

**BROMACIL ADDENDUM**

Final Report

**Task 1: Review and Evaluation of  
Individual Studies**

**Contract No. 68-01-6679**

**MARCH 7, 1985**

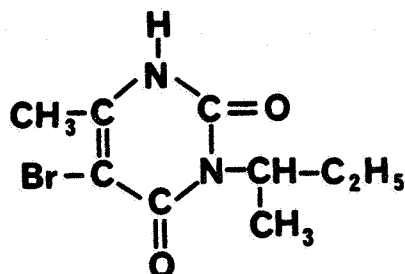
**Submitted to:**  
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Arlington, VA 22202

**Submitted by:**  
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## BROMACIL ADDENDUM

BOREA, BROMAX 4G, BROMAX 4L, CYNOGAN,  
HYVAR X, HYVAR X-L, ROUT, URAGON,  
UROX "B", UROX "HX"



5-Bromo-3-sec-butyl-6-methyluracil

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CASE GS0041 BROMACIL STUDY 1 PM

CHEM 012301 Bromacil

BRANCH EFB DISC --

FORMULATION 90 - FORMULATION NOT IDENTIFIED

FICHE/MASTER ID No MRID CONTENT CAT 01

Chrzanowski, R.L. 1984a. Photodegradation of bromacil in water. Unpublished study submitted March 10, 1983. In E.I. du Pont de Nemours and Company Response to Bromacil Registration Standard Environmental Fate. Submitted E.I. du Pont de Nemours and Company, Wilmington, DE. Accession No. 249679.

SUBST. CLASS = S.

DIRECT RVW TIME = 7 1/2 (MH) START-DATE

END DATE

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CONCLUSIONS:Degradation - Photodegradation in Water

This portion of the study is scientifically invalid because no dark controls were analyzed to confirm photolysis. In addition, this portion of the study would not fulfill EPA Data Requirements for Registering Pesticides (1983) because the purity of the test substance was not specified, the solutions were not buffered or stated to be free of live bacteria, and the sunlight was not completely characterized.

Metabolism - Anaerobic Aquatic

1. This portion of the study is scientifically valid.
2. [<sup>14</sup>C]Bromacil, at 250 ppm, degraded with a half-life of <12 weeks in sediment (uncharacterized) covered with river water (pH 6.5). After 12 weeks of incubation at 18-20 C in natural sunlight outdoors, 41% of the applied radiolabel was associated with the sediment. Of the radiolabel in the water, bromacil (1.1% of applied), 3-sec-butyl-5-acetyl-5-hydroxyhyantoin (2.8%), 3-sec-butyl-6-methyl-uracil (12%), 3-sec-butyl-ketohydantoin (1.5%), and sec-butyl-urea

(40%) predominated. Possible minor degradates included 3-sec-butyl-3H-imidazole-2,4-dione, 3-sec-butyl-5-hydroxyhydantoin, and 5-bromo-3-sec-butyl-5,6-epoxy-6-methyl-uracil.

3. This portion of the study does not fulfill EPA Data Requirements for Registering Pesticides (1983) because the purity of the test substance was not specified, characteristics of the sediment and water were not provided, and radioactive residues in the sediment were not characterized.

#### MATERIALS AND METHODS:

##### Degradation - Photodegradation in Water

Ring-labeled-[ $^{14}\text{C}$ ]bromacil (specific activity 1.0  $\mu\text{Ci}/\text{mg}$ , purity and source unspecified) was added at 250 ppm to 200 ml samples of distilled deionized water (pH 4.3), distilled water plus the photosensitizer riboflavin (pH 3.8), distilled water plus 10 ppm of the photosensitizer methylene blue (pH 4.1), or Standard Reference Water (pH 7.0). The solutions were placed in 400 ml beakers with quartz jackets (permitted passage of >95% of the available UV) and exposed to direct sunlight ( $1800 \mu\text{Em}^{-2}\text{s}^{-1}$ , sunlight not further characterized) outdoors beginning July 2, 1980. The solutions were maintained at 18-20 C during irradiation. The solutions were sampled immediately after treatment and after 1, 3, 6, and 12 weeks of irradiation.

Samples were extracted three times with ethyl acetate. Both the ethyl acetate extracts and the extracted water were analyzed for total radioactivity using LSC. Both solutions were separated using TLC with silica gel plates developed in methylene chloride:ethyl acetate (7:3). Radioactive areas were located on the plates by autoradiography and radio-scanning, quantified by LSC, and identified by comparison to bromacil, 3-sec-butyl-6-methyl-uracil (II), and sec-butyl-urea (IV) standards. Degradates for which standards were not available were identified using GC, coupled with MS.

In a related study, [ $^{14}\text{C}$ ]bromacil (specific activity 0.32 mCi/mg, purity and source unspecified) was added at 250 ppm to water containing 3 ppm of methylene blue (pH 9.4, adjusted using 0.1 N sodium hydroxide) and exposed to direct sunlight outdoors for 5 hours. The solution was aerated continuously at ambient temperatures (25-30 C). After 5 hours of irradiation, the solution was analyzed for photolysis products as described.

##### Metabolism - Anaerobic Aquatic

Ring-labeled [ $^{14}\text{C}$ ]bromacil was added at 250 ppm to 200 ml samples of river water (pH 6.3) or river water plus 1-inch of river sediment (pH 6.5) and irradiated with natural sunlight outdoors for up to 12 weeks as described.

The solutions were centrifuged to remove solids, and analyzed for total radioactivity and photolysis products using LSC, TLC, and GC/MS as described. The soil was analyzed for total radioactivity using LSC following combustion.

## REPORTED RESULTS:

### Degradation - Photodegradation in Water

[<sup>14</sup>C]Bromacil degraded with a half-life of <5 hours in distilled water plus methylene blue (pH 9.4), a half-life of 4-8 weeks in Standard Reference Water, and a half-life of 12 weeks in distilled water containing the photosensitizer riboflavin (Table 1). In distilled water and distilled water plus methylene blue (pH 4.1), ~90% of the applied bromacil remained unaltered after 12 weeks of irradiation. 3-sec-Butyl-5-acetyl-5-hydroxyhyantoin and sec-butyl-urea were the predominant degradates (Table 1, Figure 1).

### Metabolism - Anaerobic Aquatic

[<sup>14</sup>C]Bromacil degraded with a half-life of ~12 weeks in river water which did not contain sediment (Table 1). 3-sec-Butyl-5-acetyl-5-hydroxyhyantoin (20% of applied), 3-sec-butyl-ketohydantoin (13%), and sec-butyl-urea (13%) were the predominant degradates.

In the river water plus sediment, 41% of the applied radioactivity was associated with the sediment after 12 weeks of incubation (Table 2). sec-Butylurea comprised ~66% of the radioactivity in the water; 3-sec-butyl-6-methyl-uracil comprised ~20% of the radioactivity in the water.

## DISCUSSION:

### General (Both Studies)

1. The purity of the [<sup>14</sup>C]bromacil was not specified.
2. It was not specified if the flasks were sealed or open to evaporation during incubation.
3. No recovery or sensitivity data were reported for the analytical methods.

### Degradation - Photodegradation in Water

1. There were no dark controls to confirm that degradation was the result of exposure to light. Dark controls must be provided for each test solution type.
2. The solutions were not buffered. It was not specified that the solutions were free of live bacteria or that the study was conducted under sterile conditions.

3. Characteristics of the light, including hours of sunlight per day and site coordinates, were incomplete.

Metabolism - Anaerobic Aquatic

1. Radioactive residues in the sediment, which contained 41% of the applied radioactivity after 12 weeks of incubation, were not characterized. In addition, radioactivity in sediment was not quantified at any interval other than 12 weeks.
2. Complete sediment characteristics, including textural analysis, pH, organic matter content, and CEC, were not provided.
3. Complete water characteristics, including turbidity and COD, were not provided.

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Table 1. [ $^{14}\text{C}$ ]Bromacil and its radioactive degradates (% of applied) in aqueous solutions treated with [ $^{14}\text{C}$ ]bromacil at 250 ppm and irradiated by direct sunlight outdoors.

Test solutions	Sampling interval	Bromacil	I <sup>a</sup>	II <sup>b</sup>	III and Non Polars <sup>c</sup>	IV <sup>d</sup>	V, VI, VII and Polar $^{14}\text{C}$ <sup>e</sup>
Standard reference water (pH 7.0)	0 Day	94	1.7	1.4	ND <sup>f</sup>	1.2	2.0
	1 Week	86	6.0	1.6	ND	1.7	5.0
	2 Weeks	73	14.0	1.7	ND	4.0	7.0
	4 Weeks	54	27.0	ND	ND	4.1	13.0
	8 Weeks	39	34.0	ND	7.3	10.0	10.0
	12 Weeks	31	35.0	ND	2.9	14.0	17.0
Distilled water (pH 4.3)	0 Day	92	ND	5.0	ND	ND	3.0
	1 Week	96	0.4	1.3	ND	1.4	0.9
	3 Week	98	ND	1.4	ND	ND	1.0
	6 Weeks	94	0.8	1.8	ND	1.7	1.4
	12 Weeks	93	0.4	1.7	1.4	2.5	1.3
Distilled water plus methylene blue (pH 4.1)	0 Day	93	ND	3.0	ND	ND	2.0
	1 Week	95	0.7	1.4	ND	0.8	2.0
	3 Weeks	94	0.8	1.4	ND	0.7	3.1
	6 Weeks	92	1.0	1.7	ND	4.0	2.1
	12 Weeks	87	0.4	1.6	3.3	4.7	2.2
Distilled water plus riboflavin (pH 3.8)	0 Day	94	ND	2.0	ND	ND	2.4
	1 Week	81	1.2	1.0	ND	11	5.0
	3 Weeks	59	3.0	2.0	ND	32	5.0
	6 Weeks	77	0.8	1.4	ND	12	8.9
	12 Weeks	37	5.2	3.1	8.9	34	11.0
Distilled water plus methylene blue (pH 9.4)	0 Hour	100	ND	ND	ND	ND	ND
	5 Hours	ND	77.0	ND	10.0	1.0	129

<sup>a</sup> 3-sec-Butyl-5-acetyl-5-hydroxyhyanoin. (structures in Figure 1).

<sup>b</sup> 3-sec-Butyl-6-methyl-uracil.

<sup>c</sup> 3-sec-Butyl-ketohydantoin.

<sup>d</sup> sec-Butyl-urea.

<sup>e</sup> V: 3-sec-Butyl-3H-imidazole-2,4-dione; VI: 3-sec-butyl-5-hydroxyhydantoin; VII: 5-bromo-3-sec-butyl-5,6-epoxy-6-methyl-uracil.

<sup>f</sup> Not detected; detection limit was 0.1% of applied.

<sup>g</sup> The sediment contained 41% of the applied radioactivity after 12 weeks of irradiation.

Table 2. [ $^{14}\text{C}$ ]Bromacil and its radioactive degradates (% of applied) in river water treated with [ $^{14}\text{C}$ ]bromacil at 250 ppm and irradiated outdoors by direct sunlight.

Test solutions	Sampling interval	Bromacil	I <sup>a</sup>	II <sup>b</sup>	Nonpolars and III <sup>c</sup>	IV <sup>d</sup>	V, VI, VII and Polar $^{14}\text{C}$ <sup>e</sup>
River water	0 Day	92	ND <sup>f</sup>	6.0	ND	ND	1.6
	1 Week	86	3.6	1.7	ND	2.0	6.0
	3 Weeks	80	9.4	2.0	ND	2.6	5.8
	6 Weeks	66	18.0	2.5	ND	4.5	9.1
	12 Weeks	43	20.0	1.8	13.0	13.0	10.0
River water plus sediment	0 Day	86	ND	ND	ND	ND	14.0
	1 Week	50	2.1	0.7	ND	0.7	0.5
	3 Weeks	32	ND	ND	ND	1.0	3.7
	6 Weeks	14	16.0	7.2	ND	29.0	3.3
	12 Weeks <sup>9</sup>	1.1	2.8	12.0	1.5	40.0	1.9

<sup>a</sup> 3-sec-Butyl-5-acetyl-5-hydroxyhydantoin.

<sup>b</sup> 3-sec-Butyl-6-methyl-uracil.

<sup>c</sup> 3-sec-Butyl-ketohydantoin.

<sup>d</sup> sec-Butyl-urea.

<sup>e</sup> V: 3-sec-Butyl-3H-imidazole-2,4-dione; VI: 3-sec-butyl-5-hydroxyhydantoin; VII: 5-bromo-3-sec-butyl-5,6-epoxy-6-methyl-uracil.

<sup>f</sup> Not detected; detection limit was 0.1% of applied.

<sup>9</sup> The sediment contained 41% of the applied radioactivity after 12 weeks of irradiation.



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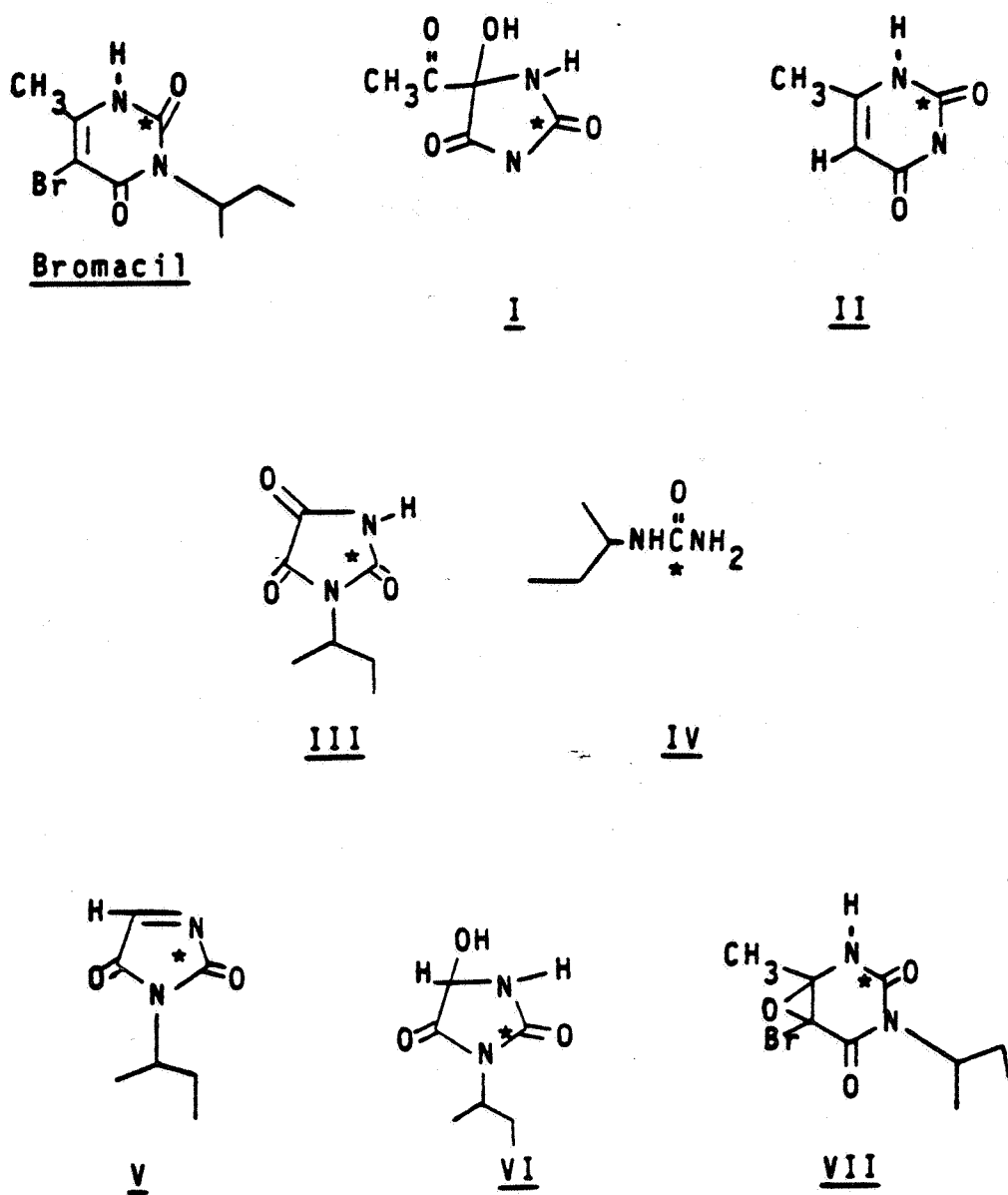


Figure 1. Bromacil and its degradates (\* denotes position of the radiolabel):  
 (I) 3-sec-butyl-5-acetyl-5-hydroxyhyantoin; (II) 3-sec-butyl-6-methyl-  
 uracil; (III) 3-sec-butyl-ketohydantoin; (IV) sec-butyl-urea, (V) 3-  
 sec-butyl-3H-imidazole-2,4-dione; (VI) 3-sec-butyl-5-hydroxyhydantoin,  
 (VII) 5-bromo-3-sec-butyl-5,6-epoxy-6-methyl-uracil.

CASE GS0041 BROMACIL STUDY 2 PM

CHEM 012301 Bromacil

BRANCH EFB DISC --

FORMULATION 00 - ACTIVE INGREDIENT

FICHE/MASTER ID No MRID CONTENT CAT 01  
Rapisarda, C. 1984. Degradation of <sup>14</sup>C-labeled Krovar Weed Killer in soil. Unpublished study submitted March 10, 1984. In E.I. du Pont de Nemours and Company Response to Bromacil Registration Standard Environmental Fate. Submitted by E.I. du Pont de Nemours and Company, Wilmington, DE. Accession No. 249679.

SUBST. CLASS = S.

DIRECT RVW TIME = 6 (MH) START-DATE END DATE

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CONCLUSIONS:Dissipation - Combination Products and Tank Mix Uses

1. This study is scientifically valid.
2. [<sup>14</sup>C]Bromacil, applied alone at 4 lb ai/A or in combination with [<sup>14</sup>C]diuron, each at 4 lb ai/A, dissipated with a half-life of ~2 months from the top 2 inches of a Keyport silt loam soil confined in stainless steel cylinders in a Delaware field plot. At the 0- to 2-inch depth of Myakka fine sand soil cylinders (Florida), [<sup>14</sup>C]bromacil dissipated with a half-life of 0.5-1 month following application with [<sup>14</sup>C]diuron (each applied at 3 lb ai/A). 5-Bromo-3-sec-butyl-6-hydroxymethyluracil; 5-bromo-3-(3-hydroxy-1-methylpropyl)-6-methyluracil; 5-bromo-3-( $\alpha$ -hydroxymethylpropyl)-6-methyluracil; 5-bromo-3-(2-hydroxy-1-methylpropyl)-6-methyluracil; 5-bromo-3-(3-hydroxy-1-methylpropyl)-6-hydroxymethyluracil; 5-bromo-6-methyluracil, and 4A, 10A-dibromo-3,9-di-sec-butyl-4B, 10B-dimethyl-cyclobutadi[1,2-D:3,4-DPR]pyrimidine-2,4,8,10-tetrone were found in soil samples from both field plots at maximum concentrations which were <2%

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of the applied at any sampling interval. Less than 30% of the radioactivity moved into lower soil depths (4-14 inches) at any sampling interval. Total radioactivity dissipated from the 14-inch soil cylinders with a half-life of <4 months in both soils treated with [ $^{14}\text{C}$ ]bromacil plus [ $^{14}\text{C}$ ]diuron, and half-life of ~8 months in the Keyport silt loam soil treated with [ $^{14}\text{C}$ ]bromacil alone.

3. Data requirements for combination products and tank mix uses are currently not being imposed for this Standard.

#### MATERIALS AND METHODS:

Stainless steel cylinders (4-inch diameter x 15-inch height) were driven into soil cleared of vegetation, leaving ~1-inch above ground level to minimize runoff and splashing. Field plots were located at Newark, Delaware, on Keyport silt loam soil (21% sand, 62% silt, 17% clay, 2.75% organic matter, pH 6.4, CEC 8.2 meq/100 g) and at Brandenton, Florida, on Myakka fine sand (97% sand, 2% silt, 1% clay, 2.43% organic matter, pH 6.3, CEC 3.9 meq/100 g). Cylinders in the Keyport silt loam soil field plot were treated as follows: 3 cylinders were treated with [ $^{14}\text{C}$ ]diuron (specific activity 4.11  $\mu\text{Ci}/\text{mg}$ , radiopurity 95.2%, source unspecified) at 4 lb ai/A; 3 cylinders were treated with [ $^{14}\text{C}$ ]bromacil (specific activity 3.96  $\mu\text{Ci}/\text{mg}$ , radiopurity >99%, source unspecified) at 4 lb ai/A; and 8 cylinders were treated with [ $^{14}\text{C}$ ]diuron plus [ $^{14}\text{C}$ ]bromacil (Krovar I Weed Killer, diuron and bromacil in 1:1 molar ratio, specific activity 4.04  $\mu\text{Ci}/\text{mg}$ , radiopurity >97%, E.I. du Pont de Nemours and Co.) at 8 lb ai/A. The Myakka fine sand field plot contained 8 cylinders which were treated with [ $^{14}\text{C}$ ]diuron plus [ $^{14}\text{C}$ ]bromacil, at 6 lb ai/A.

The cylinders were removed at intervals up to 18 months after treatment, and the soil samples were divided into 0- to 2-, 2- to 4-, 4- to 6-, 6- to 8-, 8- to 10-, 10- to 12-, and 12- to 14-inch segments. Soil segments were air dried, mixed, and aliquots were combusted. The  $^{14}\text{CO}_2$  evolved was trapped and quantified using LSC.

Additional aliquots of soil were extracted five times with methylene chloride:methanol:ammonium hydroxide (75:24.9:0.1), followed by three extractions with 0.1 N sodium hydroxide. The extracts were combined, adjusted to pH 7 with dilute hydrochloric acid, and partitioned three times with ethyl acetate in a separatory funnel. The extracted soil was then refluxed with 1 N sodium hydroxide, the extract was neutralized to pH 7, and partitioned with ethyl acetate. All ethyl acetate extracts were then combined, aliquots were analyzed for total radioactivity using LSC, and the remainder was concentrated to a small volume. Aliquots of the concentrated extracts were spotted onto silica gel TLC plates along with known standards, and the plates were developed by two-dimensional TLC using ethyl acetate in the first direction, and methylene chloride:methanol (95:5, v:v) in the second. After development, the TLC plates were autoradiographed, and radioactive areas were scraped from the plates and quantified using LSC. The extracted soil samples were combusted, and the  $^{14}\text{CO}_2$  evolved was trapped and quantified using LSC.

To confirm the identities of the degradates, a portion of the combined ethyl acetate extract was cleaned up using preparative TLC and Florisil column chromatography, and then analyzed using MS.

#### REPORTED RESULTS:

Cumulative rainfall at the test sites during the study period is shown in Table 1. In the Keyport silt loam soil, total radioactivity dissipated from the 14-inch soil columns with a half-life of <8 months when [ $^{14}\text{C}$ ]bromacil was applied alone, and 4 months when [ $^{14}\text{C}$ ]diuron was applied in combination with [ $^{14}\text{C}$ ]bromacil (Table 2). The majority of the radioactivity recovered at any sampling interval was found in the 0- to 4-inch soil depth; <10% of the applied radioactivity was found in any soil segment at lower depths. Total radioactivity dissipated from the Myakka sand soil treated with [ $^{14}\text{C}$ ]diuron plus [ $^{14}\text{C}$ ]bromacil with a half-life of 1-2 months, and <10.2% of the applied radioactivity was found in any soil segment below the 4-inch depth (Table 3).

[ $^{14}\text{C}$ ]Bromacil dissipated from the 0- to 2-inch depth of the Keyport silt loam soil with a half-life of ~2 months when applied alone or in combination with [ $^{14}\text{C}$ ]diuron, and had declined to 2.8% of the applied [ $^{14}\text{C}$ ]bromacil by 12 months after treatment (Tables 4 and 5). Dissipation of [ $^{14}\text{C}$ ]bromacil from the Myakka sand soil was more rapid, with a half-life of ~1 months (Table 6).

The bromacil degradates 5-bromo-3-sec-butyl-6-hydroxymethyluracil (Degradate A); 5-bromo-3-(3-hydroxy-1-methylpropyl)-6-methyluracil (B); 5-bromo-3-( $\alpha$ -hydroxymethylpropyl)-6-methyluracil (C); 5-bromo-3-(2-hydroxy-1-methylpropyl)-6-methyluracil (D); 5-bromo-3-(3-hydroxy-1-methylpropyl)-6-hydroxymethyluracil (E); and 5-bromo-6-methyluracil (G), plus the bromacil dimer 4A, 10A-dibromo-3,9-di-sec-butyl-4B, 10B-dimethylcyclobutadi[1,2-D:3,4-DPR]pyrimidine-2,4,8, 10-tetrone were found in both field plots (structures illustrated in Figure 1). Maximum concentrations were <1.7% of the applied, and generally occurred in the surface 2 inches of soil (Tables 4-6). Polar origin material (uncharacterized) accounted for <1.7% of the applied radioactivity in any soil sample.

#### DISCUSSION:

1. The test substances used were not typical end-use products.
2. Field plots of Keyport silt loam soil treated with [ $^{14}\text{C}$ ]bromacil alone were not sampled until 2 months after treatment. Pretreatment samples were not taken from any plot.
3. Results of bromacil dissipation in soil confined to cylinders may not be representative of dissipation under actual use conditions.
4. Field plots of Myakka fine sand soil were treated only with [ $^{14}\text{C}$ ]diuron plus [ $^{14}\text{C}$ ]bromacil; the dissipation of bromacil when applied alone and in combination with another pesticide was not compared.

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5. Field test data, such as slope of the test site, depth of the water table, and soil and air temperatures, were not reported.
6. Sample storage techniques were not reported.
7. No explanation was provided for the increased persistence of total radioactive residues in the Keyport silt loam soil treated with [ $^{14}\text{C}$ ]bromacil alone.

Table 1. Cumulative rainfall (inches) at test sites.

Sampling interval (days)	Keyport silt loam soil (Delaware)	Myakka fine sand soil (Florida)
0	0.0	0.0
0.5	0.6	2.7
1	2.1	6.2
2	6.0	9.0
4	17.1	34.9
8	27.8	43.6
12	42.1	54.1
18	57.9	83.1

Table 2. Total radioactivity (% of applied) in Keyport silt loam soil treated with bromacil alone or in combination with diuron.

Sampling depth (inches)	Sampling interval (months)							
	0	0.5	1	2	4	8	12	10
<u>[<sup>14</sup>C]Diuron plus [<sup>14</sup>C]bromacil, at 8 lb ai/A</u>								
0-2	98.1	66.1	67.7	43.8	32.4	24.2	25.6	16.9
2-4	1.9	1.6	11.7	19.7	4.7	12.0	3.0	2.4
4-6	ND <sup>a</sup>	0.2	2.4	5.6	3.0	2.9	1.2	0.9
6-8	ND	0.1	0.5	3.1	3.2	2.4	1.3	0.5
8-10	ND	ND	0.2	1.3	1.7	2.3	0.9	0.4
10-12	ND	ND	0.1	0.3	0.8	1.0	0.4	0.2
12-14	ND	ND	ND	0.2	0.6	1.0	0.4	0.1
Total <sup>14</sup> C	100.0	68.0	82.6	74.0	46.4	45.8	32.8	21.4
<u>[<sup>14</sup>C]Bromacil, at 4 lb ai/A</u>								
0-2	-- <sup>b</sup>	--	--	27.6	--	6.0	4.7	--
2-4	--	--	--	27.7	--	3.9	2.2	--
4-6	--	--	--	9.2	--	3.8	2.2	--
6-8	--	--	--	2.7	--	4.1	2.0	--
8-10	--	--	--	1.2	--	3.7	1.4	--
10-12	--	--	--	0.5	--	2.1	0.7	--
12-14	--	--	--	0.2	--	1.3	0.5	--
Total <sup>14</sup> C	--	--	--	69.1	--	24.9	13.7	--

<sup>a</sup> Not detected; detection limit is 0.1% of the applied radioactivity.

<sup>b</sup> Not sampled.

Table 3. Total radioactivity (% of applied) in Myakka fine sand treated with [ $^{14}\text{C}$ ]diuron plus [ $^{14}\text{C}$ ]bromacil, at 6 lb ai/A.

Sampling depth (inches)	Sampling interval (months)							
	0	0.5	1	2	4	8	12	10
0-2	84.9	66.1	19.6	20.9	17.1	17.6	13.8	7.1
2-4	14.5	19.3	20.7	10.3	6.9	10.8	10.4	8.1
4-6	0.6	9.4	10.2	4.1	7.0	4.6	2.7	4.6
6-8	ND <sup>a</sup>	3.9	5.3	2.1	1.8	2.7	0.6	1.1
8-10	ND	1.1	6.2	3.5	0.8	1.1	0.3	0.2
10-12	ND	0.2	6.0	3.6	0.9	0.9	0.1	0.1
12-14	ND	ND	1.2	1.3	0.6	0.2	ND	ND
Total $^{14}\text{C}$	100.0	100.0	69.2	45.8	35.1	37.9	27.9	21.3

<sup>a</sup> Not detected; detection limit is 0.1% of the applied radioactivity.



Table 4. Distribution of radioactivity (% of applied) in Keyport silt loam soil treated with [<sup>14</sup>C]diuron plus [<sup>14</sup>C]bromacil, at 8 lb ai/A.

Sampling interval (months)	Sampling depth (inches)	Bromacil	Degradate A <sup>a</sup>	Degradates B,C,D <sup>b</sup>	Degradate E <sup>c</sup>	Degradate G <sup>d</sup>	Bromacil Dimer <sup>e</sup>	Polar material	Diuron residues <sup>f</sup>	Soil bound <sup>g</sup>	Total
0	0-2	47.4	0.3	0.2	ND <sup>h</sup>	ND	ND	0.1	49.1	0.9	98.1
	2-4	1.3	ND	ND	ND	ND	ND	ND	0.5	0.1	1.9
0.5	0-2	29.0	1.7	0.5	0.5	0.3	0.1	0.5	32.2	1.3	66.1
	2-4	0.9	ND	ND	ND	ND	ND	ND	0.5	0.1	1.6
1	0-2	27.6	0.7	0.5	0.6	0.2	0.1	0.8	34.9	2.3	67.7
	2-4	7.4	0.3	ND	0.3	0.1	ND	0.2	3.0	0.3	11.7
	6-8	0.3	ND	ND	ND	ND	ND	ND	0.1	ND	0.4
2	0-2	9.4	0.1	0.1	0.2	ND	ND	0.4	30.8	2.8	43.8
	2-4	12.8	0.3	0.2	0.2	ND	0.1	0.6	4.8	0.7	19.7
	6-8	2.2	0.3	0.1	ND	ND	ND	0.2	0.2	ND	3.1
4	0-2	2.0	0.1	ND	0.1	ND	ND	0.2	26.6	3.4	32.4
	4-6	2.0	0.1	0.1	ND	ND	ND	0.3	0.4	0.1	3.0
	8-10	1.3	0.2	ND	ND	ND	ND	ND	0.1	ND	1.6
8	0-2	1.8	0.1	ND	ND	ND	ND	0.4	17.9	3.9	24.2
	4-6	2.0	0.1	ND	ND	ND	ND	0.1	0.4	0.2	2.9
	8-10	1.8	0.1	ND	ND	ND	ND	ND	0.2	0.1	2.3
	12-14	0.7	0.1	ND	ND	ND	ND	ND	0.1	0.1	1.0
12	0-2	1.4	0.1	0.1	0.1	0.1	ND	0.6	20.4	2.8	25.6
	4-6	0.6	0.2	ND	ND	ND	ND	ND	0.3	ND	1.2
	8-10	0.5	0.1	ND	ND	ND	ND	ND	0.2	ND	0.9
18	0-2	1.1	0.2	0.1	0.1	ND	ND	0.6	11.2	3.6	16.9
	4-6	0.2	ND	ND	ND	ND	ND	ND	0.5	0.1	0.9
	8-10	0.1	ND	ND	ND	ND	ND	ND	0.2	ND	0.4

<sup>a</sup> 5-Bromo-3-sec-butyl-6-hydroxymethyluracil (structures shown in Figure 1).

<sup>b</sup> 5-Bromo-3-(3-hydroxy-1-methylpropyl)-6-methyluracil (B); 5-bromo-3-(=hydroxymethylpropyl)-6-methyluracil (C); 5-bromo-3-(2-hydroxy-1-methylpropyl)-6-methyluracil (D).

<sup>c</sup> 5-Bromo-3-(3-hydroxy-1-methylpropyl)-6-hydroxymethyluracil.

<sup>d</sup> 5-Bromo-6-methyluracil.

<sup>e</sup> 4A, 10A-dibromo-3,9-di-sec-butyl-4B,10B-dimethyl-cyclobutadi[1,2-D:3,4-DPR]pyrimidine-2,4,8,10-tetrone.

<sup>f</sup> Parent diuron plus degradates.

<sup>g</sup> Unextractable radioactivity; determined by combustion analysis.

<sup>h</sup> Not detected; detection limit is 0.1% of the applied radioactivity.

Table 5. Distribution of radioactivity (% of applied) in Keyport silt loam soil treated with [<sup>14</sup>C]bromacil at 4 lb ai/A.

Sampling interval (months)	Sampling depth (inches)	Bromacil	Degradate A <sup>a</sup>	Degradates B,C,D <sup>b</sup>	Degradate E <sup>c</sup>	Degradate G <sup>d</sup>	Bromacil Dimer <sup>e</sup>	Polar material	Soil bound <sup>f</sup>	Total
2	0-2	24.0	0.6	0.5	0.1	0.1	0.3	1.7	0.2	27.6
	4-6	7.4	0.5	0.3	0.2	ND <sup>g</sup>	0.1	0.8	ND	9.6
	8-10	0.6	0.1	ND	ND	ND	ND	0.4	ND	1.2
8	0-2	4.8	0.2	0.1	0.1	ND	ND	0.1	0.6	6.0
	4-6	3.2	0.2	0.1	ND	ND	ND	ND	0.2	3.8
	12-14	0.9	0.2	ND	ND	ND	ND	ND	0.1	1.3
12	0-2	2.8	0.2	0.2	0.1	ND	ND	0.8	0.6	4.7
	4-6	1.7	0.2	0.1	0.1	ND	ND	ND	0.1	2.2
	12-14	ND	0.2	ND	ND	ND	ND	ND	0.2	0.5

a 5-Bromo-3-sec-butyl-6-hydroxymethyluracil (structures shown in Figure 1).

b 5-Bromo-3-(3-hydroxy-1-methylpropyl)-6-methyluracil (B); 5-bromo-3-(2-hydroxymethylpropyl)-6-methyluracil (C); 5-bromo-3-(2-hydroxy-1-methylpropyl)-6-methyluracil (D).

c 5-Bromo-3-(3-hydroxy-1-methylpropyl)-6-hydroxymethyluracil.

d 5-Bromo-6-methyluracil.

e 4A, 10A-dibromo-3,9-di-sec-butyl-48,108-dimethyl-cyclobutadi[1,2-D:3,4-DPR]pyrimidine-2,4,8,10-tetrone.

f Unextractable radioactivity; determined by combustion analysis.

g Not detected; detection limit is 0.1% of the applied radioactivity.

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Table 6. Distribution of radioactivity (% of applied) in Myakka fine sand soil treated with [<sup>14</sup>C]diuron plus [<sup>14</sup>C]bromacil, at 6 lb ai/A.

Sampling interval (months)	Sampling depth (inches)	Bromacil	Degradate A <sup>a</sup>	Degradates B,C,D <sup>b</sup>	Degradate E <sup>c</sup>	Degradate G <sup>d</sup>	Bromacil Dimer <sup>e</sup>	Polar material	Diuron residues <sup>f</sup>	Soil bound <sup>g</sup>	Total
0	0-2	42.5	0.9	0.3	ND <sup>h</sup>	ND	ND	0.1	40.7	0.3	84.9
	2-4	7.6	0.3	0.1	ND	ND	ND	ND	6.3	0.2	14.5
0.5	0-2	25.1	0.4	0.2	0.3	ND	ND	0.5	39.1	0.4	66.1
	4-6	7.1	0.2	0.1	0.1	ND	ND	0.2	1.6	0.1	9.4
	8-10	0.7	ND	ND	ND	ND	ND	ND	0.3	ND	1.1
1	0-2	3.6	0.1	0.1	ND	0.1	ND	0.2	15.4	0.1	19.6
	4-6	6.8	0.3	0.1	0.1	0.1	ND	0.1	2.7	ND	10.2
	8-10	5.0	0.2	0.1	0.3	0.1	ND	0.1	0.4	ND	6.2
	12-14	1.0	ND	ND	ND	ND	ND	ND	0.1	ND	1.2
2	0-2	2.1	0.2	ND	0.1	0.1	ND	0.2	17.9	0.2	20.9
	4-6	1.2	ND	ND	ND	ND	ND	0.1	2.6	0.1	4.1
	8-10	2.5	0.2	0.1	0.1	ND	ND	0.3	0.3	ND	3.5
	12-14	0.9	0.1	ND	ND	ND	ND	0.1	0.1	ND	1.3
4	0-2	1.3	0.1	ND	ND	ND	ND	0.2	12.7	2.7	17.1
	4-6	0.8	ND	ND	ND	ND	ND	0.1	4.4	1.6	7.0
	8-10	0.2	0.1	ND	ND	ND	ND	ND	0.1	0.3	0.8
8	0-2	2.7	0.4	0.5	0.2	0.2	0.1	0.1	11.7	1.7	17.6
	4-6	1.3	0.1	0.1	ND	ND	ND	ND	2.4	0.6	4.6
	8-10	0.4	0.1	ND	ND	ND	ND	ND	0.2	0.3	1.1
12	0-2	0.5	0.1	0.1	ND	ND	ND	ND	12.3	0.7	13.8
	4-6	0.2	ND	ND	ND	ND	ND	ND	3.8	0.5	4.6
18	0-2	0.2	ND	ND	ND	ND	ND	ND	6.4	0.4	7.1
	4-6	0.2	ND	ND	ND	ND	ND	ND	4.1	0.2	4.6

a 5-Bromo-3-sec-butyl-6-hydroxymethyluracil (structures shown in Figure 1).

b 5-Bromo-3-(3-hydroxy-1-methylpropyl)-6-methyluracil (B); 5-bromo-3-(2-hydroxymethylpropyl)-6-methyluracil (C); 5-bromo-3-(2-hydroxy-1-methylpropyl)-6-methyluracil (D).

c 5-Bromo-3-(3-hydroxy-1-methylpropyl)-6-hydroxymethyluracil.

d 5-Bromo-6-methyluracil.

e 4A, 10A-dibromo-3,9-di-sec-butyl-4B,10B-dimethyl-cyclobutadi[1,2-D:3,4-DPR]pyrimidine-2,4,8,10-tetrone.

f Parent diuron plus degradates.

g Unextractable radioactivity; determined by combustion analysis.

h Not detected; detection limit is 0.1% of the applied radioactivity.

-11-

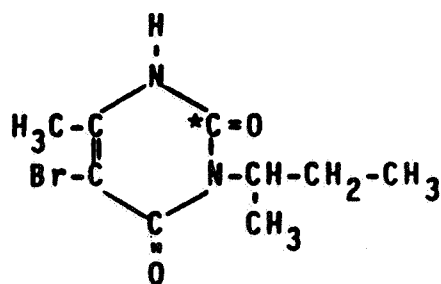
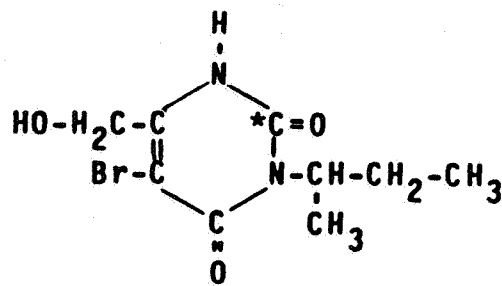
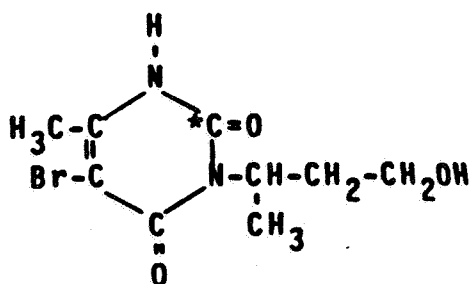
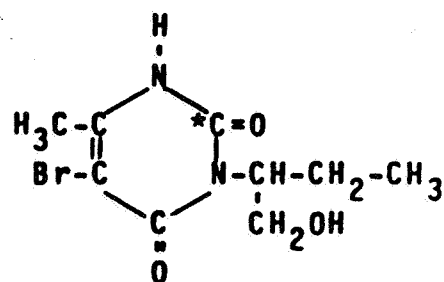
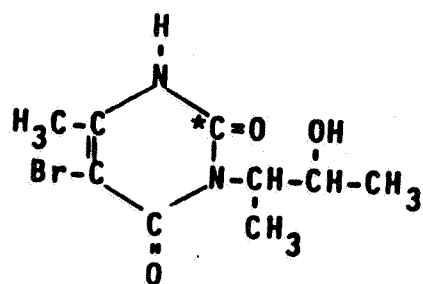
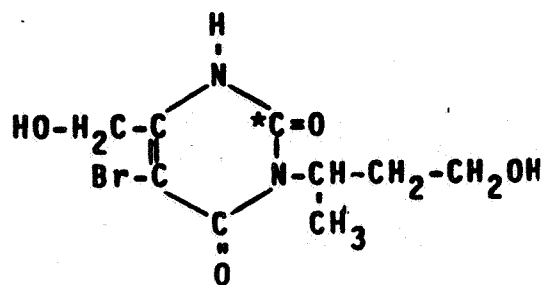
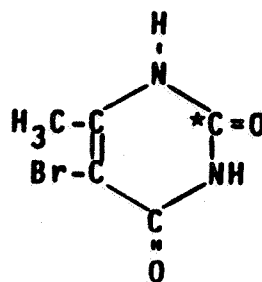
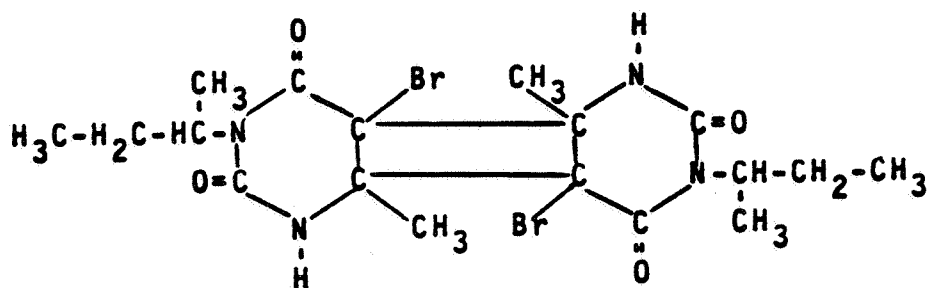
BromacilABC

Figure 1. Bromacil and its degradates (\* denotes position of radiolabel):  
 (A) 5-bromo-3-sec-butyl-6-hydroxymethyluracil; (B) 5-bromo-3-(3-hydroxy-1-methylpropyl)-6-methyluracil; (C) 5-bromo-3-( $\alpha$ -hydroxymethylpropyl)-6-methyluracil.

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DEG

Bromacil dimer

Figure 1. (Continued): (D) 5-bromo-3-(2-hydroxy-1-methylpropyl)-6-methyluracil; (E) 5-bromo-3-(3-hydroxy-1-methylpropyl)-6-hydroxymethyluracil; (G) 5-bromo-6-methyluracil; (Dimer) 4A, 10A-dibromo-3,9-di-sec-butyl-4B,10B-dimethyl-cyclobutadi[1,2-D:4-DPR]pyrimidine-2,4,8,10-tetrone.

CASE GS0041 BROMACIL STUDY 3 PM

CHEM 012301 Bromacil

BRANCH EFB DISC --

FORMULATION 90 - FORMULATION NOT IDENTIFIED

FICHE/MASTER ID No MRID CONTENT CAT 01  
Chrzanowski, R.L. 1984b. Soil column adsorption studies with 2-<sup>14</sup>C-bromacil. Unpublished study received March 10, 1984. In E.I. du Pont de Nemours and Company Response to Bromacil Registration Standard Environmental Fate. Submitted by E.I. du Pont de Nemours and Company, Wilmington, DE. Accession No. 249679.

SUBST. CLASS = S.

DIRECT RVW TIME = 4 1/2 (MH) START-DATE END DATE

REVIEWED BY: K. Patten  
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TEL: 468-2500

SIGNATURE: *K. Patten*

DATE: Jan. 18, 1985

APPROVED BY:  
TITLE:  
ORG:  
TEL:

SIGNATURE:

DATE:

CONCLUSIONS:Mobility - Leaching and Adsorption/Desorption

1. This study is scientifically valid.
2. [<sup>14</sup>C]Bromacil, at 5 lb ai/A, was very mobile in columns of sandy loam soil, with 104% of the applied detected in the leachate after ~20 inches of water percolated through the soil over a 2-day period. Aged (30-day) bromacil residues (uncharacterized) were similarly mobile, with ~80% of the radioactivity (before aging) detected in the leachate. [<sup>14</sup>C]Bromacil and aged bromacil residues were mobile in columns of silt loam and muck soils, but <2% of the applied was removed from these soils in the leachate. The maximum concentration of radioactive residues was detected at the 8-16 inch depth of the silt loam soil column and the 0-10 inch depth of the muck soil column.
3. This study does not fulfill EPA Data Requirements for Registering Pesticides (1983) because the aged residues were not characterized in the soil or leachate, radioactive residues (aged and unaged) in the sandy loam soil were not quantified, the test substance was not characterized, incubation conditions during the aging of the bromacil were incomplete, and K<sub>d</sub> values were not calculated.

#### MATERIALS AND METHODS:

Ring-labeled [ $^{14}\text{C}$ ]bromacil (0.93  $\mu\text{Ci}$ /1.1 mg, labeled in the 2-position, purity and source unspecified) in water was added to the upper soil surface of glass columns (2 inch diameter, 18 inch depth) containing Fallsington sandy loam, Flanagan silt loam, or muck soils (soil characteristics presented in Table 1) at 5 lb ai/A. Water was immediately and continuously percolated through the columns until a total of 20 inches of leachate had been collected (2 days). The leachate was collected in 0.2-inch increments and analyzed for total radioactivity using LSC. Following leaching, the soil columns were divided into 2 inch segments, and the segments were dried and analyzed for total radioactivity using LSC following combustion.

In a related study, samples (100 g) of each soil were treated with [ $^{14}\text{C}$ ]-bromacil (1.1 mg, 0.93  $\mu\text{Ci}$ ) and incubated for 30 days in a greenhouse during May in Delaware. The Fallsington sandy loam soil was maintained at 75% of the water holding capacity of the soil, the Flanagan silt loam soil at 65%, and the muck soil at 97% (no additional incubation conditions provided). The treated soils were added to the top 2-inches of columns (16 inches) containing the appropriate soil, for a total column depth of 18 inches. The columns were then leached and sampled as described.

#### REPORTED RESULTS:

During the leaching of the columns of freshly treated and aged Fallsington sandy loam soil, 104 and 80% of the applied radioactivity, respectively, was collected in the leachate (Table 2). In contrast, <2% of the applied radioactivity leached from the columns of Flanagan silt loam soil and <1.8% leached from the columns of muck soil.

Radioactive residues were most concentrated in the 8-16 inch depth in the freshly treated (62.9% of recovered after leaching) and aged (65.0%) columns of Flanagan silt loam (Table 3). Radioactive residues were most concentrated in the upper 6 inches of the column containing freshly treated muck soil and upper 10 inches of the column containing aged muck soil.

#### DISCUSSION:

1. The aged radioactive residues were not characterized when they were initially added to the soil or after leaching in the soil and leachate.
2. Radioactive residues (aged and unaged) in the Fallsington sandy loam soil columns were not measured following the leaching of the columns. Although the study reports 80-104% of the applied radioactivity was recovered in the leachate, soil data confirming the concentration of residues remaining in the soil should have been provided.
3. The purity of the test substance was not provided.

4. The temperature of the soils during the aging in the greenhouse was not specified. It was not stated if the soils were incubated in the light or dark, if they were covered, or if measures were taken to prevent loss of material by volatilization.
5.  $K_d$  values were not reported.
6. Recovery from fortified samples and the limit of detection were not reported.



Table 1. Soil characteristics.

Soil type	Sand	Silt	Clay	Organic matter	pH	CEC meq/100 g	$V_B^a$ inches
Fallsington sandy loam	67	23	10	0.79	6.6	5.22	9.8
Flanagan silt loam	5	64	31	4.02	5.0	23.4	6.8
Muck soil	--	--	--	59.6	6.6	24.3	12.4

<sup>a</sup>  $V_B$  = break-through volume for sodium chloride -  $^{36}\text{Cl}$ ; volume increment of leachate at which the maximum concentration of sodium chloride was measured for 18 inch columns of these soils.

Table 2. Radioactive residues (ppm bromacil) in leachate from columns of sandy loam, silt loam, and muck soils treated with [ $^{14}\text{C}$ ]bromacil or aged bromacil residues at 5.0 lb ai/A.<sup>a</sup>

Leachate (inches)	Fallsington sandy loam		Flanagan silt loam		Muck	
	Unaged bromacil	Aged residues	Unaged bromacil	Aged residues	Unaged bromacil	Aged residues
0-1	ND <sup>b</sup>	ND	ND	ND	ND	ND
1-2	ND	ND	ND	ND	ND	ND
2-3	ND	ND	ND	ND	ND	ND
3-4	ND	ND	ND	ND	ND	ND
4-5	ND	ND	ND	ND	ND	ND
5-6	ND	ND	ND	ND	ND	ND
6-7	0.25	0.15	ND	ND	ND	ND
7-8	1.10	1.25	ND	0.15	ND	0.10
8-9	3.50	3.95	ND	0.15	ND	0.15
9-10	6.45	4.95	ND	0.20	ND	0.15
10-11	5.25	3.50	ND	0.20	ND	0.15
11-12	3.10	2.10	ND	0.20	ND	0.15
12-13	1.75	1.30	ND	0.10	ND	0.10
13-14	1.05	0.80	ND	ND	ND	0.10
14-15	0.65	0.50	ND	ND	ND	0.10
15-16	0.50	0.45	ND	ND	ND	0.10
16-17	0.30	0.30	ND	ND	ND	ND
17-18	0.25	0.25	ND	ND	ND	ND
18-19	0.20	0.20	ND	ND	ND	ND
19-20	0.15	0.15	ND	ND	ND	ND

<sup>a</sup> Table adapted by the reviewer from graph.

<sup>b</sup> Not detected; detection limit not specified.

Table 3. Radioactive residues (% of recovered after leaching) remaining in columns of silt loam and muck soil treated with [ $^{14}\text{C}$ ]bromacil or aged bromacil residues at 5.0 lb ai/A.

Sampling depth (inches) <sup>a</sup>	Flanagan silt loam		Muck	
	Unaged bromacil <sup>b</sup>	Aged residues <sup>c</sup>	Unaged bromacil <sup>d</sup>	Aged residues <sup>e</sup>
0-2	4.73	15.3	40.6	12.6
2-4	8.09	5.2	33.9	14.2
4-6	10.3	6.3	19.2	20.7
6-8	11.8	7.1	4.05	23.9
8-10	22.3	12.6	1.43	23.4
10-12	15.5	18.7	0.12	4.19
12-14	10.8	25.1	0.02	0.39
14-16	14.3	8.6	0.16	0.30
16-18	2.15	1.0	0.28	0.23

<sup>a</sup> Increment measured as inches from the top of the column.

<sup>b</sup> 86% of applied  $^{14}\text{C}$  recovered.

<sup>c</sup> 92% of applied  $^{14}\text{C}$  recovered.

<sup>d</sup> 111% of applied  $^{14}\text{C}$  recovered.

<sup>e</sup> 98% of applied  $^{14}\text{C}$  recovered.

CASE GS0041 BROMACIL STUDY 4 PM

CHEM 012301 Bromacil

BRANCH EFB DISC --

FORMULATION 90 - FORMULATION NOT IDENTIFIED

FICHE/MASTER ID No MRID CONTENT CAT 01  
E.I. du Pont de Nemours and Company. 1984. Bromacil residue analyses soil samples (1966 to 1982). Unpublished study received March 10, 1984. In E.I. du Pont de Nemours and Company Response to Bromacil Registration Standard Environmental Fate. Submitted by E.I. du Pont de Nemours and Company, Wilmington, DE. Accession No. 249679.

SUBST. CLASS = S.

DIRECT RVW TIME = 5 1/2 (MH) START-DATE END DATE

REVIEWED BY: K. Patten  
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SIGNATURE: *K. Patten*

DATE: Jan 18, 1984

APPROVED BY:  
TITLE:  
ORG:  
TEL:

SIGNATURE:

DATE:

CONCLUSION:Field Dissipation - Terrestrial

This study is scientifically invalid because the sampling protocol was inadequate to accurately assess the dissipation of bromacil from soil, and because no methodology was available for critical review to determine if analytical procedures were adequate. In addition, this study would not fulfill EPA Data Requirements for Registering Pesticides (1983) because the soil was not completely characterized, the test substance was not characterized, field test data were not provided, no pretreatment samples were analyzed, and a nonspecific analytical method was used.

### MATERIALS AND METHODS:

Cultivated fields (uncharacterized) of Keyport silt loam soil (soil not further characterized) in Newark, Delaware, were treated with bromacil (Hyvar X, test substance uncharacterized, E.I. du Pont de Nemours and Co.) annually between 1966 and 1981 at 0.5 (1966 to 1969 only), 1.0, 2.0, or 4.0 lb ai/A (Tables 1-3). An untreated field served as the control. Soil samples (0- to 4-, 4- to 8-, 8- to 12-, and 12- to 16-inch depths) were taken from the fields before plowing at intervals between 3 and 12 months following the application of bromacil. During 1971-1975, samples were taken from plowed as well as unplowed fields.

Soil samples were analyzed for bromacil residues (methodology not provided). Recovery of bromacil from 10 soil samples fortified with bromacil at 0.04-5.0 ppm ranged from 80 to 122%. The detection limit was 0.04 ppm.

### REPORTED RESULTS:

Bromacil residues were not detected (<0.04 ppm) in the soil of the control at any sampling interval.

Data from the field treated with bromacil at 0.05 lb ai/A were provided only for the 4, 9, and 12 month intervals following the 1968 application. Bromacil residues were 0.9 ppm at the 0- to 4-inch soil depth 4 months after the application of bromacil, but were not detected at the 4- to 8- or 8- to 12-inch depths. Bromacil residues were not detected at any depth at either the 9 or 12 month sampling intervals.

In the soil treated at 1.0 lb ai/A, bromacil residues were <0.28 ppm in the surface 4 inches and were not detected below the 4- to 8-inch depth at any sampling interval (Table 1). Bromacil residues were <2.3 and <5.4 ppm in the surface 4 inches of soil from the fields treated at 2.0 and 4.0 lb ai/A, respectively. In both the 2.0 and 4.0 lb ai/A treatments, bromacil residues persisted in the soil >12 months after treatment with bromacil.

Bromacil residues in the plowed fields were less concentrated in the 0- to 4-inch soil depth and more concentrated in the 4- to 8-inch depth than the residues were in the unplowed fields.

### DISCUSSION:

1. The sampling schedule was inadequate (1-3 sampling intervals) to accurately establish the pattern of decline of bromacil and patterns of formation and decline of degradates in soil. No pretreatment soil samples were taken, immediate posttreatment samples were not analyzed to confirm bromacil application rates, and the first samples were not taken until 3-12 months after treatment.

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2. No sampling procedures (e.g. number of samples obtained at each sampling interval) or analytical methodology were received with the study; therefore it could not be determined if the methods were adequate to measure bromacil in the soil. Based on the use of the term "bromacil residues" by the researchers, the analytical method did not distinguish between bromacil and its degradates.
3. Field test data, including meteorological data and cultivation practices, were not provided.
4. Complete soil characteristics, such as pH, organic matter content, and CEC, were not provided.
5. The test substance was not characterized.
6. Although the study stated that bromacil was applied annually to the soil, no application dates were reported for 1978, 1979, or 1980. It was therefore not certain if the soil sample taken on 6/18/80 was from a field treated in 1979, or in 1977 (the last previous treatment date reported).
7. Data from the 0.5 lb ai/A treatment were reported only for the 1968 treatment period.
8. The method of storing the samples prior to analysis, and the sampling-to-analysis intervals were not specified.

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Table 1. Bromacil residues (ppm) in unplowed fields of silt loam soil in Newark Delaware, treated annually between 1966 and 1981 with bromacil (Hyvar X) at 1.0, 2.0, or 4.0 lb ai/A.

Application date	Sampling date	Sampling depth (inches)									
		1.0 lb ai/A			2.0 lb ai/A			4.0 lb ai/A			
		0-4	4-8	8-12	0-4	4-8	8-12	0-4	4-8	8-12	12-16
5/16/66	8/29/66	--	--	--	0.44	ND <sup>a</sup>	ND	--	--	--	--
	1/24/67	--	--	--	0.15	--	--	--	--	--	--
	6/14/67	--	--	--	0.15	ND	--	0.44	ND	--	--
6/15/67	11/03/67	--	--	--	0.25	--	--	1.0	0.19	--	--
	2/01/68	--	--	--	0.20	ND	--	--	--	--	--
	5/08/68	--	--	--	0.29	0.08	--	0.92	0.16	--	--
5/10/68	9/12/68	0.17	ND	ND	0.76	0.12	ND	2.5	0.80	0.04	--
	2/26/69	0.28	0.06	ND	1.00	0.12	ND	2.6	0.68	0.12	--
	5/07/69	ND	ND	ND	0.56	ND	ND	1.8	0.44	ND	--
5/22/69	9/12/69	--	--	--	0.96	0.10	ND	3.4	0.80	ND	--
	2/25/70	--	--	--	0.42	0.07	--	3.5	0.78	--	--
	5/05/70	--	--	--	0.41	ND	--	2.1	0.46	ND	--
5/08/70	9/23/70	--	--	--	2.30	0.14	--	5.0	0.64	--	--
	5/03/71	0.10	ND	--	0.46	ND	--	1.5	0.52	ND	--
5/14/71	9/09/71	--	--	--	1.50	0.16	--	5.4	0.76	--	--
	5/08/72	0.20	0.13	--	0.44	0.20	--	2.6	0.80	0.12	--
5/14/72	5/10/73	0.17	ND	--	0.70	0.45	0.07	3.9	1.50	0.07	ND
6/12/73	5/30/74	< 0.04	ND	--	0.40	0.17	0.05	2.9	0.59	0.15	< 0.05
6/05/74	5/30/75	0.07	ND	--	0.45	0.11	ND	2.8	0.66	0.16	0.80
6/10/75	6/17/76	0.19	ND	ND	0.69	0.14	ND	5.4	1.10	0.17	0.08
6/07/76	--	--	--	--	--	--	--	--	--	--	--
6/20/77	6/28/78	ND	ND	ND	0.29	0.06	ND	2.8	0.79	0.30	0.13
	6/18/80	ND	ND	ND	0.50	0.08	ND	2.3	0.54	0.08	ND
7/09/81	6/21/82	ND	ND	ND	0.21	ND	ND	3.0	0.66	0.17	ND

<sup>a</sup> Not detected; detection limit was 0.04 ppm.