Stereocontrolled approach to δ-amino acids by asymmetric hydrogenation of 5-acetylaminopent-4-enoic acid derivatives

E. V. Starodubtseva,[a] O. V. Turova,[a] O. M. Antipova,[a] M. G. Vinogradov,[a]★ Zh. R. Sagirova,[b] O. R. Malyshev,[a] and M. I. Struchkova[a]

[a]N. D. Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences, 47 Leninsky prosp., 119991 Moscow, Russian Federation. Fax: +7 (499) 135 5328. E-mail: ving@ioc.ac.ru
[b]Samara State University, 1 ul. Akad. Pavlova, 443011 Samara, Russian Federation

Asymmetric hydrogenation of the C=C bond in 5-acetylamino-5-phenylpent-4-enoic acid methyl ester or *N*,*N*-dimethylamide catalyzed by rhodium complexes with chiral bisphosphine ligands (1 mol.% of the catalyst, 20 atm. of H_2, MeOH, 50 °C) gives the corresponding saturated derivatives with enantioselectivity up to 40% *ee*.

Key words: asymmetric hydrogenation, rhodium complexes, bisphosphine chiral ligands, nonracemic 5-acetylamino-5-phenylpentanoic acid.

Amino acids and their derivatives are important natural objects and are widely used in the synthesis of various pharmacologically active compounds and ligands for asymmetric catalysis.[1—3] Widespread methods for the synthesis of chiral amino acids, including asymmetric hydrogenation of unsaturated precursors intensively developed in the last years,[4—6] in fact are mainly used for obtaining acids with the amino group in α- or β-position. Amino acids with more remote amino group, *viz.*, chiral γ- and δ-amino acids, are significantly less available. It is noteworthy that the absolute configuration of one of the simplest chiral δ-amino acids, *i.e.*, levorotating δ-aminocaprylic acid, was unambiguously established[7] only in 1965. As a rule, laborious multi-step schemes involving stoichiometric amounts of inconvenient in handling organometallic reagents and expensive chiral inductors are required for the synthesis of chiral γ- and δ-amino acids.[8—11] There are also known separate examples of solving the same problem by asymmetric hydrogenation of the C=N bond in imino carboxylic acid derivatives with the use of complex catalytic systems.[12]

Earlier, we have studied asymmetric catalytic hydrogenation of prochiral substrates containing a keto group.[13—16] In the present work, we made an attempt to use similar approach to the synthesis of chiral δ-amino acids starting from available δ-keto acids. 5-Oxo-5-phenylpentanoic acid (**1**) has been chosen as the starting compound (Scheme 1), which gave rise to two model substrates, *viz.*, ω-enamides **4** and **9**.

Reductive N-acetylation of the corresponding keto oximes **3** and **8** with finely dispersed iron metal in the presence of Ac_2O and AcOH is the key step in the synthesis of both enamides. Earlier, only simple keto oximes containing no other functional groups have been used in this reaction[17—21] with just rare exceptions.[19,21] In the present work, reductive N-acetylation, as far as we known, was for the first time accomplished for oximes with an ester and amide functional groups, and the earlier undescribed enamide **4** was isolated in the crystalline form as an individual *E*-isomer. Its two-dimensional ^{1}H NMR spectrum (500 MHz, the NOESY procedure) contains an intensive cross-signal, corresponding to the spatial interaction of the phenyl protons (δ 7.3) with the protons of the methylene group (a broad singlet at δ 2.35). At the same time, the spectrum contains no cross-signal responsible for the spatial interaction of the phenyl group with the olefin proton (δ 6.3). Unlike enamide **4**, enamide **9** was not isolated in the isomerically pure form, and it was used in the asymmetric hydrogenation reaction as a mixture of *Z*- and *E*-isomers (~1 : 2).

Comparison of rhodium-containing catalysts and catalytic systems **Rh-1**—**Rh-5** (see Scheme 1) in their activity, as well as enamides **4** and **9** in their reactivity in the hydrogenation reactions studied (30—50 °C, 20 atm of H_2) shows the absence of serious differences in both cases: the 50—100% conversion of these substrates in both the polar (MeOH) and nonpolar (toluene) solvents can be reached within 20—48 h (Table 1). At the same time, the *ee* value changes from virtually nonexistent to 40% depending on the structure of the catalyst and the nature of the solvent.

Assignment of the absolute configuration for the predominant enantiomers of **5** and **10** was made based on the

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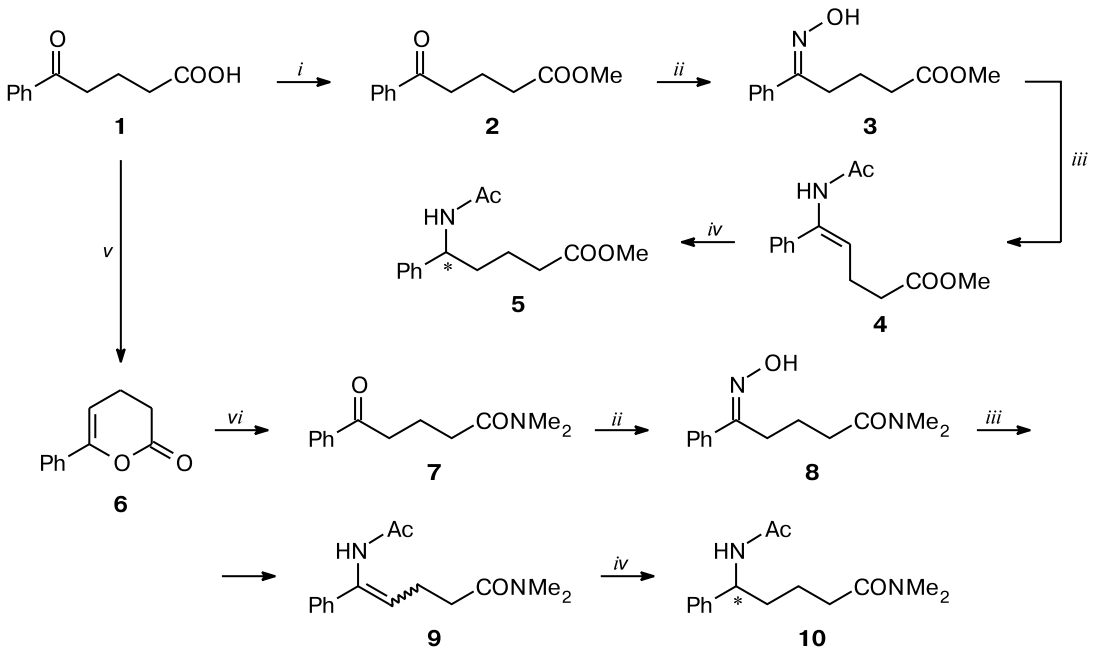
Scheme 1



Reagents: *i*. MeOH, H_2SO_4; *ii*. $NH_2OH \cdot HCl$, AcONa, MeOH, H_2O; *iii*. Fe(0), Ac_2O, AcOH, PhMe; *iv*. H_2, catalyst*; *v*. Ac_2O; *vi*. 33% aq. Me_2NH.

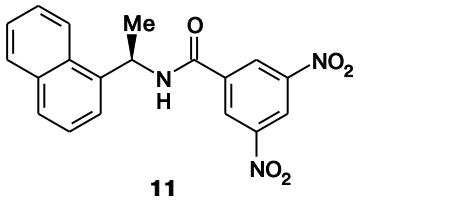
***Catalysts:** [((*S*,*S*)-Me-DuPHOS)Rh(COD)]BF_4 (**Rh-1**); [((*R*,*R*)-Et-DuPHOS)Rh(COD)]OTf (**Rh-2**); [((*R*,*R*)-Me-BPE)Rh(COD)]OTf (**Rh-3**); [$Rh(COD)_2$]OTf + (*R*)-SYNPHOS (**Rh-4**); [$Rh(COD)_2$]OTf + (*R*)-BINAP (**Rh-5**).

known data on stereochemistry of asymmetric hydrogenations of the structurally similar enamides in the presence of the same or similar rhodium catalysts with ligands of the type DuPHOS and BPE.[17—19] The *ee* values for nonracemic hydrogenation products **5** and **10** were determined by ^{1}H NMR in the presence of chiral solvating shift-reagent **11**,[22] with the *CH signal of the enantiomer (+)-(*R*)-**10**, which forms a solvate diastereomeric complex with reagent **11**, being shifted to the high field (Fig. 1).

Table 1. Asymmetric hydrogenation of enamides **4** and **9**[a]

Entry	Catalyst	Solvent	*T* /°C	τ /h	Conversion[b] (%)	Product	*ee*[c] (%)
			Hydrogenation **4**				
1	**Rh-1**	MeOH	30	45	100	(−)-**5**	14 (*S*)
2	**Rh-1**	MeOH	50	20	90	(−)-**5**	18 (*S*)
3	**Rh-1**	Toluene	50	30	100	(−)-**5**	20 (*S*)
4	**Rh-4**	MeOH	50	30	100	(+)-**5**	36 (*R*)
5	**Rh-4**	Toluene	50	30	100	(+)-**5**	21 (*R*)
6	**Rh-4**	THF	50	30	100	(+)-**5**	17 (*R*)
7	**Rh-5**	Toluene	50	30	100	—	<5
			Hydrogenation **9**				
8	**Rh-1**	MeOH	50	20	80	(−)-**10**	42 (*S*)
9	**Rh-1**	MeOH	50	45	90	(−)-**10**	40 (*S*)
10	**Rh-2**	MeOH	30	20	50	(+)-**10**	25 (*R*)
11	**Rh-3**	MeOH	50	45	60	(+)-**10**	38 (*R*)

[a] $p(H_2) = 20$ atm, [substrate] = 0.1 mol L^{-1} in entries *1—3*, *8—11* and 0.2 mol L^{-1} in entries *4—7*; [substrate]/[catalyst] = 100/1.
[b] According to the ^{1}H NMR data.
[c] The *ee* values were determined by ^{1}H NMR in the presence of a chiral solvating shift-reagent **11**.



11

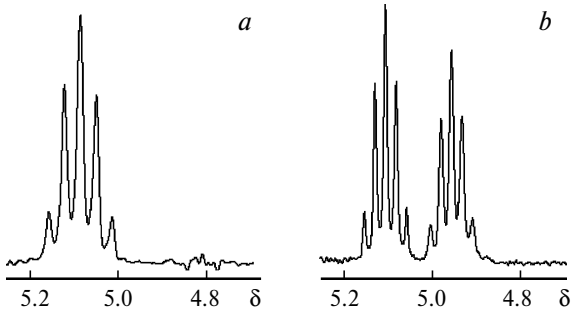


Fig. 1. A fragment of the ^{1}H NMR spectrum (500 MHz, $CDCl_3$) of racemic *N*-acetyl-δ-amino ester **5** (*CH) in the absence of the shift-reagent (*a*) and in the presence of chiral solvating shift-reagent (*R*)-**11** (0.5 equiv.) (*b*).

Earlier, shift-reagent **11** has been used for determination of enantiomeric composition of S-stereogenic chiral compounds.[23]

The results obtained look promising for the improving enantioselectivity of catalytic hydrogenation of enamides, precursors of δ-amino acid derivatives, while further optimization of the process.

Experimental

The following reactants were used in this work: bis(1,5-cyclooctadiene)rhodium(I) triflate ([Rh(COD)$_2$]OTf), (+)-1,2-bis((2*R*,5*R*)-dimethylphospholano)ethane(cyclooctadiene)rhodium triflate ([((*R*,*R*)-Me-BPE)Rh(COD)]OTf) (Acros), (*R*)-(+)-2,2′-bis(diphenylphosphino)-1,1′-binaphthyl ((*R*)-BINAP), (+)-1,2-bis((2*S*,5*S*)-dimethylphospholano)benzene(cyclooctadiene)rhodium tetrafluoroborate ([((*S*,*S*)-Me-DuPHOS)Rh(COD)]BF$_4$), (+)-1,2-bis((2*R*,5*R*)-2,5-diethylphospholano)-benzene(cyclooctadiene)rhodium triflate ([((*R*,*R*)-Et-DuPHOS)Rh(COD)]OTf), (*R*)-(+)-6,6′-bis(diphenylphosphino)-2,2′,3,3′-tetrahydro-5,5′-bi-1,4-benzodioxine ((*R*)-SYNPHOS) (Strem); $NH_2OH \cdot HCl$ (99%, Alfa Aesar); Ac_2O, AcOH, Fe (powder, 325 mesh), 5-oxo-5-phenylpentanoic acid, Me_3SiCl (Acros).

Methanol was dried by reflux over magnesium with subsequent distillation in the flow of argon. Toluene was dried by reflux over sodium in the presence of benzophenone in the flow of Ar with subsequent distillation.

Melting points were determined on a Kofler stage. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AM-300 and Bruker DRX-500 spectrometers (300.13, 500.13 (^{1}H), 75.47 MHz (^{13}C)) in $CDCl_3$. Chemical shifts are given relatively to Me_4Si. IR spectra were recorded on a Specord M82 spectrometer in KBr pellets. Mass spectra were recorded on a Kratos MS-30 instrument (70 eV). Elemental analysis was performed on a Perkin—Elmer Series II 2400 apparatus. The signs of angles of optical rotation for hydrogenation products **5** and **10** (see Table 1) were determined on a Spectropolyarimetr PU-09 instrument.

Methyl 5-oxo-5-phenylpentanoate (2). The compound was obtained according to a standard procedure[24] by esterification of acid **1** with methanol. B.p. 146 °C (5 Torr) (Ref. 24: b.p. 137 °C (2 Torr)). ^{1}H NMR ($CDCl_3$), δ: 2.07 (m, 2 H, C(O)CH$_2$C$\underline{H}_2$); 2.43 (t, 2 H, C(O)CH$_2$CH$_2$C$\underline{H}_2$, J = 7.2 Hz); 3.03 (t, 2 H, C(O)C$\underline{H}_2$, J = 7.2 Hz); 3.67 (s, 3 H, Me); 7.49 (m, 3 H, Ph); 7.95 (d, 2 H, Ph, J = 7.5 Hz).

Methyl 5-hydroxyimino-5-phenylpentanoate (3)[25]. Hydroxylamine hydrate hydrochloride (6.67 g, 96 mmol) and AcONa (7.87 g, 96 mmol) were placed into a round-bottom flask equipped with a reflux condenser and a magnetic stirring bar, followed by addition of MeOH (40 mL) and H_2O (20 mL). After stirring for 0.5 h, keto ester **2** (9.9 g, 48 mmol) was added, the mixture was refluxed for 5 h and the solvents were evaporated. Ethyl acetate was added to the residue and the mixture was washed with saturated aqueous $NaHCO_3$ and brine. The oxime obtained was crystallized from water. The yield was 8.59 g (81%), m.p. 49—50 °C. Found (%): C, 65.36; H, 6.93; N, 5.95. $C_{12}H_{15}NO_3$. Calculated (%): C, 65.14; H, 6.83; N, 6.33. ^{1}H NMR ($CDCl_3$), δ: 1.70 (m, 2 H, C(O)CH$_2$C$\underline{H}_2$); 2.32 (t, 2 H, C(O)CH$_2$CH$_2$C$\underline{H}_2$, J = 7.3 Hz); 2.73 (t, 2 H, C(O)C$\underline{H}_2$, J = 7.5 Hz); 3.59 (s, 3 H, Me); 7.40 (m, 3 H, Ph); 7.66 (m, 2 H, Ph); 11.21 (br.s, 1 H, OH). ^{13}C NMR ($CDCl_3$), δ: 21.54 (C(O)CH$_2$C$\underline{H}_2$); 25.25 (C(O)C$\underline{H}_2$); 33.58 (C(O)CH$_2$CH$_2$C$\underline{H}_2$); 51.47 (Me); 126.21 (*m*-C, Ph); 128.25 and 128.59 (*o*-C, Ph); 129.26 (*p*-C, Ph); 135.36 (C(CNOH)); 158.71 (CNOH); 173.64 (C=O). MS, m/z: 221 [M]$^+$.

Methyl 5-(*N*-acetylamino)-5-phenylpent-4(*E*)-enoate (4). Oxime **3** (5.54 g, 25 mmol) was dissolved under Ar in anhydrous toluene (50 mL) in a two-neck flask equipped with a stirrer and a reflux condenser. Then, a mixture of Ac_2O (7.2 mL, 75 mmol) and AcOH (4.4 mL, 75 mmol) was added dropwise to the solution, followed by addition of finely powdered iron metal (2.8 g, 50 mmol) and several drops of Me_3SiCl. The reaction mixture was stirred for 30 h. The color of the reaction solution turned from colorless to light green, then to reddish brown. After the reaction was complete (TLC monitoring), the reaction mixture was filtered through celite, concentrated on a rotary evaporator, and the residue was passed through a column with neutral alumina (using toluene as an eluent). A precipitate formed after partial evaporation of toluene was filtered off and washed with toluene to obtain a white crystalline product (2.1 g, 34%), m.p. 98—100 °C. Found (%): C, 68.06; H, 7.11; N, 5.66. $C_{14}H_{17}NO_3$. Calculated (%): C, 68.00; H, 6.93; N, 5.66. ^{1}H NMR ($CDCl_3$), δ: 2.02 (s, 3 H, Me); 2.37 (m, 4 H, 2 C$\underline{H}_2$); 3.63 (s, 3 H, Me); 6.35 (m, 1 H, CH); 6.71 (br.s, 1 H, NH); 7.35 (m, 5 H, Ph). ^{13}C NMR ($CDCl_3$), δ: 23.90 (Me$\underline{C}$(O)); 24.56 (C(O)CH$_2$C$\underline{H}_2$); 34.51 (C(O)C$\underline{H}_2$); 51.56 (Me); 116.84 (CH=); 128.47 (*p*-C, Ph); 128.70 (*m*-C, Ph); 128.72 (*o*-C, Ph); 135.10 ($\underline{C}$—CNH); 136.90 ($\underline{C}$=CH); 168.73 (NHC(O)); 173.32 (COO). IR, ν/cm^{-1}: 3264 (NH), 1736 (COOMe), 1660 (C=O), 1544 (C=C). MS, m/z: 247 [M]$^+$.

6-Phenyl-3,4-dihydropyran-2-one (6)[26]. A solution of 5-oxo-5-phenylpentanoic acid (**1**) (6.07 g, 31.6 mmol) in Ac_2O (37 mL, 391 mmol) was refluxed for 5 h under Ar, then excess of Ac_2O was evaporated and the residue was heated *in vacuo* for 2 h (the bath temperature was 160 °C). The compound obtained in 90% purity was used in subsequent step without additional purification. ^{1}H NMR ($CDCl_3$), δ: 2.51 (m, 2 H, C$\underline{H}_2$CH); 2.68 (m, 2 H, C$\underline{H}_2$C(O)); 5.81 (m, 1 H, CH=); 7.36 (m, 3 H, *m*- and *p*-H, Ph); 7.61 (m, 2 H, *o*-H, Ph).

***N*,*N*-Dimethyl-5-oxo-5-phenylpentanamide (7)[27].** A 33% aq. dimethylamine (30 mL) was added to lactone **6** (5.3 g, 30.6 mmol) and the mixture was vigorously stirred for 1 h. Then dimethylamine and water were evaporated and an the oily residue was distilled *in vacuo* to yield the product (5.09 g, 76%) as an oil, b.p. 160 °C (5 Torr). ^{1}H NMR ($CDCl_3$), δ: 2.09 (m, 2 H, C(O)CH$_2$C$\underline{H}_2$); 2.45 (m, 2 H, C(O)CH$_2$CH$_2$C$\underline{H}_2$); 2.95 (s, 3 H, C(O)N(M$\underline{e}$)Me); 3.01 (s, 3 H, C(O)N(Me)M$\underline{e}$); 3.11 (m, 2 H, C(O)C$\underline{H}_2$); 7.49 (m, 3 H, Ph); 7.98 (m, 2 H, Ph).

***N*,*N*-Dimethyl-5-hydroxyimino-5-phenylpentanamide (8).** A solution of AcONa (3.19 g, 46 mmol) and $NH_2OH \cdot HCl$ (3.73 g, 46 mmol) in a mixture of MeOH (15 mL) and H_2O (5 mL) was mixed with a solution of keto amide **7** (4.92 g, 22.4 mmol) in MeOH (15 mL). Further, the reaction mixture was stirred under reflux for 6 h. A solid obtained was crystallized from water to isolate a pure oxime (3.46 g, 66%), m.p. 125—127 °C. ^{1}H NMR ($CDCl_3$), δ: 1.95 (m, 2 H, C(N)CH$_2$C$\underline{H}_2$); 2.39 (m, 2 H, C(N)CH$_2$CH$_2$C$\underline{H}_2$); 2.93 (s, 8 H, 2 Me, C(N)C$\underline{H}_2$); 7.39 (m, 3 H, Ph); 7.68 (m, 2 H, Ph); 8.78 (br.s, 1 H, OH). IR, ν/cm^{-1}: 3332 (NOH), 1628 (CONMe$_2$). MS, m/z: 234 [M]$^+$.

***N*,*N*-Dimethyl-5-acetylamino-5-phenylpent-4-enamide (9).** A mixture of Ac_2O (4 mL, 42 mmol) and AcOH (2.4 mL,

42 mmol) was added dropwise to a solution of oxime **7** (3.3 g, 14 mmol) in anhydrous toluene (40 mL), followed by addition of a powdered iron metal (1.55 g, 28 mmol) and several drops of Me_3SiCl. The reaction mixture was stirred for 30 h under Ar, during which the solution gradually changed its color first to light green, then to reddish brown. The reaction mixture was filtered through celite, concentrated, and the residue was passed through a column with neutral Al_2O_3 (using toluene as an eluent). After evaporation of toluene, the product was isolated (1.8 g, 69%, a mixture of *Z*- and *E*-isomers, 1 : 2) as a light yellow oil. ^{1}H NMR ($CDCl_3$), δ: 2.02 and 2.19 (both s, 2 H and 1 H, MeC(O)); 2.50 (m, 4 H, CH_2); 2.87, 2.91, 2.93 and 2.96 (all s, 6 H, Me_2N); 5.50 (m, 0.33 H, CH=); 6.36 (m, 0.67 H, CH=); 6.71 (br.s, 0.67 H, NH); 7.37 (m, 5 H, Ph); 9.34 (br.s, 0.33 H, NH). ^{13}C NMR ($CDCl_3$), δ: 22.90 and 24.06 ($\underline{C}H_2CH$); 23.49 and 24.47 ($\underline{Me}C(O)$); 32.50 and 33.77 ($\underline{C}H_2C(O)$); 35.37 and 37.19 (Me_2N); 118.15 and 121.47 ($\underline{C}H$); 127.63 (*p*-C, Ph); 128.39 (*m*-C, Ph); 128.76 (*o*-C, Ph); 133.07 and 134.45 ($\underline{C}$—CNH); 136.95 and 137.97 ($\underline{C}CH$); 168.53 and 169.29 ($\underline{C}(O)Me$); 172.35 and 172.92 ($\underline{C}(O)NMe_2$).

Asymmetric catalytic hydrogenation of ω-enamides 4 and 9 (general procedure). ***A*** *(with the in situ formation of the catalyst).* A chiral ligand (0.006 mmol), $[Rh(COD)_2]OTf$ (0.006 mmol), and enamide (0.6 mmol) were placed into a glass tube for hydrogenation, which was evacuated and filled with argon three times, followed by addition of anhydrous methanol or other solvent (3 mL), preliminary deairated by three-fold freezing with liquid nitrogen, evacuation, defrost, and filling the vessel with argon. Then, the tube was placed into a stainless steel autoclave preliminary filled with argon, the autoclave was blown through with purified hydrogen and the pressure of hydrogen was adjusted to 20 atm. The reaction mixture was stirred with magnetic stirrer for a required time. After the experiment was over, the solvent was evaporated, the reaction mixture was dissolved in ethyl acetate and passed through a layer of silica gel. The solvent was evaporated on a rotary evaporator and the residue was analyzed by ^{1}H NMR in the presence of a chiral shift-reagent **11**.

B *(with the use of commercial rhodium catalyst).* Enamide (0.3 mmol) and a catalyst (**Rh-1**, **Rh-2**, or **Rh-3**) (0.003 mmol) were mixed in a glass tube for hydrogenation, with carrying out further manipulations as in procedure *A* when a catalyst was formed *in situ*.

Results of experiments are given in Table 1.

Methyl (±)-5-acetylamino-5-phenylpentanoate ((±)-5). This compound was synthesized to monitor the accuracy of determination of enantiomeric composition of nonracemic compound **5** by ^{1}H NMR. Racemic product was obtained by hydrogenation with Pd (1%)-on-sibunite at room temperature for 20 h at 20 atm. of H_2. ^{1}H NMR ($CDCl_3$), δ: 1.65 (m, 2 H, $C(O)CH_2C\underline{H}_2$); 1.84 (m, 2 H, $C(O)C\underline{H}_2$); 2.01 (s, 3 H, Me); 2.35 (m, 2 H, $C(O)CH_2CH_2C\underline{H}_2$); 3.66 (s, 3 H, Me); 4.99 (m, 1 H, NCH); 5.75 (br.s, 1 H, NH); 7.31 (m, 5 H, Ph).

(±)-*N*,*N*-Dimethyl-5-acetylamino-5-phenylpentanamide ((±)-10). This compound was synthesized to monitor the accuracy of determination of enantiomeric composition of nonracemic compound **10** by ^{1}H NMR. ^{1}H NMR ($CDCl_3$), δ: 1.66 (m, 2 H, $C(O)CH_2C\underline{H}_2$); 1.78 (m, 2 H, $C(O)C\underline{H}_2$); 1.96 (s, 3 H, Me); 2.31 (m, 2 H, $C(O)CH_2CH_2C\underline{H}_2$); 2.92 (s, 6 H, 2 Me); 4.93 (m, 1 H, NCH); 6.73 (br.s, 1 H, NH); 7.26 (m, 5 H, Ph).

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