# A General Approach to Indolizidine Alkaloids From 1-Benzyloxy-5-(*p*-toluenesulfonamido)-3-alken-2-ols: Synthesis of (+)-Monomorine I[+]

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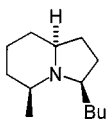
**Keywords:** Alkaloids / Asymmetric synthesis / Palladium / Amino alcohols / Natural products / Monomorine I

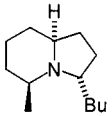
A versatile method for the preparation of indolizidine alkaloids from 1-benzyloxy-5-(*p*-toluenesulfonamido)-3-alken-2-ols as stereodefined key intermediates has been developed. The utility of this approach was demonstrated by the synthesis of (+)-monomorine I.

## Introduction

Indolizidine alkaloids have recently attracted a lot of attention due to their interesting pharmacological properties.[1] Some of these alkaloids such as Monomorine I (**1**) and Indolizidine 195B (**2**) have been the targets for synthetic organic chemists for some time.[2−4]



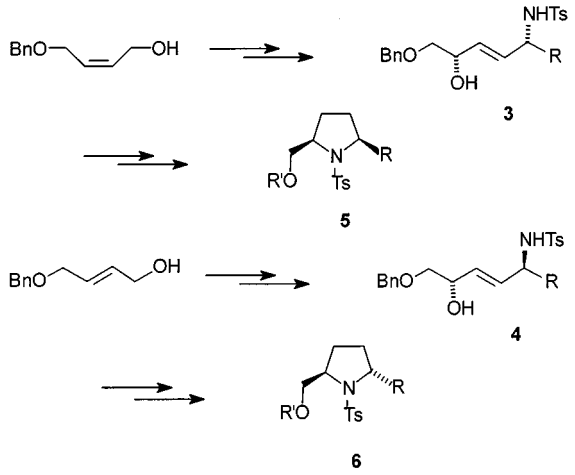
(+)-Monomorine I (**1**)



(-)-Indolizidine 195B (**2**)

(+)-Monomorine I (**1**) is a trail pheromone of the pharaoh ant *Monomorium pharaonis L.*[5] This ant, which is of tropical origin and now established in North America and western Europe, represents a health hazard particularly in hospitals. The possibility of controlling this pest by pheromone manipulations was first investigated by Ritter and co-workers,[2] and later by others.[3] The C-3 epimer, named indolizidine 195B was isolated by Daly and co-workers in 1986 from the Colombian poison frog *Dendrobates histrionicus*,[6] and has been synthesized by several groups.[4] The first asymmetric synthesis of (+)-monomorine was accomplished by Yamazaki and Kibayashi from (+)-diethyl tartrate in 1988.[3b]

Particularly attractive is the development of synthetic methodology that allows for the preparation of a large number of structurally similar analogs from *a single common intermediate*. In this regard we have recently described a simple and high yield method for the preparation of a variety of 1-benzyloxy-5-(*p*-toluenesulfonamido)-3-alken-2-ols[7] with complete control of both absolute and relative stereochemistry from the appropriate monobenzylated 2-butene-1,4-diol (Scheme 1). The stereochemistry is introduced by Sharpless epoxidation[8] of 4-benzyloxy-2-butenol and this epoxide can also be obtained from commercially available (2*S*,3*R*)-(−)-4-benzyloxy-2,3-epoxy-1-butyl 4-nitrobenzoate, by hydrolysis, in 90% yield.




Scheme 1. Stereoselective route towards 2,5-disubstituted pyrrolidines

The synthetic utility of δ-amidoalkenols such as **3** and **4** was demonstrated by the preparation of a variety of 2,5-disubstituted pyrrolidines **5** and **6**,[7][9] and more wide ranging applications to heterocyclic synthesis were envisioned for these compounds. We now report on the synthesis of (+)-monomorine I (**1**) via stereodefined intermediates **3** and **5**. A key step in the transformation of **5** to **1** is a novel Wittig coupling with a ketone-protected phosphonium salt.

## Results and Discussion

A general route into the indolizidine skeleton was envisioned to proceed from **3** and **4** via the corresponding benzyloxymethyl pyrrolidines **5** and **6**. Since the technology to prepare these pyrrolidines with a variety of R groups and with any desired stereochemistry (vide supra) had been developed previously,[7] this route has the potential to be extremely versatile. A key step in the transformations of the

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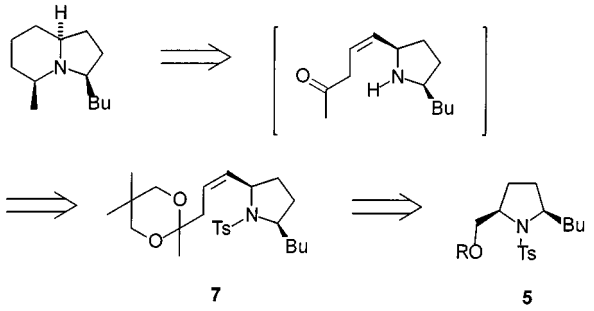
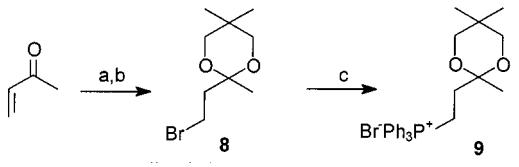
pyrrolidines **5** and **6** to the target molecules is the coupling of the hydroxymethyl pyrrolidine fragment (or more precisely, the corresponding aldehyde) with a hydrocarbon unit containing a masked carbonyl. At some later stage, after the tosylate has been removed from the nitrogen, cyclization would be initiated by liberation of the carbonyl as depicted retrosynthetically in Scheme 2.



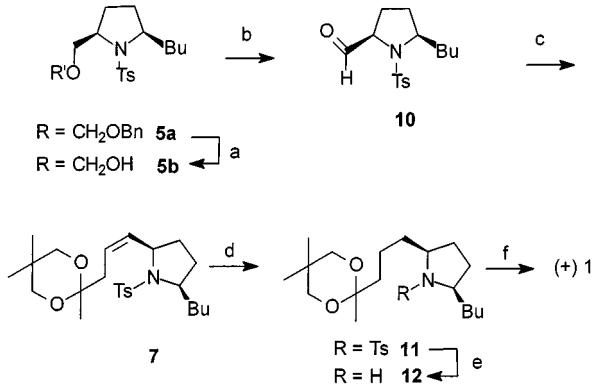

Scheme 2. Retrosynthetic analysis

As the preparation of the pyrrolidine fragment was already known,[7] our main task was to synthesize and couple an appropriately masked carbonyl fragment. The most logical method to accomplish this is by Wittig coupling of the pyrrolidine aldehyde obtained from **5** with an appropriate ylide (e.g. one based on a ketal-protected ethyl ketone). Synthesis of this phosphonium salt is shown in Scheme 3. Conversion of the methyl vinyl ketone into the bromo-ketal **8** is known[10] and can be accomplished conveniently on a multigram scale. Formation of the highly hygroscopic phosphonium salt **9** occurs readily in cyclohexane at reflux temperatures.[11] It is noteworthy that the corresponding ethylene ketal does not survive phosphonium salt formation and the propanediol-based ketal is difficult to form.




Scheme 3. (a) HBr anhydrous; (b) 2,2-dimethylpropanediol, $(EtO)_3CH$, PTSA; (c) $PPh_3$, $C_6H_{12}$, reflux

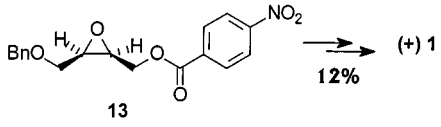
With a viable route to **9** in multigram quantities, we next turned our attention to the oxidation of pyrrolidine **5b** to the corresponding aldehyde, and subsequent Wittig coupling with phosphonium bromide **9**. Since it was known that **5b** was susceptible to radical-induced ring opening[12] a mild oxidation method was called for. In this regard both tetrapropyl ammonium perruthenate[13] (TPAP) and $SO_3$•pyridine/DMSO[14] are outstanding reagents. Although both reagents work, the $SO_3$•pyridine/DMSO method is simpler and allows for the use of the aldehyde in the next step directly, without any further purification (Scheme 4). The Wittig coupling proceeded smoothly to give olefin **7** (>97% *Z*) in 65% overall yield from **5b**.




Scheme 4. (a) Pd/C, $H_2$, MeOH; (b) pyridine−$SO_3$, DMSO, $Et_3N$, $CH_2Cl_2$; (c) **9**, *t*BuOK, THF, −78° C; (d) $PtO_2$, $H_2$, EtOH; (e) $Na/NH_3$, EtOH, −78° C to room temp.; (f) 10% Pd/C, $H_2$, 1M HCl, MeOH

In preparation for the final cyclization step the double bond was hydrogenated over $PtO_2$ to give **11** in 96% yield. The tosyl protecting group was removed from the nitrogen using $Na/NH_3$ in ethanol,[4a] to give the free amine **12** in 62% yield. Attempts to use sodium-mercury amalgam in buffered methanol[15] were less successful, leading to a slow reaction which was not complete after 48 h.

With the free amine **12** in hand, we were now ready for the final cyclization. It was envisioned that acidic cleavage of the ketal in methanol in the presence of $H_2$ and Pd/C would produce the indolizidine by spontaneous cyclization to the imine and subsequent hydrogenation of the carbon-nitrogen double bond. Treatment of **12** with Pd/C under 1 atm of $H_2$ in acidic (HCl) methanol for 6 days afforded the (+)-monomorine I HCl salt. This salt was treated with base to give the desired (+)-monomorine I (**1**) in 92% yield and with an $[\alpha]^{22}_D$ of +34.7° (*c* = 1.22, hexane). Similar approaches to monomorine I are described in the literature.[3a−d,3i,3l] The overall yield of (+) **1** from the commercially available epoxide **13** is 12%.




After completion of this work we became aware of a recent publication by Krohn and Bernhard,[16] where the Wittig salt **9** was prepared and used in olefination of aromatic aldehydes.

## Conclusions

A versatile route to indolizidine alkaloids has been developed based on the use of 1-benzyloxy-5-(*p*-toluenesulfonamido)-3-alken-2-ols (**3** and **4**) as stereodefined key intermediates. It was demonstrated that **3** can be converted into (+)-monomorine I (**1**) via **5**. A key step in this transformation is the Wittig reaction using phosphonium salt **9**. The

analogous conversion of **4** via **6** would give access to (−)-Indolizidine 195B (**2**). Because intermediates **3** and **4** are available with a variety of R groups and with any desired stereochemistry (both relative and absolute),[7] this route has the potential to be extremely flexible. Furthermore, with the accessibility of a large number of α,β-unsaturated ketones (c.f. Wittig salt **9**), preparation of a variety of indolizidines modified on the B ring should be possible.

## Experimental Section

**General:** $^1$H and $^{13}$C NMR spectra were recorded at 300 MHz (75.4 for $^{13}$C) or 400 MHz (100.6 for $^{13}$C) with $CDCl_3$ (7.26 ppm $^1$H, 77 ppm $^{13}$C) as internal standard unless otherwise stated. Elemental analyses were performed by Analytishen laboratorien, Lindlar, Germany. 2,5,5-Trimethyl-2-(2-bromoethyl)-1,3-dioxane was prepared according to a literature procedure.[10]

**(2*R*,5*R*)-2-(Hydroxymethyl)-5-butyl-*N*-(*p*-tosyl)-pyrrolidine (5b):** To a stirred solution of pyrrolidine **5a** (0.154 g, 0.40 mmol) in MeOH (2.6 mL) was added 5% palladium on carbon (41 mg) and $MeSO_3H$ (26 μL). A hydrogen pressure of 1 atm was applied and the reaction was stirred for 3 h at room temp. The reaction mixture was filtered through celite and the MeOH was removed in vacuo. The resulting oil was dissolved in $CH_2Cl_2$ and filtered through a plug of $Na_2CO_3$. Concentration of the filtrate afforded 0.112 g, 95% of the product. − $^1$H NMR ($CDCl_3$, 400 MHz): δ = 7.72 (d, *J* = 8.0 Hz, 2 H), 7.31 (d, *J* = 8.0 Hz, 2 H), 3.66−3.72 (m, 4 H), 2.63 (br s, 1 H), 2.42 (s, 3 H), 1.75−1.84 (m, 1 H), 1.56−1.71 (m, 2 H), 1.24−1.54 (m, 7 H), 0.90 (t, *J* = 7.2 Hz, 3 H). − $^{13}$C NMR ($CDCl_3$, 100.6 MHz): δ = 143.7, 134.4, 129.7 (2C), 127.6 (2C), 66.1, 63.0, 62.7, 36.4, 29.2, 28.4, 27.2, 22.5, 21.5, 14.0. − $C_{16}H_{25}NO_3S$ (311.2): calcd. C 61.71, H 8.09, N 4.50; found C 61.57, H 8.17, N 4.65. − $[\alpha]_D^{22}$ = −47 (*c* = 1.0, $CHCl_3$).

**Phosphonium Salt 9:** 2,5,5-Trimethyl-2-(2-bromoethyl)-1,3-dioxane (12.5 g, 52.7 mmol) and $PPh_3$ (27.6 g, 105.3 mmol) were dissolved in cyclohexane (30 mL) and the mixture was heated at reflux for 24 h under argon. A sticky solid was formed at the bottom of the flask. After cooling to room temp. the liquid was decanted and the remaining solid was quickly crushed to powder and then transferred to another flask which was put under an inert atmosphere. The powder was then washed in the flask with 2 × 10 mL of $Et_2O$ and 2 × 10 mL of pentane. The resulting powder was dried overnight in a $P_2O_5$ dessicator. **Caution:** The Wittig salt is extremely hygroscopic and will quickly become a sticky gum if exposed to humid air for long, thus crushing must be done quickly.

**(2*R*,5*R*)-2-(Formyl)-5-butyl-*N*-(*p*-tosyl)-pyrrolidine (10):** To a stirred solution of pyrrolidine **5b** (0.26 g, 0.84 mmol) and $Et_3N$ (0.6 mL, 4.4 mmol) in $CH_2Cl_2$ (4.4 mL) at −10°C was added a solution of pyridine−$SO_3$ (0.7 g, 4.4 mmol) in dry DMSO (4.5 mL). The cooling bath was removed and the reaction mixture was stirred vigorously. When the reaction was complete (TLC 30% $Et_2O$ in pentane, ca 40 min), the mixture was poured into a separatory funnel containing ice and brine (10 mL) and extracted with $Et_2O$ (20 mL × 3). The organic phase was first washed with a 10% aqueous solution of citric acid then with brine, and finally dried ($Na_2SO_4$). The organic phase was filtered through a silica plug and condensed in vacuo to give a fairly pure aldehyde which was used directly in the next step. − $^1$H NMR ($CDCl_3$, 400 MHz): δ = 9.62 (d, *J* = 2.3 Hz, 1 H), 7.71 (d, *J* = 8.0 Hz, 2 H), 7.33 (d, *J* = 8.0 Hz, 2 H), 3.85 (dt, *J* = 2.3, 7.4 Hz, 1 H), 3.68 (m, 1 H), 2.43 (s, 3 H), 1.2−2.1 (m, 10 H), 0.90 (t, *J* = 6.9 Hz, 3 H). − $^{13}$C NMR ($CDCl_3$, 100.6 MHz): δ = 200.3, 144.3, 134.0, 129.7 (2C), 127.6 (2C), 67.7, 62.0, 35.9, 29.8, 28.2, 25.5, 22.5, 21.5, 14.0.

**Compound 7:** *t*BuOK (0.5 g, 4.4 mmol) was dissolved in THF (12 mL) and cooled to −78°C. The Wittig salt **9** (2.2 g, 4.4 mmol) was added and the resulting dark orange solution was stirred at −78°C for 10 min. The crude aldehyde **10** from above in 5 mL of THF was added dropwise with a syringe to the reaction mixture. The solution lightened in color and was allowed to stir for 1.5 h and then warmed to room temp. for a further 1 h. The reaction was then poured into water, extracted with $Et_2O$ and washed with brine. Drying over $Na_2SO_4$ and removal of solvent in vacuo afforded the crude product, which was purified by flash chromatography [$SiO_2$, pentane/$Et_2O$ (80:20)] to give 0.25 g (65% from **5b**) of the product as an oil. − $^1$H NMR ($CDCl_3$, 400 MHz): δ = 7.71 (d, *J* = 8.0 Hz, 2 H), 7.29 (d, *J* = 8.0 Hz, 2 H), 5.43−5.59 (m, $J_{CH=CH} \approx$ 10 Hz, 2 H), 4.33 (apparent q, *J* = 7 Hz, 1 H), 3.71 (m, 1 H), 3.54 (dd, *J* = 2.0, 11.0 Hz, 2 H), 3.48 (d, *J* = 11.0 Hz, 2 H), 2.64 (dd, *J* = 7.0, 15.2 Hz, 1 H), 2.48 (dd, *J* = 8.0, 15.2, 1 H), 2.42 (s, 3 H), 1.1−1.9 (m incl. singlet at 1.36, 13 H), 0.99 (s, 3 H), 0.87−0.94 (m, 6 H). − $^{13}$C NMR ($CDCl_3$, 100.6 MHz): δ = 143.0, 135.9, 134.3, 129.5 (2C), 127.5 (2C), 124.3, 98.8, 70.5 (2C), 61.8, 58.3, 36.6, 35.5, 31.9, 30.0, 29.7, 28.4, 22.7, 22.6, 22.6, 21.5, 20.9, 14.1. − $[\alpha]_D^{22}$ = +25 (*c* = 1.0, $CHCl_3$)

**Compound 11:** The pyrrolidine **7** (28 mg, 0.06 mmol) was dissolved in 99.5% EtOH (0.6 mL) and $PtO_2$ (0.7 mg, 0.003 mmol) was added. A hydrogen pressure of 1 atm was applied and the heterogeneous system was stirred for 5 h. The catalyst was removed by filtration through celite, and the resulting solution was concentrated to afford 27 mg (96%) of pure product. − $^1$H NMR ($CDCl_3$, 300 MHz): δ = 7.71 (d, *J* = 8.0 Hz, 2 H), 7.30 (d, *J* = 8.0 Hz, 2 H), 3.42−3.56 (m, 6 H), 2.40 (s, 3 H),1.25−1.95 (m incl. a s at 1.36, 19 H), 1.02 (s, 3 H), 0.90 (m, 6 H). − $^{13}$C NMR ($CDCl_3$, 75.4 MHz): δ = 143.0, 135.3, 129.5 (2C), 127.5 (2C), 98.9, 70.3 (2C), 61.7, 61.6, 37.9, 37.5, 36.9, 30.0, 29.6, 29.5, 28.5, 22.8, 22.6, 22.5, 21.5, 20.3, 20.2, 14.1. − $C_{25}H_{41}NO_4S$ (451.3): calcd. C 66.48, H 9.15, N 3.10; found C 66.20, H 8.99, N 3.26. − $[\alpha]_D^{22}$ = +15 (*c* = 1.0, $CHCl_3$). The product was found to be >99.5% *ee* as determined by HPLC analysis using a Chiralcel OD-H column (flow rate 0.5 mL/min, hexane/2-propanol 96:4).

**Compound 12:** A solution of **11** (0.183 g, 0.41 mmol) in EtOH (3.3 mL) was added to liquid ammonia (11 mL) with stirring at −78°C. To this mixture was added sodium (0.54 g, 23.5 mmol) in small portions after which the reaction was allowed to warm to room temp. After 24 h the mixture was diluted with $Et_2O$ (15 mL) and quenched with $NH_4Cl$ (aq) (1.5 mL). The aqueous phase was separated, back extracted with ether (2 × 15 mL) and dried over $Na_2SO_4$. Concentration in vacuo afforded a residual oil which was purified by chromatography (silica gel, $Et_2O$ followed by EtOAc and finally EtOH) to give the product as an oil (0.075 g, 62%). − $^1$H NMR ($CDCl_3$, 400 MHz): δ = 3.55 (br s, 1 H), 3.52 (d, *J* = 11.7, 2 H), 3.41 (d, *J* = 11.7, 2 H), 3.08 (m, 2 H), 1.91 (m, 2 H), 1.2−1.7 (m incl. singlet at 1.34, 17 H), 0.99 (s, 3 H), 0.88 (m, 6 H). − $^{13}$C NMR ($CDCl_3$, 100.6 MHz): δ = 98.8, 70.3 (2C), 59.4 (2C), 38.1, 35.7, 35.2, 30.6 (2C), 29.9, 29.4, 22.8 (2C), 22.5, 21.1, 20.3, 14.0. − $[\alpha]_D^{22}$ = +3 (*c* = 1.0, $CHCl_3$).

**(+) Monomorine (1):** To a solution of **12** (21 mg, 0.07 mmol) in MeOH (3.5 mL) was added 10% Pd/C (22 mg) and 1M HCl (35 μL) and the mixture was stirred for 6 days under $H_2$ (1 atm). 1M HCl (90 μL) was again added to transform all amine to the HCl salt. Filtration through celite and concentration in vacuo afforded monomorine hydrochloride. The product was dissolved in $CH_2Cl_2$ (10 mL), transferred to a small separatory funnel and saturated

$Na_2CO_3$ (1.5 mL) was added. The organic phase was collected and the aqueous phase was extracted with $CH_2Cl_2$ (2 × 5 mL). Careful removal of the solvent (bp 58 °C at 0.25 Torr) gave the crude monomorine which was purified by chromatography (basic alumina, $CHCl_3$ 30% in hexane) to give (+)-monomorine I (**1**) (12.5 mg, 92%) as an oil. – $[\alpha]_D^{22}$ = +34.7 (*c* = 1.22, hexane) {ref.[3c] $[\alpha]_D^{22}$ = +34.3 (*c* = 1.02, hexane)}. Spectral data were in accordance with those previously reported.[3c,3m]

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