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Studies in Organic Fluorine Compounds Part I. Synthesis of Some N-Substituted Fluorobenzamides as Potential Fungicides

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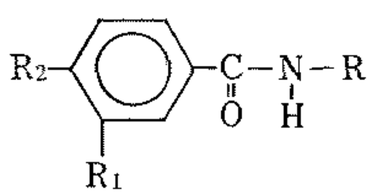
Saunders[1] and Bergmann *et al.*[2] have extensively investigated substituted fluoroacetamides of the type FCH_2CONHR as contact insecticides, potential larvaecides and rodenticides. The effective part of the molecule was found to be the CH_2F group.[3] The introduction of fluorine and other halogens in the aromatic nucleus also confers fungicidal activity[4] on the unsubstituted aromatic compounds.

Studies on the halogenated amides made by Mitchell[5] have revealed that the biological activity could be attributed both to the presence of an amide group in the side chain of the aromatic nucleus and the position and degree of halogenation in the benzene nucleus.

These views have been supplemented by the studies made on the amides of fluorophenoxy acids[6,8] and N-substituted fluorobenzamides.[7,8] Johnson *et al.*[9] have reported N,N-dialkyl and aminoalkyl benzamides as topical mosquito repellents.

Keeping these observations in view we have synthesised, as possible fungicides, N-substituted fluorobenzamides[10] from *m*-fluorobenzoic acid, 4-fluoro-3-

TABLE I. AMIDES OF FLUOROBENZOIC ACIDS



S. No.	R	R_1	R_2	mp/bp °C	Yield %	Molecular formula	% N	
							Found	Calcd.
1	*n*-Butyl	F	H	110/15 mm	67	$C_{11}H_{14}FNO$	7.00	7.17
2	3,4-Dichlorophenyl	F	H	123~124	86	$C_{13}H_8Cl_2FNO$	4.80	4.92
3[a]	*p*-Bromophenyl	F	H	167~168	74	$C_{13}H_9BrFNO$	4.75	4.76
4	*m*-Chlorophenyl	F	H	62	53	$C_{13}H_9ClFNO$	5.70	5.61
5	*o*-Tolyl	F	H	101~103	61	$C_{14}H_{12}FNO$	6.20	6.11
6	*m*-Tolyl	F	H	85	86	$C_{14}H_{12}FNO$	6.23	6.11
7	*p*-Anisyl	F	H	106	84	$C_{14}H_{12}FNO_2$	5.62	5.71
8	α-Naphthyl	F	H	146	88	$C_{17}H_{12}FNO$	5.12	5.28
9	*n*-Butyl	NO_2	F	117~118	62	$C_{11}H_{13}FN_2O_3$	11.80	11.60
10	*p*-Bromophenyl	NO_2	F	178~180	90	$C_{13}H_8BrFN_2O_3$	8.00	8.23
11[b]	*p*-Chlorophenyl	NO_2	F	174	35	$C_{13}H_8ClFN_2O_3$	9.40	9.50
12	*o*-Chlorophenyl	NO_2	F	164	64	$C_{13}H_8ClFN_2O_3$	9.36	9.50
13	*m*-Chlorophenyl	NO_2	F	172~173	86	$C_{13}H_8ClFN_2O_3$	9.32	9.50
14	Phenyl	NO_2	F	150~151	92	$C_{13}H_9FN_2O_3$	10.88	10.76
15	*p*-Tolyl	NO_2	F	165	76	$C_{14}H_{11}FN_2O_3$	10.30	10.21
16	*o*-Tolyl	NO_2	F	166~167	54	$C_{14}H_{11}FN_2O_3$	10.33	10.21
17	*p*-Anisyl	NO_2	F	169~171	86	$C_{14}H_{11}FN_2O_4$	9.51	9.65
18	*p*-Chlorophenyl	Br	F	172~173	53	$C_{13}H_8BrClFNO$	4.12	4.26
19[c]	*o*-Chlorophenyl	Br	F	136~137	57	$C_{13}H_8BrClFNO$	4.14	4.26
20	*p*-Bromophenyl	Br	F	148~149	62	$C_{13}H_8Br_2FNO$	3.58	3.75
21	*p*-Tolyl	Br	F	169~170	43	$C_{14}H_{11}BrFNO$	4.38	4.54
22	*m*-Tolyl	Br	F	180~181	40	$C_{14}H_{11}BrFNO$	4.40	4.54
23	*p*-Anisyl	Br	F	122~123	37	$C_{14}H_{11}BrFNO_2$	4.37	4.32

IR Spectral analyses ν_{max} in cm^{-1}

	Secondary N–H	Secondary amide –CONH
a)	3300	1670
b)	3200	1660
c)	3250	1630

nitrobenzoic acid[11] and 3-bromo-4-fluorobenzoic acid.[12]

Twelve of these fluorobenzamides were assayed for their antifungal activity.

EXPERIMENTAL

All the melting points are uncorrected.

N-Substituted amides of fluorobenzoic acids

A solution of the appropriate amine (0.02 M) in dry benzene was added dropwise, with frequent shaking to a solution of the acid chloride (0.01 M) in dry benzene. The reaction mixture was refluxed for two hours, left overnight, and then filtered. The precipitate was washed with a small quantity of warm benzene, which was then subsequently mixed with the original benzene extract from which the amide was obtained in the usual way.

The amides thus prepared are listed along with their relevant data in Table I.

To confirm their amide nature, some of the compounds were hydrolysed and the acids obtained were characterised by their melting points and neutralisation equivalents. In all cases the parent acids were obtained. For further structural confirmation the IR spectra of three representative compounds were taken and analysed. The spectral analyses have been reported in Table I.

Fungicidal activity. The antifungal activity of twelve compounds were evaluated against the fungus "*Aspergillus niger*" by agar plate technique[13] at three different concentrations namely 1: 1,000; 1: 10,000 and 1: 100,000. The average percentage inhibitions of radial growths of mycelial colonies by various compounds are recorded in Table II. The compounds No. 2, 3, 4, 7, 8, 10, 11, 13 and 20 were found active even at low concentrations.

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TABLE II. FUNGICIDAL ACTIVITY

Compound No.[a]	Average percentage inhibition after 96 hr		
	Concentrations used		
	1: 1000	1: 10,000	1: 100,000
2	100.00	100.00	100.00
3	100.00	100.00	100.00
4	100.00	100.00	87.61
7	100.00	92.85	80.95
8	100.00	100.00	78.57
10	100.00	88.09	80.95
11	80.95	75.81	75.61
13	97.60	71.42	50.00
18	64.28	52.38	33.33
19	66.66	52.38	47.61
20	66.66	61.92	57.14

[a] Numbers refer to those in Table I and number of replications = 3.

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