

Synthesis and Intramolecular Cyclization of N-Substituted 2-Amino-4-aryl-4-oxo-2-butenic Acids

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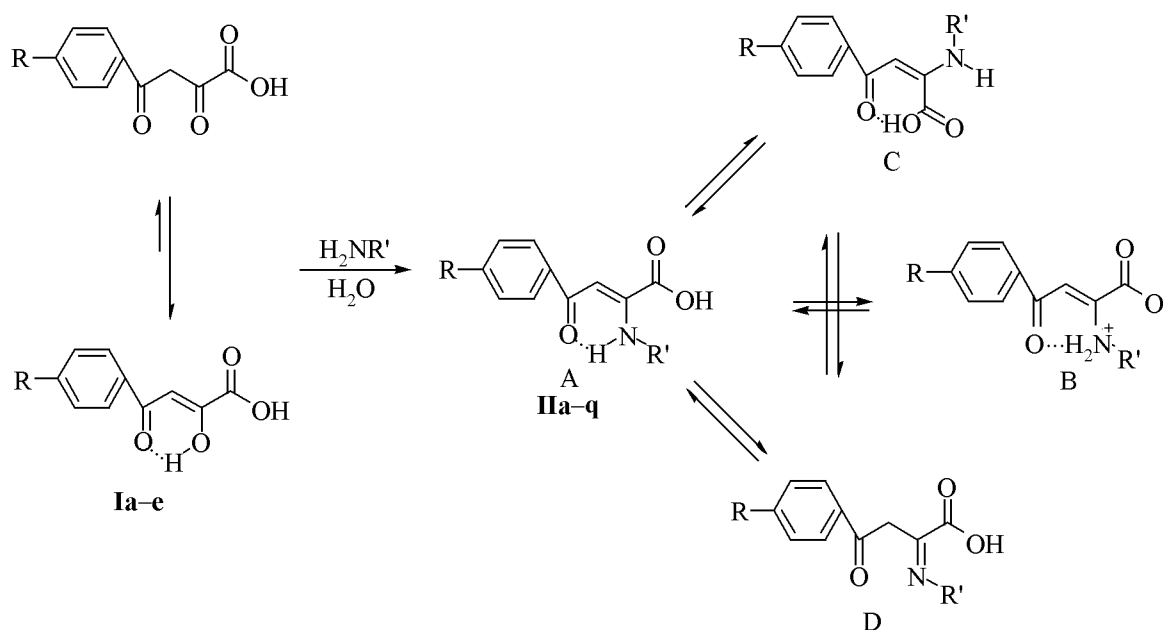
Abstract—Treating 4-aryl-2,4-dioxobutanoic acids with aromatic amines, 4-aminoantipyrine, and benzophenone hydrazone furnished N-substituted 2-amino-4-aryl-4-oxo-2-butenic acids that existed in solutions in enaminoketone and iminoketone forms. The acids obtained underwent in the presence of acetic anhydride cyclization into N-substituted 5-aryl-3-imino-3H-furan-2-ones.

Almost no published data exist on chemical reactions of 5-aryl-3-imino-3H-furan-2-ones evidently because no reliable synthetic method for preparation of such compounds have been developed up till now. For instance, derivatives of 3-R-hydrazone-3H-furan-2-ones were mentioned in [1-3], and syntheses of 5-heteryl-3-arylamino-3H-furan-2-ones and 5-aryl-3-heterylamino-3H-furan-2-ones were described in [4] and [5] respectively. Therewith this rare type of 2-furanone derivatives seems very promising because of high reactivity and possibility to find in the series

of furan derivatives substances exhibiting biological activity. The target of this study was a development of a versatile synthetic method for preparation both of 3-R-hydrazone- and 3-R-imino-5-aryl-3H-furanones.

First by reaction of 4-aryl-2,4-dioxobutanoic acids **Ia-e** with arylamines, 1-naphthylamine, 4-aminoantipyrine, and benzophenone hydrazone we prepared respectively 2-arylamino-, 2-(1-naphthylamino)-, 2-(2,3-dimethyl-5-oxo-1-phenylpyrazolin-4-ylamino)-, and 2-diphenylhydrazino-4-aryl-4-oxo-2-butenic acids **IIa-q** (Scheme 1, Tables 1, 2).

Scheme 1.



I, R = H (**a**), CH₃ (**b**), CH₃O (**c**), Cl (**d**), Br (**e**); **II**, R' = C₆H₅: R = H (**a**), CH₃ (**b**), CH₃O (**c**), Cl (**d**), Br (**e**); R = CH₃, R' = 4-ClC₆H₄ (**f**); R = H, R' = Napht (**g**); R' = 4-Ant: R = H (**h**), CH₃ (**i**), CH₃O (**j**), Cl (**k**), Br (**l**); R' = (C₆H₅)₂C=N: R = H (**m**), CH₃ (**n**), CH₃O (**o**), Cl (**p**), Br (**q**). Napht = 1-naphthyl, 4-Ant = 1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-on-4-yl.

Table 1. Yields, decomposition temperature, and elemental analyses of compounds **II**f–**q**, **III**a–**m**

Compd. no.	Yield, %	T _{decomp} , °C	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
II f	87	175–176 (ethanol)	72.60	5.40	5.00	C ₁₇ H ₁₅ NO ₃	72.58	5.37	4.98
II g	94	157–158 (ethanol)	75.73	4.72	4.42	C ₂₀ H ₁₅ NO ₃	75.70	4.76	4.41
III h	95	189–189.5 (ethanol)	67.20	5.10	11.20	C ₂₁ H ₁₉ N ₃ O ₄	67.19	5.07	11.19
III i	91	171–172 (methanol)	67.49	5.45	10.75	C ₂₂ H ₂₁ N ₃ O ₄	67.51	5.41	10.74
II j	89	158–160 (ethanol)	65.40	5.23	9.98	C ₂₂ H ₂₁ N ₃ O ₅	65.39	5.20	9.95
III k	94	182–184 (chloroform)	61.21	4.44	10.20	C ₂₁ H ₁₈ N ₃ O ₄ Cl	61.24	4.41	10.20
III l	93	170–171 (ethanol)	56.83	3.95	8.62	C ₂₁ H ₁₈ N ₃ O ₄ Br	56.80	3.98	8.64
III m	90	180–181 (ethanol)	74.60	4.92	7.57	C ₂₃ H ₁₈ N ₂ O ₃	74.58	4.90	7.56
III n	86	176–177 (acetonitrile)	75.00	5.20	7.30	C ₂₄ H ₂₀ N ₂ O ₃	74.98	5.24	7.29
III o	91	178–179 (acetonitrile)	71.97	5.00	7.00	C ₂₄ H ₂₀ N ₂ O ₄	71.99	5.03	7.00
III p	85	185–186 (acetonitrile)	68.23	4.25	6.95	C ₂₃ H ₁₇ ClN ₂ O ₃	68.24	4.23	6.92
III q	90	187–188 (acetonitrile)	61.51	3.86	6.25	C ₂₃ H ₁₇ BrN ₂ O ₃	61.48	3.81	6.23
III a	69	165–166 (acetyl acetate)	77.56	5.01	5.33	C ₁₇ H ₁₃ NO ₂	77.55	4.98	5.32
III b	45	198–199 (toluene)	68.60	4.11	4.72	C ₁₇ H ₁₂ ClNO ₂	68.58	4.06	4.70
III c	71	173–173.5 (toluene)	80.27	4.43	4.70	C ₂₀ H ₁₃ NO ₂	80.25	4.38	4.68
III d	80	235–235.5 (toluene)	70.20	4.73	11.68	C ₂₁ H ₁₇ N ₃ O ₃	70.18	4.77	11.69
III e	75	247–248 (toluene)	70.78	5.09	11.23	C ₂₂ H ₁₉ N ₃ O ₃	70.76	5.13	11.25
III f	73	260–261 (toluene)	67.87	4.89	10.78	C ₂₂ H ₁₉ N ₃ O ₄	67.86	4.92	10.79
III g	78	250–251 (toluene)	64.03	4.11	10.65	C ₂₁ H ₁₆ ClN ₃ O ₃	64.05	4.09	10.67
III h	69	255–256 (toluene)	57.57	3.65	9.60	C ₂₁ H ₁₆ BrN ₃ O ₃	57.55	3.68	9.59
III i	82	159–160 ^a (benzene–hexane)	78.40	4.60	7.97	C ₂₃ H ₁₆ N ₂ O ₂	78.39	4.58	7.95
III j	74	161–162 ^a (benzene–hexane)	78.65	4.94	7.65	C ₂₄ H ₁₈ N ₂ O ₂	78.67	4.95	7.65
III k	84	146–147 ^a (benzene–hexane)	75.39	4.75	7.35	C ₂₄ H ₁₈ N ₂ O ₃	75.38	4.74	7.33
III l	81	85–86 ^a (benzene–hexane)	71.40	3.95	7.22	C ₂₃ H ₁₅ ClN ₂ O ₂	71.41	3.91	7.24
III m	90	140–141 ^a (benzene–hexane)	64.02	3.49	6.52	C ₂₃ H ₁₅ BrN ₂ O ₂	64.05	3.51	6.50

^a For these compounds are given melting points.

In the IR spectra of acids **II**a–**g**, **m**–**q** is present a broad absorption band in the region 3201–3275 cm⁻¹ (compounds **II**a–**g**, **n**, **o**, **q**) or a plateau in the region 3100–3250 cm⁻¹ (compounds **III**m, **p**) characteristic of an amino group. In the carbonyl spectral region of acids **II**a–**g**, **m**–**q** absorption bands are lacking, but in the region 1585–1636 cm⁻¹ is present a group of broad bands with a wide sloping shoulder at the side of higher frequencies from 1615 to 1700 cm⁻¹. This pattern of IR spectra suggests that compounds **II**a–**g**, **m**–**q** exist in crystals in a B form with a ionized carboxy group and with γ -carbonyl C⁴=O involved into an intramolecular hydrogen bond. Unlike that in the IR spectra of acids **III**h–**l** are present two broad absorption bands in the region 3458–3468 and 3414–3417 cm⁻¹ (compounds **III**h, **k**) or a plateau in the region 3417–3467 cm⁻¹ (compounds **III**i, **j**, **l**) characteristic of NH group, an absorption band at 1730–1735 cm⁻¹ belonging to the stretching vibrations of a carboxy group, and a group of absorption bands in

the region 1587–1669 cm⁻¹ from lactam carbonyl of the heterocycle, carbonyl group C⁴=O involved into an intramolecular hydrogen bond, and groups C=N, C=C; assignment of the latter bands is difficult. The appearance of the carbonyl absorption band in the IR spectra and high-frequency absorption of the NH group may suggest that compounds **III**h–**m** exist in form C with an amino group not involved into a hydrogen bond. The different pattern of IR spectra of compounds **II**a–**g**, **m**–**q** and **III**h–**l** is probably due to the presence in the latter compounds of a bulky heterocyclic substituent preventing intramolecular or intermolecular protonation of the enamine nitrogen.

We investigated the ¹H NMR spectra of compounds **II**g–**q** dissolved in DMSO-*d*₆. It was established that acids **II**g–**l** existed in solutions as equilibrium mixtures of enamino-ketone forms A and C, i.e. as *Z*,*E*-isomers with *Z*-isomer prevailing (its content amounted to 80–91%). The spectrum of A(*Z*) form contains a broadened signal from the

Table 2. IR and ¹H NMR spectra of compounds **III-f-q**, **IIIa-m**

Compd. no.	IR spectra, ν , cm^{-1}	¹ H NMR spectra, δ , ppm	A: C(D), %
III-f	3201 (NH), 1700, 1653, 1635, 1598 (C ⁴ =O, C=N, C=C)	A: 6.53 s (1H, CH), 7.65 m (12H, C ₆ H ₅ , C ₁₀ H ₇), 12.63 s (1H, NH), 13.7 br.s (1H, COOH)	88:12
III-g	3275 br (NH), 1700, 1650, 1600 br (C ⁴ =O, C=N, C=C)	C: 5.88 s (1H, CH), 7.65 m (12H, C ₆ H ₅ , C ₁₀ H ₇), 10.3 s (1H, NH)	88:12
III-h	3415 (NH), 1734 (COOH), 1653, 1638, 1616, 1605, 1587 (C ³ =O pyr, C ⁴ =O, C=N, C=C)	A: 2.32 s (3H, CCH ₃), 3.09 s (3H, NCH ₃), 6.44 s (1H, CH), 7.60 m (10H, 2C ₆ H ₅), 11.63 s (1H, NH), 13.3 br.s (1H, COOH)	88:12
III-i	3467 sh, 3416 (NH), 1734 (COOH), 1653, 1635, 1632, 1624, 1617, 1599 (C ³ =O pyr, C ⁴ =O, C=N, C=C)	C: 2.25 s (3H, CCH ₃), 3.23 s (3H, NCHd3), 6.13 s (1H, CH), 7.60 m (10H, 2C ₆ H ₅), 9.71 s (1H, NH)	88:12
III-j	3417 sh (NH), 1734 (COOH), 1669, 1663, 1635, 1594 (C ³ =O pyr, C ⁴ =O, C=N, C=C)	A: 2.31 s (3H, CCH ₃), 2.41 s (1H, C-CH ₃), 3.09 s (3H, NCH ₃), 6.42 s (1H, CH), 7.60 m (9H, C ₆ H ₅ , C ₆ H ₄), 11.52 s (1H, NH), 13.3 br.s (1H, COOH)	81:19
III-k	3458, 3417 (NH), 1734 (COOH), 1669, 1663, 1635, 1603 (C ³ =O pyr, C ⁴ =O, C=N, C=C)	C: 2.25 s (3H, CCH ₃), 3.21 s (1H, C-CH ₃), 3.21 s (3H, NCH ₃), 6.10 s (1H, CH), 7.60 m (9H, C ₆ H ₅ , C ₆ H ₄), 9.51 s (1H, NH)	91:9
III-l	3427 sh (NH), 1735 (COOH), 1653, 1623, 1617, 1603 (C ³ =O pyr, C ⁴ =O, C=N, C=C)	A: 2.32 s (3H, CCH ₃), 3.11 s (3H, NCH ₃), 6.42 s (1H, CH), 7.60 m (9H, C ₆ H ₅ , C ₆ H ₄), 11.36 s (1H, NH), 13.1 br.s (1H, COOH)	86:14
III-m	3100–3250 plateau (NH), 1610 br, 1590 (C ⁴ =O, C=N, C=C)	C: 2.27 s (3H, CCH ₃), 3.23 s (3H, NCH ₃), 6.08 s (1H, CH), 7.60 m (9H, C ₆ H ₅ , C ₆ H ₄), 9.71 s (1H, NH)	80:20

Table 2. (Contd.)

Compd. no.	IR spectra, ν , cm^{-1}	^1H NMR spectra, δ , ppm	A: C(D), %
IIIa	3275 br (NH), 1615 br, 1590 ($\text{C}^d=\text{O}$, C=N, C=C)	A: 2.36 s (3H, CH_3), 6.04 c (1H, CH), 7.55 m (15H, $2\text{C}_6\text{H}_5$, C_6H_4), 12.69 s (1H, NH), D: 2.36 s (3H, CH_3), 4.39 s (2H, CH_2) 7.55 m (15H, $2\text{C}_6\text{H}_5$, C_6H_4)	83:17
IIIb	3249 (NH), 1623 br, 1617, 1599 ($\text{C}^d=\text{O}$, C=N, C=C)	A: 3.81 s (3H, O- CH_3), 6.02 s (1H, CH), 7.45 m (15H, $2\text{C}_6\text{H}_5$, C_6H_4), 12.64 s (1H, NH), D: 3.81 s (3H, O- CH_3), 4.38 s (2H, CH_2) 7.45 m (15H, $2\text{C}_6\text{H}_5$, C_6H_4)	79:21
IIIc	3100–3250 plateau (NH), 1615 br, 1600 ($\text{C}^d=\text{O}$, C=N, C=C)	A: 5.97 s (1H, CH), 7.60 m (15H, $2\text{C}_6\text{H}_5$, C_6H_4), 12.72 s (1H, NH), D: 4.39 s (2H, CH_2) 7.60 m (15H, $2\text{C}_6\text{H}_5$, C_6H_4)	77:23
IIId	3249 (NH), 1636, 1628, 1617, 1585 ($\text{C}^d=\text{O}$, C=N, C=C)	A: 5.97 s (1H, CH), 7.65 m (15H, $2\text{C}_6\text{H}_5$, C_6H_4), 12.75 s (1H, NH), D: 4.39 s (2H, CH_2) 7.65 m (15H, $2\text{C}_6\text{H}_5$, C_6H_4)	91:9
IIIe	1803 (C=O), 1605, 1585 (C=N, C=C)	2.43 s (3H, CH_3), 6.96 s (1H, CH), 7.40 m (9H, C_6H_5 , C_6H_4)	
IIIb	1799 (C=O), 1617, 1595 (C=N, C=C)	2.43 s (3H, CH_3), 6.89 s (1H, CH), 7.35 s (8H, $2\text{C}_6\text{H}_4$)	
IIIc	1815 (C=O), 1595 br (C=N, C=C)	6.95 s (1H, CH), 7.75 m (12H, C_6H_5 , C_{10}H_7)	
IIIId	1781 ($\text{C}^2=\text{O}$), 1654 ($\text{C}^3=\text{O}$ pyr) 1635, 1617, 1590 (C=N, C=C)	2.55 s (3H, CH_3), 3.41 s (3H, N CH_3), 7.55 m (10H, $2\text{C}_6\text{H}_5$), 7.85 s (1H, CH)	
IIIe	1782 ($\text{C}^2=\text{O}$), 1653 ($\text{C}^3=\text{O}$ pyr) 1638, 1617, 1615, 1607 (C=N, C=C)	2.40 (3H, CH_3), 2.55 s (3H, CH_3), 3.41 s (3H, N CH_3), 7.42 m (9H, C_6H_5 , C_6H_4), 7.74 s (1H, CH)	
IIIf	1773 ($\text{C}^2=\text{O}$), 1653 ($\text{C}^3=\text{O}$ pyr) 1646, 1636, 1617 (C=N, C=C)	2.55 s (3H, CH_3), 3.43 s (3H, N CH_3), 7.50 m (9H, C_6H_5 , C_6H_4), 7.87 s (1H, CH)	
IIIg	1785 ($\text{C}^2=\text{O}$), 1653, ($\text{C}^3=\text{O}$ pyr) 1636, 1633, 1617 (C=N, C=C)	2.55 s (3H, CH_3), 3.43 s (3H, N C_6H_3), 7.55 m (9H, C_6H_5 , C_6H_4), 7.86 s (1H, CH)	
IIIh	1781 ($\text{C}^2=\text{O}$), 1653, ($\text{C}^3=\text{O}$ pyr) 1636, 1633, 1617, 1615 (C=N, C=C)	7.11 s (1H, CH), 7.50 m (15H, $3\text{C}_6\text{H}_5$)	
IIIi	1812 (C=O), 1625, 1593 (C=N, C=C)	2.43 s (3H, CH_3), 7.16 s (1H, CH), 7.40 m (14H, $2\text{C}_6\text{H}_5$, C_6H_4)	
IIIj	1815 (C=O), 1620, 1595 (C=N, C=C)	3.89 s (3H, O CH_3), 6.92 s (1H, CH), 7.45 m (14H, $2\text{C}_6\text{H}_5$, C_6H_4)	
IIIk	1815 (C=O), 1620, 1590 (C=N, C=C)	7.20 s (1H, CH), 7.55 m (14H, $2\text{C}_6\text{H}_5$, C_6H_4)	
IIIl	1817 (C=O), 1620, 1595 (C=N, C=C)	7.21 s (1H, CH), 7.55 m (14H, $2\text{C}_6\text{H}_5$, C_6H_4)	
IIIm	1820 (C=O), 1620, 1590 (C=N, C=C)		

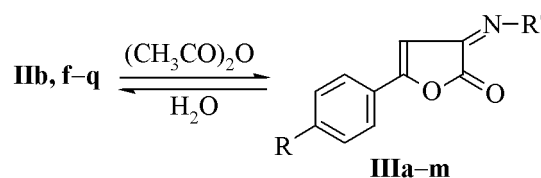
proton of COOH group at 13.1–13.7 ppm, a singlet of NH group proton involved into strong intramolecular hydrogen bond at 11.36–12.63 ppm, and a singlet of CH group proton at 6.39–6.53 ppm. The proton signals of NH and CH groups of the C(E) form appear in a stronger field at 9.51–10.30 and 5.88–6.39 ppm respectively, and the carboxy group proton of the C form is not found in the spectrum apparently due to its considerable broadening. It was shown formerly that compounds **IIa–e** could exist in solutions in A and C forms [6]. The ^1H NMR spectra of compounds **IIm–q** have another pattern. In the spectra appear signals from enamino-ketone form A(Z) and β -ketoimine form D. Characteristic signals of the A(Z) form are the singlet from the proton of NH group involved into an intramolecular hydrogen bond at 12.64–12.73 ppm and singlet of methine proton at 5.97–6.04 ppm, whereas the characteristic signal of the D form is the singlet of methylene group protons at 4.38–4.42 ppm. We failed to observe the signal from the carboxy group proton in the spectra of compounds **IIm, o, p, q** due to its strong broadening. The content of D form estimated from the integral intensity of signals is from 9 to 23%. It should be noted that for compounds of structure similar to that of acids **II**, methyl 4-aryl-2-dimethylhydrazono-4-oxobutanoates, was formerly observed the presence in solutions of β -ketoimine and ketoenamine forms, the latter prevailing [7].

In the mass spectrum of compound **IIq** is present a molecular ion peak with m/z 448, 450 [M^+] of 9.5% intensity, and also fragment ion peaks [m/z (I_{rel} , %)]: 404, 406 (85) [$M - \text{CO}_2$] $^+$, 265 (8.0) [$M - \text{BrC}_6\text{H}_4\text{CO}$] $^+$, 221 (12.0) [$M - \text{CO}_2 - \text{BrC}_6\text{H}_4\text{CO}$] $^+$, 195 (3.0) [$(\text{C}_6\text{H}_5)_2\text{C}=\text{N}=\text{NH}$] $^+$, 183, 185 (28.5) [$\text{BrC}_6\text{H}_4\text{CO}$] $^+$, 180 (100) [$(\text{C}_6\text{H}_5)_2\text{C}\equiv\text{N}$] $^+$, 155, 157 (10.0) [BrC_6H_4] $^+$, 77 (93) [C_6H_5] $^+$ in keeping with the assumed structure.

We studied the intramolecular cyclization of acids **IIb, f, g, h–q** in acetic anhydride in the temperature range from 50 to 139°C; it was established that the reaction afforded the corresponding N-substituted 5-aryl-3-imino-3H-furan-2-ones **IIIa–m** (Scheme 2, Tables 1, 2).

Furanones **IIIa–m** are brightly colored from yellow to purple crystalline substances. In the IR spectra of compounds **IIIa–m** appears an absorption band at 1773–1820 cm^{-1} characteristic of the stretching vibrations of the lactone carbonyl in the furan ring. This band in the spectra of compounds **IIIa–c, i–m** is located at higher frequencies (1799–1820 cm^{-1}) than in the spectra of 2-furanones **III d–h**

Scheme 2.



III, R = CH₃; R' = C₆H₅ (**a**), 4-ClC₆H₄ (**b**); R = H, R' = Napht (**c**); R' = 4-Ant: R = H (**d**), CH₃ (**e**), CH₃O (**f**), Cl (**g**), Br (**h**); R' = (C₆H₅)₂C=N: R = H (**i**), CH₃ (**j**), CH₃O (**k**), Cl (**l**), Br (**m**).

containing a heterocyclic substituent at the imine nitrogen (1773–1785 cm^{-1}). In the ^1H NMR spectrum of compounds **IIIa–m** signals of protons from amino or methylene groups (for compounds **IIm–q**) are lacking, and the singlet from methine proton (C⁴-H) of heterocycle is shifted downfield with respect to the corresponding methine singlet in the spectra of initial acids **II** and appears at 6.89–6.95 ppm, 7.74–7.87 ppm, and 6.92–7.21 ppm for compounds **IIIa–c, III d–h, and III i–m** respectively. The downfield location of the methine singlet in the spectra of compounds **III d–h** is likely to be caused by shielding with heterocyclic substituent attached to imine nitrogen.

The cyclization of acids **II** into furanones **III** is reversible, and the most labile are compounds **IIIa, b**. In their ^1H NMR spectra registered in DMSO-*d*₆ containing a little water in 15 min appear the signals from protons belonging to the A form of the initial acids **IIb, f**.

EXPERIMENTAL

IR spectra were recorded on spectrometers FSM-1201 (Russia) and UR-20 (DDR) from mulls in mineral oil. ^1H NMR spectra were registered on Bruker DRX500 (SF 500,13 MHz) instrument from solutions in DMSO-*d*₆, internal reference HMDS. Mass spectra were obtained on MKh-1310 device at emission current 1000 mA, ionizing electrons energy 70 eV, vaporizer temperature 120°C, ion source temperature 200°C. Chemical purity of compounds was checked and the reaction progress was monitored by TLC on Silufol 254-UV plates, eluent ether-benzene-acetone, 10:9:1.

Compounds **IIa–e** were prepared as in [6], and their constants are consistent with the published data.

2-[N-(4-Chlorophenyl)]amino-4-oxo-4-(*p*-tolyl)-but-2-enoic acid (II f). To a solution of 1.93 g (0.01 mol) of acid **Ib** in ethanol (20 ml) was added

1.29 g (0.01 mol) of *p*-chloroaniline in ethanol (20 ml), and the mixture was kept for 24 h at $-2-0^{\circ}\text{C}$. The separated precipitate was filtered off and recrystallized from ethanol. Yield 2.65 g (87%).

In a similar way from acids **Ia-e** and appropriate amines were prepared acids **Ilg-q** (Table 1).

5-*p*-Tolyl-3-phenylimino-3H-furan-2-one (IIIa).
In 8 ml of acetic anhydride was heated to 70°C for 1 h 2.81 g (0.01 mol) of acid **Iib**. The precipitate separated on cooling was filtered off, washed with anhydrous ether and recrystallized from anhydrous toluene.

Similarly from the corresponding acids **IIf-q** were prepared furanones **IIIb-m**.

Yields, decomposition temperature, and elemental analyses of compounds obtained are compiled in Table 1, the IR and ^1H NMR spectra in Table 2.

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