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Synthesis of Organic Carbonates with Alkyl/aryl 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylates and ROH/$AlCl_3$ under Ambient Condition

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We demonstrated the synthesis of organic carbonates using alkyl/aryl 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylates and alcohol in the presence of aluminum chloride. Alkyl/aryl 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylates were reacted with alcohol in the presence of $AlCl_3$ in toluene at room temperature to afford the corresponding unsymmetric and symmetric organic carbonates in good to excellent yields. These are efficient and convenient processes. Alkyl/aryl 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylates are solid, stable and non-toxic CO_2/CO_2R(Ar) source. It is noteworthy that the reaction is carry out under an ambient and acidic conditions, the easy-to prepare and readily available starting materials and the quantitative isolation of reusable 4,5-dichloropyridazin-3(2*H*)-one.

Key Words : Organic carbonate, Alkoxide equivalent, Alcohol-aluminum adduct, Alkyl/aryl 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylates, 4,5-Dichloropyridazin-3(2*H*)-one

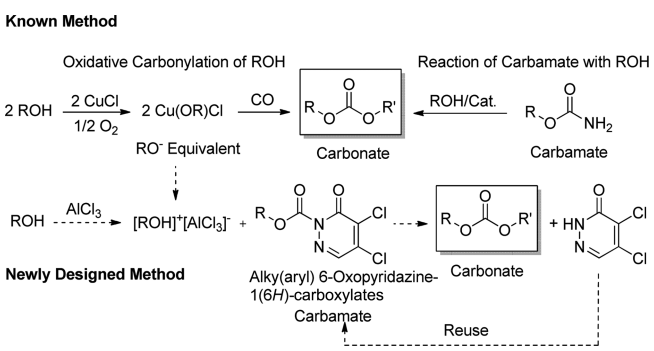
Introduction

Organic carbonates are generally safe noncorrosive molecules employed in numerous commercial and synthetic application[1-4] as eco-friendly useful reagents,[2,3,5-10] solvents for Li-ion battery[3,4] and electroanalytics.[3,11] The symmetric organic carbonates [$(RO)_2C=O$] are useful as the solvents, whereas the unsymmetric organic carbonates [ROC(=O)OR'] are used as the key-functional group in drugs and other chemicals. Various synthetic methods of organic carbonates by the phosgenation technique using $COCl_2$, the oxidative carbonylation of alcohols using CO and transition metals, the reaction of urea with alcohols, the reaction of oxiranes and CO_2, the reaction of chloroformates, the use of metal carbonate and the organic carbonate interchange reaction have been reported.[1-3,9,12,13] However, the main disadvantages of these methods are the use of toxic, gaseous and/or expensive chemicals and requirement for specific additives. The alkoxycarbonylation using organic carbonate and base be also accompanied by the undesired side reaction.[8,14] Moreover, unsymmetric organic carbonates cannot be prepared by these methods. Therefore, a great deal of research has focused on the development of a convenient and useful synthetic method for symmetric and unsymmetric organic carbonates using a nongaseous and recyclable CO_2 or CO_2R(Ar) source under non-basic conditions. To avoid the side reaction in the reaction using organic carbamate, the alkoxide or alkoxide equivalent must be prepare under aprotic acid or neutral condition.

Romano *et al.*[15] reported the synthesis of dimethyl carbonate by oxidative carbonylation of methanol using copper chloride *via* $Cu(OCH_3)Cl$ intermediate (Scheme 1). In this reaction, the $Cu(OCH_3)Cl$ acts as an equivalent of methoxide (MeO^-).

On the other hand, Ball *et al.*[16] reported the synthesis of organic carbonate by the reaction of carbamate in the presence of the catalyst. As shown in Scheme 1, alkyl/aryl 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylates[17] have a carbamate functionality. Thus, alkyl/aryl 6-oxopyridazinone-1(6*H*)-carboxylates may be use as alkoxy/aryloxy carbonyl source, and also the 4,5-dichloropyridazin-3(2*H*)-one anion as the leaving group may be act as a proton acceptor during the reaction.[18-24] Pyridazin-3(2*H*)-ones are inexpensive, very stable and good leaving group, and also can be removed and/or recycles spurred our interest in their use for other transformation according to Yoon *et al.*[18-24]

Although alkyl/aryl 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylates are good carbonyl source, however, these can



Scheme 1. Known and newly designed methods for the synthesis of organic carbonates.

[a]These authors contributed equally to this work.

not use in basic condition because of the side reaction.[25-27] Thus, we required an acidic condition for the synthesis of carbonate from alkyl/aryl 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylates and alcohols.

Inspired by the oxidative carbonyation[1,15,28] and the method of carbamate reaction,[1,16] we attempted to develop an novel convenient synthetic method for unsymmetric and symmetric organic carbonates from alkyl(or aryl) 6-oxopyridazin-1(6*H*)-one carboxylate as a carbamate and ROH in the presence of $AlCl_3$ (Scheme 1).

Herein, we report the synthesis of organic carbonates using ROH/$AlCl_3$ systems and alkyl(or aryl) 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylates system in toluene at room temperature.

Results and Discussion

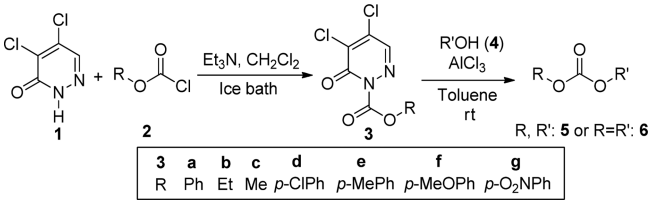
In order to demonstrate our research motivation, we firstly attempted to find a novel ROH/MCl_n system acting alkoxide equivalent. First of all, we selected aluminum chloride as the Lewis acid. Although the reaction of ROH (3 equiv.) with $AlCl_3$ (1 equiv.) yields the corresponding aluminum alkoxides $[Al(OR)_3]$,[29] (ROH-$AlCl_3$) adduct (1:1 ratio) may be easily formed in the initial step of this reaction. If only to remove the proton of (ROH-$AlCl_3$) adducts in the solvent, the residue $[(ROAlCl_3)^-]$ may be act as the alkoxide. To remove a proton of the adducts, the proton acceptor such as the organic base or the leaving group is required.

$$3ROH + AlCl_3 \rightarrow 3[(ROH)^+(AlCl_3)^-] \rightarrow (RO)_3Al + 3\ HCl$$

$$ROH + AlCl_3 \rightarrow [(ROH)^+(AlCl_3)^-] + Base \rightarrow [(ROAlCl_3)^-][H(Base)^+]$$

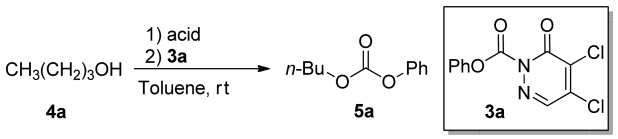
Alkyl(or aryl) 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylates **3** were prepared by the literature method[17] from 4,5-dichloropyridazin-3(2*H*)-one (**1**) and the corresponding chloroformate **2** (Scheme 2).

As a model reaction to evaluate newly designed reaction, we studied the effect of Lewis acids, protic acids and solvents in the reaction of *n*-butanol in the presence of Lewis acids or protic acids with phenyl 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylate (**3a**) as a acyl source at room temperature. Among the twelve Lewis acids investigated, one equivalent of aluminum chloride showed the best results (Entry 2, Table 1). Next, we investigated the solvent effect using the **4a**/**3a**/$AlCl_3$ system. Toluene also showed the best results among the six solvents tested (Entry 3, Table 2).



Scheme 2. Synthesis of symmetric and unsymmetric organic carbonates using alcohol-$AlCl_3$ adducts.

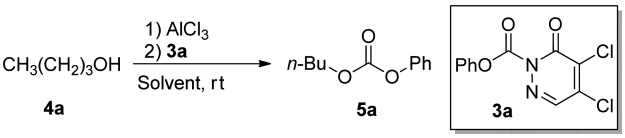
Table 1. Screening of acid for reaction of *n*-butanol with **3a**[a]



Entry	Acid (equiv.)	Time (h)	**5a** Yield (%)[b]
1	$AlCl_3$ (1.5)	3	80
2	$AlCl_3$ (1.0)	3	81
3	$AlCl_3$ (0.5)	20	26
4	$FeCl_3$ (1.5)	3	trace
5	$CuCl_2$ (1.5)	3	trace
6	CuCl (1.5)	3	trace
7	$ZnCl_2$ (1.5)	3	trace
8	$TiCl_4$ (1.5)	3	no reaction
9	$BF_3{\cdot}Et_2O$ (1.5)	3	no reaction
10	HCl (1.5)	3	no reaction
11	H_2SO_4 (1.5)	3	no reaction
12	TFA (1.5)	3	trace
13	TsOH (1.5)	3	trace
14	TfOH (1.5)	3	28

[a]Reaction condition: **4a**/**3a** (1:1 mole ratio) in toluene at room temperature. [b]Isolated yield.

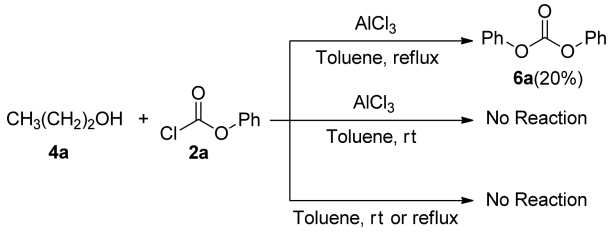
Table 2. Screening of solvent for reaction of *n*-butanol with **3a**[a]



Entry	Solvent	Time (h)	**5a** Yield (%)[b]
1	CH_3CN	8	53
2	CH_2Cl_2	16	31
3	Toluene	3	81
4	*n*-Hexane	18	69
5	THF	14	trace
6	EtOAc	3	50

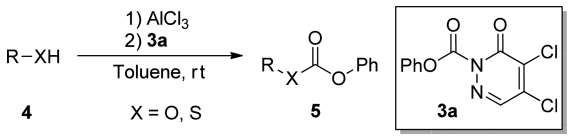
[a]Reaction condition: **4a**/$AlCl_3$/**3a** (1:1:1 mole ratio) at room temperature. [b]Isolated yield.

On the other hand, we evaluated the reactivity of phenyl chloroformate (**2a**) as carbonyl source under our condition. Although reaction of *n*-butanol (**4a**) with **2a** in the presence of $AlCl_3$ in refluxing toluene gave the carbonate **6a** in 20% yield, the reactions did not proceed in the presence of $AlCl_3$ at room temperature or in the absence of $AlCl_3$ at room



Scheme 3. Reaction of phenyl chloroformate (**2a**) with **4a**.

Table 3. Synthesis of unsymmetric organic carbonates[a]



Entry	R-XH, **4**	Time (h)	**5** Yield (%)[b]
1	MeOH	1	**5b** (88)
2	*c*-$C_6H_{11}OH$[c]	2	**5c** (90)
3	$C_6H_5(CH_2)_2OH$	2	**5d** (90)
4	*p*-(Cl)C_6H_4OH	2	**5e** (87)
5	*p*-(CH_3O)C_6H_4OH	2	**5f** (80)
6	*p*-(NO_2)C_6H_4OH	0.5	**5g** (89)
7	*p*-(C_6H_5)C_6H_4OH	1	**5h** (86)
8	*p*-(OH)$C_6H_4(CH_2)_2OH$	12	**5i** (39)[d]
9	C_6H_5SH	3	**5j** (85)

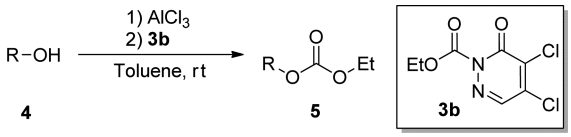
[a]Reaction condition: **4**/$AlCl_3$/**3a** (1:1:1 mole ratio) in toluene at room temperature. [b]Isolated yield. [c]Cyclohexanol. [d]We obtained diphenyl cabonate in 15% yield.

temperature and at reflux temperature in toluene.

Based on the above preliminary experimental data, we selected ROH/$AlCl_3$/**3** (1:1:1 mole ratio) system in toluene at room temperature as the optimized conditions.

To illustrate the versatility of our method, we prepared some unsymmetric organic carbonates using **3a** and alcohols under the optimized conditions. Compound **3a** was reacted with aliphatic and aromatic alcohols in the presence of aluminum chloride in toluene at room temperature to give the corresponding unsymmetric organic carbonate **5b-5h** in 80-90% yields except for 4-(2-hydroxyethyl)phenol (Table 3). Reaction of **3a** with 4-(2-hydroxyethyl)phenol in the presence of $AlCl_3$ under the optimized conditions gave the corresponding carbonate **5i** (39%) and diphenyl carbonate (15%) (Entry 8, Table 3). The long reaction time may be the cause that generated diphenyl carbonate in this reaction. Actually, the mixture of compound **3a** and $AlCl_3$ was stirred for 10 hours at room temperature to give diphenyl carbonate by the decomposition of **3a**. Reaction of **3a** with benzenethiol in the presence of $AlCl_3$ under the optimized conditions also afford the corresponding thiocarbonate **5j** (85%) (Entry 9, Table 3).

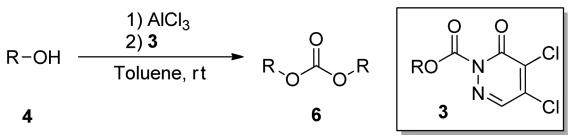
Table 4. Synthesis of unsymmetric organic carbonates[a]



Entry	R	Time (h)	**5** Yield (%)[b]
1	$CH_3(CH_2)_3$	1	**5k** (86)
2	*c*-C_6H_{11}[c]	2	**5l** (84)
3	$C_6H_5(CH_2)_2$	2	**5m** (90)
4	*p*-(Cl)C_6H_4	6	**5n** (82)
5	*p*-(CH_3O)C_6H_4	8	**5o** (83)
6	*p*-(NO_2)C_6H_4	1	**5p** (84)
7	*p*-(C_6H_5)C_6H_4	2	**5q** (86)

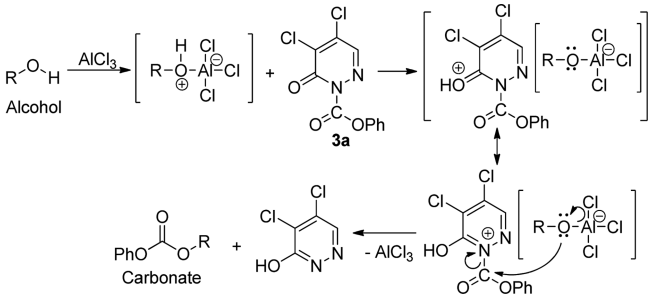
[a]Reaction condition: **4**/$AlCl_3$/**3b** (1:1:1 mole ratio) in toluene at room temperature. [b]Isolated yield. [c]Cyclohexanol.

Table 5. Synthesis of symmetric organic carbonates[a]



Entry	R	Time (min)	**6** Yield (%)[b]
1	C_6H_5	15	**6a** (94)
2	Et	10	**6b** (81)[c]
3	Me	20	**6c** (45)[c]
4	*p*-(Cl)C_6H_4	40	**6d** (94)
5	*p*-(CH_3)C_6H_4	20	**6e** (89)
6	*p*-(CH_3O)C_6H_4	40	**6f** (94)
7	*p*-(NO_2)C_6H_4	120	**6g** (70)

[a]Reaction condition: **4**/$AlCl_3$/**3** (1:1:1 mole ratio) in toluene at room temperature. [b]Isolated yield. [c]We used the corresponding alcohol as the reagent and the solvent.



Scheme 4. Plausible mechanism for the reaction of phenyl 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylate with ROH/$AlCl_3$ systems.

Next, we attempted to prepare from compound **3b** under the same conditions. Reaction of **3b** with some aliphatic and aromatic alcohols in the presence of $AlCl_3$ under the optimized conditions afford the corresponding unsymmetric carbonates **5k-5q** in good yields (Table 4).

On the other hand, we attempted the symmetric organic carbonate by our method. Alkyl/aryl 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylates **3** were reacted with alcohols in the presence of $AlCl_3$ under the optimized conditions to give the corresponding symmetric carbonates **6a-6g** in 45-94% yields.

In all case, we isolated quantitatively 4,5-dichloropyridazin-3(2*H*)-one. The structures of all prepared compounds were established by IR, NMR and HRMS. A plausible mechanism showed in Scheme 4.

In summary, an efficient and versatile method was developed for the synthesis of symmetric and unsymmetric organic carbonates. The reaction was carried out in the presence of $AlCl_3$ in toluene at room temperature, and alkyl/aryl 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylates are used as a CO or CO_2R(Ar) source. It may be considered as a novel type that could use *N*-acylazinone such as carbamate and ROH/$AlCl_3$ system at room temperature for the synthesis of

symmetric and unsymmetric organic carbonates. Our methods are efficient, convenient and practical. It is worthy to note that the reaction use ROH/$AlCl_3$ system and the stable and non-toxic CO/CO_2R(Ar) source, the easy-to prepare and readily available starting materials and the quantitative isolation of reusable 4,5-dichloropyridazin-3(2*H*)-one. We also believe that our methods would be applicable practically to industrial processes.

Experimental

General Methods. Melting points were determined with a capillary apparatus and uncorrected. NMR spectra were recorded on a 300 MHz spectrometer with chemical shift values reported in δ units (ppm) relative to an internal standard (TMS). IR spectra were obtained on a Varian 640-IR spectrophotometer. Mass spectra were recorded under electron ionization (EI). Thin–layer chromatography (TLC) analyses were performed using precoated silica gel plates. The open-bed chromatography was carried out on silica gel (70-230 mesh, Merck) using gravity flow. The column was packed with slurries made from the elution solvent.

General Procedure for the Synthesis of Alkyl/aryl 4,5-dichloro-6-oxopyridazine-1(6*H*)-carboxylate 3. To a solution of 4,5-dichloropyridazin-3(2*H*)-one (**1**, 3.0 mmol) and Et_3N (3.6 mmol) in CH_2Cl_2 (30 mL) was added dropwise the appropriate alkyl/aryl chloroformate (**2**, 3.9 mmol) and the mixture was stirred for 10 min at 5 °C (Scheme 1). The reaction mixture was washed using water (5 × 50 mL). The organic layer was dried over anhydrous magnesium sulfate, and then evaporated under reduced pressure. The resulting residue was recrystallized from THF/*n*-hexane (1:3, v/v) to give the product **3**.

Phenyl 4,5-Dichloro-6-oxopyridazine-1(6*H*)-carboxylate (3a):[17] Yield: 787 mg, 92%; white solid; mp 140 °C (lit. mp 140 °C); IR (KBr): 3074, 3043, 1793, 1681, 1602, 1483, 1272, 1188, 1070, 948, 877, 750 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 7.33-7.41 (m, 3H), 7.50-7.56 (m, 2H), 8.38 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 13.9, 66.3, 136.1, 136.7, 136.9, 150.6, 154.3; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_7H_6Cl_2N_2O_3$: 235.9755; Found: 235.9758.

Ethyl 4,5-Dichloro-6-oxopyridazine-1(6*H*)-carboxylate (3b): Yield: 704 mg, 99%; white solid; mp 78-79 °C; IR (KBr): 3061, 1783, 1683, 1603, 1242 1140, 945, 883, 757 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.46 (t, 3H, *J* = 7.1 Hz), 4.55 (q, 2H, *J* = 7.1 Hz), 7.84 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 13.95, 66.25, 136.13, 136.70, 136.94, 150.69, 154.34; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_7H_6Cl_2N_2O_3$: 235.9755; Found: *m/z* 235.9758.

Methyl 4,5-Dichloro-6-oxopyridazine-1(6*H*)-carboxylate (3c): Yield: 616 mg, 92%; white solid; mp 112 °C; IR (KBr): 3058, 3037, 2957, 1761, 1695, 1599, 1529, 1439, 1260, 1238, 1192, 927, 767 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 4.00 (s, 3H), 8.30 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 55.9, 134.7, 136.5, 137.4, 150.9, 154.0; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_6H_4Cl_2N_2O_3$: 221.9599; Found: 221.9597.

4-Chlorophenyl 4,5-Dichloro-6-oxopyridazine-1(6*H*)-carboxylate (3d): Yield: 824 mg, 86%; white solid; mp 143-144 °C; IR (KBr): 3112, 3073, 2969, 2840, 1789, 1687, 1593, 1505, 1304, 1284, 1230, 1194, 1160, 1106, 1031, 947, 831, 749 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 7.40 (d, 2H, *J* = 9.0 Hz), 7.59 (d, 2H, *J* = 9.0 Hz), 8.39 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 117.3, 122.7, 123.5, 129.6, 133.7, 133.0, 137.1, 156.7, 157.4; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{11}H_5Cl_3N_2O_3$: 317.9366; Found: 317.9362.

***p*-Tolyl 4,5-Dichloro-6-oxopyridazine-1(6*H*)-carboxylate (3e):** Yield: 835 mg, 93%; white solid; mp 107-109 °C; IR (KBr): 3107, 3075, 2951, 2919, 2864, 1793, 1693, 1596, 1502, 1287, 1244, 1194, 1166, 943, 748; ^1H NMR (300 MHz, DMSO-d_6) δ 2.35 (s, 3H), 7.21 (d, *J* = 8.7 Hz), 7.31 (d, *J* = 8.4 Hz), 8.37(s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 20.3, 120.6, 130.2, 134.8, 136.3, 137.7, 147.9, 149.1, 154.1, 156.1; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{12}H_8Cl_2N_2O_3$: 297.9912; Found: 297.9913.

4-Methoxyphenyl 4,5-Dichloro-6-oxopyridazine-1(6*H*)-carboxylate (3f): Yield: 832 mg, 88%; white solid; mp 104-105 °C; IR (KBr): 3110, 3069, 3008, 2969, 2942, 2909, 2839, 1791, 1688, 1594, 1505, 1283, 1230, 1193, 1166, 1105, 1029, 944, 834, 749; ^1H NMR (300 MHz, DMSO-d_6) δ 3.82 (s, 3H), 6.94 (d, 2H, *J* = 9.2 Hz), 7.59 (d, 2H, *J* = 9.2 Hz), 7.90 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 56.0, 115.3, 122.4, 135.3, 136.9, 138.2, 144.0, 149.8, 154.7, 158.1; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{12}H_8Cl_2N_2O_4$: 313.9861; Found: 313.9868.

4-Nitrophenyl 4,5-Dichloro-6-oxopyridazine-1(6*H*)-carboxylate (3g): Yield: 921 mg, 93%; white solid; mp 134-135 °C; IR (KBr): 3119, 3072, 1792, 1688, 1535, 1348, 1198, 1169, 862, 745; ^1H NMR (300 MHz, $CDCl_3$) δ 7.54 (d, 2H, *J* = 9.1 Hz), 7.94 (s, 1H), 8.36 (d, 2H, *J* = 9.1 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 122.0, 125.6, 136.3, 137.3, 137.6, 146.2, 148.4, 154.3, 154.4; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{11}H_5Cl_2N_3O_5$: 328.9606; Found: 328.9602.

General Procedure for the Synthesis of Unsymmetric and Symmetric Carbonates 5 and 6. To a solution of alcohol (or thiol) **4** (0.7 mmol) and $AlCl_3$ (0.7 mmol) in toluene (10 mL), compound **3** (0.7 mmol) was added. The mixture was stirred at room temperature until compound **3** was consumed, as determined by TLC. A 10% aqueous NaOH solution (50 mL) and dichloromethane (30 mL) were added to the reaction mixture with stirring. The organic layer was extracted and washed water (50 mL), and dried over anhydrous magnesium sulfate, and the solvent was evaporated under the reduced pressure. The resulting residue was transferred to an open-bed silica gel column (2.5 × 4 cm). The column was eluted with *n*-hexane/ethyl acetate (3:1, v/v) to isolate compound **5** and **6** and then ethyl acetate to isolate 4,5-dichloropyridazin-3(2*H*)-one (**1**). The column fractions containing pure compound were combined and evaporated under reduced pressure to give the respective product. 4,5-Dichloropyridazin-3(2*H*)-one was obtained quantitative yield and reused.

Butyl Phenyl Carbonate (5a): Yield: 110 mg, 81%; liquid; IR (KBr): 3068, 3041, 2959, 2932, 2870, 1759, 1591, 1490, 1456, 1388, 1250, 1208, 1067, 768 cm^{-1}; ^1H NMR

(300 MHz, $CDCl_3$) δ 0.96 (t, 3H, J = 7.3 Hz), 1.41-1.51 (m, 2H), 1.67-1.77 (m, 2H), 4.25 (t, 2H, J = 6.1 Hz), 7.16-7.25 (m, 3H), 7.34-7.40 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 13.6, 18.9, 30.5, 68.6, 121.0, 125.9, 129.4, 151.1, 153.8; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{11}H_{14}O_3$: 194.0943; Found: 194.0951.

Methyl Phenyl Carbonate (5b): Yield: 94 mg, 88%; liquid; IR (KBr): 3062, 3030, 2957, 2920, 1762, 1592, 1439, 1262, 1215, 1062, 941, 768, 704 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.88 (s, 3H), 7.16-7.26 (m, 3H), 7.35-7.41 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.4, 121.0, 126.1, 129.5, 151.1, 154.3; HRMS (EI) calcd for $C_8H_8O_3$: 152.0473; Found: 152.0475.

Cyclohexyl Phenyl Carbonate (5c): Yield: 139 mg, 90%; white solid; mp 58-60 ºC; IR (KBr): 3064, 3041, 2942, 2860, 1750, 1591, 1491, 1372, 1250, 1205, 1003, 938, 715 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.25-1.58 (m, 6H), 1.76-2.01 (m, 4H), 4.68-4.75 (m, 1H), 7.16-7.25 (m, 3H), 7.37 (t, 2H, J = 7.6 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 23.6, 25.2, 31.4, 77.7, 121.1, 125.8, 129.4, 151.1, 153.1; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{13}H_{16}O_3$: 220.1099; Found: 220.1098.

Phenethyl Phenyl Carbonate (5d):[30] Yield: 153 mg, 90%; white solid; mp 83-85 ºC (lit. mp 89 ºC); IR (KBr): 3109, 3081, 3058, 3033, 2969, 2938, 2895, 2868, 1753, 1492, 1260, 1210, 1077, 967, 778, 753 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.00 (t, 2H, J = 7.0 Hz), 4.38-4.43 (m, 2H), 7.10 - 7.35 (m, 10H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 35.1, 69.1, 121.0, 121.2, 126.1, 126.4, 126.9, 128.7, 129.1, 129.6, 129.7, 137.1, 151.1, 151.2, 153.7; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{15}H_{14}O_3$: 242.0943; Found: 242.0941.

4-Chlorophenyl Phenyl Carbonate (5e):[31] Yield: 151 mg, 87%; white solid; mp 57-59 ºC (lit. mp 82-84 ºC); IR (KBr): 3175, 3068, 1765, 1586, 1480, 1245, 1177, 1069, 1001, 906, 755 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.32-7.61 (m, 9H), 7.42-7.47 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 119.3, 120.8, 120.9, 121.7, 126.3, 126.5, 126.6, 129.6, 129.6, 130.3, 134.8, 150.9, 151.3; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{13}H_9ClO_3$: 248.0240; Found: 248.0238.

4-Methoxyphenyl Phenyl Carbonate (5f): Yield: 136 mg, 80%; white solid; mp 39-40 ºC; IR (KBr): 3065, 3011, 2967, 2932, 2910, 2839, 1769, 1511, 1241, 1179, 1031, 832, 778 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.76 (s, 3H), 6.99-7.02 (m, 2H), 7.30-7.50 (m, 7H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.4, 114.5, 121.2, 122.1, 126.4, 129.6, 144.2, 150.7, 152.0, 157.3; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{14}H_{12}O_4$: 244.0736; Found: 244.0736.

4-Nitrophenyl Phenyl Carbonate (5g):[32] Yield: 162 mg, 89%; white solid; mp 124-125 ºC (lit. mp 127-128 ºC); IR (KBr): 3116, 3087, 2923, 2855, 1764, 1618, 1596, 1527, 1490, 1351, 1274, 1187, 1158, 1008, 854, 752, 686, 496 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.33-7.40 (m, 1H), 7.41-7.44 (m, 2H), 7.46-7.53 (m, 2H), 7.69-7.72 (m, 2H), 8.34-8.40 (m, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 121.1, 122.6, 125.4, 126.6, 129.7, 145.3, 150.5, 150.6, 155.0; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{13}H_9NO_5$: 259.0481; Found: 259.0481.

[1,1'-Biphenyl]-4-yl Phenyl Carbonate (5h): Yield: 175 mg, 86%; white solid. mp 128-131 ºC. IR (KBr): 3062, 3034, 2921, 2853, 1763, 1487, 1241, 1206, 1183, 757, 688 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.32-7.50 (m, 10H) 7.67-7.78 (m, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 121.1, 121.6, 126.4, 126.6, 127.5, 127.8, 128.9, 129.6, 138.3, 139.0, 150.0, 150.5, 151.5; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{10}H_{12}O_3$: 290.0943; Found: *m/z* 290.0945.

***p*-Hydroxyphenyl Phenyl Carbonate (5i).** Yield: 78 mg, 39%; liquid; IR (KBr): 3074, 3043, 1793, 1681, 1602, 1483, 1272, 1188, 1070, 948, 877, 750, 617 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 2.87 (t, 2H, J_1 = 6.6 Hz, J_2 = 6.9 Hz), 4.33 (t, 2H, J_1 = 6.9 Hz, J_2 = 6.8 Hz), 6.69-6.73 (m, 2H), 7.07 (d, 2H, J = 8.4 Hz), 7.18-7.21 (m, 2H), 7.26-7.31 (m, 1H), 7.39-7.45 (m, 2H), 9.27 (s, 1H, D_2O exchangeable); ^{13}C NMR (75 MHz, DMSO-d_6) δ 33.3, 69.1, 115.1, 121.2, 126.0, 127.2, 129.5, 129.8, 150.6, 152.9, 155.9; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{13}H_{10}O_4$: 283.9755; Found: *m/z* 283.9758.

***O,S*-Diphenyl Thiocarbonate (5j):** Yield: 137 mg, 85%; white solid; mp 55 ºC; IR (KBr): 3058, 1731, 1588, 1484, 1440, 1243, 1187, 1160, 1107, 1080, 998, 742 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.26-7.33 (m, 3H), 7.42-7.51 (m, 5H), 7.65-7.68 (m, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 121.8, 126.8, 127.0, 130.0, 130.1, 130.2, 130.6, 135.2, 151.2, 168.4; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{13}H_{10}O_2S$: 230.0402; Found: 230.0403.

Butyl Ethyl Carbonate (5k):[33] Yield: 88 mg, 86%; liquid; IR (KBr): 2961, 2934, 2873, 1746, 1462, 1401, 1257, 1061, 1016, 934, 791 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.94 (t, 3H, J = 7.4 Hz), 1.26-1.48 (m, 5H), 1.60-1.69 (m, 2H), 4.10-4.22 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 13.6, 14.2, 18.9, 30.6, 63.7, 67.6, 155.4; MS (EI, 70 eV) *m/z*: 119 $[M]^+$ (6), 118 $[M-H]^+$ (8), 91 (8), 73 (12), 63 (31), 57 (100), 56 (46).

Cyclohexyl Ethyl Carbonate (5l):[34] Yield: 101 mg, 84%; liquid; IR (KBr): 2938, 2860, 1739, 1453, 1374, 1318, 1253, 1175, 1121, 1098, 1034, 1013, 939, 896, 792 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.29-1.51 (m, 9H), 1.75 (s, 2H), 1.92 (s, 2H), 4.15-4.19 (m, 2H), 4.55-4.61 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.2, 23.6, 23.7, 24.7, 25.2, 31.5, 31.6, 63.5, 154.6; MS (EI, 70 eV) *m/z*: 99 $[M\text{-}CH_3CH_2CO]^+$ (48), 91 (97), 83 $[M\text{-}CH_3CH_2OCO_2]^+$ (69), 82 $[M\text{-}CH_3CH_2OCO_2\text{-}H]^+$ (83), 81 (25), 67 (100), 63 (43), 57 (71), 55 (67).

Ethyl Phenethyl Carbonate (5m). Yield: 122 mg, 84%; liquid; IR (KBr): 3063, 3028, 2982, 2910, 1744, 1456, 1400, 1258, 1201, 1088, 1005, 790, 749, 700 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.27 (t, 3H, J = 7.1 Hz), 2.96 (t, 2H, J = 6.8 Hz), 4.16 (q, 2H, J = 7.1 Hz), 4.32 (t, 2H, J = 7.1 Hz), 7.21-7.31 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.2, 35.2, 63.9, 68.1, 126.6, 128.5, 128.9, 137.3, 155.1; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{11}H_{14}O_3$: 194.0943; Found: 194.0969.

4-Chlorophenyl Ethyl Carbonate (5n): Yield: 115 mg, 82%; liquid; IR (KBr): 3075, 2986, 2938, 1763, 1590, 1474, 1429, 1369, 1247, 1215, 1063, 994, 860, 780 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 1.30 (t, 3H, J = 7.1 Hz), 4.26 (q, 2H, J = 7.1 Hz), 7.23-7.27 (m, 1H), 7.36-7.39 (m, 1H), 7.44-7.50 (m, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 13.9, 64.8, 120.2, 121.8, 126.2, 130.9, 133.3, 151.3, 152.5; HRMS (EI)

m/z: $[M]^+$ calcd for $C_9H_9ClO_3$: 200.0240; Found: 200.0242.

Ethyl 4-Methoxyphenyl Carbonate (5o): Yield: 114 mg, 83%; liquid; IR (KBr): 3063, 2984, 2901, 1755, 1488, 1374, 1307, 1259, 1222, 1174, 1090, 972, 841, 762, 730 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.35 (t, 3H, *J* = 7.1 Hz), 3.75 (s, 3H), 4.28 (q, 2H, *J* = 7.1 Hz), 6.83-6.89 (m, 2H), 7.05-7.11 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.1, 55.5, 64.6, 114.4, 121.9, 144.7, 154.0, 157.3; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{10}H_{12}O_4$: 196.0736; Found: 196.0741.

Ethyl 4-Nitrophenyl Carbonate (5p):[35] Yield: 124 mg, 84%; white solid; mp 75-76 ºC (lit. mp 67-68 ºC); IR (KBr): 3118, 3085, 3004, 2923, 2855, 1758, 1521, 1378, 1352, 1283, 1230, 1001, 857 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 1.31 (t, 3H, *J* = 7.1 Hz), 4.30 (q, 2H, *J* = 7.1 Hz), 7.54-7.60 (m, 2H), 8.30-8.34 (m, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 14.3, 65.7, 123.0, 125.8, 145.6, 152.4, 155.7; HRMS (EI) *m/z*: $[M]^+$ calcd. for $C_9H_9NO_5$; 211.0481; Found: 211.0481.

[1,1'-Biphenyl]-4-yl Ethyl Carbonate (5q):[36] Yield: 146 mg, 86%; white solid; mp 75-76 ºC (lit. mp 74-74.5 ºC); IR (KBr): 3063, 2984, 1755, 1488, 1374, 1307, 1259, 1222, 1090, 1055, 973, 842, 762, 730 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 1.31 (t, 3H, *J* = 6.0 Hz), 4.28 (q, 2H, *J* = 6.0 Hz), 7.32-7.48 (m, 5H), 7.66-7.73 (m, 4H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 14.4, 65.1, 122.2, 127.1, 128.0, 128.3, 129.4, 138.5, 139.7, 150.7, 153.4; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{15}H_{14}O_3$: 242.0943; Found: 242.0941.

Diphenyl Carbonate (6a):[37] Yield: 141 mg, 94%; white solid; mp 75-76 ºC (lit. mp 77-78 ºC); IR (KBr): 3058, 1773, 1592, 1490, 1255, 1233, 1182, 1071, 1016, 996, 751 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 7.30 - 7.35 (m, 2H), 7.40-7.51 (m, 8H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 121.2, 126.4, 129.7, 150.7, 151.7; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{13}H_{10}O_3$: 214.0630; Found: 214.0634.

Diethyl Carbonate (6b):[33] Yield: 67 mg, 81%; liquid; IR (KBr): 2987, 2940, 2913, 1748, 1470, 1408, 1375, 1271, 1093, 1022, 855, 792 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.30 (t, 6H, *J* = 7.2 Hz), 4.19 (q, 4H, *J* = 7.2 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 13.9, 63.4, 154.9; MS (EI, 70 eV) *m/z*: 91 $[M+H]^+$ (61), 90 $[M]^+$ (3), 75 (3), 63 (20), 59 (6), 45 (100), 31 (58), 30 (3), 29 (90), 28 (13), 27 (28).

Dimethyl Carbonate (6c): Yield: 28 mg, 45%; liquid; IR (KBr): 3009, 2967, 2928, 2861, 1774, 1457, 1295, 989 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 3.79 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 54.5, 156.2; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_3H_6O_3$: 90.0317; Found: 90.0314.

Bis(4-chlorophenyl) Carbonate (6d):[38] Yield: 187 mg, 94%; white solid; mp 146-147 ºC (lit. 144-146 ºC); IR (KBr): 3102, 3077, 1769, 1491, 1289, 1264, 1089, 822, 783 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.20-7.23 (m, 4H), δ 7.20-7.23 (m, 4H), 7.37-7.40 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 122.2, 129.7, 131.9, 149.3, 151.5; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{13}H_8Cl_2O_3$: 281.9850; Found: 281.9851.

Di-*p*-tolyl Carbonate (6e):[39] Yield: 158 mg, 94%; white solid; mp 109-110 ºC (lit. mp 111-112 ºC); IR (KBr): 3039, 2923, 2858, 1772, 1507, 1240, 1177, 1156, 1011, 886, 812, 769 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 2.34 (s, 6H), 7.12 - 7.20 (m, 8H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 20.8, 120.6, 130.0, 135.9, 148.9, 152.4; HRMS (EI) *m/z*: $[M]^+$ calcd for $C_{15}H_{14}O_3$: 242.0943; Found: 242.0945.

Bis(4-methoxyphenyl) Carbonate (6f).[40] Yield: 171 mg, 89%; white solid; mp 94-95 ºC (lit. mp 96-97 ºC); IR (KBr): 3110, 3066, 3027, 2971, 2933, 2838, 1768, 1601, 1508, 1458, 1305, 1274, 1238, 1176, 1099, 1024, 826, 775 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 3.80 (s, 6H), 6.90 (d, 4H, *J* = 9.0 Hz), 7.18 (d, 4H, *J* = 9.0 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.6, 114.5, 121.7, 144.6, 152.8, 157.5; HRMS (EI) *m/z*: [M]+ calcd for $C_{15}H_{14}O_5$: 274.0841; Found: *m/z* 274.0840.

Bis(4-nitrophenyl) Carbonate (6g):[40] Yield: 139 mg, 70%; white solid; mp 137-139 ºC (lit. mp 138-140 ºC); IR (KBr): 3114, 3079, 1765, 1617, 1526, 1360, 1371, 1245, 1194, 1161, 1106, 1009, 891, 859, 745 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 7.50 (d, 4H, *J* = 9.1 Hz), 8.34 (d, 4H, *J* = 9.1 Hz); ^{13}C NMR (75 MHz, DMSO-d_6) δ 121.6, 125.3, 125.5, 128.2, 129.0, 145.9, 150.0, 154.8; HRMS (EI) *m/z*: [M]+ calcd for $C_{13}H_8N_2O_7$: 304.0332; Found: *m/z* 304.0331.

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