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Experimental and Theoretical Studies on Some New Pyrrol-2,3-diones Formation

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ABSTRACT: *4-Benzoyl-5-phenyl-2,3-furandione (***1***) reacts with asymmetric disubstituted urea derivatives like 1,1-dimethylurea (***2a***) and 1,1-diethylurea (***2b***) by the elimination of a H_2O molecule to give the 4-benzoyl-1-(N,N-dialkylcarbamyl)-5-phenyl-2,3-pyrroldiones* **3a** *and* **3b**. *The structures of* **3a,b** *were determined by the ^{13}C NMR, ^1H NMR, IR spectroscopic data and elemental analyses. The electronic structures of the reactants, their transition states, intermediate states, and final products of the reactions were investigated on the basis of AM1 and ab initio (DFT) methods.* © 2003 Wiley Periodicals, Inc. Heteroatom Chem 15:9–14, 2004; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.10204

INTRODUCTION

A convenient synthesis of functionalized 1*H*-pyrimidine-2-ones from 4-benzoyl-5-phenyl-2,3-furandione and some semicarbazones, thiosemicarbazones, monosubstituted ureas and thioureas have been reported previously [1]. To gain more insight into the chemical behavior of five-membered heterocyclic 2,3-diones against NH-nucleophiles has been attempted [2]. 4-Benzoyl-5-phenyl-2,3-furandione (**1**) appeared to be an important starting compound in synthetic organic chemistry [3]. The reactions of the substituted furan-2,3-diones and pyrrole-2,3-diones with various nucleophiles and dienophiles have been studied at different conditions as well [4].

In this paper, the investigations were extended to the reactions of **1** with asymmetric disubstituted urea derivatives **2a,b**. Thus, as seen from Fig. 1, the compounds 4-benzoy1-1-(*N*,*N*-dimethyl/diethylcarbamyl)-5-phenyl-2,3-pyrroldione (**3a,b**) were synthesized from the cyclocondensation reactions of *N*,*N*-dimethyl urea (**2a**) and *N*,*N*-diethylurea (**2b**), respectively, with **1**. Quantum-chemical calculations were carried out, aiming in the study of electron structure of reagents, products, and transition and intermediate states, as well. The results were used in the mechanisms of the reaction discussion.

The compounds **3a,b** have been first synthesized from these reactions, and the results obtained are discussed in this study.

EXPERIMENTAL AND CALCULATIONS

Melting points (uncorrected): Büchi 510 and Electrothermal 9200 melting points apparatus (m.p. > 360°C). IR: Perkin-Elmer 421 and Shimadzu 435 V-04; KBr, pellet. ^{1}H NMR: Varian EM 360L; TMS internal reference, ppm units. ^{13}C NMR: Varian XL 200 Gemini; TMS. Combustion analyses: Carlo-Erba 1108 of Hewlett Packard, CHNS model 105 Analysator. Rotary evaporator: Büchi RE 111. TLC: Alufolien Kieselgel G 60 F 254 Merck; Camag TLC lamp (254/366 nm). Solvents and other chemical reagents were from Merck, Sigma, Aldrich, and

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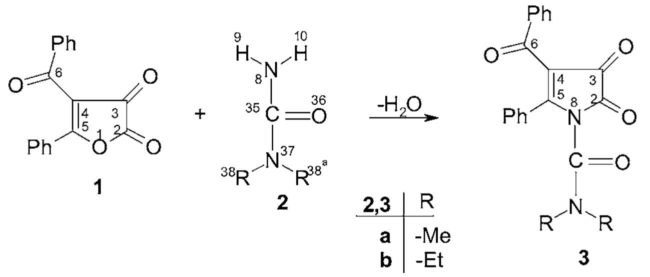

FIGURE 1 General scheme of the reaction investigated.

Fluka. Solvents were dried by refluxing with the appropriate drying agent and distilled before use.

*4-Benzoyl-5-pheny-2,3-furandione (**1**)*

For the preparation of **1**, see Ref. [3c].

*4-Benzoyl-1-(N,N-dimethylcarbamyl)-5-phenyl-2,3-pyrroldione (**3a**)*

1 (0.50 g, 1.80 mmol) and **2a** (0.16 g, 1.80 mmol) were mixed in 30 ml distilled benzene in a one-necked pear-shaped flask at room temperature and then refluxed for 4.5 h. After evaporation of the solvent under reduced pressure, the oily residue was stirred with anhydrous ether. The precipitated crude red product was separated of ether by filtering and recrystallized from toluene to give product **3a** (0.43 g, 68%) as a red powder. m.p. 188–189°C. IR (KBr): $\nu = 3100$–$2900\ \text{cm}^{-1}$ (w, aromatic and aliphatic C—H stretching, respectively), 1760 (s), 1710 (s), 1655 (s, C=O), 1600–1480 (m, C—C, aromatic rings). ^{13}C NMR (DMSO): $\delta = 187.13$ (t, C6, benzoyl), 180.25 (s, C3), 162.81 (s, C2), 160.94 (s, C35, —NCON—), 151.00 (t, C5), 139.93–128.58 (m, C-Ph's, aromatic C), 117.65 (s, C4), 37.98, 37.74 (C38, C38^a, exchangeable, N—CH$_3$). $C_{20}H_{16}N_2O_4$ (348.36): calcd C 68.96, H 4.63, N 8.04; found C 69.02, H 4.83, N 7.85.

*4-Benzoyl-1-(N,N-diethylcarbamyl)-5-phenyl-2,3-pyrroldione (**3b**)*

1 (0.50 g) and **2b** (0.21 g) (molar ratio 1:1) were mixed in benzene and refluxed for 3.5 h. The solvent was evaporated and the oily residue was washed with anhydrous ether by stirring. After filtering, the pale red product was recrystallized from toluene to give product **3b** (0.24 g, 36%). m.p. 189°C. IR (KBr): $\nu = 3100$–$2900\ \text{cm}^{-1}$ (w, aromatic and aliphatic C—H stretching, respectively), 1765 (s), 1705 (s), 1660 (s, C=O stretching), 1600–1460 (m, w, C—C aromatic rings). ^{1}H NMR (60 MHz, CDCl$_3$): $\delta = 8.0$–7.2 (m, 10H, aromatic H), 3.6 (q, 4H, aliphatic H, —CH$_2$—N—CH$_2$—), 1.8–0.9 (t, 6H, —CH$_3$). $C_{22}H_{20}N_2O_4$ (376.42): calcd C 70.20, H 5.36, N 7.44; found C 70.21, H 5.32, N 7.45.

Computational Methodology

To study the mechanism of the reaction, quantum chemical calculations were carried out by means of AM1 and ab initio (DFT) methods with full geometry optimization for reactants, intermediates, and final products. Vibrational analysis was performed for all calculated molecular systems. For transition states (TS), negative imaginary frequencies were found. Usually, one imaginary frequency is characteristic of an ordinary TS; these TSs are first-order saddle points. A more accurate way to determine what reactants and products the TS connects, is to perform an intrinsic reaction coordinate (IRC) calculation to follow the reaction path and thereby determine the reactants and products explicitly. The cycloreversion rather than the cycloaddition reaction was used, keeping the length of one of the breaking bonds constant while varying the other one. All other parameters were optimized, and stationary points were refined by the gradient norm minimization [5]. So, in each stationary point the system was characterized by the corresponding Hessian (force constant) matrix, calculated, and diagonalized. The standard parameters [6] were used in the AM1 calculations. The ab initio (B3LYP/6-31+G**) calculations were carried out by means of the GAUSSIAN 98W program package (Version 5.4 Rev A.9) [7]. The Becke's three-parameter hybrid functional using the LYP (Lee, Yang, and Parr [8]) correlation functional was taken for the B3LYP calculations.

RESULTS AND DISCUSSION

Figure 1 shows the synthesis of **3a,b**. There are four different carbonyl groups in each compound, and each of them has different electronic environment. So, the IR spectra are excellent evidences of their structures. The carbonyl groups at position 3 in pyrrole ring have high electron density. Thus, the absorption bands observed at 1760 and 1765 cm^{-1} for **3a** and **3b** must belong to these carbonyl groups, respectively [9]. The absorption bands observed at 1655 and 1660 cm^{-1} for **3a** and **3b** belong to benzoyl carbonyl groups [10]. Bonds for carbonyl groups at positions 2 and 35 have been observed at 1710 and 1705 cm^{-1} for **3a** and **3b**, respectively. The bond force constants of these carbonyl groups are smaller than that on the C3 atoms. Therefore, the absorption bands of these carbonyl groups shift to the

TABLE 1 Energy Characteristics of the Systems Participating in the Reaction (Calculated by AM1)

Systems	*Energy (au)*	*Zero Point Energy (ZPE) (au)*	*Energy + ZPE (au)*	*Imaginary Frequency for TS (cm^{-1})*
1 + 2	−0.1294515 (−1251.68)[a]	0.356367	0.226915	
TS1	−0.0921820	0.359584	0.267783	−452
I3a	−0.0976914 (−1251.84)	0.360652	0.262961	
TS2	−0.0479365	0.355582	0.319066	−419
I3b	−0.1508560 (−1251.65)	0.357643	0.206787	
TS3	−0.0680810	0.351050	0.283021	−17
3	−0.1261220 (−1251.66)	0.354134	0.228009	

[a] B3LYB energy.

lower frequency area. The structures of the reaction products were identified from analytical and spectroscopy (NMR, IR) data.

Since experimental results were insufficient to explain the mechanism of the reactions, quantum chemical calculations were used to this aim. Their results enabled us to suggest that reaction of **1** with **2** proceeded through some TSs with intermediates formation, and a substantial role in the analysis of the paths of reactions belongs to the interaction of frontier HOMO (LUMO) orbitals of reactants. The calculations have shown that in the reacting system HOMO is represented by the atoms N8, N37, and O36 belonging to urea while LUMO includes the atoms C5, C2, and O1 with the greatest values of their coefficients. The frontier orbitals localization on the reacting center forces the orbitals' energy levels to approach each other. There through, the orbitals' interaction increases. It may be suggested that it is precisely the HOMO–LUMO and their constituent atoms C5 and N8 interaction that is the first stage of the reaction.

Energy characteristics of systems participating in the reaction are given in Table 1, while geometries of the reacting centre and charge distribution on atoms are given in Table 2. The atoms spatial arrangements in intermediate (I) and transition states (TS) are shown in Fig. 2. According to Fig. 1, the mechanism of the reaction as a whole may be divided into three stages. At the first stage the urea

TABLE 2 Bond Lengths and Atomic Charges for the Molecules Being Reaction Participants

	1 + 2		**TS1**	**I3a**		**TS2**	**I3b**		**TS3**	**3**	
	AM1	*DFT*	*AM1*	*AM1*	*DFT*	*AM1*	*AM1*	*DFT*	*AM1*	*AM1*	*DFT*
Bond lengths (Å)											
N8–H10	0.99	1.02	1.02	1.02	1.08	1.03	6.23	1.08	2.80	4.20	4.95
N8–H9	0.99	1.01	1.02	1.03	1.02	1.03	1.00	1.02	2.94	4.33	4.83
C5–N8	5.31	5.52	1.74	1.57	1.53	1.56	1.38	1.45	1.31	1.42	1.43
C4–C5	1.38	1.38	1.46	1.50	1.51	1.42	1.38	1.35	1.45	1.39	1.36
C5–O1	1.41	1.41	1.43	1.45	1.45	1.73	4.54	4.02	3.63	5.73	5.60
C3–C4	1.48	1.45	1.43	1.43	1.42	1.45	1.47	1.51	1.39	1.47	1.46
C2–C3	1.53	1.55	1.53	1.53	1.55	1.53	1.51	1.53	1.52	1.55	1.55
O1–C2	1.41	1.42	1.41	1.40	1.42	1.36	1.36	1.38	2.27	3.67	3.77
O1–H10	6.42	6.01	2.42	2.33	3.21	2.32	0.97	1.00	0.96	0.96	1.01
C2–N8	6.96	7.41	3.33	3.35	3.55	2.99	4.57	2.69	2.50	1.42	1.39
O1–H9	5.77	7.68	3.23	3.19	3.21	2.40	5.59	3.32	0.96	0.96	0.99
C4–C22	1.47	1.49	1.44	1.43	1.42	1.45	1.47	1.51	1.48	1.47	1.48
Atomic charges (Mulliken populations)											
C5	0.25	0.41	0.38	0.33	0.41	0.35	0.29	0.27	0.26	0.27	0.41
O1	−0.25	−0.54	−0.30	−0.32	−0.50	−0.38	−0.36	−0.53	−0.48	−0.47	−0.65
C2	0.29	0.66	0.28	0.29	0.63	0.27	0.31	0.63	0.44	0.33	0.70
C4	−0.38	−0.09	−0.59	−0.60	−0.07	−0.61	−0.40	−0.09	−0.43	−0.36	−0.09
H10	0.28	0.31	0.29	0.31	0.34	0.32	0.29	0.34	0.26	0.23	0.35
H9	0.28	0.35	0.32	0.35	0.38	0.30	0.34	0.37	0.26	0.23	0.333
N8	−0.55	−0.81	−0.36	−0.30	−0.68	−0.29	−0.47	−0.80	−0.44	−0.44	−0.83
C22	0.41		0.41	0.41		0.44	0.38		0.41	0.40	0.38

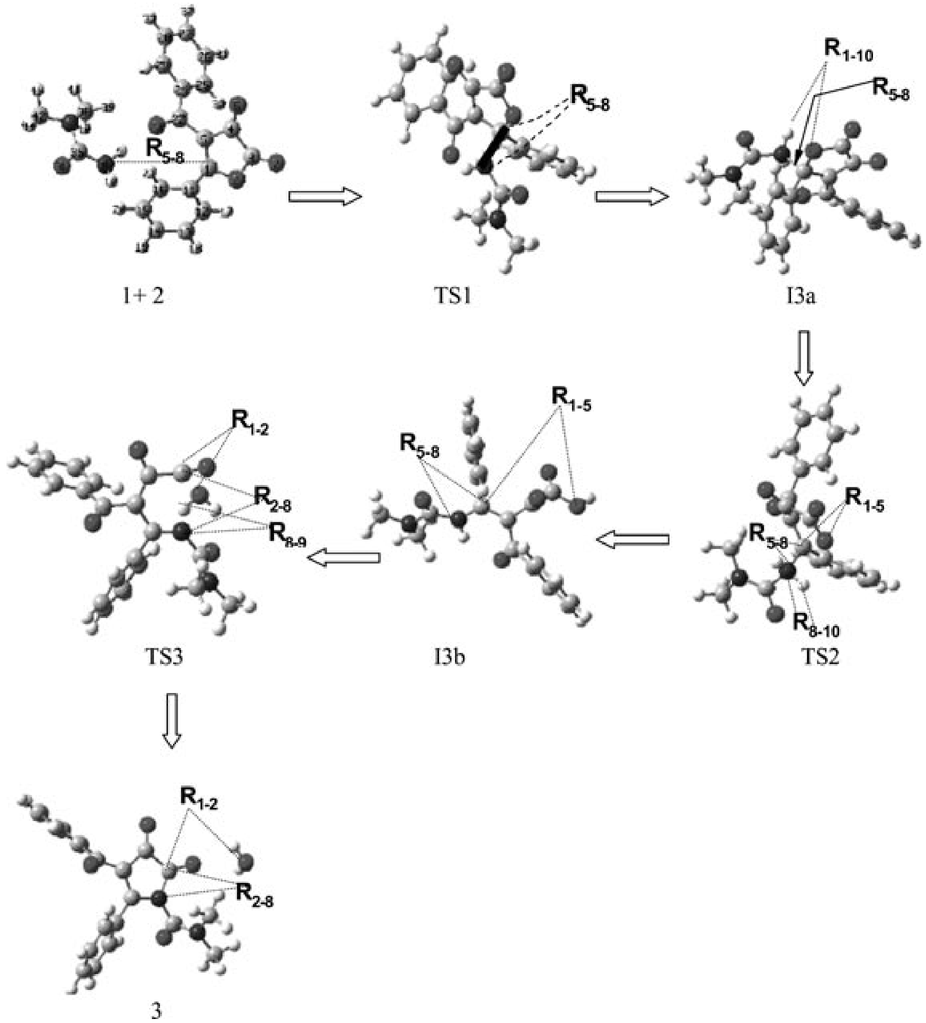

FIGURE 2 Optimized geometries of the molecular systems studied.

molecule (i.e. its atom N8) reacts with the atom C5 in furandione. Experimental data tell in favor of the fact [1a,2c,3b,4a–c,11].

The reacting molecules (**1** + **2**) being far from each other (C5—N8 = 5.31 Å) have a summary energy (as a system) of −0.1295 a.u. By this, the bond C5—N8 formation and the proton H10 attachment to O1 (see Fig. 2) may follow the synchronous concerted path or the step-by-step reaction. Both processes mentioned occur simultaneously. The results of electronic energy calculations for the TSs have shown that the step-by-step mechanism is more profitable, as to the activation energy. The step-by-step activation energy of the reaction is 23 kcal mol^{-1} and is lower than that of the synchronous one. The reaction path with a lower value of the activation energy is considered in detail below. The C5—N8 bond is chosen as the reaction coordinate at the stage Q1. The energy profile of the reactions is shown in Fig. 3.

The TS parameters are calculated by means of AM1 method. When the distance C5—N8 is equal to 1.74 Å, the system passes into the transition state **TS1**. By this, bonds C5—O1 and C5—C4 lengthen.

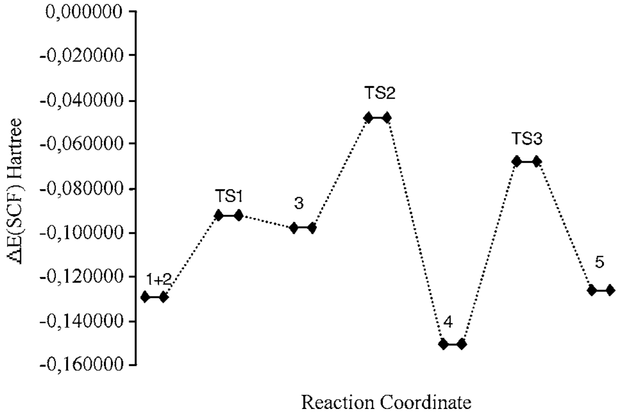

FIGURE 3 Potential energy profile for the reaction of **1** + **2**.

The atoms C5 and N8 further approach till the distance 1.57 Å transfers the system into a metastable state **I3a**. Its structure is such that the atom N8 is in a tetrahedral state, and carbon atom C5 and atoms from its nearest surrounding form a trigonal pyramid. By this, the electron density transfer from the nitrogen atom to the carbon atom happens, which causes the intermediate state **I3a** stabilization. It is seen from the charge redistribution on the reaction centre. The negative charge on the atom N8 (AM1) decreases from −0.55 to −0.30 e.u. ($\bar{e}$). Simultaneously, both the positive charge on the C5 atom and the negative charges on atoms O1 and C4 increase. In this way, a substantial polarization of bonds formed by the atom C5 can be observed. The values of bonds polarization, calculated by means of B3LYP, are higher than those obtained from the AM1 method. The C5—O1 and C5—C4 bonds demonstrate insignificant lengthening for both methods. It should be emphasized that one of hydrogen atoms (H10) attached to the nitrogen atom N8 comes close enough to the oxygen atom O1 (H10···O1 = 2.33 Å). This fact plays an important role for the discussion of the next reaction step.

The system transition to the intermediate state **I3b** happens under the bond C5···O1 full breakage and proton H10 transfer to the O1 atom. The system transition to the state **I3b** happens through the **TS2** formation. Imaginary frequency is $-419\ \text{cm}^{-1}$. For this, state is characterized by the further bonds polarization in the reaction center, which is seen from the increase of both positive and negative charges on its atoms (C5, O1, N8, and H10).

A high negative charge is concentrated on the atoms O1 ($q_{O2} = -0.38\bar{e}$) and C4 ($q_{C5} = -0.61\bar{e}$). Meanwhile, a high positive charge is concentrated on the atom H10 ($q_{H10} = +0.32\bar{e}$). The difference in the energy level for the state **I3b** in comparison with the state **I3a** in the system is 33.36 kcal/mol. In **TS2**, the system has the greatest dipole moment equal to 11.70 D. The torsion angle C5—N8—C35—O36 deviates inconsiderably from the plane one, and its value is 106°. So, the system is ready to pass to the second stage of the reaction, that is, the proton H9 passes from the atom N8 to the atom O1. At this stage, the forming bond H9···O1 is chosen as the reaction coordinate (Q2). The transition state **TS3** is being formed when the N8···C2 distance becomes equal to 2.50 Å. The seven-membered ring formation is characteristic of this stage. A proton disconnected from N8 and adjusted to O1 transfers the system into the **TS3** transition state.

The third stage is a cyclocondensation reaction resulting in the final product **3** formation. In the transition state **TS3**, bond lengths are 2.50 Å for N8—C2, 0.96 Å for O1—H9, 2.27 Å for O1—C2, and 4.33 Å for N8—H9. The atoms N8 and C2 further leads to two old bonds (N8—H9 and O1—C2) simultaneous breakage and two new bonds (N8—C2 and O1—H9) formation. The torsion angle C4—C3—C2—N8 in the five-membered cycle is 9.78°, while in the four-membered ring the angle C2—N8—H9—O1 is equal to 8.85°. This indicates that the structure of the atoms forming the transition state **TS3** is a planar one. In the final product **3** the angle N8—C2—C3—C4 becomes more planar and equals to 3.02°.

CONCLUSION

4-Benzoyl-1-(*N*,*N*-dimethyl/diethycarbamyl)-5-phenyl-2,3-pyrroldiones **3a** and **3b** were synthesized from the cyclocondensation reactions. The absorption bands observed at 1760 and 1765 cm^{-1} for **3a** and **3b** belong to carbonyl groups with high electron density, attached to the position 3 in pyrrole ring; absorption bands observed at 1655 and 1660 cm^{-1} for **3a** and **3b**, correspondingly, belong to benzoyl carbonyl groups. Bonds for carbonyl groups at positions 2 and 35 have been observed at 1710 and 1705 cm^{-1} for **3a** and **3b**, respectively.

According to the calculation carried out, the reaction between **1** and **2** passes through a few stages. At the first stage, a molecule of urea combines with furandione that causes the transition of the proton H10 from N8 to O1. At the final stage, the bonds synchronous formation and breakage proceed till the final product **3** is obtained and a molecule of water is eliminated. For reagents **1** + **2**, the total of electronic and zero point energies is 0.226915 au. For intermediates **I3a** and **I3b**, the totals of electronic and zero point energies are equal to 0.262961 and 0.206787 au, respectively. The transition of **1** + **2** into **I3a, I3b**, and, finally, into **3** happens through transition states **TS1, TS2**, and **TS3**. The values of the activation barrier ΔE for **TS1, TS2**, and **TS3** are 0.04087, 0.05611, and 0.07623 au, respectively.

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