

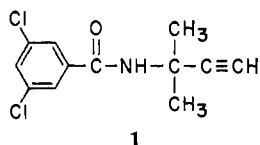
# Relationship of Chemical Structure and Herbicidal Activity in Dimethylpropynylbenzamides

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Relative preemergence herbicidal activity is reported for 40 new compounds related to the recently reported herbicide 3,5-dichloro-*N*-(1,1-dimethyl-2-propynyl)benzamide. Structure/activity studies show that herbicidal activity observed for *N*-saturated alkyl-3,5-dichlorobenzamides is enhanced

by  $\beta,\gamma$ -unsaturation and further potentiated by  $\alpha,\alpha$ -dimethyl groups. In dimethylpropynylbenzamides, chloro groups are the most desirable aromatic substituent and are optimally placed in the 3 and 5 positions. Substitutions at the 4 position are more readily tolerated than at the 2 position.

A recent report from our laboratories (Viste, *et al.*, 1970) described several dimethylpropynylbenzamides that exhibit outstanding activity as selective herbicides. Preeminent among these is 3,5-dichloro-*N*-(1,1-dimethyl-2-propynyl)benzamide (**1**) extensively field tested under the code designation RH-315 and recently assigned the trademark Kerb. Subsequent publications describe the degradation of Kerb in soils (Yih, *et al.*, 1970), and its metabolism in soils and plants (Yih and Swithenbank, 1971a) and in animals (Yih and Swithenbank, 1971b).



Benzamides as herbicides have been reported by Baker and Chupp (1963, 1967), Newallis, *et al.* (1966), and Moffett (1964). More closely related 3,5-dichlorobenzamides bearing simple alkyl substituents on nitrogen have been reported as herbicides by Lemin (1966) and Gialdi, *et al.* (1969). The effectiveness of Kerb, however, relates to a combination of structural elements in the molecule that combine to provide outstanding herbicidal activity (Table I). In this paper we shall discuss the structural parameters that lead to optimum activity in this compound.

## MATERIALS AND METHODS

**Chemical Preparation.** Benzamides of 1,1-dimethyl-2-propynylamine and variously substituted benzoic acids (**1** to **18**) were prepared *via* the Schotten-Baumann reaction, as described by Viste, *et al.* (1970). The benzoic acid precursors for these benzamides are commercially available with the exception of 3,4,5-trichlorobenzoic acid (for **2**) and 2,3,5-trichlorobenzoic acid (for **9**), whose syntheses are reported by Huntress (1948), and 3,5-difluorobenzoic acid (for **14**), prepared by Roe and Little (1955). The amine 1,1-dimethyl-

2-propynylamine is available as described by Hennion and Teach (1953).

The other benzamides (**19** to **38**) were prepared in similar fashion from 3,5-dichlorobenzoyl chloride and the appropriate amine. The amines used to prepare **19** to **29** are commercially available. The amines 1,1-dimethyl-2-propenylamine (for **30**), and *N*-methyl-1,1-dimethyl-2-propynylamine (for **38**) are reported by Hennion and DiGiovanna (1965), 1,1-diethyl-2-propynylamine (for **31**) by Hennion and Teach (1953), and 1,1-dimethyl-2-butenylamine (for **32**) by Dillard and Easton (1966). Chemical reduction of 1,1-dimethyl-2-butenylamine using sodium and liquid ammonia (Hennion and DiGiovanna, 1965) gave 1,1-dimethyl-*trans*-2-butenylamine (for **34**), and catalytic reduction using 5% palladium on barium carbonate poisoned with quinoline (Augustine, 1965) yielded 1,1-dimethyl-*cis*-2-butenylamine (for **35**). The amine 2-amino-2-cyanopropane (for **36**) is described by Jacobson (1946). The synthesis of **37** is described by Yih and Swithenbank (1971a).

The Schiff base analog (**39**) of Kerb was prepared from 3,5-dichlorobenzaldehyde and dimethylpropynylamine by standard procedures (Wagner and Zook, 1953). The ester analog (**40**) of Kerb was prepared from the alcohol by standard Fischer esterification procedure. The sulfonamide analog (**41**) of Kerb was prepared from 3,5-dichlorobenzenesulfonyl chloride and dimethylpropynylamine under Schotten-Baumann conditions. The sulfonyl chloride was prepared from 3,5-dichlorothiophenol (Metivier, 1957) by oxidative chlorination using the method of Muth (1955).

Proof of structure for all compounds was established by spectroscopic methods (ir, nmr, uv) and mass spectrometry. Purity was determined by glc, tlc, and elemental analyses. In Tables II to IV melting point data are uncorrected.

**Biological Testing.** Testing for herbicidal activity was carried out using routine greenhouse procedures. Seeds were planted in flats in soil composed of  $\frac{2}{3}$  silt loam top soil and  $\frac{1}{3}$  peat moss. The compounds dissolved in acetone were sprayed on and thoroughly mixed with the top  $\frac{3}{4}$  in. of soil at rates of 4 and 8 lb per acre. The flats were sub-irrigated in the greenhouse. Ten monocotyledonous species employed were: wild oat (*Avena fatua*), Sudangrass (*Sorghum*

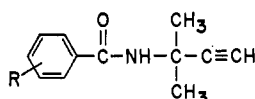
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**Table I. Herbicidal Response<sup>a</sup> of Monocotyledonous and Dicotyledonous Species to Varying Concentrations of Kerb**

		Kerb lb/Acre				
		0.5	1	2	4	8
Wild oat	( <i>Avena fatua</i> )	8	9	10	10	10
Sudangrass	( <i>Sorghum sudanensis</i> )	1	3	8	9	10
Rice	( <i>Oryza sativa</i> )	0	0	2	7	10
Millet	( <i>Setaria italica</i> )	4	6	10	10	10
Johnsongrass	( <i>Sorghum halepense</i> )	8	9	10	10	10
Ryegrass	( <i>Lolium perenne</i> )	10	10	10	10	10
Foxtail	( <i>Setaria faberii</i> )	10	10	10	10	10
Wheat	( <i>Triticum aestivum</i> )	2	3	6	8	9
Corn	( <i>Zea mays</i> )	4	9	9	10	10
Barnyardgrass	( <i>Echinochloa crusgalli</i> )	5	10	10	10	10
Average		5.2	7.0	8.5	9.4	9.8
Soybeans	( <i>Glycine max</i> )	0	0	0	7	8
Pigweed	( <i>Amaranthus retroflexus</i> )	5	7	10	10	10
Flax	( <i>Linum usitatissimum</i> )	6	9	10	10	10
Cotton	( <i>Gossypium hirsutum</i> )	0	0	0	0	0
Peas	( <i>Pisum sativum</i> )	0	0	0	0	0
Curly dock	( <i>Rumex crispus</i> )	9	10	10	10	10
Carrot	( <i>Daucus carota</i> )	0	0	10	10	10
Velvetleaf	( <i>Abutilon theophrasti</i> )	0	0	4	8	9
Alfalfa	( <i>Medicago sativa</i> )	0	0	0	7	9
Jute	( <i>Corchorus spp.</i> )	...	4	4	10	10
Average		2.0	3.0	4.8	7.2	7.6

<sup>a</sup> 0 = no effect; 10 = complete kill.

**Table II. Physical and Biological Properties of Benzamides of 1,1-Dimethylpropynylamine**



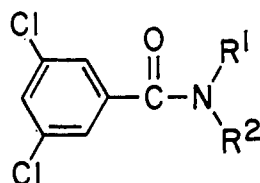
	R	Relative Herbicidal Activity at		mp	Molecular Formula	Analysis	
		8 lb/acre	4 lb/acre			Calcd, %	Found, %
1	3,5-Cl <sub>2</sub>	9	8	155-157	C <sub>12</sub> H <sub>11</sub> Cl <sub>2</sub> NO	C, 56.27 H, 4.33 N, 5.47	56.42 4.39 5.46
2	3,4,5-Cl <sub>3</sub>	8	6	151-154	C <sub>12</sub> H <sub>10</sub> Cl <sub>3</sub> NO	C, 49.61 H, 3.48 N, 4.82	49.82 3.49 4.78
3	3-Cl	6	4	110-111	C <sub>12</sub> H <sub>12</sub> ClNO	C, 65.10 H, 5.47 N, 6.33	65.37 5.47 6.19
4	2,3,6-Cl <sub>3</sub>	5	3	148-153	C <sub>12</sub> H <sub>10</sub> Cl <sub>3</sub> NO	C, 49.61 H, 3.48 N, 4.82	49.40 3.44 4.72
5	3,4-Cl <sub>2</sub>	5	2	108-110	C <sub>12</sub> H <sub>11</sub> Cl <sub>2</sub> NO	C, 56.27 H, 4.33 N, 5.47	56.22 4.33 5.51
6	4-Cl	4	2	141-143	C <sub>12</sub> H <sub>12</sub> ClNO	C, 65.10 H, 5.47 N, 6.33	65.02 5.47 6.23
7	2,5-Cl <sub>2</sub>	3	2	89-92	C <sub>12</sub> H <sub>11</sub> Cl <sub>2</sub> NO	C, 56.27 H, 4.33 N, 5.47	56.19 4.48 5.33
8	2,6-Cl <sub>2</sub>	3	1	144-146	C <sub>12</sub> H <sub>11</sub> Cl <sub>2</sub> NO	C, 56.27 H, 4.33 N, 5.47	56.31 4.21 5.26
9	2,3,5-Cl <sub>3</sub>	2	1	99-101	C <sub>12</sub> H <sub>10</sub> Cl <sub>3</sub> NO	C, 49.61 H, 3.48 N, 4.82	49.43 3.29 4.78
10	Unsub.	1	1	152-153	C <sub>12</sub> H <sub>13</sub> NO	C, 76.97 H, 7.00 N, 7.48	77.06 6.78 7.43
11	2-Cl	1	0	94-96	C <sub>12</sub> H <sub>12</sub> ClNO	C, 65.10 H, 5.47 N, 6.33	65.06 5.37 6.23
12	2,4-Cl <sub>2</sub>	0	0	108-109	C <sub>12</sub> H <sub>11</sub> Cl <sub>2</sub> NO	C, 56.27 H, 4.33 N, 5.47	56.07 4.27 5.31

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Table II. (Continued)

	R	Relative Herbicidal Activity at		mp	Molecular Formula	Analysis	
		8 lb/acre	4 lb/acre			Calcd, %	Found, %
13	3,5-Br <sub>2</sub>	7	6	162-164	C <sub>12</sub> H <sub>11</sub> Br <sub>2</sub> NO	C, 41.76 H, 3.22 N, 4.05	41.22 3.27 4.22
14	3,5-F <sub>2</sub>	6	5	74-78	C <sub>12</sub> H <sub>11</sub> F <sub>2</sub> NO	C, 64.59 H, 4.97 N, 6.28	64.45 4.95 6.35
15	3,5-(CH <sub>3</sub> ) <sub>2</sub>	6	5	126-127	C <sub>14</sub> H <sub>17</sub> NO	C, 78.12 H, 7.97 N, 6.51	78.15 7.68 6.63
16	3,5-(CF <sub>3</sub> ) <sub>2</sub>	6	4	134-136	C <sub>14</sub> H <sub>11</sub> F <sub>6</sub> NO	C, 52.00 H, 3.43 N, 4.33	51.97 3.66 4.35
17	3,5-(OCH <sub>3</sub> ) <sub>2</sub>	1	0	101.5-102.5	C <sub>14</sub> H <sub>17</sub> NO <sub>3</sub>	C, 67.99 H, 6.93 N, 5.66	68.21 7.06 5.66
18	3,5-(NO <sub>2</sub> ) <sub>2</sub>	1	0	180-180.5	C <sub>12</sub> H <sub>11</sub> N <sub>3</sub> O <sub>5</sub>	C, 51.99 H, 4.00 N, 15.16	51.97 4.04 14.89

Table III. Physical and Biological Properties of N-Substituted 3,5-Dichlorobenzamides

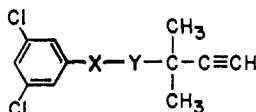


	R <sup>1</sup>	R <sup>2</sup>	Relative Herbicidal Activity at		mp	Molecular Formula	Analysis	
			8 lb/acre	4 lb/acre			Calcd, %	Found, %
19	H	H	0	0	160-161	C <sub>7</sub> H <sub>5</sub> Cl <sub>2</sub> NO	C, 44.24 H, 2.65 N, 7.37	44.25 2.56 7.40
20	H	CH <sub>3</sub>	0	1	175-176	C <sub>8</sub> H <sub>7</sub> Cl <sub>2</sub> NO	C, 47.09 H, 3.46 N, 6.86	47.09 3.49 6.82
21	H	C <sub>2</sub> H <sub>5</sub>	4	1	103-106	C <sub>9</sub> H <sub>9</sub> Cl <sub>2</sub> NO	C, 49.54 H, 4.16 N, 6.42	49.71 4.58 6.20
22	H	<i>n</i> -C <sub>3</sub> H <sub>7</sub>	3	1	102.5-103	C <sub>10</sub> H <sub>11</sub> Cl <sub>2</sub> NO	C, 51.75 H, 4.78 N, 6.03	51.75 4.80 6.01
23	H	<i>iso</i> -C <sub>3</sub> H <sub>7</sub>	5	4	157.5-158.5	C <sub>10</sub> H <sub>11</sub> Cl <sub>2</sub> NO	C, 51.75 H, 4.77 N, 6.03	51.70 4.73 5.83
24	H	<i>tert</i> -C <sub>4</sub> H <sub>9</sub>	...	4	147-152	C <sub>11</sub> H <sub>13</sub> Cl <sub>2</sub> NO	C, 53.66 H, 5.33 N, 5.69	54.00 5.49 5.66
25	H	C(CH <sub>3</sub> ) <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub>	...	3	102-103	C <sub>12</sub> H <sub>15</sub> Cl <sub>2</sub> NO	C, 55.35 H, 5.72 N, 5.41	55.32 5.87 5.35
26	CH <sub>3</sub>	CH <sub>3</sub>	2	1	54-55.5	C <sub>9</sub> H <sub>9</sub> Cl <sub>2</sub> NO	C, 49.54 H, 4.16 N, 6.42	49.55 4.12 6.37
27	<i>n</i> -C <sub>3</sub> H <sub>7</sub>	<i>n</i> -C <sub>3</sub> H <sub>7</sub>	1	1	34-35	C <sub>13</sub> H <sub>17</sub> Cl <sub>2</sub> NO	C, 56.95 H, 6.25 N, 5.10	57.07 6.29 5.05
28	H	CH <sub>2</sub> CH=CH <sub>2</sub>	6	4	95-97	C <sub>10</sub> H <sub>9</sub> Cl <sub>2</sub> NO	C, 52.20 H, 3.95 N, 6.08	52.19 3.99 6.10
29	H	CH <sub>2</sub> C≡CH	7	5	144-145	C <sub>10</sub> H <sub>7</sub> Cl <sub>2</sub> NO	C, 52.70 H, 3.09 N, 6.14	52.51 3.04 6.12
30	H	C(CH <sub>3</sub> ) <sub>2</sub> CH=CH <sub>2</sub>	7	6	114-116	C <sub>12</sub> H <sub>13</sub> Cl <sub>2</sub> NO	C, 55.80 H, 5.08 N, 5.44	55.92 5.23 5.42
31	H	C(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub> C≡CH	...	0	98-100	C <sub>14</sub> H <sub>15</sub> Cl <sub>2</sub> NO	C, 59.17 H, 5.32 N, 4.93	59.52 5.29 4.95

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Table III. (Continued)

	R <sup>1</sup>	R <sup>2</sup>	Relative Herbicidal Activity at		mp	Molecular Formula	Analysis	
			8 lb/acre	4 lb/acre			Calcd, %	Found, %
32	H	C(CH <sub>3</sub> ) <sub>2</sub> C≡CCH <sub>3</sub>	7	6	126-127.5	C <sub>13</sub> H <sub>13</sub> Cl <sub>2</sub> NO	C, 57.80 H, 4.85 N, 5.18	57.58 4.94 5.06
33	H	C(CH <sub>3</sub> ) <sub>2</sub> C≡C- <i>n</i> -C <sub>4</sub> H <sub>9</sub>	4	3	92.5-93.5	C <sub>16</sub> H <sub>19</sub> Cl <sub>2</sub> NO	C, 61.55 H, 6.13 N, 4.49	61.61 6.10 4.31
34	H	C(CH <sub>3</sub> ) <sub>2</sub> CH <sup>t</sup> =CHCH <sub>3</sub>	...	4	75-76.5	C <sub>13</sub> H <sub>13</sub> Cl <sub>2</sub> NO	C, 57.35 H, 5.55 N, 5.15	57.10 5.38 5.08
35	H	C(CH <sub>3</sub> ) <sub>2</sub> CH <sup>c</sup> =CHCH <sub>3</sub>	4	0	112-114	C <sub>13</sub> H <sub>13</sub> Cl <sub>2</sub> NO	C, 57.35 H, 5.55 N, 5.15	57.10 5.49 5.01
36	H	C(CH <sub>3</sub> ) <sub>2</sub> C≡N	...	3	138-140	C <sub>11</sub> H <sub>10</sub> Cl <sub>2</sub> N <sub>2</sub> O	C, 51.38 H, 3.92 N, 10.90	51.31 4.09 10.60
37	H	C(CH <sub>3</sub> ) <sub>2</sub> COCH <sub>3</sub>	2	1	141-142	C <sub>12</sub> H <sub>14</sub> Cl <sub>2</sub> NO <sub>2</sub>	C, 52.59 H, 4.78 N, 5.11	52.58 4.68 5.15

Table IV. Physical and Biological Properties of Miscellaneous Compounds Related to *N*-(1,1-Dimethylpropynyl)-3,5-dichlorobenzamide

	X	Y	Relative Herbicidal Activity at		mp	Molecular Formula	Analysis	
			8 lb/acre	4 lb/acre			Calcd, %	Found, %
38	CO	NCH <sub>3</sub>	...	3	72-75	C <sub>13</sub> H <sub>13</sub> Cl <sub>2</sub> NO	C, 57.80 H, 4.85 N, 5.18	57.15 4.80 5.16
39	CH=	N	...	1	38-40	C <sub>12</sub> H <sub>11</sub> Cl <sub>2</sub> N	C, 60.02 H, 4.62 N, 5.83	59.62 4.63 5.57
40	CO	O	2	1	93-95	C <sub>12</sub> H <sub>10</sub> Cl <sub>2</sub> O <sub>2</sub>	C, 56.05 H, 3.92 Cl, 27.59	56.03 3.86 27.15
41	SO <sub>2</sub>	NH	...	0	99.5-101.5	C <sub>11</sub> H <sub>11</sub> Cl <sub>2</sub> NO <sub>2</sub> S	C, 45.22 H, 3.80 N, 4.79	45.15 4.04 4.35

*sudanensis*), rice (*Oryza sativa*), millet (*Setaria italica*), Johnsongrass (*Sorghum halepense*), ryegrass (*Lolium perenne*), foxtail (*Setaria faberii*), wheat (*Triticum aestivum*), corn (*Zea mays*), and barnyardgrass (*Echinochloa crusgalli*). Ten dicotyledonous species employed were: soybean (*Glycine max*), pigweed (*Amaranthus retroflexus*), flax (*Linum usitatissimum*), cotton (*Gossypium hirsutum*), peas (*Pisum sativum*), curly dock (*Rumex crispus*), carrot (*Daucus carota*), velvetleaf (*Abutilon theophrasti*), alfalfa (*Medicago sativa*), and jute (*Corchorus* spp.).

After 14 days the treatments were evaluated on a 0 (no effect) to 10 (plants dead) scale relative to an untreated check. Twenty individual values were averaged to obtain an overall value which was adjusted to the nearest whole number.

Herbicidal data for two application rates are included to offset the nonlinearity of the herbicide scale and to facilitate comparison of activities (1 at 4 lb per acre is as active as 2 at 8 lb per acre).

#### RESULTS AND DISCUSSION

Three structural elements contribute to the excellent herbicidal activity of 3,5-dichloro-*N*-(1,1-dimethylpropynyl)benz-

amide: the 3,5-dichlorophenyl moiety; the dimethylpropynyl group; and the carbamyl function.

A wide range of herbicidal activity is observed for ring-chlorinated derivatives of *N*-(1,1-dimethylpropynyl)benzamide (Table II). Most substitution patterns are activating relative to the unsubstituted molecule, and optimum activity is attained with 3,5-substitution. The presence of a 4-substituent is tolerated more than a 2-substituent. One exception to this generalization is that amides derived from highly active parent acids, e.g., 2,3,6-trichlorobenzoic acid (4), also tend to be highly active. Bromo, fluoro, methyl, and trifluoromethyl substituents placed at the optimal 3 and 5 positions of the aromatic ring (13 to 16) produce good to excellent herbicidal activity but do not exceed chloro in potentiating herbicidal activity. Nitro and methoxy substituents similarly positioned are inactive.

The optimum substitution pattern on the phenyl ring contrasts with that of most other classes of herbicides. For example, in the chloro-substituted benzoic acids, the substitutional pattern that results in decreasing order of activity is 2,3,6 > 2,5 > 2,3 > 2,6 > all other mono- and disubstituted analogs (Smith, 1961). The requirements in benzamides

are quite different: 3,5 > 3,4,5 > 3 > 4 > 2,6. This difference is accentuated by the fact that benzamides are predominantly active on grasses (Table I), while the benzoic acids are active on dicotyledonous species.

Certain *N*-alkyl- (Lemin, 1966), and *N,N*-dialkyl-3,5-dichlorobenzamides (Gialdi, *et al.*, 1969) are reported in the patent literature. Although the parent 3,5-dichlorobenzamide is inactive (Table III, 19), introduction of a saturated alkyl group on to nitrogen (20 to 27) confers moderate herbicidal activity. Insufficient dialkyl analogs are available to define a complete trend but, in general, the dialkyl analogs appear to be less active than their monoalkyl counterparts. Introduction of unsaturation between the second and third carbon atoms of a propyl group significantly increases herbicidal activity, the increase being proportional to the degree of unsaturation introduced (22, 28, and 29). This high level of activity is further potentiated by substituting two methyl groups on the first carbon atom of the chain (29 and 1; 30 and 28), but lost completely by substituting ethyl groups (31). Substituting alkyls on the third carbon of the propynyl group also affects activity. Progressive increase in the size of these alkyl groups results in gradual loss of herbicidal activity (32 and 33). In olefinic analogs the terminal methyl group must be *trans* (34 and 35). Groups that contain other than carbon to carbon unsaturation are inactive (36 and 37).

The 3,5-dichlorophenyl ring is connected to the dimethyl-propynyl moiety most effectively *via* the carbamyl group (Table IV). Methylating the carbamyl nitrogen (Table IV, 38) decreases activity relative to the preferred material (1), but still produces greater activity than the Schiff base analog, the ester, or the sulfonamide (39, 40, and 41).

While it is clear that the amide function is indispensable to the activity of the benzamides, the inactivity of *N*-(1,1-dimethyl-2-propynyl)benzamide (10) and 3,5-dichloro-*N*-methylbenzamide (20) show that other structural elements in the molecule are important in determining herbicidal activity. One might speculate that the unsaturated center and the aromatic ring are held in a particular conformation on an

enzyme so that the carbamyl functionality is maintained in an optimum spatial configuration for interference with a specific biological process.

#### ACKNOWLEDGMENT

The authors thank A. J. Crovetti and B. W. Horrom of Abbott Laboratories for substantial synthetic support, and H. O. Bayer, R. S. Cook, S. N. Lewis, T. A. McLaughlin, D. H. McRae, and R. Y. Yih for contributions to this work.

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Received for review October 19, 1970. Accepted January 12, 1971