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Chemistry of Iminofurans: V.*
Synthesis, Structure, and Cyclization of 4-R-4-Oxo-2-[2-(2-oxo-1,2-diphenylethylidene)hydrazino]but-2-enoic Acids

O.A. Komarova[a], **N.M. Igidov**[b], **A.E. Rubtsov**[a], **V.V. Zalesov**†[a],
A. S. Makarov[b], **and Yu.S. Toksarova**[b]

[a]*Perm State University, Perm, 614990 Russia*
e-mail: Alekhsandr.Rubtsov@psu.ru
[b]*Perm State Farmaceutical Academy Perm, Russia*

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Abstract—Reactions of 1,2-diphenylethane-1,2-dione hydrazone with 4-aryl-2-hydroxy-4-oxobut-2-enoic and 2-hydroxy-5,5-dimethyl-4-oxohex-2-enoic acids provided 4-aryl-4-oxo-2-[2-(2-oxo-1,2-diphenylethylidene)hydrazino]but-2-enoic and 5,5-dimethyl-4-oxo-2-[2-(2-oxo-1,2-diphenylethylidene)hydrazino]hex-2-enoic acids. The acids derivatives can exist in solutions as *Z*- and *E*-enehydrazino- or β-ketohydrazone forms, and under the treatment with acetic anhydride they undergo cyclization into 5-aryl- and 5-*tert*-butyl-3-[2-(2-oxo-1,2-diphenylethylidene)hydrazono]-2,3-furandiones.

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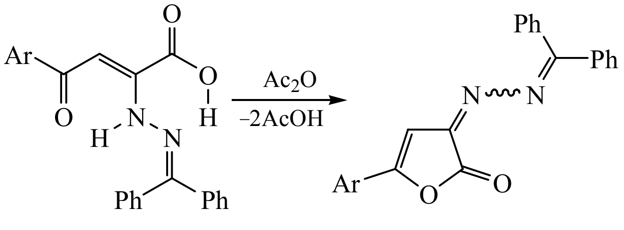
It was shown formerly that 4-aryl-2-hydroxy-4-oxobut-2-enoic acids **Ia–If** with benzophenone hydrazone formed (2*Z*)-4-aryl-2-[2-(diphenylmethylene)hydrazino]-4-oxobut-2-enoic acids **II** which in the polar solvent existed partially in the β-ketohydrazone form: 4-aryl-2-[(diphenylmethylene)hydrazono]-4-oxobutanoic acids [2] (Scheme 1). Compounds **II** in acetic anhydride underwent cyclization into 5-aryl-3-diphenylmethylenehydrazono-3*H*-furan-2-ones [2]. Compounds **II** in [2] are presented only by five examples, no information is given on their spontaneous or thermal intramolecular cyclization into N-substituted 5-aryl-3-hydrazono-3*H*-furan-2-ones in contrast to the similar in structure 4-aryl(heteryl)-2-arylamino--4-oxobut-2-enoic acids whose cyclization into 5-R-3-arylimino-3*H*-furan-2-ones is mentioned in [1, 3].

Scheme 1.



IIa–IIf

R = Ph (**a**), 4-MeC$_6$H$_4$ (**b**), 4-MeOC$_6$H$_4$ (**c**), 4-EtOC$_6$H$_4$ (**d**), 4-ClC$_6$H$_4$ (**e**), *t*-Bu (**f**).

We carried out research on the synthesis of N-substituted 4-R-2-hydrazino-4-oxobut-2-enoic acids and their tautomerism.

First from 1,2-diphenyl-1,2--ethanedione hydrazone and 4-aryl-2-hydroxy-4-oxobut-2-enoic acids **Ia–Ie** we obtained 4-aryl-4-oxo-2-[2-(2-oxo-1,2-diphenylethylidene)hydrazino]but-2-enoic acids **IIIa–IIIe** in 21–79% yields (see the scheme). Alongside the main reaction products we also isolated from the reaction mixture 1-[(2-oxo-1,2--diphenylethylidene)hydrazone]-1,2-diphenylethane-1,2--dione (benzil azine). The ratio of the main product and azine depended on the substituent in the aromatic ring of initial acids. For instance, in the reaction with 2-hydroxy-4-oxo-4-(4-fluorophenyl)but-2-enoic acid the target product did not form, and the product of the reaction of 4-(4-bromophenyl)-2-hydroxy-4oxobut-2-enoic acid totally rearranged into the benzil azine in the course of isolation and purification. In reactions of the other acids the azine formation was negligible.

In the IR spectra of compounds **IIIa–IIIe** recorded

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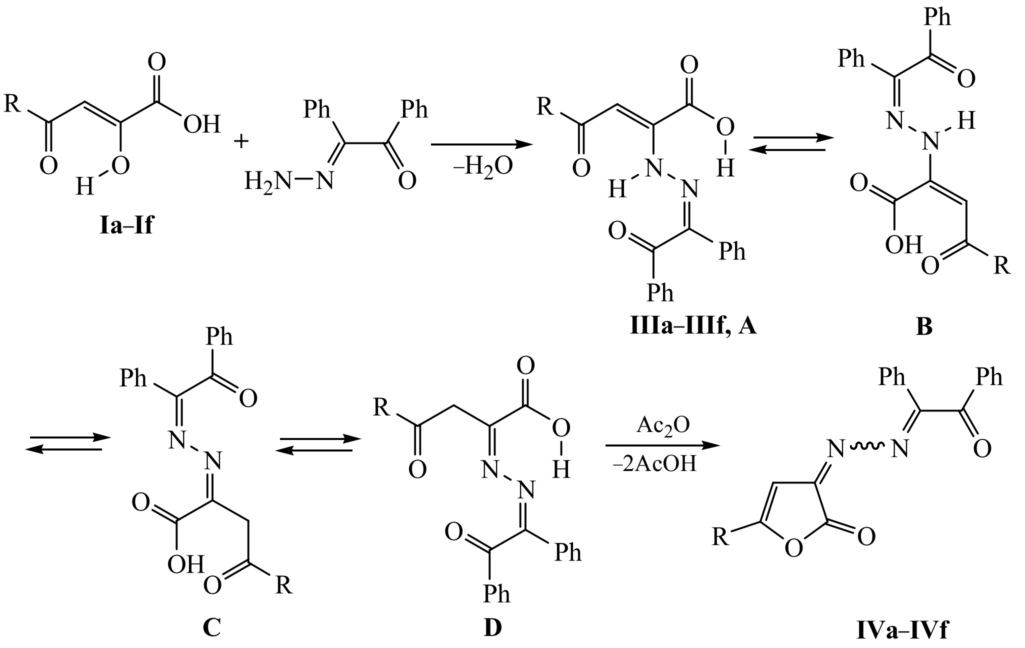
[*] For Communication IV, see [1].

† Deceased.

from mulls in mineral oil the absorption band of NH group was observed in the region 3239–3258 cm^{-1}. The absorption band of the carbonyl from the carboxy groups of compounds **IIIa–IIIe** is present in the region 1700–1707 cm^{-1}, and the band of the stretching vibrations of the ketone carbonyl, in the region 1655–1662 cm^{-1}. Like in the spectra of analogous compounds **II**, in compounds **IIIa–IIIe** due to the formation of an intramolecular hydrogen bond between the hydrogen atom of the enehydrazino group and the oxygen of the γ-carbonyl group C^4=O the absorption band of the latter is shifted to lower frequencies, 1590–1620 cm^{-1}, and it is impossible to separate it from the group of the absorption bands.

According to ^{1}H NMR data compounds **IIIa–IIIe** in DMSO-d_6 exist in *Z*- and *E*-enehydrazino forms **A**, **B** and β-ketohydrazone forms **C** and **D**, with form **A** prevailing, in contrast to the analogous acids **II** which contain a phenyl instead of a benzoyl substituent at the carbon atom of the C=N group; acids **II** in DMSO-d_6 exist in two forms (Scheme 2). Form **A** is characterized by the presence in the ^{1}H NMR spectrum of a singlet of the vinyl proton C^3–H in the range 6.21–6.43 ppm, of a multiplet of aromatic protons centered at 7.45–7.65 ppm, a singlet of NH group at 12.71–12.77 ppm, and also of a broadened singlet of the carboxy group proton in the spectra of compounds **IIIa, IIIb, IIId** at 14.25, 14.22, and 14.14 ppm respectively. The downfield position of the singlet belonging to the proton of the NH group is apparently caused by the formation of the intramolecular hydrogen bond with the carbonyl group C^4=O of the acid and (or) with the carbonyl group C^2=O of the substituent at the nitrogen atom. We failed to detect the signal of the proton of carboxy group in compounds **IIIc** and **IIIe** evidently due to its excessive broadening. Form **B** is characterized by the presence of a singlet from the vinyl proton C^3–H at 6.13–6.27 ppm, the multiplet of aromatic protons centered at 7.55–7.65 ppm, and a singlet of NH group at 13.16–13.23 ppm. The location of the singlet of NH proton is shifted downfield compared to the spectrum of form **A**. Formerly in the spectra of aroylacetones hydrazones the signal of the NH group of form **B** was observed upfield from the corresponding signal of form **A** [4]. This downfield position of the NH proton signal in the spectrum of form **B** of compounds **III** may indicate the formation of an intramolecular hydrogen bond with the carbonyl group of the benzoyl substituent attached to the C=N group. In the spectrum of β-ketohydrazone form **C** the signals of vinyl proton and that of the NH group are absent, a two-proton singlet is present from the C^3H_2 group at 4.53–4.59 ppm and a multiplet of aromatic protons centered at 7.45–7.65 ppm. In the ^{1}H NMR spectrum of form **D** appears a two-proton singlet of the

Scheme 2.



R = Ph (**a**), 4-MeC_6H_4 (**b**), 4-$MeOC_6H_4$ (**c**), 4-$EtOC_6H_4$ (**d**), 4-ClC_6H_4 (**e**), *t*-Bu (**f**).

C^3H_2 group at 4.31–4.38 ppm and a multiplet of aromatic protons centered at 7.45–7.65 ppm. ^1H NMR spectra do not allow an unambiguous assignment of signals to a definite ketohydrazone form (**C** or **D**), however, considering the data obtained in the study of hydrazones of esters of 2,4-dioxobutanoic acids [5, 6], it is possible to suggest that form **C** is predominant in the solution.

The replacement of the aryl fragment in acids **Ia–Ie** by the *tert*-butyl group did not change the direction of the reaction, and 2-hydroxy-5,5-dimethyl-4-oxohex-2-enoic acid (**If**) with benzil hydrazone in toluene afforded 5,5-dimethyl-4-oxo-2-[2-(2-oxo-1,2-diphenylethylidene)-hydrazino]hex-2-enoic acid (**IIIf**). In acetic acid, similarly to the reactions of 4-(4-fluorophenyl)-, 4-(4-bromophenyl)-2-hydroxy-4-oxobut-2-enoic acids, the process is directed to the benzil azine formation, apparently due to the rearrangement of the arising acid **IIIf** or because of predominant formation of azine under the conditions of hard acid catalysis.

In the IR spectrum of compound **IIIf** recorded from a mull in mineral oil lacks the absorption band of the NH group in the region 3200–3600 cm^{-1}. The absorption band of the carbonyl of the carboxy group is observed in the region 1699 cm^{-1}, and the bands of the stretching vibrations of the ketone carbonyls are present in the region 1681 and 1662 cm^{-1}. The absence of an amino group and the high-frequency position of the band of C^4=O carbonyl shows that acid **IIIf** in the crystal state exists in the β-ketohydrazone form **C**.

According to ^1H NMR spectra, compound **IIIf** exists in DMSO-d_6 in *Z*- and *E*-enehydrazino fotms **A**, **B** and β-ketohydrazone forms **C** and **D**, with form **C** prevailing. Form **A** is characterized by the presence in the ^1H NMR spectrum of a singlet of the vinyl proton C^3–H at 5.79 ppm, of a multiplet of aromatic protons centered at 7.55 ppm, a singlet of NH group at 12.24 ppm, and also of a broadened singlet of the carboxy group proton at 13.21 ppm. Form **B** is characterized by the presence in the ^1H NMR spectrum of a singlet of the vinyl proton C^3–H at 6.65 ppm, of a multiplet of aromatic protons centered at 7.55 ppm, a singlet of NH group at 12.78 ppm. In the spectrum of β-ketohydrazone form **C** the signals of vinyl proton and that of the NH group are absent, a two-proton singlet is present from the C^3H_2 group at 4.12 ppm and a multiplet of aromatic protons centered at 7.55 ppm. In the ^1H NMR spectrum of form **D** appears a two-proton singlet of the C^3H_2 group at 3.91 ppm and a multiplet of aromatic protons centered at 7.55 ppm. The existence of compound **IIIf** in the crystal in the form **C** and its prevalence in the solution apparently is due to the lack of the stabilization of the six-membered H-chelate ring in the **A** form by aromatic substituent in the position *4*. As a result it proved to be more feasible to form a π-conjugated system $O=C^1–C^2=N–N=C(Ph)–C(Ph)=O$ involving the substituent at the nitrogen atom.

The intramolecular cyclization of acids **IIIa–IIIf** by boiling in acetic anhydride resulted in the corresponding 5-aryl- and 5-*tert*-butyl-3-[2-(diphenylmethylene)-hydrazono]-3*H*-furan-2-ones **IVa–IVf**.

Furanones **IVa–IVf** are brightly colored from yellow to dark red crystalline substances. The IR spectra of compounds **IVa–IVf** contain the absorption band in the region 1798–1814 cm^{-1} characteristic of the stretching vibrations of the lactone carbonyl of the furan ring, the absorption band of the carbonyl group from the substituent at the nitrogen atom at 1664–1676 cm^{-1}. In the ^1H NMR spectrum of compound **IVd** a three-proton triplet of the $\mathbf{CH_3}CH_2$ group is observed at 1,3 ppm, the two-proton quartet from $\mathbf{CH_3}CH_2$ group appears at 4.1 ppm, the singlet of vinyl proton C^4H, at 7.37 ppm, and a set of aromapic protons gives rise to a multiplet centered at 7.45 ppm.

EXPERIMENTAL

IR spectra were recorded on a spectrophotometer FSM-1201 from mulls in mineral oil. ^1H NMR spectra were registered on a spectrometer Varian Mercury Plus-300 (300.05 MHz) in DMSO-d_6, internal reference HMDS. The reaction progress was monitored and the purity of compounds was checked by TLC on Silufol UV-254 or Sorbfil plates, eluent ether–benzene–acetone, 10:9:1, the spots visualized in iodine vapor.

4-Oxo-2-(2-oxo-1,2-diphenylethylidene-hydrazino)-4-phenylbut-2-enoic acids IIIa–IIIf. To a solution of 0.02 mol of acid **Ia–If** in 20 ml of ethanol was added a solution of 0.02 mol of 1,2-diphenyl-1,2-ethanedione hydrazone in 20 ml of ethanol, the mixture was kept for 24 h at 20–25°C, then cooled to 0°C, the separated precipitate was filtered off and recrystallized from acetonitrile.

4-Oxo-2-(2-oxo-1,2-diphenylethylidene-hydrazino)-4-phenylbut-2-enoic acid (IIIa). Yield 1.7 g (21%), orange crystals, t.decomp. 144– 146°C (from acetonitrile). IR spectrum, ν, cm^{-1}: 3258 (NH), 1707 (COO), 1658 (CO). ^1H NMR spectrum, δ, ppm, form **A** (62.5%): 6.43 s (1H, CH), 7.65 m (15H, 3C_6H_5), 12.77 s (1H, NH), 14.25 br.s (1H, OH); form **B** (13.1%): 6.29 s (1H, CH), 7.65 m (15H, 3C_6H_5), 13.20 s (1H, NH), 14.25 br.s (1H, OH); form **C** (18.4%): 4.59 s (2H, CH_2), 7.65 m (15H, 3C_6H_5), 14.25 br.s (1H, OH); form **D** (6%): 4.43 s (2H, CH_2), 7.65 m (15H, 3C_6H_5), 14.25 br.s (1H, OH). Found, %: C 72.40; H 4.54; N 7.03. $C_{24}H_{18}N_2O_4$. Calculated, %: C 72.36; H 4.52; N 7.04.

4-(4-Methylphenyl)-4-oxo-2-(2-oxo-1,2-diphenylethylidenehydrazino)but-2-enoic acid (IIIb). Yield 3.9 g (47%), orange crystals, t.decomp. 139–140°C (from acetonitrile). IR spectrum, ν, cm^{-1}: 3246 (NH), 1700 (COO), 1655 (CO). ^{1}H NMR spectrum, δ, ppm, form **A** (68%): 2.33 s (3H, CH_3), 6.21 s (1H, CH), 7.55 m (14H, 2C_6H_5, C_6H_4), 12.76 s (1H, NH), 14.22 br.s (1H, OH); form **B** (13%): 2.36 s (3H, CH_3), 6.13 s (1H, CH), 7.55 m (14H, 2C_6H_5, C_6H_4), 13.21 s (1H, NH), 14.22 br.s (1H, OH); form **C** (18%): 2.40 s (3H, CH_3), 4.55 s (2H, CH_2), 7.45 m(14H, 2C_6H_5, C_6H_4), 14.22 br.s (1H, OH); form **D**(1%): 2.38 C (3H, CH_3), 4.39 s (2H, CH_2), 7.45 m (14H, 2C_6H_5, C_6H_4), 14.22 br.s (1H, OH). Found, %: C 72.84; H 4.86; N 6.82. $C_{25}H_{20}N_2O_4$. Calculated, %: C 72.82; H 4.85; N 6.80.

4-(4-Methoxyphenyl)-4-oxo-2-(2-oxo-1,2-diphenylethylidenehydrazino)but-2-enoic acid (IIIc). Yield 3.8 g (44%), orange crystals, t.decomp. 168–171°C (from acetonitrile). IR spectrum, ν, cm^{-1}: 3275 (NH), 1700 (COO), 1660 (CO). ^{1}H NMR spectrum, δ, ppm, form **A** (74.6%): 3.76 s (3H, CH_3O), 6.36 s (1H, CH), 7.5 m (14H, 2C_6H_5, C_6H_4), 12.73 s (1H, NH); form **B** (9.4%): 3.78 s (3H, CH_3O), 6.21 s (1H, CH), 7.45 m (14H, 2C_6H_5, C_6H_4), 13.2 s (1H, NH); form **C** (15%): 3.81 s (3H, CH_3O), 4.49 (4.31) s (2H, CH_2), 7.45 m (14H, 2C_6H_5, C_6H_4); form **D** (1%): 3.81 s (3H, CH_3O), 4.31 C (2H, CH_2), 7.45 m (14H, 2C_6H_5, C_6H_4). Found, %: C 70.07; H 4.69; N 6.55. $C_{25}H_{20}N_2O_5$. Calculated, %: C 70.09; H 4.67; N 6.54.

4-Oxo-2-(2-oxo-1,2-diphenylethylidenehydrazino)-4-(4-ethoxyphenyl)but-2-enoic acid (IIId). Yield 3.6 g (41%), orange crystals, t.decomp. 157–159°C (from acetonitrile). IR spectrum, ν, cm^{-1}: 3239 (NH), 1665 (COO, CO). ^{1}H NMR spectrum, δ, ppm, form **A** (40.5%): 1.33 m (3H, CH_3CH_2O), 4.12 m (2H, CH_3CH_2O), 6.39 s (1H, CH), 7.59 m (14H, 2C_6H_5, C_6H_4), 12.77 s (1H, NH), 14.14 br.s (1H, OH); form **B** (21%): 1.33 m (3H, CH_3CH_2O), 4.12 m (2H, CH_3CH_2O), 6.25 s (1H, CH), 7.59 m (14H, 2C_6H_5, C_6H_4), 13.23 s (1H, NH), 14.14 br.s (1H, OH); form **C** (35.5%): 1.33 m (3H, CH_3CH_2O), 4.12 m (2H, CH_3CH_2O), 4.53 s (2H, CH_2), 7.59 m (14H, 2C_6H_5, C_6H_4), 14.14 br.s (1H, OH); form **D** (3%): 1.33 m (3H, CH_3CH_2O), 4.12 m (2H, CH_3CH_2O), 4.36 s (2H, CH_2), 7.59 m (14H, 2C_6H_5, C_6H_4), 14.14 br.s (1H, OH). Found, %: C 70.58; H 4.99; N 6.33. $C_{26}H_{22}N_2O_5$. Calculated, %: C 70.59; H 4.98; N 6.33.

4-Oxo-2-(2-oxo-1,2-diphenylethylidenehydrazino)-4-(4-chlorophenyl)but-2-enoic acid (IIIe). Yield 6.8 g (79%), orange crystals, t.decomp. 147–148°C (from acetonitrile). IR spectrum, ν, cm^{-1}: 3257 (NH), 1705 (COO), 1662 (CO). ^{1}H NMR spectrum, δ, ppm, form **A** (60%): 6.39 s (1H, CH), 7.6 m (14H, 2C_6H_5, C_6H_4), 12.71 s (1H, NH); form **B** (16%): 6.27 s (1H, CH), 7.60 m (14H, 2C_6H_5, C_6H_4), 13.16 s (1H, NH); form **C** (19.5%): 4.54 s (2H, CH_2), 7.6 m (14H, 2C_6H_5, C_6H_4); form **D** (4.5%): 4.36 s (2H, CH_2), 7.6 m (14H, 2C_6H_5, C_6H_4). Found, %: C 66.60; H 3.92; N 6.48. $C_{24}H_{17}ClN_2O_4$. Calculated, %: C 66.59; H 3.93; N 6.47.

5,5-Dimethyl-4-oxo-2-[2-(2-oxo-1,2-diphenylethylidene)hydrazino]hex-2-enoic acid (IIIf). Yield 5.07 g (67%), yellow crystals, t.decomp. 135–136°C (from toluene). IR spectrum, ν, cm^{-1}: 3257, 1699 (COO), 1681 (CO). ^{1}H NMR spectrum, δ, ppm, form **A**(24.9%): 1.01 s (9H, 3CH_3), 5.79 s (1H, CH), 7.55 m(10H, 2C_6H_5), 12.24 s (1H, NH), 13.21 br.s (1H, OH); form **B** (18.1%): 1.03 s (9H, 3CH_3), 5.65 s (1H, CH), 7.55 m (10H, 2C_6H_5), 12.78 s (1H, NH), 13.21 br.s(1H, OH); form **C** (54.2%): 1.18 s (9H, 3CH_3), 4.12 s(2H, CH_2), 7.55 m (10H, 2C_6H_5), 13.21 br.s (1H, OH); form **D** (2.8%): 1.18 s (9H, 3CH_3), 3.91 s (2H, CH_2), 7.55 m (10H, 2C_6H_5), 13.21 br.s (1H, OH). Found, %: C 69.86; H 5.84; N 7.40. $C_{22}H_{22}N_2O_4$. Calculated, %: C 69.84; H 5.82; N 7.41.

3-(2-Oxo-1,2-diphenylethylidenehydrazono)-5-phenyl-3*H*-furan-2-ones IVa–IVf. To 0.01 mol of acid **IIIa–IIIf** was added 10 ml of acetic anhydride, and the mixture was heated to boiling, On cooling the mixture to 0°C the separated precipitate was filtered off and recrystallized from toluene.

3-(2-Oxo-1,2-diphenylethylidenehydrazono)-5-phenyl-3*H*-furan-2-one (VIa). Yield 3.7 g (97%), orange crystals, mp 159–160°C (from toluene). IR spectrum, ν, cm^{-1}: 1804 ($C^2{=}O_{lactone}$), 1664 (C=O), 1592, 1578 (C=N, C=C). Found, %: C 75.77; H 4.20; N 7.37. $C_{24}H_{16}N_2O_3$. Calculated, %: C 75.79; H 4.21; N 7.37.

5-(4-Methylphenyl)-3-(2-oxo-1,2-diphenylethylidenehydrazono)-3*H*-furan-2-one (IVb). Yield

3.9 g (99%), orange crystals, mp 184–186°C(from toluene). IR spectrum, ν, cm^{-1}: 1809 ($C^2=O_{lactone}$), 1670 (C=O), 1591, 1570 (C=N, C=C). Found, %: C 76.17; H 4.59; N 7.10. $C_{25}H_{18}N_2O_3$. Calculated, %: C 76.14; H 4.57; N 7.11.

5-(4-Methoxyphenyl)-3-(2-oxo-1,2-diphenylethylidenehydrazono)-3*H*-furan-2-one (IVc). Yield 4.0 g (97%), dark red crystals, mp 93–95°C (from toluene). IR spectrum, ν, cm^{-1}: 1804 ($C^2=O_{lactone}$), 1668 (C=O), 1589, 1574 (C=N, C=C). Found, %: C 73.18; H 4.38; N 6.83. $C_{25}H_{18}N_2O_4$. Calculated, %: C 73.17; H 4.39; N 6.83.

3-(2-Oxo-1,2-diphenylethylidenehydrazono)-5-(4-ethoxyphenyl)-3*H*-furan-2-one (IVd). Yield 3.9 g (93%), orange crystals, mp 160–161°C(from toluene). IR spectrum, ν, cm^{-1} : 1811 (C=O), 1668 (C=O), 1591, 1574 (C=N, C=C). ^{1}H NMR spectrum, δ, ppm: 1.3 t (3H, CH_3), 4.10 q (2H, CH_2),7.37 s (1H, CH), 7.45 m (14H, $2C_6H_5$, C_6H_4). Found, %: C 73.59; H 4.74; N 6.58. $C_{26}H_{20}N_2O_4$. Calculated, %: C 73.58; H 4.72; N 6.60.

3-(2-Oxo-1,2-diphenylethylidenehydrazono)-5-(4-chlorophenyl)-3*H*-furan-2-one (IVe). Yield 4.1 g (98%), orange crystals, mp 174–175°C(from toluene). IR spectrum, ν, cm^{-1}: 1814 (C=O), 1664 (C=O), 1593, 1575 (C=N, C=C) . Found, %: C 69.46; H 3.60; N 6.78. $C_{24}H_{15}ClN_2O_3$. Calculated, %: C 69.48; H 3.62; N 6.76.

5-*tert*-Butyl-3-(2-oxo-1,2-diphenylethylidenehydrazono)-3*H*-furan-2-one (IVf). Yield 3.03 g (84%), orange crystals, mp 143–144°C (from toluene–hexane). IR spectrum, ν, cm^{-1}: 1798 ($C^2=O_{lactone}$), 1676 (C=O). Found, %: C 73.31; H 5.54; N 7.74. $C_{22}H_{20}N_2O_3$. Calculated, %: C 73.33; H 5.56; N 7.78.

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