

irradiation *via* Smiles rearrangement is reported. Herein, we studied the synthesis of benzo-fused [1,4]oxazin-3(4*H*)-ones and pyrido-fused [1,4]oxazin-2(3*H*)-ones series. It was found that microwave-assisted heating reproducibly resulted in high to excellent yields of the corresponding products within a few minutes.

As shown in Scheme 1, the preparation of the benzo- and pyrido-fused [1,4]oxazinones begins with commercially available 2-chlorophenol or 2-chloro-3-pyridol (**1a-p**). Initially, the reaction of **1a-p** with *N*-substituted-2-chloro-

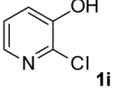
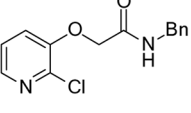
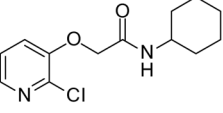
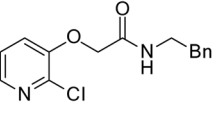
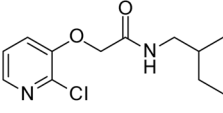
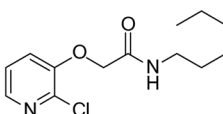
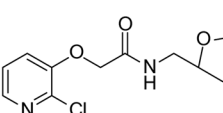
acetamide (**2a-p**) in the presence of potassium carbonate under microwave irradiation furnished the *N*-substituted-2-(2-chlorobenz-3-yloxy)acetamide or *N*-substituted-2-(2-chloropyridin-3-yloxy) acetamide (**3a-p**). Subsequent exposure of **3a-p** to cesium carbonate in DMF under microwave irradiation led to cyclization and gave the corresponding [1,4]oxazinones (**4a-p**) *via* Smiles rearrangement. These reactions were extremely rapid compared with the reactions under conventional heating. All the products were confirmed by physical and spectral data. Detailed investigations of differ-

Table 1. Comparison between microwave irradiation (M.W.) and conventional heating (C.H.) in the synthesis of **3a-j**

entry	phenols	products	C.H. ^a		M.W. ^b	
			1a-h to 3a-j	1a-h to 3a-j	1a-h to 3a-j	1a-h to 3a-j
			time (h)	yield (%)	time (min)	yield (%)
1			7	86	8	85
2	1a		10	82	8	90
3	1a		10	83	7	80
4			6	75	9	82
5			6	95	8	92
6			5	66	8	85
7			3	90	8	93
8			4	77	8	86
9			2	85	7	90
10			4	70	7	88

^aConversion of **1a-h** to **3a-j** with conventional heating (C.H.). ^bConversion of **1a-h** to **3a-j** with microwave irradiation (M.W)

Table 2. Comparison between microwave irradiation (M.W.) and conventional heating (C.H.) in the synthesis of **3k-p**

entry	pyridinol (1i)	products	C.H. ^a		M.W. ^b	
			time (h)	yield (%)	time (min)	yield (%)
1			3	90	4	92
2	1i		3	92	5	95
3	1i		5	92	5	90
4	1i		5	89	3	90
5	1i		4	90	5	93
6	1i		3	88	5	95

^aConversion of **1i** to **3k-p** with conventional heating (C.H.). ^bConversion of **1i** to **3k-p** with microwave irradiation (M.W.)**Table 3.** Comparison between microwave irradiation (M.W.) and conventional heating (C.H.) in the synthesis of **4a-j**

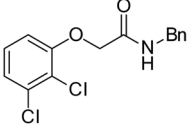
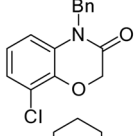
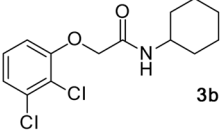
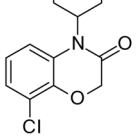
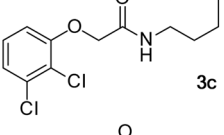
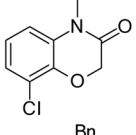
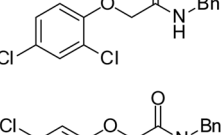
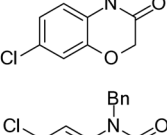
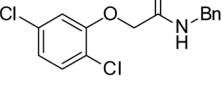
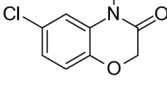
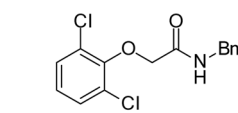
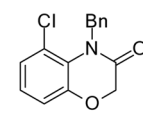
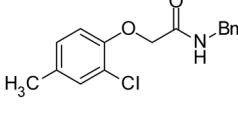
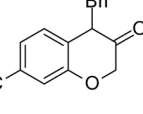
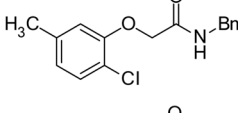
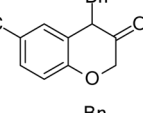
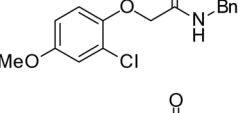
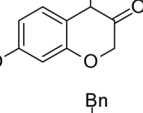
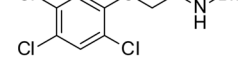
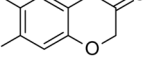
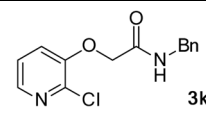
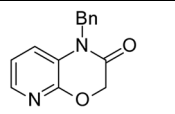
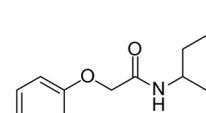
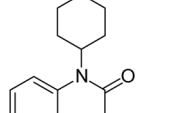
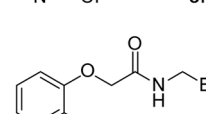
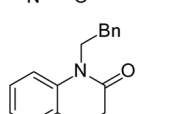
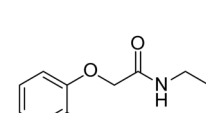
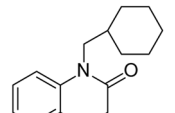
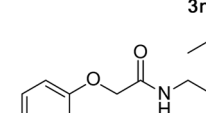
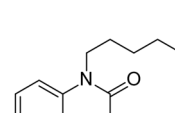
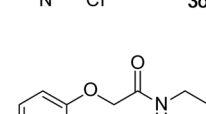
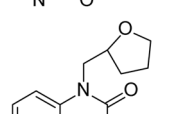
entry	acetamide	products	C.H. ^a		M.W. ^b	
			time (h)	yield (%)	time (min)	yield (%)
1			5	89	15	98
2			8	83	15	85
3			8	78	15	80
4			5	67	15	84
5			6	68	13	86

Table 3. Continued

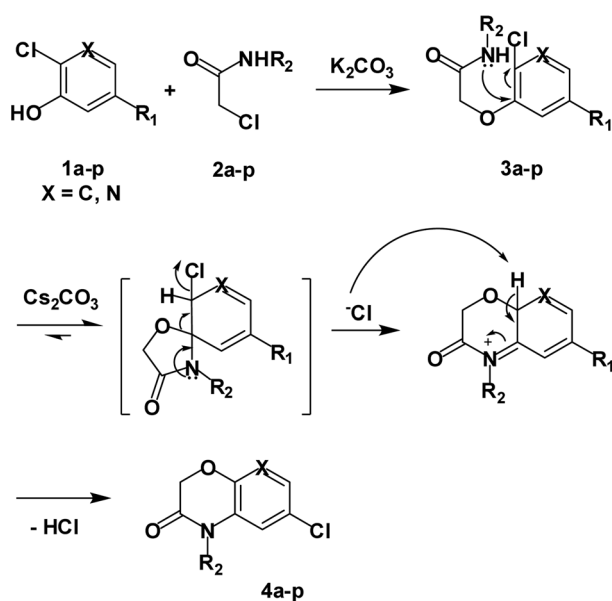
entry	acetamide	products	C.H. ^a		M.W. ^b	
			3a-j to 4a-j	3a-j to 4a-j	3a-j to 4a-j	3a-j to 4a-j
			time (h)	yield (%)	time (min)	yield (%)
6	 3f	 4f	7	92	13	95
7	 3g	 4g	5	66	13	84
8	 3h	 4h	5	70	15	88
9	 3i	 4i	3	74	15	82
10	 3j	 4j	5	63	15	90

^aConversion of **3a-j** to **4a-j** with conventional heating (C.H). ^bConversion of **3a-j** to **4a-j** with microwave irradiation (M.W)

Table 4. Comparison between microwave irradiation (M.W.) and conventional heating (C.H.) in the synthesis of oxazinone **4k-p**

entry	acetamide	products	C.H. ^a		M.W. ^b	
			3k-p to 4k-p	3k-p to 4k-p	3k-p to 4k-p	3k-p to 4k-p
			time (h)	yield (%)	time (min)	yield (%)
1	 3k	 4k	3	90	4	92
2	 3l	 4l	3	92	5	95
3	 3m	 4m	5	92	5	90
4	 3n	 4n	5	89	3	90
5	 3o	 4o	4	90	5	93
6	 3p	 4p	3	88	5	95

^aConversion of **3k-p** to **4k-p** with conventional heating (C.H). ^bConversion of **3k-p** to **4k-p** with microwave irradiation (M.W)



Scheme 2

ent substitutions of R_1 on benzyl group or diverse R_2 group on the reaction time and yields of the intermediates and products were studied and the data of the results were illustrated in Table 1-4.

As indicated, with the aim of increasing compound diversity through the integration of diverse skeletons, different substituted benzyl groups including different dichlorophenol, trichlorophenol, methyl chlorophenol, methoxy chlorophenols were used in the synthesis of benzo-fused [1,4]-oxazinones. The difference of substituted groups on benzyl ring did not have significant influence on the reaction time and yields. To study the influence of *N*-substituted-2-chloroacetamide on the rearrangement reaction, diverse R_2 groups were studied. Table 2 & 4 shows pyridine derivatives reacted more rapidly compared to benzene derivatives under the same conditions. All the reactions which take 2-10 hours under conventional heating were completed in 3-15 minutes under microwave irradiation.

Moreover, K_2CO_3 in MeCN was the most effective system for the synthesis of **3a-p**, and Cs_2CO_3 in DMF was found to be the most suitable reagent system for the preparation of **4a-p** according to our investigation of the base and solvent effects on the synthesis. Based on our previous work,^{16,17} we proposed the overall annulation is best explained by the process involved with Smiles rearrangement as shown in Scheme 2.

In summary, a novel and highly efficient synthesis of substituted benzo [*b*][1,4]oxazin-3(4*H*)-ones and pyrido-[2,3-*b*]oxazin-2(3*H*)-ones under microwave irradiation *via* Smiles rearrangement was established. Higher yields and shorter reaction time are the notable advantages of this method. The further work including expansion of new compound series and biological activity is underway in our laboratory.

Experimental Section

Typical procedure for the synthesis of *N*-benzyl-2-(2,3-dichlorophenoxy)acetamide **3a**. The solution of 2,3-dichlorophenol **1a** (1.00 g, 6.14 mmol), *N*-benzyl-2-chloroacetamide (1.13 g, 6.14 mmol), K_2CO_3 (1.02 g, 7.36 mmol) in dry CH_3CN was placed into microwave oven (KMIC-1.5 kW) at 85 °C and irradiated for the period listed in Table 1. The solvent was evaporated under reduced pressure. The residue was poured into water and then adjusted to pH = 7. It was then extracted by ethyl acetate. The combined organic layers were dried over anhydrous MgSO_4 and solvent was removed under vacuum to obtain the crude product. Pure product was obtained by further recrystallization as white solid **3a** (1.62 g, 85%). mp 129-130 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 4.57 (d, $J = 6.8$ Hz, 2H), 4.59 (s, 2H), 6.83 (dd, $J = 7.6$, 2 Hz, 1H), 7.13 (br s, 1H), 7.14-7.20 (m, 2H), 7.26-7.37 (m, 5H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 43.1, 68.4, 111.8, 122.1, 123.9, 127.6, 127.7, 128.8, 134.2, 137.6, 154.0, 167.1; MS (m/z): 309 (M^{+}).

***N*-Cyclohexyl-2-(2,3-dichlorophenoxy)acetamide (3b):** White solid, mp 163-164 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 1.23-1.31 (m, 3H), 1.37-1.47 (m, 2H), 1.60-1.64 (m, 1H), 1.70-1.76 (m, 2H), 1.92-1.96 (m, 2H), 3.85-3.94 (m, 1H), 4.51 (s, 1H), 6.75 (br s, 1H), 6.81 (dd, $J = 6.8$, 2 Hz, 1H), 7.15-7.27 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 24.6, 25.5, 32.9, 47.8, 68.3, 111.7, 122.0, 123.7, 127.8, 134.1, 154.1, 166.1; MS (m/z): 303 (M^{+}), 267 ($\text{M}^{+}-\text{Cl}$).

2-(2,3-Dichlorophenoxy)-*N*-hexylacetamide (3c): White solid, mp 96-98 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 0.89 (t, $J = 6.8$ Hz, 3H), 1.26-1.39 (m, 6H), 1.53-1.61 (m, 2H), 3.37 (dd, $J = 13.2$, 6.8 Hz, 2H), 4.53 (s, 2H), 6.82 (dd, $J = 7.6$, 2 Hz, 2H), 7.15-7.27 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 14.0, 22.6, 26.5, 29.4, 31.4, 39.1, 68.3, 111.6, 121.9, 123.7, 127.8, 134.2, 154.1, 167.0; MS (m/z): 305 (M^{+}), 269 ($\text{M}^{+}-\text{Cl}$).

***N*-Benzyl-2-(2,4-dichlorophenoxy)acetamide (3d):** Colorless crystal, mp 124-125 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 4.54-4.56 (d, 4H), 6.82 (d, $J = 8.8$ Hz, 1H), 7.11 (br s, 1H), 7.19 (dd, $J = 8.8$, 2.4 Hz, 1H), 7.26-7.37 (m, 6H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 43.1, 68.5, 114.8, 123.8, 127.5, 127.6, 128.0, 128.8, 130.2, 137.6, 151.6, 167.1; MS (m/z): 309 (M^{+}).

***N*-Benzyl-2-(2,5-dichlorophenoxy)acetamide (3e):** White solid, mp 97-99 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 4.54 (s, 2H), 4.56 (d, $J = 6$ Hz, 2H), 6.89 (d, $J = 2$ Hz, 1H), 6.96 (dd, $J = 8.4$, 2 Hz, 1H), 7.12 (br s, 1H), 7.26-7.36 (m, 6H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 43.1, 68.2, 114.6, 121.4, 123.0, 127.6, 127.7, 128.8, 131.0, 133.5, 137.6, 153.1, 167.0, 169.3; MS (m/z): 309 (M^{+}).

***N*-Benzyl-2-(2,6-dichlorophenoxy)acetamide (3f):** Colorless crystal, mp 94-96 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 4.58-4.60 (t, 4H), 7.03 (t, $J = 8$ Hz, 1H), 7.26-7.38 (m, 7H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 43.1, 71.5, 126.0, 127.6, 127.8, 128.8, 129.0, 137.7, 150.2, 167.6; MS (m/z): 309 (M^{+}).

***N*-Benzyl-2-(2-chloro-4-methylphenoxy)acetamide (3g):** Light yellow-brown crystal, mp 94-95 °C. ¹H NMR (400 MHz, CDCl₃) δ 2.25 (s, 3H), 4.52 (s, 2H), 4.54 (d, *J* = 6 Hz, 2H), 6.77 (d, *J* = 8.4 Hz, 1H), 6.98-7.00 (dd, *J* = 4.4, 2 Hz, 1H), 7.16 (d, *J* = 2 Hz, 1H), 7.22 (br, 1H), 7.26-7.35 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 20.4, 43.0, 68.5, 114.1, 122.6, 127.6, 128.5, 128.8, 130.9, 132.9, 137.8, 150.7, 167.8; MS (*m/z*): 289 (M⁺).

***N*-Benzyl-2-(2-chloro-5-methylphenoxy)acetamide (3h):** Light brown crystal, mp 113-114 °C; ¹H NMR (400 MHz, CDCl₃) δ 2.32 (s, 3H), 4.55 (s, 2H), 4.56 (d, *J* = 6 Hz, 2H), 6.71 (s, 1H), 6.77 (dd, *J* = 8, 0.8 Hz, 1H), 7.20 (br, 1H), 7.22-7.36 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 21.3, 43.0, 68.2, 114.9, 120.0, 123.6, 127.6, 126.8, 130.0, 137.7, 138.5, 152.5, 167.7; MS (*m/z*): 289 (M⁺).

***N*-Benzyl-2-(2-chloro-4-methoxyphenoxy)acetamide (3i):** White solid, mp 88-89 °C; ¹H NMR (400 MHz, CDCl₃) δ 3.75 (s, 3H), 4.52 (s, 2H), 4.55 (d, 2H), 6.75 (dd, *J* = 9.2, 2.8 Hz, 1H), 6.84 (d, *J* = 8.8 Hz, 1H), 6.93 (d, *J* = 2.8 Hz, 1H), 7.21 (br, 1H), 7.27-7.36 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 43.0, 55.9, 69.2, 113.2, 115.7, 116.1, 123.8, 127.6, 128.8, 137.8, 147.1, 155.0, 167.9; MS (*m/z*): 305 (M⁺).

***N*-Benzyl-2-(2,4,5-trichlorophenoxy)acetamide (3j):** White crystal, mp 137-138 °C; ¹H NMR (400 MHz, CDCl₃) δ 4.53 (s, 2H), 4.55 (d, *J* = 6 Hz, 2H), 6.99 (s, 1H), 7.06 (br, 1H), 7.27-7.36 (m, 5H), 7.45 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 43.2, 68.5, 115.6, 122.1, 126.0, 127.6, 127.8, 128.9, 131.1, 131.7, 137.5, 151.7, 166.6; MS (*m/z*): 343 (M⁺).

Typical procedure for the synthesis of 4-benzyl-7-methyl-2*H*-benzo[*b*][1,4]oxazin-3(4*H*)-one **4a** (Table 3). The solution of *N*-benzyl-2-(2,3-dichlorophenoxy) acetamide **3a** (0.6 g, 1.93 mmol), Cs₂CO₃ (0.76 g, 2.32 mmol) in dry DMF was placed into microwave oven at 130 °C and irradiated for 15 min. The solvent was evaporated under reduced pressure. The residue was added into water and then adjusted to pH = 7 and the mixture was extracted by ethyl acetate. The combined organic layers were dried over anhydrous MgSO₄ and the solvent was removed under vacuum to obtain crude product. Flash column chromatography on silica gel, eluting with hexanes/ethyl acetate yielded **4a** as white solid (0.46 g, 88%). Mp 98-99 °C; ¹H NMR (400 MHz, CDCl₃) δ 4.80 (s, 2H), 5.14 (s, 2H), 6.76-6.83 (m, 2H), 7.00-7.03 (dd, *J* = 7.6, 2 Hz, 1H), 7.21-7.33 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 45.2, 67.8, 114.2, 122.4, 122.8, 124.9, 126.6, 127.7, 129.0, 130.0, 135.5, 141.5, 164.2; MS (*m/z*): 273 (M⁺).

8-Chloro-4-cyclohexyl-2*H*-benzo[*b*][1,4]oxazin-3(4*H*)-one (4b): Light pink solid, mp 74-76 °C; ¹H NMR (400 MHz, CDCl₃) δ 1.22-1.43 (m, 3H), 1.71 (t, 1H), 1.79-1.91 (m, 4H), 2.30-2.40 (m, 2H), 4.10-4.18 (m, 1H), 4.56 (s, 1H), 6.95 (t, *J* = 8 Hz, 1H), 7.06-7.09 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 25.3, 26.4, 29.4, 57.5, 68.9, 114.8, 122.6, 122.7, 124.7, 131.3, 142.8, 165.7; MS (*m/z*): 265 (M⁺).

8-Chloro-4-hexyl-2*H*-benzo[*b*][1,4]oxazin-3(4*H*)-one (4c): Colorless liquid; ¹H NMR (400 MHz, CDCl₃) δ 0.89 (t, *J* = 6.8 Hz, 3H), 1.31-1.39 (m, 6H), 1.60-1.68 (m, 2H), 3.91 (t, *J* = 7.6 Hz, 2H), 4.67 (s, 2H), 6.89 (dd, *J* = 1.6 Hz, 1H), 6.96 (t, *J* = 8 Hz, 1H), 7.05 (dd, *J* = 8, 1.6 Hz); ¹³C NMR (100

MHz, CDCl₃) δ 14.0, 22.6, 26.5, 27.0, 41.5, 67.7, 113.4, 122.5, 122.7, 124.5, 130.0, 141.5, 163.6; MS (*m/z*): 267 (M⁺).

4-Benzyl-7-chloro-2*H*-benzo[*b*][1,4]oxazin-3(4*H*)-one (4d): White-off solid, mp 98-100 °C; ¹H NMR (400 MHz, CDCl₃) δ 4.72 (s, 2H), 5.13 (s, 2H), 6.76-6.78 (d, *J* = 8.4 Hz, 1H), 6.83-6.86 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.98-6.99 (d, *J* = 2.4 Hz, 1H), 7.21-7.33 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 45.0, 67.6, 116.5, 117.4, 122.7, 126.6, 127.7, 128.9, 129.0, 135.5, 145.9, 164.1; MS (*m/z*): 273 (M⁺).

4-Benzyl-6-chloro-2*H*-benzo[*b*][1,4]oxazin-3(4*H*)-one (4e): White solid, mp 113-114 °C; ¹H NMR (400 MHz, CDCl₃) δ 4.71 (s, 2H), 5.11 (s, 2H), 6.87-6.91 (m, 3H), 7.23-7.35 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 45.0, 76.8, 115.8, 118.0, 123.7, 126.7, 127.8, 129.0, 129.8, 135.3, 143.9, 164.4; MS (*m/z*): 273 (M⁺).

4-Benzyl-5-chloro-2*H*-benzo[*b*][1,4]oxazin-3(4*H*)-one (4f): Colorless liquid; ¹H NMR (400 MHz, CDCl₃) δ 4.54 (s, 2H), 5.51 (s, 2H), 6.87-6.98 (m, 3H), 7.13-7.23 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 47.0, 69.2, 115.8, 123.9, 125.5, 126.0, 127.0, 127.7, 128.4, 136.4, 150.9, 167.3; MS (*m/z*): 273 (M⁺).

4-Benzyl-7-methyl-2*H*-benzo[*b*][1,4]oxazin-3(4*H*)-one (4g): White solid; mp 90-91 °C; ¹H NMR (400 MHz, CDCl₃) δ 2.24 (s, 3H), 4.69 (s, 2H), 5.13 (s, 2H), 6.67-6.80 (m, 3H), 7.23-7.32 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 20.7, 45.0, 67.8, 115.5, 117.6, 123.3, 126.2, 126.6, 127.5, 128.9, 134.1, 136.1, 145.1, 164.6; MS (*m/z*): 253 (M⁺).

4-Benzyl-6-methyl-2*H*-benzo[*b*][1,4]oxazin-3(4*H*)-one (4h): White solid; mp 117-119 °C; ¹H NMR (400 MHz, CDCl₃) δ 2.20 (s, 3H), 4.68 (s, 2H), 5.14 (s, 2H), 6.69 (s, 1H), 6.75 (d, *J* = 8.4 Hz, 1H), 6.88 (d, *J* = 8.4 Hz, 1H), 7.24-7.34 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 45.0, 55.6, 67.8, 103.2, 107.9, 116.3, 122.2, 126.6, 127.5, 128.9, 136.0, 146.3, 156.4, 164.1; MS (*m/z*): 253 (M⁺).

4-Benzyl-7-methoxy-2*H*-benzo[*b*][1,4]oxazin-3(4*H*)-one (4i): Light pink solid; mp 71-74 °C; ¹H NMR (400 MHz, CDCl₃) δ 3.71 (s, 3H), 4.69 (s, 2H), 5.12 (s, 2H), 6.41-6.44 (dd, *J* = 8.8, 2.8 Hz, 1H), 6.56 (d, *J* = 2.8 Hz, 1H), 6.76 (d, *J* = 12.8 Hz, 1H), 7.23-7.32 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 45.0, 55.6, 67.8, 103.2, 107.9, 116.3, 122.2, 126.6, 127.5, 128.9, 136.1, 146.3, 156.4, 164.1; MS (*m/z*): 269 (M⁺).

4-Benzyl-6,7-dichloro-2*H*-benzo[*b*][1,4]oxazin-3(4*H*)-one (4j): White solid; mp 157-159 °C; ¹H NMR (400 MHz, CDCl₃) δ 4.72 (s, 2H), 5.10 (s, 2H), 6.94 (s, 1H), 7.07 (s, 1H), 7.22-7.36 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 45.1, 67.6, 116.9, 118.6, 126.1, 126.7, 126.9, 128.0, 128.5, 129.1, 135.0, 144.3, 163.9; MS (*m/z*): 307 (M⁺).

The physical and spectral data of known compounds pyrido[2,3-*b*]oxazin-2(3*H*)-ones **4k-4p** are in accordance with literature.¹⁸

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