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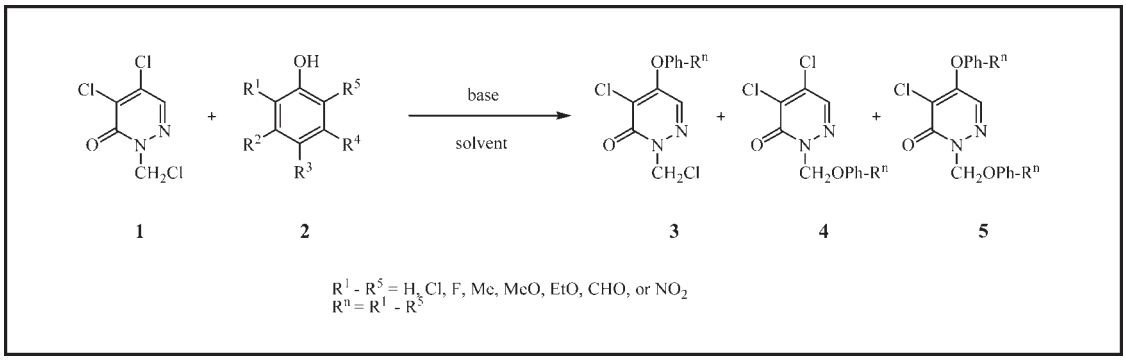

The regiochemistry was investigated for the reaction of 2-chloromethyl-4,5-dichloropyridazin-3(2*H*)-one (**1**) with some phenols **2** in the presence of a base in organic solvents. Seven diphenoxy derivatives **5** were synthesized selectively from **1** and phenols **2** under two optimized conditions. The product distributions of these reactions are dependent on the base, the solvent, and/or the substitutes of phenols.

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INTRODUCTION

Chloromethyl-4,5-dichloropyridazin-3(2*H*)-one (**1**) is a key intermediate for the synthesis of their derivatives, fluorescence molecules containing pyridazinone ring [1,2], and also biologically active compounds [3–7]. Because of involving four electrophilic carbons such as $ClCH_2N$-, C-3, C-4, and C-5, however, the reaction of compound **1** with a nucleophile such as phenol affords two or three products [8,9]. Also, the retro-ene reaction of compound **1** under basic condition was reported [9]. Based upon the atomic charges of compound **1** [10], the reactivity of the C-5 position about the nucleophile is more active than the α-C and C-4 positions (Fig. 1). Therefore, the selective synthesis of 5-phenoxy-2-phenoxymethyl-, 5-phenoxy-, or 2-phenoxymethyl derivative under basic condition is difficult.

In connection with the SAR study and the evaluation of biological activity such as cardiovascular activity or immunosuppressive activity, we need some 2-(phenoxymethyl)-5-phenoxylpyridazin-3(2*H*)-ones. Thus, we attempted to study on the regiochemistry in the reaction of compound **1** with phenols under basic condition (Scheme 1). In this article, we report the results for the title reactions.

RESULTS AND DISCUSSION

First, we examined the effect of the bases in the reaction of **1** with *o*-vaniline (**2a**) in acetonitrile. Reaction of compound **1** with two equivalents *o*-vaniline (**2a**) in the presence of base in refluxing acetonitrile gave products **3a**, **4a**, and **5a**. The distribution of the products for these reactions is shown in Table 1.

When K_2CO_3, Cs_2CO_3, and Na_2CO_3 as the base were used, compound **5a** was the main product. However, the reaction did not progress using NaH, Et_3N, and *N*,*N*-dimethylaminopyridine as base in refluxing acetonitrile. We selected potassium carbonate as the suitable base for this reaction.

On the other hand, we examined the effect of the solvents for the same reaction under different conditions such as at reflux temperature or under microwave irradiation.

Although two products were detected, the reaction of **1** with **2a** in acetonitrile, ethyl acetate, tetrahydrofuran, or acetone under the same conditions gave **5a** as the main product (entries 1, 2, 5, 6, 7, 8, 9, and 10, in Table 2). Compound **1** was reacted with **2a** in the presence of potassium carbonate in refluxing methanol or under microwave irradiation to afford selectively **5a** in

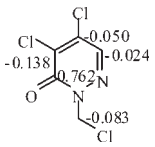
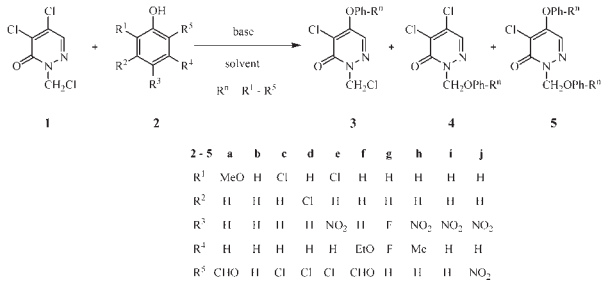


Figure 1. Atomic charge of carbons at the MP2/6-311+G** for compound **1**.

Scheme 1



2-5	a	b	c	d	e	f	g	h	i	j
R^1	MeO	H	Cl	H	Cl	H	H	H	H	H
R^2	H	H	H	Cl	H	H	H	H	H	H
R^3	H	H	H	H	NO_2	H	F	NO_2	NO_2	NO_2
R^4	H	H	H	H	H	EtO	F	Me	H	H
R^5	CHO	H	Cl	Cl	Cl	CHO	H	H	H	NO_2

excellent yield, respectively (entries 3 and 4 in Table 2). Compound **4a** was the main product when refluxing toluene was used, whereas the reaction did not occur in toluene under microwave irradiation. Even though the conversion ratio of **1** was low, **4a** was the main product in the reaction in methylene chloride and diethyl ether under microwave irradiation (entries 14 and 16 in Table 2). We did not find significant differences for the distribution of the products between the classical heating system and microwave irradiation system. According to TLC analysis, the product **5a** yielded *via* the 2-phenoxymethyl compound **4a** in the reaction of **1** with **2a**, whereas the 5-phenoxy derivative **3a** did not convert to **5a** under the same condition. Based upon the selectivity and the practical reaction conditions, therefore, we selected the optimized conditions, that is, (i) refluxing acetonitrile/potassium carbonate system and (ii) refluxing methanol/potassium carbonate system. Using the two optimized system, we investigated the effect of the substitutes of phenols. Most interestingly, reaction of **1** with 2**b** (2 equiv.) or **2e** (2 equiv.) under two optimized conditions gave selectively the corresponding 5-phenoxy derivatives **3b** or **3e** in excellent yields (entries 1, 2, 7, and 8 in Table 3). Treatment of **1** with **2j** under our two conditions also afforded selectively α-phenoxy derivative **4j** in excellent yield (entries 17 and 18 in Table 3).

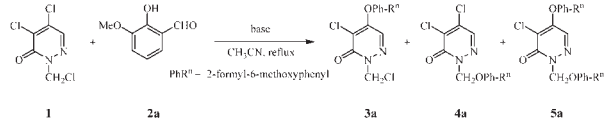
Although compound **1** was reacted with **2c** in refluxing acetonitrile to afford **4c** (48%) and **5c** (46%), the same reaction was carried out in refluxing methanol to give selectively **5c** in 94% yield (entries 3 and 4 in Table 3). On the other hand, compound **1** was reacted with **2d** or **2f–2i** under the same conditions to afford selectively α-diphenoxy derivatives **5d** or **5f–5i** in good to excellent yields, respectively (entries 5–6 and 9–16 in Table 3). The chemical structures of the all products were characterized by IR spectroscopy, NMR spectroscopy, and the elemental analysis.

CONCLUSIONS

In conclusion, the regiochemistry was investigated for the reaction of compound **1** with some phenols **2** in the presence of a base in organic solvents. Although diphenoxy derivatives **5** were synthesized selectively under two optimized conditions, we did not find any regularity. Although significant differences for the distribution of the products did not show under the classical heating system and microwave irradiation, the regiochemistry in

Table 1

Screening of bases for the reactions of one equivalent of **1** with two equivalents of **2a** in acetonitrile.[a]

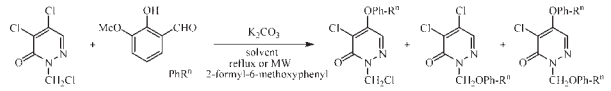


Entry	Base	Reaction time (hours)	Conversion ratio[b]	Product (%)[b]		
				3a	**4a**	**5a**
1	K_2CO_3	1.5	100	–	7	93
2	Cs_2CO_3	1	100	1	33	66
3	Na_2CO_3	13	100	12	40	48
4	NaH	48	0	–	–	–
5	Et_3N	48	0	–	–	–
6	DMAP[c]	48	0	–	–	–

[a] Mole ratio of reactants:**2/3a** = 1:2 equivalents.
[b] Determination by using proton NMR.
[c] *N*,*N*-Dimethylaminopyridine.

Table 2

The products distribution for the reactions of **1** with **2a** in the presence of K_2CO_3 in organic solvents.[a]



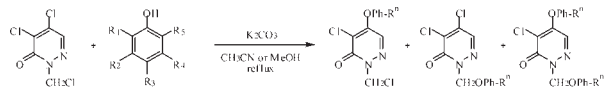
1 **2a** **3a** **4a** **5a**

Entry	Solvent	Reaction conditions[b]	Conversion ratio[c]	Product (%)[c]		
				3a	**4a**	**5a**
1	CH_3CN	Reflux,1.5 h	100	–	7	93
2		MW, 1.5 h	100	–	8	92
3	MeOH	Reflux, 1h	100	–	–	100
4		MW, 0.5 h	100	–	–	100
5	EtOAc	Reflux, 14 h	100	2	48	50
6		MW, 5 h	73	8	44	48
7	THF	Reflux, 48 h	100	45	23	32
8		MW, 5 h	90	15	42	43
9	Acetone	Reflux, 24 h	100	10	36	54
10		MW,2 h	100	8	42	50
11	Toluene	Reflux, 32 h	100	4	64	32
12		MW, 5 h	0	–	–	–
13	CH_2Cl_2	Reflux, 48 h	0	–	–	–
14		MW, 5 h	78	4	80	16
15	Ether	Reflux, 48 h	0	–	–	–
16[d]		MW, 1 h	59	25	63	12
17	*n*-Hexane	Reflux, 48 h	0	–	–	–
18		MW, 5 h	0	–	–	–

[a] Mole ratio of reactants:**2/3a** = 1:2 equivalents.
[b] MW = Microwave irradiation.
[c] Determination by using proton NMR.
[d] After 30 minutes, the solvent was refilled.

Table 3

The products distribution for the reactions of **1** with **2** in the presence of potassium carbonate in acetonitrile or methanol.[a]



1 **2** **3** **4** **5**

Entry	**2**	Reaction conditions	Product (%)[b]		
			3	**4**	**5**
1	**2b**	CH_3CN, 13 h	**3b**(93)	–	–
2		MeOH, 1 h	**3b**(94)	–	–
3	**2c**	CH_3CN, 8 h	–	**4c**(48)	**5c**(46)
4		MeOH, 5 h	–	–	**5c**(94)
5	**2d**	CH_3CN, 9 h	–	–	**5d**(93)
6		MeOH, 12 h	–	–	**5d**(94)
7	**2e**	CH_3CN, 34 h	**3e**(90)	–	–
8		MeOH, 20 h	**3e**(95)	–	
9	**2f**	CH_3CN, 4 h	–	–	**5f**(90)
10		MeOH, 2 h	–	–	**5f**(92)
11	**2g**	CH_3CN, 0.2 h	–	–	**5g**(94)
12		MeOH, 10 min	–	–	**5g**(95)
13	**2h**	CH_3CN, 1 h	–	–	**5h**(84)
14		MeOH, 20 min	–	–	**5h**(90)
15	**2i**	CH_3CN, 3 h	–	–	**5i**(89)
16		MeOH, 2h	–	–	**5i**(91)
17	**2j**	CH_3CN, 60 h	–	**4j**(85)	–
18		MeOH, 48 h	–	**4j**(90)	–

[a] Mole ratio of reactants:**2/3a** = 1:2 equivalents.
[b] Isolated yields.

the reaction is effected by the base, the solvent, and/or the substitutes of phenols. Further work including the theoretical study on the regiochemistry and the biological activity are under way in our laboratory.

EXPERIMENTAL

Melting points were determined with a capillary apparatus and uncorrected. ^{1}H and ^{13}C NMR spectra were recorded on a 300 MHz spectrophotometer with chemical shift values reported in δ units (ppm) relative to an internal standard (TMS). IR spectra were obtained on an IR spectrophotometer. Elemental analyses were performed with a CHNS-932 (Leco). 2-Chloromethyl-4,5-dichloropyridazin-3(2*H*)-one was synthesized by the literature method [2]. Microwave reaction was carried out by using a CEM, Discover microwave apparatus.

General procedure for the reaction of 2-chloromethyl-4,5-dichloropyridazin-3(2*H*)-one (1) with phenols (2).

Method 1. A solution of 2-hydroxy-3-methoxybenzaldehyde (2.8 g, 2 equiv.) and base (2 equiv.), 2-chloromethyl-4,5-dichloro-pyridazin-3(2*H*)-one (**2**) (2 g, 1 equiv.), and solvent (30 mL) was refluxed until **2** disappeared by TLC monitoring. After cooling the mixture to room temperature, the mixture was filtered and concentrated. The distribution of products was analyzed by ^{1}H NMR quantitative analysis. The products 3, 4, and 5 were also isolated by column chromatography on silica gel (2.5 × 8 cm) with *n*-hexane/EtOAc (2:1, v/v).

Method 2. 2-Chloromethyl-4,5-dichloropyridazin-3(2*H*)-one (**2**) (0.1 g, 1 equiv.) was dissolved in solvent (7 mL) in a 10 mL microwave vessel. 2-Hydroxy-3-methoxybenzaldehyde (0.14 g, 2 equiv.) and base (2 equiv.) were added. The vessel was closed and placed into a microwave apparatus. The mixture was irradiated per 10 min at 120°C with a set power of 300 W. After cooling, the mixture was filtered on a glass filter under vacuum. The distribution of products was analyzed by ^{1}H NMR quantitative analysis.

2-(5-Chloro-l-chloromethyl-6-oxo-l,6-dihydropyridazin-4-yloxy)-3-methoxybenzaldehyde (3a). Prepared by the Method 1 and 2. mp; 145°C. IR (KBr) 3081, 3045, 2935, 2878, 1663, 1581, 1479, 1452, 1387, 1254, 1213 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 3.87 (s, 3H), 5.86 (s, 2H, N-CH_2), 7.35 (d, 1H, J = 6 Hz), 7.42 (s, 1H), 7.46 (t, 1H, J = 6 Hz), 7.58 (d, 1H, J = 6 Hz), 10.30 ppm (s, CHO 1H); ^{13}C NMR ($CDCl_3$): δ 56.55, 58.29, 118.31, 121.12, 127.71, 129.26, 130.17, 142.79, 151.25, 153.83, 157.77, 187.75 ppm; Elemental analysis calcd. for $C_{13}H_{10}Cl_2N_2O_4$: C,47.44; H,3.06; N, 8.51; Found: C, 47.50; H, 3.10; N, 8.57.

4-Chloro-2-(chloromethyl)-5-phenoxypyridazin-3-(2*H*)-one (3b). Prepared by the Method 1. mp 78°C. IR (KBr) 3067, 3011, 1660, 1609, 1585, 1487, 1393, 1311, 1278, 1216, 1155 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 5.85 (s, 2H), 7.11 (d, 2H, J = 7.64 Hz), 7.32 (m, 1H), 7.48 (m, 2H), 7.53 ppm (s, 1H); ^{13}C NMR ($CDCl_3$): δ 58.29, 115.99, 119.79, 126.42, 130.55, 130.62, 131.23, 153.31, 153.72 ppm; Elemental analysis calcd. for $C_{11}H_8Cl_2N_2O_2$: C, 48.73; H, 2.97; N, 10.33. Found: C, 48.80; H, 3.00; N, 10.37.

4-Chloro-2-chloromethyl-5-[(2,6-dichloro-4-nitrophenoxy) methyl]pyridazin-3(2*H*)-one (3e). Prepared by the Method 1. mp 152°C. IR(KBr) 3086, 3054, 1678, 1588, 1523, 1506, 1349, 1293, 1249, 1215, 973, 956 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 6.11 (s, 2H), 7.77 (s, 1H), 8.21 ppm (s. 2H); ^{13}C NMR ($CDCl_3$): δ 81.58, 124.51, 129.94, 134.90, 136.66, 137.53, 144.20, 155.33, 156.82 ppm; Elemental analysis calcd. for $C_{12}H_7Cl_4N_3O_4$: C, 36.12; H, 1.77; N, 10.53. Found: C, 36.17; H, 1.79; N, 10.57.

2-[(4,5-Dichloro-6-oxopyridazin-l(6*H*)-yl)methoxy]-3-methoxybenzaldehyde (4a). Prepared by the Method 1 and 2. mp 143°C. IR (KBr) 3077, 3011, 2993, 2885, 2846, 1695, 1666, 1615, 1581, 1481, 1460, 1392, 1278, 1250,1217 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 3.91 (s, 3H), 6.09 (s, 2H, N-CH_2), 7.16 (d, 2H, J = 3 Hz), 7.34 (t, 1H, J = 5.1 Hz), 7.72 (s, 1H), 10.16 ppm (s, 1H); ^{13}C NMR ($CDCl_3$): δ 56.21, 81.44, 117.99, 119.41, 125.34, 129.93, 134.87, 136.33, 137.20, 148.32, 152.35, 156.58, 189.73 ppm; Elemental analysis calcd. for $C_{13}H_{10}Cl_2N_2O_4$: C,47.44; H,3.06; N, 8.51; Found: C, 47.49; H, 3.11; N, 8.58.

4,5-Dichloro-2-[(2,6-dichlorophenoxy)methyl]pyridazin-3(2*H*)-one (4c). Prepared by the Method 1. mp 142°C. IR (KBr) 3093, 3053, 1676, 1663, 1584, 1456, 1440, 1402, 1297, 1242, 1224, 1003, 990, 962 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 6.03 (s, 2H), 7.05 (t, 1H, J = 8.15 Hz), 7.29 (m, 2H), 7.72 ppm (s, 1H); ^{13}C NMR ($CDCl_3$): δ 81.46, 126.17, 129.07, 129.14, 134.84, 136.12, 137.17, 149.68, 156.80 ppm; Elemental analysis calcd. for $C_{11}H_6Cl_4N_2O_2$: C, 38.86; H, 1.78; N, 8.24. Found: C, 38.90; H, 1.82; N, 8.27.

4,5-Dichloro-2-((2,4-dinitrophenoxy)methyl)pyridazin-3(2*H*)-one (4j). Prepared by the Method 1. mp 147–149°C. IR (KBr) 3095, 3062, 1681, 1607, 1586, 1525, 1349, 1273, 1249, 987, 964 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 6.24 (s, 2H), 7.75 (d, 1H, J = 9.24 Hz), 7.90 (s, 1H), 8.47 (dd, 1H, J = 2.77, 6.45 Hz), 8.70 ppm (d, 1H, J = 2.74 Hz); ^{13}C NMR ($CDCl_3$): δ 79.10, 117.90, 121.72, 128.99, 135.14, 137.46, 137.85, 140.17, 141.90, 154.30, 156.62 ppm; Elemental analysis calcd. for $C_{11}H_6Cl_2N_4O_6$: C, 36.59; H, 1.67; N, 15.52. Found: C, 36.63; H, 1.70; N, 15.58.

2-(5-Chloro-l-((2-formyl-6-methoxyphenoxy)methyl)-6-oxo-l,6-dihydropyridazin-4-yloxy)-3-methoxybenzaldehyde (5a). Prepared by the Method 1 and 2. mp 165°C. IR (KBr): 3085, 3021, 2988, 2946, 2843, 1694, 1663, 1617, 1584, 1482, 1461, 1394, 1377, 1312, 1271, 1248, 990 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 3.86 (s, 3H), 3.96 (s, 3H), 6.15 (s, 2H), 7.19 (d, 2H, J = 6 Hz), 7.28 (t, 1H, J = 4.5 Hz), 7.33 (s, 1H), 7.38 (t, 1H, J = 3.8 Hz), 7.43 (s, 1H, J = 8.1 Hz), 7.56 (dd, 1H, J = 1.2, 7.7 Hz), 10.19 (s, 1H, CHO), 10.31 ppm (s, 1H, CHO); ^{13}C NMR ($CDCl_3$): δ 56.20, 56.44, 80.85, 117.91, 118.21, 119.00, 120.74, 125.27, 127.52, 129.15, 129.41, 142.98, 151.15, 152.49, 153.71, 187.80, 189.82 ppm; Elemental analysis calcd. for $C_{21}H_{17}ClN_2O_7$: C, 56.70; H, 3.85; N, 6.30; Found: C, 56.75; H, 3.87; N, 6.34.

4-Chloro-5-(2,6-dichlorophenoxy)-2-[(2,6-dichloro-phenoxy)-methyl]pyridazin-3(2*H*)-one (5c). Prepared by the Method 1. mp 145–147°C. IR (KBr): 3074, 3054, 2923, 1674, 1445, 1270 1237 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 6.04 (s, 2H), 7.05 (m, 1H), 7.29 (m, 3H), 7.44 (d, 2H, J = 8.11 Hz), 7.72 ppm (s, 1H); ^{13}C NMR ($CDCl_3$): δ 81.15, 126.04, 126.17, 128.11, 128.16, 128.98, 129.07, 129.25, 129.47, 136.12, 145.21, 152.46, 158.90 ppm; Elemental analysis calcd. for $C_{17}H_9Cl_5N_2O_3$: C, 43.77; H, 1.94; N, 6.00. Found: C, 43.79; H, 2.00; N, 6.07.

4-Chloro-5-(2,5-dichlorophenoxy)-2-[(2,5-dichlorophenoxy)-methyl]pyridazin-3(2*H*)-one (5d). Prepared by the Method 1. mp 143°C. IR (KBr): 3073, 3057, 3018, 1659, 1612, 1578,

1474, 1434, 1393, 1379, 1273, 1255, 1220, 1161, 1137 cm^{-1}. ^1H NMR ($CDCl_3$): δ 6.05 (s, 2H), 7.02 (dd, 1H, J = 2.26, 6.27 Hz), 7.22 (d, 1H, J = 2.30 Hz), 7.30 (m, 3H), 7.47 ppm (t, 2H, J = 8.59 Hz); ^{13}C NMR ($CDCl_3$): δ 79.41, 118.54, 120.22, 122.31, 123.35, 124.22, 124.72, 127.88, 129.64, 131.06, 131.96, 133.12, 134.05, 149.26, 152.68, 153.00, 158.49 ppm; Elemental analysis calcd. for $C_{17}H_9Cl_5N_2O_3$: C, 43.77; H, 1.94; N, 6.00. Found: C, 43.80; H, 2.02; N, 6.09.

2-(5-Chloro-l-[(3-ethoxy-2-formylphenoxy)methyl]-6-oxo-l,6-dihydropyridazin-4-yloxy)-6-ethoxybenzaldehyde (5f). Prepared by the Method 1. mp 188°C. IR (KBr): 2987, 2975, 2930, 2893, 2861, 1693, 1666, 1616, 1583, 1483, 1469, 1394, 1378, 1319, 1269, 1246, 1214, 1181, 1159, 1049, 987 cm^{-1}; ^1H NMR ($CDCl_3$):δ 1.26 (t, 3H, J = 6.87 Hz), 1.52 (t, 3H, J = 6.89 Hz), 4.16 (m, 4H), 6.17 (s, 2H), 7.35 (m, 7H), 10.19 (s, 1H), 10.32 ppm (s, 1H); ^{13}C NMR ($CDCl_3$): δ 14.42, 14.87, 64.84, 65.25, 80.88, 118.95, 119.01, 119.21, 120.54, 125.09, 127.37, 129.17, 129.77, 12996, 143.46, 148.79, 150.43, 153.88, 158.77, 187.87, 189.88 ppm; Elemental analysis calcd. for $C_{23}H_{22}Cl\ N_2O_7$: C, 58.29; H, 4.68; N, 5.91. Found: C, 58.32; H, 4.71; N, 5.97.

4-Chloro-5-(3,4-difluorophenoxy)-2-((3,4-difluorophenoxy) methyl)pyridazin-3(2*H*)-one (5g). Prepared by the Method 1. liquid. IR (KBr) 3083, 1681, 1668, 1513, 1270, 1252, 1206, 1160, 1132, 1031 cm^{-1}; ^1H NMR ($CDCl_3$): δ 5.98 (s, 2H), 7.26 (m, 6H), 7.56 ppm (s, 1H); ^{13}C NMR ($CDCl_3$): δ 78.45, 106.16, 106.43, 109.70, 109.97, 111.76, 115.60, 117.31, 117.55, 118.40, 118.67, 120.97, 130.29, 148.77, 151.88, 153.13, 158.54 ppm; Elemental analysis calcd. for $C_{17}H_9ClF_4N_2O_3$: C, 50.95; H, 2.26; N, 6.99.Found: C, 51.01; H, 2.30; N, 7.02.

4-Chloro-5-(3-methyl-4-nitrophenoxy)-2-[(3-methyl-4-nitrophenoxy)methyl]-pyridazin-3-(2*H*)-one (5h). Prepared by the Method 1. mp 128–129°C. IR (KBr): 3072, 3032, 1672, 1580, 1515, 1483, 1343, 1236, 1081, 1051 cm^{-1}; ^1H NMR ($CDCl_3$): δ 2.63 (d, 6H, J = 10.26 Hz), 6.12 (s, 1H), 7.09 (m, 4H), 7.68 (s, 1H), 8.12 ppm (m, 2H); ^{13}C NMR ($CDCl_3$): δ 21.02, 21.46, 53.49, 113.34, 116.58, 119.36, 122.12, 123.16, 127.46, 127.72, 131.60, 137.06, 137.62, 143.51, 146.02, 152.15, 156.46, 158.39, 159.68 ppm; Elemental analysis calcd. for $C_{19}H_{15}ClN_4O_7$: C, 51.08; H, 3.38; N, 12.54. Found: C, 51.11, H, 3.40; N, 12.57.

4-Chloro-5-(4-nitrophenoxy)-2-[(4-nitrophenoxy) methyl]-pyridazin-3(2*H*)-one (5i). Prepared by the Method 1. mp 177–178°. IR (KBr): 3073, 3057, 1659, 1612, 1578, 1474, 1393, 1379, 1273, 1220, 1095 cm^{-1}; ^1H NMR ($CDCl_3$): δ 6.15 (s, 2H), 7.26 (m, 4H), 7.69 (s, 1H), 8.33 ppm (m, 4H); ^{13}C NMR ($CDCl_3$): δ 83.90, 115.83, 118.75, 123.80, 125.99, 126.45, 131.81, 142.81, 144.98, 151.96, 158.23, 158.37, 161.27 ppm; Elemental analysis calcd. for $C_{17}H_{11}ClN_4O_7$: C, 48.76; H, 2.65; N, 13.38. Found: C, 48.79; H, 2.70; N, 13.41.

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