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Synthesis and Structural Studies of New *N*-(*p*-Toluenesulfonyl)amino Acid *o*-Phenolamides

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ABSTRACT: N-(p-*Toluenesulfonyl)glycine* o-*phenolamide* (**3a**) *and the analogous derivatives of* d,l-*alanine* (**3b**), L-*valine* (**3c**), L-*leucine* (**3d**), *and* L-*phenylalanine* (**3e**) *were synthesized in yields >80% by condensation of* N-(p-*toluenesulfonyl)amino acyl chlorides with* o-*aminophenol. The structure and conformation of these amides were established by NMR spectroscopy and X-ray crystallography.* © 2003 Wiley Periodicals, Inc. Heteroatom Chem 14:247–253, 2003; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.10135

INTRODUCTION

N-(*p*-Toluenesulfonyl)-protected amino acids and amides are useful synthetic intermediates for obtaining new substances with overhanging biological activity [1–7]. Therefore, preparation of amides with specific and conformationally constrained molecular structures constitute an alternative for development of new compounds with pharmaceutical utility [1,3]. We are interested in the synthesis and structural study of amides that can be used to give coordination complexes with optical activity [8,9]. Our work has been focused to the study of ligands where the presence of *intra*molecular interactions determine their conformation. Moreover, the coordination of a metallic ion with these compounds can lead to heterocyclic compounds [10,11]. Here, we describe the synthesis of a series of *o*-phenolamides **3** from *N*-(*p*-toluenesulfonyl)amino acids.

RESULTS AND DISCUSSION

Treatment of tosyl-amino acid (**1**) with thionyl chloride yield the acyl chlorides **2**. The subsequent condensation of **2** with *o*-aminophenol in a 1:1 molar ratio gave the *N*-(*p*-toluenesulfonyl)amino acid *o*-phenolamides **3**. Condensation of *N*-(*p*-toluenesulfonyl)glycyl chloride (**2a**) or *N*-(*p*-toluenesulfonyl)-*d,l*-alanyl chloride (**2b**) gave the achiral amide **3a** and racemate **3b** (Scheme 1), while the reaction of **2c–2e** led to the enantiomers **3c–3e** in yields >80% (Scheme 2). Evidence for the formation of these compounds was obtained from their ^{1}H, ^{13}C, and ^{15}N NMR spectra. Unequivocal ^{1}H and ^{13}C NMR assignments for **3** were achieved by 2D ^{1}H-^{13}C correlated experiments. HETCOR spectra

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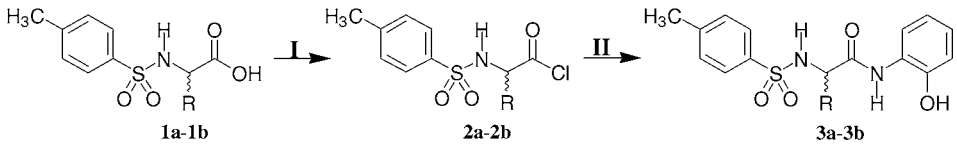


a R = H
b R = CH_3

Reagents: (I) $SOCl_2$, (II) *o*-aminophenol, THF.

SCHEME 1

allowed the complete assignment of **3**. Amide structures **3a, 3b, 3d**, and **3e** were corroborated by X-ray crystallography.

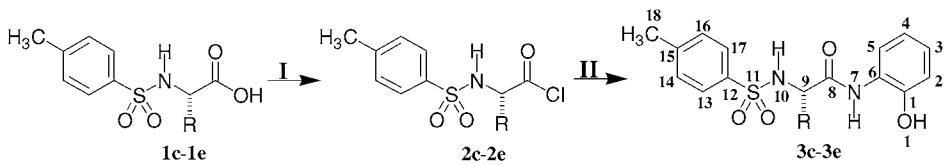
The ^{1}H NMR chemical shifts of compounds **3a–e** show the amide synthesis (Table 1). Aromatic signals have a complex pattern similar to oxamides derivatives from *o*-aminophenol and norephedrine [12,13]. For H5, $\delta = 7.56$–7.90 ppm because of the deshielding action of the carbonyl group. Similar deshielding effects has been reported in the literature and indicate that amides **3** in solution have the phenolic group in opposed position to the carbonyl group [12]. Variable temperature experiments show that chemical shift of H5 moves low frequencies when the temperature increases. The signals of the amidic and sulfonamide N—H protons have different multiplicities; but, in order to avoid a confusion with the assignment of labile protons OH, for **3e** a ^{15}N/^{1}H 2D heteronuclear correlation experiment was carried out. The chemical shifts of amidic N—H of 9.3 ppm show acid character with possibly an intramolecular hydrogen bond to O1. On the other hand, sulfonamide N—H resonances (7.93–8.27 ppm) are coupled with H9 and they have double multiplicity ($^3J = 7.9$ Hz for **3b**, 8.4 Hz for **3c**, 7.7 Hz for **3d**, and 8.7 Hz for **3e**), with the exception of **3a**. These coupling constants are due to hard N—H bonds and indicate similar conformations of amides **3a–e**.

The ^{15}N NMR spectra of **3** (Table 2) showed typical chemical shift values of amidic CN—H for *o*-phenolamides with intramolecular hydrogen bonding ($\delta = -259$ to -264 ppm) [12–14]. The coupling constants ^1J(NH) between 90 and 91 Hz for *o*-phenolamides show the residence of the proton at the nitrogen atom and also indicate a trigonal planar geometry for these nitrogen atoms [15]. Sulphonyl nitrogen atoms have small coupling constants ^1J(NH) = 84–88 Hz that show their pyramidal geometry [15].

^{2}D ^{13}C/^{1}H HETCOR experiments allowed us the unambiguous assignment of all carbon atoms of compounds **3**. Thus, carbonyl signals in ^{13}C NMR spectra (Table 3) have chemical shifts ($\delta = 166.6$–170.4 ppm) characteristic for amides and sulphonyl oligopeptides [16–19]. ^{13}C NMR signal of *o*-aminophenol fragment were assigned in comparison with oxamides [12].

The electron impact–mass spectra to 10 eV of **3** were obtained using a gas chromatograph coupled to a quadrupole mass spectrometer. In the mass spectra the M^+ was observed only for **3c**. However, the base peak in all cases was the tropylium ion (*m/e* = 91). Formation of these ions is a result of the fragmentation of the C—S bond in the molecular ion.

The X-ray molecular structure of **3a, 3b, 3d**, and **3e** (Table 4) showed similar conformation as found in solution by NMR spectroscopy (Fig. 1). These amides have the *trans* conformation with aromatic and carbonyl groups at the same side [torsion angles C6—N7—C8—O2 are 9.8°(6) for **3a**, 1.6°(6) for



c R = $CH(CH_3)_2$
d R = $CH_2CH(CH_3)_2$
e R = $CH_2C_6H_5$

Reagents: (I) $SOCl_2$, (II) *o*-aminophenol, THF.

SCHEME 2

TABLE 1 δ^1H of **3a–e** in DMSO-d_6

	H1	*H2*	*H3*	*H4*	*H5*	*H7*	*H9*	*H10*	*H13*	*H14*	*H18*	*CH*	*CH_2*	*CH_3*
3a	10.03	6.92	6.76	6.75	7.90	9.23	3.64	8.27	7.38	7.74	2.34	–	–	–
3b	9.99	6.88	6.92	6.75	7.87	9.23	3.97	8.26	7.36	7.74	2.34	–	–	1.45
3c	9.76	6.84	6.91	6.71	7.56	9.23	3.78	7.93	7.27	7.70	2.30	1.95	–	0.77 0.82
3d	9.85	6.86	6.91	6.73	7.69	9.26	3.85	8.19	7.30	7.71	2.30	1.50	1.42	0.60 0.78
3e[a]	9.85	6.88	6.93	6.74	7.68	9.37	4.27	8.25	7.12	7.45	2.26	–	3.00 2.73	–

[a]7.14–7.18 (Ph).

3b, 0.2°(6) for **3d**, and −3.2°(5) for **3e**]. The phenolic group is opposed to the carbonyl group [with torsion angles C1—C6—N7—C8 of 156.7°(3) for **3a**, 151.1°(4) for **3b**, −162.8°(4) for **3d**, and −173.4°(3) for **3e**]. Thus, the distance O2—C5 [2.939(4) Å for **3a**, 2.905(5) Å for **3b**, 2.878(5) Å for **3d**, and 2.944(4) Å for **3e**] is below the sum of the van der Waals radii (r_{VDW} = 3.20 Å) and therefore the deshielding action on H5 by O2 observed in ^{1}H NMR is explained [20].

Intramolecular hydrogen bonds play an important role in the molecular conformation of peptides and amides [21,22]. Molecular structures of amides **3** have two intramolecular hydrogen bonds. Hydrogen H7 are interacting with phenolic oxygen O1 and nitrogen N10. Hydrogen bond lengths O1···H7 [2.20(3) Å for **3a**, 2.34(4) Å for **3b**, 2.29(3) Å for **3d**, and 2.15(4) Å for **3e**] and H7···N10 [2.29(3) Å for **3a**, 2.27(4) Å for **3b**, 2.28(3) Å for **3d**, and 2.46(4) Å for **3e**] are below the sum of the van der Waals radii of hydrogen (r_{VDW} = 1.20 Å), oxygen (r_{VDW} = 1.50 Å), or nitrogen (r_{VDW} = 1.55 Å) [20]. Interaction angles O1···H7—N7 [111°(2) for **3a**, 104°(3) for **3b**, 103°(2) for **3d**, and 118°(4) for **3e**] and N10···H7—N7 [113°(3) for **3a**, 106°(3) for **3b**, 115°(2) for **3d**, and 106°(3) for **3e**] favor O1—H7 and N10—H7 hydrogen bonds [20]. The arrangement of the intramolecular interactions O···H···N forms two five-membered chelate rings where the hydrogen H7 acts as pivot and closes the fused *pseudo*-bicyclic structure.

The bond distance between N7 and N10 [2.726(4) Å for **3a**, 2.698(5) Å for **3b**, and 2.783(4) Å for **3d**] is below the sum of the van der Waals radii (r_{VDW} = 3.10 Å), and angles N10···H7—N7 show that H7 has a hydrogen bond with N10. This fact is corroborated with the geometry of the atom N10 which is pyramidal for amides **3a, 3b**, and **3d**. However, the crystal structure of **3e** shows that H7 has a more stable interaction with O3, the bond distance H7—O3 [2.41(4) Å] is similar to H7—N10 [2.46(4) Å], but the interaction angle N7—H7···O3 is almost colinear [147°(7)]. Thus, N10 for **3e** has a planar geometry and a torsional angle N7—C8—C9—N10 of 45.0°(3) [torsion angles are 16.9°(5) for **3a**, −1.2°(5) for **3b**, and −27.8°(5) for **3d**]. On the other hand, two phenyl groups are parallel in amide **3e**, but atom distances between rings [3.732(4) Å for C12—C20, 3.694 (7) Å for C13—C21, 3.667(8) Å for C14—C22, 3.641(5) Å for C15—C23, 3.648(6) Å for C16—C24, and 3.710(6) Å for C17—C25] indicate that this is the result of steric factors and not of an attractive interaction.

In conclusion, the NMR and crystallographic studies have shown that amides **3** have the same conformation in the solid state and in solution. The molecular structure of **3** is stabilized by intramolecular three-center hydrogen bonds.

TABLE 2 δ^{15}N of **3a–e** in DMSO-d_6 and $^1J(^{15}N\text{-}^1H)$ in Hz

	3a	**3b**	**3c**	**3d**	**3e**
N–CO	−262.0	−264.0	−259.0	−262.0	−260.0
	91.3	91.3	90.5	91.3	91.3
N–S	−288.0	−276.0	−282.0	−278.0	−281.0
	85.4	83.9	86.1	86.1	87.6

EXPERIMENTAL

^{1}H, ^{13}C, and ^{15}N NMR spectra were recorded with JEOL Eclipse 400 MHz (or Varian Inova 400 MHz) spectrometer. Chemical shifts (ppm) are relative to $(CH_3)_4Si$ for ^{1}H and ^{13}C NMR and CH_3NO_2 for ^{15}N NMR. Infrared spectra were recorded on a Perkin-Elmer System 200 FT-IR spectrophotometer. Mass spectra were obtained with an HP5989A equipment. Melting points were obtained on a Mel-Temp II apparatus and are uncorrected. Optical rotations were measured at 589 nm in a 10-cm path length cell with a Perkin-Elmer 341 polarimeter. HPLC was run on a Zorbax ODS C-18 semipreparative column. Elemental microanalyses were determined on

TABLE 3 δ ^{13}C of **3a–d** in DMSO-d_6/$CDCl_3$ and **3e** in DMSO-d_6

	C1	*C2*	*C3*	*C4*	*C5*	*C6*	*C8*	*C9*	*C12*	*C13*	*C14*	*C15*	*C18*
3a	146.7	115.5	124.6	119.4	120.3	125.8	166.6	46.4	143.4	129.6	126.9	136.4	21.3
3b[a]	146.5	114.8	123.9	118.8	119.8	125.9	169.8	52.8	142.8	129.3	126.6	137.2	21.0
3c[b]	147.0	115.6	124.4	118.8	120.7	125.5	169.4	62.7	142.6	129.0	126.6	137.0	20.9
3d[c]	146.8	115.3	124.3	119.0	120.2	125.8	170.4	56.0	142.9	129.2	126.7	136.9	21.0
3e[d]	147.2	115.2	125.7	118.7	121.1	124.3	169.2	58.3	142.1	129.1	126.1	137.6	20.7

[a] 17.8 (CH_3).
[b] 17.6 (CH_3); 18.6 (CH_3); 30.6 (CH).
[c] 21.1 (CH_3); 22.7 (CH_3); 23.9 (CH); 41.3 (CH_2).
[d] 38.1 (CH_2); 126.0 (*p*-Ph); 127.8 (*m*-Ph); 129.1 (*o*-Ph); 136.8 (*i*-Ph).

TABLE 4 Crystal Data of **3a, 3b, 3d**, and **3e**

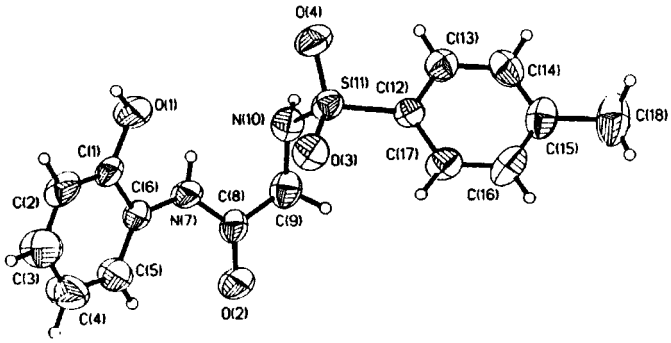
	3a	**3b**	**3d**	**3e**
Molecular formula	$C_{15}H_{16}N_2O_4S$	$C_{16}H_{18}N_2O_4S$	$C_{19}H_{24}N_2O_4S$	$C_{22}H_{22}N_2O_4S$
F_w	320.08	334.10	376.15	410.13
Space group	*Pbca*	*P*2(1)2(1)2(1)	*P*2(1)2(1)2(1)	*P*2(1)2(1)2(1)
a (Å)	14.778 (1)	10.225 (1)	10.142 (2)	7.549 (1)
b (Å)	9.905 (1)	10.677 (1)	13.477 (1)	9.584 (1)
c (Å)	21.107 (1)	19.795 (2)	14.140 (3)	27.547 (3)
V (Å^3)	3089.8 (3)	2161.0 (4)	1968.8 (7)	1993.3 (3)
Z	8	4	4	4
Absorption coefficient (mm^{-1})	0.247	0.181	0.452	2.140
F(000)	1632	864	888	1192
θ range for data collection	1.93° to 23.26°	2.06° to 26.10°	2.07° to 23.25°	1.48° to 26.04°
Reflections collected	15104	14290	4762	13431
Independent reflections	2220 [R(int) = 0.1047]	4289 [R(int) = 0.05312]	2756 [R(int) = 0.0499]	3925 [R(int) = 0.1236]
Completeness to $\theta = 23.26°$	100.0%	99.7%	98.3%	99.9%
Absorption correction	None	None	None	None
Data/restraints/parameters	2220/0/253	4289/0/268	2756/0/332	3925/0/273
Goodness-of-fit on F^2	0.874	0.869	0.814	0.818
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0393$, $wR2 = 0.0609$	$R1 = 0.0505$, $wR2 = 0.1304$	$R1 = 0.0397$, $wR2 = 0.0524$	$R1 = 0.0564$, $wR2 = 0.1031$
R indices (all data)	$R1 = 0.0920$, $wR2 = 0.721$	$R1 = 0.1157$, $wR2 = 0.1587$	$R1 = 0.0703$, $wR2 = 0.0595$	$R1 = 0.1268$, $wR2 = 0.1212$
Largest diff. peak and hole (eÅ^{-3})	0.168 and −0.174	0.214 and −0.162	0.142 and −0.164	0.185 and −0.257

a Perkin-Elmer Series II CHNS/O analyzer 2400 instrument.

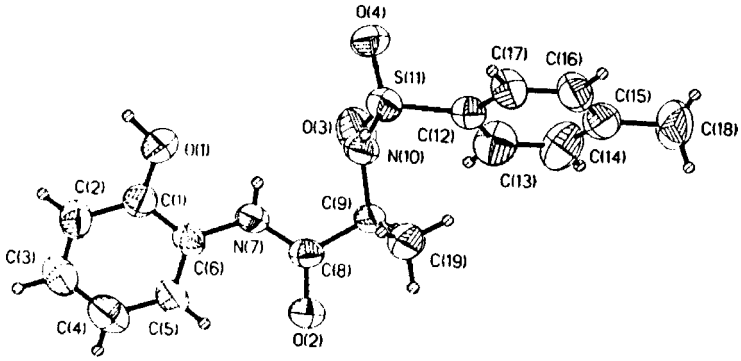
X-Ray Analysis

A selected monocrystal was set upon a Bruker Smart 6000 (or Bruker Smart 1000 for **3e**) diffractometer employing Mo Kα radiation ($\lambda = 0.71069$ Å) at room temperature. After optical alignment, the cell parameters were determined by using the reflections collected on four sets of 20 frames each [23]. Data collection was perfomed in the hemisphere mode and corrections were made for Lorentz and polarization effects. Computations were performed by using SAINT-NT [24]. Atomic form factors for neutral C, N, O, and H were taken from the *International Tables for X-ray Crystallography* [25].

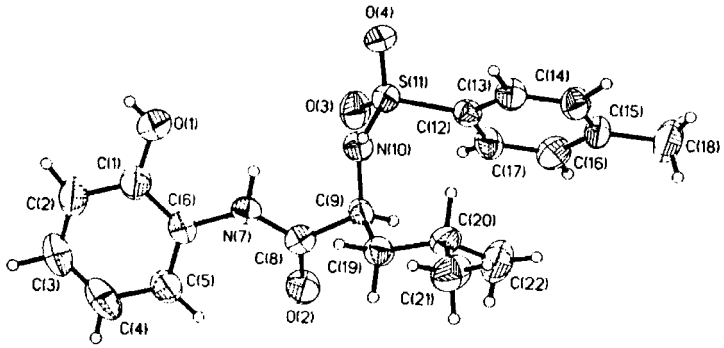
The structures were solved by direct methods using SHELXTL-NT program [26]. Full-matrix least-squares on F^2 was used as a refinement method. Hydrogen atoms were calculated and refined with an overall isotropic temperature factor. Anisotropic temperature factors were introduced for all non-hydrogen atoms, and least-squares refinements were carried out by minimizing $\Sigma w(|F_o| - |F_c|)^2$, where F_o and F_c are the observed and calculated structure factors. Model reached convergence with $R = \Sigma w(\|F_o| - |F_c\|)/|F_o|$ and $R_w = \Sigma[w(|F_o| - |F_c||)^2/\Sigma/w(F_o)2]^{1/2}$. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge



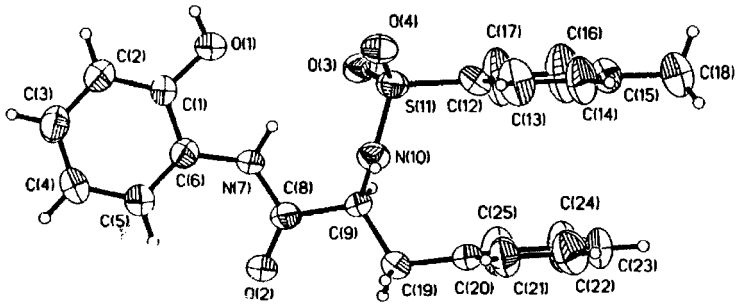
3a



3b



3d



3e

FIGURE 1 Molecular structures of **3a, 3b, 3d**, and **3e**.

Crystallographic Data Centre as supplementary publication nos. CCDC 169818–169821. Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge, CB2 IEZ, UK (fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or www:http://www.ccdc.cam.ac.uk).

*N-(p-Toluenesulfonyl)glycine o-Phenolamide (**3a**)*

The procedure described below is representative for the synthesis of compounds **3a–e**. $SOCl_2$ (1.0 ml) was added to **1a** (0.100 g, 0.43 mmol). The reaction mixture was stirred at room temperature for 1 h and then the $SOCl_2$ excess was removed under vacuum. Compound **2a** was obtained as a white solid and immediately was dissolved in a mixture of *o*-aminophenol (0.048 g, 0.43 mmol) and THF (25 ml). The reaction mixture was stirred for 30 min and the THF was removed under vacuum. Solid obtained was purified by HPLC (70:30 acetonitrile–water) to afford **3a** as a white solid (0.100 g, 80%), which was recrystallized with $CHCl_3$. mp 182–183°C. MS (EI, 10 eV) *m/z*: no M^+, 303 (1), 155 (48), 119 (35), 91 (100). IR ν_{max} (HATR) 3294 (OH), 3411, 3331, 1596, 1270 (NH—CO), 1647 (C=O), 1345, 1135 (SO_2). Anal. calcd for $C_{15}H_{16}N_2O_4S{\cdot}2H_2O$: C, 50.5; H, 5.7; N, 7.9. Found: C, 51.5; H, 4.9; N, 7.7.

*N-(p-Toluenesulfonyl)-d,l-alanine o-Phenolamide (**3b**)*

From **1b** (0.100 g, 0.41 mmol) and *o*-aminophenol (0.043 g, 0.41 mmol) to afford **3b** as a white solid (0.130 g, 95%), which was recrystallized with THF. mp 145–146°C. MS (EI, 10 eV) *m/z*: no M^+, 333 (1), 177 (100), 155 (48), 150 (37), 136 (21), 119 (24), 109 (17), 91 (98). IR ν_{max} (HATR) 3255 (OH), 1598, 1538 (NH—CO), 1663 (C=O), 1326, 1162 (SO_2), 2974, 1456 (CH_3). Anal. calcd for $C_{16}H_{18}N_2O_4S$: C, 57.5; H, 5.4; N, 8.4. Found: C, 57.6; H, 5.4; N, 8.2.

*N-(p-Toluenesulfonyl)-L-valine o-Phenolamide (**3c**)*

From **1c** (0.100 g, 0.369 mmol) and *o*-aminophenol (0.040 g, 0.369 mmol) to afford **3c** as a white solid (0.114 g, 85%). mp 158–159°C. $[\alpha]^{20}_D$ ($c = 0.006$ g/ml in MeOH): −41.6°. MS (EI, 10 eV) *m/z*: no M^+, 362 (4), 226 (48), 155 (63), 109 (59), 91 (100). IR ν_{max} (HATR) 3272 (OH), 1598, 1539 (NH—CO), 1653 (C=O), 1324, 1161 (SO_2), 2965, 2875, 1456 (CH_3). Anal. calcd for $C_{18}H_{22}N_2O_4S$: C, 59.6; H, 6.1; N, 7.7. Found: C, 59.9; H, 6.25; N, 7.6.

*N-(p-Toluenesulfonyl)-L-leucine o-Phenolamide (**3d**)*

From **1d** (100 mg, 0.351 mmol) and *o*-aminophenol (0.038 g, 0.351 mmol) to afford **3d** as a white solid (0.112 g, 85%). mp 134–135°C. $[\alpha]^{20}_D$ ($c = 0.005$ g/ml in MeOH): −75.6°. MS (EI, 10 eV) *m/z*: no M^+, 301 (1), 240 (8), 155 (67), 119 (13), 91 (100). IR ν_{max} (HATR) 3263 (OH), 3069, 1598, 1538 (NH—CO), 1771.4 (C=O), 1325, 1162 (SO_2), 2959, 2871, 1456. Anal. calcd for $C_{19}H_{24}N_2O_4S$: C, 60.6; H, 6.4; N, 7.4. Found: C, 61.4; H, 6.6; N, 7.9.

*N-(p-Toluenesulfonyl)-L-phenylalanine o-Phenolamide (**3e**)*

As before from **1e** (0.100 g, 0.313 mmol) and *o*-aminophenol (0.034 g, 0.313 mmol) to afford **3e** as a white solid (0.109 g, 85%), which was recrystallized from a mixture $CHCl_3$–THF. mp 159–161°C. $[\alpha]^{20}_D$ ($c = 0.003$ g/ml in MeOH): −31.0°. MS (EI, 10 eV) *m/z*: no M^+, 301 (32), 155 (55), 119 (8), 91 (100). IR ν_{max} (HATR) 3259 (OH), 3063, 1598, 1538 (NH—CO), 1660 (C=O), 1323, 1159 (SO_2). Anal. calcd for $C_{22}H_{22}N_2O_4S$: C, 64.4; H, 5.4; N, 6.8. Found: C, 64.5; H, 5.7; N, 6.4.

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