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Synthesis and Transamination of Enaminones: Derivatives of 1-Phenyl-4-(phenylhydroxymethylidene)-pyrrolidine-2,3,5-trione

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Summary. The reaction of 1-phenyl-4-(phenylhydroxymethylidene)-pyrrolidine-2,3,5-trione with difunctional bases (α-,β-,γ-amino acid derivatives or aminoethanol) leads to a mixture of tautomeric *Schiff* bases and enaminones. Some of the products easily undergo transamination at their enaminone moiety.

Keywords. Enaminones; Pyrrolidine-2,3,5-trione; α-,β-,γ-Amino acids; Transamination.

Synthese und Transaminierung von Enaminonen: Derivate des 1-Phenyl-4-(phenylhydroxymethyliden)-pyrrolidin-2,3,5-trions

Zusammenfassung. Die Reaktion von 1-Phenyl-4-(phenylhydroxymethyliden)pyrrolidin-2,3,5-trions mit difunktionellen Basen (α-,β-γ-Aminosäurederivativen oder Aminoethanol) führt zu einem Gemisch der tautomeren *Schiff*schen Basen und Enaminone. Einige dieser Verbindungen werden leicht am Enaminonfragment transaminiert.

Introduction

Tetramic acid derivatives have found interest because of their biological and pharmacological properties; thus, their chemistry has been extensively investigated [1–4]. In a previous study [5], the analogue of tetramic acid, 1-phenyl-4-(phenylhydroxsymethylidene)-pyrrolidine-2,3,5-trione (**1**, Scheme 1), has been synthesized. It exists only in the *exo*-enol form and is in principle susceptible to nucleophilic attack at C-2, C-3, C-5, and C-6. Reaction of **1** with amines leads to

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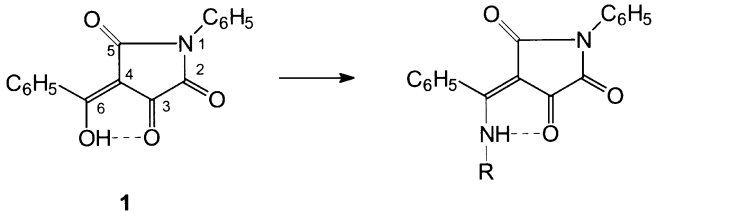
Schiff bases which are known to possess anticancer activity [6,7]. Because of the biological importance of such tetramic acid derivatives, their synthesis using different mono-, di-, and triamines varying in basicity has been studied. When strong bases were used ($pK_a > 13.6$) only salts were obtained [8–10], whereas with weaker bases (pK_a 3.43–10.71) condensation took place: for monoamines at C-6, for diamines in a first step at C-6 and then at C-3 [11,12]. In this work we have studied the reaction of **1** with weak bases containing two different functional groups such as α-, β-, or γ- amino acid derivatives and aminoethanol.

Results and Discussion

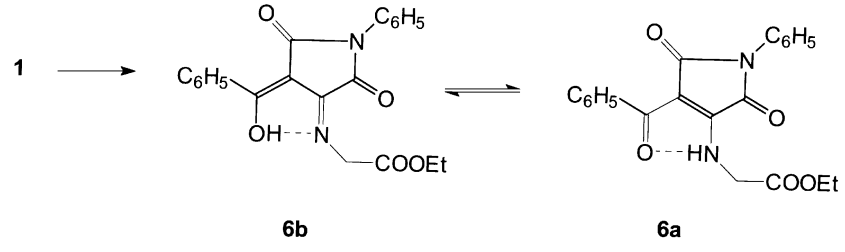
Scheme 1 shows the reaction products **2–5** obtained as a result of condensation of **1** with glycine, β-alanine, γ-aminobutyric acid, and aminoethanol in ethanolic solution. Under these conditions, condensation took place at C-6 as inferred from comparison of the spectroscopic data with those of the analogous compound 4-(phenyl-(phenylamino)-methylidene)-pyrrolidine-2,3,5-trione [5]. The crucial proof was provided by the absence of signals characteristic for a benzoyl group in their ^{13}C NMR and MS data (Table 1). The ^{1}H NMR spectra show that compounds **2** and **3** exist in two tautomeric forms (enol-imino and keto-enamino). At equilibrium, the enol-imine content amounts to 53% for **2** and 52% for **3**.



2: R = CH_2COOH
3: R = CH_2CH_2COOH
4: R = $CH_2CH_2CH_2COOH$
5: R = CH_2CH_2OH

Scheme 1

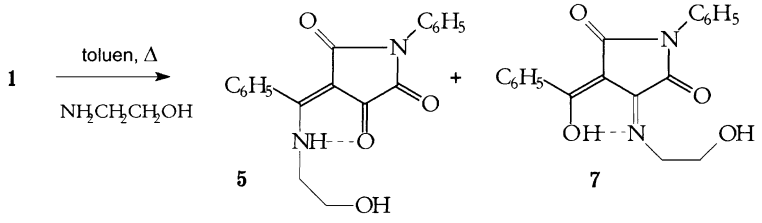
In the reaction of **1** with glycine ethyl ester hydrochloride, carried out in boiling ethanol, condensation unexpectedly occurred at C-3 (Scheme 2). Two tautomeric forms (enol-imino, **6a**; keto-enamino, **6b**) were obtained. ^{1}H NMR data showed that the keto-enamino form predominated at equilibrium (76%).



Scheme 2

In **6a**, the CH_2 protons and the NH proton resonated as doublets with a coupling constant of 6.9 Hz. It should be mentioned that recently [13] a two-step synthesis of 4-acyl-(carbamoyl)-3-amino-pyrrol-2,5-dione, the product of nucleophilic substitution at C-3, has been reported. As starting material, the *endo*-enol form of **1** has been used; reaction with $SOCl_2$ afforded the 3-chloro derivative which in turn underwent nucleophilic substitution with amine.

With the exception mentioned above, reaction of **1** with amines in ethanol always led to a condensation at C-6. However, further investigation revealed that upon changing the solvent from ethanol to toluene the reaction of **1** with aminoethanol led to two products of condensation: **5** (at C-6) and **7** (at C-3) (Scheme 3). The ^{1}H NMR spectrum of **1** in methanol-d_4 at room temperature showed the presence of 90% *exo*-enol form, whereas in benzene-d_6 only 74% of this isomer could be detected. Thus, toluene inverted the *endo-exo*-enol equilibrium of **1**, enabling formation of the C-3 product.



Scheme 3

The structure of **5** was proven by X-ray analysis (Fig. 1) as well as by its NMR and MS data. Compound **5** exists as the (*E*)-isomer stabilized by an intramolecular hydrogen bond with the following geometry: N2···O3 = 2.793(2), N2–H2 = 0.95(2), H2···O3 = 2.02(2) Å and N2–H2···O3 = 138(2)°. MS and NMR tech-

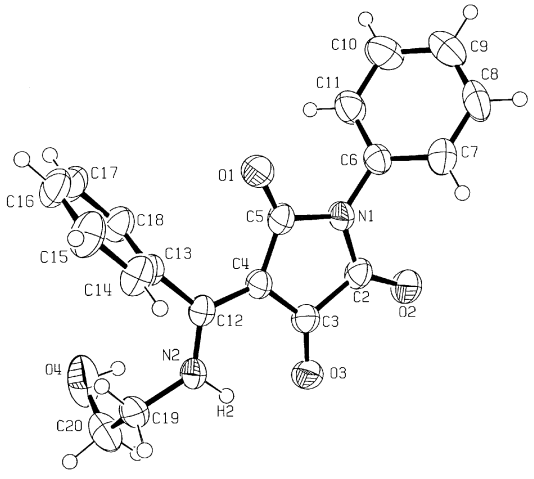
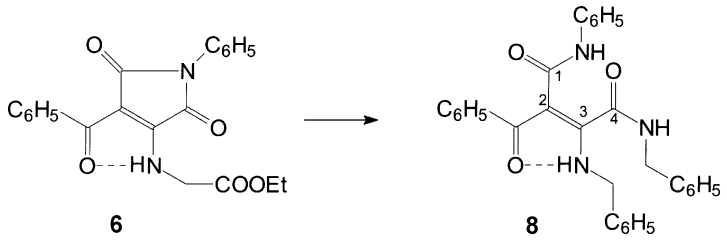


Fig. 1. Structure of **5** including the atom numbering scheme corresponding to that used in the deposited tables (ORTEP-3 [20]; thermal vibration ellipsoids are scaled to enclose 50% probability)



Scheme 4

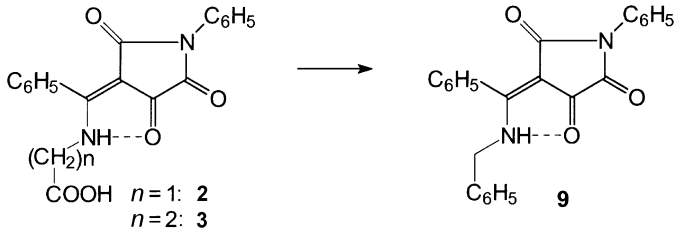
niques were also used to establish the structure of **7**, revealing especially the presence the benzoyl group.

Compounds **2**, **3**, and **6** were chosen for reactivity studies with benzylamine in ethanolic solution. As shown in Scheme 4, **6** undergoes transamination. Simultanously, cleavage of the N1–C2 bond of the succinimide group took place leading to **8**. In case of compounds **2** and **3** (Scheme 5), only transamination occurred leading to compound **9**. The structures of **8** and **9** were elucidated on the basis of elemental analysis, mass, and NMR spectroscopy (Table 1). For compound **8**, the latter method showed the presence of signals specific for the benzoyl group.

In conclusion, we have shown that the condensation of **1** with bases can take place either at C-6 or at C-3 depending on the solvent used. When the reaction of **1** with bases takes place at C-6, two tautomers are formed at the same ratio, whereas the condensation at C-3 gives a product for which the equilibrium is shifted towards the enaminone form. With benzyl amine, compounds **2**, **3**, and **6**, presumed as enaminones, undergo transamination. This means that nucleophilic substitution at the carbon atom attached to the nitrogen atom of the enaminone moiety is preferred for nucleophilic addition. That proves the lower reactivity of the latter, independently of the fact whether it is the benzoyl or a ring carbonyl group. The statement that the carbon atom attached to the nitrogen atom of the enaminone

Table 1. Selected ^{13}C NMR chemical shifts (ppm) for compounds **1–9**

	C-5	C-4	C-3	C-2	C-6
1	170.75	102.70	171.87	163.87	183.20
2 keto-enamino	169.44	93.45	175.10	161.60	166.78
enol-imino	171.35	95.46	179.88	161.89	165.96
3 keto-enamino	168.07	92.69	172.47	158.58	162.51
enol-imino	167.03	92.69	174.80	158.58	161.85
4	173.80	98.11	174.81	170.52	164.63
5	162.15	94.43	167.55	162.10	155.00
6b(enol-imino)	168.59	97.73	150.32	166.09	189.76
6a(keto-enamino)	167.73	97.73	155.68	162.51	188.91
7	166.09	98.04	156.54	162.92	191.61
9	161.85	120.45	166.98	158.59	160.13
	C-1	C-2	C-3	C-4	C-5
8	164.36	87.95	145.05	162.55	185.06



Scheme 5

moiety is more reactive towards nucleophiles is thus in accordance with earlier reports [14–17].

Experimental

The ^{1}H NMR and ^{13}C NMR spectra were recorded in *DMF*-d_7, *DMSO*-d_6, and $CDCl_3$ with a Bruker AMX 500 NMR spectrometer using *TMS* as internal standard. The IR spectra were measured in Nujol and hexachlorobutadiene (*HCBN*) with a Bruker IFS 48 spectrometer. The MS spectra were obtained on an LKB 9000 S and a Finnigan TSQ 700 triple quadruple mass spectrometer. Elemental analyses were carried out with a Perkin Elmer analyser 240 in the regional Laboratory of Physico-Chemical Analyses and Structural Research in Kraków. Their results were in good agreement with the calculated values. X-Ray intensities were collected at room temperature on an Enraf-Nonius CAD-4 diffractometer using graphite monochromated MoK_α radiation ($\lambda = 0.71073$ Å).

General procedure for the preparation of enaminones **2–6**

To a solution of 2.9 g of **1** (0.01 mol) in ethanol, 0.012 mol of the corresponding amine were added. The solution was refluxed for 2.5 h and left overnight. The precipitate was filtered off and crystallized from ethanol. Compound **6** was purified by radial chromatography (aluminum oxide, $CHCl_3$:$CH_2Cl_2 = 2{:}1$).

1-Phenyl-4-(phenyl-(carboxymethylamino)-methylidene)-pyrrolidine-2,3,5-trione (**2**;$C_{19}H_{14}N_2O_5$)

Yield: 33.7%; m.p.: 238°C; IR (*HCBN*): $\nu = 3177$ (NH), 3062 (C–H arom), 2926 (CH_2), 1746, 1669 (C=O), 1635 (C=N) cm^{-1}; ^{1}H NMR (*DMSO*-d_6, δ, 500.13 MHz): 3.9 (s, 2H, CH_2), 4.39 (s, 2H, CH_2), 7.3–7.56 (m, arom), 10.29 (s, 1H, NH), 10.85 (s, 1H, OH), 13.2 (s, 1H, COOH) ppm; ^{13}C NMR (*DMSO*-d_6, δ, 125.77 MHz): 46.05 (CH_2), 56.01 (CH_2), 119.34, 124.11, 127.0, 127.31, 127.68, 128.04, 128.31, 128.50, 128.756, 128.976, 129.61, 129.82,130.48, 130.48, 131.55, 131.80 (C-arom.), 171.31 (COOH) ppm; MS: *m/z* (%) = 350 (67) M^+, 322 (75) $[M–CO]^+$, 278 (33) $[M–CO–COO]^+$, 77(100) $C_6H_5^+$.

1-Phenyl-4-(phenyl-(2-carboxyethylamino)-methylidene)-pyrrolidine-2,3,5-trione (**3**;$C_{20}H_{16}N_2O_5$)

Yield: 21.5%; m.p.: 225°C; IR (*HCBN*): $\nu = 3104$ (NH), 1767 (CO), 1726 (CO), 1707 (CO), 1663 (CO), 1634 (C=N) cm^{-1}; ^{1}H NMR (*DMSO*-d_6, δ, 500.13 MHz): 2.6 (t, 2H, CH_2–CO), 2.85 (t, 2H, CH_2–CO), 3.87 (t, 3H, CH_2–N), 4.11 (t, 2H, CH_2–N), 7.16–7.90 (m, arom), 10.30 (s, 1H, NH), 10.88 (s, 1H, OH), 12.60 (s, 1H, COOH) ppm; ^{13}C NMR (*DMSO*-d_6, δ, 125.77 MHz): 32.52 (CH_2–N), 33.64 (CH_2–N), 40.56 (CH_2–CO), 45.81 (CH_2–CO), 120.42, 126.95, 127.19, 127.52, 127.58, 127.72, 128.04, 128.59, 129.45, 129.70, 130.31, 130.53, 131.68, 132.23, 133.90, 137.31, (C-arom), 172.47 (COOH) ppm; MS: *m/z* (%) = 364 (30.5) M^+, 336 (35.6) $[M–CO]^+$, 292 (11) $[M–CO–COO]^+$, 77(100) $C_6H_5^+$.

1-Phenyl-4-(phenyl-(3-carboxypropylamino)-methylidene)-pyrrolidine-2,3,5-trione
(**4**; $C_{21}H_{18}N_2O_5$)

Yield: 50%; m.p.: 200–202°C; IR (*HCBN*): $\nu = 3143$ (NH), 3050 (C–H arom), 2900–2485 (COOH), 1755, 1701, 1690 (CO), 1630 (C=N) cm^{-1}; ^{1}H NMR (*DMSO*-d_6,δ, 500.13 MHz): 1.78 (m, 2H, CH_2), 2.35 (t, 2H, CH_2N), 2.85 (t, 2H, CH_2CO), 3.45 (s, 1H, NH), 7.29–7.87 (m, 10H, Ph-H), 12.3 (s, 1H, COOH) ppm; ^{13}C NMR(*DMSO*-d_6, δ, 125.77 MHz): 22.57 (C-3′), 30.48 (CH_2–N), 38.53 (CH_2–CO), 187.48 (COOH) ppm; MS: m/z (%) = 378 (5.1) M^+, 350 (5.6) $[M–CO]^+$, 306 (2) $[M–CO–COO]^+$, 77(100) $C_6H_5^+$.

(E)-1-Phenyl-4-(phenyl-(2-hydroxyethylamino)-methylidene)-pyrrolidine-2,3,5-trione
(**5**; $C_{19}H_{16}N_2O_4$)

Yield: 36%; m.p.: 237°C; IR (*HCBN*): $\nu = 3525$ (OH), 3193 (NH), 1767, 1710, 1695 (CO), 1662 (C=C) cm^{-1}; ^{1}H NMR (*DMSO*-d_6, δ, 500.13 MHz): 3.26 (d, 2H, CH_2–N), 3.52 (d, 2H, CH_2–O), 5.11 (s, 1H, OH), 7.71–7.66 (m, 10H, arom), 10.30 (s, 1H, NH) ppm; ^{13}C NMR (*DMSO*-d_6, δ, 125.77 MHz): 46.92 (CH_2–N), 56.10 (CH_2–O), 126.96, 127.69 128.33, 128.47, 128.61, 129.79, 130.25, 131.70, (C-arom) ppm; MS: *m/z* (%) = 336(29.1) M^+, 308 (21.6) $[M–CO]^+$, 104 (100) $C_6H_5CNH^+$, 77 (40.5) $C_6H_5^+$.

Crystal data: $C_{19}H_{16}N_2O_4$, $MW = 336.34$, monoclinic, $P2_1/n$, $a = 6283(5)$, $b = 10.946(2)$, $c = 23.858(3)$ Å, $\alpha = 90.0$, $\beta = 96.652$, $\gamma = 90.0°$, $V = 1629.6(4)$ Å^3, $Z = 4$, $D = 1.371$ Mgm^{-3}, $\lambda(MoK_\alpha) = 0.71073$ Å, $\mu = 0.098$ mm^{-1}, $F(000) = 704$, $T = 293$ K.

Crystals of **5** suitable for X-ray analysis were grown from ethanol as gold yellow prisms. Crystal of dimensions $0.84 \times 0.44 \times 0.10$ mm were used for the measurements. Cell parameters were determined using 48 reflections in the range $4.1 < \Theta < 21.5°$. Intensity measurements were carried out in the range $2 \leq \Theta \leq 25°$ with the $\omega/2\Theta$ scan mode. The ranges of indices were $0 \leq h \leq 6$, $0 \leq k \leq 11$, $-25 \leq l \leq 24$. Three standared reflections were monitored every 197 reflections; no decay in intensities was detected. *Lorentz* and polarization, but no absorption corrections were applied. The number of reflections was 2223, and the number of symmetry independent reflections 2003. The structure was solved by direct methods using the program SIR92 [18] and refined against F^2 by full-matrix least squares using SHELXL-93 [19] with all hydrogens except those of enamine and hydroxyl group placed geometrically using the AFIX routine of the program. In the course of the refinement, their coordinates were kept riding on the atoms to which the hydrogens were attached. The temperature factors of the hydrogen atoms were set to 1.2 times U(eq) of the respective heavy atom. Hydrogens of the enamine and hydroxyl group were localized on the difference *Fourier* map and refined. The non-hydrogen atoms were refined anisotropically. The refinement of 235 parameters converged at $R = 0.0303$ and $wR = 0.0674$ for 1447 reflections with $I > 2\sigma(I)$ and $R = 0.0482$ and $wR = 0.0723$ for all data. $w = (\sigma^2(F_o^2)+(0.0377p)^2)^{-1}$ ($p = (F_o^2+2F_c^2)/3$); extinction coefficient = 0.0061; max. and min. peak on final difference *Fourier* map: 0.104 and −0.101 eÅ^{-3}. Additional material to the structure determination may be ordered from Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-76344 Eggenstein-Leopolshafen, Federal Republic of Germany, referring to the deposition number CSD-410246 and the citation of the present paper.

1-Phenyl-3-((ethoxycarbonylmethylene)-imine)-4-(phenyl-(hydroxy)-methylidene)-pyrrolidine-2,5-dione (**6**:$C_{21}H_{18}N_2O_5$)

Yield: 53%; m.p.: 175°C; IR (*HCBN*): $\nu = 3460$ (OH), 3251 (NH), 1763 (CO), 1743 (CO), 1709 (CO), 1640 (C=N), 1612 (C=C) cm^{-1}; ^{1}H NMR (*DMSO*-d_6, δ, 500.13 MHz): 0.92 (t, 3H, CH_3), 1.21 (t, 3H, CH_3), 3.8 (q, 2H, CH_2–O), 4.16 (q, 2H, CH_2–O),4.36 (s, 2H, CH_2–N), 4.68 (d, $J = 6.9$ Hz, 2H, CH_2–N), 7.31–7.75 (m, 10H, arom), 9.24 (s, 1H, OH), 10.56 (d, $J = 6.9$ Hz, 1H, NH) ppm; ^{13}C NMR

(*DMSO*-d_6, δ, 125.77 MHz): 13.59 (CH_3), 14.02 (CH_3), 45.58 (CH_2–CO), 45.6 (CH_2–CO), 60.84 (CH_2–O), 61.14 (CH_2–O), 126.92, 127.19, 127.46, 127.72, 127.79, 127.87, 128.46, 128.67, 129.40, 131.30, 131.34, 131.52, 132.46 (C-arom) ppm; MS: *m/z* (%) = 378 (37.4) M^+, 350 (12.5) $[M–CO]^+$, 332 (12.5) $[M–CH_3–CH_2O]^+$, 304 (87.5) $[M–COOCH_2CH_3–H]^+$, 105 (23.2) $[C_6H_5CO]^+$, 77 (100) $C_6H_5^+$.

General procedure for the preparation of **5** *and* **7**

To a solution of 2.9 g of **1** (0.01 mol) in toluene, 0.012 mol of 2-hydroxyethanolamine were added. The solution was refluxed for 5 h and left overnight. The excess of toluene was evaporated, and the products were filtered and washed with boiling ethanol. The filtrate was left for crystallization (compound **7**). The precipitate (compound **5**) was filtered off and crystallized from ethanol.

1-Phenyl-3-(2-hydroxyethylimino)-4-(phenyl(hydroxy)-methylidene)-pyrrolidine-2,5-dione (**7**:$C_{19}H_{16}N_2O_4$)

Yield: 14%; m.p.: 125°C; IR (*HCBN*): ν = 3387 (broad, OH), 3250 (broad, OH), 3230 (NH), 1764 (CO), 1709 (CO), 1638(C=N) cm^{-1}; ^{1}H NMR ($CDCl_3$, δ, 500.13 MHz) 2.33 (s, 1H, OH), 3.78 (t, 2H, CH_2–N), 4.23 (q, 2H, CH_2–O) 7.18–7.81 (m. 10H, arom), 10.76 (s, 1H, OH) ppm; ^{13}C NMR (*DMSO*-d_6, δ, 125.77 MHz): 45.24 (CH_2–N), 61.48 (CH_2–OH), 126.64, 127.62, 128.11, 128.72, 128.96, 131.03, 131.76, 138.46 (C-arom) ppm; MS: *m/z* (%) = 336.1 (31.8) M^+, 318 (54.7) $[M–H_2O]^+$, 317(56) $[M–H_2O–H]^+$, 105 (100) $[C_6H_5CO]^+$.

General procedure for the preparation of **8** *and* **9**

To a solution of 0.01 mol of **2 (3)** or **6** in ethanol, 0.012 mol of benzylamine were added. The solution was refluxed for 2.5 h and left overnight. The precipitate was filtered off and crystallized from ethanol.

((N-Phenyl-N′-benzyl)-3-(phenylhydroxymethylidene)-2-(iminobenzyl))-succinic diamide (**8**; $C_{31}H_{27}N_3O_3$)

Yield: 31%; m.p.: 140°C; IR(*HCBN*): ν = 3381 (OH), 3280 (NH), 3272 (NH), 1671 (CO), 1665 (CO), 1644 (C=N), 1611 (C=C) cm^{-1}; ^{1}H NMR (*DMSO*-d_6, δ, 500.13 MHz): 4.05 (d, 2H, CH_2), 4.29 (d, 2H, CH_2), 5.08 (m, 2H, 2NH), 6.98–7.53 (m, 20H, arom), 7.93 (s, 1H, NH), 8.13 (s, 1H, OH) ppm; ^{13}C NMR (*DMSO*-d_6, δ, 125.77 MHz): 42.36 (CH_2–Ph), 45.23 (CH_2–Ph), 107, 118.64, 123.2, 125.69, 126.61, 126.97, 127.35, 127.79, 128.35, 128.55, 128.88, 137.24, 139.35, 140.36 (C-arom) ppm; MS: *m/z*(%) = 471 $[M–H_2O]^+$, 364 (34) $[M–H_2O–C_6H_5CH_2NH_2]^+$, 105 (95) $C_6H_5CO^+$, 77 (100) $C_6H_5^+$.

1-Phenyl-4-(phenyl-(benzylamine)-methylidene)-pyrrolidine-2,3,5-trione (**9**; $C_{24}H_{18}N_2O_3$)

Yield: from **2**, 28%; from **3**, 70%; m.p.: 195°C; IR (*HCBN*): ν = 3470 (v. weak, OH), 3282 (NH), 1763 (CO), 1720 (CO), 1658 (CO) cm^{-1}; ^{1}H NMR (*DMSO*-d_6, δ, 500.13 MHz): 4.36 (s, 2H, CH_2), 7.18–7.89 (m, 15H, arom), 10.5 (s, 1H, NH) ppm; ^{13}C NMR (*DMSO*-d_6, δ, 125.77 MHz): 47.68(CH_2Ph), 126.97, 127.60, 127.74, 128.28, 128.49, 128.63, 128.69, 130.42, 130.66, 131.71, 136.72, 138.11, (C-arom) ppm; MS: *m/z*(%) = 382 (36.2) M^+, 354 (20.6) $[M–CO]^+$, 77 (100) $C_6H_5^+$.

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