1 H, H-4), 2.1 (m, 1 H, H-3_{syn}), 2.0 (s, 3 H, CH_3), 1.4 (ddd, 1 H, H-3_{anti}, $J_{3_{syn},3_{anti}}$ = 13 Hz, $J_{3_{anti},2} = J_{3_{anti},4}$ = 3 Hz). Anal. Calcd for $C_{14}H_{14}O_2$: C, 78.47; H, 6.60. Found: C, 78.59; H, 6.75.

Irradiations. The procedures for irradiation of all of the substrates were substantially identical. For the direct irradiation, approximately 50 mg of substrate was dissolved in 2.0 mL of acetonitrile-d_3 or acetic acid in a quartz NMR tube (5-mm i.d.). The solutions were purged with argon for 10 min. Irradiations were carried out in Rayonet–Srinivasin–Griffin photoreactors equipped with 254-nm lamps. The process and extent of photoreaction were monitored by ^1H NMR and the photoproducts isolated by preparative HPLC.

The procedure for sensitized irradiation was similar to that in acetonitrile, except that one-third of the solvent was acetone-d_6 used as a sensitizer, Pyrex NMR tubes were used, and 300-nm lamps were used in the photoreactor.

In all cases (except the chloride 8-Cl), starting with esters of 8-OH or 10-OH, only tricyclic isomers **9** and **11** were produced, even though reactions in some cases were carried out to complete disappearance of starting material. The properties of new compounds are given below.

***anti*-3,4-Benzotricyclo[3.2.1.$0^{2,7}$]oct-3-en-6-ol benzoate (9-OBz)**: mp 58–59 °C; ^1H NMR ($CDCl_3$) δ 7.7 (m, 9 H, aromatic and benzoate H), 4.8 (s, 1 H, H-6), 3.2 (d, 1 H, H-5, $J_{5,8_{anti}}$ = 5 Hz), 2.4 (ddd, 1 H, H-8_{anti}, $J_{8_{anti},8_{syn}}$ = 12 Hz, $J_{8_{anti},5}$ = 5 Hz, $J_{8_{anti},1}$ = 3 Hz), 2.3 (dd, 1 H, H-2, $J_{2,1} = J_{2,7}$ = 7 Hz), 1.9 (m, 2 H, H-1 and H-7), 1.1 (d, 1 H, H-8_{syn}, $J_{8_{syn},8_{anti}}$ = 12 Hz).

Anal. Calcd for $C_{19}H_{16}O_2$: C, 82.57; H, 5.85. Found: C, 82.53, H, 5.73.

***anti*-3,4-Benzotricyclo[3.2.1.$0^{2,7}$]oct-3-en-6-ol *p*-(trifluoromethyl)benzoate (9-OTfb)**: ^1H NMR ($CDCl_3$) δ 7.9 (m, 4 H, benzoate H), 7.1 (m, 4 H, aromatic H), 4.8 (s, 1 H, H-6), 3.2 (d, 1 H, H-5, $J_{5,8_{anti}}$ = 5 Hz), 2.3 (ddd, 1 H, H-8_{anti}, $J_{H\text{-}8_{anti},H\text{-}8_{syn}}$ = 12 Hz, $J_{8_{anti},5}$ = 5 Hz, $J_{8_{anti},1}$ = 3 Hz), 2.3 (dd, 1 H, H-2, $J_{2,1} = J_{2,7}$ = 7 Hz), 1.9 (m, 2 H, H-1 and H-7), 1.1 (d, 1 H, H-8_{syn}, $J_{8_{syn},8_{anti}}$ = 12 Hz). Anal. Calcd for $C_{20}H_{15}O_2F_3$: C, 69.75; H, 4.40. Found: C, 69.77; H, 4.64.

***syn*-3,4-Benzotricyclo[3.2.1.$0^{2,7}$]oct-3-en-6-ol benzoate 11-OBz**: mp 108–109 °C; ^1H NMR (CD_3CN) δ 7.4 (m, 9 H, aromatic and benzoate H), 5.5 (dd, H-6, $J_{6,5}$ = 5 Hz, $J_{6,7}$ = 3 Hz), 3.6 (dd, 1 H, H-5, $J_{5,6} = J_{5,8}$ = 5 Hz), 2.4 (dd, 1 H, H-2, $J_{2,7} = J_{2,1}$ = 7 Hz), 2.0 (m, 3 H, H-1, H-7 and H-8_{anti}), 1.1 (d, 1 H, H-8_{syn}, $J_{8_{syn},8_{anti}}$ = 12 Hz). Anal. Calcd for $C_{19}H_{16}O_2$: C, 82.57; H, 5.85. Found: C, 82.42; H, 6.00.

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Registry No. 1-OH, 54647-01-7; 2-OH, 64626-01-3; 8-OH, 54647-02-8; 8-OBz, 83511-40-4; 8-OTfb, 83511-41-5; 8-OAc, 16938-95-7; 9-OBz, 83511-43-7; 9-OTfb, 83527-58-6; 10-OH, 57089-48-2; 10-OBz, 83542-14-7; 11-OBz, 83542-15-8; 12-OAc, 83511-42-6.

New Synthesis of Azabufalin from C-17 Steroidal Ketones

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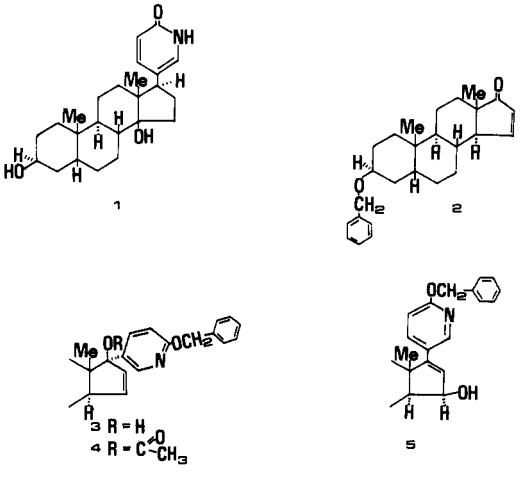
A new and superior method for the conversion of the appropriate steroidal pyridyl derivatives into azabufalin by using the known 2-(benzyloxy)pyridyl bromide at the 17-ketone is reported.

Recent studies[1,2] on synthetic cardioactive steroid analogues have shown that azabufalin (**1**) may have important use pharmacological activity. In previous work,[2,3] we demonstrated that azabufalin can be synthesized by a method which features the reaction of a steroidal ketone with 2-methoxypyridyllithium and the conversion of the pyridyl ring into a pyridone derivative. By using Wiesner's synthetic strategy[4] one can obtain the substitution and natural configuration at C-14. Our previous method[2] used 2-methoxy-5-bromopyridine in the reaction at the 17-ketone. However, the transformation of a methoxypyridyl derivative into an *N*-benzylpyridone derivative by using benzyl bromide gave only a 52% yield, with 40% of the starting material recovered. Even after recycling several times, this troublesome step gave a total yield of only 70%. The risk of formation of a 14,15-unsaturated elimination product, due to the formation of hydrogen bromide in the reaction, further complicated this synthetic route. In this paper a new and superior method for the conversion of the appropriate steroidal pyridyl derivative to azabufalin, using the known 2-(benzyloxy)pyridyl bromide,[5] is reported.

Results and Discussion

The α,β-unsaturated ketone **2**, synthesized by Wiesner's group,[4] has been successfully used for the total synthesis of cardenolide and isocardenolide compounds. Condensation of compound **2** with 5-bromo-2-(benzyloxy)pyridine



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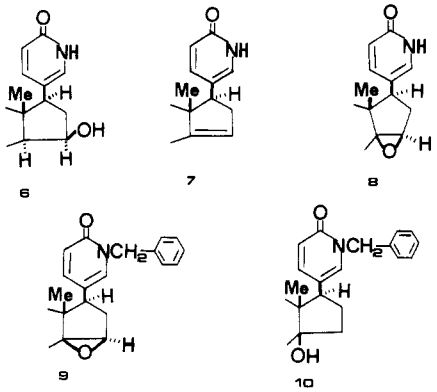
in an ethereal solution of *n*-butyllithium at −70 °C furnished the carbinol **3** in a yield of 88%.[6] Acetylation gave compound **4** in a yield of 93%. The presence of the acetyl group was indicated by the absorption band at 1735 cm^{-1} in the IR spectrum (in $CHCl_3$). Next, the rearrangement was promoted by $CaCO_3$ in aqueous solution to give an 85% yield of allylic alcohol **5**. Its IR spectrum showed the disappearance of the carbonyl band and the presence of a 3605-cm^{-1} band attributed to hydroxyl. The ^1H NMR spectrum exhibited a broad singlet for the C-15α proton at δ 4.53 and a doublet at δ 5.97 for the C-16 vinyl proton. The assignment of a β configuration for the hydroxyl group at C-15 was based on the assumption of an S_N2' mechanism for this rearrangement.[2,4]

Upon hydrogenation over palladium on $CaCO_3$, compound **5** gave in 98% yield the pyridone **6**, resulting from



reduction of the olefin at C-16,17 and selective cleavage of the *O*-benzyl group. In the IR spectrum a band appeared at 1661 cm^{-1}, characteristic of a C(O)NH group, the UV spectrum in MeOH solution had two bands at 231 and 306 nm, and the ^1H NMR spectrum verified the disappearance of the vinyl hydrogen. The alcoholic compound **6** was dehydrated with methanesulfonyl chloride–pyridine (at 60 °C for 4 h) to give in 85% yield the 14,15-unsaturated compound **7**, which, upon epoxidation by the NBA method,[7] afforded the 14β,15β-epoxide **8** in a yield of 72%. Direct reduction of this epoxide gave a poor yield of the desired product, due to the facile reduction of the pyridone carbonyl group. N-Alkylation of the pyridone **8**, by using benzyl bromide and potassium carbonate in 1,2-dimethoxyethane, gave the *N*-benzylpyridone **9** in 85% yield. Reduction of compound **9** with lithium aluminum hydride[8] at −65 °C for 1 h afforded in 80% yield compound **10**, which was shown to be identical with the compound obtained by the previous method[2] on the basis of spectroscopic evidence (NMR, IR) and its melting point. Hydrogenolysis of compound **10** over palladium on charcoal in dioxane–ethanol (1:2) raised the yield of the azabufalin **1** to 71% [the previous yield by reduction in dioxane–ethanol (1:1) was 58%]. Thus, the overall yield of azabufalin (**1**) from the α,β-unsaturated ketone **2** amounted to 21%, compared to 13.6% by the previous method.[2]

Experimental Section

Melting points were determined on a hot-stage apparatus and were uncorrected. The ^1H NMR spectra were taken with a Varian EM-360 spectrometer with Me_4Si as an internal standard and $CDCl_3$ as the solvent, unless otherwise indicated. IR spectra were recorded on a Perkin-Elmer 682 spectrophotometer in $CHCl_3$ solution, unless otherwise specified. The ultraviolet spectra in absolute methanol were recorded on a Perkin-Elmer spectrometer, Model 552. Silica gel G (80 and 100 mesh; E. Merck A G, Darmstadt) was used for column chromatography with ether–hexane, $CHCl_3$–MeOH, or AcOEt–MeOH as eluents. The elemental analyses were performed by Dr. F. Pascher, Mikroanalytisches Laboratorium, Bonn, West Germany.

3β-(Benzyloxy)-17β-hydroxy-17α-[2-(benzyloxy)-5β-pyridyl]-5β-androst-15-ene (3). *n*-Butyllithium (1.3 mL, 2.2 M solution in hexane) was added to a stirred solution of 2-(benzyloxy)-5-bromopyridine (0.79 g, 3 mmol) in absolute ether (30 mL) at −70 °C and the mixture was stirred for 1 h. An etheral solution (40 mL) of the α,β-unsaturated ketone **2**[4] (0.57 g, 1.5 mmol) was then added dropwise and the solution was stirred for 30 min at the same temperature. The excess reagent was destroyed by slow addition of water followed by washing with 5% citric acid and a 5% sodium bicarbonate solution and water, by drying ($MgSO_4$), and by evaporation to dryness. Recrystallization from hexane yielded 0.78 g (88%) of **3**: mp 65–67 °C; IR 3602 cm^{-1} (OH); ^1H NMR δ 0.97 (3 H, s, 18-Me), 1.05 (3 H, s, 19-Me), 3.73 (1 H, br s, 3α-H), 4.42 (2 H, s, benzylic protons), 5.35 (2 H, s, pyridyl benzylic protons), 5.7 (1 H, d, J = 6 Hz, 16-H), 6.2 (1 H, d, J = 6 Hz, 15-H), 6.78 (1 H, d, J = 8 Hz, C_{23} H), 7.32 (10 aromatic ring protons), 7.62 (1 H, ABq, $J_{21,22}$ = 2 Hz, $J_{22,23}$ = 8 Hz, C_{23} H), 7.98 (1 H, d, J = 2 Hz, C_{21} H). Anal. Calcd for $C_{38}H_{45}O_3N$ (mol wt 563.8): C, 80.95; H, 8.05; O, 8.51; N, 2.48. Found: C, 80.65; H, 8.05; O, 8.45; N, 2.34.

3β-(Benzyloxy)-17β-acetoxy-17α-[2-(benzyloxy)-5-pyridyl]-5β-androst-15-ene (4). Carbinol **3** (5.63 g, 0.01 mmol) was acetylated with acetic anhydride (5 mL) in pyridine (10 mL) in the presence of a catalytic amount of 4-(dimethylamino)pyridine (11 mg at room temperature for 6 h. The reaction mixture was evaporated at 50 °C in vacuo to dryness, and the residue was redissolved in ether, washed with 5% $NaHCO_3$ and then with water (several times), dried, and reduced to dryness in vacuo to give 5.6 g (93%) of **4**: mp 67–69 °C (crystallized from methanol); IR 1735 cm^{-1} (>C=O); ^1H NMR δ 0.98 (3 H, s, 18-Me), 1.11 (3 H, s, 19-Me), 2.03 (3 H, s, COMe), 3.65 (1 H, br s, 3α-H), 4.22 (2 H, s, benzylic protons), 5.32 (2 H, s, pyridyl benzylic protons), 6.33 (2 H, q, J = 6 Hz, 15-H and 16-H), 6.73 (1 H, d, J = 8 Hz, C_{23} H), 7.31 (11 H, m, 10 aromatic ring protons and C_{22} H), 7.93 (1 H, d, J = 2 Hz, C_{21} H).

3β-(Benzyloxy)-15β-hydroxy-17-[2-(benzyloxy)-5-pyridyl]-5β-androst-16-ene (5). The acetate **4** (6.05 g, 0.01 mol) was refluxed in aqueous acetone (200 mL, 25% H_2O) in the presence of $CaCO_3$ (2 g, 0.02, mol) for 24 h. The mixture was filtered, the filtrate was evaporated under reduced pressure to remove most of the acetone, and the crude product was dissolved in ether, washed with 5% $NaHCO_3$, dried ($MgSO_4$), and evaporated to dryness. The product was purified by column chromatography on silica gel and yielded pure allylic alcohol **5**: 4.7 g (85%); mp 54–56 °C (crystallized from ethanol–hexane); IR 3605 cm^{-1} (OH); ^1H NMR δ 1.05 (3 H, s, 18-Me), 1.32 (3 H, s, 19-Me), 3.7 (1 H, br s, 3α-H), 4.47 (2 H, s, benzylic protons), 4.53 (1 H, br s, 15α-H), 5.37 (2 H, s, pyridyl benzylic protons), 5.97 (1 H, d, J = 3 Hz, vinylic H), 6.73 (1 H, d, J = 8 Hz, C_{23} H), 7.32 (10 aromatic ring protons), 7.68 (1 H, ABq, $J_{21,22}$ = 2 Hz and $J_{22,23}$ = 8 Hz, C_{22} H), 8.2 (1 H, d, J = 2 Hz, C_{21} H).

3β-(Benzyloxy)-15β-hydroxy-17β-(2-hydroxy-5-pyridyl)-5β-androstane (6). The allylic alcohol **5** (4.5 g, 8 mmol) was hydrogenated in ethanol with 10% Pd/$CaCO_3$ (450 mg) at room temperature. The catalyst was removed by filtration through Celite, the filtrate was evaporated in vacuo to yield the product, and the material was purified by preparative TLC to yield, after recrystallization from dichloromethane–ether, 3.68 g (98%) of **6**: mp 255–257 °C; IR 1661 cm^{-1} (C(O)NH); UV λ_{max} 231 nm (ϵ 10.729), 306 (5.085); ^1H NMR ($CDCl_3$–Me_2SO-d_6) δ 0.75 (3 H, s, 18-Me), 0.98 (3 H, s, 19-Me), 3.73 (1 H, br s, 3α-H), 4.49 (2 H, s, benzylic protons), 6.4 (1 H, d, J = 9 Hz, C_{23} H), 7.37 (7 H, m, aromatic protons, C_{21} H and C_{22} H). Anal. Calcd for $C_{31}H_{41}O_3N$ (mol wt 475.6): C, 78.27; H, 8.69; O, 10.09; N, 2.95. Found: C, 78.34; H, 8.49; O, 10.17; N, 2.95.

3β-(Benzyloxy)-17β-(2-hydroxy-5-pyridyl)-5β-androst-14-ene (7). The pyridone hydroxy compound **6** (190 mg, 0.4 mmol) in pyridine (2 mL) was stirred with methanesulfonyl chloride (55

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mg, 0.48 mmol) at 60 °C for 4 h followed by evaporation in vacuo to dryness. The residue was dissolved in ether and washed with 5% $NaHCO_3$, dried ($MgSO_4$), and evaporated to dryness. The crude material was purified by preparative TLC to yield the pure compound: 158 mg (88%); mp 114–116 °C (recrystallized from methanol); IR 1664 cm^{-1} (C(O)NH); ^1H NMR δ 0.66 (3 H, s, 18-Me), 0.97 (3 H, s, 19-Me), 3.73 (1 H, br s, 3α-H), 4.5 (2 H, s, benzylic protons), 5.27 (1 H, br s, 15-H), 6.53 1 H, d, J = 9 Hz, C_{23} H), 7.32 (7 H, m, aromatic protons, C_{21} H and C_{22} H).

3β-(Benzyloxy)-14β,15β-oxido-17β-(2-hydroxy-5-pyridyl)-5β-androstane (8). A mixture of the pyridone compound **7** (91.6 mg, 0.2 mmol) and water (0.3 mL) in acetone (3 mL) was stirred with *N*-bromoacetamide (34.5 mg, 0.25 mmol) at room temperature for a period of 15 min. The reaction mixture was diluted with CH_2Cl_2, washed with 5% Na_2SO_3, dried over anhydrous $MgSO_4$, and evaporated at room temperature in vacuo to dryness. The residue was redissolved in a 1:1 mixture of CH_2Cl_2 and MeOH (20 mL) and stirred at room temperature with aluminum oxide (180 mg) for 4 h. The filtrate was evaporated to dryness. The crude product was purified by TLC to yield pure epoxide **8** as a yellow liquid: 68 mg (72%); IR 1659 cm^{-1} (C(O)NH); ^1H NMR δ 0.68 (3 H, s, 18-Me), 0.96 (3 H, s, 19-Me), 3.5 (1 H, s, 15α-H), 3.7 (1 H, br s, 3α-H), 4.48 (2 H, s, benzylic protons), 6.5 (1 H, d, J = 9 Hz, C_{23} H), 7.4 (7 H, m, aromatic protons, C_{21} H and C_{22} H).

3β-(Benzyloxy)-14β,15β-oxido-17β-(1-benzyl-2-hydroxy-5-pyridyl)-5β-androstane (9). To a solution of compound **8** (95 mg, 0.2 mmol) in 1,2-dimethoxyethane were added potassium carbonate (85 mg, 0.61 mmol) and benzyl bromide (36 mg, 0.21 mmol). The reaction mixture was refluxed overnight and then filtered, and the filtrate was evaporated to dryness. The crude product was purified by preparative TLC to give pure *N*-benzyl pyridone **9**: 96 mg (85%); mp 104–106 °C (recrystallized from dichloromethane–ether); IR 1662 cm^{-1} (C(O)NH); ^1H NMR δ 0.7 (3 H, s, 18-Me), 0.98 (3 H, s, 19-Me), 3.48 (1 H, s, 15α-H), 3.75 (1 H, br s, 3α-H), 4.48 (2 H, s, benzylic protons), 5.12 (2 H, s, N-benzylic protons), 6.5 (1 H, d, J = 9 HZ, C_{23} H), 7.15 (1 H, d, J = 2 Hz, C_{21} H), 7.3 (10 aromatic ring protons), 7.53 (1 H, ABq, $J_{21,22}$ = 2 Hz, $J_{22,23}$ = 9 Hz, C_{22} H). Anal. Calcd for $C_{38}H_{45}O_3N$ (mol. wt 563): C, 80.95; H, 8.05; O, 8.51; N, 2.48. Found: C, 80.91; H, 8.16; O, 8.42; N, 2.4.

3β-(Benzyloxy)-14β-hydroxy-17β-(1-benzyl-2-hydroxy-5-pyridyl)-5β-androstane (10). The *N*-benzylpyridone compound **9** (113 mg, 0.2 mmole) was dissolved in dry THF (10 mL) and lithium aluminum hydride (228 mg, 0.6 mmol) was added under a nitrogen stream at −65 °C for 1 h, followed by filtration through Celite. The filtrate was evaporated to dryness. The crude product was purified by preparative TLC and yielded 90 mg (80%) of pure compound **10** (mp 256–257 °C) which was shown to be identical with a sample prepared earlier[2] as based on NMR, IR, and UV spectral evidence and melting point.

Acknowledgment. The financial support of this work from Tunghai university and a grant from National Research Council of the Republic of China are gratefully acknowledged. I thank Dr. Teh-Chang Chou from this Department for helpful discussions.

Registry No. **1**, 81072-39-1; **2**, 73336-58-0; **3**, 83664-27-1; **4**, 83664-28-2; **5**, 83664-29-3; **6**, 83664-30-6; **7**, 83664-31-7; **8**, 83664-32-8; **9**, 83681-20-3; **10**, 81072-38-0; 2-(benzyloxy)-5-bromopyridine, 83664-33-9.

Use of Tellurium(IV) and Tellurium(VI) as Oxidants in Organic Synthesis[49]

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The oxidizing properties of TeO_2, $Te(OH)_6$, and TeO_3 in acetic acid solution containing LiBr have been explored. It was found that certain aromatic compounds were acetoxymethylated by the action of TeO_2 or, when especially activated, converted into diarylmethane derivatives. $Te(OH)_6$ and TeO_3, in contrast, mainly effected side-chain acetoxylation, as was also the case with SeO_2. In the acetoxymethylation reaction TeO_2 apparently slowly oxidized the solvent, HOAc, to a reactive species of some kind, e.g., acetoxycarbene, which attacked the aromatic compound. In the side-chain acetoxylations, Te(VI) oxidized bromide ions to Br_2, which caused benzylic bromination. The solvolysis of benzylic bromides to acetates was significantly enhanced by the presence of Te(IV) species. Both TeO_2 and TeO_3 effected more conventional oxidations like the transformation of deoxybenzil to benzil. Benzoin acetate is a probable intermediate in this oxidation.

The use of TeO_2 as an oxidant in organic synthesis was tentatively explored as early as the 1940's.[1] The results, however, were not especially encouraging due to the very low solubility of tellurium dioxide in almost all organic

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