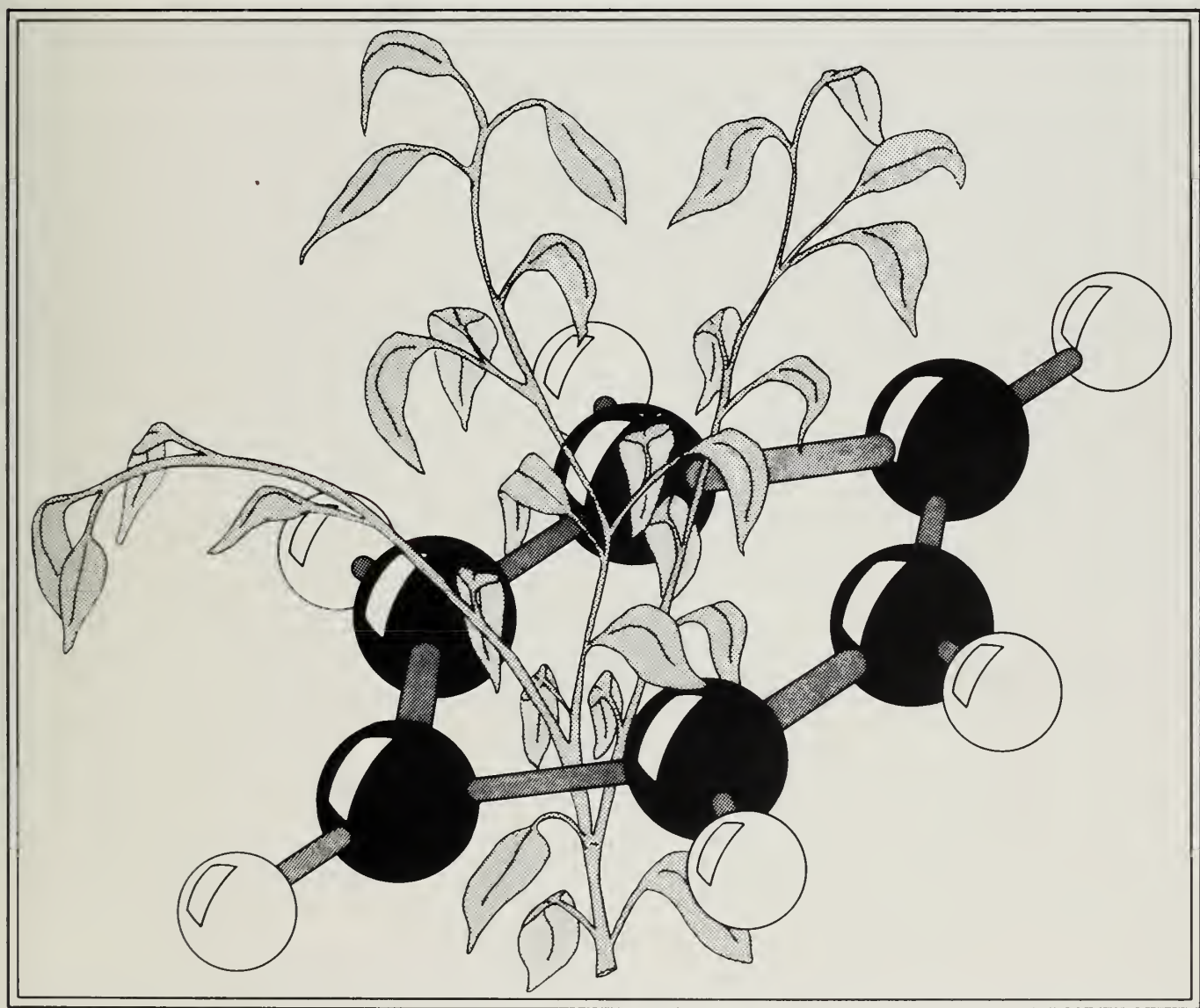


# SCREENING OF SELECTED FLUOROAROMATIC COMPOUNDS FOR USE AS AGRICHEMICALS, I

R. H. Shiley, D. R. Dickerson, Claus Grunwald, and J. R. Willard



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## ABSTRACT

A series of 214 fluoroaromatic compounds were screened for biological activity for use as fungicides and herbicides. Fungicidal activity was tested in the following four categories: foliar, curative, systemic, and soil. In the foliar fungicide evaluation, three of the subject compounds showed slight to moderate activity against leaf spot of sugarbeet and bean rust. As curative agents, six of the compounds showed good activity when compared to Benlate in controlling leaf spot of sugarbeet, bean powdery mildew, and cucumber powdery mildew. Systemically, one compound showed only slight control of tomato bacterial spot. In the soil fungicide evaluation, six of the test compounds showed moderate to good activity against fusarium root rot of bean and cucumber damping-off when compared to Benlate. Of the eleven compounds which exhibited pre- or postemergence herbicidal activity, 2,4,5-trifluorophenoxyacetic acid was the most effective. It is comparable in activity to its chloro analog. Twenty-five field crops and weeds were used in the herbicide evaluation. The testing of the gross effects on the growth of *Arabidopsis thaliana* and the ethylene evolution test were also used in the screening program.

## INTRODUCTION

Forty years of research in organic fluorine chemistry in the laboratories of the Illinois State Geological Survey has made available for research purposes a large number of aromatic fluorine compounds. The Illinois State Geological Survey traditionally has had an interest in plant growth regulators (Finger et al., 1959). As early as 1951, the Botany and Plant Pathology Section of the Illinois State Natural History Survey was testing fluorinated p-benzoquinones for their fungicidal activities (Tehon, 1951). In 1954, Forsberg tested 1-fluoro-3-methyl-4,6-dinitrobenzene and 1,3-difluoro-4,6-dinitrobenzene for protection against Fusarium rot in Gladiolus corms. These compounds were very effective.

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\*deceased

In 1974, fifty-one fluoroaromatic compounds were tested on several fungi which attack Gladiolus corms, and Poinsettia plants and cause dutch elm disease (Shiley et al., 1975). Seven of the compounds tested had a fungicidal activity equal to or greater than that of mercuric chloride on one or more of the organisms tested.

As a result of these findings and the Geological Survey's recent publication *Aromatic Fluorine Chemistry at the Illinois State Geological Survey, Research Notes 1934-1976* (Shiley et al., 1978), coupled with recent advances with the potassium fluoride-halogen exchange reaction on polychloro-benzenes (Forsberg, 1954; Shiley, Dickerson and Finger, 1972/73; Shiley, Dickerson, and Finger, 1978; Shiley et al., 1975; Tehon, 1951) which allows for increased ease of synthesis of these compounds, the desire to test the subject compounds as agrichemicals has been rekindled. Therefore, a new program designed to test 214 aromatic fluorine compounds under industrial and field conditions was undertaken in a cooperative study between the Agricultural Chemical Division of the FMC Corporation (Middleport, NY) and the Illinois State Geological Survey Geochemical Section and under the guidance of the Illinois State Natural History Survey, Botany and Plant Pathology Department. This work will be expanded in the future to include the testing of some of these compounds as insecticides and nematicides.

## EXPERIMENTAL PROCEDURES

The synthetic routes and physical constants for the compounds used in this screening program are reported in Illinois State Geological Survey Circular 501 (Shiley et al., 1978). The methods employed in this screening program are the basic testing procedures used by the FMC Corporation Agricultural Chemical Division. The various testing procedures are as follows.

### Fungicides

The organisms employed in this biological screening program are described in table 1.

The biological tests used in the screening program are as follows:

1. **Foliar disease protectant.** Test results are reported as the percentage of control of infection due to treatment of the host plant with test chemical one day prior to inoculation with the fungal organism.
2. **Curative.** Test results are reported as the percentage of control of infection due to treatment of the host plant with test chemical one day after inoculation with the fungal organism.
3. **Systemic.** Test results are reported as the percentage of control of infection of the host plant due to treatment of the soil with the test chemical one day prior to inoculation of the plant with the disease organism.
4. **Soil fungicide.**
  - a. **Fusarium root rot.** Test results are reported as the percentage of control of infection of the plant due to treatment of the soil with test chemical one day prior to inoculation of the soil with fungal organism.
  - b. **Cucumber damping-off.** Test results are reported as an efficacy index (EI) which compares the effect of the test chemical to that of a standard (Captan). EI greater than 100 indicates that the test chemical is more effective than the standard. Calculation of the efficacy index is shown below. In this test, cucumber seeds are treated with the test chemical, planted in soil, and subsequently inoculated with the disease complex.

$$EI = \frac{\text{Tot. stand}_{(\text{test chem.})} - \text{Tot. stand}_{(\text{untreated})}}{\text{Tot. stand}_{(\text{Captan stand.})} - \text{Tot. stand}_{(\text{untreated})}} \times 100$$

TABLE 1. Organisms causing diseases.

Disease	Fungus	Host plant	Cultivar
Late blight	<i>Phytophthora infestans</i> (Mont.) d By.	tomato ( <i>Lycopersicon esculentum</i> L.)	Heinz 1350
Bean rust	<i>Uromyces phaseoli</i> (Pers.) Wint.	bean ( <i>Phaseolus vulgaris</i> L.)	Pinto
Rice blast	<i>Pyricularia oryzae</i> Cav.	rice ( <i>Oryza sativa</i> L.)	Nova 66
Leaf spot	<i>Cercospora beticola</i> Sacc.	sugarbeet ( <i>Beta vulgaris</i> L.)	A436-67R
Bean powdery mildew	<i>Erysiphe polygoni</i> DC.	bean ( <i>Phaseolus vulgaris</i> L.)	Bountiful
Bacterial spot	<i>Xanthomonas vesicatoria</i> (Doidge) Dows.	tomato ( <i>Lycopersicon esculentum</i> L.)	Heinz 1350
Root rot	<i>Fusarium solani</i> (Mart.) Appel and Wr. f. <i>phaseoli</i> (Burk.) Synyd. and Hans.	bean ( <i>Phaseolus vulgaris</i> L.)	Pinto
Damping-off	<i>Pythium ultimum</i> Trow <i>Rhizoctonia solani</i> Keuhn	cucumber ( <i>Cucumis sativus</i> L.)	Straight Eight

## Herbicides

The plant species employed in the preliminary biological screening program are as follows:

Common name	Scientific name
Lima bean	<i>Phaseolus lunatus</i> L.
Sweet corn	<i>Zea mays</i> L.
Wild oats	<i>Avena fatua</i> L.
Lettuce	<i>Lactuca sativa</i> L.
Mustard	<i>Brassica juncea</i> (L.) Czerniak
Crabgrass	<i>Digitaria sanguinalis</i> (L.) Scop.

The following biological tests are used in the screening program:

5. **Preemergence.** In the preemergence test, the test chemical is sprayed onto the seeds at planting. Test results are recorded after 10 to 14 days. Test results are reported as a qualitative measure of the vigor (V) of standing plants/percentage of plants killed (K). Vigor ratings are assigned as follows:

### Vigor ratings

- 1 - Severe injury. Plants are not expected to recover.
- 2 - Moderate to severe injury. Severe injury to surviving plants.
- 3 - Moderate injury. Plants are expected to recover.
- 4 - Slight injury. Plants have recovered or are expected to fully recover.
- 5 - No effect.

The plants are also examined for any biological growth responses. These are denoted by the letter R. The growth effects screened for are as follows:

#### Biological response (R)

- 1 - Necrosis
- 2 - Stunting
- 3 - Desiccation
- 4 - Axillary growth stimulation
- 5 - Nastic responses
- 6 - Necrotic spots
- 7 - Growth stimulation
- 8 - Defoliation
- 9 - Chlorosis
- 10 - Intumescence

For example a 20/3/2 rating indicates that 20 percent of the plants were killed by the test chemical treatment, there was moderate injury to surviving plants, and the surviving plants were stunted in growth.

6. **Postemergence.** Test results are reported as in the preemergence test. In the postemergence test, 10 to 14 day old plants are sprayed with the test chemical. Test results are recorded after an additional 10 to 14 days. The same standards for vigor ratings (V) and the biological growth responses (R) are used as those for the preemergence tests.

7. *Arabidopsis thaliana* (L.) Heynh Assay. *Arabidopsis* seed is placed in a culture tube containing a solidified mixture of test chemical and agar-nutrient culture medium. The culture tubes are placed in a growth chamber and grown under controlled environmental conditions for the duration of the plant life cycle (approximately 35 days). The regulatory effects noted are: seed germination, root elongation, root geotropism, number of rosette leaves, fresh weight, abnormal leaf morphology, time to bolting, internode length, days to flowering, abnormal flower morphology, and fruit weight.

The application rates were 0.01, 0.1, 1.0, 10, and 100 ppm.

8. **Tomato pedicel test.** The pedicel with abscission layer is excised from the flower and set on a filter paper in a petri dish. A solution of the test chemical in acetone is applied to the filter paper. The test is then conducted in a controlled environment chamber, and the inhibition or acceleration of abscission is noted. The number of abscised pedicels is recorded 1, 2, 4, 5, and 7 days following the treatment.

The application rates were 0.1, 1.0, 10, 100, and 1000 ppm. Each treatment rate was replicated twice with 10 pedicels. Water was used as a control.

9. **Ethylene evolution test.** The primary leaf abscission zones including the petiolar base abscission layers are excised from 2 to 3 week old lima bean plants. One mL of test solution is added to a vial containing the test tissue and 0.1 mM of indole acetic acid. The vials are stoppered and placed in a controlled environment chamber. Ethylene evolution is analyzed by gas liquid chromatography 24 hours following treatment. The regulatory effects noted are the induction of ethylene evolution in plant tissue, or the release of ethylene by degradation of the applied chemical. The data is reported as ethylene/mg of tissue.

The rates of application were 0.01, 0.1, 1.0, 10, and 100 ppm. Cycloheximide and Ethephon were used as controls.

## RESULTS AND DISCUSSION

The fungicide testing program (table 2) involved four different methods of treatment: foliar, curative, systemic, and soil fungicide.

In the foliar fungicide evaluation, 2,6-difluoro-3,5-dinitrochlorobenzene (1)\* and 2-fluorobenzoic acid hydrazide (2) showed only slight activity in the control of leaf spot of sugarbeet. 2,2'-Difluoro-5,5'-dihydroxydiphenyl sulfide (3), however, demonstrated moderate activity against bean rust as compared to Daconil 75 WP.

When 2,6-difluoro-3,5-dinitrochlorobenzene (1) and 2-fluorobenzoic acid hydrazide (2) were tested as curative agents against leaf spot of sugarbeet, and against bean and cucumber powdery mildew, moderate to good activity was demonstrated as compared to the test standard Benlate. 2,2'-Difluoro-5,5'-dihydroxydiphenyl sulfide demonstrated good activity at the 450 ppm level against bean powdery mildew, but the activity fell off rapidly at the lower rates.

None of the tested compounds demonstrated any systemic activity on tomato bacterial spot except for 2-trifluoromethyl-4-fluoroaniline (4), and this was only slight.

Activity comparable to Benlate was demonstrated by methyl 2-fluoro-6-hydroxybenzoate (7), and 2,4-dinitro-5-methylfluorobenzene (8) in the control of root rot of bean. Three other compounds including 2-trifluoromethyl-4-fluoroaniline (4) were effective at higher rates of application.

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\*Refers to compound number listed in Appendix A.

TABLE 2. Fungicide evaluation.

Compound number	Biological test and chemical used	Percentage of disease control at indicated rate (ppm)			
		16.7	50	150	450
<b>Foliar protectant<sup>a</sup></b>					
<u>Leaf spot of sugar beet</u>					
1	2,6-Difluoro-3,5-dinitrochlorobenzene	-	0	4	13
2	2-Fluorobenzoic acid hydrazide	-	0	8	3
	Benlate 50 WP <sup>b</sup>	-	98	100	100
<u>Bean rust</u>					
3	2,2'-Difluoro-5,5'-dihydroxydiphenyl sulfide	-	0	35	71
	Daconil 75 WP <sup>b</sup>	-	80	100	100
<b>Curative<sup>c</sup></b>					
<u>Leaf spot of sugar beet</u>					
1	2,6-Difluoro-3,5-dinitrochlorobenzene	21	48	85	98
2	2-Fluorobenzoic acid hydrazide	10	71	87	94
	Benlate 50 WP <sup>b</sup>	48	92	94	98
<u>Bean powdery mildew</u>					
1	2,6-Difluoro-3,5-dinitrochlorobenzene	76	78	85	97
2	2-Fluorobenzoic acid hydrazide	71	87	87	93
3	2,2'-Difluoro-5,5'-dihydroxydiphenyl sulfide	-	0	35	95
	Benlate 50 WP <sup>b</sup>	95	100	100	100
<u>Cucumber powdery mildew</u>					
1	2,6-Difluoro-3,5-dinitrochlorobenzene	-	0	8	100
	Benlate 50 WP <sup>b</sup>	95	100	100	100
Rate (mg/pot)					
		0.4	1.3	3.8	11.3
<b>Systemic<sup>d</sup></b>					
<u>Tomato bacterial spot</u>					
4	2-Trifluoromethyl-4-fluoroaniline	-	0	4	6
<b>Soil fungicide</b>					
<u>Fusarium root rot of bean<sup>e</sup></u>					
5	N(2-Fluorophenyl)glycine hydrazide	-	-	0	89
6	4-Fluoro-3-nitrobenzoic acid	-	-	0	56
7	Methyl 2-fluoro-6-hydroxybenzoate	19	50	71	71
4	2-Trifluoromethyl-4-fluoroaniline	-	0	40	92
8	2,4-Dinitro-5-methylfluorobenzene	4	36	50	61
	Benlate 50 WP <sup>b</sup>	15	38	63	70
Efficacy index at indicated rate (mg/pot)					
			1.3	3.8	11.3
<u>Cucumber damping off<sup>f</sup></u>					
9	2-Nitro-3-trifluoromethylphenol		0	63	77
	Captan <sup>b</sup>		100	100	100

<sup>a</sup> Testing procedure number 1<sup>b</sup> Standard<sup>c</sup> Testing procedure number 2<sup>d</sup> Testing procedure number 3<sup>e</sup> Testing procedure number 4a<sup>f</sup> Testing procedure number 4b



When compared to Captan, 2-nitro-3-trifluoromethylphenol (9) showed moderate activity against cucumber damping-off.

Three other fungicidal screening tests were used in this study: the foliar and systemic tomato late blight and the curative rice blast. Little or no fungicidal activity was found in any of these three areas.

In general, the compounds evaluated in the fungicide screening program did not exhibit enough control to warrant continued testing.

The pre- and postemergence herbicide testing results can be found in table 3. The test compounds were subjected to a preliminary screening at the rate of 8.96 kilograms/hectare. Six plant species (lima bean, corn, wild oats, lettuce, mustard, and crabgrass) were used in the screening. If the compounds showed some activity, purple nutsedge was added to the list and they were tested at decreasing application rates from 8.96 to 0.56 kilograms/hectare. In some cases, as many as twenty-five field crops and weeds were used. 2,4,5-Trifluorophenoxyacetic acid (18) was extensively studied at decreasing rates from 8.96 to 0.07 kilograms/hectare in both the pre- and postemergence evaluation. 2,4,5-Trifluorophenoxyacetic acid (18) showed good activity when compared to 2,4,5-trichlorophenoxyacetic acid, especially in the preemergence test. However, most of the compounds screened in the pre- and postemergence herbicide test showed little or no effect at or below the 2.24 kilogram/hectare application rate.

A number of the compounds which exhibited some activity in the pre- and postemergence herbicide testing were further investigated for biological activity by studying their gross effects on the growth of *Arabidopsis thaliana* (table 4) and subsequently for activity in the tomato explant test (table 5) and the ethylene evolution test (table 6). *Arabidopsis thaliana* is an excellent test plant for biological testing because of its short life cycle. In a relatively short period of time (approximately 35 days), the gross biological effects starting with seed germination and ending with seed production can be monitored. In this study, 2,4,5-trifluorophenoxyacetic acid (18) exhibited the greatest effect on the growth of the *Arabidopsis thaliana* plant. This compound was toxic at application rates as low as 1 ppm and had measurable effects on the fresh weight and the fruit weight at 0.01 ppm.

In general, the other compounds tested were toxic at 10 to 100 ppm and showed little effect except on the fresh weight and the fruit weight at doses just below toxic levels. 2-Trifluoromethyl-3,4-dichloro-6-fluoroaniline (22), however, showed an increase in the fresh weight of the *Arabidopsis thaliana* specimen.

At rates less than toxic on all compounds tested for growth effects, root elongation, leaf morphology, and flower morphology were all normal. Root geotropism was positive in all of these cases. Therefore, these factors were omitted from table 4.

Because of its high activity, 2,4,5-trifluorophenoxy-

acetic acid (18) was also evaluated in the tomato pedicel explant test (table 5). In this case the number of abscissions is dramatically inhibited at application rates as low as 0.1 ppm. 2-Fluoro-6-bromobenzoic acid (23) also showed some inhibition at the 1 ppm dose level.

Isopropyl N(3,4-difluorophenyl)carbamate (12), ethyl N(4-fluorophenyl)carbamate (21), and isopropyl N(4-fluorophenyl)carbamate (13) exhibited inhibition only at higher doses near the toxic level. This activity decreased to near the control values at lower doses. The water control values started with one abscission on the first day and rose rapidly to 15 after 7 days.

2-Trifluoromethyl-3,4-dichloro-6-fluoroaniline (22) showed little if any effect on abscission rates. All of the compounds tested in the tomato pedicel explant test were toxic at the 1000 ppm application rate. This application rate was eliminated from table 5.

Five of the subject compounds were evaluated in the ethylene evolution test (table 6). This test is important in the screening of compounds for stimulating ripening of fruits and vegetables. It is difficult to get clear-cut answers from the results of this test. Although the method used in the ethylene evolution test is sound and controls are maintained on variables such as the ages of plants used, the values for cycloheximide and ethephon (controls) vary strikingly with different experiments. Therefore, each group of test plants within the ethylene evolution series must be viewed individually and the results must be considered in a general overview with the other activity evaluations done on each of the compounds. A better judgement of the gross biological effects of each compound tested can then be made.

In addition to the 23 compounds listed in tables 2 to 6, 191 other compounds were tested for biological activity, but because of their poor or complete inactivity these have been omitted from tables 2 to 6 and instead are listed in Appendix B by their chemical names.

TABLE 3. Pre- and Postemergence herbicides evaluation

Evaluation at indicated rate  
(kilograms/hectare)

Compound number	Chemical and plant species	0.07		0.14		0.28		0.56		1.12		2.24		4.48		8.96	
		K	V	K	V	K	V	K	V	K	V	K	V	K	V	K	V
10 Preemergence <sup>d</sup>	2-Chloro-6-iodofluorobenzene																
	Lima Bean																
	Corn																
	Wild Oats																
	Lettuce																
	Mustard																
	Purple Nutsedge																
11	Isopropyl N(2,5-difluorophenyl)carbamate																
	Lima Bean																
	Corn																
	Wild Oats																
	Lettuce																
	Mustard																
	Purple Nutsedge																
12	Isopropyl N(3,4-difluorophenyl)carbamate																
	Lima Bean																
	Corn																
	Wild Oats																
	Lettuce																
	Mustard																
	Purple Nutsedge																
13	Isopropyl N(4-fluorophenyl)carbamate																
	Lima Bean																
	Corn																
	Wild Oats																
	Lettuce																
	Mustard																
	Purple Nutsedge																
14	2,6-Difluorobenzonitrile																
	Lima Bean																
	Corn																
	Wild Oats																
	Lettuce																
	Mustard																
	Purple Nutsedge																
15	Ethyl N(3-trifluoromethylphenyl)carbamate																
	Lima Bean																
	Corn																
	Wild Oats																
	Lettuce																
	Mustard																
	Purple Nutsedge																
16	Isopropyl N(3-trifluoromethylphenyl)carbamate																
	Lima Bean																
	Corn																
	Wild Oats																
	Lettuce																
	Mustard																
	Purple Nutsedge																

TABLE 3. (Continued)

Evaluation at indicated rate  
(kilograms/hectare)

Compound number	Chemical and plant species	0.07		0.14		0.28		0.56		1.12		2.24		4.48		8.96			
		K	V	R	K	V	R	K	V	R	K	V	R	K	V	R	K	V	R
17	2-Nitro-4-fluorophenoxyacetic acid																		
	Lima Bean	0	4	2	100														
	Corn	0	5		0	4	2	30	3	2	40	3	2	60	3	2	100		
	Wild Oats																		
	Lettuce	80	4	2	100			90	2	2	95	1	2	100					
	Mustard	0	4	2	0	4	2	10	3	2	60	3	2	100					
	Crabgrass	0	4	2	0	4	2	0	3	2	0	4	2	0	4	2	0	4	2
	Purple Nutsedge	20	4	2	50	4		90	3	2	100								
		50	4	2	100														
		0	3	5	10	3	2	80	2	2	100								
18	2,4,5-Trifluorophenoxyacetic acid																		
	Lima Bean	0	5		0	4	2	0	3	2	70	2	2	70	2	2	100		
	Corn	0	4	2	0	4	2	0	3	2	40	3	2	60	3	2	100		
	Wild Oats																		
	Lettuce	80	4	2	100			90	2	2	95	1	2	100					
	Mustard	0	4	2	0	4	2	10	3	2	60	3	2	100					
	Crabgrass	0	4	2	0	4	2	0	3	2	0	4	2	0	4	2	0	4	2
	Purple Nutsedge	20	4	2	50	4		90	3	2	100								
		50	4	2	100														
		0	3	5	10	3	2	80	2	2	100								
19	2-Fluoro-4-nitrophenol																		
	Lima Bean	0	5		0	4	2	0	3	2	70	2	2	70	2	2	100		
	Corn	0	4	2	0	4	2	0	3	2	40	3	2	60	3	2	100		
	Wild Oats																		
	Lettuce	80	4	2	100			90	2	2	95	1	2	100					
	Mustard	0	4	2	0	4	2	10	3	2	60	3	2	100					
	Crabgrass	0	4	2	0	4	2	0	3	2	0	4	2	0	4	2	0	4	2
	Purple Nutsedge	20	4	2	50	4		90	3	2	100								
		50	4	2	100														
		0	3	5	10	3	2	80	2	2	100								
20	Ethyl N(3-chloro-4-fluorophenyl)carbamate																		
	Lima Bean	0	5		0	4	2	0	3	2	70	2	2	70	2	2	100		
	Corn	0	4	2	0	4	2	0	3	2	40	3	2	60	3	2	100		
	Wild Oats																		
	Lettuce	80	4	2	100			90	2	2	95	1	2	100					
	Mustard	0	4	2	0	4	2	10	3	2	60	3	2	100					
	Crabgrass	0	4	2	0	4	2	0	3	2	0	4	2	0	4	2	0	4	2
	Purple Nutsedge	20	4	2	50	4		90	3	2	100								
		50	4	2	100														
		0	3	5	10	3	2	80	2	2	100								

TABLE 3. (Continued)

Compound number	Chemical and plant species	Evaluation at indicated rate (kilograms/hectare)																		
		0.07		0.14		0.28		0.56		1.12		2.24		4.48		8.96				
		K	V	R	K	V	R	K	V	R	K	V	R	K	V	R	K	V	R	
18	2,4,5-Trifluorophenoxyacetic acid																			
	Lima Bean	0	4	2	0	3	10	40	3	10	100	3	10	100	3	10	100	3	10	100
	Corn	0	4	5	0	4	5	0	4	5	0	4	5	0	4	5	0	4	5	0
	Wild Oats	0	5	-	0	4	1	10	4	1	30	4	2	30	4	2	30	4	2	30
	Lettuce	-	-	-	0	5	-	100	-	-	-	-	-	-	-	-	-	-	-	-
	Mustard	80	3	2	80	3	2	100	-	-	-	-	-	-	-	-	-	-	-	-
	Crabgrass	0	4	1	0	4	1	0	4	2	0	3	1	0	3	1	0	3	1	0
	Purple Nutsedge	-	-	-	0	5	-	0	4	9	0	4	9	0	4	9	0	4	9	0
	Morning Glory	40	4	5	30	4	5	90	2	5	100	2	5	100	2	5	100	2	5	100
	Sickle Pod	0	5	-	20	4	2	70	3	5	90	2	5	100	2	5	100	2	5	100
	Soybean	0	3	2	80	2	1	80	3	1	100	-	-	-	-	-	-	-	-	-
	Field Oats	0	5	-	0	4	1	0	5	-	0	4	1	0	4	1	0	4	1	0
	Wheat	0	5	-	0	4	1	0	3	1	0	4	1	0	3	1	0	4	1	0
	Barley	0	4	1	0	4	1	0	5	-	0	4	1	0	4	1	0	4	1	0
	Onion	-	-	-	0	5	-	100	-	-	95	2	2	90	3	2	100	3	2	100
	Pea	0	3	1	70	2	1	80	1	1	100	0	0	0	0	0	0	0	0	0
	Sorghum	0	5	-	0	4	1	0	4	1	0	3	1	0	3	1	0	3	1	0
	Barn Grass	-	-	-	0	5	-	0	4	1	0	3	1	0	3	1	0	3	1	0
	Green Foxtail	0	5	-	0	3	-	30	3	1	20	3	1	30	3	1	30	3	1	30
	Rice	0	5	-	0	4	1	10	4	2	100	-	-	-	-	-	-	-	-	-
	Peanut	0	3	5	0	3	5	0	3	9	20	2	2	20	2	2	20	2	2	20
	Prickly Sida	80	3	2	80	3	2	80	3	2	90	2	2	95	2	2	95	2	2	95
	Tomato	80	4	5	100	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	Cotton	0	4	5	0	3	5	-	2	5	80	2	5	100	2	5	100	2	5	100
	Sebania	-	-	-	0	5	-	40	3	2	80	3	2	90	3	2	90	3	2	90
	Downy Brome	-	-	-	-	-	-	-	-	-	0	0	5	0	0	4	1	0	4	1

<sup>a</sup>K = % Kill

<sup>b</sup>V = Vigor

- 1 - Severe injury. Plants are not expected to recover.  
 2 - Moderate to severe injury. Severe injury to surviving plants.  
 3 - Moderate. Plants are expected to recover.  
 4 - Slight injury. Plants have recovered or are expected to fully recover.  
 5 - No effect.

<sup>c</sup>R = Biological response

- 1 - Necrosis  
 2 - Stunted  
 3 - Desiccation  
 4 - Axillary growth stimulation  
 5 - Nastic responses  
 6 - Necrotic spots  
 7 - Growth stimulation  
 8 - Defoliation  
 9 - Chlorosis  
 10 - Intumescence

<sup>d</sup>Testing procedure number 5

<sup>e</sup>Testing procedure number 6

TABLE 4. Effects on the growth of *Arabidopsis thaliana*<sup>a</sup>.

Compound number	Chemical name	Response	Average values for 4 replications treatment rate (ppm)					
			Control	0.01	0.1	1	10	100
10	2-Chloro-6-iodofluorobenzene	Seed germination, days	3	5	3	3	4	7 <sup>b</sup>
		Fresh weight (mg)	121	103	96	115	104	T
		Number of rosette leaves	11	10	11	10	10	T
		Rosette bolting, days	16	16	16	16	16	T
		Internode length (mm)	20	30	38	26	33	T
		Flowering, days	21	21	21	21	21	T
		Fruit weight (mg)	20.9 <sup>c</sup>	18	14	19	26	T
12	Isopropyl N(3,4-difluorophenyl)carbamate	Seed germination, days	3	3	3	3	3	3
		Fresh weight (mg)	110	113	111	99	T	T
		Number of rosette leaves	11	10	10	10	T	T
		Rosette bolting, days	17	17	17	17	T	T
		Internode length (mm)	30	31	25	29	T	T
		Flowering days	24	21	21	23	T	T
		Fruit weight (mg)	13.6	14.4	14.6	15.1	T	T
13	Isopropyl N(4-fluorophenyl)carbamate	Seed germination, days	3	3	3	3	3	5
		Fresh weight (mg)	110	99	118	95	67	T
		Number of rosette leaves	11	9	10	9	9	T
		Rosette bolting, days	17	17	17	17	17	T
		Internode length (mm)	30	32	32	22	24	T
		Flowering, days	24	21	22	22	24	T
		Fruit weight (mg)	13.6	16.8	15.2	17.3	3.5	T
18	2,4,5-Trifluorophenoxyacetic acid	Seed germination, days	3	3	3	3	3	5
		Fresh weight (mg)	121	80	60	T	T	T
		Number of rosette leaves	11	9	10	T	T	T
		Rosette bolting, days	16	16	16	T	T	T
		Internode length (mm)	20	34	18	T	T	T
		Flowering, days	21	21	21	T	T	T
		Fruit weight (mg)	20.9	8.8	3.7	T	T	T
21	Ethyl N(4-fluorophenyl)carbamate	Seed germination, days	4	4	4	4	4	T
		Fresh weight (mg)	111	129	125	69	T	T
		Number of rosette leaves	10	9	9	10	T	T
		Rosette bolting, days	19	19	19	19	T	T
		Internode length (mm)	26	39	35	24	T	T
		Flowering, days	24	22	24	25	T	T
		Fruit weight (mg)	23.4	25.6	19.5	5.3	T	T
22	2-Trifluoromethyl-3,4-dichloro-6-fluoroaniline	Seed germination, days	4	4	4	4	4	4
		Fresh weight (mg)	111	156	142	111	147	T
		Number of rosette leaves	9	10	10	10	10	T
		Rosette bolting, days	19	19	19	19	19	T
		Internode length (mm)	26	33	38	19	28	T
		Flowering, days	24	23	22	23	25	T
		Fruit weight (mg)	23.4	23	22.7	27	10	T
23	2-Fluoro-6-bromobenzoic acid	Seed germination, days	4	4	4	4	T	T
		Fresh weight (mg)	111	125	125	126	T	T
		Number of rosette leaves	10	9	10	10	T	T
		Rosette bolting, days	19	19	19	19	T	T
		Internode length (mm)	26	24	31	25	T	T
		Flowering, days	25	25	25	25	T	T
		Fruit weight (mg)	23.4	26.4	22.4	9.7	T	T

<sup>a</sup> Testing procedure number 7<sup>b</sup> T = Toxic<sup>c</sup> Value for a single surviving replicate

TABLE 5. Tomato pedicel explant test<sup>a</sup>

Compound number	Chemical name	Day	Number abscjsed out of 20 <sup>b</sup> rate (ppm)			
			0.1	1	10	100
12	Isopropyl N(3,4-difluorophenyl)carbamate	1	-	3	1	0
		2	-	11	5	T <sup>c</sup>
		3	-	12	5	T
		4	-	12	5	T
		5	-	12	5	T
13	Isopropyl N(4-fluorophenyl)carbamate	1	-	8	3	0
		2	-	9	7	1
		4	-	13	9	2
		5	-	13	10	2
		7	-	13	10	2
18	2,4,5-Trifluorophenoxyacetic acid	1	2	1	1	1
		2	3	1	3	1
		4	4	4	3	T
		5	4	4	4	T
		7	4	4	4	T
21	Ethyl N(4-fluorophenyl)carbamate	1	-	2	3	2
		2	-	12	11	2
		4	-	16	11	2
		5	-	16	11	2
		7	-	16	11	T
22	2-Trifluoromethyl-3,4-dichloro-6-fluoroaniline	1	-	5	7	5
		2	-	9	13	10
		4	-	11	14	11
		5	-	11	14	11
		7	-	12	14	11
23	2-Fluoro-6-bromobenzoic acid	1	-	1	2	0
		2	-	8	3	0
		4	-	8	3	1
		5	-	10	3	1
		7	-	10	3	1

<sup>a</sup>Testing procedure number 8.<sup>b</sup>Water control values were 1 on the first day and rose rapidly to 15 after 7 days.<sup>c</sup>T = Toxic.

TABLE 6. Ethylene evolution test<sup>a</sup>

Compound number	Chemical name	Ethylene/mg tissue <sup>b</sup> rate (ppm)				
		0.01	0.1	1	10	100
10	2-Chloro-6-iodofluorobenzene	8	13	15	17	3
18	2,4,5-Trifluorophenoxyacetic acid	7	28	5	11	50
	Cycloheximide <sup>c</sup>	13	7	8	4	2
	Ethephon <sup>c</sup>	9	15	28	284	2788
	Untreated check <sup>d</sup>	8	13	17	13	16
20	Ethyl N(3-chloro-4-fluorophenyl)carbamate	27	57	47	117	4
	Cycloheximide <sup>c</sup>	16	24	10	6	2
	Ethephon <sup>c</sup>	16	36	33	192	216
	Untreated check <sup>d</sup>	32	18	50	61	18
22	2-Trifluoromethyl-3,4-dichloro-6-fluoroaniline	151	71	107	118	68
23	2-Fluoro-6-bromobenzoic acid	60	79	83	89	58
	Cycloheximide <sup>c</sup>	5,663	214	244	74	9
	Ethephon <sup>c</sup>	112	90	373	2,034	25,005
	Untreated check <sup>d</sup>	112	72	145	111	58

<sup>a</sup>Testing procedure number 9.<sup>b</sup>Average of two replicates.<sup>c</sup>Standard.<sup>d</sup>Average of 5 replicates.

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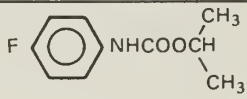
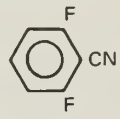
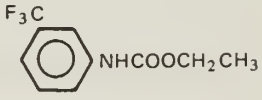

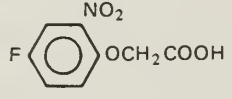
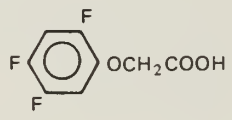
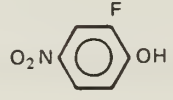
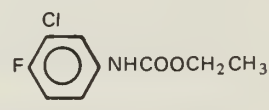

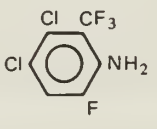
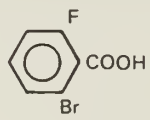
Appendix A—Compound Index

Number	Name	Empirical formula	Molecular weight	Structure
1	2,6-Difluoro-3,5-dinitrochlorobenzene	$C_6HClF_2N_2O_4$	238.53	
2	2-Fluorobenzoic acid hydrazide	$C_7H_7FN_2O$	154.14	
3	2,2'-Difluoro-5,5'-dihydroxydiphenyl sulfide	$C_{12}H_8F_2O_2S$	254.25	
4	2-Trifluoromethyl-4-fluoroaniline	$C_7H_5F_4N$	179.12	
5	N(2-Fluorophenyl)glycine hydrazide	$C_8H_{10}FN_3O$	183.19	
6	4-Fluoro-3-nitrobenzoic acid	$C_7H_4FNO_4$	185.11	
7	Methyl 2-fluoro-6-hydroxybenzoate	$C_8H_7FO_3$	170.14	
8	2,4-Dinitro-5-methylfluorobenzene	$C_7H_5FN_2O_4$	200.12	
9	2-Nitro-3-trifluoromethylphenol	$C_7H_4F_3NO_3$	207.11	
10	2-Chloro-6-iodofluorobenzene	$C_6H_3ClFI$	256.44	
11	Isopropyl N(2,5-difluorophenyl) carbamate	$C_{10}H_{11}F_2NO_2$	215.20	
12	Isopropyl N(3,4-difluorophenyl) carbamate	$C_{10}H_{11}F_2NO_2$	215.20	

(continued)



## Appendix A—continued

Number	Name	Empirical formula	Molecular Weight	Structure
13	Isopropyl N(4-fluorophenyl) carbamate	C <sub>10</sub> H <sub>12</sub> FNO <sub>2</sub>	197.21	
14	2,6-Difluorobenzonitrile	C <sub>7</sub> H <sub>3</sub> F <sub>2</sub> N	139.10	
15	Ethyl N(3-trifluoromethylphenyl) carbamate	C <sub>10</sub> H <sub>10</sub> F <sub>3</sub> NO <sub>2</sub>	233.19	
16	Isopropyl N(3-trifluoromethylphenyl) carbamate	C <sub>11</sub> H <sub>12</sub> F <sub>3</sub> NO <sub>2</sub>	247.22	
17	2-Nitro-4-fluorophenoxyacetic acid	C <sub>8</sub> H <sub>6</sub> FNO <sub>5</sub>	215.14	
18	2,4,5-Trifluorophenoxyacetic acid	C <sub>8</sub> H <sub>5</sub> F <sub>3</sub> O <sub>3</sub>	206.12	
19	2-Fluoro-4-nitrophenol	C <sub>6</sub> H <sub>4</sub> FNO <sub>3</sub>	157.10	
20	Ethyl N(3-chloro-4-fluorophenyl) carbamate	C <sub>9</sub> H <sub>9</sub> ClFNO <sub>2</sub>	217.63	
21	Ethyl N(4-fluorophenyl)carbamate	C <sub>9</sub> H <sub>10</sub> FNO <sub>2</sub>	183.18	
22	2-Trifluoromethyl-3,4-dichloro-6-fluoroaniline	C <sub>7</sub> H <sub>3</sub> Cl <sub>2</sub> F <sub>4</sub> N	248.01	
23	2-Fluoro-6-bromobenzoic acid	C <sub>7</sub> H <sub>4</sub> BrFO <sub>2</sub>	219.01	

Appendix B—Other compounds tested for biological activity

2-Fluoro-5-chloropyridine	1,2,3,4-Tetrafluorobenzene
2-Fluoro-5-bromopyridine	1,2,3,5-Tetrafluorobenzene
2-Fluoro-5-nitropyridine	1,2,4,5-Tetrafluorobenzene
2-Amino-6-fluoropyridine	2,4,6-Trifluoronitrobenzene
2,5-Dichloro-4,6-dinitro-1,3-difluorobenzene	2-Bromo-3,6-difluoro-4-chloroaniline
2,4,6-Trichloro-3,5-dinitrofluorobenzene	2-Fluoro-3-chloronitrobenzene
2,3,5,6-Tetrachloro-4-fluoronitrobenzene	2-Fluoro-4,6-dichlorophenol
2,3-Diiodo-1,4,5,6-tetrafluorobenzene	2,4-Dichloro-3,5-difluoroaniline
2,3-Difluoro-4,6-dinitrochlorobenzene	2-Fluoro-4,6-dinitrophenol
2,3-Dichloro-4,6-dinitrofluorobenzene	3-Fluoro-4,6-dinitrophenol
2,4-Dichloro-3,5-dinitrofluorobenzene	4-Fluoro-2,6-dinitrophenol
2,6-Dichloro-3,5-dinitrofluorobenzene	2,4-Difluoronitrobenzene
1,3,5-Trifluoro-2,4-dinitrobenzene	2,4,5-Trifluoro-6-nitroaniline
Pentafluorobenzene	2,4,5-Trifluorophenol
2-Chloro-3-nitro-5-bromofluorobenzene	2-Chloro-4-fluorophenol
3-Bromo-4,6-dinitrofluorobenzene	3-Chloro-4-fluorophenol
2,5-Dibromo-1,4-difluorobenzene	2,4-Dichloro-3-fluoroaniline
3-Chloro-4,6-dinitrofluorobenzene	2,5-Dichloro-4-fluoroaniline
2-Chloro-5-nitro-1,4-difluorobenzene	3,4-Dichloro-6-fluoroaniline
2,4-Dichloro-3-fluoronitrobenzene	3,5-Dichloro-4-fluoroaniline
2,4-Dichloro-5-fluoronitrobenzene	2-Fluoronicotinic acid
3,4-Dichloro-6-fluoronitrobenzene	6-Fluoronicotinic acid
2,4,6-Trichloro-3-fluorophenol	Methyl 6-Fluoronicotinate
3-Iodo-4,6-dinitrofluorobenzene	6-Fluoropicolinic acid
2-Cyano-3,5-difluoropyridine	6-Fluoropicolinic acid amide
2,3,4-Trifluoronitrobenzene	6-Fluoropicolinic acid hydrazide
2,4,5-Trifluoronitrobenzene	Methyl 6-fluoropicolinate

(continued)

2-Fluoro-6-nitrophenol	3-Fluoro-6-nitroaniline
4-Fluoro-2-nitrophenol	2,4-Difluoroacetanilide
2,5-Difluoro-4-nitroaniline	Ethyl N(2,4-difluorophenyl)carbamate
3,5-Difluoro-2-nitroaniline	N(2,4-Difluorophenyl)glycine hydrazide
2,4-Difluorophenol	Isopropyl N(2,4-difluorophenyl)carbamate
2,5-Difluorophenol	2,5-Difluoroaniline
2,5-Diacetoxy-1,4-difluorobenzene	Ethyl N(3,4-difluorophenyl)carbamate
2,3,4-Trifluoroaniline	N(3,4-Difluorophenyl)glycine hydrazide
2,4,5-Trifluoroaniline	3,5-Difluoroaniline
2,4,5-Trifluoroacetanilide	Ethyl N(3-fluorophenyl)glycinate
Ethyl N(2,4,5-trifluorophenyl)carbamate	N(4-Fluorophenyl)glycine hydrazide
Isopropyl N(2,4,5-trifluorophenyl)carbamate	N(3-Fluorophenyl)glycine hydrazide
2,4,6-Trifluoroaniline	2-Fluoro-3-methylpyridine
2,4,6-Trifluoroacetanilide	2-Fluoro-5-methylpyridine
Isopropyl N(2,4,6-trifluorophenyl)carbamate	2-Fluoro-6-methylpyridine
2-Fluoro-4-bromoaniline	2,6-Dichloro-3,5-difluorobenzotrifluoride
2-Bromo-4-fluoroaniline	2,5-Dichloro-3-nitrobenzotrifluoride
2-Bromo-4-fluoroacetanilide	3-Bromo-4-chlorobenzotrifluoride
2-Chloro-4-fluoroaniline	2,5-Dichloro-3-fluorobenzoic acid
N(2-Chloro-4-fluorophenyl)glycine hydrazide	2,6-Dichloro-3-fluorobenzoic acid
3-Chloro-2-fluoroacetanilide	2,6-Difluoro-3-nitrobenzoic acid
Ethyl N(2-fluoro-3-chlorophenyl)carbamate	3-Fluoro-6-iodobenzotrifluoride
Isopropyl N(2-fluoro-3-chlorophenyl)carbamate	2-Fluoro-5-nitrobenzotrifluoride
N(3-Chloro-4-fluorophenyl)glycine hydrazide	3-Fluoro-6-nitrobenzotrifluoride
2-Fluoro-4-nitroaniline	4-Fluoro-3-nitrobenzotrifluoride
2-Fluoro-4-nitroacetanilide	2-Fluoro-4-bromobenzoic acid
3-Fluoro-4-nitroaniline hydrochloride	2-Fluoro-4-chlorobenzoic acid

*(continued)*

2-Methyl-3-chloro-4,6-dinitrofluorobenzene	2-Fluoro-4-hydroxybenzaldehyde
2-Fluoro-3-chlorobenzoic acid	2-Fluorobenzoic acid
2-Fluoro-6-chlorobenzoic acid	Methyl 2-fluorobenzoate
2-Chlorobenzotrifluoride	3-Fluorobenzoic acid
3-Chlorobenzotrifluoride	4-Fluorobenzoic acid
4-Chlorobenzotrifluoride	3-Fluoro-4-hydroxybenzoic acid
2-Chloro-3-trifluoromethyl-5-fluoroacetanilide	3-Fluoro-4-acetoxybenzoic acid
2-Fluoro-4-nitrobenzoic acid	3-Fluoro-6-acetoxybenzoic acid
2,4-Difluorobenzoic acid	3,5-Difluoro-2-nitroanisole
Methyl 2,6-difluorobenzoate	2,4-Difluoro-5-nitroanisole
Ethyl 2,6-difluorobenzoate	3-Trifluoromethyl-4-nitroaniline
3,4-Difluorobenzoic acid	2,4,6-Trifluorobenzyl alcohol
3-Iodobenzotrifluoride	3-Trifluoromethylphenol
4-Nitro-3-trifluoromethylphenol	3-Trifluoromethylphenol (4-nitrobenzoate)
5-Trifluoromethylbenzotriazole	2-Fluoro-5-trifluoromethylaniline
2-Nitro-4-fluoro-6-trifluoromethylaniline	Ethyl N(2-fluoro-5-trifluoromethylphenyl)carbamate
2-Nitro-5-fluoro-3-trifluoromethylaniline	Isopropyl N(2-fluoro-5-trifluoromethylphenyl)carbamate
2,5-Difluoro-3-trifluoromethylaniline	Ethyl N(3-trifluoromethyl-4-fluorophenyl)glycinate
Ethyl N(2,5-difluoro-3-trifluoromethylphenyl)carbamate	Isopropyl N(4-fluoro-3-trifluoromethylphenyl)carbamate
2,5-Difluoro-3-trifluoromethylaniline hydrochloride	2-Fluoro-4-iodoanisole
Isopropyl N(2,5-difluoro-3-trifluoromethylphenyl)carbamate	4-Fluoro-2-iodoanisole
2-Bromo-5-trifluoromethylaniline	2-Fluoro-4-aminobenzoic acid
Ethyl N(2-bromo-5-trifluoromethylphenyl)glycinate	3-Fluoro-4-carboxyacetanilide
2-Trifluoromethyl-4-chloroaniline	2-Fluoro-4-nitroanisole
3-Trifluoromethyl-5-chloroaniline	4-Fluoro-2-nitroanisole
3-Methyl-4,6-dinitrofluorobenzene	N(3-Trifluoromethylphenyl)glycine hydrazide

(continued)

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2,4-Difluoro-3-methylaniline	N,N-Bis-6(fluoropyridyl) urea
3,5-Difluoro-2-methoxyacetanilide	1(4-Fluorophenyl)-2,3-dimethyl-5-pyrazolone
N(3-Fluoro-2-methylphenyl)glycine hydrazide	2,2'-Dihydroxy-5,5'-difluorobiphenyl
3-Fluoro-4-methylacetanilide	3,3'-Difluoro-4,4'-dihydroxybiphenyl
Ethyl N(3-fluoro-4-methylphenyl)glycinate	
N(3-Fluoro-4-methylphenyl)glycine hydrazide	
Isopropyl N(2-methyl-4-fluorophenyl)carbamate	
4-Fluoro-2-methylaniline	
3-Fluoro-4-methoxyaniline	
3-Fluoro-6-methoxyaniline	
Isopropyl N(3-fluoro-6-methoxyphenyl)carbamate	
3-Cyano-4-chlorobenzotrifluoride	
3,5-Bis-trifluoromethylchlorobenzene	
3-Cyano-4-fluorobenzotrifluoride	
2-Bromo-3,5-dichloro-4-fluorophenoxyacetic acid	
Ethyl N(3,5-bis(trifluoromethyl)phenyl)carbamate	
Isopropyl N(3,5-bis(trifluoromethyl)phenyl)carbamate	
2-Fluoro-6-methoxybenzoic acid	
3-Fluoro-2-methoxybenzoic acid	
3-Fluoro-4-methoxybenzoic acid	
3-Fluoro-6-methoxybenzoic acid	
4-Fluoro-2-methoxybenzoic acid	
4-Fluoromandelic acid	
2,4,6-Tris(trichloromethyl)-1,3,5-trifluorobenzene	
5(4-Fluorophenyl)-1,2-dithiole-3-one	
4-Methoxy-3-fluorobenzylcyanide	
3-Methylthio-5(4-fluorophenyl)-1,2-dithiolium iodide	

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