was not described. It was also reported that residues in mature soybean foliage and seedlings were extracted and fractionated according to a method developed for metsulfuron methyl plant residues. The method was referenced but not available for review.

Results

Table 20 summarizes data on ^{14}C residues in soil. At seeding (120 days post-treatment) ^{14}C residues of 1.2 ppb were present, dominantly as unextracted material. The parent and three degradates were also identified.

^{14}C residues accumulated by rotational crops are quantified in Table 21. The fractionation of residues by mature soybean foliage and seeds is summarized in Table 22.

Conclusions

^{14}C residues accumulated in sugarbeet, rape, oat and soybean plants that were planted in a sandy loam soil 120 days after treatment with phenyl-labeled [^{14}C]-metsulfuron methyl at 15.6 g/ha (0.22 oz/A). Maximum residues in all species were detected at maturity. For sugarbeet and rape ≤4 ppb were detected in foliage or root. In soybean, 41 ppb was reported in foliage and 2 ppb in the bean (all expressed on a fresh weight basis). In mature oats, 8 ppb was detected in foliage and 2 ppb in straw (expressed on a dry weight basis).

Fractionation of the residues in mature soybeans indicated that ^{14}C in foliage was largely methylene chloride soluble; however >50% of that in beans was butanol soluble. The registrant's assignment of residue components to various fractions is not supported by data or chromatograms. Analytical procedures are not fully described, the technique used to analyze the butanol soluble fraction is not indicated.

This study provides data on accumulation of residues containing the phenyl ring but does not address the uptake of residues containing the triazine ring by rotational crops.

In addition, soil aging conditions (temperature, moisture content) were not reported and plant growth conditions were not fully described. No indication of variability in soil or plant residue analyses was provided. A low application rate (below the highest recommended rate) was applied in the study. A higher treatment rate may have facilitated residue characterization.

STUDY 8

Anderson, J.J. 1984. Crop rotation study with ^{14}C metsulfuron methyl in the field. E.I. du Pont de Nemours and Company, Inc., Wilmington, Delaware. Document No. AMR-190-84. Acc. No. 072767. Reference G-9.

Procedure

Winter wheat (Stephens) on a 25 m^2 plot was treated with phenyl-labeled [^{14}C]-metsulfuron methyl (specific activity 8.62 µCi/mg, 99% radiochemically pure) at 30 g/ha (0.43 oz/A) on May 21, 1982 crop at booting stage. The plot was rototilled (6-8 in deep) the following spring. Crops (oats, soybeans, rape, and sorghum) were planted on separate 1-m^2 subplots within the main plot 362 days posttreatment. Soil cores (10 over the 25 m^2 plot) were taken immediately prior to sampling. Soils were air-dried then ground. Red beets (garden variety) were planted 384 and 417 days posttreatment.

The beet crops failed soon after germination. Other crops were sampled at intervals up to maturity. Soil samples were taken after the final harvest (no details were provided).

Methodology

Total ^{14}C activity in aliquots of the air-dried ground soil samples was determined by combustion and LSC. The ten soil subsamples were mixed and an aliquot extracted with filtered methylene chloride:methanol:2M ammonium carbonate (3:4:1) with stirring for 1 hour. After filtration the soil was re-extracted by refluxing with filtered acetone:2M ammonium carbonate (3:1) for 1 hour. The filtered soil was air-dried, and residual ^{14}C activity quantified (combustion and LSC). The two extracts were combined, and the solvent removed (rotary evaporation). Residues were taken up in water and subsamples assayed (LSC) for ^{14}C activity. Aqueous residues were then acidified (pH 3) and extracted three times with methylene chloride. Radioactivity in the aqueous fraction and in the methylene chloride fraction (after concentration) was quantified by LSC.

Plant samples were weighed, freeze-dried and ground. Total ^{14}C was determined in subsamples of dried, ground plant material by combustion. Aliquots of plant material were extracted three times with acetone:water (4:1) in a tissue homogenizer. Combined extracts were taken to dryness (rotary evaporator). Residues were dissolved in acidified (pH 3, phosphoric acid) water and partitioned three times with methylene chloride. Aliquots of the combined methylene chloride fraction and of the aqueous fraction were assayed for ^{14}C activity (LSC).

Results

^{14}C residues in soils were 2 ppb (pre-planting) and 1 ppb (post harvest) (metsulfuron methyl equivalents). ^{14}C residues were reported in all plant samples (Table 23). The highest residue value recorded was for mature rape seed (31 ppb); maxima for other crops ranged from 4 ppb to 8 ppb (all expressed as metsulfuron methyl).

Fractionation of ^{14}C residues in treated soil and in mature rotational crops are summarized in Table 24. Methylene chloride soluble residues constituted 27 to 55% of the activity in mature rape, soybean and sorghum tissue; 8% of that in oat grain; and 79% of that in oat straw. Thus the range of radioactivity in plant tissue extracted into methylene chloride was 8% to 79% not 27 to 79% as stated in the report. The registrant states that previous work (ref-

Table 23. ^{14}C residues in rotational crops to winter wheat treated with phenyl-labeled [^{14}C]metsulfuron methyl at 30 g/ha.

Crop	Sampling interval (days)[a]	^{14}C residues (ppb)[b]
Oats		
Foliage	426	2
Straw	448[c]	9
Grain	448[c]	8
Rape		
Foliage	426	9
Foliage	448	4
Straw	462[c]	5
Seed	462[c]	31
Soybeans		
Foliage	426	4
Foliage	448	4
Foliage	448	1
Foliage	476	3
Foliage	535[c]	4
Foliage	535[c]	2
Bean		
Sorghum		
Foliage	426	3
Foliage	448	4
Foliage	448	4
Straw	489[c]	4
Grain	489[c]	2

[a] Days after last treatment.

[b] Expressed as metsulfuron methyl.

[c] Maturity date of crop.

Table 24. Partitioning of ^{14}C residues in rotational crops and in soil treated with phenyl-labeled [^{14}C]metsulfuron methyl.

Sample	Percent of total ^{14}C residues		
	Water soluble	Methylene Chloride soluble	Unextracted
Rape			
Seed	31	35	34
Straw	35	33	32
Soybean			
Seed	19	27	54
Foliage	16	55	29
Sorghum			
Seed	2	43	55
Straw	4	44	51
Oat			
Seed	<1	8	92
Straw	4	79	16
Soil	17	68	16

erenced but reference not available for review) indicated that the parent partitions into the methylene chloride fraction.

Conclusions

This study does not fulfill EPA criteria for field accumulation studies in rotational crops. Treatment was not made with a typical end use product and very small (1 m^2) plots were used. It was therefore evaluated as a confined accumulation study.

^{14}C residues accumulated in oats, soybeans, rape and sorghum planted 362 days posttreatment in soil treated at 30 g/ha (0.43 oz/A) with phenyllabeled [^{14}C]-metsulfuron methyl. Immature plant tissues contained 2-9 ppb. Residues in mature tissues were ≤4 ppb for soybeans and sorghum and ≤9 ppb for oats. Mature rape contained 31 ppb in seeds and 5 ppb in straw.

The study does not fulfill EPA data requirements because no soil characterization or climatic data were provided. Also no estimate of variability is provided for the soil analysis data.

Additional deficiencies were present. Insufficient details of pesticide application were given (if the application was made in solution, and if so, what solvent was used). It was not indicated whether residues were expressed on a fresh or dry weight basis.

Residues in mature plant tissues were fractionated but were reportedly too low for characterization. The results of one soil fractionation were provided but it was not clear if this was for the pre-sowing or post-harvest sample.

This study provides data on accumulation of residues containing the phenyl ring but does not address the uptake of residues containing the triazine ring by rotational crops.

REVIEW INFORMATION ON STUDIES INCLUDED IN THE SUBMISSION BY REFERENCE

Friedman, P. 1982. Hydrolysis of ^{14}C-phenyl DPX-T6376. Document No. AMR-62-82. Acc. No. 071434.

This study was reviewed by EAB (E. Regelman) on 5/20/83. It was determined that the study did not satisfy data requirements because it did not address the fate of the triazine moiety.

Friedman, P. 1983. Hydrolysis of ^{14}C-4-methoxy-6-methyl-1,3,5-triazin-2-amine. Document No. AMR-136-83. Acc. No. 252492.

This study was reviewed by EAB (E. Regelman) on 7/12/84. It was concluded that this study addresses the EAB's concerns on the fate of the triazine moiety when metsulfuron methyl is hydrolysed.

Rapisarda, C. 1981. Microbial degrdataion of ^{14}C-DPX-4189 in soil. Document No. AMR-43-81. Acc. No. 250928. The registrant has requested that this study be withdrawn without prejudice (E. Regelman, Memorandum of Meeting, 10/17/84).

Friedman, P. 1982. Aerobic soil metabolism of ^{14}C-phenyl-labeled-DPX-T6376. Document No. AMR-75-82. Acc. No. 071434.

This study was reviewed by EAB (E. Regelman) on 5/20/83. It was concluded that the study inadequately defined metsulfuron methyl metabolism because there was no monitoring of the triazine moiety.

Han, J. C-Y. 1981. ^{14}C-DPX-W4189. Soil disappearance studies in the field. Document No. AMR-54-81. Acc. No. 250928.

This study was reviewed by EAB (E. Regelman) on 7/12/84. This review concluded that the field dissipation of the aminotriazine moiety was not adequately defined by this study for full registration. EAB has since (E. Regelman, Memorandum of Meeting, 10/17/84) deferred assessment of the significance of residual levels of the moiety to the Residue Chemistry and Toxicological Branches.

Han, J. C-Y. 1982. Residue studies with [^{14}C]-DPX-T6376 in bluegill sunfish. Document No. AMR-81-82. Acc. No. 252492.

This study was reviewed by EAB (E. Regelman) on 7/12/84. Insufficient analytical and procedural details were provided to allow the study to fulfill data requirements. A submission of raw data for review was also requested.

EXECUTIVE SUMMARY

A previously reviewed study demonstrated that metsulfuron methyl was stable to hydrolysis at pH 7 and 9 at both 15°C and 25°C. Estimated half-lives of the parent at pH 5 were 3 weeks (25°C) and >30 days (15°C). The primary degradate was methyl 2-(aminosulfonyl)-benzoate. The hydrolytic stability of the triazine moiety was addressed in a study that showed 4-methoxy-6-methyl-1,3,5-triazin-2-amine was stable at pH 5, 7, and 9. No reliable quantitative data were submitted for photolysis in water or on soil.

No new aerobic metabolism studies were submitted. Previously, the estimated aerobic half-life of metsulfuron methyl in a silt loam soil was ~4 weeks. $^{14}CO_2$ was the major metabolite (36%); methyl 2-(aminosulfonyl)-benzoate, 2-(aminosulfonyl)-benzoic acid and saccharin were also identified. The fate of the triazine moiety was not addressed.

Anaerobic aquatic metabolism tests demonstrated that degradation rates may vary. Half-lives in three simulated pond systems varied from 7 to 37 weeks. Submitted data were considered inadequate to define metabolite distribution. The fate of the triazine moiety was not addressed.

Column leaching studies indicated that metsulfuron methyl was mobile in a range of soil types. Mobility was reduced by aerobic aging. In batch and soil TLC studies metsulfuron methyl was less extensively sorbed and was more mobile than terbacil or diuron.

Conclusions on mobility are tentative because the submitted studies contained insufficient information to meet guideline requirements.

The field dissipation data submitted did not meet data requirements. The study did, however, confirm that metsulfuron methyl may be mobile in silt loam and sandy soils.

Confined rotational crop studies indicate that residues may be taken up by sugarbeet, rape, oat, and soybeans planted in sandy loam soil 120 days after treatment at 0.22 oz ai/A.

Tentative conclusions from a previously reviewed study suggest that metsulfuron methyl does not bioaccumulate in bluegill sunfish.

In summary, metsulfuron methyl is stable to hydrolysis at pH 7 and 9 but is hydrolyzed at pH 5. Limited data suggest a soil aerobic half-life of ~4 weeks. Degradation is slower under anaerobic aquatic conditions.

Metsulfuron methyl appears to be mobile in a range of soil types. The environmental fate of the triazine moiety has not been adequately investigated.

RECOMMENDATIONS

Available data are insufficient to fully assess the environmental fate of metsulfuron methyl as well as the potential for exposure of humans and non-target organisms to metsulfuron methyl. The submission of data to fulfill registration requirements (Subparts N and K) is summarized below:

Hydrolysis studies: Two previously submitted and reviewed studies were cited in this submission. One study (Friedman, 1982, Document AMR-62-82, Acc. No. 071434) was scientifically valid, and partially fulfilled data requirements, but did not address the fate of the triazine moiety. The second study (Friedman, 1982, Acc. No. 252492) supplied appropriate information. Data requirements are satisfied; no further hydrolysis data are required.

Photodegradation studies in water: One study (Friedman, 1984, Document No. AMR-102-82, Acc. No. 072767) was submitted and reviewed. This study does not satisfy data requirements because metsulfuron methyl is not hydrolytically stable at the pH of the test solution. All data are required.

Photodegradation studies on soil: One study (Friedman, 1984, Document No. AMR-134-83, Acc. No. 072767) was submitted and reviewed. This study does not satisfy data requirements. The temperature at which the study was conducted was not reported. It was not indicated that control and irradiated samples were maintained at the same temperature. All data are required.

Photodegradation studies in air: No studies were submitted, all data maybe required pending reentry considerations.

Aerobic soil metabolism studies: Two previously submitted and reviewed studies were cited in this submission. One study (Rapisanda, C. 1981, Acc. No. 250928) was not considered because it has been withdrawn by the registrant. The second study (Friedman, 1982 Document AMR-62-82, Acc. No. 071434) partially satisfies data requirements but does not address the fate of the triazine moiety.

Anaerobic soil metabolism studies: No data were provided, however, an anaerobic aquatic metabolism study was submitted.

Anaerobic aquatic metabolism studies: One study (Friedman, 1984, Document No. AMR-134-83) was submitted and reviewed. Residues were not sufficiently characterized by this study to meet all data requirements. All data are required.

Aerobic aquatic metabolism studies: No data were submitted, but these studies are not required because metsulfuron methyl does not have an aquatic or aquatic impact use.

Leaching and adsorption/desorption studies: Two studies were submitted and reviewed. One study (Friedman, 1984 Acc. No. 072767, Reference G-6) providing information on the mobility of the parent compound only is judged scientifically valid. Characterization of test substances and further analytical details are required. Insufficient procedural and analytical information was provided to allow the second study (Chrzanowski, 1984, Acc. No. 072767) to fulfill data requirements on aged residues. In addition,the mobility of the triazine fragment was not investigated.

Laboratory and field volatility studies: No data were submitted. Requirements for these data depend upon toxicity data, product chemistry data, soil adsorption data, and methods of application.

Terrestrial dissipation studies: One new study (Anderson and Harvey, 1984, Acc. No. 072767) was submitted and reviewed. This study does not fulfill data requirements because treatment was not made with a typical end use product, pretreatment soil samples were not analyzed, and insufficient climatic data were reported. All data are required. One previously submitted and reviewed study was cited in this submission. This study (Han, 1981, Acc. No. 250928) provides data on the dissipation of the triazine moiety. Assessment of the significance of residual levels of this group has been deferred (E. Regelman Memorandum of Meeting, 10/17/84) to the Residue Chemistry and Toxicological Branches.

Aquatic field dissipation studies: No data were submitted, but no data are required because metsulfuron methyl does not have an aquatic or an aquatic impact use.

Forestry dissipation studies: No data were submitted, but no data are required because metsulfuron methyl does not have a forestry use.

Long-term field dissipaton studies: No data were submitted. Requirements for these data depend upon the results from the terrestrial field dissipation data.

Confined accumulation studies on rotational crops: Two studies were submitted and reviewed. One study (Anderson, 1984, Acc. No. 072767) does not fulfill data requirements because no soil characterization or climatic data were provided. The treatment rate in the second study (Harvey, 1984. Acc. No. 072767) was too low. Neither study addressed uptake of degradates containing the triazine moiety by rotational crops.

Field accumulation studies on rotational crops: No data were submitted. Data requirements are dependent upon confined accumulation studies on rotational crops.

Accumulation studies on irrigated crops: No data were submitted; however, data are not required because metsulfuron methyl has no aquatic food crop or aquatic noncrop use, is not used in and around holding ponds used for irrigation purposes, and has no uses involving effluents or discharges to water used for crop irrigation.

Laboratory studies of accumulation in fish: One previously submitted and reviewed study (Han, 1982, Acc. No. 252492) was cited in this submission. The study did not fulfill data requirements because insufficient analytical and procedural details were provided.

Field accumulation studies on nontarget organisms: No data were submitted; requirements for these studies depend upon the results from laboratory studies of accumulation in fish and toxicological data.

References

Anderson, J.J. 1984. Crop rotation study with ^{14}C metsulfuron methyl in the field. Document No. AMR-190-84. Acc. No. 072767.

Anderson, J.J. and J. Harvey. 1984. Field dissipation study of DPX-T6376 in Delaware, North Carolina, Florida and Mississippi. Document No. AMR-117-83. Acc. No. 072767.

Chrzanowski, R.L. 1984. Soil column leaching studies with [^{14}C]-DPX-T6376. Document No. AMR-82-82. Acc. No. 072767.

Friedman, P. 1982. Hydrolysis of ^{14}C-phenyl-DPX-T6376. Document No. AMR-62-82. Acc. No. 071434. (Included by reference not reviewed here).

Friedman, P. 1983. Aerobic soil metabolism of phenyl-labeled. DPX-T6376. Document No. AMR-75-82. Acc. No. 071434. (Included by reference not reviewed here).

Friedman, P. 1983. Hydrolysis of ^{14}C-4-methoxy-6-methyl-1,3,5-triazin-2-amine. Document No. AMR-136-83. Acc. No. 25492. (Included by reference not reviewed here).

Friedman, P. 1984. Aqueous photolysis of ^{14}C-DPX-T6376. Document No. AMR-102-82. Acc. No. 072767.

Friedman, P. 1984. Photodegradation of ^{14}C-phenyl-DPX-T6376 on soil. Document No. AMR-77-82. Acc. No. 072767.

Friedman, P.L. 1984. Anaerobic aquatic metabolism of [^{14}C-phenyl]-metsulfuron methyl. Document No. AMR-134-83. Acc. No. 072767.

Friedman, P.L. 1984. Adsorption of ^{14}C-DPX-T6376 on soil. Acc. No. 072767.

Han, J. C-Y. 1981. ^{14}C-DPX-W4189. Soil disappearance studies in the field. Document No. AMR-54-81. Acc. No. 250928. (Included by reference not reviewed here).

Han, J. C-Y. 1982. Residue studies with [^{14}C]-DPX-T6376 in bluegill sunfish. Document No. AMR-81-81. Acc. No. 252492. (Included by reference not reviewed here).

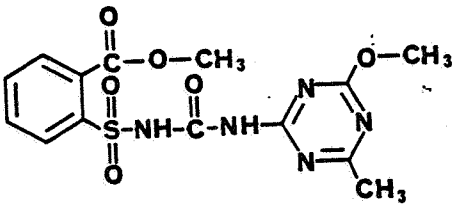
Harvey, J. 1984. Crop rotation study with ^{14}C-DPX-T6376 in the greenhouse. Document No. AMR-120-83. Acc. No. 072767.

Rapisarda, C. 1981. Microbial degradation of ^{14}C-DPX-4189 in soil. Report No. AMR-43-81. Acc. No. 250928.

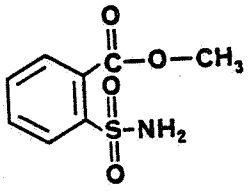
APPENDIX

STRUCTURES OF REFERENCE COMPOUNDS

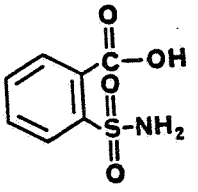
Structures of metsulfuron methyl and its proposed decomposition products.



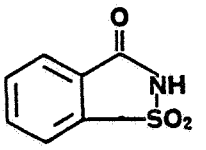
Methyl 2-[[[[(4-methoxy-6-methyltriazin-2-yl)-amino]carbonyl]amino]sulfonyl]-benzoate. (Metsulfuron methyl).



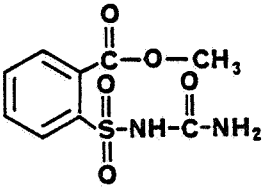
Methyl 2-(aminosulfonyl)-benzoate



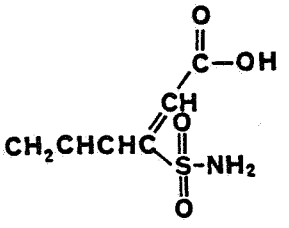
2-(Aminosulfonyl)-benzoic acid



Saccharin



Methyl 2-[N-(aminocarbonyl)aminsulfonyl]-benzoate



Proposed structure of the polar degradation product (Study 1).