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1-Butyl-1-methylpyrrolidinium hydrogen sulfate-promoted preparation of 1,5-diaryl-3-(arylamino)-1*H*-pyrrol-2(5*H*)-one derivatives

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Abstract A convenient preparative approach for synthesis of various 1,5-diaryl-3-(arylamino)-1*H*-pyrrol-2(5*H*)-ones promoted by 1-butyl-1-methylpyrrolidinium hydrogen sulfate is developed which involves cyclocondensation of aldehydes with amines and ethyl pyruvate under ambient conditions.

Keywords 1,5-Diaryl-3-(arylamino)-1*H*-pyrrol-2(5*H*)-one · γ-Lactam · Ionic liquid · 1-Butyl-1-methylpyrrolidinium hydrogen sulfate

Introduction

Synthesis of pyrroles and their analogs has been an area of interest for chemists as well as biologists because these are versatile precursors for synthesis of pharmacologically significant molecules and natural products [1–6]. A useful class of pyrrole skeletons is 1,5-dihydro-2*H*-pyrrol-2-ones, which are an appealing family of lactams. These compounds and their 3-amino substituents (which also can act as enamine) are used increasingly as valuable intermediates in organic synthesis and offer major synthetic opportunities in synthesis of biological and pharmaceutical compounds [1–6]. Accordingly, many methods for assembly of 1,5-diaryl-3-(arylamino)-1*H*-pyrrol-2(5*H*)-ones have been developed which involve two-component condensation of amines with pyrrolidine-2,3-diones [7] and *β*,*γ*-unsaturated α-oxo esters [8] or tricomponent reaction of amines, aldehyde, and pyruvate using acidic catalysts such as H_2SO_4 [9], thiourea and phosphoric acid analogs [10], SiO_2–$FeCl_3$ [11], and triethylammonium hydrogen sulfate [12].

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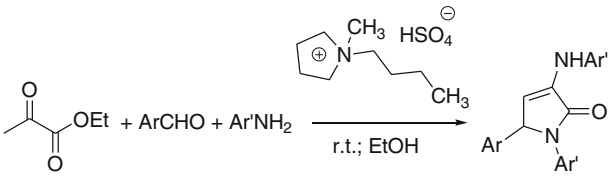


Scheme 1 Preparation of 1,5-diaryl-3-(arylamino)-1*H*-pyrrol-2(5*H*)-one derivatives

In recent years, use of ionic liquid media in organic chemistry has gained considerable attention because these reactions are attractive in terms of procedural simplicity and high product yield. This also contributes towards development of clean and green chemical processes and investigations of new and efficient catalysts [13–18]. Ionic liquids have shown advantages of high thermal stability, negligible vapor pressure, and recyclability [13–18]. As media, they have shown great promise as an attractive alternative to conventional solvents. In many cases, the products are weakly soluble in the ionic phase so that the products can be easily separated by simple filtration and washing. Therefore, much attention is currently being focused on organic reactions promoted by ionic liquids.

The overall aim of this project is to develop and validate an efficient protocol for synthesis and easy purification of 1,5-diaryl-3-(arylamino)-1*H*-pyrrol-2(5*H*)-ones from cyclocondensation reaction of aldehydes, aryl amines, and ethyl pyruvate in the presence of 1-butyl-1-methylpyrrolidinium hydrogen sulfate as an efficient acidic ionic liquid (for the preparation of 1-butyl-1-methylpyrrolidinium hydrogen sulfate, see [19–21]) (Scheme 1).

Results and discussion

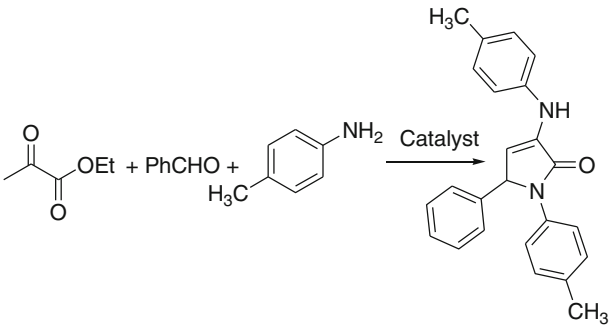
To study the effect of solvent, the reaction of benzaldehyde, 4-methylaniline, and ethyl pyruvate was examined in different solvents (Table 1, entries 1–7) and solvent-free condition (Table 1, entry 8). 1-Butyl-1-methylpyrrolidinium hydrogen sulfate was applied as effective promoter (Scheme 2). Among the various solvents tested, ethanol was chosen in terms of higher yield. Then the effect of the amount of promoter on this transformation was examined, and the results demonstrated that 1 mmol catalyst appeared to give the optimum yield (Table 1, entries 9–11). Thus, the best yield, cleanest reaction, and most facile workup were achieved by employing 1 mmol catalyst in ethanol under ambient condition (Table 1, entry 5).

To show the generality and scope of this protocol, we used various aldehydes and amines (Scheme 1; Table 2). Notably, a wide range of aldehydes (aromatic, heteroaromatic, and aliphatic) and aromatic amines were well permitted under the reaction conditions (Table 2, products **a**–**y**). In all cases, aromatic aldehydes containing electron-donating groups gave shorter times and higher yields than those with electron-withdrawing groups. Our results show that aliphatic aldehydes react with 4-methyl aniline and ethyl pyruvate successfully in good yield (Table 2, product

Table 1 Optimization of the reaction conditions in the synthesis of 3-(*p*-tolylamino)-5-phenyl-1-*p*-tolyl-1*H*-pyrrol-2(5*H*)-one (Scheme 2)

Entry	Catalyst (mmol)	*T* (°C)	Solvent (5 mL)	Yield (%)[a]
1	1	r.t.	*n*-Hexane	57
2	1	r.t.	CH_2Cl_2	67
3	1	r.t.	Et_2O	70
4	1	r.t.	EtOAc	75
5	1	r.t.	EtOH	89
6	1	r.t.	MeOH	80
8	1	r.t.	–	45
9	0.5	r.t.	EtOH	70
10	0.25	r.t.	EtOH	45
11	–	r.t.	EtOH	7

[a] Isolated yields, reaction time: 2 h



Scheme 2 Preparation of 3-(*p*-tolylamino)-5-phenyl-1-*p*-tolyl-1*H*-pyrrol-2(5*H*)-one

y). The reactions were clean, and all the products were easily isolated simply by filtration and washing with diethyl ether.

The reaction of cyclohexyl amine with benzaldehyde and ethyl pyruvate failed to give any product (Table 2, entry 23).

To explain the formation of 1,5-diaryl-3-(arylamino)-1*H*-pyrrol-2(5*H*)-ones via the one-pot multicomponent reaction, we have proposed a plausible reaction mechanism, which is illustrated in Scheme 3. Firstly, catalytic coupling of amine with aldehyde by Brønsted acid occurs to form intermediate **a**. In the same manner, ethyl pyruvate can react with amine to form intermediate **b**, which is equilibrated with their enamine form (**c**). Thereafter, the formation of intermediate **d** resulting from condensation of intermediate **a** with intermediate **c** was established. Finally, the imine–enamine conversion of intermediate **d** followed by loss of ethanol from enamine form results in synthesis of 1,5-diaryl-3-(arylamino)-1*H*-pyrrol-2(5*H*)-one derivatives (Scheme 3).

To estimate the efficiency and generality of this methodology, the result obtained for synthesis of 3-(*p*-tolylamino)-5-phenyl-1-*p*-tolyl-1*H*-pyrrol-2(5*H*)-one by this method was compared with those of previously reported methods. The results are

Table 2 Synthesis of 1,5-diaryl-3-(arylamino)-1*H*-pyrrol-2(5*H*)-one derivatives

Entry	Aldehyde	Amine	Product	Time (h)	Yield (%)[a]
1	Benzaldehyde	4-Methylaniline	**a**	2	89
2	Benzaldehyde	4-Methoxyaniline	**b**	2	82
3	Benzaldehyde	4-Chloroaniline	**c**	3	88
4	Benzaldehyde	3-Methylaniline	**d**	2	87
5	4-Chlorobenzaldehyde	4-Chloroaniline	**e**	4	79
6	4-Methoxybenzaldehyde	4-Chloroaniline	**f**	3	89
7	4-Methylbenzaldehyde	4-Chloroaniline	**g**	3	82
8	4-*tert*-Butylbenzaldehyde	4-Chloroaniline	**h**	3	75
9	3,4,5-Trimethoxybenzaldehyde	4-Chloroaniline	**i**	3	84
10	4-Chlorobenzaldehyde	4-Methoxyaniline	**j**	3	83
11	4-Nitrobenzaldehyde	4-Methoxyaniline	**k**	5	69
12	4-Methoxybenzaldehyde	4-Methoxyaniline	**l**	2	90
13	4-Methylbenzaldehyde	4-Methoxyaniline	**m**	2	91
14	4-Chlorobenzaldehyde	4-Methylaniline	**n**	3	77
15	4-Methoxybenzaldehyde	4-Methylaniline	**o**	2	93
16	4-Methylbenzaldehyde	4-Methylaniline	**p**	2	92
17	3,4,5-Trimethoxybenzaldehyde	4-Methylaniline	**q**	2	88
118	4-Methylbenzaldehyde	3-Methylaniline	**r**	2	70
19	3,4,5-Trimethoxybenzaldehyde	3-Methylaniline	**s**	2	89
20	Furfural	4-Methylaniline	**t**	4	74
21	Furfural	4-Methoxyaniline	**w**	4	72
22	Butyraldehyde	4-Methylaniline	**y**	2	70
23	Benzaldehyde	Cyclohexyl amine	**z**	2	–

[a] Isolated yields. All known products have been reported previously in the literature and were characterized by comparison of infrared (IR) and nuclear magnetic resonance (NMR) spectra with authentic samples [8–12]

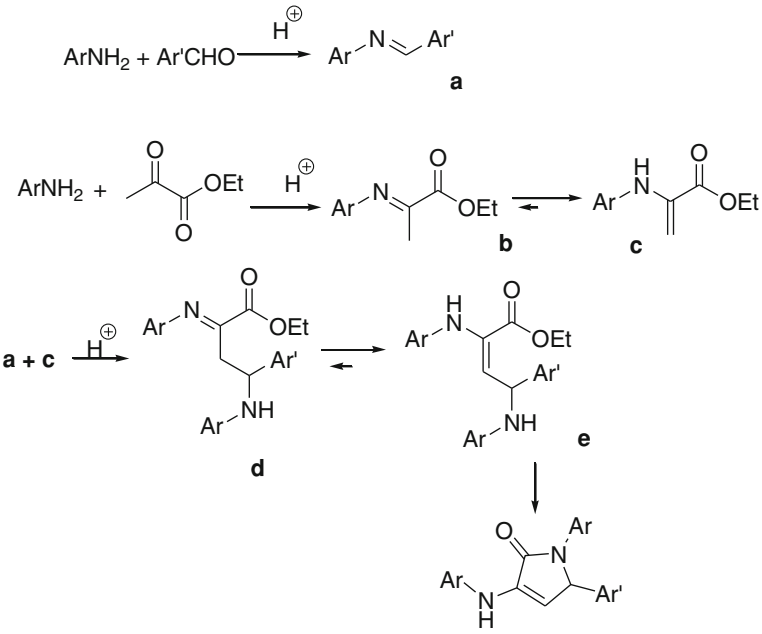
summarized in Table 3. It was found that the present method is convincingly superior to the reported methods in terms of reaction time and product yield.

Our protocol uses ionic liquids during the reaction process, making it superior to reactions that use hazardous liquid acidic catalysts.

In summary, simple one-pot three-component synthesis of 1,5-diaryl-3-(arylamino)-1*H*-pyrrol-2(5*H*)-one derivatives has been developed by coupling of aldehydes, amines, and ethyl pyruvate in ethanol promoted by 1-butyl-1-methylpyrrolidinium hydrogen sulfate. This protocol is endowed with several advantages such as improved yield, clean reaction, and simple purification.

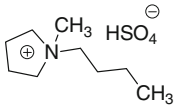
Experimental

All reagents were purchased from Merck and Aldrich and used without further purification. All yields refer to isolated products after purification. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Avance DPX 400 MHz



Scheme 3 Proposed mechanism for preparation of 1,5-diaryl-3-(arylamino)-1*H*-pyrrol-2(5*H*)-ones

Table 3 Comparison results of 1-butyl-1-methylpyrrolidinium hydrogen sulfate with other catalysts reported in the literature

Entry	Catalyst	Condition	Yield (%)[a]	Reference
1	Et_3NHHSO_4	*n*-Hexane; r.t.; 120 min	74	[12]
2	Thiourea	Toluene; r.t.; 18 h	84	[10]
3	Phosphoric acid	Toluene; r.t.; 18 h	84	[10]
4	SiO_2–$FeCl_3$	Solvent-free; r.t. (40 min) to 100 °C (20 min)	70	[11]
5	$Al(H_2PO_4)_3$	EtOH; r.t.; 120 min	25	This work
6	$Mg(HSO_4)_2$	EtOH; r.t.; 120 min	35	This work
7	SiO_2–H_2SO_4	EtOH; r.t.; 120 min	27	This work
8		EtOH; r.t.; 120 min	89	This work

[a] Isolated yield; based on preparation of 3-(*p*-tolylamino)-5-phenyl-1-*p*-tolyl-1*H*-pyrrol-2(5*H*)-one

instrument. Spectra were measured in $CDCl_3$ relative to tetramethylsilane (TMS, 0.00 ppm). Infrared (IR) spectra were recorded on a PerkinElmer 781 spectrophotometer. Elemental analysis was performed on a Heraeus CHN-O-Rapid analyzer. Melting points were determined in open capillaries with a BUCHI 510 melting point

apparatus. Thin-layer chromatography (TLC) was performed on silica gel polygram SIL G/UV 254 plates.

General procedure

To a mixture of aldehyde (1 mmol), aromatic amine (2 mmol), and ethyl pyruvate (1.5 mmol) in ethanol (5 mL), 1-butyl-1-methylpyrrolidinium hydrogen sulfate (1 mmol) as catalyst was added, and the mixture was stirred for appropriate time at ambient condition. Progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was displaced to a filter paper and washed with diethyl ether to afford the pure product (Table 2, products **a**–**r**).

Selected data

*3-(m-Tolylamino)-5-phenyl-1-m-tolyl-1H-pyrrol-2(5H)-one (product **d**)*

^{1}H NMR (400 MHz, $CDCl_3$): 2.29 (s, 3H), 2.36 (s, 3H), 5.69 (d, $J = 2.3$ Hz, 1H), 6.08 (d, $J = 2.3$ Hz, 1H), 6.31 (s, 1H), 6.51 (d, $J = 8.6$ Hz, 1H), 6.69 (s, 1H), 6.93 (d, $J = 8.6$ Hz, 1H), 7.01 (t, $J = 8.3$ Hz, 1H), 7.14–7.28 (m, 7H), 7.53-7.59 (m, 2H) ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 20.8$, 21.2, 64.5, 105.3, 108.2, 112.8, 116.3, 121.4, 124.9, 126.9, 127.3, 127.7, 128.7, 129.3, 129.7, 138.7, 139.0, 139.2, 139.5, 143.4, 144.6, 166.6 ppm; IR (KBr): 3316, 3014, 2954, 1674, 1589, 1164, 887, 751, and 728 cm^{-1}; Found: C, 81.45; H, 6.38; N, 7.99 $C_{24}H_{22}N_2O$; requires: C, 81.33; H, 6.26; N, 7.90 %.

*3-(4-Chlorophenylamino)-1-(4-chlorophenyl)-5-(4-methoxyphenyl)-1H-pyrrol-2(5H)-one (product **f**)*

^{1}H NMR (400 MHz, $CDCl_3$): 3.79 (s, 3H), 5.63 (d, $J = 2.5$ Hz, 1H), 6.06 (d, $J = 2.5$ Hz, 1H), 6.69 (s, 1H), 6.84 (d, $J = 8.6$ Hz, 2H), 7.02 (d, $J = 8.6$ Hz, 2H), 7.25–7.31 (m, 6H), 7.54 (d, $J = 8.7$ Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 55.4$, 63.9, 108.4, 114.9, 117.0, 121.7, 126.1, 128.1, 129.0, 129.2, 131.5, 134.6, 136.8, 140.0, 151.6, 158.5, 167.5 ppm; IR (KBr): 3319, 1673, 1648, 1597, 1524, 1490, 1169, 1014, 824, and 775 cm^{-1}; Found: C, 65.07; H, 4.38; N, 6.67 $C_{23}H_{18}Cl_2N_2O_2$; requires: C, 64.95; H, 4.27; N, 6.59 %.

*3-(4-Chlorophenylamino)-1-(4-chlorophenyl)-5-p-tolyl-1H-pyrrol-2(5H)-one (product **g**)*

^{1}H NMR (400 MHz, $CDCl_3$): 2.31 (s, 3H), 5.63 (d, $J = 2.5$ Hz, 1H), 6.04 (d, $J = 2.5$ Hz, 1H), 6.69 (s, 1H), 7.01 (d, $J = 8.8$ Hz, 2H), 7.12 (d, $J = 8.4$ Hz, 2H), 7.25–7.26 (m, 4H), 7.32 (d, $J = 8.0$ Hz, 2H), 7.54 (d, $J = 8.8$ Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 21.4$, 63.5, 108.7, 116.7, 119.6, 122.5, 125.8, 126.5, 129.1, 129.4, 130.0, 131.5, 133.8, 136.7, 139.9, 151.7, 167.4 ppm; IR (KBr): 3319, 1674, 1653, 1597, 1524, 1490, 1169, 1092, 817, and 775 cm^{-1}; Found: C, 67.62; H, 4.55; N, 6.95 $C_{23}H_{18}Cl_2N_2O$; requires: C, 67.49; H, 4.43; N, 6.84 %.

*3-(4-Chlorophenylamino)-5-(4-tert-butylphenyl)-1-(4-chlorophenyl)-1H-pyrrol-2(5H)-one (product **h**)*

m.p.: 235–237 °C, ^{1}H NMR (400 MHz, $CDCl_3$): 1.30 (s, 9H), 5.65 (d, $J = 2.4$ Hz, 1H), 6.06 (d, $J = 2.4$ Hz, 1H), 6.70 (s, 1H), 7.01 (d, $J = 8.8$ Hz, 2H), 7.13 (d, $J = 8.4$ Hz, 2H), 7.25–7.27 (m, 4H), 7.33 (d, $J = 8.0$ Hz, 2H), 7.56 (d, $J = 8.8$ Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 31.3$, 34.6, 63.8, 108.9, 117.9, 122.4, 126.1, 126.2, 126.3, 129.0, 129.3, 130.1, 131.6, 133.6, 135.9, 139.8, 151.5, and 167.1 ppm; IR (KBr): 3320, 3065, 2965, 2927, 2867, 1670, 1650, 1599, 1541, 1495, 1419, 1393, 1318, 1297, 1268, 1164, 1092, 1015, 919, 870, 833, 819, 796, and 778 cm^{-1}; Found: C, 69.25; H, 5.41; N, 6.27 $C_{26}H_{24}Cl_2N_2O$; requires: C, 69.18; H, 5.36; N, 6.21 %.

*3-(4-Chlorophenylamino)-1-(4-chlorophenyl)-5-(3,4,5-trimethoxyphenyl)-1H-pyrrol-2(5H)-one (product **i**)*

^{1}H NMR (400 MHz, $CDCl_3$): 3.77 (s, 6H), 3.79 (s, 3H), 5.66 (d, $J = 2.5$ Hz, 1H), 6.09 (d, $J = 2.5$ Hz, 1H), 6.37 (s, 2H), 6.70 (s, 1H), 7.26-7.32 (m, 6H), 7.55 (d, $J = 8.6$ Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 55.4$, 56.2, 64.2, 104.0, 108.5, 115.2, 117.1, 121.5, 126.3, 128.2, 129.0, 129.5, 131.7, 140.0, 151.6, 154.4, 157.2, and 167.6 ppm; IR (KBr): 3321, 1677, 1645, 1596, 1524, 1168, 1014, 824, and 776 cm^{-1}; Found: C, 61.99; H, 4.67; N, 5.89 $C_{25}H_{22}Cl_2N_2O_4$; requires: C, 61.87; H, 4.57; N, 5.77 %.

*3-(p-Tolylamino)-5-(3,4,5-trimethoxyphenyl)-1-p-tolyl-1H-pyrrol-2(5H)-one (product **q**)*

^{1}H NMR (400 MHz, $CDCl_3$): 2.23 (s, 3H), 2.27 (s, 3H), 3.76 (s, 6H), 3.79 (s, 3H), 5.58 (d, $J = 2.4$ Hz, 1H), 6.05 (d, $J = 2.4$ Hz, 1H), 6.36 (s, 2H), 6.66 (s, 1H), 7.02 (d, $J = 8.5$ Hz, 2H), 7.18 (d, $J = 8.1$ Hz, 2H), 7.23–7.28 (m, 4H) ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 21.1$, 21.4, 55.4, 55.5, 65.1, 103.9, 106.6, 116.9, 126.0, 129.7, 129.9, 131.0, 133.4, 133.8, 136.9, 137.8, 138.5, 154.4, 157.2, and 168.4 ppm; IR (KBr): 3319, 3021, 2940, 1675, 1650, 1610, 1543, 1454, 1322, 1185, 1123, 831, 774, and 740 cm^{-1}; Found: C, 73.09; H, 6.46; N, 6.39 $C_{27}H_{28}N_2O_4$; requires: C, 72.95; H, 6.35; N, 6.30 %.

*3-(m-Tolylamino)-1-m-tolyl-5-p-tolyl-1H-pyrrol-2(5H)-one (product **r**)*

^{1}H NMR (400 MHz, $CDCl_3$): 2.23 (s, 3H), 2.29 (s, 3H), 2.35 (s, 3H), 5.67 (d, $J = 2.4$ Hz, 1H), 6.09 (d, $J = 2.4$ Hz, 1H), 6.30 (s, 1H), 6.49 (d, $J = 8.2$ Hz, 1H), 6.68 (s, 1H), 6.91 (d, $J = 8.2$ Hz, 1H), 7.02 (t, $J = 8.4$ Hz, 1H), 7.10–7.28 (m, 6H), 7.51–7.57 (m, 2H) ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 20.9$, 21.4, 21.6, 63.7, 105.7, 108.5, 113.0, 116.1, 118.9, 119.2, 121.5, 126.8, 127.1, 129.0, 129.6, 136.5, 138.9, 139.2, 139.4, 140.7, 144.4, 144.7, and 167.5 ppm; IR (KBr): 3318, 3021, 2849, 1671, 1602, 1563, 1171, 1052, and 749 cm^{-1}; Found: C, 81.58; H, 6.71; N, 7.71 $C_{25}H_{24}N_2O$; requires: C, 81.49; H, 6.57; N, 7.60 %.

*3-(m-Tolylamino)-5-(3,4,5-trimethoxyphenyl)-1-m-tolyl-1H-pyrrol-2(5H)-one (product **s**)*

^{1}H NMR (400 MHz, $CDCl_3$): 2.31 (s, 3H), 2.39 (s, 3H), 3.75 (s, 6H), 3.77 (s, 3H), 5.51 (d, $J = 2.3$ Hz, 1H), 6.02 (d, $J = 2.3$ Hz, 1H), 6.35 (s, 2H), 6.42 (s, 1H), 6.50 (d, $J = 8.3$ Hz, 1H), 6.69 (s, 1H), 6.93 (d, $J = 8.3$ Hz, 1H), 7.05 (t, $J = 8.5$ Hz, 1H), 7.12–7.26 (m, 2H), 7.50 (d, $J = 8.4$ Hz, 1H), 7.55 (s, 1H) ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 21.1$, 21.4, 55.4, 55.5, 65.1, 103.9, 106.0, 108.6, 112.9, 116.2, 119.0, 121.5, 126.8, 127.1, 128.8, 129.0, 129.6, 138.8, 139.2, 140.3, 144.4, 154.4, 157.2, and 167.4 ppm; IR (KBr): 3315, 3019, 2941, 1677, 1649, 1612, 1541, 1455, 1322, 1186, 1125, 832, 775, and 742 cm^{-1}; Found: C, 73.07; H, 6.44; N, 6.38 $C_{27}H_{28}N_2O_4$; requires: C, 72.95; H, 6.35; N, 6.30 %.

*3-(p-Tolylamino)-5-(furan-2-yl)-1-p-tolyl-1H-pyrrol-2(5H)-one (product **t**)*

^{1}H NMR (400 MHz, $CDCl_3$): 2.35 (s, 6H), 5.78 (d, $J = 2.4$ Hz, 1H), 6.04 (d, $J = 2.4$ Hz, 1H), 6.21 (d, $J = 2.8$ Hz, 1H), 6.28 (s, 1H), 6.65 (s, 1H), 7.04 (d, $J = 8.0$ Hz, 2H), 7.15–7.19 (m, 4H), 7.33 (s, 1H), 7.37 (d, $J = 8.4$ Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 20.7$, 21.0, 58.1, 102.9, 108.4, 110.5, 116.9, 122.6, 129.6, 129.9, 130.8, 133.5, 134.4, 135.3, 138.8, 142.6, 150.1, and 166.7 ppm; IR (KBr): 3314, 3032, 2920, 2856, 1679, 1650, 1616, 1590, 1572, 1543, 1515, 1461, 1422, 1401, 1323, 1304, 1276, 1238, 1212, 1165, 1150, 1101, 1071, 929, 883, 867, 849, 821, 800, and 777 cm^{-1}; Found: C, 76.82; H, 5.93; N, 8.17 $C_{22}H_{20}N_2O_2$; requires: C, 76.72; H, 5.85; N, 8.13 %.

*3-(4-Methoxyphenylamino)-5-(furan-2-yl)-1-(4-methoxyphenyl)-1H-pyrrol-2(5H)-one (product **w**)*

^{1}H NMR (400 MHz, $CDCl_3$): 3.73 (s, 3H), 3.77 (s, 3H), 5.76 (d, $J = 2.6$ Hz, 1H), 6.05 (d, $J = 2.6$ Hz, 1H), 6.21 (d, $J = 3.0$ Hz, 1H), 6.28 (d, $J = 3.0$ Hz, 1H), 6.65 (s, 1H), 6.89 (d, $J = 8.4$ Hz, 2H), 7.03 (d, $J = 8.4$ Hz, 2H), 7.11 (d, $J = 8.6$ Hz, 2H), 7.28–7.34 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 55.3$, 55.8, 60.1, 103.1, 105.9, 110.6, 114.8, 118.5, 124.6, 129.6, 129.9, 133.6, 135.3, 142.6, 150.2, 154.9, 157.2, and 166.9 ppm; IR (KBr): 3321, 3031, 2925, 1675, 1651, 1614, 1591, 1541, 1462, 1275, 1234, 1210, 1163, 1103, 1070, 881, 866, 823, and 776 cm^{-1}; Found: C, 70.31; H, 5.47; N, 7.53 $C_{22}H_{20}N_2O_4$; requires: C, 70.20; H, 5.36; N, 7.44 %.

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