Synthesis of Some New 3-Pyrrolidinylquinoline Derivatives via 1,3-Dipolar Cycloaddition of Stabilized Azomethine Ylides to Quinolinyl α,β-Unsaturated Ketones

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Abstract: N-Metallated azomethine ylide generated from methyl (E)-N-benzylidendeglycinate, LiBr and triethylamine underwent cycloaddition to quinolyl α,β-unsaturated ketones with excellent diastereoselectivity to afford new functionalised 3-pyrrolidinylquinoline derivatives.

Keywords: 1,3-dipolar cycloaddition, quinoline, pyrrolidine synthesis, azomethine ylides.

INTRODUCTION
Quinolines derivatives have attracted considerable interest for many years due to their presence in the skeleton of a large number of bioactive compounds and natural products [1]. For example, quinoline alkaloids, such as quinine, chloroquine, mefloquine and amodiaquine, are used as efficient drugs for the treatment of malaria [2].

On the other hand, 1,3-dipolar cycloaddition reactions of azomethine ylides with olefinic dipolarophiles had resulted in a number of novel heterocyclic scaffolds which are particularly useful for the creation of diverse chemical libraries of drug-like molecules for biological screening [3]. Functionalized pyrrolidine containing compounds are also of significant importance because of their biological activities and widespread employment in catalysis [4].

The coupling of this chemical entity with quinoline unit might as well be envisioned to bring with some biological activities. In this context, some limited investigations have been carried out which involved the combination of the quinolyl moiety and the pyrrolidine unit.

As a part of our program related to the preparation and biological evaluation of quinolyl derivatives [5], we have recently described a practical and an efficient synthesis of some 3-pyrrolidinylquinoline derivatives from quinolinyl α,β-unsaturated esters as starting materials via 1,3-dipolar cycloaddition [6]. In a continuation of our efforts in this area, we report here an efficient procedure for the preparation of new pyrrolidine derivatives bearing a quinoline ring at C-3, aroyl or acetyl group at C-4, and a phenyl substituent at C-5 via an 1,3-dipolar cycloaddition reaction of a stabilized metallo-azomethine ylide to quinolinyl α,β-unsaturated ketones [7].

RESULTS AND DISCUSSION

Starting from the corresponding 2-chloro-3-formylquinoline derivatives 1, chalcones 2a-2h were synthesized by Claisen-Schmidt condensation reactions of appropriately substituted acetophenone in ethanol in the presence of 10% of aqueous NaOH [8]. The methylketone derivatives 2i-2j were prepared from the aldehydes 1 via a Wittig reaction using methyl(triphenylphosphoranylidene) acetate and were obtained in good yields (Scheme 1).

The E-configured dipolarophiles (2a-2j) reacted with azomethine ylide, generated from methyl (E)-N-benzylidendeglycinate in the presence of LiBr and triethylamine at room temperature, employing dry THF as the solvent (Scheme 2).

In accordance with literature reports [9, 10], this 1,3-dipolar cycloaddition reaction of the in situ generated metallo-azomethine ylide, exhibited high regio and stereoselectivity leading to the expected syn-endo cycloadduct (3a-3j).

All results reported below shown that pyrrolidines were obtained with conservation of the stereochemistry of starting alkenes [11], giving only one diastereoisomer with no evidence of any other isomers in the 1H NMR spectra of the crude products (Table 1).

The structure of compounds 3a-3j has been established by analogy and by comparison of their 1H NMR with those reported [12]. The shielding of the protons of the methyl connected to the aroyl group attached at C-4 by the adjacent 5-phenyl ring (δ=1.83 ppm) confirms the regiochemistry and demonstrated the 4,5-cis configuration relationship [6, 13].
Scheme 1. Synthesis of chalcone and methyl(vinylquinoline) ketone derivatives.

Scheme 2. Synthesis of quinolylpyrrolidine N-H derivatives (3a-3j).

Table 1. Synthesis of 3-pyrrolidinylquinolines 3

<table>
<thead>
<tr>
<th>Compound</th>
<th>R¹</th>
<th>R²</th>
<th>R³</th>
<th>R⁴</th>
<th>R</th>
<th>Yield (%)</th>
</tr>
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<tbody>
<tr>
<td>3a</td>
<td>H</td>
<td>H</td>
<td>H</td>
<td>Me</td>
<td>2-MeC₆H₄</td>
<td>70</td>
</tr>
<tr>
<td>3b</td>
<td>H</td>
<td>H</td>
<td>H</td>
<td>H</td>
<td>2-MeC₆H₄</td>
<td>58</td>
</tr>
<tr>
<td>3c</td>
<td>OMe</td>
<td>H</td>
<td>H</td>
<td>OMe</td>
<td>2-MeC₆H₄</td>
<td>71</td>
</tr>
<tr>
<td>3d</td>
<td>H</td>
<td>Me</td>
<td>H</td>
<td>H</td>
<td>2-MeC₆H₄</td>
<td>62</td>
</tr>
<tr>
<td>3e</td>
<td>H</td>
<td>H</td>
<td>H</td>
<td>Me</td>
<td>4-OMeC₆H₄</td>
<td>60</td>
</tr>
<tr>
<td>3f</td>
<td>H</td>
<td>OMe</td>
<td>H</td>
<td>H</td>
<td>4-OMeC₆H₄</td>
<td>65</td>
</tr>
<tr>
<td>3g</td>
<td>H</td>
<td>H</td>
<td>H</td>
<td>Me</td>
<td>3,4-diMeOC₆H₄</td>
<td>53</td>
</tr>
<tr>
<td>3h</td>
<td>H</td>
<td>H</td>
<td>H</td>
<td>Me</td>
<td>3,4,5-triMeOC₆H₄</td>
<td>54</td>
</tr>
<tr>
<td>3i</td>
<td>H</td>
<td>Me</td>
<td>H</td>
<td>H</td>
<td>Me</td>
<td>67</td>
</tr>
<tr>
<td>3j</td>
<td>H</td>
<td>H</td>
<td>H</td>
<td>H</td>
<td>Me</td>
<td>59</td>
</tr>
</tbody>
</table>

The structure of compound 3a, as representative example, was elucidated by detailed NMR studies (Table 2). The \(^1\)H and \(^13\)C NMR assignments were made on the basis of high-field one and two-dimensional methods (HSQC, COSY, and NOESY H, H). The ‘2,4,5-cis’ configuration of these pyrrolidines was confirmed by the observed NOE enhancement between the two pairs (H-2 and H-5) and (H-2 and H-4) (Table 2).

X-ray crystallography of 3e (Fig. 1) showed an asymmetric unit which contains two independent molecules and the analysis demonstrate that the two stereoisomers have for each one, the absolute stereochemistry (2S,3R,4S,5R) and

Table 2. Significant \(^1\)H, \(^{13}\)C NMR Data, Selected H-H Coupling NOE for 3a

<table>
<thead>
<tr>
<th></th>
<th>(^{13})C</th>
<th>(^1)H (m, J)</th>
<th>(^1)H(^{1})H n.O.e(^a)</th>
<th>(^1)H(^{1})H cosy</th>
</tr>
</thead>
<tbody>
<tr>
<td>H-2</td>
<td>66.5</td>
<td>4.40 (d, 7.9)</td>
<td>H-5, H-4 Qui, 2-CO₂Me</td>
<td>4.69</td>
</tr>
<tr>
<td>H-5</td>
<td>66.4</td>
<td>4.96 (d, 8.1)</td>
<td>H-2, H-6 Ar, H-2 Ar</td>
<td>4.77</td>
</tr>
<tr>
<td>H-4</td>
<td>60.8</td>
<td>4.77 (t, 8.0)</td>
<td>H-2, H-4 Qui</td>
<td>4.69, 4.96</td>
</tr>
<tr>
<td>H-3</td>
<td>50.5</td>
<td>4.69 (t, 8.0)</td>
<td>H-4 Qui</td>
<td>4.40, 4.77</td>
</tr>
</tbody>
</table>

\(^a\)Obtained by 2D-NOESY spectroscopy.
(2R,3S,4R,5S) of the new stereocenters created in the cycloaddition reactions [14].

**General Method for the Synthesis of Chalcone Derivatives (2a-2b)**

To a solution of 10% NaOH (520 mg, 13 mmol) in 95% ethanol (20 mL) was added 500 mg (2.61 mmol) of 2-chloro-3-formylquinoline and the acetophenone derivative (1.0 eq., 2.61 mmol). The mixture was stirred at 25 °C for 24 h. The contents were then cooled and poured into cold water then neutralized with dilute HCl. The solid obtained was filtered, washed, and dried on air to afford the crude chalcone.

(E)-3-(2-Chloro-8-methylquinolin-3-yl)-1-o-tolylprop-2-en-1-one (2a)

Yd 94%. Rf (CH2Cl2): 0.60. Mp 165-167 °C. IR \( \nu_{\text{max}} \) (KBr) 1654 cm\(^{-1}\) (C=O, ketone). \(^1\)H NMR (250 MHz, CDCl\(_3\)) \( \delta \) 8.48 (s, 1H), 8.00 (d, \( J=16.1, 1H \)), 7.75 (d, \( J=8.9, 1H \)), 7.52 (dd, \( J=9.1, J=2.4, 1H \)), 7.50-7.43 (m, 3H), 7.40 (ddd, \( J=8.9, J=8.1, J=2.3, 1H \)), 7.38 (dd, \( J=9.4, J=1.2, 1H \)), 7.25 (d, \( J=16.1, 1H \)), 2.75 (s, 3H), 2.50 (s, 3H). \(^13\)C NMR (62.5 MHz, CDCl\(_3\)) \( \delta \) 195.2 (C=O), 149.1 (C), 147.1 (C), 140.5 (CH), 138.2 (C), 137.4 (C), 136.7 (CH), 136.4 (CH), 131.7 (C), 131.5 (CH), 130.9 (CH), 130.2 (C), 128.4 (CH), 127.4 (CH), 127.3 (C), 125.0 (CH), 125.8 (CH), 20.4 (CH\(_3\)), 17.7 (CH\(_3\)). HRMS (EI): m/z [M\(^+\)] Calcd. for C\(_{19}\)H\(_{14}\)NO\(_3\)\(_5\)Cl: 307.0769, found 307.0769.

(E)-3-(2-Chloroquinolin-3-yl)-1-o-tolylprop-2-en-1-one (2b)

Yd 88%. Rf (CH2Cl2): 0.65. Mp 120-124 °C. IR \( \nu_{\text{max}} \) (KBr) 1685 cm\(^{-1}\) (C=O, ketone). \(^1\)H NMR (250 MHz, CDCl\(_3\)) \( \delta \) 8.50 (s, 1H), 7.95 (d, \( J=15.9, 1H \)), 7.79 (dd, \( J=8.7, J=8.4, 1H \)), 7.52 (dd, \( J=8.9, J=2.4, 1H \)), 7.50-7.32 (4H, m), 7.37 (dd, \( J=8.4, J=1.2, 1H \)), 7.29 (d, \( J=15.9, 1H \)), 6.82 (td, \( J=9.1, J=2.1, 1H \)), 2.50 (s, 3H). \(^13\)C NMR (62.5 MHz, CDCl\(_3\)) \( \delta \) 195.2 (C=O), 150.2 (C), 147.8 (C), 140.1 (CH), 138.1 (C), 137.5 (C), 137.3 (CH), 136.1 (CH), 133.4 (CH), 132.0 (CH), 131.6 (CH), 131.4 (CH), 130.6 (CH), 129.9 (CH), 127.2 (C), 125.5 (CH), 124.8 (CH), 20.4 (CH\(_3\)). HRMS (EI): m/z [M\(^+\)] Calcd. for C\(_{20}\)H\(_{16}\)NO\(_3\)\(_5\)Cl: 312.0924, found 312.0928.

(E)-3-(2-Chloro-8-methylquinolin-3-yl)-1-o-tolylprop-2-en-1-one (2c)

Yd 87%. Rf (CH2Cl2): 0.62. Mp 169-170 °C. IR \( \nu_{\text{max}} \) (KBr) 1672 cm\(^{-1}\) (C=O, ketone). \(^1\)H NMR (300 MHz, CDCl\(_3\)) \( \delta \) 8.90 (s, 1H), 7.97 (d, \( J=16.0, 1H \)), 7.60 (d, \( J=7.5, 1H \)), 7.49 (d, \( J=7.4, 1H \)), 7.19-7.42 (m, 3H), 7.05 (d, \( J=16.0, 1H \)), 6.89 (d, \( J=8.6, 1H \), 4.04 (s, 3H), 4.00 (s, 3H), 2.51 (s, 3H). \(^13\)C NMR (75.4 MHz, CDCl\(_3\)) \( \delta \) 197.5 (C=O), 151.3 (C), 148.9 (C), 148.4 (C), 140.6 (CH), 138.7 (C), 135.6 (CH), 132.1 (C), 131.5 (C), 131.5 (CH), 130.9 (CH), 130.4 (CH), 128.4 (CH), 127.9 (CH), 125.5 (CH), 121.8 (C), 109.8 (CH), 104.8 (CH), 56.2 (OCH\(_3\)), 55.8 (OCH\(_3\)), 20.4 (CH\(_3\)). HRMS (EI): m/z [M\(^+\)] Calcd. for C\(_{21}\)H\(_{18}\)NO\(_3\)\(_5\)Cl: 307.0769, found 307.0769.

(E)-3-(2-Chloro-6-methylquinolin-3-yl)-1-o-tolylprop-2-en-1-one (2d)

Yield 91%. Rf (CH2Cl2): 0.59. Mp 143-145 °C. IR \( \nu_{\text{max}} \) (KBr) 197.5 (C=O), 150.1 (C), 146.6 (C), 140.5 (C).
(CH), 138.2 (C), 136.0 (C), 136.5 (CH), 135.4 (CH), 131.7 (C), 131.5 (CH), 130.9 (CH), 130.2 (C), 128.4 (CH), 128.1 (CH), 127.4 (C), 127.0 (CH), 125.6 (CH), 125.2 (CH), 20.4 (CH3), 18.1 (CH3). HRMS (EI): m/z [M+] Calcd. for C20H16NO3 Cl: 321.09204, found 321.0928.

(E)-3-(2-Chloro-8-methylquinolin-3-yl)-1-(4-methoxyphenyl)prop-2-en-1-one (2g)

Yield 70%. Rf 0.72. Mbp 175-177 °C. IR v max (KBr) 1651 cm –1 (C=O, ketone). 1H NMR (300 MHz, CDCl3) δ 8.42 (s, 1H), 8.04 (d, J=8.4, 1H), 7.98 (d, J=16.3, 1H), 7.88 (d, J=8.2, 1H), 7.80 (t, J=7.9, 1H), 7.62 (t, J=7.3, 1H), 6.82 (d, J=16.3, 1H), 2.48 (s, 3H). 13C NMR (75.4 MHz, CDCl3) δ 197.6 (C=O), 148.5 (C), 138.0 (CH), 136.1 (CH), 133.4 (C), 131.9 (C), 131.7 (CH), 131.0 (C), 128.4 (CH), 128.0 (CH), 127.7 (C), 126.4 (CH), 125.9 (2xCH), 121.6 (CH), 113.1 (2xCH), 108.6 (CH), 55.5 (OCH3), 54.4 (OCH3). HRMS (EI): m/z [M+] Calcd. for C13H18NO3Cl: 245.06074, found 245.0607.

(E)-4-(2-Chloro-6-methylquinolin-3-yl)-but-3-en-2-one (2i)

Yield 90%. Rf (CH2Cl2): 0.65. Mp 183-184 °C. IR v max (KBr) 1658 cm –1 (C=O, ketone). 1H NMR (250 MHz, CDCl3) δ 8.50 (s, 1H), 8.25 (d, J=15.7, 1H), 7.75-7.67 (m, 4H), 7.52 (d, J=15.7, 1H), 7.50 (s, 1H), 7.00 (d, J=8.4, 1H), 4.00 (s, 3H), 3.73 (s, 3H). 13C NMR (75.4 MHz, CDCl3) δ 195.5 (C=O), 153.5 (C), 150.0 (C), 149.2 (C), 146.9 (C), 145.7 (C), 138.7 (CH), 136.6 (CH), 136.4 (CH), 136.3 (CH), 131.5 (C), 131.8 (CH), 127.0 (CH), 126.2 (C), 125.9 (2xCH), 121.6 (CH), 113.1 (2xCH), 108.6 (CH), 55.5 (OCH3), 54.4 (OCH3). HRMS (EI): m/z [M+] Calcd. for C20H16NO3 Cl: 353.08187, found 353.0825.
Methyl 4-(2-methylbenzoyl)-3-(2-chloroquinolin-3-yl)-5-phenylpyrrolidine-2-carboxylate (3b)

Yield 58%. Rf (CHCl₃): 0.34. Mp 79-81 °C. IR (ATR) v 3347, 2926, 1735, 1676, 1567, 1488, 1454, 1331, 1202, 1133, 731 cm⁻¹. ¹H NMR (300 MHz, CDCl₃) δ 8.31 (s, 1H), 8.04 (d, J = 8.5, 1H), 8.86 (d, J = 8.1, 1H), 7.72 (dd, J = 7.7, J = 1.1, 1H), 7.59 (t, J = 7.4, 1H), 7.46 (d, J = 7.7, 1H), 7.24 (t, J = 7.6, 1H), 7.17-7.11 (m, 6H), 6.99 (d, J = 7.5, 1H), 4.96 (d, J = 8.2, 1H), 4.81 (t, J = 8.1, 1H), 4.70 (t, J = 8.1, 1H), 3.47 (d, J = 8.3, 1H), 3.79 (s, 3H), 2.88 (brs, 1H), 1.83 (s, 3H). ¹³C NMR (75.4 MHz, CDCl₃) δ 201.7 (C=O), 173.2 (C=O), 151.3 (C), 147.1 (C), 139.9 (C), 139.0 (C), 138.4 (C), 137.8 (C), 137.5 (C), 132.8 (C), 132.2 (CH), 131.9 (CH), 130.9 (CH), 129.0 (CH), 128.7 (2CH), 128.6 (CH), 128.6 (CH), 128.1 (CH), 127.7 (2CH), 127.7 (CH), 125.8 (CH), 126.6 (CH), 126.5 (CH), 125.7 (CH), 124.7 (CH), 131.8 (CH), 128.3 (CH), 128.1 (CH), 127.8 (C), 127.7 (2CH), 127.5 (CH), 125.7 (CH), 125.4 (CH), 125.3 (CH), 125.1 (CH), 124.8 (CH), 124.7 (CH), 123.7 (CH), 123.6 (CH), 123.5 (CH), 123.4 (CH), 123.1 (CH), 121.1 (C), 108.6 (CH), 104.9 (CH), 67.1 (CH, C-2), 66.5 (CH, C-5), 61.0 (CH, C-4), 55.8 (OCH₃), 55.7 (OCH₃), 53.0 (OCH₃), 50.6 (CH, C-3), 18.1 (CH). MS (ESI): m/z 485.2 (MH⁺, 100), 425 (8), 350 (3), 280 (28), 178 (75), 174 (46), 119 (56), 91 (8), 60 (7). HRMS (ESI): m/z [MH⁺] Calcd. for C₃₂H₂₃N₂O₈: 485.16320, found 485.1628.

Methyl 4-(2-methylbenzoyl)-3-(2-chloro-5,8-dimethoxyquinolin-3-yl)-5-phenylpyrrolidine-2-carboxylate (3c)

Yield 71%. Rf (CHCl₃): 0.34. Mp 153-154 °C. IR (ATR) v 3339, 2953, 1733, 1674, 1617, 1592, 1481, 1330, 1263, 1014, 725 cm⁻¹. ¹H NMR (300 MHz, CDCl₃) δ 8.66 (s, 1H), 7.39 (d, J = 7.7, 1H), 7.24 (td, J = 7.4, J = 1.2, 1H), 7.15-7.11 (m, 6H), 6.98 (m, 2H), 6.78 (d, J = 8.5, 1H), 4.96 (d, J = 8.1, 1H), 4.73 (t, J = 7.9, 1H), 4.72 (t, J = 8.0, 1H), 4.35 (d, J = 8.2, 1H), 4.03 (s, 3H), 3.98 (s, 3H), 3.77 (s, 3H), 3.23 (brs, 1H), 1.80 (s, 3H). ¹³C NMR (75.4 MHz, CDCl₃) δ 202.14 (C=O), 173.2 (C=O), 151.3 (C), 148.9 (C), 148.6 (C), 139.7 (C), 139.1 (C), 139.0 (C), 138.0 (C), 132.7 (C), 132.1 (C), 131.8 (CH), 128.9 (CH), 128.7 (2CH), 128.0 (CH), 127.7 (2CH), 127.4 (CH), 125.7 (CH), 121.1 (C), 108.6 (CH), 104.9 (CH), 67.1 (CH, C-2), 66.5 (CH, C-5), 61.0 (CH, C-4), 55.6 (OCH₃), 56.1 (OCH₃), 52.9 (OCH₃), 51.0 (CH, C-3), 20.9 (CH). MS (ESI): m/z 545.1 (MH⁺, 100), 509 (10), 485 (5), 368 (38), 332 (3), 178 (38), 119 (15), 91 (1), 60 (1). HRMS (ESI): m/z [MH⁺] Calcd. for C₃₃H₂₉N₂O₈Cl: 545.18432, found 545.1839.

Methyl 4-(2-methylbenzoyl)-3-(2-chloro-6-methoxyquinolin-3-yl)-5-phenylpyrrolidine-2-carboxylate (3d)

Yield 62%. Rf (CHCl₃): 0.29. Mp 83-85 °C. IR (ATR) v 3336, 2952, 1736, 1677, 1597, 1494, 1338, 1215, 1219, 1044, 731 cm⁻¹. ¹H NMR (300 MHz, CDCl₃) δ 8.21 (s, 1H), 7.92 (d, J = 8.6, 1H), 7.62 (s, 1H), 7.57 (dd, J = 8.7, J = 1.7, 1H), 7.43 (d, J = 7.7, 1H), 7.26 (td, J = 7.4, J = 1.2, 1H), 7.17-7.14 (m, 6H), 6.99 (d, J = 7.5, 1H), 4.95 (d, J = 8.0, 1H) 4.78 (t, J = 8.0, 1H), 4.67 (t, J = 8.1, 1H), 4.37 (d, J = 7.9, 1H), 3.79 (s, 3H), 2.78 (brs, 1H), 2.55 (s, 3H), 1.83 (s, 3H). ¹³C NMR (75.4 MHz, CDCl₃) δ 201.8 (C=O), 173.3 (C=O), 150.3 (C), 145.7 (C), 139.8 (C), 139.0 (C), 137.8 (C), 137.8 (C), 136.9 (CH), 133.1 (CH), 132.6 (C), 132.2 (CH), 131.9 (CH), 129.0 (CH), 128.7 (2xCH), 128.3 (CH), 128.1 (CH), 127.8 (C), 127.7 (2xCH), 126.6 (CH), 125.7 (CH), 124.7 (CH), 124.6 (CH), 124.5 (CH), 124.4 (CH), 124.4 (CH), 124.3 (CH), 124.2 (CH), 123.8 (CH), 122.9 (CH), 122.7 (2xCH), 127.1 (CH), 125.3 (CH), 122.9 (CH), 121.0 (CH), 109.5 (CH), 66.2 (CH), 65.4 (CH), 58.4 (CH), 55.9 (OCH₃), 55.7 (OCH₃), 52.6 (OCH₃), 49.1 (CH), 17.7 (CH₃). HRMS (ESI): m/z [MH⁺] Calcd. for C₃₃H₂₉N₂O₈Cl: 545.18433, found 545.1843.
Methyl 4-(3,4,5-trimethoxybenzoyl)-3-(2-chloro-8-methylquinolin-3-yl)-5-phenyl pyridine-2-carboxylate (3j)

Yield 54%. Rf (CHCl3): 0.38. Mp 209-210 °C. IR (ATR) ν 3354, 2949, 1732, 1648, 1494, 1250, 1185, 915, 708 cm⁻¹. 1H NMR (300 MHz, CDCl3) δ 8.28 (1H, s), 7.71 (d, J = 7.9, 1H), 7.59 (d, J = 7.9, 1H), 7.48 (t, J = 7.9, 1H), 7.16-7.10 (m, 5H), 6.77 (s, 2H), 4.95 (d, J = 8.1, 1H), 4.71 (t, J = 8.0, 1H), 4.24-4.44 (m, 2H), 3.87 (s, 3H), 3.83 (s, 3H), 3.76 (s, 6H), 3.30 (s, 3H), 2.78 (s, 3H). 13C NMR (75.4 MHz, CDCl3) δ 198.0 (C=O), 173.4 (C=O), 152.5 (C), 149.8 (C), 145.9 (C), 142.4 (C), 137.9 (C), 136.8 (CH), 136.4 (C), 132.4 (C), 132.2 (C), 130.5 (CH), 128.3 (2×CH), 128.0 (CH), 127.3 (2×CH), 127.2 (C), 127.2 (CH), 125.3 (CH), 105.8 (2×CH), 66.4 (CH), 65.3 (CH), 60.8 (OCH3), 58.8 (CH), 56.2 (OCH3), 52.7 (OCH3), 50.0 (CH), 17.7 (CH3). HRMS (EI): m/z [M+H]+ Calcd. for C23H21N2O3: 375.1549, found 375.1517.

REFERENCES
[13] The single-crystal growth was carried out in CHCl3 at room temperature. X-ray diffraction data have been collected with an Enraf–Nonius KAPPA CCD at 293 K using Mo Kα radiation (λ = 0.71073 Å). The crystal 3e belong to the triclinic space group Pc, with unit-cell parameters α = 12.4892(2)Å, β = 13.6473(4)Å, c = 18.3062(2)Å, α = 110.709(3)°, β = 105.647(4)° and γ = 119.690(10)°. The asymmetric unit contains two molecules. Bond angles, bond distances and other crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary Publication No. CCDC-662754 for compound 3e. Copies of the data can be obtained free of charge on application to the CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK.


Received: December 14, 2009 Revised: February 27, 2010 Accepted: March 12, 2010

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