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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

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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 hydroisoindol-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 (PBr_3/DMF/$CHCl_3$) (Scheme 2).[7,8] Allyl alcohols **3** prepared by treating **2** with $NaBH_4$/MeOH were easily brominated to **4** under PBr_3 in CCl_4.[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 $PdCl_2(PPh_3)_2$ (4 mol%) along with K_2CO_3 under carbon monoxide pressure (10 atm) afforded 2-phenyl-2,3,4,5,6,7-hexahydro-1*H*-isoindol-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 Bu_3N, the yield of **6a** was lower than that when K_2CO_3 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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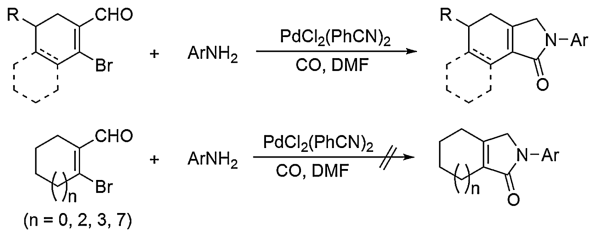
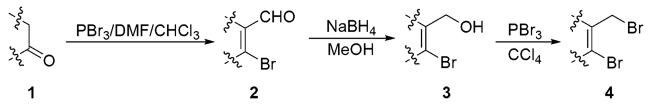


Scheme 1. Palladium-catalyzed carbonylative cyclization leading to hydroisoindol-1-ones



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 carbonylatively cyclized with anilines under carbon monoxide pressure in the presence of a catalytic amount of $PdCl_2(PPh_3)_2$ along with K_2CO_3 to give 1-aryl-1*H*-pyrrol-2(5*H*)-ones. The present reaction provides a promising route for 1-aryl-1*H*-pyrrol-2(5*H*)-ones from readily available starting ketones. Further study of synthetic applications to heterocycles by using this ketone as starting compound is in progress.

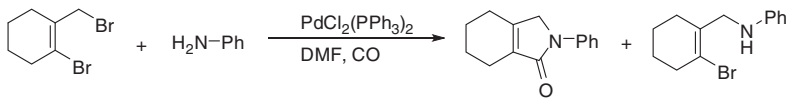
Experimental

^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 Standford 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 × 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 PBr_3/DMF/$CHCl_3$[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), $PdCl_2(PPh_3)_2$ (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]

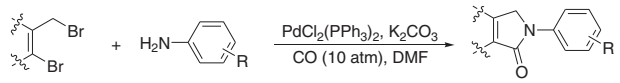


4a **5a** **6a** **7**

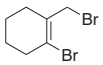
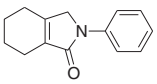
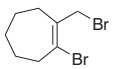
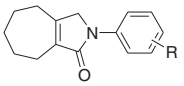
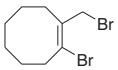
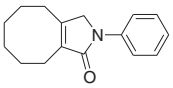
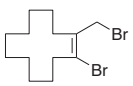
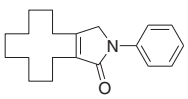
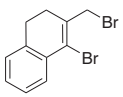
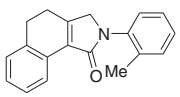
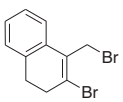
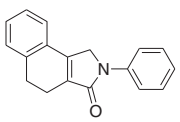
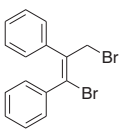
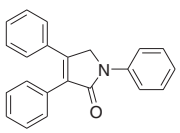
Entry	Base	Temp. (°C)	Pressure (atm)	Time (h)	Yield (%)	
					6a	**7**
1	K_2CO_3	100	10	20	48	25
2	K_2CO_3	100	20	20	49	31
3	K_2CO_3	120	10	40	55	44
4[b]	K_2CO_3	130	10	20	48	47
5	NaO^tBu	100	10	20	1	45
6	NaOAc	100	10	20	41	32
7	Bu_3N	100	10	20	38	29

[a]Reaction conditions: **4a** (0.5 mmol), **5a** (0.5 mmol), $PdCl_2(PPh_3)_2$ (0.02 mmol), base (1 mmol), DMF (10 ml).
[b]**5a** (1 mmol).

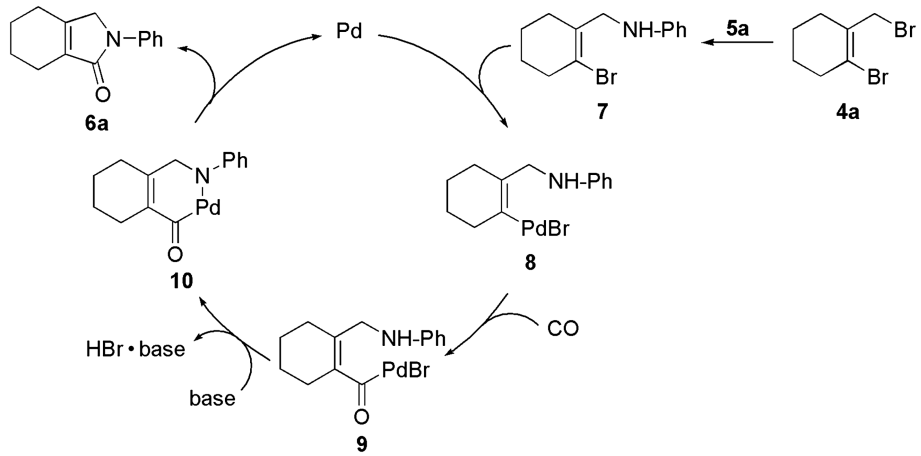
Table 2. Palladium-catalyzed carbonylative cyclization of 1,3-dibromopropenes **4** with anilines **5** leading to 1-aryl-1*H*-pyrrol-2(5*H*)-ones **6**[a]



4 **5a** R =H; **5b** R = 4-Me; **5c** R = 3-Me; **5d** R = 2-Me; **5e** R = 4-Cl; **6**

1,3-Dibromopropenes **4**	Anilines **2**	Conditions	1-Aryl-1H-pyrrol-2(5*H*)-ones **6**	Yield (%)
				
4a	**5a**	100 °C, 20 h	**6a**	48
		120 °C, 40 h		55
				
4b	**5a**	100 °C, 20 h	**6b**	51
		100 °C, 40 h		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
				
4c	**5a**	100 °C, 20 h	**6g**	25
		150 °C, 40 h		56
				
4d	**5a**	100 °C, 20 h	**6h**	0
		150 °C, 20 h		24
				
4e	**5d**	100 °C, 20 h	**6i**	71
				
4f	**5a**	100 °C, 20 h	**6j**	0
		150 °C, 40 h		25
				
4g	**5a**	100 °C, 20 h	**6k**	30

[a]Reaction conditions: 4 (0.5 mmol), 5 (0.5 mmol), $PdCl_2(PPh_3)_2$ (0.02 mmol), K_2CO_3 (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-1*H*-pyrrol-2(5*H*)-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). ^{1}H NMR ($CDCl_3$) δ 1.62–1.74 (m, 4H, 2CH_2), 1.80–1.86 (m, 2H, CH_2), 2.45–2.48 (m, 4H, 2CH_2), 4.19 (s, 2H, CH_2-N), 7.07–7.11 (m, 1H, C*H*), 7.33–7.37 (m, 2H, 2C*H*), 7.72–7.74 (m, 2H, 2C*H*). ^{13}C NMR ($CDCl_3$) δ 25.11 (CH_2), 27.33 (CH_2), 27.39 (CH_2), 29.92 (CH_2), 31.22 (CH_2), 54.05 (CH_2-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 $C_{15}H_{17}NO$: 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–CH_2Cl_2). ^{1}H NMR ($CDCl_3$) δ 1.61–1.73 (m, 4H, 2CH_2), 1.79–1.85 (m, 2H, CH_2), 2.32 (s, 3H, CH_3), 2.44–2.46 (m, 4H, 2CH_2), 4.15 (s, 2H, CH_2-N), 7.15 (d, J_{HH} = 8.5 Hz, 2H, 2C*H*), 7.59 (d, J_{HH} = 8.5 Hz, 2H, 2C*H*). ^{13}C NMR ($CDCl_3$) δ 20.97 (CH_3), 25.11 (CH_2), 27.34 (CH_2), 27.40 (CH_2), 29.89 (CH_2), 31.21 (CH_2), 54.18 (CH_2-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 $C_{16}H_{19}NO$: 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–CH_2Cl_2). ^{1}H NMR ($CDCl_3$) δ 1.61–1.73 (m, 4H, 2CH_2), 1.78–1.85 (m, 2H, CH_2), 2.36 (s, 3H, CH_3), 2.45–2.47 (m, 4H, 2CH_2), 4.17 (s, 2H, CH_2-N), 6.91 (d, J_{HH} = 7.8 Hz, 1H, C*H*), 7.49 (d, J_{HH} = 7.8 Hz, 1H, C*H*), 7.59 (s, 1H, C*H*), 7.23 (t, J_{HH} = 7.8 Hz, 1H, C*H*). ^{13}C NMR ($CDCl_3$) δ 21.89 (CH_3), 25.11 (CH_2), 27.33 (CH_2), 27.40 (CH_2), 29.91 (CH_2), 31.20 (CH_2), 54.20 (CH_2-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 $C_{15}H_{17}NO$: 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). ^{1}H NMR ($CDCl_3$) δ 1.63–1.75 (m, 4H, 2CH_2), 1.81–1.87 (m, 2H, CH_2), 2.22 (s, 3H, CH_3), 2.44–2.49 (m, 4H, 2CH_2), 4.08 (s, 2H, CH_2-N), 7.12–7.15 (m, 1H, C*H*), 7.19–7.24 (m, 2H, 2C*H*), 7.26–7.28 (m, 1H, C*H*). ^{13}C NMR ($CDCl_3$) δ 18.51 (CH_3), 25.41 (CH_2), 27.42 (CH_2), 27.46 (CH_2), 30.02 (CH_2), 31.31 (CH_2), 56.53 (CH_2-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 $C_{15}H_{17}NO$: 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). ^{1}H NMR ($CDCl_3$) δ 1.61–1.74 (m, 4H, 2CH_2), 1.80–1.86 (m, 2H, CH_2), 2.45–2.48 (m, 4H, 2CH_2), 4.16 (s, 2H, CH_2-N), 7.27–7.32 (m, 2H, 2C*H*), 7.67–7.70 (m, 2H, 2C*H*). ^{13}C NMR ($CDCl_3$) δ 25.08 (CH_2), 27.28 (CH_2), 27.34 (CH_2), 29.74 (CH_2), 31.17 (CH_2), 53.97 (CH_2-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 $C_{15}H_{16}ClNO$: 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). ^{1}H NMR ($CDCl_3$) δ 1.54–1.56 (m, 4H, 2CH_2), 1.67–1.73 (m, 2H, CH_2), 1.76–1.82 (m, 2H, CH_2), 2.50–2.56 (m, 4H, 2CH_2), 4.21 (s, 2H, CH_2-N), 7.07–7.11 (m, 1H, C*H*), 7.33–7.38 (m, 2H, 2C*H*), 7.74–7.77 (m, 2H, 2C*H*). ^{13}C NMR ($CDCl_3$) δ 22.22 (CH_2), 25.83 (CH_2), 26.12 (CH_2), 26.90 (CH_2), 27.72 (CH_2), 27.75 (CH_2), 53.36 (CH_2-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 $C_{16}H_{19}NO$: 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–CH_2Cl_2). ^{1}H NMR ($CDCl_3$) δ 1.35–1.46 (m, 12H, 6CH_2), 1.64–1.77 (m, 4H, 2CH_2), 2.87 (t, J_{HH} = 6.7 Hz, 2H, CH_2), 2.47 (t, J_{HH} = 7.3 Hz, 2H, CH_2), 4.20 (s, 2H, CH_2-N), 7.06–7.09 (m, 1H, C*H*), 7.33–7.37 (m, 2H, 2C*H*), 7.74–7.76 (m, 2H, 2C*H*). ^{13}C NMR ($CDCl_3$) δ 21.25 (CH_2), 21.95 (CH_2), 22.86 (CH_2),

23.80 (CH_2), 24.62 (CH_2), 24.83 (CH_2), 25.22 (CH_2), 25.24 (CH_2), 25.63 (CH_2), 26.20 (CH_2), 52.67 (CH_2-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 $C_{20}H_{27}NO$: 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–CH_2Cl_2). ^{1}H NMR ($CDCl_3$) δ 2.61–2.66 (m, 2H, CH_2), 3.00 (t, J_{HH} = 8.3 Hz, 2H, CH_2), 4.65 (t, J_{HH} = 2.3 Hz, 2H, CH_2-N), 7.11–7.15 (m, 1H, C*H*), 7.21–7.31 (m, 4H, 4C*H*), 7.38–7.42 (m, 2H, 2C*H*), 7.79–7.82 (m, 2H, 2C*H*). ^{13}C NMR ($CDCl_3$) δ 18.65 (CH_2), 28.09 (CH_2), 50.06 (CH_2-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 $C_{18}H_{15}NO$: 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). ^{1}H NMR ($CDCl_3$) δ 4.75 (s, 2H, CH_2-N), 7.13–7.17 (m, 1H, C*H*), 7.29–7.46 (m, 12H, 12C*H*), 7.84–7.86 (m, 2H, 2C*H*). ^{13}C NMR ($CDCl_3$) δ 52.82 (CH_2-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 $C_{22}H_{17}NO$: C, 84.86; H, 5.50; N, 4.50. Found: C, 84.67; H, 5.47; N, 4.51.

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