Synthesis of Organic Carbonates with Alkyl/aryl 4,5-dichloro-6-oxypyridazine-1(6H)-carboxylates and ROH/AlCl₃ under Ambient Condition

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We demonstrated the synthesis of organic carbonates using alkyl/aryl 4,5-dichloro-6-oxypyridazine-1(6H)-carboxylates and alcohol in the presence of aluminum chloride. Alkyl/aryl 4,5-dichloro-6-oxopyridazine-1(6H)-carboxylates were reacted with alcohol in the presence of AlCl₃ 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(6H)-carboxylates are solid, stable and non-toxic CO₂/CO₂R(Ar) source. It is noteworthy that the reaction is carried 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(2H)-one.

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

Introduction

Organic carbonates are generally safe noncorrosive molecules employed in numerous commercial and synthetic application as eco-friendly useful reagents, solvents for Li-ion battery and electroanalytics. The symmetric organic carbonates [(RO)₂C=O] are useful as the solvents, whereas the unsymmetric organic carbonates [ROC(O)=O] are used as the key-functional group in drugs and other chemicals. Various synthetic methods of organic carbonates by the phosgenation technique using COCl₂, the oxidative carbonylation of alcohols using CO and transition metals, the reaction of urea with alcohols, the reaction of oxiranes and CO₂, the reaction of chlorofornates, the use of metal carbonate and the organic carbonate interchange reaction have been reported. However, the main disadvantages of these methods are the use of toxic, gaseous and/or expensive chemicals and requirement for specific additives. The alkoxy carbonylation using organic carbonate and base be also accompanied by the undesired side reaction. 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 CO₂ or CO₂R(Ar) source under non-basic conditions. To avoid the side reaction in the reaction using organic carbonate, the alkoxy or alkoxide equivalent must be prepared under aprotic acid or neutral condition.

Romano et al. reported the synthesis of dimethyl carbonate by oxidative carbonylation of methanol using copper chloride via Cu(OCH₃)Cl intermediate (Scheme 1). In this reaction, the Cu(OCH₃)Cl acts as an equivalent of methoxide (MeO⁻).

On the other hand, Ball et al. 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(6H)-carboxylates have a carbamate functionality. Thus, alkyl/aryl 6-oxopyridazine-1(6H)-carboxylates may be used as alkoxy/aryloxy carbonyl source, and also the 4,5-dichloropyridazin-3(2H)-one anion as the leaving group may be act as a proton acceptor during the reaction. Pyridazin-3(2H)-ones are inexpensive, very stable and good leaving group, and also can be removed and/or recycled spurred our interest in their use for other transformation according to Yoon et al.

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

Scheme 1. Known and newly designed methods for the synthesis of organic carbonates.
not use in basic condition because of the side reaction.\textsuperscript{25-27} Thus, we required an acidic condition for the synthesis of carbonate from alkyl/aryl 4,5-dichloro-6-oxopyridazine-1(6H)-carboxylates and alcohols.

Inspired by the oxidative carbonylation\textsuperscript{1,15,28} and the method of carbamate reaction,\textsuperscript{1,16} we attempted to develop a novel convenient synthetic method for unsymmetric and symmetric organic carbonates from alkyl(or aryl) 6-oxopyridazine-1(6H)-one carboxylate as a carbamate and ROH in the presence of AlCl\textsubscript{3} (Scheme 1).

Herein, we report the synthesis of organic carbonates using ROH/AlCl\textsubscript{3} systems and alkyl(or aryl) 4,5-dichloro-6-oxopyridazine-1(6H)-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\textsubscript{3} system acting alkoxide equivalent. First of all, we selected aluminum chloride as the Lewis acid. Although the reaction of ROH (3 equiv.) with AlCl\textsubscript{3} (1 equiv.) yields the corresponding aluminum alkoxides [Al(OR)\textsubscript{3}]\textsuperscript{−},\textsuperscript{29} (ROH-AlCl\textsubscript{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\textsubscript{3}) adducts in the solvent, the residue [(ROAlCl\textsubscript{3})\textsuperscript{−}] may 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.

\[
3\text{ROH} + \text{AlCl}_3 \rightarrow \{\text{ROH}^+\}^+\{\text{AlCl}_2^−\} \rightarrow \{\text{RO}_2\text{Al} + 3 \text{HCl}
\]

Alkyl(or aryl) 4,5-dichloro-6-oxopyridazine-1(6H)-carboxylates 3 were prepared by the literature method\textsuperscript{17} from 4,5-dichloropyridazin-3(2H)-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 3-butanol in the presence of Lewis acids or protic acids with phenyl 4,5-dichloro-6-oxopyridazine-1(6H)-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\textsubscript{3} system. Toluene also showed the best results among the six solvents tested (Entry 3, Table 2).

<table>
<thead>
<tr>
<th>Entry</th>
<th>Acid (equiv.)</th>
<th>Time (h)</th>
<th>5a Yield (%)\textsuperscript{a}</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>AlCl\textsubscript{3} (1.5)</td>
<td>3</td>
<td>80</td>
</tr>
<tr>
<td>2</td>
<td>AlCl\textsubscript{3} (1.0)</td>
<td>3</td>
<td>81</td>
</tr>
<tr>
<td>3</td>
<td>AlCl\textsubscript{3} (0.5)</td>
<td>20</td>
<td>26</td>
</tr>
<tr>
<td>4</td>
<td>FeCl\textsubscript{3} (1.5)</td>
<td>3</td>
<td>trace</td>
</tr>
<tr>
<td>5</td>
<td>CuCl\textsubscript{2} (1.5)</td>
<td>3</td>
<td>trace</td>
</tr>
<tr>
<td>6</td>
<td>CuCl (1.5)</td>
<td>3</td>
<td>trace</td>
</tr>
<tr>
<td>7</td>
<td>ZnCl\textsubscript{2} (1.5)</td>
<td>3</td>
<td>28</td>
</tr>
<tr>
<td>8</td>
<td>TiCl\textsubscript{3} (1.5)</td>
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<td>no reaction</td>
</tr>
<tr>
<td>9</td>
<td>BF\textsubscript{3}Et2O (1.5)</td>
<td>3</td>
<td>no reaction</td>
</tr>
<tr>
<td>10</td>
<td>HCl (1.5)</td>
<td>3</td>
<td>no reaction</td>
</tr>
<tr>
<td>11</td>
<td>H\textsubscript{2}SO\textsubscript{4} (1.5)</td>
<td>3</td>
<td>no reaction</td>
</tr>
<tr>
<td>12</td>
<td>TFA (1.5)</td>
<td>3</td>
<td>trace</td>
</tr>
<tr>
<td>13</td>
<td>TsOH (1.5)</td>
<td>3</td>
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</tr>
<tr>
<td>14</td>
<td>TfOH (1.5)</td>
<td>3</td>
<td>28</td>
</tr>
</tbody>
</table>

\textsuperscript{a}Reaction condition: 4a/3a (1:1 mole ratio) in toluene at room temperature. \textsuperscript{b}Isolated yield.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Solvent</th>
<th>Time (h)</th>
<th>5a Yield (%)\textsuperscript{a}</th>
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<tr>
<td>1</td>
<td>CH\textsubscript{3}CN</td>
<td>8</td>
<td>53</td>
</tr>
<tr>
<td>2</td>
<td>CH\textsubscript{2}Cl\textsubscript{2}</td>
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<td>31</td>
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<tr>
<td>3</td>
<td>Toluene</td>
<td>3</td>
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<td>4</td>
<td>n-Hexane</td>
<td>18</td>
<td>69</td>
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<tr>
<td>5</td>
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<td>trace</td>
</tr>
<tr>
<td>6</td>
<td>EtOAc</td>
<td>3</td>
<td>50</td>
</tr>
</tbody>
</table>

\textsuperscript{a}Reaction condition: 4a/AlCl\textsubscript{3}/3a (1:1:1 mole ratio) at room temperature. \textsuperscript{b}Isolated yield.

On the other hand, we evaluated the reactivity of phenyl chloroformate 2a as carbonyl source under our condition. Although reaction of 3-butanol (4a) with 2a in the presence of AlCl\textsubscript{3} in refluxing toluene gave the carbonate 6a in 20% yield, the reactions did not proceed in the presence of AlCl\textsubscript{3} at room temperature or in the absence of AlCl\textsubscript{3} at room

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**Scheme 2.** Synthesis of symmetric and unsymmetric organic carbonates using alcohol-AlCl\textsubscript{3} adducts.

**Scheme 3.** Reaction of phenyl chloroformate 2a with 4a.
temperature and at reflux temperature in toluene.

Based on the above preliminary experimental data, we selected ROH/AlCl\(_3\) (1:1:1 mole ratio) system in toluene at room temperature as the optimized conditions.

Table 3. Synthesis of unsymmetric organic carbonates

<table>
<thead>
<tr>
<th>Entry</th>
<th>R-XH, ROH</th>
<th>Time (h)</th>
<th>Yield (%)</th>
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<tbody>
<tr>
<td>1</td>
<td>MeOH</td>
<td>1</td>
<td>5b (88)</td>
</tr>
<tr>
<td>2</td>
<td>c-C(_6)H(_11)OH</td>
<td>2</td>
<td>5c (90)</td>
</tr>
<tr>
<td>3</td>
<td>C(_6)H(_5)(CH(_2)_3)OH</td>
<td>2</td>
<td>5d (90)</td>
</tr>
<tr>
<td>4</td>
<td>(p)-(Cl)C(_6)H(_4)OH</td>
<td>2</td>
<td>5e (87)</td>
</tr>
<tr>
<td>5</td>
<td>(p)-(CH(_3)O)C(_6)H(_4)OH</td>
<td>2</td>
<td>5f (80)</td>
</tr>
<tr>
<td>6</td>
<td>(p)-(NO(_2))C(_6)H(_4)OH</td>
<td>0.5</td>
<td>5g (89)</td>
</tr>
<tr>
<td>7</td>
<td>(p)-(C(_6)H(_5))C(_6)H(_4)OH</td>
<td>1</td>
<td>5h (86)</td>
</tr>
<tr>
<td>8</td>
<td>(p)-(OH)C(_6)H(_5)(CH(_2)_3)OH</td>
<td>12</td>
<td>5i (39)</td>
</tr>
<tr>
<td>9</td>
<td>C(_6)H(_5)SH</td>
<td>3</td>
<td>5j (85)</td>
</tr>
</tbody>
</table>

*Reaction condition: 4/AlCl\(_3\)/3a (1:1:1 mole ratio) in toluene at room temperature. *Isolated yield. *Cyclohexanol. *We obtained diphenyl carbonate in 15% yield.

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 benzene-thiol in the presence of AlCl\(_3\) under the optimized conditions also afforded the corresponding thiocarbonate 5j (85%) (Entry 9, Table 3).

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 afforded 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(6H)-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(2H)-one. The structures of all prepared compounds were established by IR, NMR and HRMS. A plausible mechanism showed in Scheme 4.

Scheme 4. Plausible mechanism for the reaction of phenyl 4,5-dichloro-6-oxopyridazine-1(6H)-carboxylate with ROH/AlCl\(_3\) systems.

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(6H)-carboxylates are used as a CO or CO\(_2\)R(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/AlCl₃ system and the stable and non-toxic CO₂ system (Ar) source, the easy-to-prepare and readily available starting materials and the quantitative isolation of reusable 4,5-dichloropyridazin-3(2H)-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. Electron ionization (EI). Thin–layer chromatography (TLC) was carried out on silica gel 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. Electron ionization (EI). Thin–layer chromatography (TLC) was carried out on silica gel 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. Electron ionization (EI). Thin–layer chromatography (TLC) was carried out on silica gel 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. Electron ionization (EI). Thin–layer chromatography (TLC) was carried out on silica gel 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. Electron ionization (EI). Thin–layer chromatography (TLC) was carried out on silica gel 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. Electron ionization (EI). Thin–layer chromatography (TLC) was carried out on silica gel 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. Electron ionization (EI). Thin–layer chromatography (TLC) was carried out on silica gel 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. Electron ionization (EI). Thin–layer chromatography (TLC) was carried out on silica gel 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. Electron ionization (EI). Thin–layer chromatography (TLC) was carried out on silica gel capillary apparatus and uncorrected.

**General Procedure for the Synthesis of Alkyl/aryl 4,5-dichloropyridazin-3(2H)-one (3a):** Yield: 787 mg, 92%; 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-d₆) δ 7.21 (s, 3H), 7.31 (d, J = 8.4 Hz), 7.31 (d, J = 8.4 Hz), 8.37 (s, 1H); 13C NMR (75 MHz, DMSO-d₆) δ 20.3, 115.3, 122.4, 135.3, 135.4, 138.2, 144.0, 149.8, 154.7, 158.1; HRMS (EI) m/z: [M⁺] calcd for C₁₂H₁₀Cl₂N₀₂O: 297.9912; Found: 297.9913.

**4-Methoxyphenyl 4,5-dichloropyridazin-3(2H)-one (3b):** 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-d₆) δ 7.40 (d, J = 8.7 Hz), 7.59 (d, J = 9.2 Hz), 7.94 (s, 1H), 8.36 (d, J = 9.2 Hz), 7.59 (s, 1H); 13C NMR (75 MHz, DMSO-d₆) δ 56.0, 115.3, 122.4, 135.3, 135.4, 138.2, 144.0, 149.8, 154.7, 158.1; HRMS (EI) m/z: [M⁺] calcd for C₁₂H₁₀Cl₂N₀₂O: 313.9861; Found: 313.9868.

**4-Methylphenyl 4,5-dichloropyridazin-3(2H)-one (3c):** Yield: 110 mg, 81%; liquid; IR (KBr): 3068, 3041, 2959, 2932, 2870, 1759, 1591, 1490, 1456, 1388, 1250, 1067, 768 cm⁻¹; 1H NMR

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Methyl Phenyl Carbonate (5b): Yield: 94 mg, 88%; white solid; mp 39-40 °C; IR (KBr): 3063, 3024, 2983, 1763, 1487, 1241, 1206, 1183, 757, 688 cm⁻¹; 1H NMR (300 MHz, DMSO-d₆) δ 7.16-7.25 (m, 3H), 1.33-1.40 (m, 2H), 0.90-0.97 (t, 3H, J = 7.1 Hz), 7.14 (d, 1H, J = 6.8 Hz). 13C NMR (75 MHz, CDCl₃) δ 130.0, 124.9, 121.6, 126.7, 127.5, 127.8, 128.9, 129.6, 138.3, 139.0, 150.0, 150.5, 151.3, 152.5; HRMS (EI) m/z: [M]+ calecd for C₁₈H₂₃O₂: 290.0943; Found: m/z 290.0945.

-p-Hydroxyphenyl Phenyl Carbonate (5i): Yield: 78 mg, 39%; white solid; mp 55-56 °C; IR (KBr): 3058, 3047, 1739, 1600, 1272, 1188, 1070, 948, 877, 750, 617 cm⁻¹; 1H NMR (300 MHz, DMSO-d₆) δ 7.28-7.32 (m, 2H, J = 7.1 Hz), 7.04 (d, 1H, J = 7.1 Hz), 6.99 (d, 1H, J = 6.8 Hz). 13C NMR (75 MHz, CDCl₃) δ 126.7, 126.8, 127.0, 130.0, 130.1, 130.2, 130.6, 135.2, 151.2, 168.4; HRMS (EI) m/z: [M]+ calecd for C₁₈H₁₇O₃S: 283.0402; Found: 283.0403.

O,S-Diphenyl Thiocarbonate (5j): Yield: 137 mg, 85%; white solid; mp 55 °C; IR (KBr): 3058, 3171, 1588, 1484, 1440, 1243, 1187, 1160, 1107, 1080, 998, 742 cm⁻¹; 1H NMR (300 MHz, DMSO-d₆) δ 7.28-7.33 (m, 3H, J = 7.1 Hz), 7.05-7.10 (m, 3H, J = 6.8 Hz), 7.10-7.20 (m, 3H, J = 6.8 Hz). 13C NMR (75 MHz, CDCl₃) δ 123.3, 123.4, 127.8, 129.9, 139.8, 150.5, 152.9, 155.9; HRMS (EI) m/z: [M]+ calecd for C₁₈H₁₇O₃S: 283.0497; Found: m/z 283.0475.

1H NMR (300 MHz, CDCl3): δ 1.35 (t, J = 7.1 Hz), 3.75 (s, 3H), 4.28 (q, 2H, J = 7.1 Hz), 6.38-6.89 (m, 2H), 7.05-7.11 (m, 2H); 13C NMR (75 MHz, CDCl3): δ 11.41, 11.44, 120.7, 144.7, 154.0, 157.3; HRMS (EI) m/z: [M]+ calec for C14H12O2: 196.0736; Found: 196.0741.

1H NMR (300 MHz, CDCl3): δ 1.30 (t, 3H, J = 7.1 Hz), 3.38 (q, 2H, J = 7.1 Hz), 7.24-7.30 (m, 1H), 7.38 (m, 1H), 7.42 (m, 1H); 13C NMR (75 MHz, CDCl3): δ 27.8, 31.8, 98.6, 129.7, 129.8, 131.9, 142.2, 154.0; HRMS (EI) m/z: [M]+ calec for C16H15NO2: 258.1084; Found: 258.1082.

1H NMR (300 MHz, CDCl3): δ 1.30 (t, 3H, J = 7.1 Hz), 3.38 (q, 2H, J = 7.1 Hz), 7.24-7.30 (m, 1H), 7.38 (m, 1H), 7.42 (m, 1H); 13C NMR (75 MHz, CDCl3): δ 27.8, 31.8, 98.6, 129.7, 129.8, 131.9, 142.2, 154.0; HRMS (EI) m/z: [M]+ calec for C16H15NO2: 258.1084; Found: 258.1082.

1H NMR (300 MHz, CDCl3): δ 1.30 (t, 3H, J = 7.1 Hz), 3.38 (q, 2H, J = 7.1 Hz), 7.24-7.30 (m, 1H), 7.38 (m, 1H), 7.42 (m, 1H); 13C NMR (75 MHz, CDCl3): δ 27.8, 31.8, 98.6, 129.7, 129.8, 131.9, 142.2, 154.0; HRMS (EI) m/z: [M]+ calec for C16H15NO2: 258.1084; Found: 258.1082.

1H NMR (300 MHz, CDCl3): δ 1.30 (t, 3H, J = 7.1 Hz), 3.38 (q, 2H, J = 7.1 Hz), 7.24-7.30 (m, 1H), 7.38 (m, 1H), 7.42 (m, 1H); 13C NMR (75 MHz, CDCl3): δ 27.8, 31.8, 98.6, 129.7, 129.8, 131.9, 142.2, 154.0; HRMS (EI) m/z: [M]+ calec for C16H15NO2: 258.1084; Found: 258.1082.

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