Chemistry of Heterocyclic Compounds, Vol. 43, No. 7, 2007

SYNTHESIS OF 1-ALKYL-3-ARYLAMINO-PYRROLE-2,5-DIONES

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Reaction of 1-alkyl-3-alkylaminopyrrole-2,5-diones with primary arylamine hydrochlorides in methanol or DMSO gives 1-alkyl-3-arylaminopyrrole-2,5-diones which could also be obtained from the reaction of arylaminofumarates with primary aliphatic amines.

Keywords: arylaminofumarates, maleimide, pyrrole-2,5-dione, transamination.

3-Arylaminopyrrole-2,5-dione derivatives show fungistatic [1], herbicidal, insecticidal [2], and antitumor activity [3] and are used for the treatment and/or prophylaxis of cancer as well as conditions needing inhibition of the enzyme GSK-3 (diabetes, Alzheimer disease, and acute stroke) [4].

The synthesis of 3-arylaminopyrrole-2,5-diones and their 1-alkyl derivatives is based on the cyclization of arylaminofumarates with ammonia [5], treatment of maleimides with arylhydroxylamines [6], or on the reaction of 3-halo- [4, 5, 7, 8] or 3-hydroxy- [4, 8] pyrrole-2,5-diones with arylamines. It is also known that enamines are transaminated by amines under conditions of acid or base catalysis: 1,4-bis(dimethylamino)-1,3-butadiene by aryl and alkylamines in the presence of catalytic amounts of acetic and/or hydrochloric acid [9] and 1-nitro-2-dimethylaminopropylene by ammonia and alkylamines (without acid) or arylamines in the presence of an equimolar amount of *p*-toluenesulfonic acid [10].

We have studied the reaction of 1-alkyl-3-alkylaminopyrrole-2,5-diones with arylamines with the aim of preparing 1-alkyl-3-arylaminopyrrole-2,5-diones.

It was found that 1-methyl-3-methylaminopyrrole-2,5-dione **1** does not react with aniline at a reagent ratio from 1: 1 to 1: 3 under various conditions (refluxing in methanol, heating at 100-120ºC in DMSO, or at 100ºC without solvent over 5-40 h in the absence or presence of a catalytic or an equimolar amount of acetic acid. By contrast maleimide **1** reacts rapidly with an equimolar amount of the arylamine hydrochlorides **2a-m** by heating for 1 h in methanol or DMSO to give good yields of the corresponding 3-arylamino-1-methylpyrrole-2,5-diones **3a-m** (Tables 1, 2). In a similar reaction 1-(2-hydroxyethyl)-3-(2-hydroxyethylamino)pyrrole-2,5-dione reacts with *p*-anisidine hydrochloride to give 1-(2-hydroxyethyl)-3-(*p*-methoxyphenylamino)pyrrole-2,5-dione (**3n)** (Tables 1, 2). The reaction of maleimide **1** with an equimolar amount of aniline in the presence of 15 mole % of aniline hydrochloride (refluxing in methanol for 1 h) gave a low yield (5%) of the 1-methyl-3-phenylaminopyrrole-2,5-dione **3a**.

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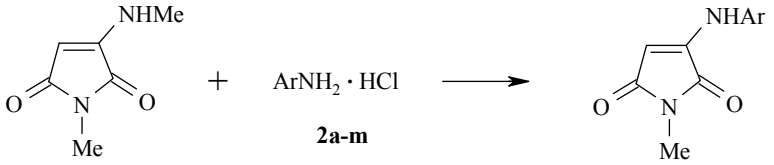
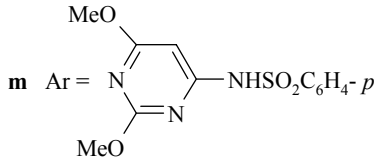
 0009-3122/07/4307-0844

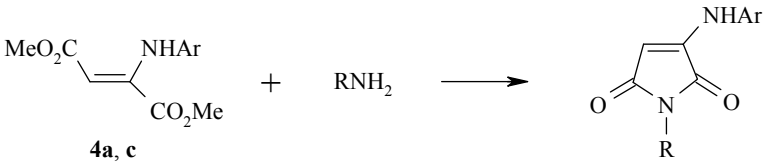
According to the ^{1}H NMR spectra of the reaction products the maleimide **1** does not react with benzylamine (molar ratio of reagents 1: 1 or 1: 2, 90% aqueous methanol, 18 days at 20ºC or 1: 2, 90% aqueous methanol, 10 h at 20ºC with subsequent refluxing for 8 h) and the 1-benzyl-3-benzylaminopyrrole-2,5-dione is inert towards methylamine (molar ratio of reagents 1: 3, 90% aqueous methanol, 10 h at 20ºC and 8 h at 95ºC in a sealed ampul). At the same time, refluxing maleimide **1** in a 5 fold excess of morpholine gave a 1: 1 mixture of maleimide **1** and 1-methyl-3-morpholinopyrrole-2,5-dione (**3o)** (Tables 1, 2) (from the corresponding CH signals at 4.73 and 5.13 ppm in the 300 MHz ^{1}H NMR spectrum). Treatment of maleimide **1** with an equimolar amount of benzylamine hydrochloride gave a satisfactory yield of 3-benzylamino-1-methylpyrrole-2,5-dione **3p** (Tables 1, 2). By contrast 1-methyl-3-phenylaminopyrrole-2,5-dione does not react with an equimolar amount of benzylamine or its hydrochloride (refluxing in methanol for 1 h).



1 **3a-m**

2,**3 a** Ar = Ph, **b** Ar = *p*-BrC_6H_4, **c** Ar = *p*-MeC_6H_4, **d** Ar = *m*-MeC_6H_4, **e** Ar = *o*-$MeOC_6H_4$,
f Ar = *p*-HOC_6H_4, **g** Ar = *p*-$O_2NC_6H_4$, **h** Ar = *m*-$O_2NC_6H_4$, **i** Ar = *m*-$HOCH_2CH_2OC_6H_4$,
j Ar = *p*-$Me_2NC_6H_4$, **k** Ar = *p*-$Me_2NC_6H_4$·HCl, **l** Ar = *p*-$H_2NSO_2C_6H_4$,





4a, **c** **3a**, **5a–c**

5 a Ar = Ph, R = $PhCH_2$; **b** Ar = p-MeC_6H_4, R = $PhCH_2$,
c Ar = *p*-MeC_6H_4, R = CH_2CH_2OH

It is known that the reaction of anilinofumarate **4a** with ammonia gives 2-anilinomaneimide [5] whereas treatment of aminofumarate with primary aliphatic amines gives principally the 1-alkyl-3-alkylaminopyrrole-2,5-dione [11] and not the likely 1-alkyl-3-amino derivative. We found that reaction of arylaminofumarates with primary aliphatic amines gives exclusively the 1-alkyl-3-arylaminopyrrole-2,5-diones **3a, 5a-c** (Tables 1, 2), i.e. in this reaction an exchange of arylamino group for alkylamine does not occur.

The ^{1}H NMR spectra of compounds **3a-n, 5a-c** show singlet signals for the vinyl proton (5.35-6.06) and NH group (8.24-10.21 ppm). The IR spectra show stretching bands for the N–H bond (3365-3310) and C=C

TABLE 1. Characteristics of Compounds **3a-p, 5a-c**

Compound	Empirical formula	Found, % / Calculated, %			mp, °C	Yield, %
		C	H	N		
3a	$C_{11}H_{10}N_2O_2$	65.28 / 65.34	4.95 / 4.98	13.84 / 13.85	199-200	70
3b	$C_{11}H_9BrN_2O_2$	47.11 / 47.00	3.25 / 3.23	10.02 / 9.97	237-238 (dec.)	70
3c	$C_{12}H_{12}N_2O_2$	66.60 / 66.65	5.58 / 5.59	12.89 / 12.96	208-210	67
3d	$C_{12}H_{12}N_2O_2$	66.54 / 66.65	5.52 / 5.59	12.90 / 12.96	206-208	56
3e	$C_{12}H_{12}N_2O_3$	62.12 / 62.06	5.25 / 5.21	12.06 / 12.06	162-164	50
3f	$C_{11}H_{10}N_2O_3$	60.57 / 60.55	4.63 / 4.62	12.91 / 12.84	251-254 (dec.)	57
3g	$C_{11}H_9N_3O_4$	53.39 / 53.44	3.69 / 3.67	17.12 / 17.00	289-291 (dec.)	51
3h	$C_{11}H_9N_3O_4$	53.52 / 53.44	3.70 / 3.67	17.05 / 17.00	286-287 (dec.)	45
3i	$C_{13}H_{14}N_2O_4$	59.55 / 59.54	5.43 / 5.38	10.74 / 10.68	165-167	45
3j	$C_{13}H_{15}N_3O_2$	63.80 / 63.66	6.22 / 6.16	17.00 / 17.13	207-209	82
3k	$C_{13}H_{16}ClN_3O_2$	55.54 / 55.42	5.75 / 5.72	14.87 / 14.91	195-198	75
3l	$C_{11}H_{11}N_3O_4S$	46.95 / 46.97	3.90 / 3.94	14.85 / 14.94	282-285 (dec.)	64
3m	$C_{17}H_{17}N_5O_6S$	48.72 / 48.68	4.13 / 4.09	16.75 / 16.70	215-217	72
3n	$C_{13}H_{14}N_2O_4$	59.62 / 59.54	5.40 / 5.38	10.65 / 10.68	169-171	78
3o	$C_9H_{12}N_2O_3$	55.15 / 55.09	6.18 / 6.16	14.37 / 14.28	128-129	12
3p	$C_{12}H_{12}N_2O_2$	66.65 / 66.65	5.58 / 5.59	12.97 / 12.96	144-146	56
5a	$C_{17}H_{14}N_2O_2$	73.32 / 73.37	5.10 / 5.07	10.18 / 10.07	165-166	56
5b	$C_{18}H_{16}N_2O_2$	73.84 / 73.95	5.55 / 5.52	9.63 / 9.58	195-196.5	60
5c	$C_{13}H_{14}N_2O_3$	63.52 / 63.40	5.70 / 5.73	11.44 / 11.38	162-162.5	75

bonds (1660-1625) and also the asymmetric (1770-1750) and symmetric (1720-1695 cm^{-1}) carbonyl group stretching vibrations of the pyrrole-2,5-dione ring in agreement with data in [2, 4, 5] (Table 2).

In our opinion, the experimental data indicates that the driving force for the transamination of the 1-alkyl-3-alkylaminopyrrole-2,5-diones by the amine hydrochlorides is likely to be formation of the less basic maleimide and the salt of the weaker conjugated acid i.e. alkylammonium. Hence the pK_a values of the amines increase in the series aniline (4.60), benzylamine (9.35), and methylamine (10.64) [12] and the values of the pK_a calculated by us using the *ACD/pK$_a$DB:Chem.Sketch* program [13] show that the basicity decreases in the series aniline (4.61±0.20), maleimide **1** (3.38±0.20) and maleimide **3a** (-2.69±0.20).

The transamination reaction evidently occurs as the result of a preliminary protonation of the 1-alkyl-3-alkylaminopyrrole-2,5-diones by the amine hydrochlorides leading to an increase in the electrophilicity of the carbon atom bound to the alkylamino group with subsequent attack of the formed cation by the free base and separation of the alkylamine.

TABLE 1. Spectroscopic Characteristics of Compounds **3a-p**, **5a-c**

Compound	IR spectrum, ν, cm^{-1}				^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz)*
	NH	$(CO)_{as}$	$(CO)_s$	C=C	
3a	3315	1750	1720	1645	2.90 (3H, s, NCH_3); 5.63 (1H, s, CH); 7.06-7.44 (5H, m, C_6H_5); 9.60 (1H, br. s, NH)
3b	3320	1755	1710	1640	2.90 (3H, s, NCH_3); 5.70 (1H, s, CH); 7.37 (2H, d, ^3J = 9.0, Ar); 7.52 (2H, d, ^3J = 9.0, Ar); 9.78 (1H, br. s, NH)
3c	3320	1770	1720	1660	2.25 (3H, s, CH_3); 2.87 (3H, s, NCH_3); 5.56 (1H, s, CH); 7.15 (2H, d, ^3J = 8.6, Ar); 7.28 (2H, d, ^3J = 8.6, Ar); 9.62 (1H, br. s, NH)
3d	3310	1760	1705	1645	2.28 (3H, s, CH_3); 2.87 (3H, s, NCH_3); 5.65 (1H, s, CH); 6.85-6.94 (1H, m, Ar) and 7.14-7.28 (3H, m, Ar); 9.58 (1H, br. s, NH)
3e	3365	1760	1705	1645	2.90 (3H, s, NCH_3); 3.90 (3H, s, OCH_3); 5.57 (1H, s, CH); 6.96-7.06 (1H, m, Ar); 7.08-7.22 (2H, m, Ar); 7.32-7.42 (1H, m, Ar); 8.24 (1H, br. s, NH)
3f	3330	1755	1715	1650	2.85 (3H, s, NCH_3); 5.38 (1H, s, CH); 6.74 (2H, d, ^3J = 9.0, Ar); 7.19 (2H, d, ^3J = 9.0, Ar); 9.39 (1H, br. s, OH); 9.52 (1H, br. s, NH)
3g	3315	1760	1710	1640	2.92 (3H, s, NCH_3); 6.06 (1H, s, CH); 7.63 (2H, d, ^3J = 9.3, Ar); 8.21 (2H, d, ^3J = 9.3, Ar); 10.21 (1H, br. s, NH)
3h	3320	1760	1695	1635	2.91 (3H, s, NCH_3); 5.85 (1H, s, CH); 7.60-7.67 (1H, m, Ar); 7.78-7.96 (2H, m, Ar); 8.23-8.30 (1H, m, Ar); 10.08 (1H, br. s, NH)
3i	3315	1760	1705	1640	2.90 (3H, s, NCH_3); 3.72 (2H, d. t, ^3J = 4.8, ^3J = 5.4, CH_2OH); 3.99 (2H, t, ^3J = 4.8, OCH_2);4.87 (1H, br. t, ^3J = 5.4, OH); 5.62 (1H, s, CH); 6.66-6.74 (1H, m, Ar); 6.94-7.04 (2H, m, Ar); 7.22-7.34 (1H, m, Ar); 9.63 (1H, br. s, NH)
3j	3310	1755	1695	1625	2.85 (9H, s, NCH_3 and $N(CH_3)_2$); 5.35 (1H, s, CH); 6.71 (2H, d, ^3J = 9.2, Ar); 7.22 (2H, d, ^3J = 9.2, Ar); 9.52 (1H, br. s, NH)
3k	3325	1760	1700	1645	2.87 (3H, s, NCH_3); 3.03 (6H, s, $N(CH_3)_2$); 5.71 (1H, s, CH); 7.42-7.68 (4H, m, Ar); 9.78 (1H, br. s, NH)
3l	3310	1765	1705	1640	2.92 (3H, s, NCH_3); 5.91 (1H, s, CH); 7.28 (2H, s, NH_2); 7.57 (2H, d, ^3J = 9.0, Ar); 7.80 (2H, d, ^3J = 9.0, Ar); 9.92 (1H, br. s, NH)
3m	3340	1755	1710	1650	2.92 (1H, s, NCH_3);3.79 (3H, s, OCH_3); 3.82 (3H, s, OCH_3);5.95 (1H, s, CH);5.98 (1H, s, CH); 7.62 (2H, d, ^3J = 8.1, Ar); 7.92 (2H, d, ^3J = 8.1, Ar); 10.01 (1H, br. s, NH); 11.56 (1H, br. s, SO_2NH)
3n	3330	1765	1705	1655	3.40-3.52 (4H, m, $(CH_2)_2$); 3.73 (3H, s, OCH_3); 4.79 (1H, br. s, OH); 5.46 (1H, s, CH); 6.92 (2H, d, ^3J = 8.8, Ar); 7.32 (2H, d, ^3J = 8.8, Ar); 9.58 (1H, br. s, NH)
3o	–	1760	1725	1625	2.75 (3H, s, CH_3); 3.45-3.78 (8H, m, $O(CH_2CH_2)_2$); 5.15 (1H, s, CH)
3p	3360	1745	1705	1640	2.81 (3H, s, CH_3); 4.32 (2H, d, ^3J = 6.6, CH_2); 4.86 (1H, s, CH); 7.24-7.44 (5H, m, C_6H_5);8.35 (1H, br. t, ^3J = 6.6, NH)
5a	3310	1765	1710	1650	4.62 (2H, s, CH_2); 5.56 (1H, s, CH); 7.03-7.42 (10H, m, $2C_6H_5$); 9.60 (1H, br. s, NH)
5b	3320	1765	1705	1650	2.32 (3H, s, CH_3); 4.62 (2H, s, CH_2); 5.48 (1H, s, CH); 7.12 (2H, d, ^3J = 8.4, Ar); 7.19-7.35 (7H, m, C_6H_5 + Ar); 9.54 (1H, br. s, NH)
5c	3315	1750	1700	1635	2.28 (3H, s, CH_3); 3.44-3.61 (4H, m, $(CH_2)_2$); 4.81 (1H, br. t, ^3J = 5.5, OH); 5.57 (1H, s, CH); 7.17 (2H, d, ^3J = 8.4, Ar); 7.30 (2H, d, ^3J = 8.4, Ar); 9.60 (1H, br. s, NH)

* ^{1}H NMR Spectra recorded on a Bruker DRX-500 instrument (compound **3a**), Varian VXR-300 (compounds **3b, e ,g-i, l, m, p, 5a-c**), or Varian VX-200 (compounds **3c, d, f, j, k, n, o**).

EMPERIMENTAL

^{1}H NMR Spectra were taken on Bruker DRX-500 (500 MHz), Varian VXR-300 (300 MHz), and Varian VX-200 (200 MHz) spectrometers with TMS as internal standard. IR Spectra were recorded on a UR-20 spectrometer for KBr tablets. CHN-analysis was performed on a Perkin Elmer 2400 instrument. 2-(3-Aminophenoxy)ethanol was prepared by method [14] and 1-methyl-3-methylaminopyrrole-2,5-dione **1**, 1-benzyl-3-benzylaminopyrrole-2,5-dione, and 1-(2-hydroxyethyl-3-(2-hydroxyethylamino)pyrrole-2,5-dione by [11]. *p*-Aminobenzenesulfamide and 4-amino-N-(2,6-dimethoxypyrimidin-4-yl)benzenesulfamide were prepared from the medicines *streptocid* and *sulfadimethoxine*. Monitoring of the reaction course and purity of the materials was carried out by TLC on Silufol UV-254 plates in the system chloroform-methanol (10: 1) and revealed using UV light and/or iodine vapor.

1-Methyl-3-phenylaminopyrrole-2,5-dione (3a). A solution of compound **2a** (prepared from aniline (0.73 g, 7.8 mmol), hydrochloric acid (8.371 N, 0.85 ml), and methanol (10 ml)) was added to a solution of maleimide **1** (1.0 g, 7.1 mmol) in methanol (10 ml), refluxed for 1 h, and then left overnight. The precipitate was filtered off, washed with methanol, and recrystallized from 2-propanol to give compound **3a** (1.0 g). Using the hydrochloride of **2a** the yield of maleimide **3a** was 1.08 g (75%).

Compounds 3b-k,n,p were prepared similarly. **Compounds 3g,h** were crystallized from acetic acid, **compound 3p** from methanol, and **compounds 3f,k** were purified by refluxing in methanol.

1-Methyl-3-(*p*-sulfamidophenylamino)pyrrole-2,5-dione (3l). A solution of compound **2l** (prepared from *p*-aminobenzenesulfamide (1.34 g, 7.8 mmol), hydrochloric acid (8.371 N, 0.85 ml), and DMSO (15 ml)) was added to a solution of maleimide **1** (1.0 g, 7.1 mmol) in DMSO (5 ml), heated for 1 h at 50ºC, left overnight, and poured into methanol (100 ml). The precipitate was filtered off, washed with methanol, and refluxed in methanol to give compound **3l** (1.28 g).

Compound **3m** was prepared similarly.

1-Methyl-3-morpholinopyrrole-2,5-dione (3o). Maleimide (3.5 g, 25 mmol) was added to morpholine (11 ml, 11 g, 126 mmol), refluxed for 11.5 h, allowed to stand overnight, and morpholine (7.8 ml) was removed by distillation *in vacuo*. The residue was dissolved in benzene (5 ml), cooled to 0ºC and the precipitate formed was filtered off and washed with cold benzene to give a 1: 1 mixture of maleimides **1** and **3o** (1.5 g) with mp 99-101ºC. The latter was recrystallized from ethanol with the addition of active charcoal to give the product (0.71 g) with mp 125-128ºC which was recrystallized from a mixture of benzene and petroleum ether. Yield of maleimide **3o** 0.58 g.

Dimethyl Phenylaminofumarate (4a). A solution of aniline (12.8 g, 137 mmol) in benzene (50 ml) was added dropwise over 1 h with stirring at 5-10ºC to a solution of dimethyl acetylenedicarboxylate (19.4 g, 137 mmol) in benzene (50 ml). Solvent was evaporated at reduced pressure and the residue was distilled *in vacuo* to give compound **4a** (25.7 g, 80%) with bp 140.5-141.5 (0.5 mm Hg) and n_D^{20} 1.5883 (bp 105-107ºC at 0.05 to 0.07 mm Hg [5]). ^{1}H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm: 3.64 (3H, s, OCH_3); 3.70 (3H, s, OCH_3); 5.38 (1H, s, CH); 6.83-7.28 (5H, m, C_6H_5); 9.67 (1H, br. s, NH).

Dimethyl *p*-tolylaminofumarate (4b) was prepared similarly, yield 70%, mp 89-91ºC, bp 175-177ºC (2 mm Hg) (mp 89ºC, bp 140ºC (0.5 mm Hg) [15]). ^{1}H NMR spectrum, (300 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 2.30 (3H, s, CH_3); 3.69 (3H, s, OCH_3); 3.73 (3H, s, OCH_3); 5.33 (1H, s, CH); 6.82 (2H, d, ^3J = 8.4, Ar); 7.06 (2H, d, ^3J = 8.4, Ar); 9.62 (1H, br. s, NH).

1-Methyl-3-phenylaminopyrrole-2,5-dione (3a). An aqueous solution of methylamine (12.7 N, 1.5 ml, 19.1 mmol) was added to a solution of phenylaminofumarate **4a** (1.5 g, 6.4 mmol) in methanol (15 ml) and held for 7 days at 20ºC. The precipitate was filtered off, washed with methanol, and recrystallized from 2-propanol to give the maleimide **3a** (0.90 g, 70%).

1-Benzyl-3-phenylaminopyrrole-2,5-dione (5a). Benzylamine (3.5 ml, 3.4 g, 31.7 mmol) was added to a solution of phenylaminofumarate (5.0 g, 21.3 mmol) in methanol (20 ml) and held for 10 days at 20ºC. The precipitate was filtered off, washed with methanol, and recrystallized from 2-propanol to give the maleimide **5a** (3.3 g).

Compound 5b was prepared similarly.

1-(2-Hydroxyethyl)-3-(*p*-tolylamino)pyrrole-2,5-dione (5c). Ethanolamine (2.0 ml, 2.0 g, 33.4 mmol) was added to a solution of *p*-tolylaminofumarate **4b** (5.0 g, 20 mmole) in methanol (30 ml), held for 0.5 h at 60ºC, and then left for 10 days at 20ºC. The precipitate was filtered off, washed with methanol, and recrystallized from methanol to give compound **5c** (28 g, 57%). The filtrate was evaporated *in vacuo* and the residue was extracted with chloroform (60 ml), washed with water (3 x 10 ml), dried over $CaCl_2$, and the chloroform was evaporated. The residue was crystallized from methanol to give an additional yield of compound **5c** (1.1 g, 18%).

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