Facile Access to 2-Arylindolines and 2-Arylindoles by Microwave-Assisted Tandem Radical Cyclization

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Abstract: A new route providing access to 2-arylindoles has been developed. The synthesis consists of a tin-mediated tandem radical cyclization of appropriate precursors to form 2,3-disubstituted dihydroindoles, which in turn are oxidized to yield the corresponding 2-arylindoles. The reactions proceed smoothly under microwave irradiation, furnishing the desired products in good yields.

Key words: indoles, cyclizations, heterocycles, radical reactions, dehydrogenations, microwave irradiation

The indole moiety is a privileged scaffold in medicinal chemistry, being present in a wide range of biologically active natural and artificial compounds.1 Recently, a novel class of highly selective ERα ligands (SERAMs) such as 1 derived from the 2-(2-phenyl-1H-indol-3-yl)acetic acid moiety 2 as a crucial building block was disclosed, with this key intermediate having potential utilization as a lead structure towards the treatment of breast cancer (Scheme 1).2

To date, several elegant methods for assembling 2-arylindol-3-ylacetic acids have been established. These strategies consist of transition-metal-assisted intermolecular annulation and concomitant derivatization,3 cyclization of imidoyl radicals4 in combination with arylations,5 twofold elimination protocols of vinyl sulfones,6 base-catalyzed cyclization exploiting umpolung of imines,7 or the Fischer indolization approach.8

We report here a different approach that allows the direct synthesis of 2-(2-arylindol-3-yl)acetic acid derivatives 3 as well as their corresponding indoline derivatives 4 from 3-(2-aminophenyl)acrylates 5 by tandem radical cyclization involving 1,6-hydrogen transfer followed by 5-exo ring closure (Scheme 2), a synthetic method that was pioneered by Parsons et al. for the synthesis of mitomycins.9

Anilines 8 were readily synthesized in high yields from commercially available 2-nitrobenzaldehydes 6, which formed acrylates 7 by a Horner–Wadsworth–Emmons reaction with triethyl a-bromophosphonoacetate generated in situ (Scheme 3). Subsequent reduction of the thus formed acrylates 7 with iron powder under acidic conditions following a literature procedure3,9 gave anilines 8 (Scheme 3).

The direct monobenzylation of 8 by reaction with benzyl bromide failed: a complex mixture of unchanged starting material, and dibenzylated product along with the desired monobenzylated product was observed. To circumvent this lack of selectivity, the introduction of an appropriate protecting group was first necessary. For this, aniline 8

Scheme 1

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was initially N-Boc-protected; however, the successive benzylation produced only small amounts of desired product, presumably due to the steric effects of the space-demanding protecting group. Therefore, the methoxycarbonyl group (Moc) was introduced by reaction of the crude reaction mixture containing a reduced nitro group could be detected in the crude reaction mixture. Performing the cyclization under microwave heating in an open vessel resulted in some shortening of the reaction time (3.5 h instead of 5 h for complete consumption of starting material), but, more importantly, yields were significantly improved to 76–89% (Table 2, entries 2, 4, 7). Consequently, these conditions were also applied for the cyclization of 10e–h to give the corresponding indolines 14e–h in 25–73% yield (Table 2, entries 8–11).

The cis/trans ratios of indolines 14 (Scheme 5, Table 2) were determined by 1H NMR spectroscopy, with the final assignments accomplished by NOESY experiments. Diastereomers of 14a–e could be separated by crystallization of the product mixture, and the cis-diastereomer of 14b could be additionally characterized by X-ray crystallography (Scheme 5). The cis-diastereomers of 14 are kinetically favored (Table 2, and thus formed predominantly (cis/trans ca. 2:1) in the course of the reaction, with the exception being naphthyl derivative 14f, which was obtained in a reversed cis/trans diastereoselectivity (1:3), albeit in low yields of 25% along with reduced but uncyclized starting material (Table 2, entry 9). This may be a consequence of a relatively long lifetime of the uncyclized radical intermediate 12f (cf. Scheme 5) stabilized by the adjacent naphthyl group, leading to a preferred formation of the sterically less hindered, but thermodynamically favored trans-diastereomer.

The transformation of indolines 14 into the desired 2-arylindoles 15 (Scheme 6) was investigated with several dehydrogenation reagents (Table 3), of which 2,3-dichloro-5,6-dicyano-1,4-benzoquinone proved to be most effective, with respect to yields as well as ease of workup and purification of the products (Table 3, entries
Table 2  Radical Cyclization of 10a–h to 2,3-Substituted Indolines

<table>
<thead>
<tr>
<th>Entry</th>
<th>Conditions</th>
<th>Indoline</th>
<th>R</th>
<th>Ar</th>
<th>Yield (%)</th>
<th>Ratio cis/trans</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>A</td>
<td>14a</td>
<td>H</td>
<td>Ph</td>
<td>71</td>
<td>66:34</td>
</tr>
<tr>
<td>2</td>
<td>B</td>
<td>14a</td>
<td>H</td>
<td>Ph</td>
<td>86</td>
<td>66:36</td>
</tr>
<tr>
<td>3</td>
<td>A</td>
<td>14b</td>
<td>H</td>
<td>3-ClC₆H₄</td>
<td>62</td>
<td>67:33</td>
</tr>
<tr>
<td>4</td>
<td>B</td>
<td>14b</td>
<td>H</td>
<td>3-ClC₆H₄</td>
<td>76</td>
<td>64:36</td>
</tr>
<tr>
<td>5</td>
<td>A</td>
<td>14c</td>
<td>H</td>
<td>4-O₂NC₆H₄</td>
<td>&amp;b</td>
<td>-</td>
</tr>
<tr>
<td>6</td>
<td>A</td>
<td>14d</td>
<td>H</td>
<td>PMP</td>
<td>76</td>
<td>63:37</td>
</tr>
<tr>
<td>7</td>
<td>B</td>
<td>14d</td>
<td>H</td>
<td>PMP</td>
<td>89</td>
<td>60:40</td>
</tr>
<tr>
<td>8</td>
<td>B</td>
<td>14e</td>
<td>H</td>
<td>3-MeOC₆H₄</td>
<td>73</td>
<td>60:40</td>
</tr>
<tr>
<td>9</td>
<td>B</td>
<td>14f</td>
<td>H</td>
<td>Naph</td>
<td>25</td>
<td>25:75</td>
</tr>
<tr>
<td>10</td>
<td>B</td>
<td>14g</td>
<td>H</td>
<td>2-furyl</td>
<td>68</td>
<td>67:33</td>
</tr>
<tr>
<td>11</td>
<td>B</td>
<td>14h</td>
<td>OMe</td>
<td>Ph</td>
<td>64</td>
<td>59:41</td>
</tr>
</tbody>
</table>

* Reagents and conditions: Bu₃SnH (1.4 equiv), AIBN (2 equiv), toluene; Method A: reflux, 5 h; Method B: open vessel, 100–105 °C, microwave (2.45 GHz), 3.5 h.

Microwave-Induced, 2,3-Dichloro-5,6-dicyano-1,4-benzoquinone-Promoted Aromatization of Indolines 14 to Indoles 15*.

Table 3  Conditions for the Dehydrogenation of Indole 14a to Indole 15a

<table>
<thead>
<tr>
<th>Entry</th>
<th>Reaction conditions</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>DDQ (2 equiv), benzene, 50 °C, 2 d</td>
<td>48</td>
</tr>
<tr>
<td>2</td>
<td>DDQ (2 equiv), toluene, reflux, 5 d</td>
<td>63</td>
</tr>
<tr>
<td>3</td>
<td>Mn₃O (10 equiv), benzene, reflux, 2 d</td>
<td>11</td>
</tr>
<tr>
<td>4</td>
<td>NBS (1.5 equiv), CCl₄, reflux, 12 h</td>
<td>53</td>
</tr>
<tr>
<td>5</td>
<td>CAN (2 equiv), MeCN–H₂O, 70 °C, 3 h</td>
<td>32</td>
</tr>
<tr>
<td>6</td>
<td>DDQ (2 equiv), toluene, sealed vessel, 120 °C, MW, 4 h</td>
<td>66</td>
</tr>
</tbody>
</table>

Table 4  Microwave-Induced, 2,3-Dichloro-5,6-dicyano-1,4-benzoquinone-Promoted Aromatization of Indolines 14 to Indoles 15*.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Starting material 14*</th>
<th>Product 15</th>
<th>R</th>
<th>Ar</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>14a</td>
<td>15a</td>
<td>H</td>
<td>Ph</td>
<td>66</td>
</tr>
<tr>
<td>2</td>
<td>14b</td>
<td>15b</td>
<td>H</td>
<td>3-ClC₆H₄</td>
<td>60</td>
</tr>
<tr>
<td>3</td>
<td>14d</td>
<td>15c</td>
<td>H</td>
<td>PMP</td>
<td>61</td>
</tr>
<tr>
<td>4</td>
<td>14e</td>
<td>15d</td>
<td>H</td>
<td>3-MeOC₆H₄</td>
<td>60</td>
</tr>
<tr>
<td>5</td>
<td>14f</td>
<td>15e</td>
<td>H</td>
<td>Naph</td>
<td>55</td>
</tr>
<tr>
<td>6</td>
<td>14g</td>
<td>15f</td>
<td>H</td>
<td>2-furyl</td>
<td>&amp;b</td>
</tr>
<tr>
<td>7</td>
<td>14h</td>
<td>15g</td>
<td>OMe</td>
<td>Ph</td>
<td>69</td>
</tr>
</tbody>
</table>

* Reagents and conditions: DDQ (2 equiv), toluene, sealed vessel, 120 °C, MW, 4 h.

Scheme 6

1, 2, and 6). Treatment of 14a with 2,3-dichloro-5,6-dicyano-1,4-benzoquinone in benzene at 50 °C for two days provided the desired indole 15a in 48% yield along with unchanged, exclusively trans-configured starting material 14a (25%) (Table 3, entry 1). Because of the obvious lower reactivity of the trans-diastereomer towards aromatization, the reaction temperature was increased by changing the solvent to toluene. The reaction was carried out at reflux temperature for five days until no starting material could be detected by TLC, and 15a was isolated in 63% yield (Table 3, entry 2). Further improvements were possible by carrying out this transformation under microwave heating in a sealed vessel at 120 °C, resulting again in slightly improved yields, but, moreover, the reaction time could be shortened to four hours (Table 3, entry 6). Under the same conditions, indolines 14b–d–h were also subjected to dehydrogenation, and, with the exception of furyl derivative 14g, smoothly yielded the corresponding indoles 15b–e–g (Scheme 6, Table 4).

In conclusion, a facile method for the synthesis of the (2-arylindol-3-yl)acetic acid moiety, representing a potential lead structure for the development of drugs against breast cancer, could be developed.

All nonaqueous reactions were carried out in oven-dried glassware under N₂. Toluene and benzene were distilled from Na. THF was distilled from Na/benzophenone ketyl under N₂. All other reagents were commercially available and were used without further purification. Preparative flash chromatography was performed on Silica Gel 60 (Merck Gederan 60, 0.063–0.200 mm). ¹H and ¹³C NMR spectra were recorded on Bruker Avance 300, 400, and 600 instruments; CDCl₃ or DMSO-d₆ was used as solvent. IR spectra were recorded on an FT IR spectrometer (Excalibur Series FT3000MX), melting points were recorded on a Büchi-SMP-20 apparatus and are uncorrected, HRMS was performed on Varian MAT 311A, Finnigan MAT95, and Thermoquest Finnigan TSQ 7000 spectrometers, and elemental analysis was carried out on a Vario EL II or Mikro-Rapid CHN (Heraeus) instrument. Microwave-assisted reactions were performed in a CEM Focused Microwave Synthesis System, Discover. Reactions were monitored by TLC on Merck 60 F₂₅₄ silica gel TLC plates. Spots were visualized by UV light at 254 and 365 nm. All solvents used for column chromatography were distilled.

2-Bromo-3-(2-nitrophenyl)acrylates 7; General Procedure
A 60% suspension of NaH in fuel oil (3.7 g, 92.6 mmol, 1.40 equiv) was added to THF (50 mL) at 0 °C under N₂. (EtO)₂P(O)CH₂CO₂Et (18.08 g, 86.1 mmol, 1.30 equiv) dissolved in THF (30 mL) was added dropwise over 15 min, and stirring of the resulting mixture continued for 45 min. Br₂ (14.29 g, 89.4 mmol, 1.35 equiv) was addi-
ed dropwise over 15 min, and stirring of the reaction mixture continued for 30 min at 0 °C. A 60% NaHa in N-hexyl oil (3.7 g, 92.6 mmol, 1.40 equiv) was added over 20 min, and stirring continued for 1 h at 0 °C. 2-Nitrobenzaldehyde 6 (66.17 mmol, 1.0 equiv) dissolved in THF (20 mL) was added and the reaction mixture was slowly warmed to r.t. and stirred for 5.5 h. Sat. aq NH₄Cl (15 mL) was added and the mixture was extracted with Et₂O (3 × 50 mL). The combined organic phases were washed with brine (30 mL), dried (Na₂SO₄), filtered, and concentrated. The crude product was recrystallized from i-ProH (25 mL) to give pale yellow crystals of 7.

**Ethyl (Z)-2-Bromo-(2-nitrophenyl)acrylate (7a)**

Yield: 18.9 g (95%); mp 18.9°C; Rf = 0.4 (hexanes–EtOAc, 3:1).

IR (KBr): 488, 575, 612, 706, 757, 833, 860, 908, 945, 1037, 1069, 1109, 1190, 1248, 1273, 1364, 1450, 1537, 1581, 1616, 1702, 2934, 2986, 3030, 3301 cm⁻¹.

**Ethyl (Z)-2-Bromo-(3-(2-amino-4,5-dimethoxyphenyl)acrylate (8b)**

Yield: 4.4 g (91%); mp 72–73 °C; Rf = 0.2 (hexanes–EtOAc, 5:1).

IR (KBr): 488, 575, 612, 707, 755, 833, 860, 908, 945, 1037, 1069, 1109, 1190, 1248, 1273, 1364, 1450, 1537, 1581, 1616, 1702, 2934, 2986, 3030, 3301 cm⁻¹.

**Ethyl (Z)-2-Bromo-3-(2-(methoxycarbonylamino)phenyl)acrylate (9b)**

Yield: 4.9 g (85%); mp 141 °C; Rf = 0.3 (hexanes–EtOAc, 3:1).

IR (KBr): 488, 575, 612, 707, 755, 833, 860, 908, 945, 1037, 1069, 1109, 1190, 1248, 1273, 1364, 1450, 1537, 1581, 1616, 1702, 2934, 2986, 3030, 3301 cm⁻¹.

**Ethyl (Z)-2-Bromo-3-(2-(methoxycarbonylamino)phenyl)acrylate (9c)**

Yield: 4.4 g (91%); mp 72–73 °C; Rf = 0.2 (hexanes–EtOAc, 5:1).

IR (KBr): 488, 575, 612, 707, 755, 833, 860, 908, 945, 1037, 1069, 1109, 1190, 1248, 1273, 1364, 1450, 1537, 1581, 1616, 1702, 2934, 2986, 3030, 3301 cm⁻¹.

**Ethyl (Z)-2-Bromo-3-(2-(methoxycarbonylamino)phenyl)acrylate (9d)**

Yield: 4.9 g (85%); mp 141 °C; Rf = 0.3 (hexanes–EtOAc, 3:1).

IR (KBr): 488, 575, 612, 707, 755, 833, 860, 908, 945, 1037, 1069, 1109, 1190, 1248, 1273, 1364, 1450, 1537, 1581, 1616, 1702, 2934, 2986, 3030, 3301 cm⁻¹.
13C NMR (75.5 MHz, CDCl3): δ = 14.2 (CH3), 52.6 (CH3), 56.0 (CH2), 62.9 (CH2), 111.2 (CH), 115.3 (C), 130.4 (C), 136.3 (CH), 150.7 (C), 154.5 (C), 163.0 (C).

Anal. Calcd for C9H8BrNO3: C, 46.41; H, 4.67; N, 3.61. Found: C, 46.68; H, 4.91; N, 3.50.

3-[2-[(Arylmethyl)(methoxy carbonyl)amino]phenyl]-2-bromoacrylates 10; General Procedure
LiOH (1.0 mmol, 1.5 equiv) was added portionwise over 30 min to a solution of 9 (1.0 mmol, 1.0 equiv) in THF (10 mL), washed with sat. aq NH4Cl (10 mL), and extracted with EtOAc (3 × 15 mL). The combined organic phases were dried (Na2SO4), filtered, and concentrated. The crude mixture was purified by chromatography (silica gel, hexanes–EtOAc); this gave 10.

Ethyl (Z)-3-[(Benzyloxy carbonyl)amino]phenyl]-2-bromoacrylates (10a)
Colorless oil; yield: 91%; Rf = 0.3 (hexanes–EtOAc, 5:1).

IR (KBr): 3427, 2961, 2925, 2854, 1734, 1609, 1589, 1522, 1457, 1385, 1314, 1283, 1254, 1210, 1167, 1122, 1075, 983, 872, 753 cm−1.


Indolines 114 by Radical Cyclization of 2-Bromo-3-phenylacrylates 10; General Procedure
A mixture of n-Bu3SnH (185 μL, 0.7 mmol, 1.4 equiv) and AIBN (22 mg, 0.13 mmol, 0.25 equiv) in toluene (10 mL) was added via a syringe pump over 3 h to a refluxing (microwave irradiation at 100–110 °C under open-vessel conditions) solution of 10 (0.5 mmol, 1.0 equiv) and AIBN (22 mg, 0.13 mmol, 0.25 equiv) in toluene (10 mL). The solvent was evaporated and the residue was dissolved in Et2O (100 mL). DBU (2.7 equiv, 1.35 mmol) was added to the solution, which was titrated with 0.1 M I2 in Et2O and filtered through silica gel.14 The crude product was purified by chromatography (silica gel, hexanes–EtOAc); this gave 14 as white solids.

Methyl (3-[(Ethoxy-2-oxoethyl)-2-phenylindolino]-1-carboxylate (14a)
Rf = 0.3 (hexanes–EtOAc, 5:1).


Methyl 2-(3-Chlorophenyl)-3-(2-ethoxy-2-oxoethyl)indoline-1-carboxylate (14b)
IR (KBr): 480, 509, 579, 631, 706, 752, 791, 849, 888, 934, 1024, 1056, 1080, 1138, 1180, 1254, 1271, 1308, 1335, 1381, 1442, 1483, 1575, 1597, 1717, 2957 cm−1.


Methyl 2-(3-Chlorophenyl)-3-(2-ethoxy-2-oxoethyl)indoline-1-carboxylate (14b)
IR (KBr): 480, 509, 579, 631, 706, 752, 791, 849, 888, 934, 1024, 1056, 1080, 1138, 1180, 1254, 1271, 1308, 1335, 1381, 1442, 1483, 1575, 1597, 1717, 2957 cm−1.


Methyl 2-(3-Chlorophenyl)-3-(2-ethoxy-2-oxoethyl)indoline-1-carboxylate (14b)
IR (KBr): 480, 509, 579, 631, 706, 752, 791, 849, 888, 934, 1024, 1056, 1080, 1138, 1180, 1254, 1271, 1308, 1335, 1381, 1442, 1483, 1575, 1597, 1717, 2957 cm−1.


Methyl 2-(3-Chlorophenyl)-3-(2-ethoxy-2-oxoethyl)indoline-1-carboxylate (14b)
IR (KBr): 480, 509, 579, 631, 706, 752, 791, 849, 888, 934, 1024, 1056, 1080, 1138, 1180, 1254, 1271, 1308, 1335, 1381, 1442, 1483, 1575, 1597, 1717, 2957 cm−1.


Methyl 2-(3-Chlorophenyl)-3-(2-ethoxy-2-oxoethyl)indoline-1-carboxylate (14b)
IR (KBr): 480, 509, 579, 631, 706, 752, 791, 849, 888, 934, 1024, 1056, 1080, 1138, 1180, 1254, 1271, 1308, 1335, 1381, 1442, 1483, 1575, 1597, 1717, 2957 cm−1.
Hz. 1.7 Hz, 1 H, CH), 3.76 (s, 3 H, CH3), 4.19 (q, J = 7.2 Hz, 2 H, CH2), 5.16 (d, J = 1.7 Hz, 1 H, CH), 6.79 (d, J = 8.8 Hz, 2 H, CH), 6.98–7.17 (m, 2 H, CH), 7.12 (d, J = 8.8 Hz, 2 H, CH), 7.25–7.30 (m, 6 H, CH), 7.85 (s, 1 H, CH).

13C NMR (75.5 MHz, CDCl3): δ (cis-14d) = 14.2 (CH3), 34.1 (CH3), 41.1 (CH3), 52.7 (CH3), 55.2 (CH3), 60.7 (CH3), 113.7 (CH), 114.6 (CH), 123.1 (CH), 123.4 (CH), 128.1 (CH), 128.3 (CH), 130.4 (C), 132.1 (C), 153.5 (C), 159.1 (C), 172.3 (C).


Methyl 3-(2-Ethoxy-2-oxoethyl)-2-(3-methoxyphenyl)indoline-1-carboxylate (14e)

Rf = 0.3 (hexanes–EtOAc, 9:1).


Anal. Calcd for C21H23NO5: C, 68.28; H, 6.28; N, 3.79. Found: C, 67.88; H, 6.48; N, 3.61.

Methyl 3-(2-Ethoxy-2-oxoethyl)-5,6-dimethoxy-2-phenylindoline-1-carboxylate (14f)

Rf = 0.4 (hexanes–EtOAc, 3:1).


Anal. Calcd for C21H23NO5: C, 74.02; H, 5.95; N, 3.60. Found: C, 73.95; H, 6.22; N, 3.58.

Methyl 3-(2-Ethoxy-2-oxoethyl)-2-(2-furyl)indoline-1-carboxylate (14g)

Rf = 0.3 (hexanes–EtOAc, 2:1).


Methyl 3-(2-Ethoxy-2-oxoethyl)-5,6-dimethoxy-2-phenylindoline-1-carboxylate (14h)

Rf = 0.3 (hexanes–EtOAc, 2:1).

Indoles 15 by Dehydrogenation of Indolines 14; General Procedure
A soln of indoline 14 (0.3 mmol, 1.0 equiv) and DDQ (0.6 mmol, 2.0 equiv) in toluene (7 mL) was placed in a sealed vessel in a microwave and irradiated at 120 °C for 4 h. After completion of the reaction, the solvent was removed and the residue was washed with sat. aq NaHCO3 (5 mL) and extracted with CH2Cl2 (3 × 10 mL). The combined organic phases were dried (Na2SO4), filtered, and concentrated. The crude product was purified by chromatography (silica gel, hexanes–EtOAc); this gave indoles 15 as oils.

**Methyl 3-(2-Ethoxy-2-oxoethyl)-2-phenyl-1-indole-1-carboxylate (15a)**

Rf = 0.4 (hexanes–EtOAc, 5:1).

**IR (KBr):** 625, 752, 822, 940, 1028, 1076, 1154, 1230, 1330, 1360, 1411, 1605, 1732, 1896, 2854, 2929, 2956, 2981, 3055 cm−1.

**HRMS–FAB: m/z [M + H]+ calcd for C21H21NO5: 367.1420; found: 367.1417.**

**Methyl 3-(2-Ethoxy-2-oxoethyl)-2-(2-naphthyl)-1-indole-1-carboxylate (15b)**

Rf = 0.4 (hexanes–EtOAc, 5:1).

**IR (KBr):** 699, 755, 785, 1021, 1090, 1145, 1222, 1326, 1356, 1439, 1595, 1730, 2960 cm−1.

**HRMS–FAB: m/z [M + H]+ calcd for C24H21NO4: 387.1471; found: 387.1471.**

**Methyl 3-(2-Ethoxy-2-oxoethyl)-2-(3-methoxyphenyl)-1H-indole-1-carboxylate (15d)**

Rf = 0.4 (hexanes–EtOAc, 5:1).

**IR (KBr):** 733, 819, 862, 910, 1025, 1072, 1150, 1227, 1321, 1358, 1440, 1734, 2932, 2990, 3055 cm−1.

**HRMS–FAB: m/z [M + H]+ calcd for C24H21NO4: 387.1471; found: 387.1471.**

**Methyl 3-(2-Ethoxy-2-oxoethyl)-2-(3-chlorophenyl)-1H-indole-1-carboxylate (15e)**

Rf = 0.4 (hexanes–EtOAc, 5:1).

**IR (KBr):** 702, 849, 1030, 1080, 1167, 1205, 1312, 1364, 1442, 1485, 1514, 1603, 1707, 1725, 2841, 2956 cm−1.

**HRMS–FAB: m/z [M + H]+ calcd for C24H21NO4: 387.1471; found: 387.1471.**
Acknowledgment

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References

(11) Details on the X-ray crystal structure of 14b can be obtained from the Cambridge Crystallographic Data Centre (CCDC 691332).