One-Pot Synthesis of 4-(Alkylamino)-1-(arylsulfonyl)-3-benzoyl-1,5-dihydro-5-hydroxy-5-phenyl-2*H*-pyrrol-2-ones *via* a Multicomponent Reaction

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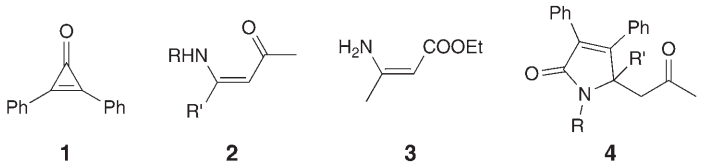
An effective route to novel 4-(alkylamino)-1-(arylsulfonyl)-3-benzoyl-1,5-dihydro-5-hydroxy-5-phenyl-2*H*-pyrrol-2-ones **10** is described (*Scheme 2*). This involves the reaction of an enamine, derived from the addition of a primary amine **5** to 1,4-diphenylbut-2-yne-1,4-dione, with an arenesulfonyl isocyanate **7**. Some of these pyrrolones **10** exhibit a dynamic NMR behavior in solution because of restricted rotation around the C–N bond resulting from conjugation of the side-chain N-atom with the adjacent α,β-unsaturated ketone group, and two rotamers are in equilibrium with each other in solution (**10** $\rightleftharpoons$ **11**; *Scheme 3*). The structures of the highly functionalized compounds **10** were corroborated spectroscopically (IR, ^{1}H- and ^{13}C-NMR, and EI-MS), by elemental analyses, and, in the case of **10a**, by X-ray crystallography. A plausible mechanism for the reaction is proposed (*Scheme 4*).

1. Introduction. – The 1,5-dihydro-2*H*-pyrrol-2-ones (= 3-pyrrolin-2-ones) are important structural units of the structurally related indolocarbazole alkaloids, *e.g.*, (+)-staurosporine [1] and (+)-K252a [2], which are strong kinase inhibitors widely used as molecular tools. On the other hand, 3,4-diaryl- and 1,3,4-triaryl-1,5-dihydro-2*H*-pyrrol-2-ones, have been shown to be a prospective new type of selective COX-2 inhibitors [3][4]. Moreover, the α,β-unsaturated γ-butyrolactam moiety can be utilized as a *Michael* acceptor for a variety of nucleophiles [5]. Therefore, the synthesis of 1,5-dihydro-2*H*-pyrrol-2-ones is currently receiving considerable attention [6], and several interesting methods have been reported to prepare 1,5-dihydro- and 1,3-dihydro-2*H*-pyrrol-2-ones with two aryl groups at adjacent positions [7–19].

One of the methods developed for the synthesis of 1,5-dihydro-2*H*-pyrrol-2-ones is based on a formal [2+3] cycloaddition reaction of diphenylcyclopropenone (**1**) [20] with imines [7] or diimines [8][19]. In fact, some years ago, it was found that the reaction of **1** with acyclic enaminones **2** and amino ester **3** in refluxing toluene leads to the formation of the 5-functionalized 3,4-diphenyl-1,5-dihydro-2*H*-pyrrol-2-ones **4** in good yields [7].

Enaminones are widely used building blocks for the synthesis of various organic compounds [21], especially for bioactive natural products and their analogs [22]. Enamines belong to the most important intermediates for C–C bond-formation in both organic chemistry and the biological world. In organic synthesis, pyrrolidine

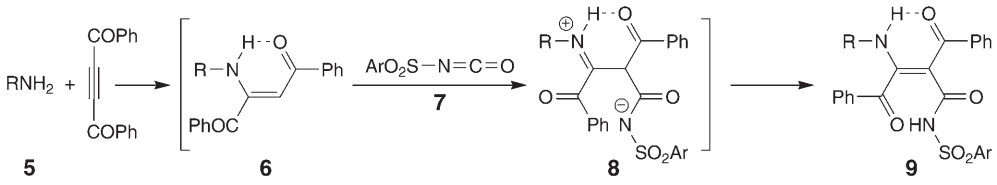


1 2 3 4

derivatives are used to efficiently form enamines with carbonyl compounds in many reactions.

In the context of our ongoing studies on heterocyclic synthesis mediated by enamine intermediates, the possibility of trapping the 1:1 intermediate **6**, formed between 1,4-diphenylbut-2-yne-1,4-dione (=dibenzoylacetylene) and a primary amine **5**, with an arenesulfonyl isocyanate **7** appeared attractive from the viewpoint of devising a novel multicomponent reaction. Although the trapping of the 1:1 intermediate formed between 1,4-diphenylbut-2-yne-1,4-dione and a secondary amine, with an arenesulfonyl isocyanate **7** has been studied in detail by our research group [23][24], the analogous reaction with primary amines has not been reported. We expected that compound **9** should result from the reaction of an enaminone of type **6** with **7** (*Scheme 1*).

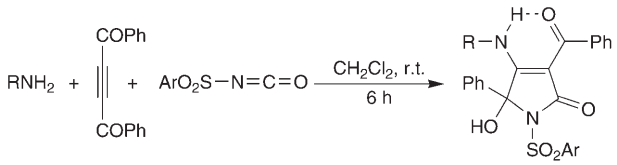
Scheme 1



5 6 8 9

Herein, we report a simple one-pot reaction between primary amines **5**, 1,4-diphenylbut-2-yne-1,4-dione, and an arenesulfonyl isocyanate **7** leading to 1,5-dihydro-5-hydroxy-2*H*-pyrrol-2-one derivatives **10** (*Scheme 2*).

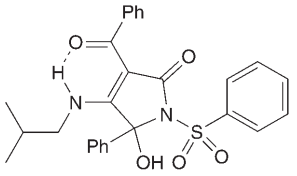
Scheme 2



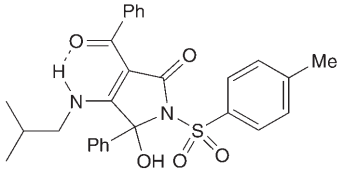
5 7 10

2. Results and Discussion. – The one-pot reaction proceeded *via* a smooth 1:1:1 addition of the primary amine **5**, 1,4-diphenylbut-2-yne-1,4-dione, and arenesulfonyl isocyanate **7** in anhydrous CH_2Cl_2 at room temperature to give 1,5-dihydro-5-hydroxy-2*H*-pyrrol-2-one derivatives **10** in 90–98% yields (*Scheme 2* and *Table*). The structures

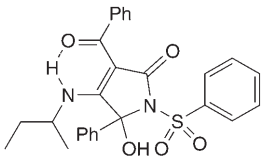
Table. *Condensation Cyclization Reactions of 1,4-Diphenylbut-2-yne-1,4-dione with Primary Amines* **5** *in the Presence of Arenesulfonyl Isocyanates* **7** *yielding 1,5-Dihydro-5-hydroxy-2*H*-pyrrol-2-ones* **10**



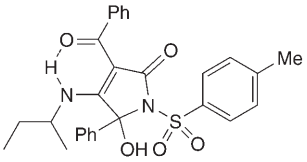
10a



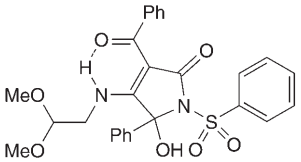
10b



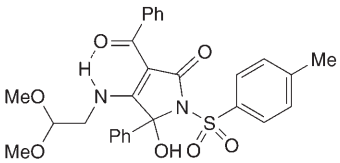
10c



10d



10e



10f

5	**7**	Product	Yield [%][a])
$Me_2CHCH_2NH_2$	$PhSO_2NCO$	**10a**	95
$Me_2CHCH_2NH_2$	4-$MeC_6H_4SO_2NCO$	**10b**	95
$MeCH_2CH(Me)NH_2$	$PhSO_2NCO$	**10c**	95
$MeCH_2CH(Me)NH_2$	4-$MeC_6H_4SO_2NCO$	**10d**	95
$(MeO)_2CHCH_2NH_2$	$PhSO_2NCO$	**10e**	90
$(MeO)_2CHCH_2NH_2$	4-$MeC_6H_4SO_2NCO$	**10f**	98

[a]) Isolated yields.

of compounds **10a–f** were deduced from elemental analysis, IR, ^{1}H- and ^{13}C-NMR, and mass spectra. The ^{1}H- and ^{13}C-NMR spectra of the crude precipitate clearly indicated the formation of derivatives **10** as the only product. The structure of **10a** was elucidated by a single crystal X-ray diffraction analysis (*Fig.*).

The mass spectrum of **10a** displayed the molecular-ion peak at *m*/*z* 490, which is consistent with the structure of a 3-benzoyl-1,5-dihydro-5-hydroxy-4-(isobutylamino)-5-phenyl-1-(phenylsulfonyl)-2*H*-pyrrol-2-one. The IR spectrum exhibited absorption bands for the carbonyl groups of the pyrrol-2-one and benzoyl moieties at 1705 and 1620 cm^{-1}, respectively, for OH and NH groups at 3410 and 3030 cm^{-1}, and for the sulfonyl moiety at 1375 and 1185 cm^{-1}. The ^{1}H-NMR spectrum of compound **10a** exhibited seven sharp signals readily recognized as arising from two Me (δ 1.03 and 1.05), a CH (δ 2.21), a CH_2 (δ 3.41 and 3.60), an OH (δ 6.14), and an NH group (δ 8.35). The ^{1}H-decoupled ^{13}C-NMR spectrum of **10a** showed 21 distinct resonances in agreement with the 3-benzoyl-1,5-dihydro-5-hydroxy-4-(isobutylamino)-5-phenyl-1-(phenylsulfonyl)-2*H*-pyrrol-2-one structure.

The ^{1}H- and ^{13}C-NMR spectra of compounds **10b–f** are similar to those of **10a**, except for the amine moieties, which exhibit characteristic signals with appropriate chemical shifts.

The compounds **10b–d** indicate dynamic NMR behavior in solution because of restricted rotation around the C–N bond, resulting from conjugation of the side-chain N-atom with the adjacent α,β-unsaturated ketone group, and two rotamers (**10** $\rightleftharpoons$ **11**) are in equilibrium with each other in solution (*Scheme 3*).

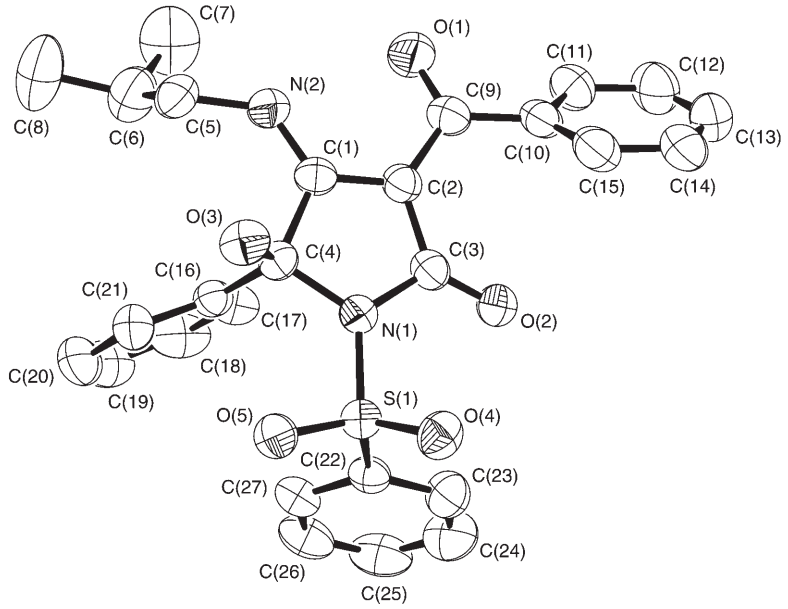
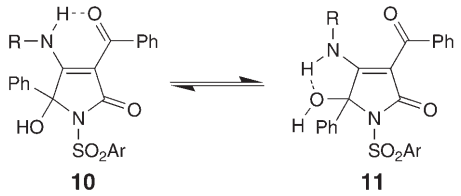


Figure. *Molecular structure of compound* **10a**

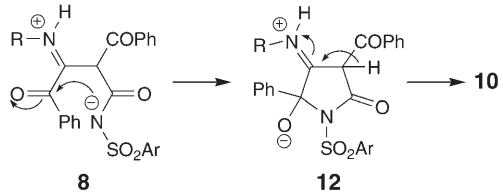
Scheme 3



Although we have not established the mechanism of the reaction of enamines **6** and an arenesulfonyl isocyanate **7** in an experimental manner, a possible explanation is proposed in *Scheme 4*. Compounds **10** apparently result from the initial addition of the primary amine to the 1,4-diphenylbut-2-yne-1,4-dione and subsequent attack of the resulting reactive enamine **6** (*Scheme 1*) on the arenesulfonyl isocyanate **7** [25][26] to yield a betaine **8**, which cyclizes to give **12** (*Scheme 4*), finally, a proton shift gives the 1,5-dihydro-5-hydroxy-2*H*-pyrrol-2-one **10**.

In summary, the reaction between a primary amine and 1,4-diphenylbut-2-yne-1,4-dione in the presence of an arenesulfonyl isocyanate provides a simple one-pot entry into the synthesis of 1,5-dihydro-5-hydroxy-2*H*-pyrrol-2-one derivatives of potential synthetic and pharmaceutical interest. The present method has the advantage of being performed under neutral conditions and requiring no activation or modification of the starting materials. The simplicity of the procedure makes it an interesting alternative to complex multistep approaches [27].

Scheme 4



Experimental Part

General. Benzenesulfonyl isocyanate and 4-methylbenzenesulfonyl isocyanate were obtained from *Merck* (Germany) and *Fluka* (Switzerland) and were used without further purification. The 1,4-diphenylbut-2-yne-1,4-dione was prepared according to a published procedure [28][29]. CC = Column chromatography. Melting points: *Electrothermal 9100* apparatus. IR Spectra: *Shimadzu IR-460* spectrometer. ^{1}H- and ^{13}C-NMR Spectra ($CDCl_3$): *Bruker DRX-500-Avance* spectrometer; at 500.1 and 125.7 MHz, resp.; δ in ppm, J in Hz. Mass spectra: *Finnigan MAT-8430* mass spectrometer; ionization potential 20 eV. Elemental analyses: *Herpes CHN-O-Rapid* analyzer.

Compounds **10**: *General Procedure*, *Exemplified for* **10a**. A soln. of 1,4-diphenylbut-2-yne-1,4-dione (0.23 g, 1 mmol) and isobutylamine (=2-methylpropan-1-amine; 0.073 g, 1 mmol) in anh. CH_2Cl_2 (5 ml) was magnetically stirred for 1 h. Then a soln. of benzenesulfonyl isocyanate (**7a**; 0.18 g, 1 mmol) in anh. CH_2Cl_2 (3 ml) was added dropwise at r.t. The mixture was stirred for 5 h. The solvent was evaporated, and the residue separated by CC (silica gel (*Merck* 230–240 mesh), hexane/AcOEt mixtures).

*3-Benzoyl-1,5-dihydro-5-hydroxy-4-[(2-methylpropyl)amino]-5-phenyl-1-(phenylsulfonyl)-2*H*-pyrrol-2-one* (**10a**). Yield 470 mg (95%). Colorless crystals. M.p. 170–172°. IR (KBr): 3410 (OH), 3030 (NH), 1754 (C=O), 1626 (NC=C), 1575 and 1440 (Ar), 1375 and 1185 (SO_2), 1212 and 1082 (CO). ^{1}H-NMR ($CDCl_3$): 1.03 (*d*, *J* = 6.5, 3 H); 1.05 (*d*, *J* = 6.5, 3 H); 2.19–2.22 (*m*, 1 H); 3.41 (*dd*, *J* = 13.7, 7.0, 1 H); 3.60 (*dd*, *J* = 13.8, 7.5, 1 H); 6.14 (*s*, 1 H); 7.29–7.56 (*m*, 11 H); 7.69 (*d*, *J* = 7.2, 2 H); 7.80 (*d*, *J* = 7.3, 2 H); 8.35 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 19.54; 19.68; 25.91; 48.18; 90.32; 94.85; 125.77; 126.97; 127.49; 127.84; 127.94; 128.29; 128.31; 132.49; 133.04; 137.21; 137.34; 138.40; 150.76; 160.90; 189.58. EI-MS: 490 (3, $M^{+\cdot}$), 333 (6), 264 (5), 228 (5), 202 (6), 146 (19), 105 (100), 91 (9), 57 (14), 51 (19), 41 (17). Anal. calc. for $C_{27}H_{26}N_2O_5S$ (490.56): C 66.11, H 5.34, N 5.71; found: C 67.00, H 5.26, N 5.63.

Crystal Data of **10a** ($C_{27}H_{26}N_2O_5S$)[1]): M_w 490.56, triclinic, space group $P\bar{1}$, a = 8.3282(9) Å, b = 12.2023(13) Å, c = 13.5584(14) Å, α = 111.607(1), β = 93.525(1), γ = 103.671(1)°, V = 1227.8(2) Å^3, Z = 2, D_c = 1.327 mg/m^3, $F(000)$ = 516, crystal dimension 0.38 × 0.26 × 0.17 mm, radiation, MoK_α (λ 0.71073 Å), $2.55 \leq 2\theta \leq 25.50$. Intensity data were collected at 295 K with a *Bruker APEX*-area-detector diffractometer by the $\omega/2\theta$ scanning technique, in the range of $-7 \leq h \leq 10$, $-14 \leq k \leq 14$, and $-16 \leq l \leq 15$; the structure was solved by a direct method, all non-H-atoms were positioned and anisotropic thermal parameters refined from 3796 observed reflections with $R(\text{int}) = 0.0125$ by a full-matrix least-squares technique converged to $R = 0.0439$ and $R_w = 0.1104$ ($I > 2\sigma(I)$).

*3-Benzoyl-1,5-dihydro-5-hydroxy-1-[(4-methylphenyl)sulfonyl]-4-[(2-methylpropyl)amino]-5-phenyl-2*H*-pyrrol-2-one* (**10b**). Yield 480 mg (95%). Colorless crystals. M.p. 155–157°. IR (KBr): 3480 (OH), 3030 (NH), 1748 (C=O), 1619 (NC=C), 1581 and 1462 (Ar), 1365, 1163 (SO_2), 1210, 1082 (CO). EI-MS: 504 (4, $M^{+\cdot}$), 440 (7), 349 (6), 333 (10), 246 (8), 228 (9), 172 (7), 155 (5), 146 (7), 105 (100), 91 (32), 77 (33), 57 (11), 41 (9). Anal. calc. for $C_{28}H_{28}N_2O_5S$ (504.60): C 66.65, H 5.59, N 5.55; found: C 66.45, H 5.63, N 5.48.

Major Rotamer of **10b** (60%): ^{1}H-NMR ($CDCl_3$): 0.68 (*d*, *J* = 6.6, 3 H); 0.81 (*d*, *J* = 6.6, 3 H); 1.44–1.47 (*m*, 1 H); 2.33 (*s*, 3 H); 2.98–3.03 (*m*, 1 H); 3.27–3.32 (*m*, 1 H); 5.46 (*s*, 1 H); 7.01 (*d*, *J* = 7.8, 2 H);

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[1]) CCDC-642464 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge *via* www.ccdc.cam.ac.uk/data_request/cif.

7.21 (*d*, *J* = 7.80, 2 H); 7.25 – 7.69 (*m*, 8 H); 7.8 (*d*, *J* = 7.8, 2 H); 8.36 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 19.43; 20.25; 21.55; 28.44; 51.46; 88.85; 95.40; 126.16; 127.66; 127.85; 128.73; 128.77; 129.02; 129.41; 131.61; 136.16; 136.53; 138.53; 144.38; 161.70; 172.39; 192.88.

Minor Rotamer of **10b** (40%): ^{1}H-NMR ($CDCl_3$): 1.02 – 1.13 (*m*, 6 H); 2.19 – 2.25 (*m*, 1 H); 2.40 (*s*, 3 H); 3.39 (*dd*, *J* = 14.7, 6.8, 1 H); 3.60 (*dd*, *J* = 14.1, 7.5, 1 H); 6.13 (*s*, 1 H); 7.00 – 7.78 (*m*, 14 H); 10.69 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 19.57; 20.37; 21.66; 26.61; 48.84; 90.94; 96.22; 126.45; 127.60; 128.15; 128.58; 128.83; 128.90; 129.13; 133.11; 136.17; 137.96; 138.05; 144.80; 161.70; 164.16; 190.21.

*3-Benzoyl-1,5-dihydro-5-hydroxy-4-[(1-methylpropyl)amino]-5-phenyl-1-(phenylsulfonyl)-2*H*-pyrrol-2-one* (**10c**). Yield 460 mg (95%). Colorless crystals. M.p. 180 – 182°. IR (KBr): 3420 (OH), 3215 (NH), 1718 (C=O), 1616 (NC=C), 1547, 1442 (Ar), 1361, 1166 (SO_2), 1241, 1081 (CO). EI-MS: 491 (3, $[M+1]^+$), 333 (9), 247 (3), 190 (5), 172 (5) 105 (100), 77 (55), 57 (5), 51 (9), 41 (10). Anal. calc. for $C_{27}H_{26}N_2O_5S$ (490.57): C 66.11, H 5.34, N 5.71; found: C 66.00, H 5.26, N 5.63.

Major Rotamer of **10c** (80%): ^{1}H-NMR ($CDCl_3$): 0.41 (*d*, *J* = 6.4, 3 H); 0.97 (*t*, *J* = 7.3, 3 H); 1.53 – 1.59 (*m*, 2 H); 3.90 – 3.99 (*m*, 1 H); 5.50 (*s*, 1 H); 7.21 (*t*, *J* = 7.4, 2 H); 7.30 (*t*, *J* = 7.2, 2 H); 7.34 – 7.53 (*m*, 9 H); 7.78 (*d*, *J* = 7.6, 2 H); 10.45 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 10.03; 19.89; 30.51; 52.42; 88.80; 95.47; 126.03; 127.68; 127.72; 128.43; 128.84; 128.86; 129.39; 131.61; 133.28; 136.95; 138.55; 139.12; 164.17; 177.45; 192.83.

Minor Rotamer of **10c** (20%): ^{1}H-NMR ($CDCl_3$): 0.24 (*t*, *J* = 7.3, 3 H); 1.29 (*d*, *J* = 6.3, 3 H); 1.66 – 1.72 (br. *m*, 2 H); 3.79 – 3.85 (*m*, 1 H); 5.52 (*s*, 1 H); 7.19 – 7.79 (*m*, 15 H); 10.47 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 9.46; 21.96; 31.26; 52.66; 88.95; 95.51; 126.07; 127.68; 127.73; 128.43; 128.79; 128.87; 129.44; 131.64; 133.28; 136.96; 138.56; 139.13; 164.17; 177.63; 192.85.

*3-Benzoyl-1,5-dihydro-5-hydroxy-1-[(4-methylphenyl)sulfonyl]-4-[(1-methylpropyl)amino]-5-phenyl-2*H*-pyrrol-2-one* (**10d**). Yield 480 mg (95%). Colorless crystals. M.p. 177 – 179°. IR (KBr): 3375 (OH), 3050 (NH), 1717 (C=O), 1617 (NC=C), 1549, 1443 (Ar), 1365, 1161 (SO_2), 1210, 1000 (CO). EI-MS: 505 (1, $[M+1]^+$), 333 (6), 278 (6), 172 (5), 105 (11), 41 (5). Anal. calc. for $C_{28}H_{28}N_2O_5S$ (504.60): C 66.65, H 5.59, N 5.55; found: C 65.90, H 5.73, N 5.43.

Major Rotamer of **10d** (70%): ^{1}H-NMR ($CDCl_3$): 0.42 (*d*, *J* = 6.5, 3 H); 0.98 (*t*, *J* = 7.4, 3 H); 1.53 – 1.59 (*m*, 2 H); 2.33 (*s*, 3 H); 3.92 – 3.98 (*m*, 1 H); 5.46 (*s*, 1 H); 7.01 (*d*, *J* = 8.00, 2 H); 7.27 (*d*, *J* = 8.30, 2 H); 7.28 – 7.60 (*m*, 8 H); 7.78 (*d*, *J* = 8.10, 2 H); 10.45 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 10.02; 19.92; 21.96; 30.53; 52.39; 88.94; 95.51; 126.05; 127.65; 127.86; 128.82; 128.85; 129.02; 129.35; 131.58; 136.19; 137.12; 138.58; 144.36; 164.16; 171.46; 192.83.

Minor Rotamer of **10d** (30%): ^{1}H-NMR ($CDCl_3$): 0.25 (*t*, *J* = 7.4, 3 H); 1.30 (*d*, *J* = 6.4, 3 H); 1.61 – 1.68 (br. *m*, 2 H); 2.33 (*s*, 3 H); 3.85 – 3.85 (*m*, 1 H); 5.48 (*s*, 1 H); 7.00 – 7.78 (*m*, 14 H); 10.47 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 9.45; 21.96; 29.55; 31.25; 52.62; 88.79; 96.00; 126.10; 127.70; 127.88; 128.77; 128.86; 129.06; 129.38; 131.60; 136.09; 136.99; 138.32; 144.30; 164.02; 171.64; 192.98.

*3-Benzoyl-4-[(2,2-dimethoxyethyl)amino]-1,5-dihydro-5-hydroxy-5-phenyl-1-(phenylsulfonyl)-2*H*-pyrrol-2-one* (**10e**). Yield 470 mg (90%). Colorless crystals. M.p. 150 – 152°. IR (KBr): 3410 (OH), 3035 (NH), 1751 (C=O), 1631 (NC=C), 1574, 1436 (Ar), 1375, 1167 (SO_2), 1214, 1079 (CO). ^{1}H-NMR ($CDCl_3$): 3.46 (*s*, 3 H); 3.47 (*s*, 3 H); 3.58 (*dd*, *J* = 14.6, 7.1, 1 H); 3.99 (*dd*, *J* = 14.3, 7.1, 1 H); 4.60 – 4.64 (*m*, 1 H); 6.47 (*s*, 1 H); 7.26 – 7.55 (*m*, 11 H); 7.72 (*d*, *J* = 6.9, 2 H); 7.77 (*d*, *J* = 6.8, 2 H); 8.37 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 43.44; 55.12; 55.71; 91.10; 96.73; 102.28; 126.35; 127.48; 128.23; 128.48; 128.49; 128.50; 128.89; 133.16; 133.71; 137.74; 137.81; 138.80; 151.11; 160.81; 190.35. EI-MS: 307 (5), 141 (7), 105 (21), 91 (6), 75 (100), 51 (17). Anal. calc. for $C_{27}H_{26}N_2O_7S$ (522.57): C 62.06, H 5.01, N 5.36; found: C 62.00, H 5.10, N 5.40.

*3-Benzoyl-4-[(2,2-dimethoxyethyl)amino]-1,5-dihydro-5-hydroxy-1-[(4-methylphenyl)sulfonyl]-5-phenyl-2*H*-pyrrol-2-one* (**10f**). Yield 520 mg (98%). Colorless crystals. M.p. 145 – 147°. IR (KBr): 3390 (OH), 3160 (NH), 1754 (C=O), 1636 (NC=C), 1570, 1435 (Ar), 1373, 1187 (SO_2), 1212, 1070 (CO). ^{1}H-NMR ($CDCl_3$): 2.41 (*s*, 3 H); 3.49 (*s*, 3 H); 3.50 (*s*, 3 H); 3.59 (*dd*, *J* = 14.6, 5.6, 1 H); 3.99 (*dd*, *J* = 14.6, 4.4, 1 H); 4.65 (*t*, *J* = 4.7, 1 H); 6.47 (*s*, 1 H); 7.21 (*d*, *J* = 7.9, 2 H); 7.28 – 7.29 (*m*, 3 H); 7.38 (*t*, *J* = 7.3, 2 H); 7.51 (*t*, *J* = 7.7, 1 H); 7.58 – 7.59 (*m*, 2 H); 7.69 (*d*, *J* = 7.5, 2 H); 7.72 (*d*, *J* = 7.9, 2 H); 8.36 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 21.67; 43.62; 55.28; 55.77; 91.21; 96.70; 102.50; 126.47; 127.53; 128.30; 128.55; 128.74; 128.89; 129.16; 133.16; 136.19; 137.95; 138.11; 144.84; 151.27; 161.06; 190.42. EI-MS: 308 (6), 307 (12), 105 (23),

91 (11), 75 (100), 47 (8). Anal. calc. for $C_{28}H_{28}N_2O_7S$ (536.59): C 62.67, H 5.26, N 5.22; found: C 61.52, H 5.10, N 5.11.

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