Asymmetric Photochemical Synthesis of Chiral 5-(*R*)-(*l*)-Menthyloxy-4-Cycloaminobutyrolactones

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Through photocatalysed regiospecific and stereoselective additions of cycloamines to 5-(*R*)-(*l*)-menthyloxy-2(5*H*)-furanone (3), chiral 5-(*R*)-(*l*)-menthyloxy-4-cycloaminobutyrolactones were synthesized. In the new asymmetric photoaddition of compound 3, the *N*-methyl cyclic amines (4) gave novel chiral C—C photoadducts (5) in 24—50% isolated yields with *d.e.* ≥98%. However, the secondary cyclic amines (6) afforded optically active N—C photoadducts (7) in 34—58% isolated yields with *d.e.* ≥98% under the same condition. All the synthesized optically active compounds were identified on the basis of their analytical data and spectroscopic data, such as $[\alpha]_{589}^{20}$, IR, ^{1}H NMR, ^{13}C NMR, MS and elementary analysis. The photosynthesis of chiral butyrolactones and its mechanism were discussed in detail.

Keywords 5-(*R*)-(*l*)-Menthyloxy-4-cycloaminobutyrolactones, asymmetric photocatalysed conjugate addition, chiral C—C photoadduct and N—C photoadduct

Introduction

It has been known that the synthetic organic photochemistry constitutes a research area with exceptional importance for the development of efficient and selective transformations for the preparation of natural products as well as unnatural and complicated molecules.[1] The synthesis of γ-substituted-butyrolactones has attracted much attention owing to its presence in a variety of biologically active natural products[2] and utility as the valuable synthetic intermediates.[3] Recently, asymmetric photoinduced conjugate additions to 5-substituted-2(5*H*)-furanones have been used for the construction of some important natural products.[4] On the basis of our previous work,[5] we further studied the photocatalysed conjugate additions of cyclic amines to chiral 5-(*R*)-(*l*)-menthyloxy-2(5*H*)-furanone (3). The novel chiral C—C photoadducts (5) were obtained from the *N*-methyl cyclic amines (4). However, the secondary cyclic amines (6) gave optically active N—C photoadducts (7) under the same condition (Scheme 1). These results provide a valuable synthetic route to potentially interesting enantiomerically pure compounds. The aim of the present study would be proposed groundwork for future applications on the photoreaction to synthesis of more complex molecules.

Experimental

Instruments and materials

Infrared spectra were recorded on a Fourier 170-sx and Hitachi 260-50 spectrophotometer. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Varian-300 MHz spectrometer, and the chemical shifts were expressed in δ-values using TMS as the internal standard. Mass spectra were determined with Finnigan MHT4500 mass spectrometer. Microanalyses were performed in a Perkin-Elemer 240-C Elemental Analyser. Na-D line polarimetry was carried out at 25℃ in a Perkin-Elemer 241-C polarimeter. Melting points were determined on a Liuben microthermopan and are uncorrected.

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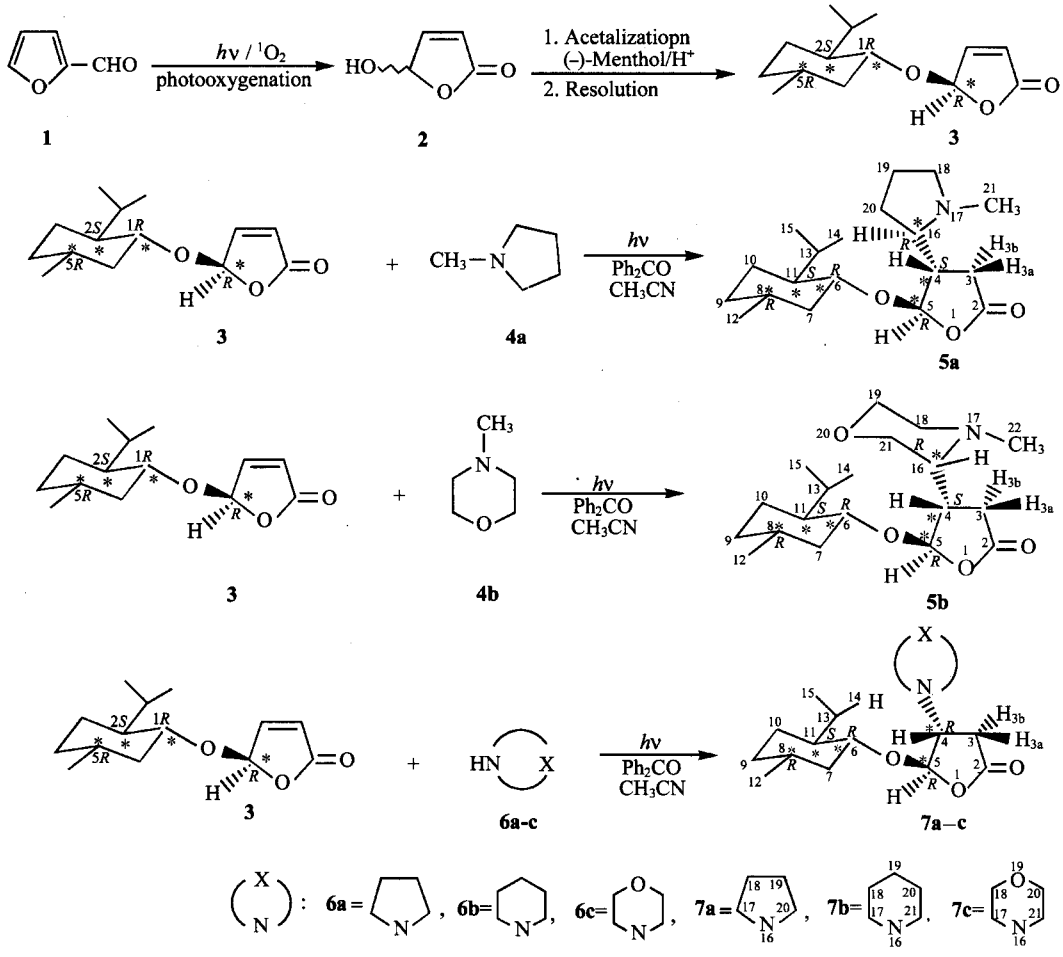
Scheme 1



All chemical reagents were commercially available and treated with standard method before use. Solvents were dried in a routine way and redistilled.

Preparation of chiral 5-(*R*)-(*l*)-*menthyloxy*-2(5*H*)-*furanone* (**3**)

The preparation of **3** has been reported as shown in Scheme 1.[5c-5e]

Synthesis of 5-(*R*)-(*l*)-*menthyloxy*-4-(*S*)-*N*-*methyl*-2′-*pyrrolidinobutyrolactone* (**5a**)

The chiral synthon **3** (3.81 g, 16 mmol), benzophenone (2.92 g, 16 mmol) and *N*-methyl pyrrolidine **4a** (4 mL, 38.5 mmol) were dissolved in acetonitrile (70 mL). The solution was placed in a Pyrex vessel and degassed by passage of a steady stream of nitrogen, and then was irradiated with a medium pressure mercury lamp (350 nm, 450 W) for 4.5 h. The solvent was evaporated under reduced pressure and the residue was purified by column chromatography with gradient elution [eluent: L.P (light petroleum, 30—60℃)-EtOAc (9:1, *V*/*V*)] and recrystallization with a mixed solvent of light petroleum ether and ethyl acetate to give the title compound **5a** as a colorless sheet crystal (1.22 g, isolated yield 24%), recovered benzophenone (2.1 g, 71%) and benzopinacol [0.2 g, m.p. 190—192℃, (Lit.[6] m.p.184—186℃); ^{1}H NMR (60 MHz, $CDCl_3$) δ: 3.10 (s, 2H, OH, the proton signal lost after D_2O exchanged), 7.03—7.67 (m, 20H, ArH)].

5a m.p. 110—112℃. $[\alpha]_{589}^{20}$ = − 104.5 ($CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$)δ: 0.78 (d, *J* = 6.6 Hz, CH_3), 0.87 (d, *J* = 6.6 Hz, CH_3), 0.93 (d, *J* = 6.3 Hz, CH_3), 0.95—1.08 (m, 3H, 2H-10, H-9), 1.13—1.27 (m, 1H, H-9), 1.20—1.53 (m,

1H, H-7), 1.60—1.77 (m, 2H, H-7, H-8), 1.80—1.90 (m, 4H, 2H-19, 2H-20), 2.03—2.15 (m, 2H, H-11, H-13), 2.30 (s, 3H, N-CH_3), 2.18—2.38 (m, 2H, 2H-18), 2.40—2.60 (m, 2H, H-16, H-3b), 2.82 (dd, J = 9.9, 9.0 Hz, 1H, H-4), 3.03—3.13 (m, 1H, H-3a), 3.50 (ddd, J = 10.8, 7.2, 3.6 Hz, 1H, H-6), 5.60 (1H, s, H-5). ^{13}C NMR (75 MHz, $CDCl_3$)δ: 15.6, 20.9, 22.2, 23.0, 25.4, 27.0, 31.2, 31.3, 34.3, 39.9, 40.8, 42.7, 47.7, 57.1, 66.5, 77.1, 102.3, 176.4. IR (KBr)ν: 2960, 2925, 2800, 1782, 1170, 962 cm^{-1}. MS (70 eV) m/z (%): 323(M^+, 2), 184($M^+ - C_{10}H_{19}$, 100), 84 ($C_5H_{10}N^+$, 100); Anal. calcd. for $C_{19}H_{33}NO_3$: C 70.55, H 10.28, N 4.33; found: C 70.70, H 10.43, N 4.09.

Synthesis of 5-(*R*)-(*l*)-*menthyloxy*-4-(*S*)-*N*-*methyl*-2′-*morpholinobutyrolactone* (**5b**)

As the procedure of **5a** above, the chiral synthon **3** (1.19 g, 5 mmol), benzophenone (0.91 g, 5 mmol) and *N*-methyl morpholine **5b** (2 mL, 18.2 mmol) were dissolved in acetonitrile (70 mL), and the solution was irradiated under a medium pressure mercury lamp (350 nm, 450 W) in a Pyrex water-cooled immersion well (250 mL) for 1 h. The solvent was evaporated in vacuum to give crude product which was purified by column chromatography gradient elution [eluent: L.P-EtOAc (9 :1, V/V)] and recrystallization with a mixture of light petroleum and diethyl ether to give the title compound **5b** as a colorless small sheet crystal (0.85 g, isolated yield 50%), recovered benzophenone (0.46 g, 51%) and benzopinacol [0.23 g, m.p. 190—192℃, (Lit.[6] m. p.184—186℃)].

5b m. p. 106—107℃ $[\alpha]_{589}^{20}$ = − 104.6 ($CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$)δ: 0.77 (d, J = 6.3 Hz, CH_3), 0.88 (d, J = 7.5 Hz, CH_3), 0.94 (d, J = 6.3 Hz, CH_3), 0.93—1.10 (m, 3H, 2H-10, H-9), 1.14—1.27 (m, 1H, H-9), 1.30—1.47 (m, 1H, H-7), 1.58—1.73 (m, 2H, H-7, H-8), 2.00—2.15 (m, 2H, H-11, H-13), 2.17—2.32 (m, 2H, 2H-18), 2.33—2.40 (m, H, H-16), 2.42—2.60 (m, 5H, N-CH_3, H-4, H-3b), 2.77 (dd, J = 9.3, 9.0 Hz, 1H, H-3a), 3.47 (ddd, J = 10.8, 10.8, 4.8 Hz, 1H, H-6), 3.63—3.73 (m, 4H, 2H-19, 2H-21), 5.57 (1H, s, H-5). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 15.7, 21.0, 22.4, 23.2, 25.6, 31.4, 31.7, 34.4, 38.7, 40.1, 47.8, 53.6, 59.8, 66.9, 77.1, 103.2, 176.2. IR (KBr)ν: 2950, 2920, 2850, 1791, 1128, 952 cm^{-1}. MS (70 eV) m/z (%): 339 (M^+, 12), 200 ($M^+ - C_{10}H_{19}$, 75), 100 ($C_5H_{10}NO^+$, 100). Anal. calcd. for $C_{19}H_{33}NO_4$: C 67.22, H 9.80, N 4.13; found: C 67.30, H 9.92, N 3.77.

Synthesis of 5-(*R*)-(*l*)-*menthyloxy*-4-(*R*)-1-*pyrrolidinobutyrolactone* (**7a**)

The chiral synthon **3** (1.19 g, 5 mmol), benzophenone (0.91 g, 5 mmol) and pyrrolidine **6a** (2 mL, 24 mmol) were dissolved in acetonitrile (70 mL) and the solution was placed in a Pyrex vessel and degassed by passage of a steady stream of nitrogen, then was irradiated under a medium pressure mercury lamp (350 nm, 450 W) in a Pyrex water-cooled immersion well (250 mL) for 1 h. The solvent was evaporated under reduced pressure and the residue was purified by column chromatography with gradient elution [eluent: L.P-EtOAc (9 : 1, V/V)] and recrystallization with acetonitrile to offer the title compound **7a** as a colorless small sheet crystal (0.52 g, isolated yield 34%), recovered benzophenone (0.65 g, 71%), benzopinacol [0.26 g, m.p. 190—192℃ (Lit.[6] m.p. 184—186℃). ^{1}H NMR (60 MHz, $CDCl_3$)δ: 3.10 (s, 2H, OH, the proton signal lost after D_2O exchanged), 7.03—7.67 (m, 20H, ArH)].

7a m.p. 134.5—135℃. $[\alpha]_{589}^{20}$ = − 154.3 ($CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$)δ: 0.78 (d, J = 6.9 Hz, CH_3), 0.88 (d, J = 7.2 Hz, CH_3), 0.94 (d, J = 6.6 Hz, CH_3), 0.97—1.10 (m, 3H, 2H-10, H-9), 1.17—1.28 (m, 1H, H-9), 1.29—1.50 (m, 1H, H-7), 1.58—1.72 (m, 2H, H-7, H-8), 1.74—1.86 (m, 4H, CH_2CH_2), 2.01—2.18 (m, 2H, H-11, H-13), 2.47—2.67 (m, 5H, H-3b, CH_2N-CH_2), 2.76 (dd, J = 7.5, 7.2 Hz, H-3a), 3.04 (dd, J = 7.5, 7.2 Hz, H-4), 3.53 (ddd, J = 10.8, 10.8, 4.2 Hz, 1H, H-6), 5.60 (s, 1H, H-5). ^{13}C NMR (50 MHz, $CDCl_3$)δ: 15.7, 20.9, 22.4, 23.0, 23.2, 25.4, 31.3, 33.5, 34.3, 39.6, 47.7, 51.5, 65.5, 77.0, 102.7, 174.5. IR (KBr)ν: 2960, 2921, 2871, 1776, 1125, 948 cm^{-1}. MS (70 eV) m/z(%): 309 (M^+,15), 112 ($C_7H_{14}N^+$, 13), 97 ($C_6H_{11}N^+$, 100), 41 ($C_3H_5^+$, 16). Anal. calcd. for $C_{18}H_{31}NO_3$: C 69.86, H 10.10, N 4.53; found: C 69.86, H 10.24, N 4.53.

Synthesis of 5-(*R*)-(*l*)-*menthyloxy*-4-(*R*)-1-*piperidinobutyrolactone* (**7b**)

As the procedure of **7a** above, the solution of chiral synthon **3** (1.19 g, 5 mmol), benzophenone (0.91 g, 5 mmol) and piperidine **6b** (2 mL, 23 mmol) was degassed by passage of a steady stream of nitrogen, and then was irradiated for 1 h. The solvent was evaporated under reduced pressure and the residue was purified by column chromatography with gradient elution [eluent: L. P-EtOAc (9 : 1, *V*/*V*)] and recrystallization with ethyl acetate to offer the title compound **7b** as a colorless small sheet crystal (0.94 g, isolated yield 58%), recovered benzophenone (0.49 g, 71%), benzopinacol [0.48 g, m.p. 190—192℃ (Lit.[6] m.p. 184—186℃)].

7b m. p. 114—116℃. $[\alpha]_{589}^{20}$ = − 133.8 (CCl_4). ^{1}H NMR (60 MHz, CCl_4)δ: 0.67—2.60 (m, 29H, menthyl's H, H-3b, piperidyl's H), 2.77—3.20 (m, 2H, H-4, H-3a), 3.27—3.70 (m, 1H, H-6), 5.40 (s, 1H, H-5). IR (KBr)ν: 2953, 2922, 2867, 1971, 1109, 947 cm^{-1}. MS (70 eV) *m*/*z*(%): 323 (M^+, 30), 324 (M^+ + 1, 20), 156 ($C_{10}H_{20}O^+$, 20), 111 ($C_7H_{13}N^+$, 100). Anal. calcd. for $C_{19}H_{33}NO_3$: C 70.55, H 10.28, N 4.33; found: C 70.53, H 10.91, N 4.28.

Synthesis of 5-(*R*)-(*l*)-*menthyloxy*-4-(*R*)-1-*morphorlinobutyrolactones* (**7c**)

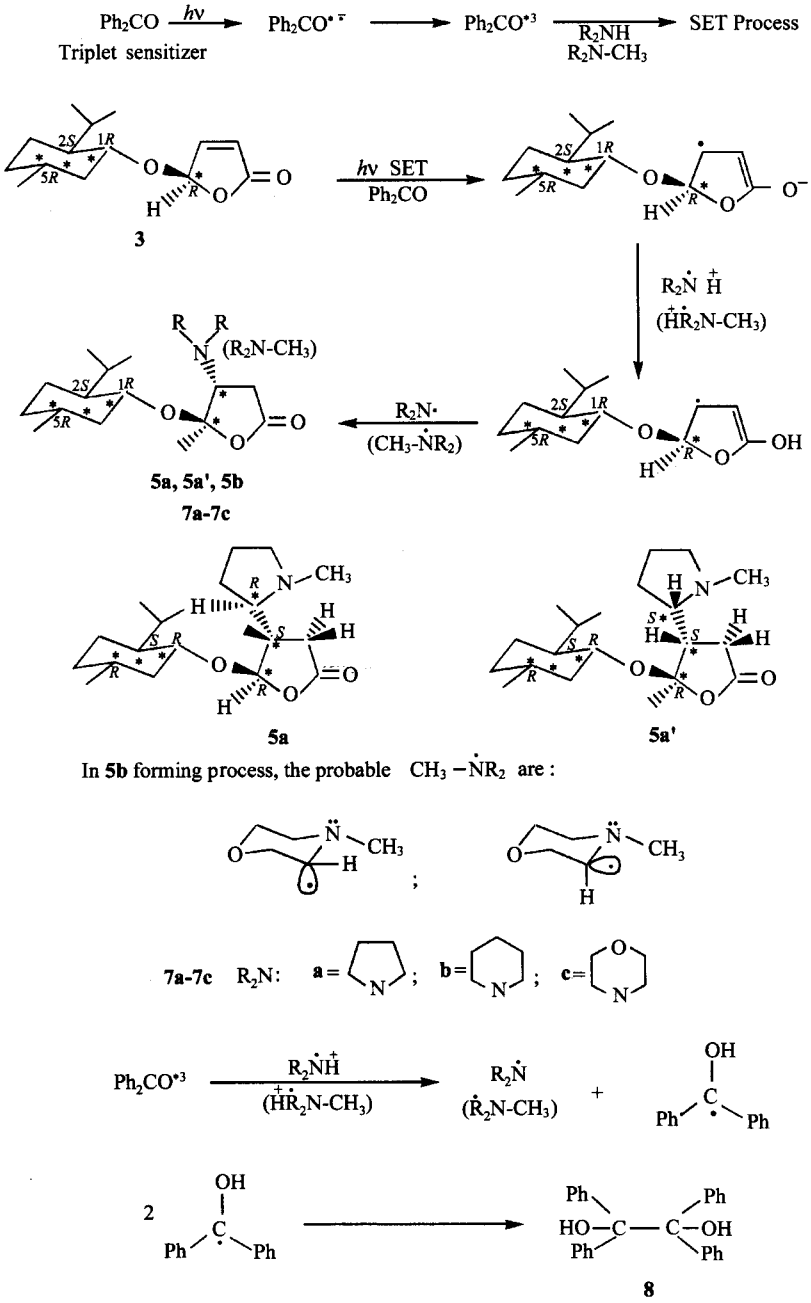
As the procedure of **7a** above, the solution of chiral synthon **3** (3.81 g, 16 mmol), benzophenone (2.92 g, 16 mmol) and morpholine **6c** was degassed by pasage of a steady stream of nitrogen, and then was irradiated for 1 h. The solvent was evaporated under reduced pressure and the residue was purified by column chromatography with gradient elution [eluent: L. P-EtOAc (9 : 1, *V*/*V*)] and recrystallization with a mixture of light petroleum ether and ethyl acetate to offer the title compound **7c** as colorless needle crystal (2.58 g, isolated yield 50%), recovered benzophenone (2.0 g, 70%), benzopinacol [0.28 g, m.p. 190—192℃ (Lit.[6] m.p. 184—186℃)].

7c m. p. 116—117℃. $[\alpha]_{589}^{20}$ = − 166.9 ($CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$)δ: 0.77(d, *J* = 7.2 Hz, CH_3), 0.88 (d, *J* = 6.6 Hz, CH_3), 0.94 (d, *J* = 6.6 Hz, CH_3), 0.97—1.10 (m, 3H, 2H-10, H-9), 1.15—1.20 (m, 1H, H-9), 1.32—1.47 (m, 1H, H-7), 1.60—1.77(m, 2H, H-7, H-8), 1.93—2.17(m, 2H, H-11, H-13), 2.42—2.63 (m, 5H, CH_2NCH_2, H-3b), 2.76 (dd, *J* = 8.1, 7.2 Hz, 1H, H-3a), 3.10—3.20 (m, 1H, H-4), 3.55 (ddd, *J* = 10.8, 10.5, 4.2 Hz, 1H, H-6), 3.63—3.80 (m, 4H, CH_2OCH_2), 5.63 (s, 1H, H-5). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 15.6, 20.8, 22.2, 23.0, 25.4, 31.3, 34.2, 39.5, 47.7, 50.2, 66.3, 77.1, 101.4, 174.3. IR (KBr)ν: 2960, 2923, 2857, 1781, 1122, 942 cm^{-1}. MS(70 eV) *m*/*z* (%): 325(M^+, 15), 113 ($C_6H_{11}NO^+$, 100). Anal. calcd. for $C_{18}H_{31}NO_4$: C 66.43, H 9.60, N 4.31; found: C 66.77, H 9.85, N 4.48.

Results and discussion

The reaction of benzophenone with amines[7] can be fundamentally different from general photochemical reaction and presents an alternative mechanism for hydrogen atom transfer. Photophysical studies demonstrated that: (1) the rate of quenching of triplet benzophenone by amines is several orders of magnitude larger than for other substrates and approaches the diffusion-controlled limit; (2) the ketone whose triplet states are long lived and quenched efficiently by amines; (3) the rate of triplet quenching can be correlated with ionization potential of the amines; and (4) the products of such quenching include ground-state reactants and radical ions. Extensive studies have led to acceptance of a mechanistic picture which spans the extremes of initial single electron transfer (SET) between amine and triplet ketone to conventional hydrogen atom transfer. It is likely that this reaction is initiated by a single-electron transfer (SET),[8] though it then proceeds *via* radical chain reaction as shown in Scheme 2. The mechanism of this photocatalysed reaction would appear to be analogous to that proposed by Fraser-Reid,[4e,9] and involves excitation of benzophenone to the triplet (n, π^*) state with subsequent abstraction of a hydrogen atom from the various cyclic amines and Michael addition of the resultant radical to the chiral synthon **3**. The photocatalysed conjugate additions of the tertiary amines to **3** offer chiral active C—C adducts **5**, and the result is similar to Mann's report,[4d] in which the photocatalysed addition of *N*-methyl pyrro-

Scheme 2



lidine to 5-*O*-TBDMS-butenolide under the same condition obtained the photoadduct, 3-(*N*-methyl-ptrrolidin-2′-yl)-4-(*tert*-butyldimethylsiloxy-methyl)-butan-4-olide in isolated yield of 40%. Thus, the photoadduct **5a** is produced stereoselectively in considerable preference as major product in which the two heterocyclic groups occur in *trans* form, as shown in Scheme 2. However, the photoproduct **5a′** in which the two heterocyclic groups occur in *cis* form may be also formed theoretically during the photoinduced reaction, but it is so trivial that we could not find it after purification. In **5b** forming process, the probable N—C radicals are axial and equatorial conformers respectively, as shown in Scheme 2. The equatorial radical conformer has steric interference

and could not undergo radical coupling reaction. The axial radical conformer considers the anomeric effect as a stabilizing electronic effect, which occurs when an electron pair of a nitrogen atom is oriented antiperiplanar[10] to a radical bond and finally leads to form the optically pure compound **5b**. Similarly, the benzophenone-photoinduced reactions occurring between conjugated **3** and the second cycloamines **6a—6c** are believed to proceed *via* the mechanistic pathway outlined in Scheme 2. In this process, the triplet excited state of benzophenone, formed by excitation and efficient intersystem crossing, abstracts a hydrogen atom from the second cycloamines to form the cycloamine radical and ketyl radicals, and then the N radical of the secondary cyclic amines **6** undergoes the 1,4-addition to the α,β-unsaturated butenolide **3** to offer N—C photoadducts **7a—7c**.

The enantiomerically pure **3** was easily obtained though single oxygen photooxygenation of furfural followed by acetalization of the resulting 4-hydroxybutenolide with the chiral auxiliaries (−)-(*l*)-menthol and subsequent crystallization of the epimeric mixture from light petroleum. Compound **3** was identified on the basis of its satisfactory elemental data and spectroscopic data (IR, UV, ^{1}H NMR, ^{13}C NMR, Mass). The absolute configuration at the acetal carbon of **3** was proved to be *R* by mean of an X-ray structure analysis.[5c-5e]

These experimental results show clearly that the chiral synthon **3**, undergoes efficient benzophenone-initiated photoaddition of various cycloamines to give the regiospecific and stereospecific adducts **5a**, **5b** and **7a-7c**. Irradiation of chiral synthon **3** with *N*-methyl cycloamines **4a**, **4b** in the presence of benzophenone (mol. equivalent) obtained the novel chiral C—C photoadducts **5a**, **5b** respectively in isolated yields of 24% and 50% with *d. e.* ≥98%. A small amount of benzopinacol **8** was obtained, but about 70% of benzophenone was recovered. However, we discovered that with the second cycloamines **6a—6c**, it was possible to convert the chiral synthon **3** into optically purity N—C photoadducts **7a—7c** in isolated yields of 34—58% with *d. e.* ≥98%. Around 70% of benzophenone could be recovered unchanged, but variable amounts of benzopinacol were also obtained, together with by-products, like menthol as a photolysised product from chiral synthon **3**.

Most remarkable is the high stereoselectivity of the reaction process. This is easily explained when a bulky protecting group of *l*-menthyloxy is present as in substrate **3**. On basis of literature precedent[5b] most probably the radical group of cycloamine attacks stereoselectively the 4-position of **3** from the side opposite to the menthyloxy group, namely the less hindered, to offer the novel optically pure C—C and N—C photoadducts **5a**, **5b** and **7a—7c** which were identified on the basis of their spectroscopic data.

As a extension of the above work, other *N*-methyl cyclic amines such as *N*-methyl piperidine, nicotine and *N*-trimethylsilyl cyclic amines,[4d] and some kinds of containing oxygen atom compounds such as alcohols, aldehydes, acetals[11] and ethers[12] can be also used in the asymmetric photocatalysed conjugate additions to **3** for synthesizing more new optically active compounds. The application of this synthetic route to some interesting optically pure compounds in asymmetric synthesis is currently in progress.

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