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REACTION OF SOME FURAN-2,3-DIONES WITH VARIOUS 1,2-PHENYLENEDIAMINES

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Abstract - Condensation of 4-aroyl-5-aryl-2,3-furandiones **(1a,b)** and ethyl 4,5-dioxo-2-phenyl-4,5-dihydrofuran-3-carboxylate **(1c)** with various 1,2-phenylenediamines **(2d-f)** gave 2(1*H*)-quinoxalinone derivatives, 1,3-diaryl-2-(3-oxo-3,4-dihydro-2-quinoxalinyl)-1,3-propanediones **(3ae,af,bd,be,bf)** and ethyl 3-aryl-3-oxo-2-(2-oxo-1,2-dihydro-2-quinoxalinyl)propanoate **(3cd,ce)**. Alkaline treatment of 1,3-propanediones **(3af,bd,bf)** gave corresponding 3-aroylmethyl-2(1*H*)-quinoxalinones **(4af,bd,bf)**.

Introduction

Quinoxaline derivatives are an important class of nitrogen-containing heterocycles and they constitute useful intermediates in organic synthesis. Some of them are applied for dyes[1] and for building blocks in the synthesis of organic semiconductors,[2] and show interesting biological properties (antibacterial, antiviral, anticancer, antifungal, antihelmintic, insecticidal).[3] Oxidation of both nitrogens of the quinoxaline ring dramatically increased the diversity of certain biological properties, such as antibacterial activity,[4] promotion of animal growth in feed additives,[5] and hypoxia-selective anticancer activity.[6] Several kinds of compounds that were activated under hypoxic conditions are at various stages of development including agents derived from 1,2,4-benzotriazine 1,4-di-*N*-oxide,[7] and quinoxaline 1,4-di-*N*-oxide.[8] 2,3-Disubstituted quinoxalines can be prepared by condensation of aryl-1,2-diamines and α-dicarbonyl compounds.[9] Recently, reactions of cyclic oxalyl compounds have been reported to give corresponding heterocyclic compounds.[10] The reactions of 4-benzoyl-5-phenyl-2,3-furandione **(1a)**, prepared easily from dibenzoylmethane and oxalyl dichloride,[10a] with several semicarbazones, ureas and their thio-anologues, oximes, hydrazines and various phenylhydrazones have been reported to cause the cleavage of the furan ring.[11] The reactions might be caused by Michael-attack of nitrogen atom on C-5 in the furandione ring.[12] The general reactivity of 4-benzoyl-5-phenylfuran-2,3-dione **(1a)** and the

mechanism of the reactions with NH-nucleophiles have recently been reviewed with semi-empirical (AM1 and PM3) calculations.[13]

Results and Discussion

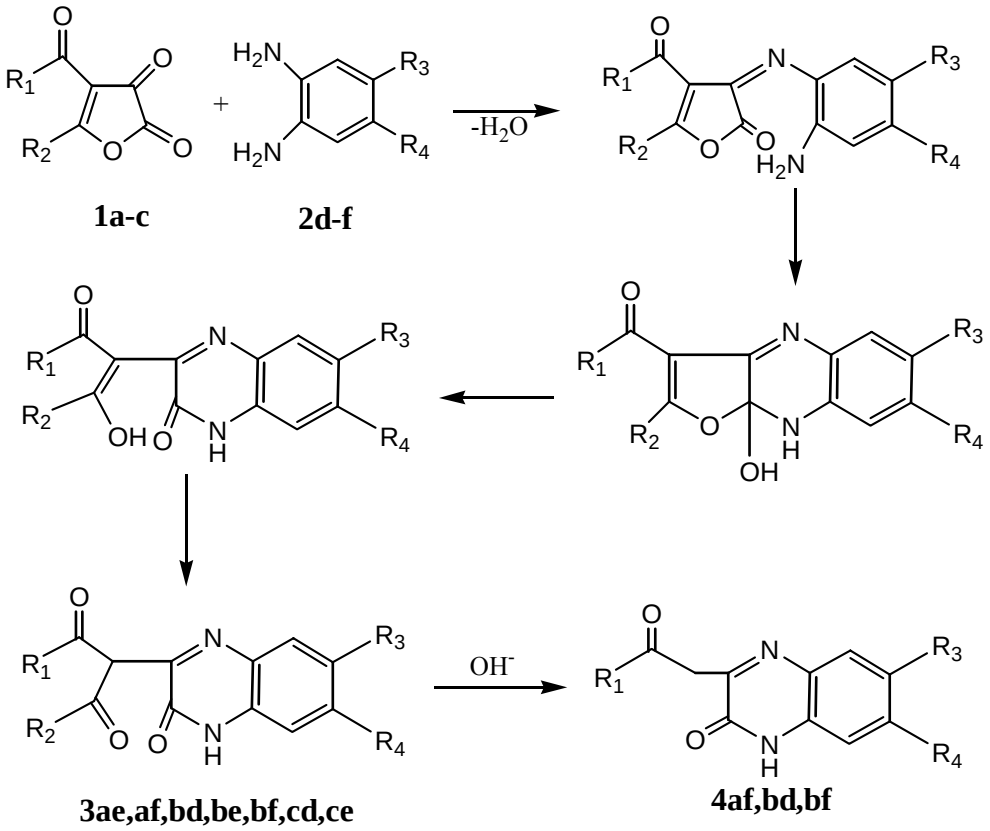
In compounds (**1a-c)** carbon atoms C-2, C-3 and C-5 represent electrophilic sites of different reactivity and could be used for the construction reaction with nucleophiles.[11f,13a,14] The reactions of the furan-2,3-diones **(1a-c)** with 1,2-phenylenediamine derivatives **(2d-f)** were carried out and corresponding 2(1*H*)-quinoxalinone derivatives, 1,3-diaryl-2-(3-oxo-1,2-dihydro-2-quinoxalinyl)-1,3-propanediones **(3ad,af, bd,be,bf)** and ethyl 3-aryl-3-oxo-2-(2-oxo-1,2-dihydro-3-quinoxalinyl)propanoate **(3cd,ce)**, in which the binucleophlic attack of **2d,e,f** occurred on C-3 followed by attack on C-2 of **1a,b,c**, with opening of the lactone ring,[15] were obtained, as shown in **Scheme .**

Condensation of **1b,c** with *1,2*-phenylenediamine **(2d)** by stirring in benzene at room temperature for 12 h or 24 h gave 2(1*H*)-quinoxalinone **(3bd)** (25% yield) and **(3cd)** (52% yield), and similar condensation of **1a-c** with 4,5-dimethyl-*1,2*-phenylenediamine **(2e)** for 24 h gave 2(1*H*)-quinoxalinone **(3ae)** (37% yield), **(3be)** (35% yield), and (**3ce)** (48% yield). Similar condensation of **1a,b** with 4-nitro-*1,2*-phenylenediamine **(2f)** in benzene at room temperature for 18 or 24 h gave 2(1*H*)-quinoxalinone **(3af)** (75% yield) and **(3bf)** (80% yield). The lower yields in **3bd,cd,ae,be,ce** may be explained by the lower nucleophilicity of the amine of the *1,2*-phenylendiamines **(2d,e)**. Product **(3ae)** was obtained by treating **1a** with 4,5-dimethyl-*1,2*-phenylendiamine **(2e)**. In the IR spectra of compound **(3ae)**, the -NH absorption band was found to be at about 3450-3300 cm^{-1}. The quinoxaline C=O and benzoyl C=O absorptions were at 1658 and 1620 cm^{-1}, respectively. The ^1H NMR signals were found to be at 12.58 (br, -NH), 8.26-7.11 (m, 12H, ArH), 6.11 (s, C-H), 2.28 and 2.23 ppm (s, $2CH_3$) and the ^{13}C NMR signals were at δ 195.91 (t, PhCO), 157.84 (s, lactam C), 155.61 (s, C-3), 21.52 and 20.46 ppm ($2CH_3$) and elemental analytical data (see EXPERİMENTAL) confirm the structure of **3ae**.

The alkaline treatment of 3-(1-benzoyl-2-hydroxy-2-phenylethenyl)-2-oxo-1,2-dihydroquinoxaline, by the removal of the benzoyl group, results in 2-oxo-3-phenacylidene-1,2,3,4-tetrahydroquinoxaline. [15] Similar to the alkaline treatment of 1,3-propanediones **(3af**, **3bd** and **3bf)** gave corresponding 3-aroylmethly-2(1*H*)-quinoxalinones (**4af,bd,bf)** (see Scheme). The alkaline cleavage of 1,3-diketones **(3af**,**3bd**,**3bf)** with 2N NaOH by refluxing in ethanol for 2 h gave 2(1*H*)-quinoxalinone **(4af)** (52% yield), **(4bd)** (70% yield) and **(4bf)** (57% yield). In the IR spectra of compound **(4af)**, the –NH absorption band was found to be at about 3300-3100 cm^{-1}. The quinoxaline C=O and benzoyl C=O absorptions were at 1693 and 1616 cm^{-1}, respectively. The ^1H NMR signals were found to be at 12.35 (br, -NH); 8.19-7.15 (m, 8H, ArH), 6.85 ppm (s, C-H), and the ^{13}C NMR signals were at δ 191.47 (t, ArCO), 157.36 (s, lactam C),

144.47 (s, C-3), 143.01-112.05 ppm (m, aromatic C) and elemental analysis data (see experimental EXPERIMENTAL) confirm the structure of **4af**.

Scheme



a) R_1=R_2=Ph, **b)** R_1=R_2=*p*-$MeOC_6H_4$, **c)** R_1=Ph, R_2=OEt
d) R_3=R_4=H, **e)** R_3=R_4=Me, **f)** R_3=NO_2, R_4=H

EXPERIMENTAL

Solvents were dried by refluxing with the appropriate drying agent and distilled before use. Melting points were determined on the Electrothermal 9200 apparatus and are uncorrected. Microanalyses were performed on a Carlo Erba elemental analyser, Model 1108. The IR spectra were recorded on a Shimadzu Model 435 V-04 spectrophotometer in potassium bromide discs. The ^{1}H and ^{13}C NMR spectra were recorded on a Gemini-Varian 200 instrument in DMSO-d_6 solutions. The chemical shifts are reported in ppm from tetramethylsilane and given in δ units.

General Condensation Procedure of 2,3-Furandiones (1a-c) with 1,2-Phenylenediamine (2d-f).

A benzene solution (30 mL) of 2,3-furandione **(1a-c)** (1.5-2.0 mmol) and 1.02 equivalent of 1,2-phenylenediamine **(2d-f)** was stirred at 25°C for 12 h to **3bd**, 18 h to **3af**, 24 h to **3ae,be,bf,cd,ce** or

refluxed for 1 h to **3cd,ce**. The white crystaline product was filtered and washed through ethyl acetate and recrystallized from ethanol and allowed to dry on P_2O_5.

2-(6,7-Dimethyl-2-oxo-1,2-dihydroquinoxalin-3-yl)-1,3-diphenylpropane-1,3-dione (3ae).

Yield 0.26 g (37%); mp 274-276°C; IR (KBr): 3450-3300 (NH), 1658 (C=O), 1620 cm^{-1} (C=O); ^1H NMR (DMSO-d_6): δ= 12.58 (br, NH), 8.26-7.11 (m, 12H, ArH), 6.11 (s, C-H), 2.28 and 2.23 (CH_3); ^{13}C NMR (DMSO-d_6): δ= 195.91 (t, PhCO), 157.84 (s, lactam C), 155.61 (s, C-3), 142.07-117.32 (m, Aromatic C), 21.52 and 20.46 (CH_3). Anal. Calcd for $C_{25}H_{20}N_2O_3$: C, 75.74; H, 5.08; N, 7.07. Found: C, 75.55; H, 4.96; N, 7.21.

2-(7-Nitro-2-oxo-1,2-dihydroquinoxalin-3-yl)-1,3-diphenylpropane-1,3-dione (3af).

Yield 0.555 g (75%); mp 280-283°C; IR (KBr): 3400-3230 (NH), 1680 (C=O), 1630 cm^{-1} (C=O); ^1H NMR (DMSO-d_6): δ= 12.06 (NH), 8.55-7.19 (m, 13H, ArH), 5.40 (s, C-H); ^{13}C NMR (DMSO-d_6): δ= 195.62 (t, ArCO), 157.02 (s, lactam C), 146.21 (s, C-3), 145.01 (C-NO_2), 144.00-112.00 (m, aromatic C). Anal. Calcd for $C_{23}H_{15}N_3O_5$: C, 66.83; H, 3.66; N, 10.16. Found: C, 66.62; H, 3.38; N, 9.88.

2-(2-Oxo-1,2-dihydroquinoxalin-3-yl)-1,3-di(4-methoxyphenyl)propane-1,3-dione (3bd).

Yield 0.25 g (25%); mp 208°C; IR (KBr): 3300-3250 (NH), 1690 (C=O), 1610 cm^{-1} (C=O); ^1H NMR (DMSO-d_6): δ= 11.54 (br, NH), 7.99-7.01 (m, 13H, ArH), 6.84 (s, C-H), 3.84 and 3.72 (s, 6H, CH_3O); ^{13}C NMR (DMSO-d_6): δ= 192.01 (t, ArCO), 157.00 (s, lactam C), 154.00 (s, C-3), 132.00-113.00 (m, Aromatic C), 55.01 and 54.65 (q, $2CH_3O$). Anal. Calcd for $C_{25}H_{20}N_2O_5$: C, 70.08; H, 4.70; N, 6.54. Found: C, 70.01; H, 4.66; N, 6.24.

2-(6,7-Dimethyl-2-oxo-1,2-dihydroquinoxalin-3-yl)-1,3-di(4-methoxyphenyl)propane-1,3-dione (3be).

Yield 0.234 g (35%); mp 262-264°C; IR (KBr): 3450-3250 (NH), 1648 (C=O), 1600 cm^{-1} (C=O); ^1H NMR (DMSO-d_6): δ= 12.25 (br, NH), 7.96-7.01 (m, 10H, ArH), 6.24 (s, C-H), 3.95 and 3.67 (s, 6H, $2CH_3O$), 2.28 and 2.16 (CH_3); ^{13}C NMR (DMSO-d_6): δ 194.26 (t, ArCO), 158.01 (s, lactam C), 155.65 (s, C-3), 60.50 and 57.24 (q, $2CH_3O$), 20.55 and 20.44 (s, $2CH_3$). Anal. Calcd for $C_{27}H_{24}N_2O_5$: C, 71.04; H, 5.30; N, 6.14. Found: C, 71.28; H, 5.58; N, 5.98.

2-(7-Nitro-2-oxo-1,2-dihydroquinoxalin-3-yl)-1,3-di(4-methoxyphenyl)propane-1,3-dione (3bf).

Yield 0.56 g (80%); mp 288-290°C; IR (KBr): 3433-3208 (NH), 1700 (C=O), 1567 cm^{-1} (C=O); ^1H NMR (DMSO-d_6): δ= 11.99 (br, NH), 8.36-6.78 (m, 11H, ArH), 3.83 and 3.73 (s, 6H, $2CH_3O$); ^{13}C NMR

(DMSO-d_6): δ= 194.21 (t, ArCO), 165.37 (s, lactam C), 157.03 (s, C-3), 138.91 (s, C-NO_2), 146.00-112 (m, aromatic C), 60.15 and 57.05 (q, 2CH_3O). Anal. Calcd for $C_{25}H_{19}N_3O_7$: C, 63.42; H, 4.05; N, 8.88. Found: C, 63.40; H, 4.13; N, 8.65.

Ethyl 3-oxo-2-(2-oxo-1,2-dihydroquinoxalin-3-yl)-3-phenylpropanoate (3cd).

Yield 0.35 g (52%); mp 164-166°C; IR (KBr): 3300 (NH), 1680 (C=O), 1655 cm^{-1} (C=O); ^{1}H NMR (DMSO-d_6): δ= 11.85 (br, NH), 7.51-7.21 (m, 10H, ArH), 7.05 (s, C-H), 4.21 (q, O-CH_2), 1.35 and 1.19 (s, 2CH_3); ^{13}C NMR (DMSO-d_6): δ= 194.28 (t, ArCO), 169.40 (s, lactam C), 160.32 (s, C-3), 144.06-116.72 (m, aromatic C), 59.63 (-CH_2), 21.52 and 20.50 (s, 2CH_3). Anal. Calcd for $C_{19}H_{16}N_2O_4$; C, 67.85; H, 4.79; N, 8.33. Found; C, 67.62; H, 4.55; N, 8.14.

Ethyl 2-(6,7-dimethyl-2-oxo-1,2-dihydroquinoxalin-3-yl)-3-oxo-3-phenylpropanoate (3ce).

Yield 0.21 g (48%); mp 184-186°C; IR (KBr): 3400-3250 (NH), 1680 (C=O), 1654 cm^{-1} (C=O); ^{1}H NMR (DMSO-d_6): δ= 12.35 (br, NH), 7.98-7.06 (m, 8H, ArH), 4.11 (q, O-CH_2) 1.89 and 1.02 (s, 2CH_3); ^{13}C NMR (DMSO-d_6): δ= 194.35 (t, ArCO), 160.36 (s, lactam C), 155.42 (s, C-3), 142.25-117.36 (m, aromatic C), 62.44 (-CH_2), 21.52 and 20.50 (s, 2CH_3). Anal. Calcd for $C_{21}H_{20}N_2O_4$; C, 69.22; H, 5.53; N, 7.69. Found; C, 68.96; H, 5.61; N, 7.88.

General Procedure for Alkaline Cleavage of 1,3-Diketones (3af,bd,bf).

The ethanolic solution (25 mL) of diketone **(3af,bd,bf)** (0.2 g, 0.35-0.5 mmol) and 2N NaOH (0.1 mL) was refluxed for 2 h. The solution was cooled and it was acidified to pH 4 with 2N HCl. The crude crystaline product was filtered and recrystallized from butanol and allowed to dry on P_2O_5.

7-Nitro-3-(2-oxo-2-phenylethyl)quinoxalin-2(1*H*)-one (4af).

Yield 0.078 g (52 %); mp 335-338 °C; IR (KBr): 3300-3100 (NH), 1693 (C=O), 1616 cm^{-1} (C=O); ^{1}H NMR (DMSO-d_6): δ= 12.35 (br, NH), 8.19-7.15 (m, 9H, ArH), 6.85 (s, C-H); ^{13}C NMR (DMSO-d_6): δ= 191.47 (t, ArCO), 157.36 (s, lactam C), 144.47 (s, C-3), 143.01-112.05 (m, aromatic C). Anal. Calcd for $C_{16}H_{11}N_3O_4$: C, 62.14; H, 3.58; N, 13.58. Found: C, 61.79; H, 3.39; N, 13.21.

3-[2-(4-Methoxyphenyl)-2-oxoethyl]quinoxalin-2(1*H*)-one (4bd).

Yield 0.07 g (70%,); mp 249 °C; IR (KBr): 3500-3300 (NH), 1675 (C=O), 1600 cm^{-1} (C=O); ^{1}H NMR (DMSO-d_6): δ= 11.90 (NH), 8.01-7.01 (m, 9H, ArH), 6.85 (s, C-H), 3.8 (s, CH_3O); ^{13}C NMR (DMSO-d_6): δ 188.01 (t, ArCO), 156.01 (s, lactam C), 145.07 (s, C-3), 131.01-114.01 (m, aromatic C), 57.35 (s, CH_3O). Anal. Calcd for $C_{17}H_{14}N_2O_3$: C, 69.38; H, 4.79; N, 9.52. Found: C, 69.41; H, 4.72; N, 9.35.

3-[2-(4-Methoxyphenyl)-2-oxoethyl]-7-nitroquinoxalin-2(1*H*)-one (4bf).

Yield 0.08 g (57%); mp 328-331 ^{o}C; IR (KBr): 3200-3140 (NH), 1695 (C=O), 1620 cm^{-1} (C=O); ^{1}H NMR (DMSO-d_6) : δ= 11.99 (NH), 7.98-7.02 (m, 8H, ArH), 6.87 (s, C-H), 3.86 (s, CH_3O); ^{13}C NMR (DMSO-d_6): δ= 190.42 (t, ArCO), 157.51 (s, lactam C), 144.83 (s, C-3), 144.01-112.05 (m, aromatic C), 58.66 (s, CH_3O). Anal. Calcd for $C_{17}H_{13}N_3O_5$; C, 60.18; H, 3.86; N, 12.38. Found; C, 60.39; H, 3.90; N, 12.00.

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