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Palladium-catalyzed carbonylative cyclization of 1-bromoallyl bromides with anilines leading to 1-aryl-1*H*-pyrrol-2(5*H*)-ones

Chan Sik Cho^{a*}, Jong Ik Son^a and Nam Sik Yoon^b

1-Bromoallyl bromides are carbonylatively cyclized with anilines under carbon monoxide pressure in DMF in the presence of a catalytic amount of a palladium catalyst along with a base to give the corresponding 1-aryl-1*H*-pyrrol-2(5*H*)-ones in moderate to good yield. Copyright © 2012 John Wiley & Sons, Ltd.

Keywords: anilines; 1-aryl-1*H*-pyrrol-2(5*H*)-ones; 1-bromoallyl bromides; carbonylative cyclization; palladium catalyst

Introduction

Transition metal-catalyzed carbonylation followed by cyclization (carbonylative cyclization) has been widely explored and used as a promising synthetic tool for the construction of the structural core of many pharmacologically and biologically active lactones and lactams.^[1–6] In connection with this report, it is known that β -bromo- α,β -unsaturated aldehydes and their derivatives, which are readily prepared from the corresponding ketones by Vilsmeier–Haack reaction^[7,8] and subsequent transformation, are used as a building block for the construction of versatile cyclic compounds.^[9–31] As part of our continuing studies directed towards palladium-catalyzed cyclization reactions using β -bromo- α,β -unsaturated aldehydes and their derivatives,^[32–35] we reported on the synthesis of several heterocycles via such a carbonylative cyclization.^[36–39] Among them, 2-bromocyclohex-1-enecarbaldehydes were found to be carbonylatively cyclized with anilines in the presence of a palladium catalyst under carbon monoxide pressure to give hydroisindol-1-ones (Scheme 1).^[38] However, unfortunately, the carbonylative cyclization did not take place at all with other cyclic β -bromovinyl aldehydes except for six-membered ones under the employed conditions. This led us to seek for a synthetic method for such heterocycles using other starting compounds under similar palladium-catalyzed carbonylative cyclization conditions. This report shows a palladium-catalyzed carbonylative cyclization of 1-bromoallyl bromides with anilines leading to 1-aryl-1*H*-pyrrol-2(5*H*)-ones.^[40]

Results and Discussion

The starting 1-bromoallyl bromides **4** were synthesized by initial conversion of the corresponding α -methylene containing ketones **1** into β -bromovinyl aldehydes **2** under bromination conditions of the Vilsmeier–Haack reaction (PBr₃/DMF/CHCl₃) (Scheme 2).^[7,8] Allyl alcohols **3** prepared by treating **2** with NaBH₄/MeOH were easily brominated to **4** under PBr₃ in CCl₄.^[26]

The results of several attempted carbonylative cyclizations of 1-bromo-2-(bromomethyl)cyclohex-1-ene (**4a**) with aniline (**5a**) are listed in Table 1. Treatment of equimolar amounts of **4a**

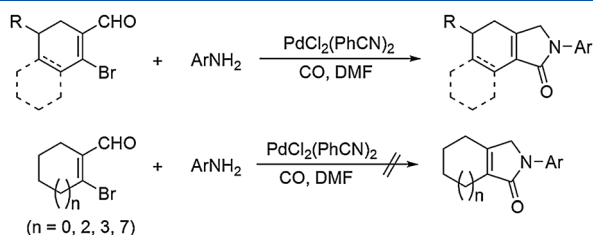
and **5a** in DMF in the presence of a catalytic amount of PdCl₂(PPh₃)₂ (4 mol%) along with K₂CO₃ under carbon monoxide pressure (10 atm) afforded 2-phenyl-2,3,4,5,6,7-hexahydro-1*H*-isindol-1-one (**6a**) in 48% isolated yield with concomitant formation of nucleophilic substitution product **7** (entry 1). Higher carbon monoxide pressure did not affect the product yield and distribution (entry 2). Performing the reaction under either higher reaction temperature for 40 h or changing the molar ratio of [5a]/[4a] gave no significant change to **6a** yield, whereas **7** was produced in a considerably increased yield (entries 3 and 4). Among bases examined, NaO^{*t*}Bu was not effective at all toward the formation of **6a** (entry 5). With other bases such as NaOAc and Bu₃N, the yield of **6a** was lower than that when K₂CO₃ was employed (entries 6 and 7).

Based on reaction conditions of Table 1, various 1-bromoallyl bromides **4** were subjected to the reaction with anilines **5** in order to investigate the reaction scope, and several representative results are summarized in Table 2. With cyclic 1-bromoallyl bromides (**4a–d**) having various ring sizes, the carbonylative cyclized products (**6a–h**) were formed in the range of 24–64% yield and the product yield was considerably affected by the ring size of **4a–d**. The product yield gradually decreases with increase in the ring size of **4a–d**. In the reaction with **4c** and **4d**, a higher reaction temperature was needed for the allowable yield of products. Judging from the reaction of **4b** with anilines **5a–e**, the position and electronic nature of the substituent on the aromatic ring of **5a–e** had no relevance to the product yield. To test for the effect of the position of bromide and bromomethyl group on 1-bromoallyl bromides, **4e** and **4f** were employed. The carbonylative cyclization readily took place with **4e**, whereas the reaction with **4f** did not proceed toward the desired carbonylative cyclization

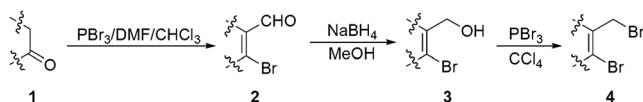
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Scheme 1. Palladium-catalyzed carbonylative cyclization leading to hydroisoindol-1-ones



Scheme 2. Synthesis of 1-bromoallyl bromides

under the same reaction conditions. Performing the reaction with **4f** under the conditions of 150 °C and 40 h, as is the case for the reaction with **4d**, afforded **6j** in only 25% yield. Similar reactivity was observed by our recent report: palladium-catalyzed carbonylative cyclization of 2-bromocyclohex-1-enecarbaldehydes with anilines leading to hydroisoindol-1-ones.^[38] Similar treatment of acyclic 1,3-dibromoprop-1-ene **4g** under the employed conditions also afforded the carbonylative cyclized product **6k**; however, the yield was lower than that when previously described cyclic 1,3-dibromoprop-1-enes were used except for **4d** and **4f**.

A plausible reaction pathway is presented in Scheme 3. Oxidative addition of the carbon–bromide bond of allyl amine **7**, initially formed *in situ* between **4a** and **5a**, to palladium(0), produces a vinylpalladium(II) complex **8**, where carbon monoxide coordination to palladium and then vinyl migration from palladium to the carbon of carbon monoxide occurs to give an acylpalladium(II) intermediate **9**. Intermediate **9** reacts with a base to produce a palladacycle intermediate **10** which can reductively eliminate to give **6a**. We confirmed in a separate experiment that treatment of **7** under the employed conditions afforded **6a** in 8% yield.

Conclusion

In summary, 1-bromoallyl bromides, which are readily prepared from ketones by three steps, are carbonylative cyclized with anilines under carbon monoxide pressure in the presence of a catalytic amount of $\text{PdCl}_2(\text{PPh}_3)_2$ along with K_2CO_3 to give 1-aryl-1H-pyrrol-2(5H)-ones. The present reaction provides a promising route for 1-aryl-1H-pyrrol-2(5H)-ones from readily available starting ketones. Further study of synthetic applications to heterocycles by using this ketone as starting compound is in progress.

Experimental

^1H and ^{13}C NMR (400 and 100 MHz) spectra were recorded on a Bruker Avance Digital 400 spectrometer using tetramethylsilane (TMS) as an internal standard. Melting points were determined on a Stanford Research Inc. MPA100 automated melting point apparatus. Gas–liquid chromatographic analyses were carried out with a Shimadzu GC-17A (FID) equipped with CBP10-S25-050 column (Shimadzu, silica fused capillary column, 0.33 mm \times 25 m, 0.25 μm film thickness) using N_2 as carrier gas. The isolation of pure products was carried out via thin-layer chromatography (silica gel 60 GF_{254} , Merck). 1-Bromoallyl bromides **4** were synthesized in three steps, initial treatment of ketones **1** with $\text{PBr}_3/\text{DMF}/\text{CHCl}_3$ ^[7,8] to produce β -bromovinyl aldehydes **2**, followed by reduction of **2** to allyl alcohols **3** using NaBH_4 and bromination using PBr_3 .^[20] Commercially available organic and inorganic compounds were used without further purification.

General Experimental Procedure for Palladium-Catalyzed Carbonylative Cyclization of 1-Bromoallyl Bromides with Anilines

To a 50 ml stainless steel autoclave were added 1-bromoallyl bromide **4** (0.5 mmol), aniline **5** (0.5 mmol), $\text{PdCl}_2(\text{PPh}_3)_2$ (0.014 g, 0.02 mmol), K_2CO_3 (0.138 g, 1 mmol) and DMF (10 ml). After the system was flushed and then pressurized with carbon monoxide, the reaction mixture was allowed to react under appropriate

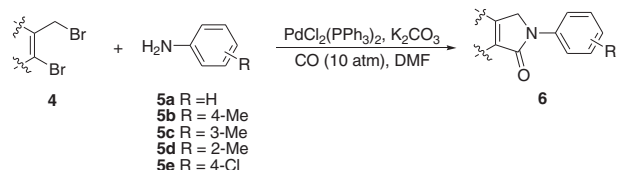

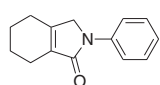

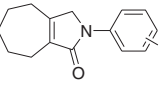

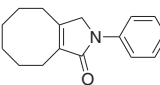

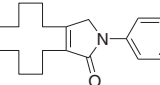
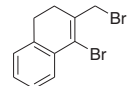
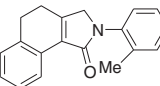
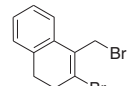
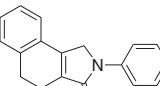
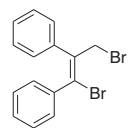
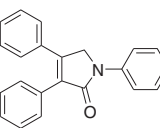
Table 1. Palladium-catalyzed carbonylative cyclization of **4a** with **5a**^a

Entry	Base	Temp. (°C)	Pressure (atm)	Time (h)	Yield (%)	
					6a	7
1	K_2CO_3	100	10	20	48	25
2	K_2CO_3	100	20	20	49	31
3	K_2CO_3	120	10	40	55	44
4 ^b	K_2CO_3	130	10	20	48	47
5	NaO^tBu	100	10	20	1	45
6	NaOAc	100	10	20	41	32
7	Bu_3N	100	10	20	38	29

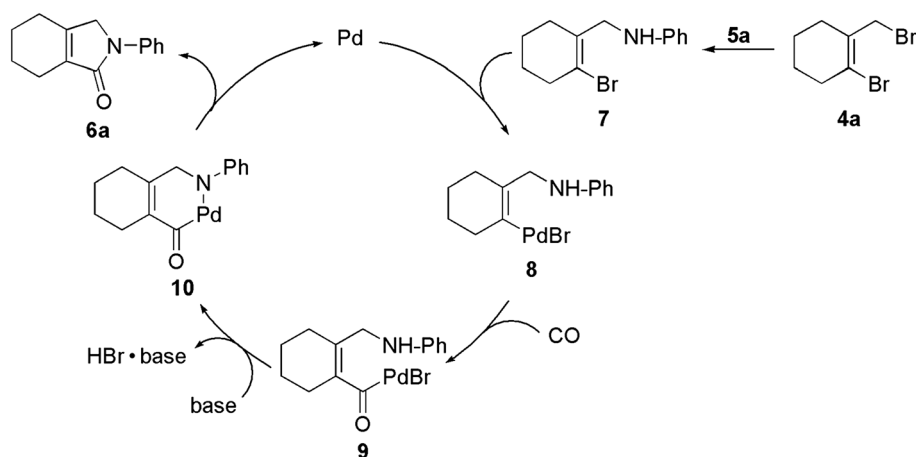
^aReaction conditions: **4a** (0.5 mmol), **5a** (0.5 mmol), $\text{PdCl}_2(\text{PPh}_3)_2$ (0.02 mmol), base (1 mmol), DMF (10 ml).

^b**5a** (1 mmol).

Table 2. Palladium-catalyzed carbonylative cyclization of 1,3-dibromopropenes **4** with anilines **5** leading to 1-aryl-1*H*-pyrrol-2(5*H*)-ones **6**^a

				
1,3-Dibromopropenes 4	Anilines 2	Conditions	1-Aryl-1 <i>H</i> -pyrrol-2(5 <i>H</i>)-ones 6	Yield (%)
	5a	100 °C, 20 h 120 °C, 40 h		48 55
	5a	100 °C, 20 h 100 °C, 40 h		51 64
	5b	100 °C, 20 h	6c	52
	5c	100 °C, 20 h	6d	44
	5d	100 °C, 20 h	6e	50
	5e	100 °C, 40 h	6f	45
	5a	100 °C, 20 h 150 °C, 40 h		25 56
	5a	100 °C, 20 h 150 °C, 20 h		0 24
	5d	100 °C, 20 h		71
	5a	100 °C, 20 h 150 °C, 40 h		0 25
	5a	100 °C, 20 h		30

^aReaction conditions: **4** (0.5 mmol), **5** (0.5 mmol), PdCl₂(PPh₃)₂ (0.02 mmol), K₂CO₃ (1 mmol), DMF (10 ml), CO (10 atm).

**Scheme 3.** A catalytic cycle

reaction temperature and time. The reaction mixture was filtered through a short silica gel column (ethyl acetate–hexane mixture) to eliminate inorganic salts. Removal of the solvent left a crude mixture, which was separated by thin-layer chromatography (silica gel, ethyl acetate–hexane mixture) to give 1-aryl-1H-pyrrol-2(5H)-ones **6**. Except for **6a** and **6i**, which were characterized by gas–liquid chromatography and spectroscopic comparison with authentic samples synthesized by our recent report,^[38] all products prepared by the above procedure were characterized spectroscopically as shown below.

2-Phenyl-2,3,5,6,7,8-hexahydrocyclohepta[c]pyrrol-1(4H)-one (6b)

Solid; m.p. 126–127°C (from hexane–ethyl acetate). ¹H NMR (CDCl₃) δ 1.62–1.74 (m, 4H, 2CH₂), 1.80–1.86 (m, 2H, CH₂), 2.45–2.48 (m, 4H, 2CH₂), 4.19 (s, 2H, CH₂-N), 7.07–7.11 (m, 1H, CH), 7.33–7.37 (m, 2H, 2CH), 7.72–7.74 (m, 2H, 2CH). ¹³C NMR (CDCl₃) δ 25.11 (CH₂), 27.33 (CH₂), 27.39 (CH₂), 29.92 (CH₂), 31.22 (CH₂), 54.05 (CH₂-N), 118.42 (aromatic C), 123.59 (aromatic C), 129.22 (aromatic C), 135.92 (vinyl C), 139.84 (aromatic C), 152.16 (vinyl C), 171.51 (C-O). Anal. Calcd for C₁₅H₁₇NO: C, 79.26; H, 7.54; N, 6.16. Found: C, 79.25; H, 7.47; N, 6.14.

2-(4-Methylphenyl)-2,3,5,6,7,8-hexahydrocyclohepta[c]pyrrol-1(4H)-one (6c)

Solid; m.p. 120–121°C (from hexane–CH₂Cl₂). ¹H NMR (CDCl₃) δ 1.61–1.73 (m, 4H, 2CH₂), 1.79–1.85 (m, 2H, CH₂), 2.32 (s, 3H, CH₃), 2.44–2.46 (m, 4H, 2CH₂), 4.15 (s, 2H, CH₂-N), 7.15 (d, *J*_{HH} = 8.5 Hz, 2H, 2CH), 7.59 (d, *J*_{HH} = 8.5 Hz, 2H, 2CH). ¹³C NMR (CDCl₃) δ 20.97 (CH₃), 25.11 (CH₂), 27.34 (CH₂), 27.40 (CH₂), 29.89 (CH₂), 31.21 (CH₂), 54.18 (CH₂-N), 118.57 (aromatic C), 129.70 (aromatic C), 133.14 (aromatic C), 135.86 (vinyl C), 137.35 (aromatic C), 151.89 (vinyl C), 171.36 (C-O). Anal. Calcd for C₁₆H₁₉NO: C, 79.63; H, 7.94; N, 5.80. Found: C, 79.54; H, 7.85; N, 5.73.

2-(3-Methylphenyl)-2,3,5,6,7,8-hexahydrocyclohepta[c]pyrrol-1(4H)-one (6d)

Solid; m.p. 81–83°C (from hexane–CH₂Cl₂). ¹H NMR (CDCl₃) δ 1.61–1.73 (m, 4H, 2CH₂), 1.78–1.85 (m, 2H, CH₂), 2.36 (s, 3H, CH₃), 2.45–2.47 (m, 4H, 2CH₂), 4.17 (s, 2H, CH₂-N), 6.91 (d, *J*_{HH} = 7.8 Hz, 1H, CH), 7.49 (d, *J*_{HH} = 7.8 Hz, 1H, CH), 7.59 (s, 1H, CH), 7.23 (t, *J*_{HH} = 7.8 Hz, 1H, CH). ¹³C NMR (CDCl₃) δ 21.89 (CH₃), 25.11 (CH₂), 27.33 (CH₂), 27.40 (CH₂), 29.91 (CH₂), 31.20 (CH₂), 54.20 (CH₂-N), 115.60 (aromatic C), 119.29 (aromatic C), 124.46 (aromatic C), 129.01 (aromatic C), 135.89 (vinyl C), 139.03 (aromatic C), 139.76 (aromatic C), 152.07 (vinyl C), 171.48 (C-O).

Anal. Calcd for C₁₅H₁₇NO: C, 79.63; H, 7.94; N, 5.80. Found: C, 79.56; H, 7.89; N, 5.75.

2-(2-Methylphenyl)-2,3,5,6,7,8-hexahydrocyclohepta[c]pyrrol-1(4H)-one (6e)

Solid; m.p. 113–114°C (from hexane–ethyl acetate). ¹H NMR (CDCl₃) δ 1.63–1.75 (m, 4H, 2CH₂), 1.81–1.87 (m, 2H, CH₂), 2.22 (s, 3H, CH₃), 2.44–2.49 (m, 4H, 2CH₂), 4.08 (s, 2H, CH₂-N), 7.12–7.15 (m, 1H, CH), 7.19–7.24 (m, 2H, 2CH), 7.26–7.28 (m, 1H, CH). ¹³C NMR (CDCl₃) δ 18.51 (CH₃), 25.41 (CH₂), 27.42 (CH₂), 27.46 (CH₂), 30.02 (CH₂), 31.31 (CH₂), 56.53 (CH₂-N), 126.79 (aromatic C), 127.55 (aromatic C), 127.78 (aromatic C), 131.26 (aromatic C), 134.82 (vinyl C), 136.49 (aromatic C), 137.56 (aromatic C), 153.21 (vinyl C), 177.77 (C-O). Anal. Calcd for C₁₅H₁₇NO: C, 79.63; H, 7.94; N, 5.80. Found: C, 79.50; H, 7.88; N, 5.70.

2-(4-Chlorophenyl)-2,3,5,6,7,8-hexahydrocyclohepta[c]pyrrol-1(4H)-one (6f)

Solid; m.p. 165–168°C (from hexane–ethyl acetate). ¹H NMR (CDCl₃) δ 1.61–1.74 (m, 4H, 2CH₂), 1.80–1.86 (m, 2H, CH₂), 2.45–2.48 (m, 4H, 2CH₂), 4.16 (s, 2H, CH₂-N), 7.27–7.32 (m, 2H, 2CH), 7.67–7.70 (m, 2H, 2CH). ¹³C NMR (CDCl₃) δ 25.08 (CH₂), 27.28 (CH₂), 27.34 (CH₂), 29.74 (CH₂), 31.17 (CH₂), 53.97 (CH₂-N), 119.44 (aromatic C), 128.51 (aromatic C), 129.19 (aromatic C), 135.91 (vinyl C), 138.43 (aromatic C), 152.34 (vinyl C), 171.47 (C-O). Anal. Calcd for C₁₅H₁₆ClNO: C, 68.83; H, 6.16; N, 5.35. Found: C, 68.67; H, 6.10; N, 5.32.

2-Phenyl-2,3,4,5,6,7,8,9-octahydro-1H-cycloocta[c]pyrrol-1-one (6g)

Solid; m.p. 110–111°C (from hexane–ethyl acetate). ¹H NMR (CDCl₃) δ 1.54–1.56 (m, 4H, 2CH₂), 1.67–1.73 (m, 2H, CH₂), 1.76–1.82 (m, 2H, CH₂), 2.50–2.56 (m, 4H, 2CH₂), 4.21 (s, 2H, CH₂-N), 7.07–7.11 (m, 1H, CH), 7.33–7.38 (m, 2H, 2CH), 7.74–7.77 (m, 2H, 2CH). ¹³C NMR (CDCl₃) δ 22.22 (CH₂), 25.83 (CH₂), 26.12 (CH₂), 26.90 (CH₂), 27.72 (CH₂), 27.75 (CH₂), 53.36 (CH₂-N), 118.23 (aromatic C), 123.54 (aromatic C), 129.23 (aromatic C), 134.13 (vinyl C), 139.91 (aromatic C), 150.78 (vinyl C), 171.35 (C-O). Anal. Calcd for C₁₆H₁₉NO: C, 79.63; H, 7.94; N, 5.80. Found: C, 79.59; H, 7.84; N, 5.62.

2-Phenyl-2,3,4,5,6,7,8,9,10,11,12,13-dodecahydro-1H-cyclododeca[c]pyrrol-1-one (6h)

Solid; m.p. 99–101°C (from hexane–CH₂Cl₂). ¹H NMR (CDCl₃) δ 1.35–1.46 (m, 12H, 6CH₂), 1.64–1.77 (m, 4H, 2CH₂), 2.87 (t, *J*_{HH} = 6.7 Hz, 2H, CH₂), 2.47 (t, *J*_{HH} = 7.3 Hz, 2H, CH₂), 4.20 (s, 2H, CH₂-N), 7.06–7.09 (m, 1H, CH), 7.33–7.37 (m, 2H, 2CH), 7.74–7.76 (m, 2H, 2CH). ¹³C NMR (CDCl₃) δ 21.25 (CH₂), 21.95 (CH₂), 22.86 (CH₂),

23.80 (CH₂), 24.62 (CH₂), 24.83 (CH₂), 25.22 (CH₂), 25.24 (CH₂), 25.63 (CH₂), 26.20 (CH₂), 52.67 (CH₂-N), 118.23 (aromatic C), 123.54 (aromatic C), 129.17 (aromatic C), 133.93 (vinyl C), 139.83 (aromatic C), 150.82 (vinyl C), 171.46 (C-O). Anal. Calcd for C₂₀H₂₇NO: C, 80.76; H, 9.15; N, 4.71. Found: C, 80.55; H, 9.09; N, 4.68.

2-Phenyl-4,5-dihydro-1H-benzo[e]isoindol-3(2H)-one (**6j**)

Solid; m.p. 130–131°C (from hexane–CH₂Cl₂). ¹H NMR (CDCl₃) δ 2.61–2.66 (m, 2H, CH₂), 3.00 (t, J_{HH} = 8.3 Hz, 2H, CH₂), 4.65 (t, J_{HH} = 2.3 Hz, 2H, CH₂-N), 7.11–7.15 (m, 1H, CH), 7.21–7.31 (m, 4H, 4CH), 7.38–7.42 (m, 2H, 2CH), 7.79–7.82 (m, 2H, 2CH). ¹³C NMR (CDCl₃) δ 18.65 (CH₂), 28.09 (CH₂), 50.06 (CH₂-N), 118.72 (aromatic C), 122.99 (aromatic C), 123.96 (vinyl C), 127.09 (aromatic C), 128.77 (aromatic C), 129.32 (aromatic C), 129.48 (aromatic C), 130.00 (aromatic C), 132.93 (aromatic C), 137.38 (aromatic C), 139.88 (aromatic C), 146.01 (vinyl C), 170.20 (C-O). Anal. Calcd for C₁₈H₁₅NO: C, 82.73; H, 5.79; N, 5.36. Found: C, 82.66; H, 5.70; N, 5.32.

1,3,4-Triphenyl-1H-pyrrol-2(5H)-one (**6k**)

Solid; m.p. 187–189°C (from hexane–ethyl acetate). ¹H NMR (CDCl₃) δ 4.75 (s, 2H, CH₂-N), 7.13–7.17 (m, 1H, CH), 7.29–7.46 (m, 12H, 12CH), 7.84–7.86 (m, 2H, 2CH). ¹³C NMR (CDCl₃) δ 52.82 (CH₂-N), 118.70 (aromatic C), 124.26 (vinyl C), 127.90 (aromatic C), 128.50 (aromatic C), 128.67 (aromatic C), 129.00 (aromatic C), 129.35 (aromatic C), 129.73 (aromatic C), 129.83 (aromatic C), 131.74 (aromatic C), 132.87 (aromatic C), 133.62 (aromatic C), 139.54 (aromatic C), 147.09 (vinyl C), 169.69 (C-O). Anal. Calcd for C₂₂H₁₇NO: C, 84.86; H, 5.50; N, 4.50. Found: C, 84.67; H, 5.47; N, 4.51.

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