Palladium-Catalysed Reductive Aminocarbonylation of Aryl Bromides and Iodides with Nitroarenes

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Abstract
Amide functional groups are a structural feature in a vast array of beneficial organic molecules. This has resulted in a surge in new methodologies developed to enable access to this functional group using a broad range of coupling partners. Herein, we report a palladium-catalysed reductive aminocarbonylation of aryl bromides and iodides with nitroarenes to afford the respective amide products. The developed protocol employs Mo(CO)₆ as a carbonyl source and a combination of Zn and TMSCl as co-reducing agents. For most substrates, the anticipated amide products were obtained in modest to high amide product yields.

Key words
amides, reductive aminocarbonylation, palladium, nitroarenes, C–N bond formation

The amide functional group is present in several interesting compounds, including natural products, peptides and agrochemicals.1–3 Furthermore, this functional group is found to be present in two-thirds of drug candidates and in over 25% of commercially available drugs.4,5 Some of the drugs are highlighted in Figure 1 and these include moclobemide (1), used for the treatment of depression, nevirapine (2), an antiretroviral drug for HIV, valsartan (3), which is known as a treatment for hypertension and cardiovascular diseases, and paracetamol (4), which is a commonly used analgesic.6–9

Thus, research and development of new amide synthesis procedures has become a key research area due to the prevalence of amide functional groups in beneficial organic molecules.10,11 Traditionally, amides are synthesised from the condensation of carboxylic acids and amines. However, some of these methods are associated with drawbacks, such as using stoichiometric amounts of carboxylic acid activators. These usually generate undesired products in high quantities.12,13 Furthermore, one of the commonly used activators, 1-[bis(dimethylamino)methylene]-1H-1,2,3-triazolo[4,5-b]pyridinium 3-oxide hexafluorophosphate (hexafluorophosphate azabenzotriazole tetramethyl uronium, HATU), has been reported to cause allergies, raising health concerns.14 Protocols using alternative functional groups instead of carboxylic acids have been established to try to overcome some of these challenges and these include reactions that utilise catalytic activators, esters, aldehydes, alcohols and amides as amine coupling partners.15–18 The direct use of nitroarenes as arylamine surrogates has also gained momentum in the amidation reactions as they offer step-economic access to amides.19,20 Nitroarenes are cheaper and more stable than their amine analogues and are ideal for

Figure 1
Commercially available amide-containing drugs

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late-stage transformation in multistep syntheses due to their ‘chemical inertness’. They have been successfully coupled to various functional groups such as esters, carboxylic acids, amides, aldehydes, aryl iodides and alcohols to afford the respective amide products.\(^{21-26}\) In addition, amides have been obtained from reactions between nitroarenes and aryl halides (or equivalent) in the presence of a carbonyl source, and in most cases, nickel was employed as the catalyst of choice.\(^{27-29}\) Although nickel offers a more sustainable option, the general superior robustness of palladium catalysts in cross-coupling reactions prompted us to explore herein an optimisation study for a palladium-catalysed reductive aminocarbonylation of aryl halides with nitroarenes and a subsequent application of the protocol in the synthesis of a variety of amides.\(^{30}\)

Our optimal reaction conditions revealed that palladium(II) acetate [Pd(OAc)\(_2\)] catalyst, 1,3-bis(diphenylphosphino)propane (dppp) as a supporting ligand for the metal, molybdenum hexacarbonyl [Mo(CO)\(_6\)] as a carbonyl source, pyridine as a base and Zn/TMSCl as additives were essential for the successful coupling of the model substrates, 4-bromoanisole (5\(_a\)) and nitrobenzene (6\(_a\)), to afford the amide product 7\(_a\) in 95% yield at 130 °C (Table 1, entry 1). Alternative inorganic and organic bases did not afford better yields in comparison to pyridine (entries 2–7). For example, the use of NaOH, Cs\(_2\)CO\(_3\) and K\(_2\)CO\(_3\) resulted in lowered product yields (entries 2–4). In comparison to the inorganic bases, Et\(_3\)N, quinoline and 2-quinolone afforded slightly improved yields of 50%, 63% and 55%, respectively (entries 5–7). The stronger basicity of pyridine in comparison to its derivatives quinoline and 2-quinolone could be the reason for the observed reactivity trend.\(^{31,32}\) Although pyridine has been reported as a suitable ligand in other studies, such as the C–H activation reaction, phosphine-free reactions in our case afforded poor yields (entry 8).\(^{33}\) Monodentate ligand 2-dicyclohexylphosphino-2',6',6'-dimethoxy-1,1'-biphenyl (SPhos) and bidentate ligand 1,2-bis(diphenylphosphino)ethane (dppe) were also investigated as suitable alternative ligands. However, these did not outperform dppp as lower amide product yields of 54% and 60% for dppe and SPhos, respectively, were obtained (entries 9 and 10).

Other palladium catalysts were also explored and, in our case, Pd(acac)\(_2\), PdCl\(_2\) and PdCl\(_2\)(cod) afforded low yields to no product (entries 11–13). A different choice of a metal catalyst, NiCl\(_2\), showed no reactivity as no amide product was formed (entry 14). Furthermore, different solvents were investigated and these included CH\(_3\)CN, DMF and THF which all gave diminished product yields of 23%, 32% and 43%, respectively (entries 15–17). The use of NMP afforded no amide product at all while DMSO only gave trace amounts of the amide product (entries 18 and 19). Lastly, reducing the reaction temperature to below 130 °C afforded trace amounts of the amide product.

### Table 1 Optimisation Study for the Reductive Aminocarbonylation Reaction

<table>
<thead>
<tr>
<th>Entry</th>
<th>Change in reaction conditions</th>
<th>Product yield (%)</th>
<th>a</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>none</td>
<td>95</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>NaOH instead of pyridine</td>
<td>25</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>K(_2)CO(_3) instead of pyridine</td>
<td>11</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Cs(_2)CO(_3) instead of pyridine</td>
<td>15</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Et(_3)N instead of pyridine</td>
<td>50</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>quinoline instead of pyridine</td>
<td>63</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>2-quinolone instead of pyridine</td>
<td>55</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>no ligand</td>
<td>12</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>dppe instead of dppp</td>
<td>54</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>SPhos instead of dppp</td>
<td>60</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>Pd(acac)(_2) instead of Pd(OAc)(_2)</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>PdCl(_2) instead of Pd(OAc)(_2)</td>
<td>14</td>
<td></td>
</tr>
<tr>
<td>13</td>
<td>PdCl(_2)(cod) instead of Pd(OAc)(_2)</td>
<td>16</td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>NiCl(_2) instead of Pd(OAc)(_2)</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>CH(_3)CN instead of dioxane</td>
<td>23</td>
<td></td>
</tr>
<tr>
<td>16</td>
<td>THF instead of dioxane</td>
<td>43</td>
<td></td>
</tr>
<tr>
<td>17</td>
<td>DMF instead of dioxane</td>
<td>32</td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>NMP instead of dioxane</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td>19</td>
<td>DMSO instead of dioxane</td>
<td>trace</td>
<td></td>
</tr>
</tbody>
</table>

a Dash (–) indicates no amide product formed.

The substrate scope and limitations of our protocol were then explored by varying the aryl halide and nitroarene (Scheme 1). Firstly, we investigated the reactivities of variously substituted aryl bromides with nitrobenzene (6\(_a\)). Substituted aryl bromides containing an electron-donating group (EDG) afforded products 7b–7d and 7g in good to excellent yields. However, product 7e was obtained in a lower product yield and the anticipated product 7f was not formed due to the possible steric hindrance presented by the ortho-substituted aryl bromide utilised. Aryl bromides with a methylene- or ethylenedioxy substituent afforded products 7i and 7j in 37% and 59% yield, respectively. Aryl bromides possessing an electron-withdrawing group (EWG) were less reactive than those with an EDG, and in some cases completely inactive. This could be due to a reduced inductive stabilisation of the intermediate complexes formed between the metal, carbonyl, halide, ligand and phenyl ring. This further results in reduced rates of carbonylation and thus, the observed poor reactivities.\(^{34,35}\) This trend was prominent in para-substituted aryl bromides which did not react to afford the anticipated products 7k.
and 7f. Amides 7m and 7n bearing an EWG on the para and ortho positions were obtained in 41% and 23% yield, respectively. Reactions between both substituted bromoarenes and nitroarenes afforded products 7s–7w in average to excellent yields. The reactivities of electron-rich nitroarenes and an ortho-substituted bromobenzene resulted in higher product yields compared to yields obtained with neutral nitroarenes (product 7m vs 7s). The product yields of 7w and 7m also revealed a similar trend, whereby the reactivities of an aryliodide possessing an EWG at the para position were improved when reacted with an electron-rich nitroarene. Aryliodides also reacted using the established reaction conditions and, in most cases, were found to be reactive at a lower reaction temperature of 90 °C (Scheme 1). Product 7q was obtained in 91% yield using iodobenzene as the coupling partner compared to the 51% yield obtained with bromobenzene at 130 °C. A similar trend was observed with the yields of product 7t. However, the use of aryliodides for the synthesis of products 7p and 7u resulted in unimproved yields. Increasing the reaction temperature from 90 °C to 130 °C resulted in an improved product yield from 35% to 91% for product 7t.

Heterocyclic aryl halides and nitropyridines were also investigated; however, reactions using these substrates did not afford the desired products (Scheme 1). 3-Bromopyridine was reacted with various nitroarenes resulting in no formation of anticipated products 7v and 7e. Similarly, coupling reactions with 2-nitropyridine did not afford the anticipated amide products 7ab and 7aa. We also went on to investigate the reductive aminocarbonylation of aryliodides and aryl triflates and, unfortunately, these substrates did not afford the anticipated amide products. Increasing the catalyst loading (20 mol%), carbonyl source moles (5 eq) and reaction time (24 h), as well as the reaction temperature (150 °C), did not afford the anticipated product.

Insights into the reductive aminocarbonylation reaction mechanism have been sought. A variety of possible reactive nitroarene intermediates have also been identified. To establish the reaction mechanism, we undertook a brief study to identify the possible reactive intermediates that are formed under the current protocol. GC analysis of products obtained from the reaction of nitrobenzene (6a) under the optimal conditions, as highlighted in Scheme 2 a, revealed aniline (8) as the reduction intermediate. However, and similarly to reports by Wu and co-workers, the direct use of aniline as a coupling partner under these reaction conditions was unsuccessful.

Scheme 1 Substrate scope study. *Reaction at 90 °C.
conditions did not afford the desired product (Scheme 2, b). Interestingly, when the reaction was repeated in the presence of nitrobenzene 6b, the amide products 7a and 7u were successfully formed in 100% GC yield (Scheme 2, c). Although specific mechanistic studies are yet to be established, this indicates that the presence of the nitro group plays a vital role in the reductive aminocarbonylation reaction.

Scheme 2 Control experiments. * GC yield.

In addition, coupling aryl bromides with other possible intermediates, such as azobenzene (9a), azoxybenzene (9b), 1,2-diphenylhydrazine (9c) and nitrosobenzene (9d), did not afford the anticipated amide products. However, these intermediates apart from 9d were also successfully coupled to in the presence of nitroarene 6b and afforded the anticipated amide products (Table 2, entries 2–4). This further demonstrates the possible role of the nitro functional group in activating the coupling of the proposed intermediates.

Scheme 3 outlines a general proposed mechanism of the reductive aminocarbonylation reaction. Initially, the Pd(II) species is converted into Pd(0) by the ligand. This is then followed by the oxidative addition of the aryl halide to give the aryl–Pd–halide intermediate i. CO liberated from Mo(CO)₆ then coordinates to Pd affording the aryl–Pd–CO intermediate ii that undergoes a rearrangement and carbonylation to form the phenacyl–Pd–halide complex iv. Zn or Mo and TMSCl reduce nitroarene v to the possible intermediates of type 8 and 9a–9c. These undergo amidation followed by a base-promoted reductive elimination to afford the target amide product vi and regeneration of the active Pd(0) catalyst.

Table 2 Reductive Aminocarbonylation between Nitroarene 6b, Intermediates and Aryl Bromide 5a

<table>
<thead>
<tr>
<th>Entry</th>
<th>Intermediate</th>
<th>Yield (%) a</th>
<th>Yield (%) a</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>2</td>
<td>9a</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>3</td>
<td>9b</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>4</td>
<td>9c</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>5</td>
<td>9d</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

Table 2: Reductive Aminocarbonylation between Nitroarene 6b, Intermediates and Aryl Bromide 5a.

In conclusion, we have demonstrated the potential applicability of Pd as a metal catalyst in reductive aminocarbonylation of nitroarenes with aryl bromides and iodides. Overall, products were obtained in modest to excellent yields. Aryl iodides showed better reactivities at lower reaction temperatures. Detailed mechanistic studies to establish the role of nitroarenes in activating the possible reduction intermediates are underway and these will be reported in the future. We will also focus on the development of more active catalysts that will promote the reductive coupling of a broader substrate scope.
(20 mL) was added and the mixture was extracted with EtOAc (3 × 10 mL). The extracts were combined and dried over anhydrous MgSO₄, filtered and concentrated using a rotary evaporator. The concentrated crude material was then purified using silica gel column chromatography (30% EtOAc/hexane) to afford the desired amide product 7. The following notes apply to the yields below: a Aryl halide used was Br. b Aryl halide used was I. * Reaction temperature was 90 °C.

4-Methoxy-N-phenylbenzamide (7a)
Isolated yield: 175 mg (95%); mp 173 °C.
IR (neat): 3336 (N-H), 1652 cm–1 (C=O).
1H NMR (500 MHz, CDCl₃): δ = 7.83 (d, J = 8.50 Hz, 2 H, Ar), 7.75 (br, 1 H, -NH), 7.62 (d, J = 8.00 Hz, 2 H, Ar), 7.15–7.10 (m, 1 H, Ar), 6.96 (d, J = 8.50 Hz, 2 H, Ar), 3.85 (s, 3 H, -OCH₃).
13C NMR (126 MHz, CDCl₃): δ = 165.20 (-C=O), 162.51 (-Ar), 138.14 (-Ar), 129.04 (-Ar), 128.88 (-Ar), 127.22 (-Ar), 124.32 (-Ar), 124.17 (-Ar), 113.99 (-Ar), 55.45 (-OCH₃).
HRMS: m/z [M + H]+ calcd: 228.1026; found: 228.1025.
Data in agreement with literature.37

4-Methyl-N-phenylbenzamide (7b)
Isolated yield: 145 mg (85%)a; mp 148 °C.
IR (neat): 3350 (N-H), 1648 cm–1 (C=O).
1H NMR (500 MHz, CDCl₃): δ = 7.94 (br, 1 H, -NH), 7.75 (d, J = 8.00 Hz, 2 H, Ar), 7.63 (d, J = 8.00 Hz, 2 H, Ar), 7.39–7.30 (m, 4 H, -Ar), 7.14–7.10 (m, 1 H, -Ar), 2.39 (s, 3 H, -CH₃).
13C NMR (126 MHz, CDCl₃): δ = 165.70 (-C=O), 142.27 (-Ar), 138.06 (-Ar), 132.10 (-Ar), 129.88 (-Ar), 127.22 (-Ar), 124.32 (-Ar), 120.17 (-Ar), 113.99 (-Ar), 55.45 (-OCH₃).
HRMS: m/z [M + H]+ calcd: 212.1078; found: 212.1078.

4-Isopropyl-N-phenylbenzamide (7c)
Isolated yield: 130 mg (67%); mp 168 °C.
IR (neat): 3368 (N-H), 1630 cm–1 (C=O).
1H NMR (500 MHz, CDCl₃): δ = 7.79 (d, J = 8.00 Hz, 2 H, Ar), 7.75–7.32 (m, 4 H, -Ar), 7.15–7.11 (m, 1 H, -Ar), 3.00–2.90 (m, 1 H, -Ar), 1.27 (d, J = 7.00 Hz, 6 H, -CH₃).
13C NMR (126 MHz, CDCl₃): δ = 165.63 (-C=O), 153.20 (-Ar), 138.04 (-Ar), 132.52 (-Ar), 129.08 (-Ar), 127.12 (-Ar), 126.87 (-Ar), 124.41 (-Ar), 120.09 (-Ar), 34.12 (-CH), 23.75 (-CH₃).
HRMS: m/z [M + H]+ calcd: 240.1390; found: 240.1380.

2-Methyl-N-phenylbenzamide (7d)
Isolated yield: 113 mg (66%)a; mp 124 °C.
IR (neat): 3307 (N-H), 1643 cm–1 (C=O).
1H NMR (500 MHz, CDCl₃): δ = 7.61 (d, J = 7.50 Hz, 2 H, Ar), 7.51 (br, 1 H, -NH), 7.48–7.32 (m, 1 H, -Ar), 7.32–7.30 (m, 2 H, -Ar), 7.28–7.20 (m, 3 H, -Ar), 7.16–7.10 (m, 1 H, -Ar), 2.48 (s, 3 H, -CH₃).
13C NMR (126 MHz, CDCl₃): δ = 168.02 (-C=O), 154.79 (-Ar), 137.99 (-Ar), 136.44 (-Ar), 131.27 (-Ar), 130.27 (-Ar), 129.19 (-Ar), 126.58 (-Ar), 125.90 (-Ar), 124.54 (-Ar), 119.88 (-Ar), 18.78 (-CH₃).
HRMS: m/z [M + H]+ calcd: 228.1026; found: 228.1028.

2-Methoxy-N-phenylbenzamide (7e)
Isolated yield: 61 mg (33%)a; mp 147 °C.
IR (neat): 3336 (N-H), 1728 cm–1 (C=O).
1H NMR (500 MHz, CDCl₃): δ = 9.78 (br, 1 H, -NH), 8.29 (dd, J = 2.00, 8.00 Hz, 1 H, -Ar), 7.67 (d, J = 8.00 Hz, 2 H, Ar), 7.50–7.45 (m, 1 H, -Ar), 7.36 (t, J = 7.50 Hz, 2 H, Ar), 7.16–7.09 (m, 2 H, -Ar), 7.02 (d, J = 8.00 Hz, 1 H, -Ar), 4.04 (s, 3 H, -CH₃).
13C NMR (126 MHz, CDCl₃): δ = 163.19 (-C=O), 157.19 (-Ar), 138.38 (-Ar), 133.20 (-Ar), 132.52 (-Ar), 129.89 (-Ar), 124.17 (-Ar), 121.76 (d, J = 19.2 Hz, -Ar), 120.43 (-Ar), 111.53 (-Ar), 56.21 (-OCH₃).
HRMS: m/z [M + H]+ calcd: 228.1026; found: 228.1028.

3-Methyl-N-phenylbenzamide (7g)
Isolated yield: 140 mg (82%); mp 126 °C.
IR (neat): 3368 (N-H), 1647 cm–1 (C=O).
1H NMR (500 MHz, CDCl₃): δ = 7.92 (br, 1 H, -NH), 7.69–7.71 (m, 4 H, -Ar), 7.37–7.30 (m, 4 H, -Ar), 7.13–7.10 (m, 1 H, -Ar), 2.39 (s, 3 H, -CH₃).
1H NMR (500 MHz, CDCl3): δ = 7.76 (br, 1 H, –NH), 7.60 (d, J = 7.00 Hz, 2 H, –Ar), 7.40 (d, J = 2.0 Hz, 1 H, –Ar), 7.38–7.33 (m, 3 H, –Ar), 7.13–7.08 (m, 1 H, –Ar), 6.91 (d, J = 8.50 Hz, 1 H, –Ar), 4.29 (dd, J = 5.0, 10.5 Hz, 1 H, –Ar).

IR (neat): 3255 (N-H), 1650 cm–1 (C=O).

HRMS: m/z [M + H]+ calcd: 212.1077; found: 212.1075.

N-(o-Tolyl)benzamide (7p)αβ

Isolated yield: 65 mg (42%); mp 146 °C.

IR (neat): 3344 (N-H), 1655 cm–1 (C=O).

1H NMR (500 MHz, CDCl3): δ = 7.90–7.82 (m, 3 H, Ar), 7.64 (d, J = 8.00 Hz, 2 H, –Ar), 7.50–7.45 (m, 3 H, –Ar), 7.38–7.33 (m, 3 H, –Ar), 7.16–7.10 (m, 1 H, –Ar).

13C NMR (126 MHz, CDCl3): δ = 156.69 (-C=O), 147.68 (-Ar), 139.93 (-Ar), 137.36 (-Ar), 135.02 (-Ar), 131.82 (-Ar), 129.09 (-Ar), 128.78 (-Ar), 126.99 (-Ar), 124.95 (-Ar), 124.55 (-Ar), 120.19 (-Ar).

HRMS: m/z [M + H]+ calcd: 198.0921; found: 198.0917.

N-(4-Methoxyphenyl)-4-methylbenzamide (7s)α

Isolated yield: 89 mg (55%); mp 172 °C.

IR (neat): 3304 (N-H), 1606 cm–1 (C=O).

1H NMR (500 MHz, CDCl3): δ = 7.81 (d, J = 8.50 Hz, 2 H, –Ar), 7.60 (br, 1 H, –NH), 7.33 (d, J = 1.0 Hz, 1 H, –Ar), 6.96 (d, J = 8.50 Hz, 2 H, –Ar), 6.89–6.83 (m, 3 H, –Ar), 6.77 (d, J = 8.00 Hz, 1 H, –Ar), 5.95 (s, 2 H, –CH3), 3.85 (s, 3 H, –OCH3).

13C NMR (126 MHz, CDCl3): δ = 162.48 (-C=O), 147.93 (-Ar), 144.40 (-Ar), 132.36 (-Ar), 128.81 (-Ar), 127.09 (-Ar), 113.99 (-Ar), 113.39 (-Ar), 108.10 (-Ar), 103.17 (-Ar), 101.30 (-CH3), 55.47 (-OCH3).

HRMS: m/z [M + H]+ calcd: 272.0925; found: 272.0921.

N-(4-Methoxyphenyl)-4-methylbenzamide (7t)

Isolated yield: 104 mg (62%); 151 mg (90%)αβ.

IR (neat): 3336 (N-H), 1657 cm–1 (C=O).

1H NMR (500 MHz, CDCl3): δ = 7.77 (br, 1 H, –NH), 7.74 (d, J = 8.00 Hz, 2 H, –Ar), 7.52 (d, J = 9.00 Hz, 2 H, –Ar), 7.38–7.32 (m, 2 H, –Ar), 6.88 (d, J = 9.00 Hz, 2 H, –Ar), 3.79 (s, 3 H, –OCH3), 2.39 (s, 3 H, –CH3).

N-(4-Methoxyphenyl)-4-methylbenzamide (7t)
1H NMR (500 MHz, CDCl₃): δ = 7.84 (d, 1 H, -NH), 7.82 (d, J = 9.00 Hz, 2 H, -Ar), 7.55–7.40 (m, 3 H, -Ar), 7.32 (s, 1 H, -Ar), 6.88 (d, J = 8.00 Hz, 1 H, -Ar), 6.74 (d, J = 8.00 Hz, 1 H, -Ar), 5.04 (s, 2 H, -CH₂).

13C NMR (126 MHz, CDCl₃): δ = 165.67 (-C=O), 147.87 (-Ar), 144.52 (-Ar), 134.87 (-Ar), 132.13 (-Ar), 131.73 (-Ar), 128.71 (-Ar), 126.96 (-Ar), 113.60 (-Ar), 108.07 (-Ar), 103.32 (-Ar), 101.30 (-CH₂).


Conflict of Interest

The authors declare no conflict of interest.

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