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Chemistry of Iminofurans: XI.* Synthesis, Structure, and Cyclization of 4-Substituted 2-(Aroylhydrazinylidene)-4-oxobutanoic Acids

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Abstract—Aromatic hydrazides reacted with 4-aryl-2-hydroxy-4-oxobut-2-enoic and 2-hydroxy-5,5-dimethyl-4-oxohex-2-enoic acids to give 4-aryl-2-(2-aroylhydrazinylidene)-4-oxobutanoic and 5,5-dimethyl-2-(aroylhydrazinylidene)-4-oxohexanoic acids. The products were found to exist in solution as mixtures of *Z*/*E*-hydrazinylidene and cyclic dihydropyrazole tautomers, and they underwent intramolecular cyclization to 5-aryl- and 5-*tert*-butyl-3-(aroylhydrazinylidene)furan-2(3*H*)-ones by the action of acetic anhydride.

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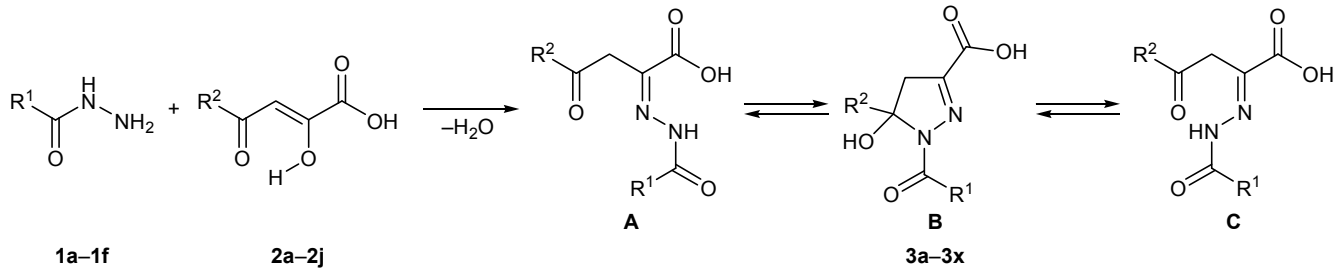
3-Hydrazinylidene(imino)furan-2(3*H*)-ones constitute a class of furan derivatives that are interesting from the viewpoints of both their chemical transformations [2–13] and preparation of various biologically active compounds [14–18]. One of the most convenient methods for the synthesis of 5-aryl-3-hydrazinylidenefuran-2(3*H*)-ones with various substituents in the hydrazone fragment is cyclization of 2-(methylidenehydrazinyl)-4-aryl-4-oxobut-2-enoic acids by the action of aliphatic carboxylic acid anhydrides [19–21]. Herein, we report the synthesis of 3-(aroylhydrazinylidene)furan-2(3*H*)-ones via intramolecular cyclization of 2-(aroylhydrazinylidene)-4-oxobutanoic acids. Introduction of an aroylhydrazinylidene functionality into the 3-position was expected to further extend the synthetic potential of imino (hydrazinylidene) derivatives of furan.

Initially, by reacting 4-substituted 2-hydroxy-4-oxobut-2-enoic acids **2a**–**2j** with aromatic hydrazides **1a**–**1f** we obtained 4-substituted 2-(aroylhydrazinylidene)-4-oxobutanoic acids **3a**–**3x** (Scheme 1). The reaction regioselectively involved the C^2=O carbonyl

Scheme 1.



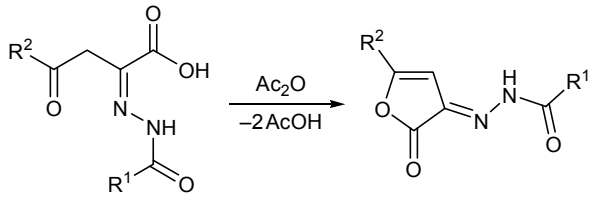
1a–1f **2a–2j** **3a–3x**

1, R^1 = Ph (**a**), 4-MeC_6H_4 (**b**), 4-$MeOC_6H_4$ (**c**), 4-BrC_6H_4 (**d**), 4-$O_2NC_6H_4$ (**e**), 3-$O_2NC_6H_4$ (**f**); **2**, R^2 = *t*-Bu (**a**), Ph (**b**), 4-MeC_6H_4 (**c**), 2,4-$Me_2C_6H_3$ (**d**), 4-$MeOC_6H_4$ (**e**), 4-$EtOC_6H_4$ (**f**), 4-FC_6H_4 (**g**), 4-ClC_6H_4 (**h**), 3,4-$(MeO)_2C_6H_3$ (**i**), naphthalen-1-yl (**j**); **3**, R^1 = Ph, R^2 = *t*-Bu (**a**), Ph (**b**), 4-MeC_6H_4 (**c**), 4-$MeOC_6H_4$ (**d**), 4-$EtOC_6H_4$ (**e**), 4-ClC_6H_4 (**f**), 3,4-$(MeO)_2C_6H_3$ (**g**), naphthalen-1-yl (**h**); R^1 = 4-MeC_6H_4, R^2 = *t*-Bu (**i**), Ph (**j**), 4-MeC_6H_4 (**k**), 2,4-$Me_2C_6H_3$ (**l**), 4-ClC_6H_4 (**m**); R^1 = 4-$MeOC_6H_4$, R^2 = *t*-Bu (**n**), 4-MeC_6H_4 (**o**), 2,4-$Me_2C_6H_3$ (**p**), 4-$EtOC_6H_4$ (**q**), 4-FC_6H_4 (**r**); R^1 = 4-BrC_6H_4, R^2 = 4-$MeOC_6H_4$ (**s**), 4-ClC_6H_4 (**t**); R^1 = 4-$O_2NC_6H_4$, R^2 = *t*-Bu (**u**), 4-$MeOC_6H_4$ (**v**); R^1 = 3-$O_2NC_6H_4$, R^2 = *t*-Bu (**w**), 4-$MeOC_6H_4$ (**x**).

* For communication X, see [1].

group. Acids **3a**–**3x** are colorless or yellow crystalline substances. According to the ^{1}H NMR data, they exist in solution (DMSO-d_6) as equilibrium mixtures of three tautomers **A**–**C**. The composition of the equilibrium mixture does not change with time. Unlike structurally related compounds [20–26], no enehydrazine tautomer was detected. Structure **A** showed a singlet at δ 3.83–4.34 ppm from the methylene protons and a broadened signal at δ 12.95–13.91 ppm due to the NH proton involved in intramolecular hydrogen bond with the C^1=O carbonyl group. Tautomer **B** is characterized by two unsymmetrical *AB* doublets at δ 2.87–3.34 and 3.22–3.46 ppm (^2J = 17.9–20.3 Hz), which is typical of cyclic 5-hydroxy-4,5-dihydropyrazole structure with a chiral carbon atom in the 5-position, and a singlet at δ 5.72–8.46 ppm (OH). The signals at δ 4.07–4.58 (s) and 10.31–11.64 ppm were assigned, respectively, to the methylene and NH groups of tautomer **C**. Electron-donating substituents in the aromatic ring of the hydrazide fragment favor formation of tautomer **B**, whereas electron-withdrawing substituents shift the equilibrium toward structure **C**. This may be rationalized by reduced nucleophilicity of the amino group participating in the cyclization via attack on C^4 due to effect of electron-withdrawing substituent and vice versa. Variation of the substituent on C^4 in butanoic acids **3** is also accompanied by shift of the tautomeric equilibrium. Aromatic substituents with electron-withdrawing groups shift the equilibrium toward structure **B**, and those containing electron-donating groups, toward structure **C**. The latter is the major component of the equilibrium mixture of 5,5-dimethylhexanoic acid derivatives **3a**, **3i**, **3n**, **3u**, and **3w**. Presumably, electron-withdrawing substituents enhance the electrophilicity of the C^4=O carbon atom thus favoring nucleophilic attack thereon by the hydrazide amino group; by contrast, electron-donating groups in the aromatic ring on C^4, as well as *tert*-butyl group, reduce the electrophilicity of C^4, and the formation of cyclic structure **B** is hindered. Analogous tautomers were identified previously for the condensation products of acylhydrazines with acylpyruvic acid esters [27–29] and 5,5-dimethyl-2,4-dioxohexanoic acid [30].

Scheme 2.



3a–3d, **3f**, **3g**, **3i** **3j**, **3n**, **3s**, **3t**, **3v** — **4a–4l**

4, R^1 = Ph, R^2 = *t*-Bu (**a**), Ph (**b**), 4-MeC$_6$H$_4$ (**c**), 4-MeOC$_6$H$_4$ (**d**), 3,4-(MeO)$_2$C$_6$H$_3$ (**e**), 4-ClC$_6$H$_4$ (**f**); R^1 = 4-MeC$_6$H$_4$, R^2 = *t*-Bu (**g**), Ph (**h**); R^1 = 4-MeOC$_6$H$_4$, R^2 = *t*-Bu (**i**); R^1 = 4-BrC$_6$H$_4$, R^2 = 4-MeOC$_6$H$_4$ (**j**), 4-ClC$_6$H$_4$ (**k**); R^1 = 4-O$_2$NC$_6$H$_4$, R^2 = 4-MeOC$_6$H$_4$ (**l**).

Heating of acids **3a**–**3d**, **3f**, **3g**, **3i**, **3j**, **3n**, **3s**, **3t**, and **3v** in acetic anhydride at 70°C for 20 min led to the formation of 3-(aroylhydrazinylidene)furan-2(3*H*)-ones **4a**–**4l** (Scheme 2). Compounds **4a**–**4l** are yellow crystalline substances. They showed in the IR spectra absorption bands typical of stretching vibrations of NH, lactone carbonyl, and hydrazide carbonyl groups in the regions 3177–3288, 1772–1804, and 1623–1682 cm^{-1}, respectively. The NH proton resonated in the ^{1}H NMR spectra of **4a**–**4l** as a singlet at δ 11.49–12.87 ppm, and the proton on C^4 of the furan ring appeared as a singlet at δ 6.54–7.55 ppm.

EXPERIMENTAL

The IR spectra were recorded in mineral oil on FSM-1201 and Specord M-80 spectrometers. The ^{1}H NMR spectra were measured on Varian Mercury Plus-300 (300.05 MHz), Bruker Avance III (400 MHz), and Tesla BS-567A (100 MHz) instruments relative to hexamethyldisiloxane as internal standard. The elemental analyses were obtained on a Leco CHNS-932 analyzer. The progress of reactions was monitored, and the purity of the isolated compounds was checked, by TLC on Sorbfil PTSKh P-A-UF-254 plates (diethyl ether–benzene–acetone, 10:9:1; detection under UV light or by treatment with iodine vapor). The melting points were determined on an SMP40 melting point apparatus. The mass spectra (electron impact, 70 eV) were recorded on a Kratos MS-30 spectrometer (ion source temperature 200°C).

2-(2-Aroylhydrazinylidene)-4-aryl-4-oxobutanoic acids 3a–3x (*general procedure*). A solution of 0.01 mol of hydrazide **1a**–**1f** in 15 mL of ethanol or acetonitrile was added to a solution of 0.01 mol of aroylpyruvic acid **2a**–**2j** in 20 mL of ethanol. The mixture was stirred for 5–10 min at 40–50°C and kept for 24 h at 20–25°C, and the precipitate was filtered off and purified by recrystallization.

2-(2-Benzoylhydrazinylidene)-5,5-dimethyl-4-oxohexanoic acid (3a). Yield 2.09 g (72%), colorless crystals, mp 182–183°C (from *i*-PrOH). IR spectrum, ν, cm^{-1}: 3202 br (NH), 1701 sh (COOH), 1638 (CONH),

1601 (C^4=O). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: **A** (43%): 1.17 s (9H, *t*-Bu), 3.85 s (2H, CH_2), 7.60 m (5H, H_{arom}), 13.54 br.s (1H, NH); **B** (2%): 1.05 s (9H, *t*-Bu), 2.92 d and 3.44 d (1H each, CH_2, J_{AB} = 19.54 Hz), 5.72 (1H, OH), 7.60 m (5H, H_{arom}); **C** (55%): 1.15 s (9H, *t*-Bu), 4.10 s (2H, CH_2), 7.60 m (5H, H_{arom}), 11.23 s (1H, NH). Found, %: C 62.02; H 6.26; N 9.72. $C_{15}H_{18}N_2O_4$. Calculated, %: C 62.06; H 6.25; N 9.65.

2-(2-Benzoylhydrazinylidene)-4-oxo-4-phenylbutanoic acid (3b). Yield 2.07 g (67%), yellow crystals, mp 171–172°C (from PhMe). IR spectrum, ν, cm^{-1}: 3257 br (NH), 1682 sh (COOH, CONH), 1612 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (16%): 4.25 s (2H, CH_2), 7.50 m (10H, H_{arom}), 13.39 br.s (1H, NH); **B** (64%): 3.10 d and 3.30 d (1H each, CH_2, J_{AB} = 17.9 Hz), 7.50 m (11H, OH, H_{arom}); **C** (20%): 4.51 s (2H, CH_2), 7.50 m (10H, H_{arom}), 11.18 s (1H, NH). Found, %: C 65.77; H 4.51; N 9.07. $C_{17}H_{14}N_2O_4$. Calculated, %: C 65.80; H 4.55; N 9.03.

2-(2-Benzoylhydrazinylidene)-4-(4-methylphenyl)-4-oxobutanoic acid (3c). Yield 3.01 g (93%), yellow crystals, mp 170–171°C (from *i*-PrOH). IR spectrum, ν, cm^{-1}: 3249 br (NH), 1693 (COOH), 1675 (CONH), 1603 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (40%): 2.38 s (3H, Me), 4.23 s (2H, CH_2), 7.50 m (9H, H_{arom}), 13.42 br.s (1H, NH); **B** (50%): 2.28 s (3H, Me), 3.10 d and 3.30 d (1H each, CH_2, J_{AB} = 18 Hz), 7.50 m (10H, OH, H_{arom}); **C** (29%): 2.45 s (3H, Me), 4.55 s (2H, CH_2), 7.5 m (9H, H_{arom}), 10.31 s (1H, NH). Found, %: C 66.72; H 5.01; N 8.59. $C_{18}H_{16}N_2O_4$. Calculated, %: C 66.66; H 4.97; N 8.64.

2-(2-Benzoylhydrazinylidene)-4-(4-methoxyphenyl)-4-oxobutanoic acid (3d). Yield 3.06 g (90%), yellow crystals, mp 156–158°C (from *i*-PrOH). ^{1}H NMR spectrum, δ, ppm: **A** (21%): 3.86 s (3H, MeO), 4.28 s (2H, CH_2), 7.50 m (9H, H_{arom}), 13.68 br.s (1H, NH); **B** (26%): 3.78 s (3H, MeO), 3.25 d and 3.32 d (1H each, CH_2, J_{AB} = 18.9 Hz), 7.22 br.s (1H, OH), 7.50 m (9H, H_{arom}); **C** (53%): 3.88 s (3H, MeO), 4.55 s (2H, CH_2), 7.50 m (9H, H_{arom}), 11.35 s (1H, NH). Found, %: C 63.55; H 4.77; N 8.25. $C_{19}H_{16}N_2O_5$. Calculated, %: C 63.53; H 4.74; N 8.23.

2-(2-Benzoylhydrazinylidene)-4-(4-ethoxyphenyl)-4-oxobutanoic acid (3e). Yield 2.73 g (77%), yellow crystals, mp 138–140°C (from PhH). IR spectrum, ν, cm^{-1}: 3420 (OH), 3240 br (NH), 1714 (COOH), 1666 (CONH), 1600 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (17%): 1.37 m (3H, Me), 4.12 m (2H, CH_2O), 4.34 s (2H, CH_2), 7.50 m (10H, H_{arom}), 13.37 br.s (1H, NH); **B** (31%): 1.37 m (3H, Me), 3.20 d and 3.22 d (1H each, CH_2, J_{AB} = 7.8 Hz), 4.12 m (2H, CH_2O), 7.50 m (10H, OH, H_{arom}); **C** (52%): 1.37 m (3H, Me), 4.12 m (2H, CH_2O), 4.52 s (2H, CH_2), 7.50 m (9H, H_{arom}), 11.18 s (1H, NH). Found, %: C 64.46; H 5.15; N 7.87. $C_{19}H_{18}N_2O_5$. Calculated, %: C 64.40; H 5.12; N 7.91.

2-(2-Benzoylhydrazinylidene)-4-(4-chlorophenyl)-4-oxobutanoic acid (3f). Yield 2.17 g (63%), yellow crystals, mp 164–166°C (from MeCN). IR spectrum, ν, cm^{-1}: 3420 (OH), 3250 br (NH), 1728 (COOH), 1682 (CONH), 1602 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (9%): 4.24 s (2H, CH_2), 7.50 m (9H, H_{arom}), 13.34 br.s (1H, NH); **B** (79%): 3.23 d and 3.25 d (1H each, CH_2, J_{AB} = 18.6 Hz), 7.50 m (10H, OH, H_{arom}); **C** (12%): 4.49 s (2H, CH_2), 7.50 m (9H, H_{arom}), 11.17 s (1H, NH). Found, %: C 59.22; H 3.76; N 8.12. $C_{17}H_{13}N_2O_4$. Calculated, %: C 59.23; H 3.80; N 8.13.

2-(2-Benzoylhydrazinylidene)-4-(3,4-dimethoxyphenyl)-4-oxobutanoic acid (3g). Yield 3.03 g (82%), yellow crystals, mp 108–110°C (from MeCN). IR spectrum, ν, cm^{-1}: 3349 br (OH), 3196 br (NH), 1711 (COOH), 1687 (CONH), 1640 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (19%): 3.75 s (3H, MeO), 3.89 s (3H, MeO), 4.30 s (2H, CH_2), 7.50 m (8H, H_{arom}), 13.70 br.s (1H, NH); **B** (33%): 3.74 s (3H, MeO), 3.84 s (3H, MeO), 3.23 d and 3.25 d (1H, CH_2, J_{AB} = 19.3 Hz), 7.23 br.s (1H, OH), 7.50 m (8H, H_{arom}); **C** (48%): 3.76 s (3H, MeO), 3.89 s (3H, MeO), 4.58 s (2H, CH_2), 7.50 m (8H, H_{arom}), 11.37 s (1H, NH). Found, %: C 61.72; H 4.96; N 7.52. $C_{19}H_{18}N_2O_6$. Calculated, %: C 61.62; H 4.90; N 7.56.

2-(2-Benzoylhydrazinylidene)-4-(naphthalen-1-yl)-4-oxobutanoic acid (3h). Yield 2.41 g (67%), yellow crystals, mp 156–157°C (from MeCN). IR spectrum, ν, cm^{-1}: 3204 br (NH), 1704 (COOH), 1676 (CONH), 1604 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (18%): 4.31 s (2H, CH_2), 7.60 m (12H, H_{arom}), 13.53 br.s (1H, NH); **B** (56%): 3.34 d and 3.38 d (1H each, CH_2, J_{AB} = 18.2 Hz), 7.60 m (12H, H_{arom}), 8.46 br.s (1H, OH); **C** (26%): 4.60 s (2H, CH_2), 7.60 m (12H, H_{arom}), 11.17 s (1H, NH). Found, %: C 69.92; H 4.55; N 7.81. $C_{21}H_{16}N_2O_4$. Calculated, %: C 69.99; H 4.48; N 7.77.

5,5-Dimethyl-2-[2-(4-methylbenzoyl)hydrazinylidene]-4-oxohexanoic acid (3i). Yield 2.52 g (83%), colorless crystals, mp 190–191°C (from *i*-PrOH). IR spectrum, ν, cm^{-1}: 3192 br (NH), 1700 (COOH), 1668 (CONH), 1640 (C^4=O). ^{1}H NMR spectrum

(DMSO-d_6), δ, ppm: **A** (46%): 1.19 s (9H, *t*-Bu), 2.43 s (3H, Me), 3.86 s (2H, CH_2), 7.37 d and 7.77 d (2H each, H_{arom}, J = 8.1 Hz), 13.56 br.s (1H, NH); **B** (3%): 1.07 s (9H, *t*-Bu), 2.40 s (3H, Me), 2.93 d and 3.46 d (1H each, CH_2, J_{AB} = 19.4 Hz), 5.73 s (1H, OH), 7.30 d and 7.60 d (2H each, H_{arom}, J = 8.1 Hz); **C** (51%): 1.20 s (9H, *t*-Bu), 2.42 s (3H, Me), 4.13 s (2H, CH_2), 7.42 d and 7.78 d (2H, H_{arom}, J = 8.1 Hz), 11.21 s (1H, NH). Found, %: C 63.10; H 6.65; N 9.22. $C_{16}H_{20}N_2O_4$. Calculated, %: C 63.14; H 6.62; N 9.20.

2-[2-(4-Methylbenzoyl)hydrazinylidene]-4-oxo-4-phenylbutanoic acid (3j). Yield 2.17 g (67%), yellow crystals, mp 154–155°C (from 1,2-dichloroethane). IR spectrum, ν, cm^{-1}: 3210 br (NH), 1706 (COOH), 1672 (CONH), 1604 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (15%): 2.36 s (3H, Me), 4.32 s (2H, CH_2), 7.50 m (9H, H_{arom}), 13.43 br.s (1H, NH); **B** (63%): 2.39 s (3H, Me), 3.26 d and 3.29 d (1H each, CH_2, J_{AB} = 18.2 Hz), 7.50 m (9H, OH, H_{arom}); **C** (22%): 2.36 s (3H, Me), 4.58 s (2H, CH_2), 7.50 m (9H, H_{arom}), 11.21 s (1H, NH). Mass spectrum, *m*/*z* (I_{rel}, %): 306 (2.1) $[M - H_2O]^+$, 279 (5.2) $[M - COOH]^+$, 119 (95.2), 105 (48.3), 91 (100), 77 (78.3), 65 (71.8), 51 (48.3), 39 (49.7). Found, %: C 66.70; H 5.03; N 8.63. $C_{18}H_{16}N_2O_4$. Calculated, %: C 66.66; H 4.97; N 8.64.

2-[2-(4-Methylbenzoyl)hydrazinylidene]-4-(4-methylphenyl)-4-oxobutanoic acid (3k). Yield 2.77 g (82%), yellow crystals, mp 194–195°C (from MeCN). IR spectrum, ν, cm^{-1}: 3210 br (NH), 1704 (COOH), 1678 (CONH), 1604 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (32%): 2.36 s (3H, Me), 2.40 s (3H, Me), 4.30 s (2H, CH_2), 7.50 m (8H, H_{arom}), 13.59 br.s (1H, NH); **B** (26%): 2.29 s (3H, Me), 2.40 s (3H, Me), 3.22 d and 3.28 d (1H each, CH_2, J_{AB} = 19.2 Hz), 7.34 br.s (1H, OH), 7.50 m (8H, H_{arom}); **C** (42%): 2.39 s (3H, Me), 2.40 s (3H, Me), 4.58 s (2H, CH_2), 7.50 m (8H, H_{arom}), 11.27 s (1H, NH). Mass spectrum, *m*/*z* (I_{rel}, %): 293 (25.2) $[M - COOH]^+$, 119 (15.3), 91 (100). Found, %: C 67.42; H 5.39; N 8.33. $C_{19}H_{18}N_2O_4$. Calculated, %: C 67.45; H 5.36; N 8.28.

4-(2,4-Dimethylphenyl)-2-[2-(4-methylbenzoyl)-hydrazinylidene]-4-oxobutanoic acid (3l). Yield 2.68 g (76%), yellow crystals, mp 158–159°C (from MeCN). IR spectrum, ν, cm^{-1}: 3200 br (NH), 1701 (COOH), 1679 (CONH), 1612 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (28%): 2.10–2.40 m (9H, Me), 4.14 s (2H, CH_2), 7.40 m (7H, H_{arom}), 13.36 br.s (1H, NH); **B** (32%): 2.10–2.40 m (9H, Me), 3.12 d and 3.18 d (1H each, CH_2, J_{AB} = 18.2 Hz), 7.38 br.s (1H, OH), 7.40 m (7H, H_{arom}); **C** (40%): 2.10–2.40 m (9H, Me), 4.40 s (2H, CH_2), 7.40 m (7H, H_{arom}), 11.07 s (1H, NH). Found, %: C 68.15; H 5.69; N 8.00. $C_{20}H_{20}N_2O_4$. Calculated, %: C 68.17; H 5.72; N 7.95.

2-[2-(4-Methylbenzoyl)hydrazinylidene]-4-(4-chlorophenyl)-4-oxobutanoic acid (3m). Yield 2.83 g (79%), yellow crystals, mp 192–193°C (from AcOH). IR spectrum, ν, cm^{-1}: 3202 br (NH), 1683 (COOH), 1662 (CONH), 1601 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (32%): 2.32 s (3H, Me), 4.24 s (2H, CH_2), 7.40 m (8H, H_{arom}), 13.41 br.s (1H, NH); **B** (26%): 2.32 s (3H, Me), 3.21 d and 3.23 d (1H each, CH_2, J_{AB} = 19.0 Hz), 7.40 m (9H, OH, H_{arom}); **C** (42%): 2.32 s (3H, Me), 4.51 s (2H, CH_2), 7.40 m (8H, H_{arom}), 11.12 s (1H, NH). Found, %: C 68.15; H 5.69; N 8.00. $C_{20}H_{20}N_2O_4$. Calculated, %: C 68.17; H 5.72; N 7.95.

2-[2-(4-Methoxybenzoyl)hydrazinylidene]-5,5-dimethyl-4-oxohexanoic acid (3n). Yield 2.18 g (68%), colorless crystals, mp 164–165°C (from MeCN). IR spectrum, ν, cm^{-1}: 3225 br (NH), 1696 br (COOH, CONH), 1632 (C^4=O). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: **A** (44%): 1.14 s (9H, *t*-Bu), 3.84 s (2H, CH_2), 3.83 s (3H, MeO), 7.40 m (4H, H_{arom}), 13.48 br.s (1H, NH); **B** (4%): 1.02 s (9H, *t*-Bu), 2.87 d and 3.41 d (1H each, CH_2, J_{AB} = 20.1 Hz), 3.82 s (3H, MeO), 5.67 s (1H, OH), 7.40 m (4H, H_{arom}); **C** (52%): 1.16 s (9H, *t*-Bu), 3.82 s (3H, MeO), 4.08 s (2H, CH_2), 7.40 m (4H, H_{arom}), 11.07 s (1H, NH). Found, %: C 59.93; H 6.25; N 8.75. $C_{16}H_{20}N_2O_5$. Calculated, %: C 59.99; H 6.29; N 8.74.

2-[2-(4-Methoxybenzoyl)hydrazinylidene]-4-(4-methylphenyl)-4-oxobutanoic acid (3o). Yield 3.19 g (90%), yellow crystals, mp 151–152°C (from MeCN). IR spectrum, ν, cm^{-1}: 3267 br (NH), 1748 (COOH), 1677 (CONH), 1605 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (14%): 2.37 s (3H, Me), 3.77 s (3H, MeO), 4.22 s (2H, CH_2), 7.40 m (8H, H_{arom}), 13.38 br.s (1H, NH); **B** (65%): 2.25 s (3H, Me), 3.18 d and 3.20 d (1H each, CH_2, J_{AB} = 18.7 Hz), 3.77 s (3H, MeO), 7.40 m (9H, OH, H_{arom}); **C** (21%): 2.37 s (3H, Me), 3.77 s (3H, MeO), 4.49 s (2H, CH_2), 7.40 m (8H, H_{arom}), 11.20 s (1H, NH). Found, %: C 64.40; H 5.13; N 8.69. $C_{19}H_{18}N_2O_5$. Calculated, %: C 64.40; H 5.12; N 7.91.

4-(2,4-Dimethylphenyl)-2-[2-(4-methoxybenzoyl)hydrazinylidene]-4-oxobutanoic acid (3p). Yield 2.72 g (74%), yellow crystals, mp 164–165°C (from MeCN). IR spectrum, ν, cm^{-1}: 3241 br (NH), 1698 (COOH), 1681, 1652 (CONH), 1605 (C^4=O).

^{1}H NMR spectrum, δ, ppm: **A** (20%): 2.10–2.40 m (6H, Me), 3.77 s (3H, MeO), 4.14 s (2H, CH_2), 7.40 m (7H, H_{arom}), 13.44 br.s (1H, NH); **B** (42%): 2.10–2.40 m (6H, Me), 3.16 d and 3.18 d (1H each, CH_2, J_{AB} = 18.2 Hz), 3.77 s (3H, MeO), 7.48 br.s (1H, OH), 7.40 m (7H, H_{arom}); **C** (28%): 2.10–2.40 m (6H, Me), 3.77 s (3H, MeO), 4.42 s (2H, CH_2), 7.40 m (7H, H_{arom}), 11.07 s (1H, NH). Found, %: C 65.16; H 5.49; N 7.65. $C_{20}H_{20}N_2O_5$. Calculated, %: C 65.21; H 5.47; N 7.60.

4-(4-Ethoxyphenyl)-2-[2-(4-methoxybenzoyl)hydrazinylidene]-4-oxobutanoic acid (3q). Yield 2.31 g (60%), yellow crystals, mp 168–169°C (from MeCN). IR spectrum, ν, cm^{-1}: 3357 (OH), 3265 (NH), 3225 br (NH), 1695 (COOH), 1677 (CONH), 1605 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (12%): 1.36 m (3H, Me), 3.76 s (3H, MeO), 4.22 s (2H, CH_2), 4.18 m (2H, CH_2), 7.40 m (8H, H_{arom}), 13.36 br.s (1H, NH); **B** (31%): 1.36 m (3H, Me), 3.18 d and 3.20 d (1H each, CH_2, J_{AB} = 18.4 Hz), 3.77 s (3H, MeO), 4.18 m (2H, OCH_2), 7.40 m (9H, OH, H_{arom}); **C** (57%): 1.36 m (3H, Me), 3.76 s (3H, MeO), 4.18 m (2H, CH_2), 4.46 s (2H, CH_2), 7.40 m (8H, H_{arom}), 11.04 s (1H, NH). Found, %: C 62.43; H 5.23; N 7.33. $C_{20}H_{20}N_2O_6$. Calculated, %: C 62.49; H 5.24; N 7.29.

4-(4-Fluorophenyl)-2-[2-(4-methoxybenzoyl)hydrazinylidene]-4-oxobutanoic acid (3r). Yield 2.04 g (57%), yellow crystals, mp 136–137°C (from MeCN). IR spectrum, ν, cm^{-1}: 3212 br (NH), 1701 (COOH), 1679, 1642 (CONH), 1601 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (18%): 3.78 s (3H, MeO), 4.23 s (2H, CH_2), 7.40 m (8H, H_{arom}), 13.34 br.s (1H, NH); **B** (60%): 3.19 d and 3.21 d (1H each, CH_2, J_{AB} = 19.4 Hz), 3.78 s (3H, MeO), 7.40 m (9H, OH, H_{arom}); **C** (22%): 3.78 s (3H, MeO), 4.48 s (2H, CH_2), 7.40 m (8H, H_{arom}), 11.03 s (1H, NH). Found, %: C 60.40; H 5.33; N 7.83. $C_{18}H_{15}FN_2O_5$. Calculated, %: C 60.34; H 4.22; N 7.82.

2-[2-(4-Bromobenzoyl)hydrazinylidene]-4-(4-methoxyphenyl)-4-oxobutanoic acid (3s). Yield 3.85 g (92%), yellow crystals, mp 168–169°C (from EtOH–$CHCl_3$). IR spectrum, ν, cm^{-1}: 3225 br (NH), 1711 (COOH), 1687 (CONH), 1597 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (16%): 3.87 s (3H, MeO), 4.30 s (2H, CH_2), 7.50 m (8H, H_{arom}), 13.45 br.s (1H, NH); **B** (36%): 3.29 d and 3.31 d (1H each, CH_2, J_{AB} = 19.4 Hz), 3.79 s (3H, MeO), 7.34 br.s (1H, OH), 7.50 m (8H, H_{arom}); **C** (48%): 3.90 s (3H, MeO), 4.57 s (2H, CH_2), 7.40 m (8H, H_{arom}), 11.44 s (1H, NH). Found, %: C 51.52; H 3.63; N 6.73. $C_{18}H_{15}BrN_2O_5$. Calculated, %: C 51.57; H 3.61; N 6.68.

2-[2-(4-Bromobenzoyl)hydrazinylidene]-4-(4-chlorophenyl)-4-oxobutanoic acid (3t). Yield 3.76 g (89%), yellow crystals, mp 179–180°C (from EtOH–$CHCl_3$). IR spectrum, ν, cm^{-1}: 3195 br (NH), 1683 (COOH, CONH), 1596 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (9%): 4.28 s (2H, CH_2), 7.50 m (8H, H_{arom}), 12.95 br.s (1H, NH); **B** (75%): 3.34 d and 3.36 d (1H each, CH_2, J_{AB} = 20.3 Hz), 7.60 m (9H, OH, H_{arom}); **C** (16%): 4.61 s (2H, CH_2), 7.60 m (8H, H_{arom}), 11.45 s (1H, NH). Found, %: C 48.22; H 2.84; N 6.65. $C_{17}H_{12}BrClN_2O_4$. Calculated, %: C 48.20; H 2.86; N 6.61.

5,5-Dimethyl-2-[2-(4-nitrobenzoyl)hydrazinylidene]-4-oxohexanoic acid (3u). Yield 2.81 g (84%), colorless crystals, mp 191–192°C (from EtOAc). IR spectrum, ν, cm^{-1}: 3237 br (NH), 1703 (COOH), 1639 (CONH), 1600 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (42%): 1.14 s (9H, *t*-Bu), 3.84 s (2H, CH_2), 7.90 m (4H, H_{arom}), 13.66 br.s (1H, NH); **B** (8%): 1.02 s (9H, *t*-Bu), 2.93 d and 3.45 d (1H each, CH_2, J_{AB} = 19.8 Hz), 6.67 s (1H, OH), 7.90 m (4H, H_{arom}); **C** (50%): 1.16 s (9H, *t*-Bu), 4.07 s (2H, CH_2), 7.90 m (4H, H_{arom}), 11.46 s (1H, NH). Found, %: C 53.75; H 5.07; N 12.56. $C_{15}H_{17}N_3O_6$. Calculated, %: C 53.73; H 5.11; N 12.53.

4-(4-Methoxyphenyl)-2-[2-(4-nitrobenzoyl)hydrazinylidene]-4-oxobutanoic acid (3v). Yield 3.19 g (83%), yellow crystals, mp 115–116°C (from EtOH–$CHCl_3$). IR spectrum, ν, cm^{-1}: 3275 br (NH), 1709 (COOH), 1692 (CONH), 1598 (C^4=O). ^{1}H NMR spectrum, δ, ppm: **A** (14%): 3.88 s (3H, MeO), 4.31 s (2H, CH_2), 7.60 m (8H, H_{arom}), 13.75 br.s (1H, NH); **B** (42%): 3.32 d and 3.36 d (1H each, CH_2, J_{AB} = 19.1 Hz), 3.80 s (3H, MeO), 7.50 m (9H, OH, H_{arom}); **C** (44%): 3.90 s (3H, MeO), 4.58 s (2H, CH_2), 7.60 m (8H, H_{arom}), 11.64 s (1H, NH). Found, %: C 56.12; H 3.87; N 10.95. $C_{18}H_{15}N_3O_7$. Calculated, %: C 56.11; H 3.92; N 10.91.

5,5-Dimethyl-2-[2-(3-nitrobenzoyl)hydrazinylidene]-4-oxohexanoic acid (3w). Yield 2.21 g (66%), colorless crystals, mp 173–174°C (from MePh). IR spectrum, ν, cm^{-1}: 3184 br (NH), 1699 (COOH), 1662 (CONH), 1635 (C^4=O). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: **A** (20%): 1.14 s (9H, *t*-Bu), 3.83 s (2H, CH_2), 8.10 m (4H, H_{arom}), 13.84 br.s (1H, NH); **B** (18%): 1.08 s (9H, *t*-Bu), 2.95 d and 3.46 d (1H each, CH_2, J_{AB} = 19.4 Hz), 6.66 s (1H, OH), 8.10 m (4H, H_{arom}); **C** (62%): 1.17 s (9H, *t*-Bu), 4.08 s (2H, CH_2), 8.10 m (4H, H_{arom}), 11.49 s (1H, NH). Found, %: C 53.78; H 5.09; N 12.59. $C_{15}H_{17}N_3O_6$. Calculated, %: C 53.73; H 5.11; N 12.53.

4-(4-Methoxyphenyl)-2-[2-(3-nitrobenzoyl)hydrazinylidene]-4-oxobutanoic acid (3x). Yield 2.66 g (69%), yellow crystals, mp 183–184°C (from AcOH). ^{1}H NMR spectrum, δ, ppm: **A** (21%): 3.68 s (3H, MeO), 4.28 s (2H, CH_2), 7.60 m (8H, H_{arom}), 13.75 br.s (1H, NH); **B** (26%): 3.26 d and 3.31 d (1H each, CH_2, J_{AB} = 18.9 Hz), 3.75 s (3H, MeO), 7.60 m (9H, OH, H_{arom}); **C** (53%): 3.88 s (3H, MeO), 4.55 s (2H, CH_2), 7.60 m (8H, H_{arom}), 11.35 s (1H, NH). Found, %: C 56.12; H 3.87; N 10.95. $C_{18}H_{15}N_3O_7$. Calculated, %: C 56.11; H 3.92; N 10.91.

5-Substituted 3-(2-aroylhydrazinylidene)furan-2(3*H*)-ones 4a–4l (*general procedure*). A solution of 0.01 mol of acid **3a**–**3d**, **3f**, **3g**, **3i**, **3j**, **3n**, **3s**, **3t**, or **3v** in 3–4 mL of acetic anhydride was heated for 15–20 min at 70°C with stirring. After cooling, the precipitate was filtered off, washed with anhydrous diethyl ether, and recrystallized from anhydrous toluene.

***N′*-[5-*tert*-Butyl-2-oxofuran-3(2*H*)-ylidene]benzohydrazide (4a).** Yield 1.58 g (58%), yellow crystals, mp 203–204°C (from PhMe). IR spectrum, ν, cm^{-1}: 3220 br (NH), 1798 (C^2=O), 1670 (CONH). ^{1}H NMR spectrum, δ, ppm: 1.26 s (9H, *t*-Bu), 6.54 s (1H, 4-H), 7.60 m (5H, H_{arom}), 11.82 s (1H, NH). Found, %: C 66.12; H 5.88; N 10.95. $C_{15}H_{16}N_2O_3$. Calculated, %: C 66.16; H 5.92; N 10.29.

***N′*-[2-Oxo-5-phenylfuran-3(2*H*)-ylidene]benzohydrazide (4b).** Yield 1.46 g (50%), yellow crystals, mp 218–219°C (from PhMe). IR spectrum, ν, cm^{-1}: 3177 br (NH), 1802 (C^2=O), 1662 (CONH). ^{1}H NMR spectrum, δ, ppm: 7.21 s (1H, 4-H), 7.60 m (10H, H_{arom}), 12.41 s (1H, NH). Found, %: C 69.78; H 4.09; N 9.65. $C_{17}H_{12}N_2O_3$. Calculated, %: C 69.86; H 4.14; N 9.58.

***N′*-[5-(4-Methylphenyl)-2-oxofuran-3(2*H*)-ylidene]benzohydrazide (4c).** Yield 1.65 g (54%), yellow crystals, mp 240–241°C (from dioxane). IR spectrum, ν, cm^{-1}: 3195 br (NH), 1804 (C^2=O), 1660 (CONH). ^{1}H NMR spectrum, δ, ppm: 2.40 s (3H, Me), 7.55 s (1H, 4-H), 7.60 m (9H, H_{arom}), 11.79 s (1H, NH). Found, %: C 70.66; H 4.69; N 8.99. $C_{18}H_{14}N_2O_3$. Calculated, %: C 70.58; H 4.61; N 9.15.

***N′*-[5-(4-Methoxyphenyl)-2-oxofuran-3(2*H*)-ylidene]benzohydrazide (4d).** Yield 2.19 g (68%), yellow crystals, mp 228–229°C (from PhMe). IR spectrum, ν, cm^{-1}: 3271 br (NH), 1806 (C^2=O), 1694 (CONH). ^{1}H NMR spectrum, δ, ppm: 3.85 s (3H, MeO), 7.11 s (1H, 4-H), 7.50 m (9H, H_{arom}), 12.49 s (1H, NH). Found, %: C 64.11; H 4.34; N 8.66. $C_{18}H_{14}N_2O_4$. Calculated, %: C 64.07; H 4.38; N 8.69.

***N′*-[5-(3,4-Dimethoxyphenyl)-2-oxofuran-3(2*H*)-ylidene]benzohydrazide (4e).** Yield 1.93 g (55%), yellow crystals, mp 245–247°C (from PhMe). IR spectrum, ν, cm^{-1}: 3180 br (NH), 1798 (C^2=O), 1653 (CONH). ^{1}H NMR spectrum, δ, ppm: 3.80 s (6H, MeO), 7.29 s (1H, 4-H), 7.60 m (8H, H_{arom}), 12.35 s (1H, NH). Found, %: C 64.68; H 4.64; N 7.87. $C_{19}H_{16}N_2O_5$. Calculated, %: C 64.77; H 4.58; N 7.95.

***N′*-[5-(4-Chlorophenyl)-2-oxofuran-3(2*H*)-ylidene]benzohydrazide (4f).** Yield 2.12 g (65%), yellow crystals, mp 243–244°C (from PhMe). IR spectrum, ν, cm^{-1}: 3280 br (NH), 1772 (C^2=O), 1682 (CONH). ^{1}H NMR spectrum, δ, ppm: 7.23 s (1H, 4-H), 7.60 m (8H, H_{arom}), 12.39 s (1H, NH). Found, %: C 62.44; H 3.46; N 8.63. $C_{17}H_{11}ClN_2O_3$. Calculated, %: C 62.49; H 3.39; N 8.57.

***N′*-[5-*tert*-Butyl-2-oxofuran-3(2*H*)-ylidene]-4-methylbenzohydrazide (4g).** Yield 1.46 g (51%), yellow crystals, mp 200–201°C (from PhMe). IR spectrum, ν, cm^{-1}: 3210 br (NH), 1797 (C^2=O), 1677 (CONH). ^{1}H NMR spectrum, δ, ppm: 1.21 s (9H, *t*-Bu), 2.36 s (3H, Me), 6.81 s (1H, 4-H), 7.25 d and 7.69 d (2H each, H_{arom}, J = 8.1 Hz), 11.54 s (1H, NH). Mass spectrum, *m*/*z* (I_{rel}, %): 201 (45.2), 119 (100), 91 (76.2), 57 (42.4). Found, %: C 67.18; H 6.28; N 9.71. $C_{16}H_{18}N_2O_3$. Calculated, %: C 67.12; H 6.34; N 9.78.

4-Methyl-*N′*-[2-oxo-5-phenylfuran-3(2*H*)-ylidene]benzohydrazide (4h). Yield 2.11 g (69%), yellow crystals, mp 208–210°C (from PhMe). IR spectrum, ν, cm^{-1}: 3200 br (NH), 1772 (C^2=O), 1698 (CONH). ^{1}H NMR spectrum, δ, ppm: 2.40 s (3H, Me), 6.60 s (1H, 4-H), 7.50 m (9H, H_{arom}), 12.67 s (1H, NH). Mass spectrum, *m*/*z* (I_{rel}, %): 201 (45.2), 119 (100), 91 (71.1), 77 (56.5). Found, %: C 70.49; H 4.69; N 9.08. $C_{18}H_{14}N_2O_3$. Calculated, %: C 70.58; H 4.61; N 9.15.

***N′*-[5-*tert*-Butyl-2-oxofuran-3(2*H*)-ylidene]-4-methoxybenzohydrazide (4i).** Yield 1.72 g (57%), yellow crystals, mp 211–212°C (from PhMe). IR spectrum, ν, cm^{-1}: 3230 br (NH), 1798 (C^2=O), 1668 (CONH). ^{1}H NMR spectrum, δ, ppm: 1.21 s (9H, *t*-Bu), 3.81 s (3H, MeO), 6.79 s (1H, 4-H), 6.82 d and 7.79 d (2H each, H_{arom}, J = 8.2 Hz), 11.49 s (1H, NH). Found, %: C 63.61; H 6.09; N 9.19. $C_{16}H_{18}N_2O_4$. Calculated, %: C 63.56; H 6.00; N 9.27.

***N′*-[5-(4-Methoxyphenyl)-2-oxofuran-3(2*H*)-ylidene]benzohydrazide (4j).** Yield 2.88 g (72%), orange crystals, mp 237–238°C (from $CHCl_3$). IR spectrum, ν, cm^{-1}: 3281 br (NH), 1776 (C^2=O), 1669

(CONH). ^{1}H NMR spectrum, δ, ppm: 3.87 s (3H, MeO), 6.55 s (1H, 4-H), 7.50 m (8H, H_{arom}), 12.81 s (1H, NH). Found, %: C 53.84; H 3.36; N 7.05. $C_{18}H_{13}BrN_2O_4$. Calculated, %: C 53.89; H 3.27; N 6.98.

4-Bromo-*N*′-[5-(4-chlorophenyl)-2-oxofuran-3(2*H*)-ylidene]benzohydrazide (4k). Yield 2.88 g (79%), orange crystals, mp 267–269°C (from $CHCl_3$). IR spectrum, ν, cm^{-1}: 3288 br (NH), 1774 (C^2=O), 1672 (CONH). ^{1}H NMR spectrum, δ, ppm: 6.99 s (1H, 4-H), 7.50 m (8H, H_{arom}), 12.84 s (1H, NH). Found, %: C 50.29; H 2.55; N 6.85. $C_{17}H_{10}BrClN_2O_3$. Calculated, %: C 50.34; H 2.49; N 6.91.

***N*′-[5-(4-Methoxyphenyl)-2-oxofuran-3(2*H*)-ylidene]-4-nitrobenzohydrazide (4l).** Yield 2.79 g (76%), orange crystals, mp 249–250°C (from $CHCl_3$). IR spectrum, ν, cm^{-1}: 3282 br (NH), 1778 (C^2=O), 1671 (CONH). ^{1}H NMR spectrum, δ, ppm: 3.83 s (3H, MeO), 7.14 s (1H, 4-H), 7.80 m (8H, H_{arom}), 12.06 s (1H, NH). Found, %: C 58.79; H 3.51; N 11.49. $C_{18}H_{13}N_3O_6$. Calculated, %: C 58.86; H 3.57; N 11.44.

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