2,4,5-Substituted Furan-3(2H)-ones: Synthesis, Reactions with Amino Acid and Hydrazine Derivatives

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ABSTRACT: *Furan-3(2H)-ones (**3**) were obtained from some 2,3-dihydro-furan-2,3-diones with a few Wittig reagents (**2**). The compounds of **3** with glycin and hydrazines (**4a,b**) produced 2,3-dihydro-1H-pyrrol-3-ones (**5a–d**). All the reaction mechanisms were discussed by utilizing the similar reaction pathways. Structures of these compounds were determined by the IR, NMR, elemental analysis, and X-ray diffraction method.* © 2005 Wiley Periodicals, Inc. Heteroatom Chem 16:235–241, 2005; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20115

INTRODUCTION

A convenient synthesis of functionalized furan-3(2H)-one (**3a**) [1a] and its reactions with primary amines, which produced 2,3-dihydro-1H-pyrrol-3-ones, has been reported previously [1b–d]. Since 2,3-dihydo-furan-3-ones and 2,3-dihydro-1H-pyrrol-3-ones in general have much more interest for biological and medicinal reasons [2–11], we have now extended our investigation to derivatizations and reactions of furan-3(2H)-one with amino acid and hydrazine derivatives.

RESULTS AND DISCUSSION

A number of 2,4,5-substitued furan-3(2H)-ones (**3b–d**) were obtained in good yields (67–88%) from the reactions of the 2,3-dihydrofuran-2,3-diones (**1**) and the Wittig reagents (**2**) (Scheme 1). According to spectroscopic data of **3**, the Wittig reactions of **1** with **2** afford stereo- and regio-selectively products (**3**). In this reaction, Wittig reagents attack on ester's carbonyl of 2,3-dihydrofuran-2,3-diones, but not on keton's carbonyls. The reactions of **2** with ester's carbonyl group of **1** produce various intermediates. These intermediates have relatively low energy by means of intramolecular hydrogen bonds, and the distribution of charges on various atoms is shown in Scheme 2. Therefore, the reaction pathway of **1** and **2** follows Scheme 2.
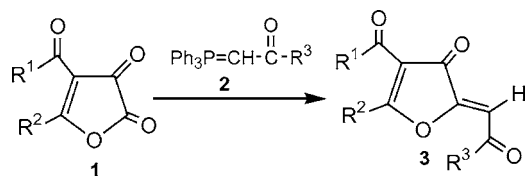
In the reaction of 4-benzoyl-5-phenyl-2,3-dihydro-furan-2,3-dione with methylenetriphenylphosphorane, the formation of the above-mentioned similar intermediates was previously reported by Kollenz et al. [12] as shown in Scheme 3. Also, the stable one intermediate type was isolated from reactions of alkoxy carbonylmethylenetriphenylphosphoranes with some 2,3-dihydrofuran-2,3-diones
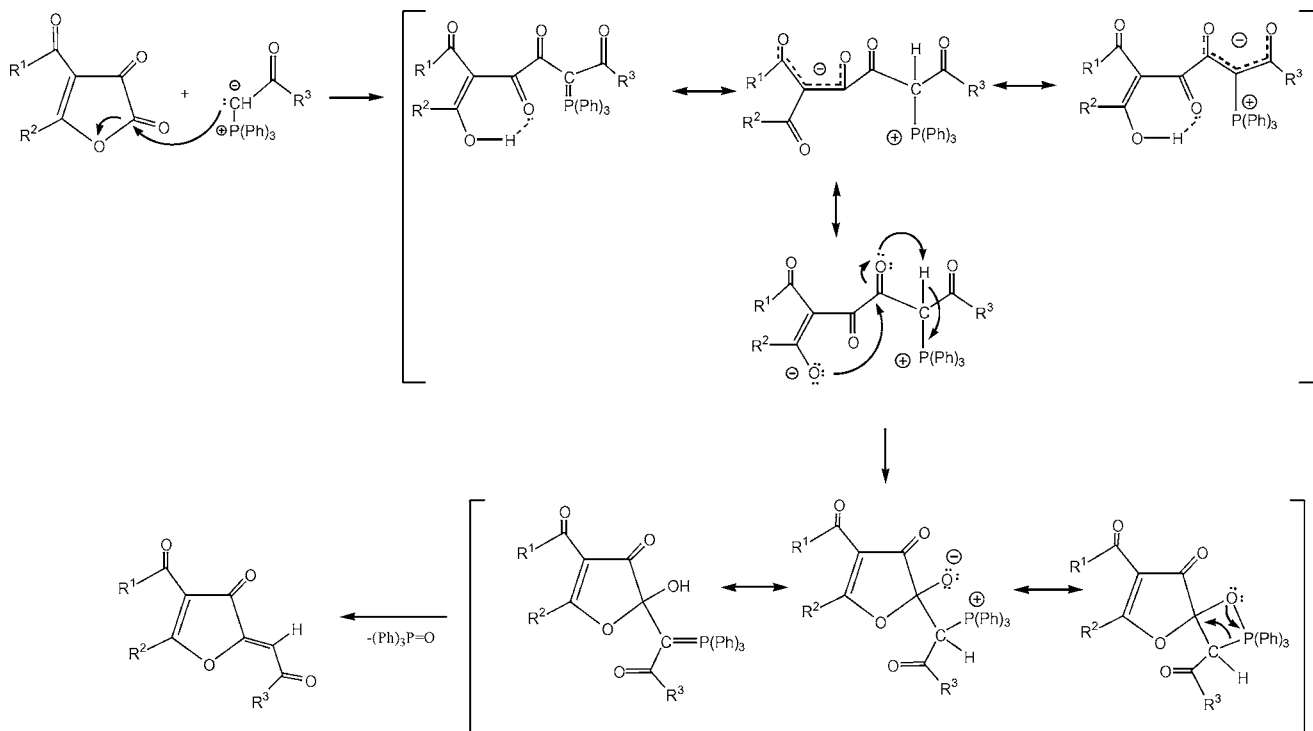
SCHEME 1

by Kozminky et al. [5]. This reaction pathway is illustrated in Scheme 4.

In the light of this, based on the proposed reaction pathway, we showed in detail the reaction pathway of **1** with **2** as outlined in Scheme 2.

The structures of **3** were elucidated mainly from elemental analyses, IR, and NMR spectroscopic data (see Experimental). We have performed an X-ray single-crystal study on compound **3d**. There are four different groups connected to furan-3(2H)-one

ring of **3d**. These groups are 4-methoxybenzoyl, 4-methoxyphenyl, oxo and 2-oxopropylidene. The molecule (**3d**) contains two 4-methoxyphenyl rings A (C9–C14) and B (C16–C21) connected to the furan-3(2H)-one ring C (C1–C4, O1), see Fig. 1. The 2-oxopropylidene group D (C5–C7, O5) is also connected to ring C. A least-square plane analysis shows that all rings are fairly planar. The dihedral angles between the planes are as follows: A/B = 66.65(12), A/C = 60.90(14), A/D = 74.14(14), B/C = 11.69(16), B/D = 9.10(18), and C/D = 14.28(17)°. The molecules in the crystal structure are connected by van der Waals interaction. There are three intermolecular and one intramolecular hydrogen bond in the unit cell (Table 1). Compound **3a** [1a] shows similar structures to the compound **3d** apart from 2-oxopropylidene group. A comparison of the bond lengths and angles of the present work (Table 2,3) with similar values in [1a] shows that they are to be very similar.

The reactions of **3a** with phenylhydrazine and hydrazine hydrate produced very complex products which cannot be isolated. These hydrazines contain very active two N-nucleophilic atoms, and for this reason the reactions lead to a variety of non isolable products. But, in the event that there are more strongly an electron-withdrawing groups attached to N atom of hydrazine, this type of hydrazines may be reacted to compound **3** by using
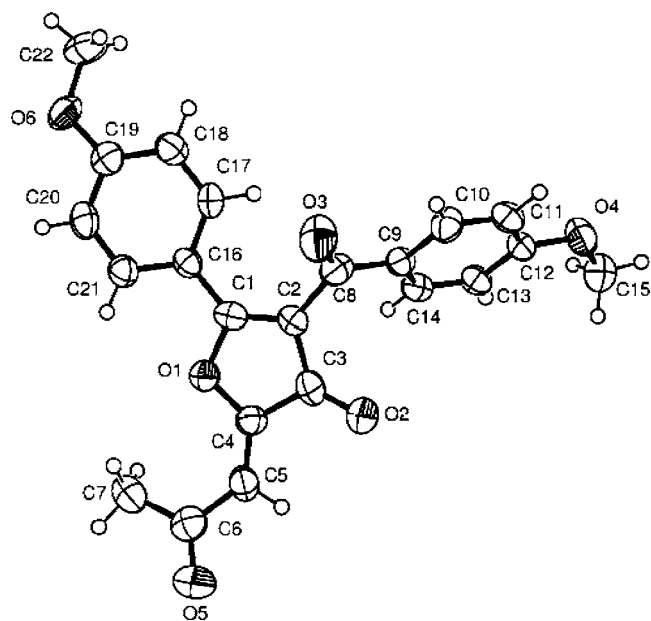

SCHEME 2
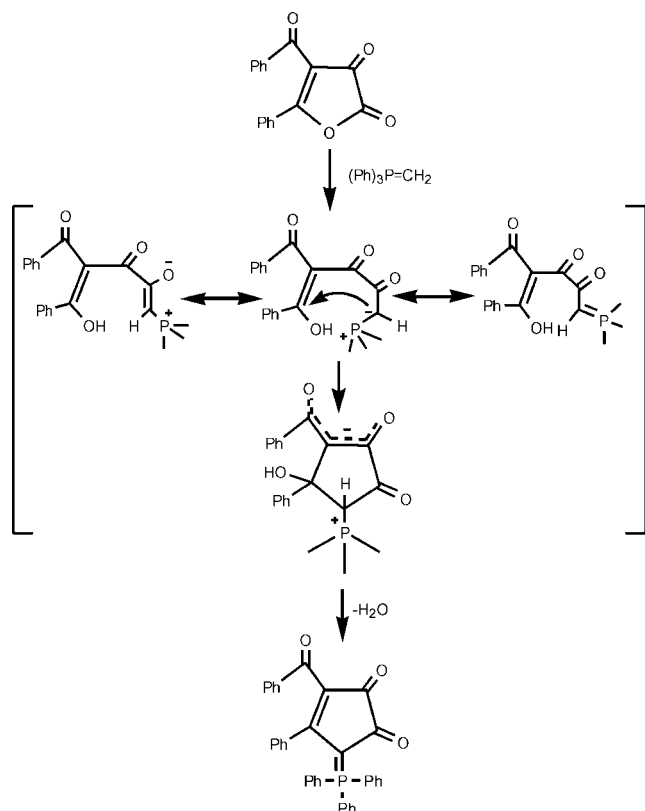
SCHEME 3 Reaction pathway of [12] for Wittig reaction of 2,3-dihydrofuran-2,3-dione with a Wittig reagent.

FIGURE 1 ORTEP drawing of **3d** with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

only NH$_2$ nucleophilic group in one reaction pathway. In this way, enough pure crude product can be obtained.

Really, the reaction product **5b** was obtained as a relatively pure crude product from the reaction of **3a** with **4b**. In addition, compounds **3a,b** gave the nucleophilic addition reaction by using NH$_2$ group of *N*,*N*-dimethyl substituted hydrazine and only one type product *N*-amino substituted 2,3-dihydro-1*H*-pyrrol-3-one (**5a,c**) was obtained. Very interestingly, compounds **3b** with **4b** and **3d** with **4a,b** did not give isolable products (Scheme 5).

On the other hand, **3a** and glycine were produced with a similar pathway involving amino acid

moiety, the corresponding *N*-acetic acid substituted 2,3-dihydro-1*H*-pyrrol-3-one (**5d**) in methanol, and the presence of pyridine (Scheme 6). In the ^{1}H NMR spectra, acid proton of **5d** was detected at 3.42 ppm as a broad band.

Formation of **5** proceeds a reaction pathway as shown in Scheme 7. In compounds **5**, elimination of water did not occur in the reaction medium; because, α-hydrogens of esters of compound **5** are not adequately acidic and also pH of the reaction medium did not cause elimination of water in these reactions.

EXPERIMENTAL

1a [13], **1b** [14], and **3a** [1a] were prepared according to the published method. Melting points were determined on an electrothermal 9200 apparatus and uncorrected. Elemental analysis was performed with a Carlo Erba elemental analyzer, 1108. FT-IR
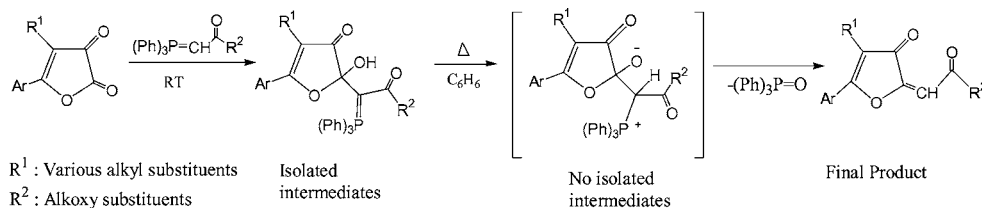
SCHEME 4 Reaction pathway of [5] for Wittig reactions of 2,3-dihydrofuran-2,3-diones with Wittig reagents.

TABLE 1 Hydrogen Bonding Geometry (Å,°)

D	- $H \cdots$	A	D-H	$H \cdots A$	$D \cdots A$	D-$H \cdots A$
C7	H7C	O3^i	0.9602	2.4873	3.309 (6)	143.52
C13	H13	O5^{ii}	0.9301	2.5481	3.467 (6)	169.64
C15	H15B	O5^{ii}	0.9600	2.5612	3.333 (7)	137.59
C21	H21	O1	0.9296	2.4505	2.771 (6)	100.25

Symmetry codes: (i) $1 - x, -y, -z$ (ii) $2 - x, \frac{1}{2} + y, \frac{1}{2} - z$.

spectrum was measured on a Jasco plus model 460 spectrometer, using potassium bromide pellet. The ^{1}H and ^{13}C NMR spectra were obtained on a Varian AS 400-mercury instrument. The chemical shifts are reported in ppm from tetramethylsilane and are given in δ units. All experiments were followed by TLC using DC Alufolien Kieselgel 60 F 254 Merck and Camag TLC lamp (254/366 nm).

Crystallography

The compound crystallizes in the monoclinic system with space group $P2_1/c$, $a = 11.8677(12)$, $b = 12.1778(13)$, $c = 13.3113(14)$ Å, $\beta = 107.226(3)°$, $V = 1837.5(3)$ Å^3, $Z = 4$. The intensity data were collected at room temperature using an Enraf-Nonius CAD 4 diffractometer [15] with Mo K$_\alpha$ radiation using $\omega/2\,\theta$ scan mode. The cell parameters were determined from least-squares analysis using 25 centered reflections. Three standard reflections were periodically measured (every 120 min) during data collection and showed no significant intensity variations. The structure was solved by direct methods using the solution program SHELXS97 [16] in the WinGX package [17], and was refined using SHELXL97 [16]. All non-hydrogen atoms were refined, first with isotropic and then with anisotropic thermal displacement parameters by full-matrix least squares (see Table 4). All hydrogen atoms were placed geometrically and were refined as riding with $U_{iso}(H) = 1.2 U_{eq}(C)$. The final

TABLE 2 Selected Bond Lengths for Non-hydrogen Atoms (Å)

C1	O1	1.396 (5)
C2	C8	1.495 (6)
C3	O2	1.231 (5)
C4	O1	1.377 (5)
C6	O5	1.217 (5)
C8	O3	1.218 (5)
C12	O4	1.364 (5)
C15	O4	1.420 (5)
C19	O6	1.355 (5)
C22	O6	1.435 (5)

TABLE 3 Selected Bond Angles for Non-hydrogen Atoms (°)

|			
C2	C1	O1	112.6 (4)
O1	C1	C16	113.8 (4)
O2	C3	C2	130.7 (5)
O2	C3	C4	124.0 (5)
C5	C4	O1	126.3 (4)
O1	C4	C3	107.6 (4)
O5	C6	C5	117.3 (5)
O5	C6	C7	122.2 (5)
O3	C8	C9	122.1 (4)
O3	C8	C2	119.0 (4)
O4	C12	C11	114.8 (4)
O4	C12	C13	124.3 (4)
O6	C19	C20	116.4 (5)
O6	C19	C18	123.9 (5)
C4	O1	C1	107.3 (3)
C12	O4	C15	118.1 (4)
C19	O6	C22	118.3 (4)

cycle of the refinement included 253 variable parameters and gave $R = 0.056$, $wR = 0.125$, and goodness of fit $= 1.016$. The minimum and maximum residual electron densities were -0.258 and 0.449 eÅ^{-3}, respectively.

General Procedure for **3**

A solution of 2,3-dihydro-furan-2,3-diones **1** (10 mmole) and **2** (10 mmole) in dry benzene (70 mL) were refluxed for 30 min. After removal of the solvents, the residues were washed with cool methanol (20 mL, 0–5°) and crystallized to give pure **3**.
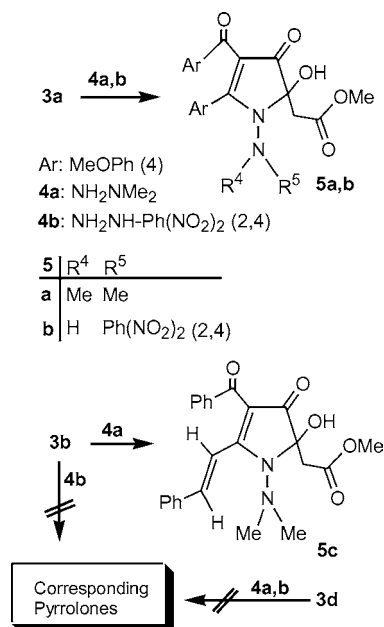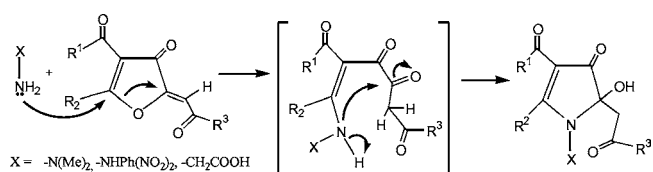

SCHEME 5
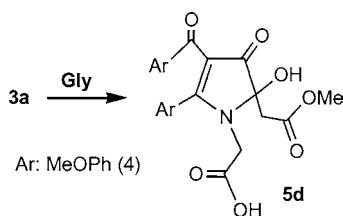
SCHEME 6

Methyl (2Z)-[4-benzoyl-3-oxo-5-[(E)-2-phenylvin-yl]furan-2(3H)-ylidene]acetate (3b). Compound **3b** was prepared from 3.04 g **1b** and 3.94 g **2a**. Orange crystals from methanol. Yield (2.56 g, 68%), mp 153°C. IR (KBr): υ = 1725, 1703, 1645 cm^{-1} (C=O), 1673, 1612, 1605, 1577 (C=C). ^{1}H NMR (400 MHz, CDCl$_3$): δ = 3.89 (s, 3H, OCH$_3$), 6.15 (s, 1H, =CH–CO), 7.26–8.09 ppm (m, 12H, Ar-H, and styryl-CH). ^{13}C NMR (CDCl$_3$) δ = 52.64 (OCH$_3$), 100.55 (ArCO–C̲=C), 114.12 (COC̲H=C), 115.73 (Ar–CH=C̲H), 128.56–137.44 and 164.13 (Ar-C), 146.23 (Ar–C̲H=CH), 152.17 (COCH=C̲), 181.45 (C̲OOMe), 182.84 (furanon's C=O), 188.91 (benzoyl's C=O). C$_{23}$H$_{20}$O$_5$(376): calcd C 73.39, H 5.36; found C 73.32, H 5.35.

Ethyl (2Z)-[4-benzoyl-3-oxo-5-[(E)-2-phenylviny-l]furan-2(3H)-ylidene]acetate (3c). Compound **3c** was prepared from 3.04 g **1b** and 3.48 g **2c**. Orange crystals from methanol. Yield (2.61 g, 67%), mp 148°C. IR (KBr): υ = 1722, 1700, 1644 cm^{-1} (C=O), 1674, 1610, 1604, 1575 (C=C). ^{1}H NMR (400 MHz, CDCl$_3$): δ = 1.39, 1.40, 1.42 (t, 3H, CH$_3$), 4.32, 4.33, 4.35, 4.37 (q, 2H, OCH$_2$), 6.12 (s, 1H, CO–CH=C), 7.26–8.08 (m, 12H, Ar-H, and styryl-H). ^{13}C NMR (CDCl$_3$) δ = 14.49 (CH$_3$), 61.66 (OCH$_2$), 101.09 (ArCO–C̲=C), 114.16 (COC̲H=C), 115.73 (Ar–CH=C̲H), 128.55–137.46 and 163.72 (Ar-C), 146.11 (Ar–C̲H=CH), 151.10 (COCH=C̲), 181.36 (C̲OOEt), 182.88 (furanon's C=O), 188.92 (benzoyl's CO). C$_{24}$H$_{22}$O$_5$(390): calcd C 73.83, H 5.68; found C 73.88, H 5.68.

(2Z)-4-(4-Methoxybenzoyl)-5-(4-methoxyphenyl)-2-(2-oxopropylidene)furan-3(2H)-one (3d). Compound **3d** was prepared from 3.38 g **1a** and 3.18 g

SCHEME 7

TABLE 4 Final Atomic Coordinates and Equivalent Anisotropic Thermal Parameters for Non-hydrogen Atoms

Atom	x	y	z	Ueq
O1	0.6675 (3)	0.0738 (3)	0.0350 (2)	0.0452 (8)
O2	0.8285 (3)	−0.0272 (3)	0.2812 (3)	0.0734 (12)
O3	0.4956 (3)	0.0320 (3)	0.2742 (3)	0.0751 (12)
O4	0.8488 (3)	0.2040 (3)	0.6954 (3)	0.0617 (10)
O5	0.9304 (3)	−0.1182 (3)	−0.0491 (3)	0.0823 (13)
O6	0.1991 (3)	0.3493 (3)	−0.1080 (3)	0.0622 (10)
C1	0.5998 (4)	0.1055 (4)	0.0998 (4)	0.0417 (12)
C2	0.6475 (4)	0.0727 (4)	0.2009 (3)	0.0423 (12)
C3	0.7560 (4)	0.0174 (4)	0.2066 (4)	0.0505 (13)
C4	0.7641 (4)	0.0178 (4)	0.0968 (4)	0.0460 (13)
C5	0.8451 (4)	−0.0329 (4)	0.0639 (4)	0.0511 (13)
C6	0.8570 (4)	−0.0516 (5)	−0.0413 (4)	0.0535 (14)
C7	0.7818 (4)	0.0090 (5)	−0.1332 (4)	0.0676 (16)
C8	0.5932 (4)	0.0721 (4)	0.2890 (4)	0.0482 (13)
C9	0.6631 (4)	0.1119 (4)	0.3926 (3)	0.0380 (12)
C10	0.6239 (4)	0.0891 (4)	0.4793 (4)	0.0488 (14)
C11	0.6870 (4)	0.1202 (4)	0.5788 (4)	0.0504 (14)
C12	0.7918 (4)	0.1773 (4)	0.5935 (4)	0.0457 (13)
C13	0.8319 (4)	0.2043 (4)	0.5087 (4)	0.0439 (12)
C14	0.7663 (4)	0.1713 (4)	0.4093 (4)	0.0424 (12)
C15	0.9663 (4)	0.2438 (5)	0.7194 (4)	0.0662 (16)
C16	0.4958 (4)	0.1685 (4)	0.0461 (4)	0.0409 (12)
C17	0.4337 (4)	0.2255 (4)	0.1037 (4)	0.0521 (14)
C18	0.3348 (4)	0.2867 (4)	0.0564 (4)	0.0531 (14)
C19	0.2961 (4)	0.2929 (4)	−0.0529 (4)	0.0491 (13)
C20	0.3590 (4)	0.2397 (4)	−0.1115 (4)	0.0517 (14)
C21	0.4565 (4)	0.1788 (4)	−0.0633 (3)	0.0463 (13)
C22	0.1257 (4)	0.3976 (4)	−0.0515 (4)	0.0674 (16)

2b. Yellow crystals from CCl$_4$. Yield (3.33 g, 88%), mp 165°C. IR (KBr): υ = 1688 cm^{-1}(MeC=O), 1667 (pyrrol's C=O), 1640 (Ar–C=O), ^{1}H NMR (400 MHz, CDCl$_3$): δ = 2.64 (s, 3H, COCH$_3$), 3.86, 3.95 (s, 6H, OCH$_3$), 6.21 (s, 1H, MeCOCH=), 6.91–7.92 (m, 8H, Ar-H). ^{13}C NMR (CDCl$_3$)δ = 31.68 (CH$_3$), 55.48, 55.60 (OCH$_3$), 107.12–164.56 (C=C, arom. and aliph.), 184.72 (furanon's C=O), 188.24 (COMe), 196.24 (anisoyl's C=O). C$_{22}$H$_{18}$O$_6$(378): calcd C 69.83, H 4.79; found C 69.97, H 4.88.

Reactions of 3 with Hydrazines (4a,b)

General Procedure. To solutions of **3** (10 mmole) in dry benzene (50 mL) were added **4a,b** (10 mmole) and stirred for 1 h at room temperature. After solvents were removed by evaporation, the oily residue was treated with dry diethyl ether and *n*-hexane (2:1) to get corresponding crude pyrrolones (**5a–c**), which were purified by recrystallization.

Methyl [1-(dimethylamino)-2-hydroxy-4-(4-meth-oxybenzoyl)-5-(4-methoxyphenyl)-3-oxo-2,3-dihydro-1H-pyrrol-2-yl]acetate (5a). Compound **5a** was

prepared from 0.60 g **4a** and 3.94 g **3a**. Yellow crystals from CCl$_4$. Yield (2.59 g, 57%), mp 134°C. IR (KBr): υ = 3213 (OH), 1743 (C=O, ester), 1684 (Ar—C=O), 1638 (pyrrol's C=O). ^{1}H NMR (400 MHz, CDCl$_3$): δ = 2.66 (s, 6H, N(CH$_3$)$_2$), 3.12 (s, 2H, CH$_2$), 3.70, 3.78, 3.79 (s, 9H, OCH$_3$), 6.06 (s, 1H, broad OH), 6.74–7.74 (m, 8H, Ar-H). ^{13}C NMR (CDCl$_3$) δ = 40.21 (CH$_2$), 45.05 (N(CH$_3$)$_2$), 51.95, 55.14, 55.20 (OCH$_3$), 90.72 (C-OH), 110.58–162.72 (arom. and aliph. C=C), 179.10 (COOMe), 188.49 (pyrrol's C=O), 192.18 (anisoyl's C=O). C$_{24}$H$_{26}$N$_2$O$_7$(454): calcd C 63.43, N 6.16, H 5.77; found C 63.30, N 6.25, H 5.75.

*Methyl {1-[(2,4-dinitrophenyl)amino]-2-hydroxy-4-(4-methoxybenzoyl)-5-(4-methoxyphenyl)-3-oxo-2,3-dihydro-1H-pyrrol-2-yl}acetate (**5b**).* Compound **5b** was prepared from 1.98 g **4b** and 3.94 g **3a**. Red crystals from cyclohexane-diethylether (3:1). Yield (3.37 g, 57%), mp 182°C. IR (KBr): υ = 3427 (broad OH), 3290 (NH), 1729 (C=O, ester), 1712 (Ar—C=O), 1651 (pyrrol's C=O). ^{1}H NMR (400 MHz, DMSO): δ = 3.28, 3.33, 3.77, 3.81 (q, 2H, CH$_2$), 3.37, 3.77, 3.83 (s, 9H, OCH$_3$), 6.29 (OH), 7.02–8.81 (m, 11H, Ar-H), 10.90 (NH). ^{13}C NMR (DMSO) δ = 53.60, 57.19, 57.44 (OCH$_3$), 103.95 (C—OH), 116.07–165.61 (arom. and aliph. C=C), 169.99 (COOMe), 181.45 (pyrrol's C=O), 197.90 (anisoyl's C=O), due to DMSO signals there is no detection of CH$_2$ signal. C$_{28}$H$_{24}$N$_4$O$_{11}$(592): calcd C 56.76, N 9.46, H 4.08; found C 56.70, N 9.53, H 4.05.

*Methyl{4-benzoyl-1-(dimethylamino)-2-hydroxy-3-oxo-5-[(E)-2-phenylvinyl]-2,3-dihydro-1H-pyrrol-2-yl}acetate (**5c**).* Compound **5c** was prepared from 0.60 g **4a** and 3.76 g **3b**. Yellow crystals from benzene. Yield (2.36 g, 52%), mp 174°C. IR (KBr): υ = 3194 (OH), 1738 (C=O, ester), 1689 (Ar-C=O), 1636 (pyrrol's C=O). ^{1}H NMR (400 MHz, CDCl$_3$): δ = 2.86 (s, 6H, N(CH$_3$)$_2$), 3.01, 3.05, 3.17, 3.21 (q, 2H, CH$_2$), 3.58 (s, 3H, OCH$_3$), 7.25–7.70 (m, 10H, Ar-H), no OH detection. ^{13}C NMR (DMSO) δ = 41.54 (CH$_2$), 46.13 (N(CH$_3$)$_2$), 52.44 (OCH$_3$), 90.56 (C-OH), 108.17–169.52 (arom. and aliph. C=H C), 173.95 (COOMe), 189.88 (pyrrol's C=O), 192.73 (benzoyl's C=O). C$_{24}$H$_{24}$N$_2$O$_5$(420): calcd C 68.56, N 6.66, H 5.75; found C 68.50, N 6.60, H 5.85.

*[2-Hydroxy-4-(4-methoxybenzoyl)-2-(2-methoxy-2-oxoethyl)-5-(4-methoxyphenyl)-3-oxo-2,3-dihydro-1H-pyrrol-1-yl] Acetic Acid (**5d**).* 0.103 g (1 mmol) glycine and 0.394 g (1 mmol) **3a** were refluxed in mixture of 50 mL methanol and 5 mL pyridin for 6 h. After solvents were removed by evaporation, the oily residue was treated with dry diethyl ether to get

a corresponding crude pyrrolone (**5d**), which was purified by recrystallization. White crystals from water. Yield (0.356 g, 76%), mp 182°C. IR (KBr): υ = 3419 (OH, acid), 3153 (OH, alcohol), 1747, 1728, 1689 (C=O). ^{1}H NMR (400 MHz, DMSO): δ = 2.92, 2.93 (d, 2H, N-CH$_2$), 3.42 (s, 1H, broad OH), 3.57, 3.75, 3.77 (s, 9H, OCH$_3$), 3.88, 3.93, 4.12, 4.17 (q, 2H, C$\underline{H}_2$COOMe), 6.86–7.67 (Ar-H). ^{13}C NMR (DMSO) δ = 39.47 ($\underline{C}H_2$COOMe), 44.38 (N-CH$_2$), 52.19, 55.96, 56.06 (OCH$_3$), 88.33 (C-OH), 111.41 (Ar-C=$\underline{C}$), 113.65–162.99 (arom-C), 169.01 (Ar-$\underline{C}$=C), 179.85 (COOMe), 188.04 (pyrrol's C=O), 194.10 (anisoyl's C=O). C$_{24}$H$_{23}$NO$_9$(469): calcd C 61.40, N 2.98, H 4.94; found C 61.50, N 2.95, H 4.98.

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