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THE REACTION OF BENZOYLACRYLIC ACID WITH *ortho*-PHENYLENEDIAMINES

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The reaction of β-benzoylacrylic acid with substituted o-phenylenediamines gives substituted quinoxal-2-ones. The structure of one of the products has been proved using X-ray analysis.

Keywords: benzoylacrylic acid, 3-phenacylquinoxal-2-ones, *o*-phenylenediamines, cyclocondensation.

In a continuation of our investigation of the reactivity of enone systems towards nitrogen-containing binucleophiles [1, 2] we have studied the reaction of β-benzoylacrylic acid (**1**) with substituted *o*-phenylenediamines **2a-g**. The presence of different kinds of electrophilic centers in the molecule **1** leads us to suggest that six- or seven-membered annelated, nitrogen-containing heterocycles of types **5** or **6** can be formed via the intermediate products of addition **3** and/or amidation **4** respectively. Substituents in the starting diamine set up the ground for the formation of the isomeric products.

Refluxing acid **1** with diamines **2a-g** in alcohol gives satisfactory yields (57-75%) of quinoxalones **5a-j** (Table 1). As a rule, similar reactions of other unsaturated carbonyl compounds need the participation of an acid- (HOAc) or base-type (triethylamine) catalyst [3]. We have found that neither the direction of the reaction nor the yield of the product in this reaction depend on the presence of catalyst. The activity of the carboxyl group of acid **1** is not decreased on conversion to the salt form. The reaction of the sodium (or triethylamine) salt of acid **1** with diamine **2a** gives exclusively the product **5a** (Scheme 1).

The structure of the products **5** agrees with their spectroscopic parameters. Hence the IR spectra, taken for KBr tablets, show two bands in the region of 1660-1690 cm^{-1} which are assigned to the vibrations of the carbonyl groups and also a band for the stretching vibrations of the secondary amino group at 3336-3383 cm^{-1}. A new absorption band at 3398-3415 cm^{-1} appears in the spectra taken in CCl_4 along with the regular shift of the considered bands to the higher frequencies. The mass spectrum of the compound **5e** shows a molecular ion peak with *m/z* 311.

The ^{1}H NMR spectra (see Table 2) show aromatic proton multiplets together with double doublets for the methylene group protons, double doublets for the methine proton (J_{AB} = 17.1-17.4, J_{AX} = 6.9, J_{BX} = 4.3 Hz), a broadened signal for the amino group (δ = 5.9-6.65 ppm), and also a lowfield singlet (δ ≥ 10 ppm) which is assigned to the amido NH proton.

We should mention that the ^{1}H NMR spectra of the products obtained from diamines **2b,d,e** show doubling of the signals for the methine proton and the proton of the secondary amino group and this confirms the formation of a mixture of the 6- (**5b,d,e**) and 7-substituted (**5h,i,j**) 3-phenacylquinoxal-2-ones.

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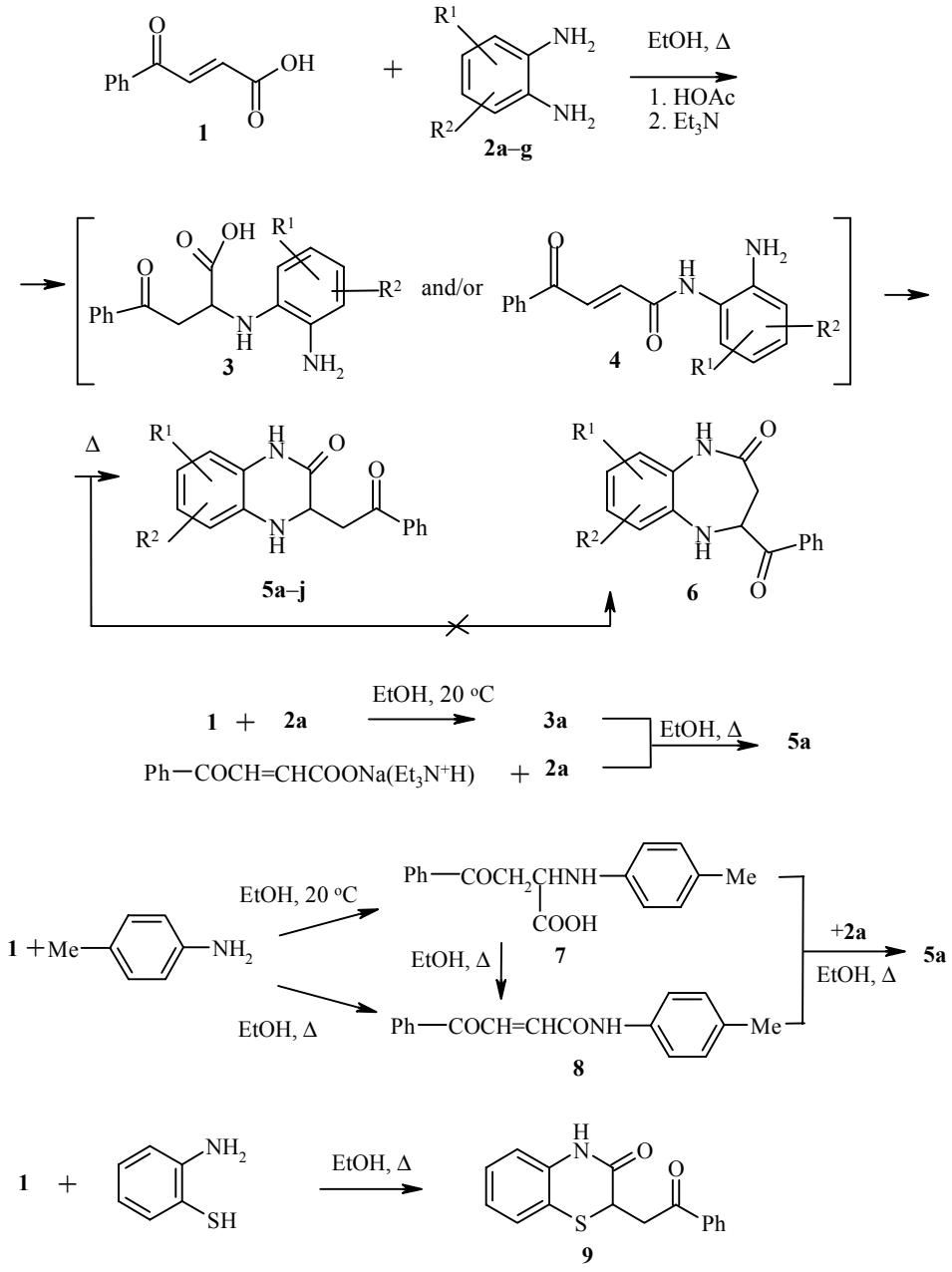
Scheme 1



2-4 a-f R^2 = H, **a** R^1 = H, **b** R^1 = 4-CN, **c** R^1 = 4-Br, **d** R^1 = 4-Me, **e** R^1=4-NO_2, **f** R^1 = 3-NO_2, **g** R^1 = 4-Me, R^2 = 5-Me; **5 a-f** R^2 = H, **a** R^1 = H, **b** R^1 = 6-CN, **c** R^1 = 6-Br, **d** R^1 = 6-Me, **e** R^1 = 6-NO_2, **f** R^1 = 8-NO_2, **g** R^1 = 6-Me, R^2 = 7-Me, **h-j** R^2 = H, **h** R^1 = 7-CN, **i** R^1 = 7-Me, **j** R^1 = 7-NO_2

The ratio of isomers can be evaluated from the integrated intensity of the signals for the mentioned groups. Identification of the isomers takes into account the electronic effect of the substituent on the protons of both NH groups and also the fact that the signal for amide group proton is observed to low field in comparison with an imino group proton. The contribution of the 6-isomer increases with an increase in the electron-acceptor properties of the substituent R (see Table 2). Hence the isomers **5d** and **5i** are formed in approximately equal

TABLE 1. Characteristics of the Compounds Synthesized

Compound	Empirical formula	Found N, % / Calculated N, %	mp, °C	IR spectrum, cm^{-1}*		Yield, %
				$\nu_{C=O}$	ν_{N-H}	
3a	$C_{16}H_{16}N_2O_3$	9.9 / 9.8	160	1660, 1700[a]	3280, 3380[a]	69
5a	$C_{16}H_{14}N_2O_2$	10.3 / 10.5	171	1678, 1685[a] 1680, 1702[b]	3383[a]3413, 3373[b]	61[*2]
5b+5h	$C_{17}H_{13}N_3O_2$	14.3 / 14.4	225-226	1662, 1674[a] 1667, 1677[b]	3368[a] 3353[b]	64
5c	$C_{16}H_{13}BrN_2O_2$	8.0 / 8.1	194	1672, 1682[a] 1680, 1700[b]	3362[a] 3407, 3371[b]	72
5d+5i	$C_{17}H_{16}N_2O_2$	10.0 / 9.9	222-224	1667, 1682[a] 1681, 1697[b]	3376[a] 3413, 3378[b]	61*[2]
5g	$C_{18}H_{18}N_2O_2$	9.5 / 9.5	218-220	1667, 1680[a] 1682, 1697[b]	3369[a] 3415, 3388[b]	63
5e+5j	$C_{16}H_{13}N_3O_4$	13.3 / 13.5	225	1662, 1672[a] 1679, 1712[b]	3383[a] 3403, 3378[b]	71
5f	$C_{16}H_{13}N_3O_4$	13.4 / 13.5	175	1677, 1692[a] 1687, 1717[b]	3336[a] 3398, 3350[b]	75
7	$C_{17}H_{17}NO_3$	4.6 / 4.9	138	1680, 1730[a]	3360[a]	80
8	$C_{17}H_{15}NO_2$	5.1 / 5.3	153	1660[a]	3220[a]	53[*3]
9	$C_{16}H_{13}NO_2S$	4.7 / 4.9	171	1677,1695[b]	3376[b]	76

* a) for KBr tablets; b) for solution in CCl_4.

*[2] Yield using method A. The yields for methods B-E are 59, 57, 58, and 53% respectively.

*[3] Yield using method A. The yield for method B is 52%.

amounts (45:55, see Table 2) but the ratio of the isomers **5e** and **5j** is 85:15. Separation of the isomeric quinoxalones **5b,d,e** and **5h,i,j** using thin layer chromatography did not prove possible. For the same reason, compound **5c** should probably be assigned as the 6-Br isomer. In the reaction of diamine **2f** with acid **1** 8-nitro isomer **5f** is isolated, in the molecule of which an intramolecular hydrogen bond is realized, and this explains the marked diamagnetic shift of the amido group proton.

However, the given spectroscopic evidence could correspond, equally, to 4-benzoyltetrahydrodiazepin-2-ones (**6**). Hence, in order to obtain an unambiguous answer to the structure of the products, an X-ray structural analysis was carried out for compound **5a** (Tables 2-5) and this confirmed it to be 3-phenacylquinoxal-2-one.

The pyrazine ring in the product **5a** is found to exist in distorted chair-type conformation (Figure 1). Atoms $C_{(6)}$ and $C_{(7)}$ deviate from the plane of the remaining ring atoms by 0.23 and 0.59 Å respectively. The substituent at atom $C_{(7)}$ is planar (the mean deviation from the plane not exceeding 0.01 Å) and is axially orientated (torsion angle $N_{(1)}$–$C_{(6)}$–$C_{(7)}$–$C_{(9)}$ 95.7 (2)°). In the crystalline state, molecule **5a** forms infinite chains due to the intermolecular hydrogen bonds $N_{(1)}$–$H_{(1N)}$···$O_{(1)}$ (-*x*, -*y*, 1-*z*) (H···O distance 2.02 Å, N–H···N angle 179°) and $N_{(1)}$–$H_{(2N)}$···$O_{(1)}$ (1-*x*, -*y*, -*z*) (H···O 2.34 Å, N–H···O 145°).

Formation of the isomeric quinoxal-2-one derivatives can be explained in the following way. The more basic amino group takes part in both α-addition reactions (formation of compound type **3**) and amidation reactions (formation of compound type **4**) such that, in the molecules of certain diamines **2,** the difference in the basicity of the amino groups is not large and, even in the first stage, the isomeric intermediates of type **3** or **4** are formed. The first route is confirmed by an experiment with diamine **2a** in which the α-adduct **3a** is formed in its reaction with acid **1** in ethanol at 20°C.

TABLE 2. ^{1}H NMR Spectral Characteristics of the Synthesized Compounds (DMSO-d_6), δ, ppm

Compound	CH_2, dd		CH, dd	NH	NHCO, s	H_{arom}	Isomer content, %	
	A	B	X	br. s			6-	7-
5a	3.60	3.30	4.36	5.90	10.30	8.10-6.60	—	—
5b+5h	3.60	3.53	4.58 4.44	6.45 br.	10.78 10.61	8.00-6.40	60	40
5c	3.56	3.35	4.40	6.28	10.48	8.00-6.69	100	—
5d+5i*	3.58	3.28	4.32 4.28	5.92 5.81	10.32 10.28	8.10-6.40	45	55
5e+5j	3.60	3.40	4.50 4.62	6.52 6.75	10.85 10.63	8.10-6.80	85	15
5f	3.58	3.43	4.55	6.65	9.99	8.10-6.88	—	—
5g*	3.80	3.27	4.30	5.74	10.28	8.10-6.50	—	—
7*	3.53	3.48	4.45	3.39	—	8.00-6.70	—	—
8*	—	—	6.40 6.10	—	10.00	7.80-7.00	—	—
9	4.10	3.70	4.45	—	10.65	8.00-7.00	—	—

* Signals for the CH_3 group protons: 2.16 (**5d** + **5j**), 2.10 (**5g**), 2.10 (**7**), 2.30 (**8**).

The possibility of realizing the second route is indicated by the following data. In the reaction of acid **1** with *p*-toluidine (for which the basicity is close to that of *o*-phenylenediamine (pK_a for *o*-phenylenediamine = 4.47, pK_a of *p*-toluidine = 4.39 [4]) but cyclization reaction is excluded) the product 2-(*p*-methylanilino)-3-benzoylpropionic acid (**7**) is formed at room temperature.

Refluxing the mentioned compounds in alcohol gives amide **8**. The latter is also obtained in 53% yield by heating acid **7** in alcohol. Hence acid **7** is kinetically controlled and compound **8** is thermodynamically controlled reaction product. When the products **7** and **8** are refluxed with diamine **2a** they undergo transamination and subsequent cyclization to the thermodynamically stable quinoxalone ring. In our opinion, these results allow us to propose that the formation of the 6-isomer occurs *via* addition stage while the formation of the 7-isomer may also include initial amidation step.

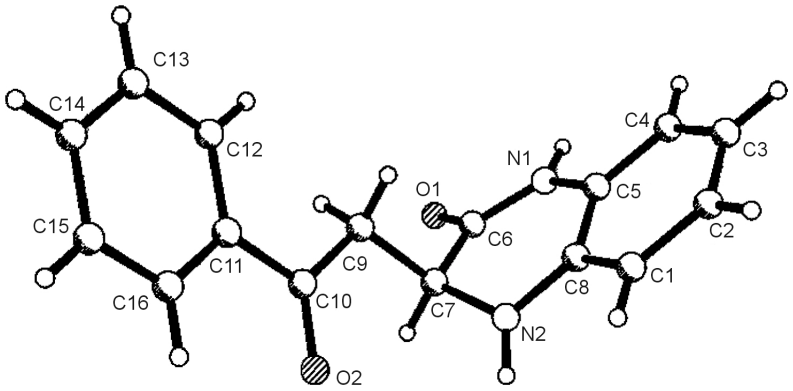


Fig. 1. General view of the molecule of compound **5a**.

TABLE 3. Bond lengths (d) in the Structure of Compound **5a**

Bond	*d*, Å	Bond	*d*, Å
$N_{(1)}$–$C_{(6)}$	1.343(3)	$N_{(1)}$–$C_{(5)}$	1.411(3)
$N_{(2)}$–$C_{(8)}$	1.389(3)	$N_{(2)}$–$C_{(7)}$	1.465(3)
$O_{(1)}$–$C_{(6)}$	1.239(3)	$O_{(2)}$–$C_{(10)}$	1.218(3)
$C_{(1)}$–$C_{(8)}$	1.384(3)	$C_{(1)}$–$C_{(2)}$	1.385(3)
$C_{(2)}$–$C_{(3)}$	1.385(4)	$C_{(3)}$–$C_{(4)}$	1.386(3)
$C_{(4)}$–$C_{(5)}$	1.386(3)	$C_{(5)}$–$C_{(8)}$	1.402(3)
$C_{(6)}$–$C_{(7)}$	1.524(3)	$C_{(7)}$–$C_{(9)}$	1.534(3)
$C_{(9)}$–$C_{(10)}$	1.515(3)	$C_{(10)}$–$C_{(11)}$	1.507(3)
$C_{(11)}$–$C_{(12)}$	1.386(3)	$C_{(11)}$–$C_{(16)}$	1.390(3)
$C_{(12)}$–$C_{(13)}$	1.384(4)	$C_{(13)}$–$C_{(14)}$	1.394(4)
$C_{(14)}$–$C_{(15)}$	1.361(5)	$C_{(15)}$–$C_{(16)}$	1.376(4)

Our data is in good agreement with the results of studying the reaction of arylamides and benzoylpyruvic acid esters with *o*-phenylenediamine [5, 6], in which derivatives of 3-phenacylidenequinoxal-2-one and not those of benzodiazepine are formed. In this case reactions occur *via* an initial stage of amination of the α-keto group and this is in agreement with the sequence of stages proposed by us for kinetically controlled conditions.

We have also studied the reaction of acid **1** with *o*-aminothiophenol under the same conditions as for the synthesis of compound **5**. According to the spectroscopic parameters (see Table 1) the product **9** is 2-phenylacylbenzo-1,4-thiazin-3-one and not benzothiazepine derivative as claimed in the report [7]. The first stage of nucleophilic α-addition of the mercapto group is clear in this case [8] and it serves as an indirect confirmation of the proposed reaction scheme.

TABLE 4. Bond Angles (ω) in the Structure of Compound **5a**

Angle	ω, deg.	Angle	ω, deg.
$C_{(6)}$–$N_{(1)}$–$C_{(5)}$	124.3(2)	$C_{(8)}$–$N_{(2)}$–$C_{(7)}$	118.9(2)
$C_{(8)}$–$C_{(1)}$–$C_{(2)}$	120.7(2)	$C_{(3)}$–$C_{(2)}$–$C_{(1)}$	120.5(2)
$C_{(2)}$–$C_{(3)}$–$C_{(4)}$	119.6(3)	$C_{(3)}$–$C_{(4)}$–$C_{(5)}$	120.0 (2)
$C_{(4)}$–$C_{(5)}$–$C_{(8)}$	120.6(2)	$C_{(4)}$–$C_{(5)}$–$N_{(1)}$	120.9(2)
$C_{(8)}$–$C_{(5)}$–$N_{(1)}$	118.5(2)	$O_{(1)}$–$C_{(6)}$–$N_{(1)}$	122.7(2)
$O_{(1)}$–$C_{(6)}$–$C_{(7)}$	120.3(2)	$N_{(1)}$–$C_{(6)}$–$C_{(7)}$	116.9(2)
$N_{(2)}$–$C_{(7)}$–$C_{(6)}$	110.8(2)	$N_{(2)}$–$C_{(7)}$–$C_{(9)}$	113.3(2)
$C_{(6)}$–$C_{(7)}$–$C_{(9)}$	107.6(2)	$C_{(1)}$–$C_{(8)}$–$N_{(2)}$	123.1(2)
$C_{(1)}$–$C_{(8)}$–$C_{(5)}$	118.6(2)	$N_{(2)}$–$C_{(8)}$–$C_{(5)}$	118.2(2)
$C_{(10)}$–$C_{(9)}$–$C_{(7)}$	113.4(2)	$O_{(2)}$–$C_{(10)}$–$C_{(11)}$	120.6(2)
$O_{(2)}$–$C_{(10)}$–$C_{(9)}$	120.7(2)	$C_{(11)}$–$C_{(10)}$–$C_{(9)}$	118.6(2)
$C_{(12)}$–$C_{(11)}$–$C_{(16)}$	118.8(2)	$C_{(12)}$–$C_{(11)}$–$C_{(10)}$	122.3(2)
$C_{(16)}$–$C_{(11)}$–$C_{(10)}$	118.9(2)	$C_{(13)}$–$C_{(12)}$–$C_{(11)}$	120.8(3)
$C_{(12)}$–$C_{(13)}$–$C_{(14)}$	118.9(3)	$C_{(15)}$–$C_{(14)}$–$C_{(13)}$	120.6(3)
$C_{(14)}$–$C_{(15)}$–$C_{(16)}$	120.3(3)	$C_{(15)}$–$C_{(16)}$–$C_{(11)}$	120.5(3)

TABLE 5. Coordinates ($\times 10^4$) and Equivalent Isotropic Thermal Parameters for the Non-hydrogen Atoms in Structure of Compound **5a**

Atom	*x*	*y*	*z*	$U_{(eq)}$
$N_{(1)}$	2257(3)	620(2)	3922(1)	45(1)
$N_{(2)}$	3430(4)	717(2)	1742(2)	50(1)
$O_{(1)}$	-329(3)	-862(2)	3927(1)	53(1)
$O_{(2)}$	4830(4)	-1411(2)	692(1)	65(1)
$C_{(1)}$	6608(5)	2121(2)	1821(2)	55(1)
$C_{(2)}$	7769(5)	2829(2)	2411(2)	61(1)
$C_{(3)}$	7119(5)	2811(2)	3506(2)	62(1)
$C_{(4)}$	5288(5)	2080(2)	4011(2)	54(1)
$C_{(5)}$	4123(4)	1371(2)	3422(2)	41(1)
$C_{(6)}$	1299(4)	-211(2)	3462(2)	44(1)
$C_{(7)}$	2470(4)	-405(2)	2366(2)	45(1)
$C_{(8)}$	4756(4)	1395(2)	2310(2)	44(1)
$C_{(9)}$	4577(4)	-1511(2)	2637(2)	45(1)
$C_{(10)}$	5713(5)	-1901(2)	1615(2)	45(1)
$C_{(11)}$	7990(5)	-2892(2)	1762(2)	51(1)
$C_{(12)}$	9008(5)	-3489(2)	2801(2)	67(1)
$C_{(13)}$	11103(6)	-4394(3)	2907(3)	82(1)
$C_{(14)}$	12215(6)	-4682(3)	1949(4)	87(1)
$C_{(15)}$	11237(6)	-4094(3)	926(3)	88(1)
$C_{(16)}$	9130(5)	-3207(2)	824(3)	70(1)

EXPERIMENTAL

^{1}H NMR spectra were recorded on a Bruker AM-300 (300 MHz) instrument for solutions in DMSO-d_6 with TMS as internal standard. IR spectra were taken on a Specord IR-75 spectrometer for KBr tablets and solutions in CCl_4. Mass spectra were recorded on a Finnigan MAT INCOS 50 instrument (70 eV). Monitoring of the reaction course and the degree of purity of the products was carried out using TLC on Silufol UV-254 plates in the system ethyl acetate-chloroform (1:4).

2-(*o*-Aminophenylamino)-3-benzoylpropionic Acid (3a). Solution of acid **1** (0.44 g, 2.5 mmol) and diamine **2a** (0.3 g, 2.5 mmol) in ethanol (10 ml) was stirred at room temperature for 30-40 min to give acid **3a** (0.49 g).

3-Phenacylquinoxal-2-one (5a). A. Solution of acid **1** (1 g, 5.6 mmol) and diamine **2a** (0.61 g, 5.6 mmol) in ethanol (20 ml) was refluxed for 1 h. The precipitated solid was crystallized from ethyl acetate to give the product **5a** (0.92 g).

Compounds **5b-j** were obtained similarly from the corresponding amines **2b-j**.

B. Solution of acid **1** (1 g, 5.6 mmol), diamine **2a** (0.61 g, 5.6 mmol), and acetic acid (0.5 ml) in ethanol (20 ml) was refluxed for 1 h. The precipitated solid was crystallized from ethyl acetate to give the product **5a** (0.88 g).

C. Solution of acid **1** (1 g, 5.6 mmol) in ethanol (10 ml) was added to solution of sodium hydroxide (0.2 g) in ethanol (2 ml) and diamine **2a** (0.61 g, 5.6 mmol) in ethanol (2 ml). The mixture was refluxed for 1 h. The precipitate formed was filtered off and the filtrate was diluted with water, acidified with acetic acid, and an additional amount of product separated. The overall yield of the product **5a** was 0.85 g,

D. Solution of acid **3a** (1 g) in ethanol (10 ml) was refluxed for 1 h. The precipitate was filtered off and crystallized from ethyl acetate to give quinoxalone **5a** (0.55 g).

E. Solution of acid **7** (1.2 g, 4.6 mmol) (see below) and diamine **2a** (0.5 g, 4.6 mmol) in ethanol (10 ml) was refluxed for 1 h. The precipitate was filtered off and crystallized from ethyl acetate to give quinoxalone **5a** (0.6 g).

The product **5a** was synthesized similarly from amide **8**. Mixed samples of compound **5a** prepared using the different methods did not give a depression of melting point.

2-(*p*-Methylphenylamino)-3-benzoylpropionic Acid (7). Mixture of acid **1** (1.5 g, 8.4 mmol) and *p*-toluidine (0.9 g, 8.4 mmol) in ethanol (15 ml) was stirred for 30-40 min at room temperature. The precipitate was filtered off and crystallized from chloroform. Yield 1.9 g.

Benzoylacrylic acid N-(4-methylphenyl)amide (8). A. Mixture of acid **1** (1.5 g, 8.4 mmol), *p*-toluidine (0.9 g, 8.4 mmol), and triethylamine (1.2 ml) in ethanol (11 ml) was refluxed for 30 min. The amide formed was filtered off and recrystallized from ethanol to give the product **8** (1.18 g).

B. Refluxing the product **7** (1 g, 3.5 mmol) in ethanol (10 ml) for 1-1.5 h gave amide **8** (0.49 g, from ethanol) which did not give the melting point depression with a sample obtained by method A.

2-Phenacylbenzo-1,4-thiazin-3-one (9). Solution of acid **1** (1.5 g, 8.5 mmol), *o*-aminothiophenol (1.06 g, 8.5 mmol), and acetic acid (0.5 ml) in ethanol (10 ml) was stirred at room temperature for 15-20 min. The precipitate was filtered off and crystallized from ethanol to give the product **9** (1.8 g).

X-Ray Structural Investigation of Compound 5a. Crystals of the compound **5a** ($C_{16}H_{14}N_2O_2$) are triclinic. At 20°C a = 5.201 (1), b = 11.255 (4), c = 12.337 (4) Å; α = 77.11 (3), β = 89.26 (2), γ = 82.98 (2)°; V = 698.6 (4) Å^3; d_{calc} = 1.266 g/cm^3; space group $P1$; Z = 2. The unit cell parameters and intensities of 2451 independent reflection (R_{ind} = 0.004) were measured using a Siemens P3/PS automatic four-circle diffractometer (λMoKα, graphite monochromator, $\theta/2\theta$ scanning, $2\theta_{max}$ = 50°).

The structure was solved by a direct method using the SHELXTL PLUS [9] computer package. The positions of the hydrogen atoms were calculated geometrically and refined using the "rider" model with fixed $U_{iso} = 1.2U_{eq}$ of the non-hydrogen atom bonded to the given hydrogen atom. Refinement of F^2 by full-matrix, least-squares analysis in the anisotropic approximation for non-hydrogen atoms gave ωR_2 = 0.1486 (R_1 = 0.051 for 1242 reflections with $F > 4\sigma$ (F), S = 0.908). The coordinates for the non-hydrogen atoms are given in Table 5.

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