New Efficient Synthesis of 2-Substituted Benzothieno[3,2-d]pyrimidin-4(3H)-ones via a Tandem Aza-Wittig Reaction

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Abstract: 1-Aryl-3-[2-(ethoxycarbonyl)benzothien-3-yl]carbodiimides 4, obtained from aza-Wittig reactions of iminophosphorane 3 with aryl isocyanates, reacted with secondary amines, phenols, or alkanols in the presence of a catalytic amount of potassium carbonate or sodium alkoxide to give 2-substituted benzothieno[3,2-d]pyrimidin-4(3H)-ones 6 in good yields. The reaction of carbodiimides 4 with primary amines RNH₂ (R = H, Me) in the presence of sodium ethoxide selectively produced one regioisomer 8 via a base-catalyzed cyclization mechanism. However, a different regioisomer 9 was obtained when primary amines RNH₂ (R = H, Me) were used in the absence of sodium ethoxide via a direct cyclization mechanism.

Key words: benzothieno[3,2-d]pyrimidin-4(3H)-one, iminophosphorane, carbodiimide, aza-Wittig reaction, synthesis

The derivatives of fused pyrimidinones have been the focus of great interest for many years. This is probably due to the fact that many compounds containing a fused pyrimidinone ring play an important part in the biochemistry of the living cell.¹ Thienopyrimidines are of great importance because of their significant antifungal and antibacterial activities, as well as their good anticonvulsant and angiotensin or H₁ receptor antagonistic activities.²–⁶ Although some derivatives of benzothienopyrimidines have shown good antithrombotic, cardiotonic, and α-adrenergic antagonistic activities,⁷–¹⁰ there are few reports on the synthesis of benzothieno[3,2-d]pyrimidin-4(3H)-ones, which are of considerable interest as potential biologically active compounds or pharmaceuticals. The methods described for the preparation of some representative derivatives of this ring system involve the cyclization of ethyl 3-aminobenzothiophene-2-carboxylate with an orthoformate and an amine, or with formamide.¹¹–¹³ However, 2-substituted benzothieno[3,2-d]pyrimidin-4(3H)-ones are not easily accessible by current routes.

The aza-Wittig reactions of iminophosphoranes have received increased attention in view of their utility in the synthesis of nitrogen-containing heterocyclic compounds.¹⁴–¹⁷ Annulation of ring systems containing nitrogen heterocycles by means of an aza-Wittig reaction has been widely utilized because of the availability of functionalized iminophosphoranes. We are currently interested in the synthesis of quinazolinones, thienopyrimidinones, and imidazolinones via the aza-Wittig reaction of α- or β-ethoxycarbonyl substituted iminophosphoranes with aryl isocyanates and subsequent reaction with various nucleophiles under mild conditions.¹⁸–²¹ Herein we wish to report a new efficient synthesis of benzothieno[3,2-d]pyrimidin-4(3H)-ones.

Ethyl 3-aminobenzothiophene-2-carboxylate (2), readily obtained by cyclization of 2-chlorobenzonitrile (1) with ethyl 2-sulfonylacetate under basic conditions (Scheme 1),²² was converted into iminophosphorane 3 via reaction with triphenylphosphine, hexachloroethane, and triethylamine.

Scheme 1

Iminophosphorane 3 reacted with aryl isocyanates to give carbodiimides 4, which were allowed to react with secondary amines to provide guanidine intermediates 5 (Scheme 2). Even in refluxing toluene, guanidines 5 did not cyclize, however, in the presence of a catalytic amount of sodium ethoxide guanidines 5 were readily converted into 3-aryl-2-(dialkylamino)benzothieno[3,2-d]pyrimidin-4(3H)-ones 6 in satisfactory yields at room temperature. The results are listed in Table 1.

The direct reaction of carbodiimides 4 with phenols also did not produce 3-aryl-2-(arylxy)benzothieno[3,2-d]pyrimidin-4(3H)-ones 6. However, when carried out in the presence of a catalytic amount of potassium carbonate, the reaction took place to give 6 in good yields. The formation of 6 can be rationalized in terms of initial nucleophilic addition of phenoxy to the carbodiimide derivatives 4 to give the intermediates 5 that cyclize to give 6. Regardless of whether the substituents on the phenols are electron-withdrawing (Table 1, entries 13, 14, and 17) or electron-donating (Table 1, entries 15 and 16), the cyclization can
be completed smoothly under mild conditions. The direct reaction of carbodiimides 4 with alkanols gave a complex mixture; however, when the reaction was carried out in the presence of a catalytic amount of sodium alkoxide the reaction took place smoothly and 2-alkoxybenzothieno[3,2-d]pyrimidin-4(3H)-ones 6 were obtained in satisfactory yields.

Table 1 Preparation of 2-Substituted 3-Arylbenzothieno[3,2-d]pyrimidin-4(3H)-ones

<table>
<thead>
<tr>
<th>Entry</th>
<th>Product</th>
<th>Ar</th>
<th>Y</th>
<th>Conditions</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>6a</td>
<td>Ph</td>
<td>NEt₂</td>
<td>r.t., 5 h</td>
<td>84</td>
</tr>
<tr>
<td>2</td>
<td>6b</td>
<td>Ph</td>
<td>NP₆₂</td>
<td>r.t., 6 h</td>
<td>75</td>
</tr>
<tr>
<td>3</td>
<td>6c</td>
<td>Ph</td>
<td>N(i-Bu)₂</td>
<td>r.t., 6 h</td>
<td>70</td>
</tr>
<tr>
<td>4</td>
<td>6d</td>
<td>Ph</td>
<td>N(t-C₅H₁₁)₂</td>
<td>r.t., 6 h</td>
<td>66</td>
</tr>
<tr>
<td>5</td>
<td>6e</td>
<td>Ph</td>
<td>N(Me)Ph</td>
<td>r.t., 6 h</td>
<td>78</td>
</tr>
<tr>
<td>6</td>
<td>6f</td>
<td>Ph</td>
<td>morpholin-4-yl</td>
<td>r.t., 5 h</td>
<td>90</td>
</tr>
<tr>
<td>7</td>
<td>6g</td>
<td>Ph</td>
<td>piperidin-1-yl</td>
<td>r.t., 5 h</td>
<td>87</td>
</tr>
<tr>
<td>8</td>
<td>6h</td>
<td>4-MeC₆H₄</td>
<td>morpholin-4-yl</td>
<td>r.t., 5 h</td>
<td>92</td>
</tr>
<tr>
<td>9</td>
<td>6i</td>
<td>4-ClC₆H₄</td>
<td>piperidin-1-yl</td>
<td>r.t., 5 h</td>
<td>88</td>
</tr>
<tr>
<td>10</td>
<td>6j</td>
<td>4-ClC₆H₄</td>
<td>NEt₂</td>
<td>r.t., 6 h</td>
<td>81</td>
</tr>
<tr>
<td>11</td>
<td>6k</td>
<td>4-ClC₆H₄</td>
<td>NEt₂</td>
<td>r.t., 6 h</td>
<td>80</td>
</tr>
<tr>
<td>12</td>
<td>6l</td>
<td>Ph</td>
<td>OPh</td>
<td>60 °C, 5 h</td>
<td>75</td>
</tr>
<tr>
<td>13</td>
<td>6m</td>
<td>Ph</td>
<td>4-CIC₆H₄O</td>
<td>60 °C, 8 h</td>
<td>66</td>
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<tr>
<td>14</td>
<td>6n</td>
<td>Ph</td>
<td>4-BrC₆H₄O</td>
<td>60 °C, 8 h</td>
<td>73</td>
</tr>
<tr>
<td>15</td>
<td>6o</td>
<td>Ph</td>
<td>4-MeOC₆H₄O</td>
<td>60 °C, 5 h</td>
<td>87</td>
</tr>
<tr>
<td>16</td>
<td>6p</td>
<td>Ph</td>
<td>4-MeSC₆H₄O</td>
<td>60 °C, 6 h</td>
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<tr>
<td>17</td>
<td>6q</td>
<td>Ph</td>
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<td>60 °C, 10 h</td>
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<tr>
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<td>r.t., 4 h</td>
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<tr>
<td>19</td>
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<td>Ph</td>
<td>OEt</td>
<td>r.t., 6 h</td>
<td>76</td>
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<tr>
<td>20</td>
<td>6t</td>
<td>4-ClC₆H₄</td>
<td>OMe</td>
<td>r.t., 5 h</td>
<td>87</td>
</tr>
</tbody>
</table>

* Isolated yields based on iminophosphorane 3.
or methylamine (R = H, Me) gave directly 2-(phenylamino)benzothieno[3,2-d]pyrimidin-4(3H)-ones 9, another of the possible regioisomers, as the sole product in the absence of sodium ethoxide (Table 2, entries 7 and 8). The reversed, selective formation of 9 can be rationalized in terms of direct cyclization of the guanidine intermediates 7 to give 9 across the sterically smaller amino or methylamino group rather than the arylamino group. The same selectivity has been observed in similar cases. 23

\[ \text{COOEtS} \rightarrow \text{RNH}_2 \rightarrow \text{NHR}\]

![Scheme 3](image)

**Table 2 Reaction of Carbodiimides with Amines**

<table>
<thead>
<tr>
<th>Entry</th>
<th>Product</th>
<th>R</th>
<th>Conditions</th>
<th>Yield (a) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8a</td>
<td>Et</td>
<td>r.t., 4 h</td>
<td>79</td>
</tr>
<tr>
<td>2</td>
<td>8b</td>
<td>Pr</td>
<td>r.t., 4 h</td>
<td>88</td>
</tr>
<tr>
<td>3</td>
<td>8c</td>
<td>iPr</td>
<td>r.t., 6 h</td>
<td>81</td>
</tr>
<tr>
<td>4</td>
<td>8d</td>
<td>Bu</td>
<td>r.t., 4 h</td>
<td>84</td>
</tr>
<tr>
<td>5</td>
<td>8e</td>
<td>t-Bu</td>
<td>r.t., 8 h</td>
<td>85</td>
</tr>
<tr>
<td>6</td>
<td>8f</td>
<td>Bn</td>
<td>r.t., 5 h</td>
<td>80</td>
</tr>
<tr>
<td>7</td>
<td>9a</td>
<td>H</td>
<td>r.t., 1 h</td>
<td>87</td>
</tr>
<tr>
<td>8</td>
<td>9b</td>
<td>Me</td>
<td>r.t., 1 h</td>
<td>82</td>
</tr>
</tbody>
</table>

(a) Isolated yields based on iminophosphorane 3.

In conclusion, we have developed a new efficient and selective synthesis of 2-substituted benzothieno[3,2-d]pyrimidin-4(3H)-ones via reaction of functionalized carbodiimides with various amines, phenols, or alcohols. Due to the easily accessible and versatile starting material, this method can potentially be used for the synthesis of many biologically and pharmaceutically active benzothienopyrimidine derivatives.

Melting points are uncorrected. MS were measured on a Finnigan Trace MS spectrometer. IR spectra were recorded on a PE-983 spectrophotometer as KBr pellets. NMR spectra were recorded in CDCl₃ on a Varian Mercury 400 spectrometer with TMS as the internal reference. Elemental analyses were taken on a Vario EL III elementary analysis instrument.

**Ethyl 3-[(Triphenylophosphorylidenamino]benzothieno[3,2-d]pyrimidin-2-carboxylate (3)**

To a mixture of ethyl 3-aminobenzothieno[3,2-d]pyrimidin-2-carboxylate (2) 22 (1.77 g, 8 mmol), Ph₂P (3.14 g, 12 mmol), and C₂Cl₆ (2.84 g, 12 mmol) in anhyd MeCN (40 mL), was added dropwise Et₃N (2.42 g, 24 mmol) at r.t.; the mixture rapidly became yellow in color. The mixture was stirred for 4–6 h and then the solvent was removed under reduced pressure and the residue was recrystallized (EtOH) to give iminophosphorane 3 as white crystals; yield: 3.27 g (85%); mp 153–154 °C.

\[ \text{H NMR (400 MHz, CDCl₃): } \delta = 7.90–7.16 \text{ (m, 19 H, Ar-H), 3.82} \]

MS: m/z (%) = 481 (100) [M⁺], 452 (17), 408 (61), 183 (57), 77 (55).

Anal. Calcd for C₉H₉N₅O₃P: C, 72.33; H, 5.02; N, 2.91. Found: C, 72.58; H, 5.17; N, 2.84.

**3-Aryl-2-(dialkylamino)- and 3-Aryl-2-(diarylaminobenzothieno[3,2-d]pyrimidin-4(3H)-ones 6a–k; General Procedure**

To a solution of iminophosphorane 3 (1.44 g, 3 mmol) in anhyd CH₂Cl₂ (10 mL) was added the aryl isocyanate (3 mmol) under N₂ at 0–5 °C; the mixture was left to stand until the treatment reached 8–12 h. The solvent was then removed under reduced pressure and Et₂O–petroleum ether (1:2, 12 mL) was added to precipitate Ph₃PO. Removal of the solvent gave carbodiimides 4, which were used directly without further purification.

To a solution of 4 in CH₂Cl₂ (10 mL) was added dialkylamine (3 mmol); the mixture was left to stand until the treatment reached 2–3 h. The solvent was then removed and anhyd EtOH (8 mL) and EtONa (0.3 mmol, 10% equiv) in EtOH were added. The mixture was stirred at r.t. for 4–6 h. The solution was condensed and the residue was recrystallized (EtOH) to give 6a–k.

**2-(Diethylamino)-3-phenylbenzothieno[3,2-d]pyrimidin-4(3H)-one (6a)**

White crystals; mp 128–129 °C.

**2-(Diisopropylamino)-3-phenylbenzothieno[3,2-d]pyrimidin-4(3H)-one (6b)**

White crystals; mp 108–110 °C.

**2-(Diethylamino)-3-phenylbenzothieno[3,2-d]pyrimidin-4(3H)-one (6a)**

White crystals; mp 128–129 °C.

**2-(Diisopropylamino)-3-phenylbenzothieno[3,2-d]pyrimidin-4(3H)-one (6b)**

White crystals; mp 108–110 °C.

**Entry Product R Conditions Yield (a) (%)**

1. 8a Et r.t., 4 h 79
2. 8b Pr r.t., 4 h 88
3. 8c i-Pr r.t., 6 h 81
4. 8d Bu r.t., 4 h 84
5. 8e t-Bu r.t., 8 h 85
6. 8f Bn r.t., 5 h 80
7. 9a H r.t., 1 h 87
8. 9b Me r.t., 1 h 82

*Isolated yields based on iminophosphorane 3.*
MS: *m/z* (%) = 377 (63) [M⁺], 277 (72), 201 (20), 146 (100), 77 (58).

Anal. Calcd for C₁₆H₁₄N₂O₂S: C, 70.00; H, 6.14; N, 11.13. Found: C, 70.18; H, 6.25; N, 11.08.

2-(Dibutylamino)-3-phenylbenzothieno[3,2-d]pyrimidin-4(3H)-one (6c)

White crystals; mp 154–155 °C.

IR (KBr): 1678 (C=O), 1542, 1340, 729 cm⁻¹.

1H NMR (400 MHz, CDCl₃): δ = 8.28–7.33 (m, 9 H, Ar-H), 3.10 (q, \( J = 6.9 \) Hz, 4 H, 2 × NCH₂), 1.32–1.09 (m, 12 H, 6 × CH₃), 0.84 (t, \( J = 7.0 \) Hz, 6 H, 2 × CH₂).

MS: *m/z* (%) = 433 (67) [M⁺], 277 (100), 201 (16), 146 (90), 77 (25).


2-(Morpholin-4-yl)-3-phenylbenzothieno[3,2-d]pyrimidin-4(3H)-one (6h)

White crystals; mp 195–197 °C.

IR (KBr): 1681 (C=O), 1533, 1369, 754 cm⁻¹.

1H NMR (400 MHz, CDCl₃): δ = 8.27–7.21 (m, 8 H, Ar-H), 3.21 (q, \( J = 4.8 \) Hz, 4 H, 2 × NCH₂), 2.41 (s, 3 H, CH₃), 0.90 (t, \( J = 7.0 \) Hz, 6 H, 2 × CH₂).

MS: *m/z* (%) = 395 (100) [M⁺], 291 (10), 146 (39), 111 (15).


2-(Dipentylamino)-3-phenylbenzothieno[3,2-d]pyrimidin-4(3H)-one (6j)

White crystals; mp 134–135 °C.

IR (KBr): 1670 (C=O), 1542, 1340, 754 cm⁻¹.

1H NMR (400 MHz, CDCl₃): δ = 8.26–7.34 (m, 8 H, Ar-H), 3.17 (q, \( J = 5.2 \) Hz, 4 H, 2 × NCH₂), 1.60–1.34 (m, 6 H, 3 × CH₂).

MS: *m/z* (%) = 419 (100) [M⁺], 310 (20), 146 (25), 111 (15).

Anal. Calcd for C₂₃H₂₃N₃OS: C, 72.05; H, 4.47; N, 10.96. Found: C, 72.05; H, 4.54; N, 10.80.

2-(Morpholin-4-yl)-3-phenylbenzothieno[3,2-d]pyrimidin-4(3H)-one (6k)

White crystals; mp 166–168 °C.

IR (KBr): 1674 (C=O), 1530, 1381, 695 cm⁻¹.

1H NMR (400 MHz, CDCl₃): δ = 8.28–7.41 (m, 9 H, Ar-H), 3.48 (t, \( J = 4.8 \) Hz, 4 H, 2 × NCH₂), 3.21 (t, \( J = 4.8 \) Hz, 4 H, 2 × OCH₂).

13C NMR (100 MHz, CDCl₃): δ = 159.4 (C=O), 156.7, 151.9, 142.0, 136.9, 134.4, 129.0, 128.7, 128.6, 128.5, 124.7, 123.6, 123.4, 117.4, 65.9, 49.4.

MS: *m/z* (%) = 363 (48) [M⁺], 306 (31), 277 (49), 146 (100), 103 (38).

Anal. Calcd for C₁₅H₁₂N₂O₂S: C, 66.10; H, 4.71; N, 11.56. Found: C, 66.02; H, 4.94; N, 11.47.

3-Phenyl-2-(piperidin-1-yl)benzothieno[3,2-d]pyrimidin-4(3H)-one (6p)

White crystals; mp 177–178 °C.

IR (KBr): 1673 (C=O), 1540, 1374, 700 cm⁻¹.
2-Phenoxo-3-phenylbenzothieno[3,2-d]pyrimidin-4(3H)-one (6l)
White crystals; mp 215–216 °C.
IR (KBr): 1668 (C=O), 1488, 1348, 704 cm⁻¹.

1H NMR (400 MHz, CDCl₃): δ = 7.99 (d, J = 7.6 Hz, 1 H, Ar-H), 7.87 (d, J = 8.0 Hz, 1 H, Ar-H), 7.59–7.20 (m, 12 H, Ar-H).
13C NMR (100 MHz, CDCl₃): δ = 158.8 (C=O), 154.8, 151.9, 150.8, 141.8, 134.8, 133.9, 129.4, 129.1, 128.8, 128.1, 125.9, 124.7, 123.7, 123.2, 121.4, 118.0.

MS: m/z (%) = 416 (71) [M⁺], 277 (62), 200 (11), 146 (92), 139 (100), 77 (71).
Anal. Calcd for C₂₃H₁₉N₅O₂S: C, 66.32; H, 3.87; N, 6.73. Found: C, 66.60; H, 3.94; N, 6.67.

2-(3-Nitrophenoxy)-3-phenylbenzothieno[3,2-d]pyrimidin-4(3H)-one (6q)
White crystals; mp 226–227 °C.
IR (KBr): 1676 (C=O), 1520, 1346, 695 cm⁻¹.

1H NMR (400 MHz, CDCl₃): δ = 8.28–7.28 (m, 9 H, Ar-H), 4.05 (s, 3 H, OCH₃).
13C NMR (100 MHz, CDCl₃): δ = 159.0 (C=O), 155.3, 151.3, 141.9, 134.9, 134.2, 129.2, 128.8, 128.7, 128.6, 128.1, 124.6, 123.5, 123.4, 117.0, 65.1, 14.0.

MS: m/z (%) = 322 (4) [M⁺], 293 (6), 200 (9), 145 (100), 102 (32), 77 (50).

2-Alkoxy-3-phenylbenzothieno[3,2-d]pyrimidin-4(3H)-ones 6r–v

General Procedure
To the soln of 4 (ca. 3 mmol) in anhyd alkanoil ROH (8 mL) was added EtONa (0.3 mmol, 10% equiv.) in EtOH. The mixture was stirred for at r.t 4–6 h. The soln was condensed and the residue was recrystallized (ROH) to give 6r–v.

2-Methoxy-3-phenylbenzothieno[3,2-d]pyrimidin-4(3H)-one (6r)
White crystals; mp 212–214 °C.
IR (KBr): 1676 (C=O), 1520, 1346, 695 cm⁻¹.

1H NMR (400 MHz, CDCl₃): δ = 8.28–7.28 (m, 9 H, Ar-H), 4.05 (s, 3 H, OCH₃).

MS: m/z (%) = 308 (100) [M⁺], 277 (51), 200 (11), 146 (93), 77 (58).

2-Ethoxy-3-phenylbenzothieno[3,2-d]pyrimidin-4(3H)-one (6s)
White crystals; mp 190–192 °C.
IR (KBr): 1673 (C=O), 1522, 1334, 698 cm⁻¹.

1H NMR (400 MHz, CDCl₃): δ = 8.26–7.26 (m, 9 H, Ar-H), 4.58 (q, J = 6.9 Hz, 2 H, OCH₂).

MS: m/z (%) = 342 (100) [M⁺], 293 (6), 200 (9), 145 (100), 102 (32), 77 (50).
Anal. Calcd for C₁₇H₁₄N₂O₂S: C, 67.06; H, 4.38; N, 8.69. Found: C, 67.20; H, 4.29; N, 8.77.

3-(4-Chlorophenyl)-2-methoxybenzothieno[3,2-d]pyrimidin-4(3H)-one (6t)
White crystals; mp 174–176 °C.
IR (KBr): 1668 (C=O), 1523, 1346, 694 cm⁻¹.

1H NMR (400 MHz, CDCl₃): δ = 8.19–7.41 (m, 13 H, Ar-H).

MS: m/z (%) = 349 (5) [M⁺], 277 (9), 200 (11), 146 (37), 77 (100).

2-Allylribino[3,2-d]pyrimidin-4(3H)-one 6u

General Procedure
To the soln of 4 (ca. 3 mmol) in CH₂Cl₂ (10 mL) was added alkylamine (3 mmol) and the mixture was allowed to stand unstirred for at 4–6 h. The soln was stirred at r.t 4–6 h. The soln was condensed and the residue was recrystallized (EtOH) to give 6u–v.
several minutes. The solvent was removed and anhyd EtOH (8 mL) with EtONa (0.3 mmol, 10% equiv) in EtOH was added. The mixture was stirred at r.t. for 4–6 h. The soln was condensed and the residue was recrystallized (EtOH) to give 8a-f.

2-(Ethylamino)-3-phenylbenzothieno[3,2-d]pyrimidin-4(3H)-one (8a)
White crystals; mp 191–193 °C.

IR (KBr): 1673 (C=O), 1544, 1321, 756 cm⁻¹.

1H NMR (400 MHz, CDCl₃): δ = 8.27–7.34 (m, 9 H, Ar-H), 4.17 (s, 1 H, NH), 3.56–3.45 (m, 2 H, NCH₂), 1.34–1.28 (m, 4 H, 2 × CH₂), 0.90 (t, J = 7.0 Hz, 3 H, CH₃).

MS: m/z (%) = 321 (55) [M⁺], 277 (32), 200 (9), 146 (62), 77 (24).

2-(Butylamino)-3-phenylbenzothieno[3,2-d]pyrimidin-4(3H)-one (8b)
White crystals; mp 167–169 °C.

IR (KBr): 1675 (C=O), 1546, 1324, 756 cm⁻¹.

1H NMR (400 MHz, DMSO): δ = 8.27–7.33 (m, 9 H, Ar-H), 4.59 (s, 1 H, NH), 3.56–3.51 (m, 2 H, NCH₂), 1.62–1.56 (q, J = 7.0 Hz, 2 H, CH₂), 0.90 (t, J = 7.0 Hz, 3 H, CH₃).

MS: m/z (%) = 335 (58) [M⁺], 277 (22), 200 (8), 146 (9), 77 (26).

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References

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