Cobalt-catalyzed Arylation of Azole Heteroarenes via Direct C-H Bond Functionalization

Bengü Sezen and Dalibor Sames*

Department of Chemistry, Columbia University, 3000 Broadway, New York, NY 10027

Supporting Information

I. Results for Cobalt and Palladium-catalyzed Arylation of Benzo-azoles

Table 1. Cobalt-catalyzed arylation of benzothiazole. A comparison with palladium-based methods

entry	conditions	yield
1	$\text{Co}(\text{OAc})_2$, SALEN (10 mol%), dioxane, CuI (0.2 eq)	87 %
2	$\text{Co}(\text{OAc})_2$, IMes (10 mol%), dioxane, CuI (0.2 eq)	90 %
3	$\text{Pd}(\text{OAc})_2$, Ph_3P (20 mol%), DMF, CuI (0.2 eq)	89 %
4	control (no cat./ligand), dioxane, CuI (0.2 eq)	traces

Table 2. Cobalt-catalyzed arylation of benzoxazole. A comparison with palladium-based methods

entry	conditions	yield
1	$\text{Co}(\text{OAc})_2$, SALEN (10 mol%), dioxane, CuI (0.2 eq)	91 %
2	$\text{Co}(\text{OAc})_2$, IMes (10 mol%), dioxane, CuI (0.2 eq)	92 %
3	$\text{Pd}(\text{OAc})_2$, Ph_3P (20 mol%), DMF, CuI (0.2 eq)	92 %
4	control (no cat./ligand), dioxane, CuI (0.2 eq)	38 %

Table 3. Cobalt-catalyzed arylation of benzimidazole. A comparison with palladium-based methods

entry	conditions	yield
1	Co(OAc) ₂ , SALEN (10 mol%), DMF, CuI (0.2 eq)	53 %
2	Co(OAc) ₂ , IMes (10 mol%), DMF, CuI (0.2 eq)	78 %
3	Pd(OAc) ₂ , Ph ₃ P (20 mol%), dioxane, CuI (0.2 eq)	90 %
4	control (no cat./ligand), DMF, CuI (0.2 eq)	traces

II. Experimental Procedures and Characterization Data

Anhydrous Co(OAc)₂ was purchased from Aldrich and used without any further treatment. All starting heterocycles were purchased from Aldrich or Lancaster and used as received except for imidazole which was recrystallized from 1:2 mixture of dichloromethane and diethylether prior to use in arylation reactions. All aryl donors were purchased from Aldrich and used as received. Dioxane was dried over Na-benzophenone and DMF was dried by the alumina/copper catalyst solvent purification column.

Co(OAc)₂ and Cs₂CO₃ are extremely hygroscopic and must be kept away from moisture. Anhydrous Co(OAc)₂ was used throughout this study. Use of Co(OAc)₂.xH₂O for the described methodology gives low to trace amount of yields. Cs₂CO₃ was dried at 150 °C under *vacuo* for 2 hours prior to each use.

Thiazole, oxazole and their mono-arylation products are volatile, care must be taken during the solvent evaporation step of arylation reactions of these compounds. The reactions were performed in closed systems (using pressure vessels having screw caps equipped with a resealable PTFE/silicone liner) due to the low boiling points of the starting materials and dioxane.

Procedure A: Catalytic arylation of thiazole at C-5 with Co(OAc)₂ / SALEN

In a flame dried flask, anhydrous Co(OAc)₂ (3.7 mg, 0.021 mmol, 5 mol %) and SALEN [ethylenebis(salicylimine)] (11.0 mg, 0.041 mmol, 0.1 equiv) in 0.5 ml dry DMF were stirred for 10 min at room temperature. Thiazole (35 mg, 0.411 mmol) in 1 ml dry DMF and anhydrous Cs₂CO₃ (160.7 mg, 0.493 mmol, 1.2 equiv) were added consecutively to the above mixture under argon. Phenyl iodide (100.7 mg, 0.493 mmol, 1.2 equiv), which was dissolved in 0.5 ml dry DMF, was added dropwise next and the reaction mixture was heated to 150 °C under argon. The reaction propagation was monitored by TLC and upon completion (after about 12 hours, when the relative amount of desired product reached its maximum, longer reaction times may lead to further arylation of the product) of the reaction, the resulting reaction mixture was diluted with CHCl₃ (20 ml) and filtered through a pad of celite. The solvents were evaporated and **5-phenylthiazole 2** (27.2 mg, 41 %) was isolated by flash column chromatography (gradient elution hexanes □ 10 % ethyl acetate / 90 % hexanes). The spectral data for this compound are in agreement with the literature values.¹

Procedure B: Catalytic arylation of thiazole at C-5 with Co(OAc)₂ / IMes

In a flame dried flask, anhydrous Co(OAc)₂ (3.7 mg, 0.021 mmol, 5 mol %), 1,3-bis(2,4,6-trimethylphenyl)imidazolium chloride (14.0 mg, 0.041 mmol, 0.1 equiv) and K₃PO₄ (8.8 mg, 0.041 mmol, 0.1 equiv) in 0.5 ml dry DMF were stirred for 10 min at room temperature. Thiazole (35 mg, 0.411 mmol) in 1 ml dry DMF and anhydrous Cs₂CO₃ (160.7 mg, 0.493 mmol, 1.2 equiv) were added consecutively to the above mixture under argon. Phenyl iodide (100.7 mg, 0.493 mmol, 1.2 equiv), which was dissolved in 0.5 ml dry DMF, was added dropwise next and the reaction mixture was heated to 150 °C under argon. The reaction propagation was monitored by TLC and upon completion (after about 12 hours, when the relative amount of desired product reached its maximum, longer reaction times may lead to further arylation of the product) of the reaction, the resulting reaction mixture was diluted with CHCl₃ (20 ml) and filtered through a pad of celite. The solvents were evaporated and **5-phenylthiazole 2** (42.4 mg, 64 %) was isolated by flash column chromatography (gradient elution hexanes □ 10 % ethyl acetate / 90 % hexanes). The spectral data for this compound are in agreement with the literature values.¹

¹H NMR data (400MHz, CDCl₃)

H-1	8.78, 1H, brs
H-2	8.10, 1H, brs
H-3	7.58-7.60, 2H, m
H-4, H-5	7.33-7.44, 3H, m

¹³C NMR data (100MHz, CDCl₃)

126.6 (quat. C)
126.9 (2 C-H's)
128.3 (C-H)
128.9 (2 C-H's)
131.0 (quat. C)
139.1 (C-H)
152.4 (C-H)

IR (NaCl plate) $\bar{\nu}_{\text{max}}$ 3061, 2957, 1653, 1559, 1540, 1457, 1379, 1286 cm⁻¹.

HRMS (FAB ⁺) calculated for C₉H₈NS m/z 162.0377, observed m/z 162.0365.

Procedure C: Catalytic arylation of thiazole at C-2 with Co(OAc)₂ / SALEN / CuI

In a flame dried flask, anhydrous Co(OAc)₂ (3.7 mg, 0.021 mmol, 5 mol %) and SALEN [ethylenebis(salicylimine)] (11.0 mg, 0.041 mmol, 0.1 equiv) in 0.5 ml dry dioxane were stirred for 10 min at room temperature. Thiazole (35 mg, 0.411 mmol) in 1 ml dry dioxane, anhydrous Cs₂CO₃ (160.7 mg, 0.493 mmol, 1.2 equiv) and CuI (156.5 mg, 0.822 mmol, 2 equiv) were added consecutively to the above mixture under argon. Phenyl iodide (100.7 mg, 0.493 mmol, 1.2 equiv), which was dissolved in 0.5 ml dry dioxane, was added dropwise next and the reaction mixture was heated to 150 °C under argon. The reaction propagation was monitored by TLC and upon completion (after about 10 hours, when the relative amount of desired product reached its maximum, longer reaction times may lead to further arylation of the product) of the reaction, the resulting reaction mixture was diluted with CHCl₃ (20 ml) and filtered through a pad of celite. The solvents were evaporated and **2-phenylthiazole 3** (57.7 mg, 87 %) was isolated by flash column chromatography (gradient elution hexanes $\square$ 10 % ethyl acetate / 90 % hexanes). The spectral data for this compound are in agreement with the literature values.²

Procedure D: Catalytic arylation of thiazole at C-2 with Co(OAc)₂ / IMes / CuI

In a flame dried flask, anhydrous Co(OAc)₂ (3.7 mg, 0.021 mmol, 5 mol %), 1,3-bis(2,4,6-trimethylphenyl)imidazolium chloride (14.0 mg, 0.041 mmol, 0.1 equiv) and K₃PO₄ (8.8 mg, 0.041 mmol, 0.1 equiv) in 0.5 ml dry dioxane were stirred for 10 min at room temperature. Thiazole (35 mg, 0.411 mmol) in 1 ml dry dioxane, anhydrous Cs₂CO₃ (160.7 mg, 0.493 mmol, 1.2 equiv) and CuI (156.5 mg, 0.822 mmol, 2 equiv) were added consecutively to the above mixture under argon. Phenyl iodide (100.7 mg, 0.493 mmol, 1.2 equiv), which was dissolved in 0.5 ml dry dioxane, was added dropwise next and the reaction mixture was heated to 120 °C under argon. The reaction propagation was monitored by TLC and upon completion (after about 10 hours, when the relative amount of desired product reached its maximum, longer reaction times may lead to further arylation of the product) of the reaction, the resulting reaction mixture was diluted with CHCl₃ (20 ml) and filtered through a pad of celite. The solvents were evaporated and **2-phenylthiazole 3** (55.7 mg, 84 %) was isolated by flash column chromatography (gradient elution hexanes □ 10 % ethyl acetate / 90 % hexanes). The spectral data for this compound are in agreement with the literature values.²

¹H NMR data (400MHz, CDCl₃)

H-1	7.87, 1H, d, J= 3.3 Hz
H-2	7.33, 1H, d, J= 3.3 Hz
H-3	7.95-7.98, 2H, m
H-4 and H-5	7.39-7.45, 3H, m

¹³C NMR data (100MHz, CDCl₃)

118.9 (C-H)
126.7 (2 C-H's)
129.1 (2 C-H's)
130.1 (C-H)
133.7 (quat. C)
143.8 (C-H)
168.4 (quat. C)

IR (NaCl plate) $\bar{\nu}_{\text{max}}$ 3111, 3081, 1509, 1479, 1442, 1411, 1240 cm⁻¹.

HRMS (EI⁺) calculated for C₉H₇SN *m/z* 161.0299, observed *m/z* 161.0308.

2,5-Diphenylthiazole 4 was obtained as a side product of the Pd(OAc)₂ catalyzed arylation reaction of thiazole. The spectral data for this compound are in agreement with the literature values.²

¹H NMR data (400MHz, CDCl₃)

H-1	8.02, 1H, s
H-2	7.97, 2H, dd, J ₁ = 1.6, J ₂ = 7.7 Hz
H-3, H-4, H-6	7.39-7.47, 5H, m
H-5	7.61, 2H, J ₁ = 1.4, J ₂ = 7.3 Hz
H-7	7.34, 1H, tt, J ₁ = 2.1, J ₂ = 7.4 Hz

¹³C NMR data (100MHz, CDCl₃)

126.2 (2 C-H's)
126.4 (quat. C)
126.5 (2 C-H's)
128.2 (C-H)
128.8 (2 C-H's)
129.0 (2 C-H's)
129.9 (C-H)
131.3 (quat.C)
133.6 (quat. C)
139.1 (C-H)
167.0 (quat. C)

IR (NaCl plate) $\bar{\nu}_{\text{max}}$ 3056, 2920, 1521, 1480, 1446, 1066 cm⁻¹.

HRMS (EI⁺) calculated for C₁₅H₁₁SN *m/z* 237.0612, observed *m/z* 237.0602.

5-Phenyloxazole 6 was obtained from oxazole in 18 % yield by following procedure **A** and in 34 % yield by following procedure **B**. The spectral data for this compound are in agreement with the literature values.³

¹H NMR data (400MHz, CDCl₃)

H-1	7.88, 1H, brs
H-2, H-4, H-5	7.23-7.46, 4H, m
H-3	7.60-7.63, 2H, m

¹³C NMR data (100MHz, CDCl₃)

124.4 (C-H)
127.8 (2 C-H's)
128.7 (C-H)
128.9 (2 C-H's)
150.6 (quat. C)
151.6 (C-H)

IR (NaCl plate) $\bar{\nu}_{\text{max}}$ 3068, 1725, 1669, 1465, 1373, 1249 cm⁻¹.

HRMS (FAB⁺) calculated for C₉H₈ON *m/z* 146.0606, observed *m/z* 146.0602.

2-Phenyloxazole 7 was obtained in 53 % yield from oxazole by following procedure **C** and in 73 % yield by following procedure **D**. The spectral data for this compound are in agreement with the literature values.⁴

¹H NMR data (400MHz, CDCl₃)

H-1	7.22, 1H, d, J= 0.7 Hz
H-2	7.68, 1H, d, J= 0.7 Hz
H-3	8.03-8.06, 2H, m
H-4 and H-5	7.41-7.45, 3H, m

¹³C NMR data (100MHz, CDCl₃)

126.4 (2 C-H's)
127.6 (quat. C)
128.5 (C-H)
128.8 (2 C-H's)
130.3 (C-H)
38.6 (C-H)
62.0 (quat. C)

IR (NaCl plate) $\bar{\nu}_{\max}$ 3162, 3125, 3065, 1724, 1557, 1483, 1449, 1070 cm⁻¹.

HRMS (FAB⁺) calculated for C₉H₈ON *m/z* 146.0606, observed *m/z* 146.0608.

2,5-Diphenyloxazole 8 was obtained as a side product of the Pd(OAc)₂ catalyzed arylation reaction of oxazole. The spectral data for this compound are in agreement with the literature values.²

¹H NMR data (400MHz, CDCl₃)

H-1	7.45, 1H, s
H-2	8.10-8.13, 2H, m
H-3, H-4, H-6	7.43-7.50, 5H, m
H-5	7.71-7.74, 2H, m
H-7	7.34, 1H, tt, J ₁ = 1.2, J ₂ = 7.4 Hz

¹³C NMR data (100MHz, CDCl₃)

123.6 (C-H)
124.3 (2 C-H's)
126.4 (2 C-H's)
127.6 (quat. C)
128.1 (quat. C)
128.6 (C-H)
129.0 (2 C-H's)
129.1 (2 C-H's)
130.5 (C-H)
151.4 (quat. C)
161.3 (quat. C)

IR (NaCl plate) $\bar{\nu}_{\max}$ 3118, 3059, 1587, 1481, 1445, 1133, 1069, 1024 cm⁻¹.

HRMS (EI⁺) calculated for C₁₅H₁₁ON *m/z* 221.0841, observed *m/z* 221.0835.

2-Phenylbenzothiazole 14 was obtained in 87 % yield from benzothiazole by following procedure **C** and in 90 % yield by following procedure **D** (using 0.2 equiv of CuI instead of 2 equiv). The spectral data for this compound are in agreement with the literature values.⁵

¹H NMR data (400MHz, CDCl₃)

H-1	7.86, 1H, d, J= 8.0 Hz
H-2 – H-4	8.06-8.09, 3H, m
H-5 – H-7	7.31-7.37, 1H, m
	7.43-7.49, 4H, m

¹³C NMR data (100MHz, CDCl₃)

121.5 (C-H)
123.2 (C-H)
125.1 (C-H)
126.2 (C-H)
127.5 (2 C-H's)
128.9 (2 C-H's)
130.9 (C-H)
133.6 (quat. C)
135.0 (quat. C)
154.1 (quat. C)
168.0 (quat. C)

IR (NaCl plate) $\bar{\nu}_{\text{max}}$ 3056, 1623, 1572, 1441, 1214, 1053 cm⁻¹.

2-Phenylbenzoxazole 16 was obtained in 91 % yield from benzoxazole by following procedure **C** and in 92 % yield by following procedure **D** (using 0.2 equiv of CuI instead of 2 equiv). The spectral data for this compound are in agreement with the literature values.⁶

¹H NMR data (400MHz, CDCl₃)

H-1 – H-4	7.55-7.57, 1H, m
	7.76-7.78, 1H, m
	8.23-8.26, 2H, m
H-5 – H-7	7.32-7.35, 2H, m
	7.49-7.52, 3H, m

¹³C NMR data (100MHz, CDCl₃)

110.5 (C-H)
120.0 (C-H)
124.5 (C-H)
125.0 (C-H)
127.1 (quat. C)
127.6 (2 C-H's)
128.8 (2 C-H's)
131.4 (C-H)
142.1 (quat. C)
150.7 (quat. C)
163.0 (quat. C)

IR (NaCl plate) $\bar{\nu}_{\text{max}}$ 3052, 1632, 1544, 1452, 1427 cm⁻¹.

Procedure E: Catalytic arylation of imidazole at C-5 with Co(OAc)₂ / SALEN

In a flame dried flask, anhydrous Co(OAc)₂ (3.9 mg, 0.022 mmol, 5 mol %) and SALEN [ethylenebis(salicylimine)] (11.8 mg, 0.044 mmol, 0.1 equiv) in 0.5 ml dry DMF were stirred for 10 min at room temperature. Imidazole (30 mg, 0.441 mmol) in 1 ml dry DMF and MgO (21.3 mg, 0.529 mmol, 1.2 equiv) were added consecutively to the above mixture under argon and the resultant mixture was stirred vigorously for an additional 10 min. (A homogeneous suspension must be obtained at this point, without any purple undissolved Co(OAc)₂ or big particles of MgO at the bottom of the flask). Phenyl iodide (107.9 mg, 0.529 mmol, 1.2 equiv), which was dissolved in 0.5 ml dry DMF, was added dropwise next and the reaction mixture was heated to 150 °C under argon. The reaction propagation was monitored by TLC and upon completion (after about 12 hours, when the relative amount of desired product reached its maximum, longer reaction times may lead to further arylation of the product) of the reaction, the resulting reaction mixture was diluted with CHCl₃ (20 ml) and filtered through a pad of celite. The solvents were evaporated and **4(5)-phenylimidazole 10** (17.2 mg, 27 %) was isolated by flash column chromatography (gradient elution hexanes □ 20 % ethyl acetate / 80 % hexanes). The spectral data for this compound are in agreement with the literature values.⁷

Procedure F: Catalytic arylation of imidazole at C-5 with Co(OAc)₂ / IMes

In a flame dried flask, anhydrous Co(OAc)₂ (3.9 mg, 0.022 mmol, 5 mol %), 1,3-bis(2,4,6-trimethylphenyl)imidazolium chloride (15.0 mg, 0.044 mmol, 0.1 equiv) and K₃PO₄ (9.4 mg, 0.044 mmol, 0.1 equiv) in 0.5 ml dry DMF were stirred for 10 min at room temperature. Imidazole (30 mg, 0.441 mmol) in 1 ml dry DMF and ZnO (43.0 mg, 0.529 mmol, 1.2 equiv) were added consecutively to the above mixture under argon and the resultant mixture was stirred vigorously for an additional 10 min. (A homogeneous suspension must be obtained at this point, without any purple undissolved Co(OAc)₂ or big particles of ZnO at the bottom of the flask). Phenyl iodide (107.9 mg, 0.529 mmol, 1.2 equiv), which was dissolved in 0.5 ml dry DMF, was added dropwise next and the reaction mixture was heated to 150 °C under argon. The reaction propagation was monitored by TLC and upon completion (after about 12 hours, when the relative amount of desired product reached its maximum, longer reaction times may lead to further arylation of the product) of the reaction, the resulting reaction mixture was diluted with CHCl₃ (20 ml) and filtered through a pad of celite. The solvents were evaporated and **4(5)-phenylimidazole 10** (26.0 mg, 41 %) was isolated by flash column chromatography (gradient elution hexanes □ 20 % ethyl acetate / 80 % hexanes). The spectral data for this compound are in agreement with the literature values.⁷

¹H NMR data (400MHz, d₆-DMSO)

H-1	7.75, 1H, s
H-2	7.60, 1H, s
H-3	7.78, 2H, d, J= 7.8 Hz
H-4	7.36, 2H, t, J= 7.7 Hz
H-5	7.19, 1H, t, J= 7.4 Hz
N-H	12.33, 1H, brs

¹³C NMR data (100MHz, d₆-DMSO)

114.7 (C-H)
124.1 (2 C-H's)
126.0 (C-H)
128.4 (2 C-H's)
133.9 (quat. C)
135.9 (C-H)
138.2 (quat. C)

IR (NaCl plate) $\bar{\nu}_{\text{max}}$ 3091, 3060, 1605, 1462, 1312, 1118, 1068 cm⁻¹.

Procedure G: Catalytic arylation of imidazole at C-2 with Co(OAc)₂ / SALEN / CuI

In a flame dried flask, anhydrous Co(OAc)₂ (3.9 mg, 0.022 mmol, 5 mol %) and SALEN [ethylenebis(salicylimine)] (11.8 mg, 0.044 mmol, 0.1 equiv) in 0.5 ml dry DMF were stirred for 10 min at room temperature. Imidazole (30 mg, 0.441 mmol) in 1 ml dry DMF and MgO (21.3 mg, 0.529 mmol, 1.2 equiv) were added consecutively to the above mixture under argon and the resultant mixture was stirred vigorously for an additional 10 min. (A homogeneous suspension must be obtained at this point, without any purple undissolved Co(OAc)₂ or big particles of MgO at the bottom of the flask). CuI (167.8 mg, 0.881 mmol, 2 equiv) and followed by phenyl iodide (107.9 mg, 0.529 mmol, 1.2 equiv), which was dissolved in 0.5 ml dry DMF, were added next and the reaction mixture was heated to 150 °C under argon. The reaction propagation was monitored by TLC and upon completion (after about 12 hours, when the relative amount of desired product reached its maximum, longer reaction times may lead to further arylation of the product) of the reaction, the resulting reaction mixture was diluted with CHCl₃ (20 ml) and filtered through a pad of celite. The solvents were evaporated and **2-phenylimidazole 11** (34.3 mg, 54 %) was isolated by flash column chromatography (gradient elution hexanes $\square$ 20 % ethyl acetate / 80 % hexanes). The spectral data for this compound are in agreement with the literature values.⁸

Procedure H: Catalytic arylation of imidazole at C-2 with Co(OAc)₂ / IMes / CuI

In a flame dried flask, anhydrous Co(OAc)₂ (3.9 mg, 0.022 mmol, 5 mol %), 1,3-bis(2,4,6-trimethylphenyl)imidazolium chloride Cl (15.0 mg, 0.044 mmol, 0.1 equiv) and K₃PO₄ (9.4 mg, 0.044 mmol, 0.1 equiv) in 0.5 ml dry DMF were stirred for 10 min at room temperature. Imidazole (30 mg, 0.441 mmol) in 1 ml dry DMF and ZnO (43.0 mg, 0.529 mmol, 1.2 equiv) were added consecutively to the above mixture under argon and the resultant mixture was stirred vigorously for an additional 10 min. (A homogeneous suspension must be obtained at this point, without any purple undissolved Co(OAc)₂ or big particles of ZnO at the bottom of the flask). CuI (167.8 mg, 0.881 mmol, 2 equiv) and followed by phenyl iodide (107.9 mg, 0.529 mmol, 1.2 equiv), which was dissolved in 0.5 ml dry DMF, were added next and the reaction mixture was heated to 150 °C under argon. The reaction propagation was monitored by TLC and upon completion (after about 12 hours, when the relative amount of desired product reached its maximum, longer reaction times may lead to further arylation of the product) of the reaction, the resulting reaction mixture was diluted with CHCl₃ (20 ml) and filtered through a pad of celite. The solvents were evaporated and **2-phenylimidazole 11** (49.6 mg, 78 %) was isolated by flash column chromatography (gradient elution hexanes □ 20 % ethyl acetate / 80 % hexanes). The spectral data for this compound are in agreement with the literature values.⁸

¹H NMR data (400MHz, CDCl₃)

H-1	7.13, 2H, s
H-2	7.86-7.90, 2H, m
H-3, H-4	7.28-7.35, 3H, m
N-H	11.23, 1H, brs

¹³C NMR data (100MHz, CDCl₃)

123.2 (2 C-H's)
125.5 (2 C-H's)
128.5 (C-H)
128.8 (2 C-H's)
130.3 (quat. C)
147.2 (quat. C)

IR (NaCl plate) □_{max} 3053, 2906, 1564, 1504, 1488, 1416, 1106, 948 cm⁻¹.

2-Phenylbenzimidazole 18 was obtained in 53 % yield from benzimidazole by following procedure **G**, and in 78 % yield by following procedure **H** (using 0.2 equiv of CuI instead of 2 equiv) and the spectral data for this compound are in agreement with the literature values.⁹

¹ H NMR data (400MHz, d ₆ -DMSO)		¹³ C NMR data (100MHz, d ₆ -DMSO)	
H-1	8.22, 2H, d, J= 7.6 Hz		115.3 (2 C-H's)
H-2 – H-5	7.20-7.23, 2H, m		122.0 (2 C-H's)
	7.47-7.66, 5H, m		126.3 (2 C-H's)
N-H	12.98, 1H, brs		128.8 (2 C-H's)
			129.7 (C-H)
			130.1 (quat. C)
			134.9 (quat. C)
			143.7 (quat. C)
			151.2 (quat. C)

IR (NaCl plate) $\bar{\nu}_{\text{max}}$ 3166, 3056, 1661, 1443, 1050, 1037 cm⁻¹.

References

1. Pavlik, J. W.; Tongcharoensirikul, P.; Bird, N. P.; Day, A. C.; Barltrop, J. A. *J. Am. Chem. Soc.* **1994**, *116*, 2292.
2. Pivsa-Art, S.; Satoh, T.; Kawamura, Y.; Miura, M. Nomura, M. *Bull. Chem. Soc. Jpn.* **1998**, *71*, 467.
3. Kulkarni, B. A.; Ganesan, A. *Tet. Lett.* **1999**, *40*, 5633.
4. Prager, R. H.; Smith, J. A.; Weber, B.; Williams, C. M. *J. Chem. Soc. Perkin Trans. 1* **1997**, *17*, 2665.
5. The Aldrich Library of ¹³C and ¹H FT NMR Spectra Ed.1, Pouchert, C. J.; Behnke, J. **1993**, 3, 202-C.
6. The Aldrich Library of ¹³C and ¹H FT NMR Spectra Ed.1, Pouchert, C. J.; Behnke, J. **1993**, 3, 198-B.
7. The Aldrich Library of ¹³C and ¹H FT NMR Spectra Ed.1, Pouchert, C. J.; Behnke, J. **1993**, 3, 82A.
8. Foces, C.; Llamas-Saiz, A. L.; Claramunt, R. M.; Cabildo, P.; Elguero, J. *J. Mol. Str.* **1998**, *440*, 193.
9. The Aldrich Library of ¹³C and ¹H FT NMR Spectra Ed.1, Pouchert, C. J.; Behnke, J. **1993**, 3, 180A.