

# Quinolizidines. XXVI. : A Synthesis of ( $\pm$ )-Deplancheine

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## Quinolizidines. XXVI.<sup>1)</sup> A Synthesis of (±)-Deplancheine

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A formal racemic synthesis of the *Alstonia* and *Aspidosperma* alkaloid deplancheine (12) has been achieved in the form of the synthesis of the tetracyclic methyl ketone 11 through the "3-acetylpyridine route." The route started with an initial quaternization of the ketal 5 with 2-(3-indolyl)ethyl bromide and proceeded through the intermediates 7 and 8.

**Keywords** quaternization; pyridinium salt catalytic reduction; piperidine mercuric acetate–edetic acid oxidation; oxidative cyclization; deketalization; sodium borohydride reduction; indoloquinolizidine alkaloid synthesis

The tetracycle 4 represents the parent framework common to indolo[2,3-*a*]quinolizidine alkaloids, which are often referred to as indoloquinolizidine alkaloids.<sup>2)</sup> This base itself is also a natural product isolated in partially racemized form from the leaves of *Dracontomelum mangiferum* BL. (Anacardiaceae).<sup>3)</sup> It has been a model target selected for synthesis by many investigators<sup>4)</sup> who are interested in the syntheses of more complex indoloquinolizidine alkaloids. In 1962, Wenkert and Wickberg<sup>5)</sup> prepared the tetracycle 4 from 3-(2-piperidinoethyl)indole (1) in 63% yield by oxidative cyclization with 10 molar eq of Hg(OAc)<sub>2</sub> in hot 5% aqueous AcOH for 1 h, followed by treatment with H<sub>2</sub>S and NaBH<sub>4</sub> reduction of the over-oxidized product 3 present as a minor component. The procedure and yield of 4 in this oxidative cyclization (1→2→4) were recently improved by us through the use of 3 molar eq of Hg(OAc)<sub>2</sub>–edetate disodium (EDTA·2Na) in boiling aqueous EtOH for 3 h.<sup>6)</sup> In the present study, the applicability of the modified oxidative cyclization procedure to syntheses of more complex indoloquinolizidine alkaloids was tested in a synthesis of (±)-deplancheine (12),<sup>7,8)</sup> whose (+)-enantiomer was isolated from *Alstonia* and *Aspidosperma* plants<sup>7c,9)</sup> belonging to the family Apocynaceae and was recently assigned the (*R*) configuration.<sup>8)</sup>

Quaternization of the ketal 5, a synthetic equivalent for 3-acetylpyridine, with 2-(3-indolyl)ethyl bromide was effected in HCONMe<sub>2</sub> at 80–85 °C for 50 h, and the crude quaternary salt 6 that formed was hydrogenated over Adams catalyst in aqueous EtOH at 35 °C and atmospheric

pressure to give the piperidine derivative 7 in 78% overall yield (from 5). Treatment of 7 with 3 molar eq of Hg(OAc)<sub>2</sub>–EDTA·2Na in boiling aqueous EtOH for 3 h furnished, after reduction of the over-oxidized product (type 3) with NaBH<sub>4</sub>, the tetracyclic ketal 8 in 65% yield. In this oxidative cyclization, the isomeric ketal 9 would be another possible product, but we were unable to find it in the reaction mixture. Such high regioselectivity in the Hg(OAc)<sub>2</sub>–EDTA oxidation of 7 is comparable to that observed for the structurally analogous 1-(3,4-dimethoxyphenethyl)piperidine system.<sup>10)</sup> The relative configuration of 8 was assigned from a consideration of the thermo-

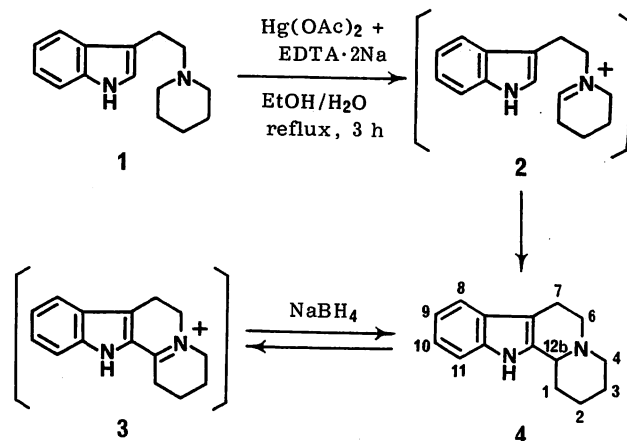


Chart 1

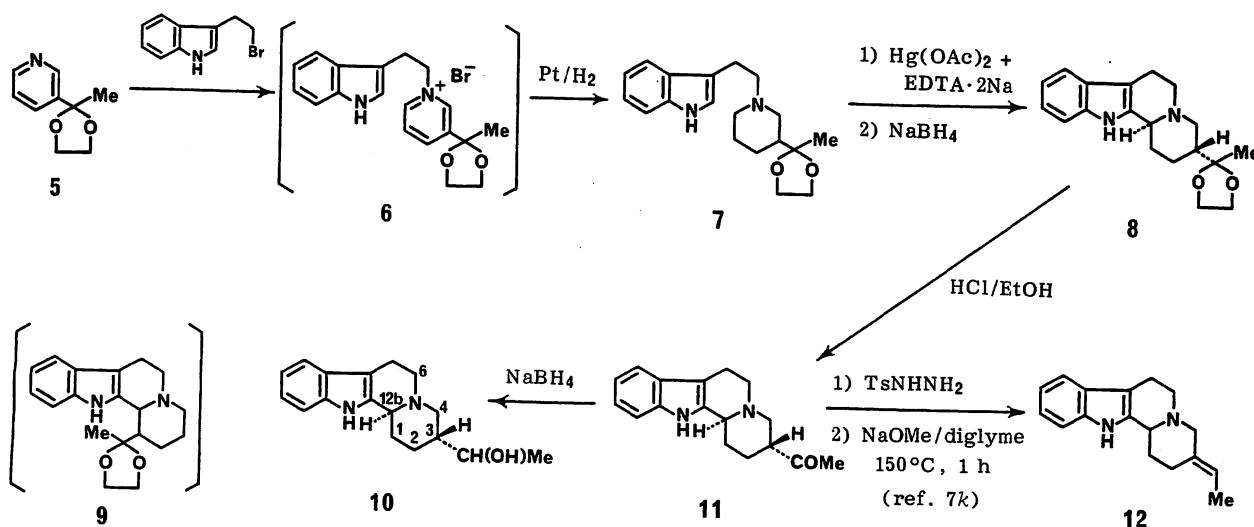


Chart 2

dynamic stabilities of the possible diastereomers, and the whole structure was eventually substantiated by the following self-consistent reaction sequence.

Deketalization of **8** with 1N aqueous HCl in boiling EtOH for 2 h afforded the methyl ketone **11** in 95% yield. Reduction of **11** with NaBH<sub>4</sub> in tetrahydrofuran produced a 1:1 mixture of the diastereomeric alcohols **10**<sup>7k,m</sup> (89% yield), from which one isomer (mp 202–204 °C) was separated by recrystallization. The infrared (IR) spectrum of this isomer in CHCl<sub>3</sub> at 0.004 M concentration indicated the presence of a *trans*-quinolizidine ring<sup>11</sup>) as well as the absence of intramolecular hydrogen bonding between the hydroxy group and N(5), suggesting the hydrogen at C(12b) and the side chain at C(3) to be axial and equatorial, respectively, and hence the same in the methyl ketone **11**. Judging from this IR spectroscopic feature and the melting point, it seemed to be the same diastereomer as the one (mp 199–200 °C) reported by Winterfeldt's group.<sup>12</sup>) Finally, the correctness of the whole structure of **11** was confirmed by a direct comparison with an authentic sample.<sup>7k,12a)</sup>

Since the methyl ketone **11** has been converted into (±)-deplancheine (**12**) by Pakrashi's group<sup>7k)</sup> through the *p*-tosylhydrazone formation followed by thermolysis with sodium methoxide (Chart 2), the above synthesis of **11** from **5** through **6**, **7**, and **8** is tantamount to a new formal racemic synthesis of this alkaloid. It should be emphasized that the present study has confirmed the applicability of the "3-acetylpyridine route",<sup>13)</sup> originally designed for the racemic syntheses of all of the benzo[*a*]quinolizidine-type *Alangium* alkaloids, to the syntheses of structurally related indoloquinolizidine alkaloids.

## Experimental

**General Notes** All melting points were taken on a Yamato MP-1 capillary melting point apparatus and are corrected. Spectra reported herein were recorded on a JASCO A-202 IR spectrophotometer, a Hitachi M-80 mass spectrometer, or a JEOL JNM-FX-100 nuclear magnetic resonance (NMR) spectrometer, equipped with a <sup>13</sup>C Fourier transform NMR system, at 25 °C with Me<sub>4</sub>Si as an internal standard. Elemental analyses were performed by Mr. Y. Itatani and his associates at Kanazawa University. The following abbreviations are used: br = broad, d = doublet, m = multiplet, s = singlet.

(±)-3-[2-[3-(1,1-Ethylenedioxyethyl)-1-piperidinyl]ethyl]-1H-indole (**7**) A solution of 3-(1,1-ethylenedioxyethyl)pyridine (**5**)<sup>14)</sup> (6.61 g, 40 mmol) and 2-(3-indolyl)ethyl bromide<sup>15)</sup> (9.41 g, 42 mmol) in HCONMe<sub>2</sub> (40 ml) was stirred at 80–85 °C for 50 h. The solvent was removed by evaporation under reduced pressure, and the residual oil was dissolved in H<sub>2</sub>O (80 ml). The aqueous solution was washed with benzene and concentrated to dryness *in vacuo* to leave **6** as a brown glass, which was dissolved in H<sub>2</sub>O (100 ml). The resulting aqueous solution was hydrogenated over Adams catalyst (300 mg) at 35 °C and atmospheric pressure for 21 h, and EtOH (20 ml) was added to the reaction mixture in order to dissolve the precipitate that deposited during this period. The hydrogenation was then continued for a further 9 h. The catalyst was removed by filtration and washed with H<sub>2</sub>O (30 ml). The filtrate and washings were combined, made alkaline with 10% aqueous NaOH, and extracted with CHCl<sub>3</sub>. The combined CHCl<sub>3</sub> extracts were washed with saturated aqueous NaCl, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated to dryness *in vacuo*, leaving a pale brown glass. Crystallization of the glass from benzene-hexane (1:1, v/v) gave **7** (9.80 g, 78% from **5**) as slightly yellowish prisms. Recrystallization in a similar manner provided an analytical sample as colorless prisms, mp 118–119 °C; MS *m/z*: 314 (M<sup>+</sup>); IR  $\nu_{\text{max}}^{\text{CHCl}_3}$  3500 cm<sup>-1</sup> (NH); <sup>1</sup>H-NMR (CDCl<sub>3</sub>)  $\delta$ : 1.28 (3H, s, Me), 1.6–2.1 (7H, m, two CH<sub>2</sub>'s, CH, and ArCH<sub>2</sub>), 2.6–3.3 (6H, m, three NCH<sub>2</sub>'s), 3.8–4.0 (4H, m, two OCH<sub>2</sub>'s), 6.95–7.7 (5H, m, aromatic protons), 8.02 (1H, br, NH). *Anal.* Calcd for C<sub>19</sub>H<sub>26</sub>N<sub>2</sub>O<sub>2</sub>: C, 72.58; H, 8.33; N, 8.91. Found: C, 72.47; H, 8.46; N, 8.76.

(±)-3 $\alpha$ -(1,1-Ethylenedioxyethyl)-1,2,3,4,6,7,12,12b $\alpha$ -octahydroindolo-

[2,3-*a*]quinolizine (**8**) A stirred mixture of **7** (3.14 g, 10 mmol) and 0.1 M aqueous Hg(OAc)<sub>2</sub>-EDTA·2Na·2H<sub>2</sub>O [1:1 (molar ratio)] (300 ml) in EtOH (150 ml) was heated under reflux for 3 h. After cooling, the mixture was made alkaline (pH 9) with 10% aqueous NH<sub>3</sub> and stirred, after addition of NaBH<sub>4</sub> (3.78 g, 100 mmol), at room temperature for 4 h. The reaction mixture was shaken with CHCl<sub>3</sub> (80 ml), and an insoluble substance was removed from the mixture by filtration and washed with CHCl<sub>3</sub>. The aqueous layer of the filtrate was separated from the CHCl<sub>3</sub> layer and extracted with CHCl<sub>3</sub>. The CHCl<sub>3</sub> washings and extracts were combined, washed with saturated aqueous NaCl, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated *in vacuo* to leave a dark brown oil. The oil was passed through an alumina (50 g) column using CHCl<sub>3</sub> as the eluent, and the eluate was concentrated *in vacuo* to give a brown glass. Crystallization of the glass from MeOH yielded a first crop (1.50 g) of **8** as slightly yellowish prisms. The mother liquor of this crystallization was concentrated *in vacuo*, and purification of the resulting brown glass by column chromatography [alumina, CHCl<sub>3</sub>-hexane (2:1, v/v)], followed by crystallization from MeOH, gave a second crop (0.52 g) of **8**. The total yield was 2.02 g (65%). Recrystallization of crude **8** from MeOH furnished an analytical sample as colorless prisms, mp 206–207 °C; MS *m/z*: 312 (M<sup>+</sup>); IR  $\nu_{\text{max}}^{\text{CHCl}_3}$  cm<sup>-1</sup>: 3490 (NH), 2820, 2770 (*trans*-quinolizidine ring<sup>11</sup>); <sup>1</sup>H-NMR (CDCl<sub>3</sub>)  $\delta$ : 1.31 (3H, s, Me), 1.3–3.3 (12H, m, quinolizidine protons), 3.8–4.0 (4H, m, two OCH<sub>2</sub>'s), 7.0–7.5 (4H, m, aromatic protons), 7.72 (1H, br, NH). *Anal.* Calcd for C<sub>19</sub>H<sub>24</sub>N<sub>2</sub>O<sub>2</sub>: C, 73.05; H, 7.74; N, 8.97. Found: C, 73.04; H, 7.69; N, 8.97.

*cis*-(±)-1-(1,2,3,4,6,7,12,12b-Octahydroindolo[2,3-*a*]quinolizine-3-yl)ethanone (**11**) A mixture of **8** (1.87 g, 6 mmol) and 1N aqueous HCl (20 ml) in EtOH (40 ml) was heated under reflux for 2 h. The reaction mixture was concentrated *in vacuo*, and the residue was partitioned by extraction with a mixture of 10% aqueous Na<sub>2</sub>CO<sub>3</sub> (30 ml) and CHCl<sub>3</sub>. The CHCl<sub>3</sub> extracts were washed with saturated aqueous NaCl, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated to dryness *in vacuo* to leave a pale orange glass. Crystallization of the glass from AcOEt gave **11** (1.12 g) as colorless pillars. The mother liquor of this crystallization was concentrated *in vacuo*, and the residue was purified by column chromatography [silica gel, CH<sub>2</sub>Cl<sub>2</sub>-MeOH (40:1, v/v)] to yield a second crop (0.40 g) of **11**. The total yield was 1.52 g (95%). Recrystallization of crude **11** from AcOEt and drying over P<sub>2</sub>O<sub>5</sub> at 2 mmHg and 60 °C for 10 h afforded an analytical sample as colorless pillars, mp 155–157 °C<sup>16)</sup>; MS *m/z* (relative intensity): 268 (M<sup>+</sup>) (98), 267 (90), 239 (22), 225 (59), 223 (17), 197 (37), 184 (61), 170 (100), 169 (77), 168 (22), 156 (25); IR  $\nu_{\text{max}}^{\text{KBr}}$  cm<sup>-1</sup>: 3485 (NH), 2825, 2770 (*trans*-quinolizidine ring<sup>11</sup>), 1693 (CO); <sup>1</sup>H-NMR (Me<sub>2</sub>SO-*d*<sub>6</sub>)  $\delta$ : 1.3–1.55 and 2.0–3.4 (2H and 10H, m each, quinolizidine protons), 2.15 (3H, s, COMe), 6.8–7.1 and 7.2–7.4 (2H each, m, aromatic protons), 10.72 (1H, br, NH). *Anal.* Calcd for C<sub>17</sub>H<sub>20</sub>N<sub>2</sub>O: C, 76.09; H, 7.51; N, 10.44. Found: C, 76.08; H, 7.35; N, 10.31. This sample was identical [by comparison of the <sup>1</sup>H-NMR and mass spectra and thin-layer chromatographic mobility [on a silica gel plate with CH<sub>2</sub>Cl<sub>2</sub>-MeOH (20:1, v/v), CHCl<sub>3</sub>-MeOH (10:1, v/v), AcOEt-EtOH (20:1, v/v), or Et<sub>2</sub>O-EtOH (20:1, v/v)] with that (mp 146 °C)<sup>12b)</sup> sent by Prof. Winterfeldt or the one (mp 128 °C)<sup>7k)</sup> from Dr. Pakrashi.<sup>17)</sup>

[3 $\alpha$ (R\*),12b $\alpha$ ]-(-) and [3 $\alpha$ (S\*),12b $\alpha$ ]-(-)-1,2,3,4,6,7,12,12b-Octahydro- $\alpha$ -methylindolo[2,3-*a*]quinolizine-3-methanols (**10**) A solution of **11** (537 mg, 2 mmol) in tetrahydrofuran (20 ml) was stirred under ice-cooling, and NaBH<sub>4</sub> (76 mg, 2 mmol) was added portionwise in 10 min. After having been stirred at room temperature for 1.5 h, the reaction mixture was poured into H<sub>2</sub>O (30 ml), and the aqueous mixture was extracted with CHCl<sub>3</sub>. The CHCl<sub>3</sub> extracts were washed with saturated aqueous NaCl, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated *in vacuo* to leave a slightly yellowish glass. Purification of the glass by means of column chromatography [silica gel, CH<sub>2</sub>Cl<sub>2</sub>-MeOH (15:1, v/v)] gave **10** (480 mg, 89%) as a colorless solid [mp 180–185 °C (lit. mp 159–160 °C<sup>7k)</sup>; mp 192–194 °C<sup>7m)</sup>], which was presumed to be a 1:1 mixture of two diastereomers on the basis of its <sup>1</sup>H-NMR spectrum in Me<sub>2</sub>SO-*d*<sub>6</sub>. Recrystallization of the solid from EtOH and drying over P<sub>2</sub>O<sub>5</sub> at 2 mmHg and 70 °C for 8 h yielded a pure sample of one diastereomer as colorless minute needles, mp 202–204 °C (lit. <sup>12b)</sup> mp 199–200 °C); MS *m/z* (relative intensity): 271 (M<sup>+</sup>+1) (15), 270 (M<sup>+</sup>) (83), 269 (100), 225 (33), 197 (9), 184 (14), 171 (10), 170 (33), 169 (29), 156 (13), 144 (11); IR  $\nu_{\text{max}}^{\text{KBr}}$  cm<sup>-1</sup>: 3540 (NH), 3420–3280 (OH), 2815, 2770 (*trans*-quinolizidine ring<sup>11</sup>);  $\nu_{\text{max}}^{\text{CHCl}_3}$  (at 0.004 M concentration) cm<sup>-1</sup>: 3620 (OH), 3490 (NH), 2810, 2760 (*trans*-quinolizidine ring<sup>11</sup>); <sup>1</sup>H-NMR (Me<sub>2</sub>SO-*d*<sub>6</sub>)  $\delta$ : 1.06 [3H, d, *J* = 6.3 Hz, CH(OH)Me], 1.1–3.1 (12H, m, quinolizidine protons), 3.3–3.6 [1H, m, CH(OH)Me], 4.38 (1H, d, *J* = 4.9 Hz, OH), 6.8–7.1 and 7.15–7.4 (2H each, m, aromatic protons), 10.67 (1H, s, NH); <sup>13</sup>C-NMR (Me<sub>2</sub>SO-*d*<sub>6</sub>)

$\delta$ : 20.7 [CH(OH)Me], 21.5 [C(7)], 26.0 [C(2)], 29.1 [C(1)], 43.1 [C(3)], 53.1 [C(6)], 57.7 [C(4)], 60.0 [C(12b)], 68.0 [CH(OH)Me], 106.0 [C(7a)], 110.8 [C(11)], 117.2 [C(8)], 118.1 [C(9)],<sup>18</sup> 120.1 [C(10)],<sup>18</sup> 126.5 [C(7b)], 135.7 [C(11a) or C(12a)], 135.9 [C(12a) or C(11a)]. Anal. Calcd for C<sub>17</sub>H<sub>22</sub>N<sub>2</sub>O: C, 75.52; H, 8.20; N, 10.36. Found: C, 75.46; H, 8.34; N, 10.29.

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- 16) When crystallized out of a considerably dilute solution, 11 sometimes gave colorless minute needles of almost the same melting point (mp 156–157 °C) but with an IR spectrum not completely identical in the solid state, suggesting the existence of another crystal form.
- 17) The difference of these three samples in melting point is most likely due to polymorphism of 11. According to a private communication from Prof. E. Winterfeldt (Hannover, West Germany), his group of workers have also observed a higher melting point (mp 151 °C) for their later preparations. The IR spectrum (in KBr) of our sample described in footnote 16 was superimposable on those of the two authentic specimens.
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