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Reaction of 5-Arylfuran-2(3*H*)-ones with Amines

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Abstract—5-(4-Chlorophenyl)- and 5-phenylfuran-2(3*H*)-ones reacted with guanidine carbonate at the methylene group in the unsaturated lactone molecule, leading to the formation of 4-(2-aryl-5-oxo-2,5-dihydrofuran-2-yl)-5-aryltetrahydrofuran-2-ones, while 5-(4-methylphenyl)furan-2(3*H*)-one under analogous conditions gave rise to *N*,*N*′-bis[4-(4-methylphenyl)-4-oxobutanoyl]guanidine. The reactions of 5-arylfuran-2(3*H*)-ones with thioacetamide afforded 4-aryl-*N*-{1-[5-aryl-2-oxo-2,3-dihydrofuran-3-ylidene]ethyl}-4-oxobutanamides. The corresponding *N*-(4-aryl-4-oxobutanoyl)thioureas were obtained by heating 5-arylfuran-2(3*H*)-ones with thiourea.

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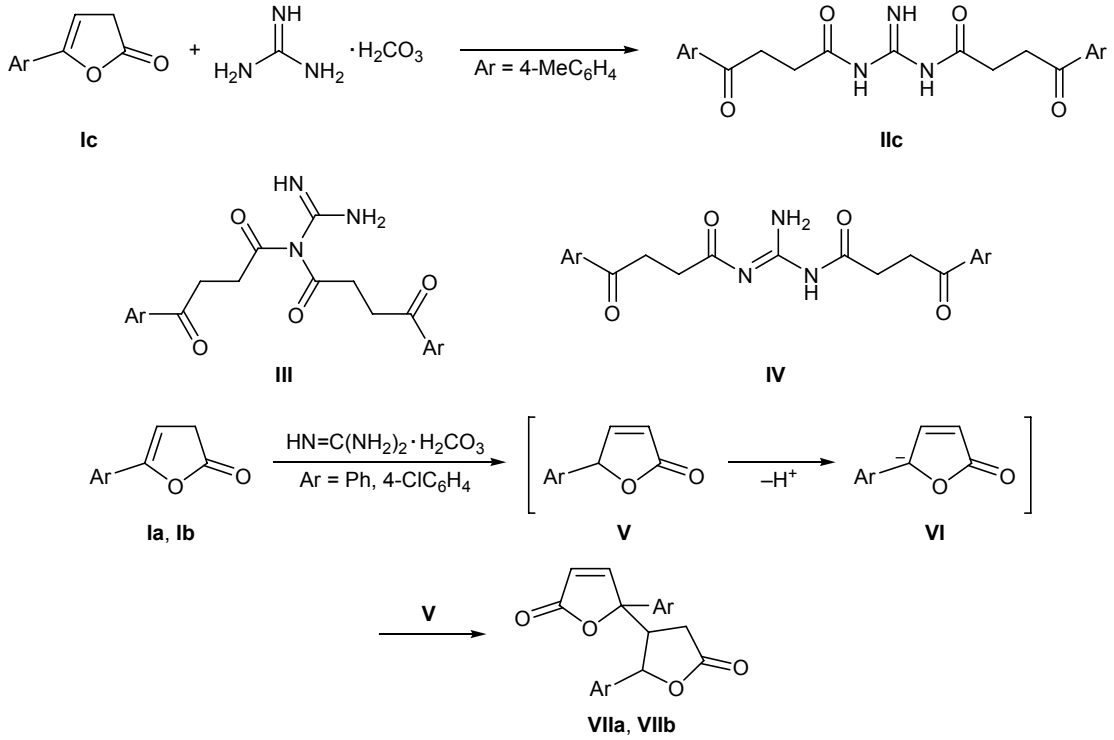
Various nitrogen-containing heterocyclic systems are often synthesized following an approach based on the reaction of equimolar amounts of difunctional electrophiles with amines. 5-Arylfuran-2(3*H*)-ones **I** are synthetic equivalents of 1,4-bielectrophiles. Although studies on the properties of these compounds have been initiated long ago by Biedermann [1] and Fittig [2] in the end of the XIXth century, known reactions of 5-arylfuran-2(3*H*)-ones **I** with amines are strongly limited; examples of their reactions with hydrazine derivatives and primary and secondary alkyl- and arylamines have been reported; in some cases, these reactions led to the formation of pyrrole [3–5] and pyridazine derivatives [6]. We now report on the reactions of 5-arylfuran-2(3*H*)-ones **Ia**–**Ic** with 1,3-binucleophiles, namely guanidine, thiourea, and thioacetamide, which were expected to produce nitrogen- and sulfur-and-nitrogen-containing heterocycles.

It is known that heating of 5-arylfuran-2(3*H*)-ones **I** with amines results in opening of the lactone ring and formation of the corresponding 4-aryl-4-oxobutanoic acid amides [2, 3, 5]. 5-(4-Methylphenyl)furan-2(3*H*)-one (**Ic**) reacted with guanidine carbonate according to a similar scheme: fusion of a mixture of the reactants at 120–130°C gave a 4-(4-methylphenyl)-4-oxobutanamide derivative. The product structure followed from its ^{1}H NMR spectrum which contained signals from methylene protons as two triplets at δ 2.70 and 3.22 ppm (^3J = 6.5 Hz) and NH proton signal at δ 9.33 ppm, br.s. This pattern is typical of 4-aryl-4-oxobutanoic acid derivatives [7]. In the IR spectrum of the product we observed ν_{NH} bands at 3350 and 3289 cm^{-1}. In keeping with the mass-spectral data, the product was diacyl-substituted guanidine (*m*/*z* 408 $[M + 1]^+$). The above spectral data are consistent with three isomeric structures: *N*,*N*′-bis[4-(4-methylphenyl)-4-oxobutanoyl]guanidine (**IIc**), *N*,*N*-bis[4-(4-methylphenyl)-4-oxobutanoyl]guanidine (**III**), and *N*,*N*″-bis[4-(4-methylphenyl)-4-oxobutanoyl]guanidine (**IV**) (Scheme 1). Taking into account that the ^{1}H NMR spectrum of the product contained only one set of signals from 4-aryl-4-oxobutanoyl fragment (i.e., the product has a symmetric structure) and that its IR spectrum lacked strong absorption bands at 1700 cm^{-1} and above (which could be assigned to imido group [8]), it was identified as isomer **IIc**.

The reactions of 5-arylfuran-2(3*H*)-ones **Ia** and **Ib** with guanidine carbonate under analogous conditions (120–130°C) followed a different path. According to the data of elemental analysis and mass spectrometry, the products were previously unknown bis-lactones. They displayed in the IR spectra absorption bands due to stretching vibrations of carbonyl groups in the region 1757–1773 cm^{-1}, while no strong absorption bands were present in the region 1665–1685 cm^{-1} typical of the Ar–C=O fragment in compounds resulting from opening of the lactone ring [7]; these findings indicated conservation of the cyclic lactone structure [9]. The ^{1}H NMR spectra of the isolated compounds, apart from signals belonging to protons in two non-

Scheme 1.



Ar = Ph (**a**), 4-ClC_6H_4 (**b**), 4-MeC_6H_4 (**c**).

equivalent phenyl groups, characteristically contained signals from olefinic protons as two doublets with a coupling constant *J* of 6 Hz and signals from aliphatic protons as an *ABMX* spin system. The spectral pattern suggests that the product molecule consists of two fragments of initial compound **I** linked to each other and partially reduced.

Examples of dimerization of furan-2(3*H*)-one derivatives on heating in acetic anhydride [10, 11], under irradiation [12], and in reactions with bases [13, 14] have been reported. We presumed that the reactions of lactones **Ia** and **Ib** with guanidine are examples of dimerization by the action of bases. According to the mechanism proposed in [13] for the dimerization of α-angelicalactone, β,γ-unsaturated lactone **I** on heating with a base (in our case, guanidine) is converted into α,β-unsaturated lactone **V**; proton abstraction from the sp^3-carbon atom in **V** gives anion **VI** which adds at the double bond of **V** with formation of the corresponding dimeric products, 5-aryl-4-(2-aryl-5-oxo-2,5-dihydrofuran-2-yl)tetrahydrofuran-2-ones **VIIa** and **VIIb** (Scheme 1).

To elucidate the structure of compounds **VII**, we examined the ^{13}C NMR spectrum of **VIIa**, as well as its two-dimensional COSY, HMQC, HMBC, and NOESY spectra. Figure 1 illustrates assignment of signals and structurally significant correlations (shown with arrows) in the HMBC (**A**) and NOESY spectra (**B**). Thus the ^1H signal at δ 3.52 ppm displayed numerous NOEs with protons in both furan rings and *ortho*-protons in the corresponding phenyl substituents. The same proton showed correlations in the HMBC spectrum with four quaternary carbon atoms (carbonyl carbon atom, δ_C 175.5 ppm, C^5, and carbon atoms in the two benzene rings), with the sp^3-hybridized carbon atom resonating at δ_C 91.2 ppm, and with sp^3-carbon atoms in the CH and CH_2 groups. These findings reliably indicated the presence of a 4-substituted 2-oxo-5-phenyltetrahydrofuran fragment in molecule **VIIa**. The presence of another structural fragment, 5-substituted 5-phenyl-2-oxo-2,5-dihydrofuran moiety, follows from the cross peaks between protons resonating at δ 6.40 and 8.18 ppm and carbonyl carbon atom (δ_C 172.0 ppm) and between the proton resonating at δ 8.18 ppm and quaternary carbon atoms (δ_C 91.2 and 137.6 ppm). Some specific features of the steric configuration of molecule **VIIa** were established on the basis of the NOESY data. The 4-H proton displayed coupling with *ortho* protons in the phenyl substituent on C^5. This means that the substituents on C^4 and C^5

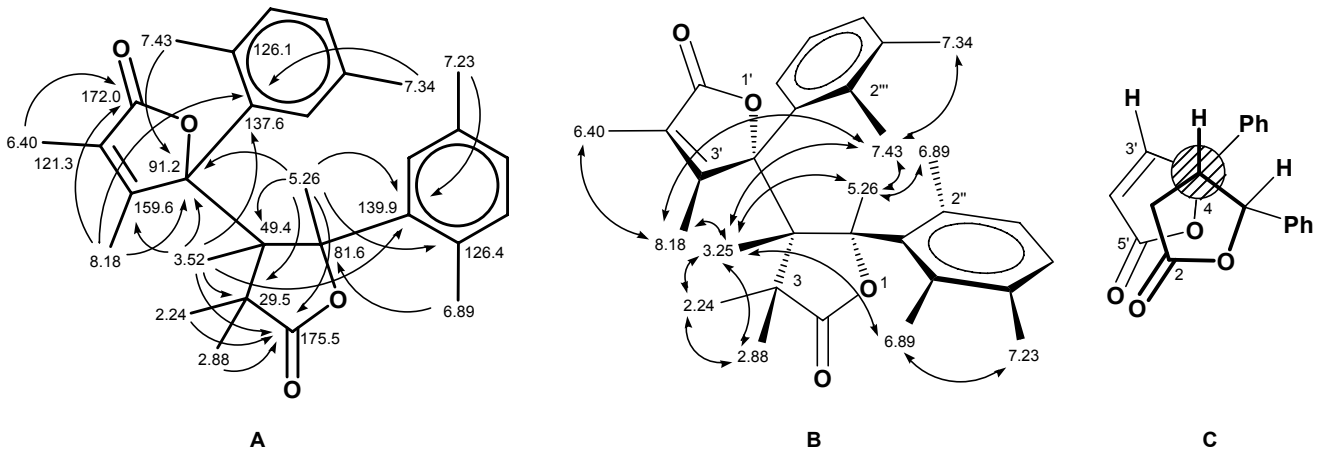


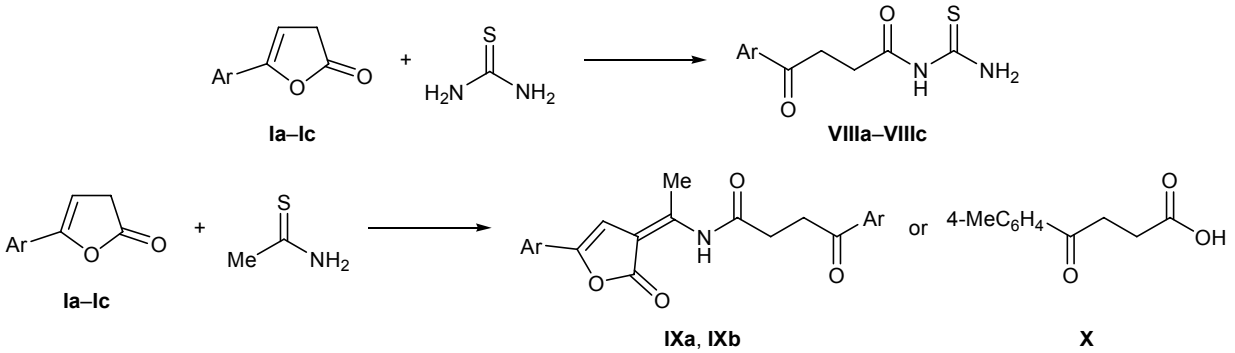
Fig. 1. Structurally significant correlations in the HMBC (**A**) and NOESY spectra (**B**) and the most populated conformation (**C**) of 4-(5-oxo-2-phenyl-2,5-dihydrofuran-2-yl)-5-phenyltetrahydrofuran-2-one (**VIIa**).

are oriented *trans*. The 4-H atom also appears spatially close to the 3′-H proton and *ortho* protons in the benzene ring attached to $C^{2'}$, and the latter protons exhibit NOE due to coupling with 5-H, so that the most populated conformation of molecule **VIIa** may be illustrated by structure **C** (Fig. 1).

Lactones **Ia**–**Ic** reacted with thiourea at a higher temperature (140–150°C) than in the reaction with guanidine, and the products were the corresponding *N*-(4-aryl-4-oxobutanoyl)thioureas **VIIIa**–**VIIIc** (Scheme 2). Rotation about formally single bonds in the H_2N–CS–NH–CO– fragment is likely to be restricted due to formation of intramolecular hydrogen bond. As a result, protons in the NH_2 group become nonequivalent, and they appear in the ^{1}H NMR spectra as differently broadened signals at δ 9.20 and 9.58 ppm. The NH stretching vibration frequency in the IR spectra of **VIIIa**–**VIIIc** (ν_{NH} 3300 cm^{-1} and lower) also indicates hydrogen bonding with participation of the amino groups.

The reactions of 5-arylfuran-2(3*H*)-ones **Ia** and **Ib** with thioacetamide were accompanied by opening of the lactone ring, but the products had a more complicated structure than simple 4-aryl-4-oxobutanoic acid amides. According to the spectral data, the products were formed from two molecules of the lactone and one thoacetamide molecule. Their ^{1}H NMR spectra contained signals from two nonequivalent phenyl rings, four methylene protons in 4-oxobutanoic acid fragment, and methyl protons (δ 2.63 ppm), as well as a singlet at δ 7.02 ppm from an olefinic proton. In the IR spectra of these compounds we observed three strong absorption bands in the region corresponding to carbonyl stretching vibrations, at 1731, 1709, and 1687 cm^{-1}. It was shown previously [15–18] that 5-arylfuran-2(3*H*)-ones readily react with carbonyl compounds to form condensation products at the α-methylene group. Taking into account the above stated, as well as the absence of sulfur in the product molecules (according to their elemental analyses), the

Scheme 2.



Ar = Ph (**a**), 4-ClC_6H_4 (**b**), 4-MeC_6H_4 (**c**).

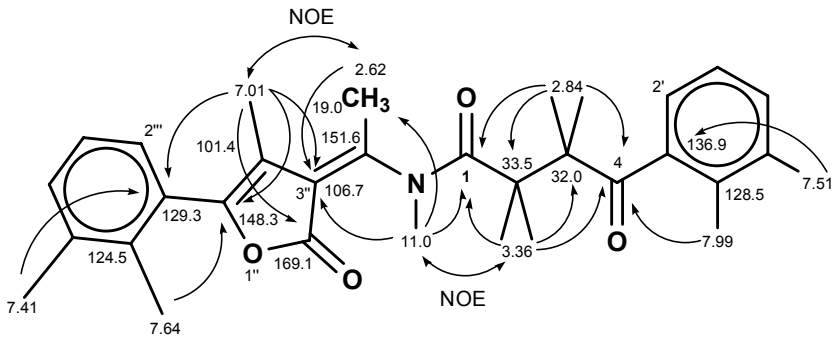


Fig. 2. Structurally significant correlations in the HMBC and NOESY spectra of 4-oxo-*N*-[1-(2-oxo-5-phenyl-2,3-dihydrofuran-3-ylidene)ethyl]-4-phenylbutanamide (**IXa**).

products obtained from lactones **Ia** and **Ib** and thioacetamide were assigned the structure of 4-aryl-*N*-[1-(5-aryl-2-oxo-2,3-dihydrofuran-3-ylidene)ethyl]-4-oxobutanamides **IXa** and **IXb**, respectively (Scheme 2).

The structure of compound **IXa** was confirmed by two-dimensional NMR spectra (COSY, HMQC, HMBC; Fig. 2). The HMBC spectrum of **IXa** showed correlations of the olefinic 4″-H proton (δ 7.01 ppm) with three carbon atoms in the lactone ring (δ_C 169.1, 148.3, 106.7 ppm) and quaternary carbon atom in the phenyl substituent (δ_C 129.3 ppm), indicating the presence of a 5-aryl-2oxo-2,3-dihydrofuran fragment in molecule **IXa**. Correlations were also observed between protons in the methyl group and amino group (δ 11.0 ppm), on the one hand, and quaternary carbon atom in the lactone ring (δ_C 106.7 ppm), which is consistent with the formation of condensation product at the methylene group of **Ia**. The steric configuration of molecule **IXa** was reliably determined on the basis of the NOESY spectrum which revealed coupling between the methyl protons and 4″-H. The existence of such coupling suggests *Z* configuration of the 1-aminoethylidene fragment.

In the reaction of 5-(4-methylphenyl)furan-2(3*H*)-one (**Ic**) with thioacetamide we isolated 4-(4-methylphenyl)-4-oxobutanoic acid (**X**) which was identified by comparison of its physical constants and spectral parameters with those reported in [19]. Presumably, initially formed 4-oxobutanoic acid derivative underwent hydrolysis during the isolation process (the product was purified by recrystallization from aqueous ethanol).

Our results led us to draw some conclusions concerning the effect of substituent in the benzene ring of 5-arylfuran-2(3*H*)-ones on their reactivity. The presence of an electrophilic group (such as 4-chlorophenyl or unsubstituted phenyl) on C^5 in the furan ring makes protons in the C^2H_2 methylene group fairly acidic, so that the reaction with guanidine (which is a strong base) occurs as base-catalyzed dimerization. In the reaction with thioacetamide, the initial reaction step is condensation at the methylene group. If a donor substituent (methyl group) is present, the main reaction pathway is nucleophilic attack on the carbonyl carbon atom, which is followed by cleavage of the lactone ring with formation of 4-(4-methylphenyl)-4-oxobutanoic acid derivative.

EXPERIMENTAL

The IR spectra were recorded in KBr on a Perkin–Elmer Spectrum BX instrument. The ^1H and ^{13}C NMR spectra were measured from solutions in DMSO-d_6 on a Varian Mercury 400 spectrometer at 400 MHz for ^1H and 100 MHz for ^{13}C using tetramethylsilane as internal reference. The melting points were determined on a Boetius melting point apparatus and are uncorrected. The purity of the isolated compounds was checked by TLC on Silufol UV-254 plates using chloroform as eluent and by HPLC–MS on an Agilent 1100 Series instrument coupled with an Agilent LC/MSD SL mass-selective detector (samples were introduced in a trifluoroacetic acid matrix). Initial 5-arylfuran-2(3*H*)-ones **Ia**–**Ic** were synthesized according to the procedure reported in [19].

***N*,*N*′-Bis[4-(4-methylphenyl)-4-oxobutanoyl]-guanidine (IIc).** A mixture of 0.33 g (1.87 mmol) of lactone **Ic** and 0.23 g (1.87 mmol) of guanidine carbonate was fused at 120–130°C over a period of 2 h. After cooling, the solid product was recrystallized from acetic acid. Yield 0.19 g (52%), mp 216–217°C (from AcOH). IR spectrum, ν, cm^{-1}: 3350 (NH), 3289 (NH), 1687 (C=O), 1667 (C=O), 1642 (C=N), 1516, 1329, 1158, 788, 638. ^1H NMR spectrum, δ, ppm: 2.41 s (6H, CH_3), 2.70 t (4H, 3-H, J = 6.5 Hz), 3.22 t

(4H, 2-H, J = 6.5 Hz), 7.29 d (4H, *m*-H, J = 8.0 Hz), 7.86 d (4H, *o*-H, J = 8.0 Hz), 9.33 br.s (3H, NH). Mass spectrum, m/z (I_{rel}, %): 408 (100) $[M + 1]^+$, 410 (20). Found, %: C 67.50; H 6.07; N 10.33. $C_{23}H_{25}N_3O_4$. Calculated, %: C 67.80; H 6.18; N 10.31.

4-(5-Oxo-2-phenyl-2,5-dihydrofuran-2-yl)-5-phenyltetrahydrofuran-2-one (VIIa). A mixture of 0.3 g (1.87 mmol) of lactone **Ia** and 0.23 g (1.87 mmol) of guanidine carbonate was fused at 120–130°C over a period of 2 h. After cooling, the solid product was recrystallized from acetic acid. Yield 0.21 g (71%), mp 179–180°C (decomp.; from AcOH). IR spectrum, ν, cm^{-1}: 3087, 1771 (C=O), 1757 (C=O), 1449, 1211 (C–O), 1172, 1119, 1015, 962, 914, 819, 761, 699. ^{1}H NMR spectrum, δ, ppm: 2.24 d.d (1H, 3-H, ^2J = 18.0, ^3J = 5.2 Hz), 2.88 d.d (1H, 3-H, ^2J = 18.0, ^3J = 10.0 Hz), 3.52 m (1H, 4-H), 5.26 d (1H, 5-H, J = 4.8 Hz), 6.40 d (1H, 4′-H, J = 5.6 Hz), 6.89 m (2H, *o*-H), 7.23 m (3H, *m*-H, *p*-H), 7.34 m (3H, *m*′-H, *p*′-H), 7.43 d (2H, *o*′-H, J = 7.5 Hz), 8.18 d (1H, 3′-H, J = 5.6 Hz). ^{13}C NMR spectrum, δ_C, ppm: 29.5 (C^3), 49.4 (C^4), 81.6 (C^5), 91.2 ($C^{2'}$), 121.3 ($C^{4'}$), 126.0 ($C^{o'}$), 126.4 (C^o), 128.9 (C^p), 129.1 (C^m), 129.2 ($C^{p'}$), 129.6 ($C^{m'}$), 137.6 ($C^{i'}$), 139.9 (C^i), 159.6 ($C^{3'}$), 172.0 ($C^{5'}$), 175.5 (C^2). Mass spectrum: m/z 321 (I_{rel} 100%) $[M + 1]^+$. Found, %: C 74.86; H 4.89. $C_{20}H_{16}O_4$. Calculated, %: C 74.99; H 5.03.

5-(4-Chlorophenyl)-4-[2-(4-chlorophenyl)-5-oxo-2,5-dihydrofuran-2-yl]tetrahydrofuran-2-one (VIIb) was synthesized in a similar way from 0.37 g (1.87 mmol) of lactone **Ib**. Yield 0.25 g (68%), mp 178–180°C (decomp.; from AcOH). IR spectrum, ν, cm^{-1}: 3081, 1773 br.s (C=O), 1757, 1491, 1200 (C–O), 1175, 1094, 1013, 828. ^{1}H NMR spectrum, δ, ppm: 2.28 d.d (1H, 3-H, ^2J = 18.0, ^3J = 7.2 Hz), 2.75 d.d (1H, 3-H, ^2J = 18.0, ^3J 9.2 Hz), 3.57 m (1H, 4-H), 5.23 d (1H, 5-H, J = 6.0 Hz), 6.30 d (1H, 4′-H, J = 5.6 Hz), 6.98 d (2H, *o*-H, J = 8.0 Hz), 7.23 d (2H, *m*-H, J = 8.0 Hz), 7.30 d (2H, *m*′-H, J = 8.0 Hz), 7.43 d (2H, *o*′-H, J = 8.0 Hz), 8.14 d (1H, 3′-H, J = 5.6 Hz). Found, %: C 61.54; H 3.50; Cl 18.24. $C_{20}H_{14}Cl_2O_4$. Calculated, %: C 61.72; H 3.63; Cl 18.22.

***N*-(4-Aryl-4-oxobutanoyl)thioureas VIIIa–VIIIc (*general procedure*).** A mixture of 2.0 mmol of lactone **Ia**–**Ic** and 0.15 g (2.0 mmol) of thiourea was fused at 140–150°C over a period of 5 h. After cooling, the solid product was recrystallized from dimethylformamide.

***N*-(Aminocarbonothioyl)-4-oxo-4-phenylbutanamide (VIIIa).** Yield 0.26 g (56%), mp 187–188°C (from DMF). IR spectrum, ν, cm^{-1}: 3300 (NH), 3171 (NH), 3042, 1703, 1678 br.s (C=O), 1611, 1597, 1530, 1407, 1147, 973, 755, 691, 638. ^{1}H NMR spectrum, δ, ppm: 2.75 t (2H, 3-H, J = 6.0 Hz), 3.27 t (2H, 2-H, J = 6.0 Hz), 7.49 t (2H, *m*-H, J = 8.0 Hz), 7.60 t (1H, *p*-H, J = 8.0 Hz), 7.96 d (2H, *o*-H, J = 8.0 Hz), 9.21 s and 9.57 s (1H each, $CSNH_2$, 11.16 s (1H, CONH). Found, %: C 55.84; H 5.09; N 11.86; S 13.61. $C_{11}H_{12}N_2O_2S$. Calculated, %: C 55.91; H 5.12; N 11.86; S 13.57.

***N*-(Aminocarbonothioyl)-4-(4-chlorophenyl)-4-oxobutanamide (VIIIb).** Yield 0.28 g (51%), mp 252–254°C (from DMF). IR spectrum, ν, cm^{-1}: 3300 (NH), 3160 (NH_2), 1681 br.s (C=O), 1611, 1589, 1533, 1401, 1150, 1085, 985, 663. ^{1}H NMR spectrum, δ, ppm: 2.75 t (2H, 3-H, J = 6.0 Hz), 3.25 t (2H, 2-H, J = 6.0 Hz), 7.49 d (2H, *m*-H, J = 8.0 Hz), 7.96 d (1H, *o*-H, J = 8.0 Hz), 9.17 s and 9.58 s (1H each, $CSNH_2$), 11.10 s (1H, CONH). Found, %: C 48.73; H 4.05; Cl 13.12; N 10.34; S 11.89. $C_{11}H_{11}ClN_2O_2S$. Calculated, %: C 48.80; H 4.10; Cl 13.09; N 10.35; S 11.84.

***N*-(Aminocarbonothioyl)-4-(4-methylphenyl)-4-oxobutanamide (VIIIc).** Yield 0.3 g (60%), mp 223–225°C (from DMF). IR spectrum, ν, cm^{-1}: 3300 (NH), 3148 (NH_2), 1676 br.s (C=O), 1606, 1536, 1404, 1152, 646, 610. ^{1}H NMR spectrum, δ, ppm: 2.41 s (3H, CH_3), 2.74 t (2H, 3-H, J = 6.0 Hz), 3.23 t (2H, 2-H, J = 6.0 Hz), 7.29 d (2H, *m*-H, J = 8.0 Hz), 7.85 d (1H, *o*-H, J = 8.0 Hz), 9.18 s and 9.58 s (1H each, $CSNH_2$), 11.14 s (1H, CONH). Found, %: C 57.50; H 5.59; N 11.21; S 12.86. $C_{12}H_{14}N_2O_2S$. Calculated, %: C 57.58; H 5.64; N 11.19; S 12.81.

4-Oxo-*N*-[1-(2-oxo-5-phenyl-2,3-dihydrofuran-3-ylidene)ethyl]-4-phenylbutanamide (IXa). A mixture of 0.3 g (1.87 mmol) of lactone **Ia** and 0.14 g (1.87 mmol) of thioacetamide was fused at 120–130°C over a period of 4 h. After cooling, the solid product was recrystallized from nitromethane. Yield 0.16 g (48%), mp 183–185°C (from $MeNO_2$). IR spectrum, ν, cm^{-1}: 3266 (NH), 2913, 1731 (C=O), 1709 (C=O), 1687 (C=O), 1631, 1594, 1343, 1228, 1141, 1119, 750, 688. ^{1}H NMR spectrum, δ, ppm: 2.62 s (3H, CH_3), 2.84 t (2H, 3-H, J = 6.0 Hz), 3.36 t (2H, 2-H, J = 6.0 Hz), 7.01 s (1H, *p*-H), 7.32 t (1H, *p*′-H, J = 8.0 Hz), 7.41 t (2H, *m*′-H, J = 8.0 Hz), 7.51 t (2H, *m*′-H, J = 7.5 Hz), 7.61 t (1H, 4′-H, J = 7.5 Hz), 7.64 d (2H, *o*′-H, J = 8.0 Hz), 7.99 d (2H, *o*-H, J = 7.5 Hz), 11.01 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 19.0 (CH_3), 32.0 (C^3), 33.5 (C^2), 101.4 ($C^{4'}$), 106.7 ($C^{3'}$), 124.5 ($C^{o'}$), 128.5 (C^o), 129.1 (C^m, $C^{m'}$), 129.2 ($C^{p'}$), 129.3 ($C^{i'}$), 133.6 (C^p), 136.9 (C^i), 148.3 ($C^{5'}$), 151.6

(=**C**CH_3), 169.1 ($C^{2''}$), 171.4 (C^1), 198.1 (C^4). Found, %: C 73.02; H 5.28; N 3.90. $C_{22}H_{19}NO_4$. Calculated, %: C 73.12; H 5.30; N 3.88.

4-(4-Chlorophenyl)-*N*-{1-[5-(4-chlorophenyl)-2-oxo-2,3-dihydrofuran-3-ylidene]ethyl}-4-oxobutanamide (IXb) was synthesized in a similar way from 0.37 g (1.87 mmol) of lactone **Ib**. Yield 0.17 g (43%), mp 208–209°C (from $MeNO_2$). IR spectrum, ν, cm^{-1}: 3260 (NH), 2924, 1731 (C=O), 1709 (C=O), 1681 (C=O), 1631, 1603, 1348, 1231, 1124, 1091, 1010, 831, 780. ^{1}H NMR spectrum, δ, ppm: 2.61 s (3H, CH_3), 2.84 t (2H, 3-H, J = 6.0 Hz), 3.34 t (2H, 2-H, J = 6.0 Hz), 7.10 s (1H, 4′-H), 7.42 d (2H, *m*′-H, J = 8.5 Hz), 7.53 d (2H, *m*-H, J 8.0 Hz), 7.65 d (2H, *o*′-H, J = 8.5 Hz), 7.99 d (2H, *o*-H, J = 8.0 Hz), 10.98 s (1H, NH). Found, %: C 61.35; H 3.92; Cl 16.50; N 3.27. $C_{22}H_{17}Cl_2NO_4$. Calculated, %: C 61.41; H 3.98; Cl 16.48; N 3.26.

4-(4-Methylphenyl)-4-oxobutanoic acid (X) was isolated in the reaction of 0.33 g (1.87 mmol) of lactone **Ic** with thioacetamide according to the procedure described above for the synthesis of compound **IXa**. Yield 0.16 g (45%), mp 125–126°C; published data [20]: mp 127°C.

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