Chiral Bicyclic Lactams for Asymmetric Synthesis of Quaternary Carbons. The Total Synthesis of (–)-α-Cuparenone

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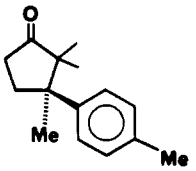
An asymmetric synthesis of the title compound has been achieved in >98% ee using chiral bicyclic lactams **14** derived from (*S*)-valinol and γ-keto acids. Thus, cyclocondensation of racemic 2-*p*-tolyl-4-oxopentanoic acid with (*S*)-valinol gave a 1:1 mixture of endo and exo *p*-tolyl-substituted lactams **14**, which were transformed, via their common lithio enolate, to the quaternary substituted lactams **15**–**16** as a 93:7 ratio of diastereomers. Crystallization gave pure **15**, which was carried forward to the enantiomerically pure sesquiterpene (–)-α-cuparenone.

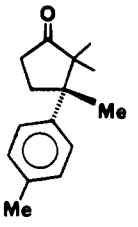
Asymmetric syntheses of quaternary carbon centers have only recently been addressed as an important structural component found in many naturally occurring compounds.[1] Our studies on the readily available chiral bicyclic lactams **1**, **6**, and **9** have provided a useful entry into a variety of compounds containing a stereochemically defined quaternary carbon center. Thus, **1**, when sequentially alkylated to give the α,α-disubstituted lactam **2**, may be hydrolyzed to the chiral γ-keto esters **3** in >99% ee,[1c] or alternatively, reduction to the keto aldehydes **4** leads to 4,4-disubstituted cyclopentenones **5** in >99% ee[1d] (Scheme I).

Further synthetic studies on chiral bicyclic lactams have led to an asymmetric total synthesis of (+)-mesembrine (**8**) utilizing the readily available lactam **6** (Scheme II). The latter, after reduction, hydrolysis, and aldolization, furnished chiral 4,4-disubstituted cyclohexenone **7**, a ready precursor to mesembrine.[1e] The unsaturated bicyclic lactam **9**, underwent (2 + 2) photocyclization with ethylene, affording the cyclobutane-fused system **10** in 93% diastereoselectivity, and then was carried forward to (–)-grandisol (**11**).[1f]

Taber[2] recently described an enantioselective approach to (+)-cuparenone (**12**), a naturally occurring sesquiterpene found as the (+)-enantiomer in the essential oil of the *Mayur Pankhi* tree.[3] The (–)-enantiomer, also naturally

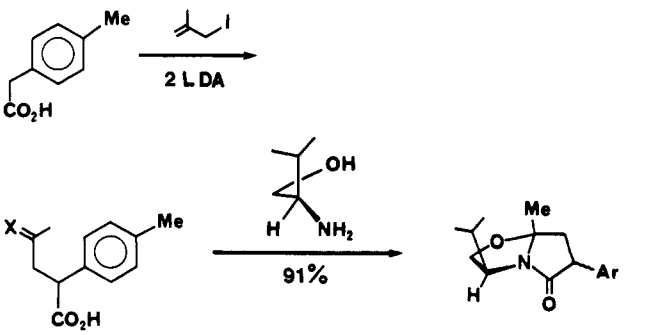


(–)-**12**



(+)-**12**

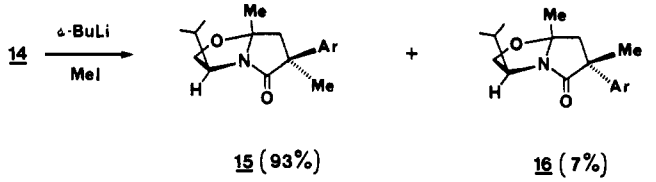
occurring, has been isolated from the liverwort *Mannia fragrans*.[4] The (+)-enantiomer of **12** has also been prepared in 71% ee by Posner.[5] We now describe an asymmetric synthesis of (–)-cuparenone, in greater than 97% ee, utilizing these chiral bicyclic lactams as starting materials. The requisite bicyclic lactam **14** was prepared by alkylation of *p*-tolylacetic acid with methallyl iodide with 2.0 equiv lithium diisopropyl amide. The resulting α-methallyl derivative **13** (X = CH_2) was transformed into the keto acid **13** (X = O) via ozonolysis, both steps proceeding in over 90% yield. Treatment of the keto acid



(±)-**13**, X = CH_2, 92%
(±)-**13**, X = O, 93%

14, Ar = p-Tolyl

13 with (*S*)-valinol in benzene at reflux produced the bicyclic lactam **14** in 91% yield as a 1:1 mixture of endo–exo aryl-substituted diastereomers. The mixture was of no consequence since further metalation of the α-position would generate a single lithium enolate. This has been termed a "deracemizing alkylation" since racemic keto acid **13** (X = O) will be *completely* utilized in the synthesis.[1g] In the event, the 1:1 mixture of **14** was treated with *sec*-butyllithium at –100 °C and 3 equiv of methyl iodide. The products **15** and **16** were obtained in 90% yield as a 93:7 mixture in favor of **15**. Recrystallization afforded pure



15 (93%) **16** (7%)

15 (60–70%) as a crystalline material confirmed by HPLC (>99.0%). The stereochemical assignment of **15** and **16** was easily performed by observing the chemical shift of the angular methyl group in each diastereomer. At 270 MHz, the major isomer showed the methyl singlet at 1.15 ppm, whereas the minor isomer exhibited the methyl singlet further downfield at 1.54 ppm. Obviously the methyl group is strongly shielded by the tolyl group in **15**. Other alkylations in related bicyclic lactams[1d-f] have always occurred predominantly from the endo-face (α-alkylation),

(1) (a) Hashimoto, S.-i.; Koga, K. *Tetrahedron Lett.* **1978**, 573. (b) Kogan, H.; Tomioka, K.; Hashimoto, S.-i.; Koga, K. *Tetrahedron* **1981**, *37*, 3951. (c) Meyers, A. I.; Harre, M.; Garland, R. *J. Am. Chem. Soc.* **1984**, *106*, 1146. (d) Meyers, A. I.; Wanner, K. T. *Tetrahedron Lett.* **1985**, *26*, 2047. (e) Meyers, A. I.; Hanreich, R.; Wanner, K. T. *J. Am. Chem. Soc.* **1985**, *107*, 7776. (f) Meyers, A. I.; Fleming, S. A. *J. Am. Chem. Soc.* **1985**, *107*, 306. (g) Pfau, M.; Revial, G.; Guingant, A.; d'Angelo, J. *J. Am. Chem. Soc.* **1985**, *107*, 273. (h) Schultz, A. G.; Sundararaman, P.; Macielag, M.; Lavieri, F. P.; Welch, M. *Tetrahedron Lett.* **1985**, *26*, 4575.

(2) Taber, D. F.; Petty, E. H.; Raman, K. *J. Am. Chem. Soc.* **1985**, *107*, 196.

(3) Chetty, G. L.; Dev, S. *Tetrahedron Lett.* **1964**, 3.

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(5) Posner, G. H.; Kogan, T. P.; Hulce, M. *Tetrahedron Lett.* **1984**, *25*, 383.

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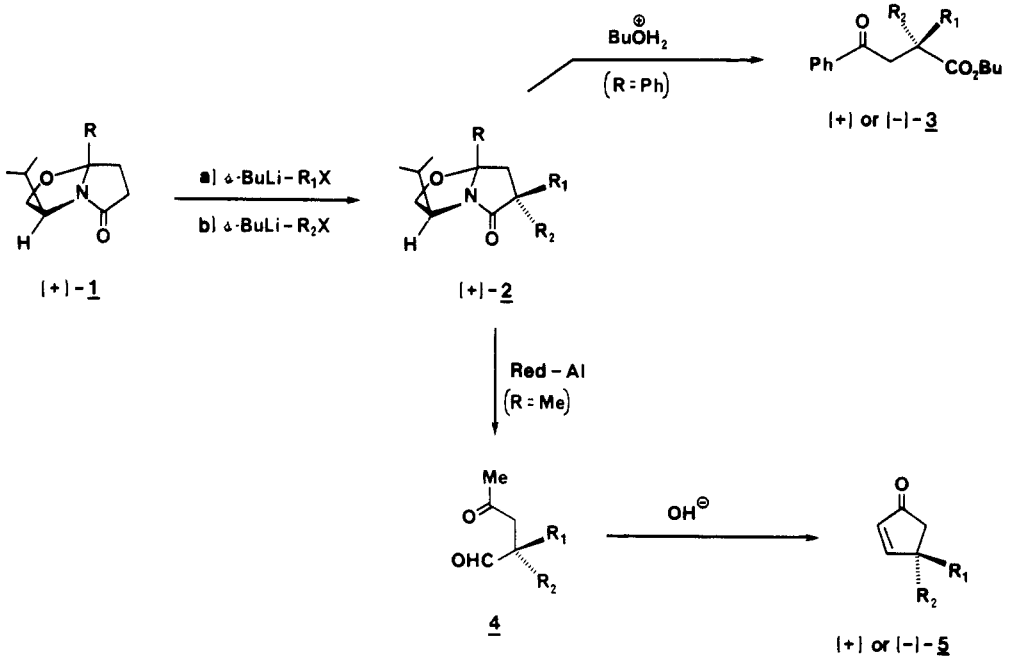
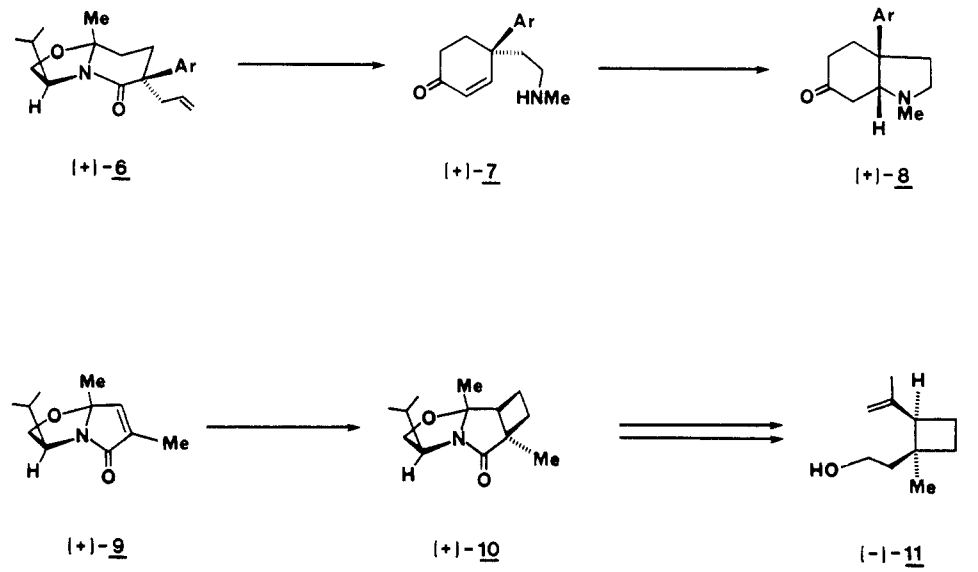
Scheme I



Scheme II



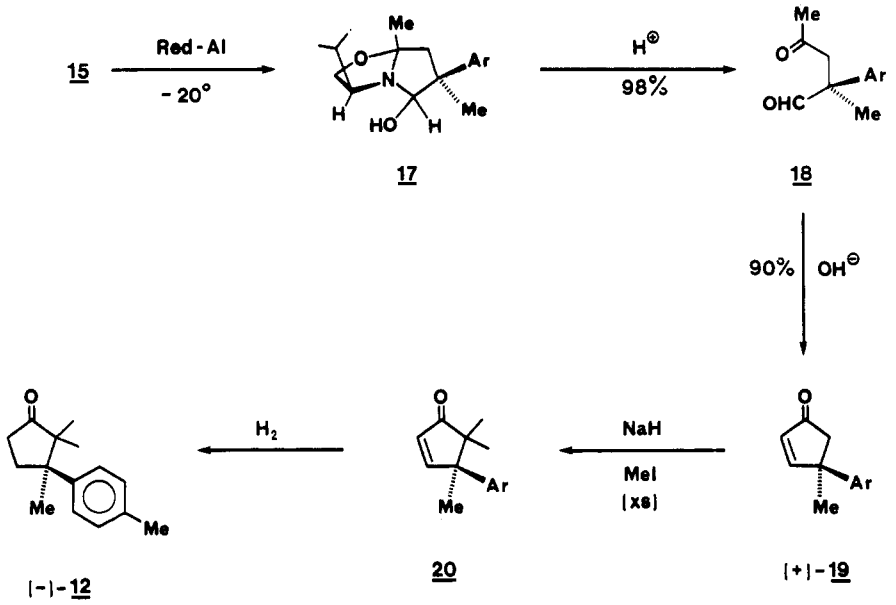
consistent with the quaternary carbon in **15**.

Reduction of the single diastereomeric lactam **15** was performed with Red-Al at −20 °C furnishing the carbinolamine **17**, which was hydrolyzed with tetrabutylammonium dihydrogen phosphate to the keto aldehyde **18** (Scheme III). The reduction to the carbinolamine proceeded in quantitative yield only when pure crystalline lactam **15** was employed. When purified material of an oily nature was treated with Red-Al, poor yields of **17**, accompanied by extensive overreduced products, were obtained. After hydrolysis to the keto aldehyde (98%), the aldol cyclization to the cyclopentenone **19** was accomplished in 90% with 1 M potassium hydroxide in ethanol. Attempts to introduce the *gem*-dimethyl groups to **20** proved to be more difficult than expected. Generation of the enolate of **19** with LDA–methyl iodide, with or without HMPA as a cosolvent, led to mixtures of starting material and mono- and dialkylated cyclopentenones. Wenkert,[6] in his synthesis of racemic α-cuparenone alkylated (±)-**19** using sodium hydride–methyl iodide and reported (±)-**20** in 74% yield. Repeating this process gave (−)-**20** in 48% purified yield. The final step in the synthesis required reduction of the unsaturation, and this proceeded smoothly (87%) to give (−)-α-cuparenone (**12**). The specific rotation observed was −166° in chloroform ($[\alpha]^{20}_{D}$ +170° ($CHCl_3$))[4,7] which indicated optical purity of at least 97.6%. Since the HPLC analysis of the intermediate **15** indicated at least 98% enantiomeric purity and racemization in any of the subsequent steps to α-cuparenone was unlikely, it may be assumed that the optical purity of the final product is at least 98%. Since the levorotary enantiomer of cuparenone was obtained in this study and the absolute configuation has been earlier established by Ito[7] to be *R*-(+) for the natural sesquiterpene, we may conclude that the endo-alkylation of **14** to **15** is in accord with our earlier obser-

(6) Wenkert, E.; Buckwater, B. L.; Craviero, A. A.; Sanchez, E. L.; Sathe, S. S. *J. Am. Chem. Soc.* **1978**, *100*, 1267.

(7) The absolute configuration of (+)-α-cuparenone has been established as *R*: Irie, T.; Suzuki, T.; Ito, S.; Kurosawa, E. *Tetrahedron Lett.* **1967**, 3187.

Scheme III



vations on chiral bicyclic lactam alkylations. The reasons for predominant endo-alkylation on the lithium enolates of these lactams is still not clear and further studies to clarify this behavior are still in progress.

Experimental Section

2-*p*-Tolyl-4-methyl-4-pentenoic Acid (13, X = CH_2). To a stirred solution of 6.3 mL (45 mmol) of diisopropylamine in 100 mL of dry THF at 0 °C was added 4.20 mL (44.1 mmol) of 10.5 M *n*-butyllithium. The solution was stirred for 15 min and then cooled to −78 °C. *p*-Tolylacetic acid (3 g, 20 mmol) in 10 mL of dry THF was added to the LDA solution over 10 min. The solution was warmed to 0 °C and then cooled to −78 °C, at which time 3-iodo-2-methylpropene (3 mL, 30 mmol) was added in one portion. The solution was stirred for 1 h at −78 °C, and a colorless precipitate appeared. The reaction was quenched with 3 mL of water, and the THF was removed in vacuo. The residue was dissolved in 75 mL of water and extracted with 2 × 75-mL portions of ether. The aqueous layer was acidified to pH 2 with 6 N concentrated HCl and extracted with two 75-mL portions of ether. The combined organic layers were washed twice with brine, dried ($MgSO_4$), and concentrated to yield 3.73 g (92%) of colorless crystals, mp 89–91 °C: ^{1}H NMR ($CDCl_3$) δ 7.17 (AB q, *J* = 7.9 Hz, 4 H), 4.70 (br s, 1 H), 4.74 (br s, 2 H), 3.76 (dd, *J* = 8.7, 6.7 Hz, 1 H), 2.80 (dd, *J* = 8.7, 14.7 Hz, 1 H), 2.42 (dd, *J* = 14.7, 6.7 Hz, 1 H), 2.32 (s, 3 H), 1.70 (s, 3 H). Anal. Calcd for $C_{13}H_{16}O_2$: C, 76.44; H, 7.89. Found: C, 76.18; H, 7.76.

2-*p*-Tolyl-4-oxopentanoic Acid (13, X = O). To a magnetically stirred solution of 2.5 g (12.3 mmol) of the above pentenoic acid in 300 mL of EtOAc at −78 °C was introduced ozone via a gas dispersion tube until the solution turned blue. The excess ozone was allowed to dissipate, and 1 mL of dimethyl sulfide was added. The ethyl acetate was removed in vacuo, and the residue was dissolved in 50 mL of 5% NaOH. The aqueous solution was washed with two 50-mL portions of ether, acidified to pH 2 with 6 N HCl, and extracted with two 50-mL portions of ether. The combined ether layers were extracted twice with brine, dried ($MgSO_4$), and concentrated to yield 2.34 g (93%) of colorless crystals, mp 133–134 °C: ^{1}H NMR ($CDCl_3$) δ 7.14 (AB q, *J* = 8.4 Hz, 4 H), 4.08 (dd, *J* = 10.1, 4.4 Hz, 1 H), 3.33 (dd, *J* = 18.0, 10.1 Hz, 1 H), 2.70 (dd, *J* = 18.0, 4.4 Hz, 1 H), 2.32 (s, 3 H), 2.16 (s, 3 H). Anal. Calcd for $C_{12}H_{14}O_3$: C, 69.89; H, 6.84. Found: C, 69.92; H, 6.83.

Exo–Endo *p*-Tolyl Bicyclic Lactam 14. A stirred solution of 2.30 g (11.2 mmol) of keto acid **13** and 1.17 g (11.3 mmol) of (*S*)-valinol in 200 mL of dry benzene was heated under azeotropic removal of water with a Dean–Stark trap for 16 h. The solution was cooled and concentrated, and the residue was dissolved in 100 mL of ether. The solution was washed with saturated NH_4Cl, saturated Na_2CO_3, and brine, dried ($MgSO_4$), and concentrated to yield 2.81 g (91%) of a yellow oil, which was a 1:1 mixture of exo–endo epimers: ^{1}H NMR ($CDCl_3$) δ [*exo*-**14**] 7.15 (s, 4 H), 4.24 (dd, *J* = 8.3, 8.1 Hz, 1 H), 3.87 (m, 1 H), 3.85 (m, 1 H), 3.72 (m, 1 H), 2.73 (dd, *J* = 13.9, 10.8 Hz, 1 H), 2.33 (s, 3 H), 2.22 (dd, *J* = 13.9, 3.9 Hz, 1 H), 1.71 (m, 1 H), 1.45 (s, 3 H), 1.10 (d, *J* = 6.6 Hz, 3 H), 0.91 (d, *J* = 6.6 Hz, 3 H) [*endo*-**14**] 7.13 (s, 4 H), 4.23 (dd, *J* = 8.7, 7.4 Hz, 1 H), 4.04 (dd, *J* = 11.6, 8.7 Hz, 1 H), 3.92 (dd, *J* = 8.8, 6.3 Hz, 1 H), 3.70 (ddd, *J* = 10.1, 6.8, 6.8 Hz, 1 H), 2.63 (dd, *J* = 12.2, 12 Hz, 1 H), 2.32 (s, 3 H), 1.70 (m, 1 H), 1.56 (s, 3 H), 1.04 (d, *J* = 6.7 Hz, 3 H), 0.90 (d, *J* = 6.6 Hz, 3 H).

Bicyclic Lactams 15 and 16. To a stirred solution of 0.55 g (2.01 mmol) of lactam mixture **14** in 50 mL of THF at −100 °C was added 2.38 mL (3.19 mmol) of 1.34 M *sec*-BuLi. The mixture was stirred for 30 min, at which time 0.40 mL (6.42 mmol) of MeI was added. After 1 h, the reaction was quenched with 2 mL of water, concentrated, dissolved in ether, and washed with water and brine. The ether layer was dried ($MgSO_4$) and concentrated to yield 0.52 g (90%) of a yellow oil (93:7 ratio of **15** to **16**). The oil was crystallized from *n*-heptane to yield 0.35 g (60%) of colorless prisms, **15**, mp 71.5–72.5 °C, $[\alpha]^{20}_D$ +109° (*c* 1.16, EtOH).

Major product **15**: ^{1}H NMR ($CDCl_3$) δ 7.19 (AB q, *J* = 8.0 Hz, 4 H), 4.23 (dd, *J* = 8.3, 7.5 Hz, 1 H), 3.84 (dd, *J* = 8.3, 6.6 Hz, 1 H), 3.73 (m, 1 H), 2.69 (d, *J* = 13.0 Hz, 1 H), 2.32 (s, 3 H), 2.30 (d, *J* = 13.0 Hz, 1 H), 1.62 (m, 1 H), 1.47 (s, 3 H), 1.15 (s, 3 H), 1.09 (d, *J* = 6.7 Hz, 3 H), 0.90 (d, *J* = 6.6 Hz, 3 H). Anal. Calcd for $C_{18}H_{25}NO_2$: C, 75.24; H, 8.77; N, 4.87. Found: C, 75.30; H, 8.89; N, 4.80.

Minor product **16**: ^{1}H NMR ($CDCl_3$) δ 7.20 (AB q, *J* = 8.0 Hz, 4 H), 4.25 (dd, *J* = 8.6, 7.9 Hz, 1 H), 3.83 (dd, *J* = 8.6, 7.1 Hz, 1 H), 3.66 (m, 1 H), 2.75 (d, *J* = 13.9 Hz, 1 H), 2.34 (d, *J* = 13.9 Hz, 1 H), 2.31 (s, 3 H), 1.72 (s, 3 H), 1.59 (s, 3 H), 1.07 (d, *J* = 6.7 Hz, 3 H), 0.89 (d, *J* = 6.6 Hz, 3 H).

Carbinolamine 17. To a stirred solution of 0.70 g (2.44 mmol) of crystalline lactam **15** in 33 mL of THF at −60 °C was added 0.90 mL (1.53 mmol) of 1.7 M Red-Al in toluene. The solution was kept at −20 °C for 20 h, quenched with 0.5 mL of MeOH, and concentrated. The residue was dissolved in 50 mL of hexane–ether (1:1), washed with 10% NaOH, water, and brine, dried ($MgSO_4$), and concentrated to yield 0.74 g (100%) of a colorless oil: ^{1}H NMR ($CDCl_3$) δ 7.17 (AB q, *J* = 8.3 Hz, 4 H), 4.55 (d, *J* = 4.4 Hz, 1 H), 4.23 (dd, *J* = 8.7, 7.8 Hz, 1 H), 3.65 (t, *J* = 8.7 Hz, 1 H), 3.09 (q, *J* = 8.6 Hz, 1 H), 2.46 (AB q, *J* = 13.8 Hz, 2 H), 2.32 (s, 3 H), 1.53 (d, *J* = 4.4 Hz, 1 H), 1.42 (s, 6 H), 1.06 (d, *J* = 6.5 Hz, 3 H), 0.84 (d, *J* = 6.6 Hz, 3 H). This material was carried on directly to the next step.

(*S*)-2-*p*-Tolyl-2-methyl-4-oxovaleraldehyde (18). The carbinolamine **17** (0.74 g, 2.44 mmol) was added to a mixture of 50 mL of ethanol and 50 mL of 1 M $Bu_4NH_2PO_4$ (in water) and stirred at room temperature for 12 h. The solution was concen-

trated and extracted with two 50-mL portions of ether. The combined organic layers were washed with water and brine, dried ($MgSO_4$), and concentrated to yield 0.49 g of a yellow liquid (98%): ^1H NMR ($CDCl_3$) δ 9.51 (s, 1 H), 7.15 (AB q, J = 5.9 Hz, 4 H), 3.10 (AB q, J = 17.2 Hz, 2 H), 2.32 (s, 3 H), 2.06 (s, 3 H), 1.57 (s, 3 H). No further purification of this material was attempted.

(*S*)-4-*p*-Tolyl-4-methylcyclopentenone (19). To a stirred solution of 0.49 g (2.40 mmol) of keto aldehyde **18** in 60 mL of dry THF was added 200 μL of 1 M KOH in ethanol. The solution was stirred at room temperature for 1 h and concentrated in vacuo. The residue was dissolved in ether, washed with water and brine, dried ($MgSO_4$), and concentrated. The yellow liquid was purified by column chromatography (EM Merck 7747 silica gel) to yield 0.40 g (90%) of cyclopentenone as a colorless liquid: $[\alpha]^{20}_D$ +114° (c 1.36, EtOH); ^1H NMR ($CDCl_3$) δ 7.66 (d, J = 5.5 Hz, 1 H), 7.15 (s, 4 H), 6.19 (d, J = 5.5 Hz, 1 H), 2.58 (AB q, J = 18.7 Hz, 2 H), 2.33 (s, 3 H), 1.62 (s, 3 H). Spectral properties were identical with those reported for racemic material.[6]

(*S*)-4,5,5-Trimethyl-4-*p*-tolylcyclopentenone (20). A solution of 100 mg (0.54 mmol) of cyclopentenone **19** in 0.3 mL of DMF was added over 0.25 h to a stirred solution of 0.33 g (1.3 mmol) of NaH in 0.3 mL of DMF. The reaction was stirred for 0.5 h, and 0.17 mL (2.70 mmol) MeI was added dropwise. The mixture was stirred for 16 h at 25 °C, and the excess hydride was decomposed by adding 0.30 mL of MeOH. The solution was diluted with 30 mL of ether, washed with water and brine, dried ($MgSO_4$), and concentrated. The yellow residue was purified by column chromatography on silica gel to yield 54 mg (48%) of dimethylated cyclopentenone **20**: ^1H NMR ($CDCl_3$) δ 7.75 (d, J = 5.9 Hz, 1 H), 7.10 (AB q, J = 9.3 Hz, 4 H), 6.22 (d, J = 5.9 Hz, 1 H), 2.34 (s, 3 H), 1.45 (s, 3 H), 1.19 (s, 3 H), 0.53 (s, 3 H). Spectral properties were identical with those reported for racemic material.[6]

(*S*)-(−)-α-Cuparenone (12). A stirred solution of 54 mg (0.25 mmol) cyclopentenone **20** and 10 mg of 10% Pd/C in 2 mL of ethyl acetate was stirred under 3 atm of hydrogen for 2 h. The solution was then filtered through a 1-in. plug of silica gel and concentrated to yield 47 mg (87%) of α-cuparenone: $[\alpha]^{20}_D$ −166° (c 0.200, $CHCl_3$) (lit.[4,7] $[\alpha]_D$ −170°); ^1H NMR ($CDCl_3$) δ 7.22 (AB q, J = 8.4 Hz, 4 H), 2.60 (m, 1 H), 2.40 (m, 2 H), 2.32 (s, 3 H), 1.90 (m, 1 H), 1.26 (s, 3 H), 1.17 (s, 3 H), 0.61 (s, 3 H); ^{13}C NMR ($CDCl_3$) δ 221.6, 142.2, 135.8, 128.9, 126.4, 53.2, 48.5, 33.7, 30.0, 25.4, 22.1, 20.7, 18.6. Anal. Calcd for $C_{15}H_{20}O$: C, 83.33; H, 9.33. Found: C, 83.18; H, 9.48.

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An Efficient and Novel Approach to the Synthesis of 3,4,5,6-Tetraphenyl-2(1*H*)-pyridinone

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The reaction of tetracyclone (**1a**) with sodium azide under acidic conditions was found to give 3,4,5,6-tetraphenyl-2(1*H*)-pyridinone (**2a**) in 90% isolated yield. The reaction does not proceed by a Schmitt mechanism. By adjusting the reaction conditions, the intermediate 1,5,7,8-tetraphenyl-2,3,4-triazabicyclo[3.3.0]octa-2,7-dien-6-one (**3a**) was isolated in 94% yield. Treatment of **3a** under acidic conditions resulted in loss of nitrogen and formation of **2a**. When the acidification was carried out in methanol, 2-methoxy-3,4,5,6-tetraphenylpyridine (**4a**) was also produced. Treatment of the N-methyl intermediate (**6a**) under similar conditions produced 1-methyl-3,4,5,6-tetraphenyl-2(1*H*)-pyridinone (**5a**) in good yield. Reaction of 2-methyl-3,4,5-triphenyl-2,4-cyclopentadien-1-one (**1b**) with sodium azide gave similar results.

Tetraphenyl-2(1*H*)-pyridinone (**2a**) has been prepared by a variety of methods. Wajon and Arens[2] described the condensation of α-benzoylbenzyl cyanide with benzyl phenyl ketone under acidic conditions to produce the title compound in 50% yield. Jagt and Van Leusen[3] reported the Diels–Alder cycloaddition of tosyl cyanide to tetracyclone, followed by base hydrolysis to yield the pyridinone in 69% yield. Abramovitch and Knaus[4] described the addition of singlet sulfonyl nitrene to tetracyclone which resulted in a 13% yield of the pyridinone, while Martin and Bauer[5] obtained the title compound in 76% yield by the Diels–Alder addition of trichloroethyl cyanate to tetracyclone, with subsequent hydrolysis. The above syntheses of tetraphenylpyridinone all suffer from either poor yields, multiple steps, or the use of exotic reagents. In this paper we report a highly efficient, one-pot synthesis of tetraphenylpyridinone using readily available and relatively inexpensive starting materials.

Results and Discussion

Treatment of tetracyclone (**1a**) with sodium azide under acidic conditions resulted in a 90% isolated yield of the corresponding tetraphenylpyridinone (**2a**) (Scheme I). However, when the reaction was run under milder conditions (i.e., lower temperature, shorter reaction time), the bicyclic triazoline **3a** was obtained in 94% yield. Subsequent acidification of **3a** resulted in elimination of nitrogen and formation of the pyridinone **2a**. Thus it appears that **3a** is an intermediate in the formation of **2a** and that the reaction does not take place by a classical Schmitt reaction pathway.[6]

(1) Presented at the 36th Southeastern Regional Meeting of the American Chemical Society, Raleigh, NC, October 25, 1984.
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