material, and GC analysis confirmed significant benzophenone production. The dichloromethane layer, after washing with water, evaporation, and crystallization from petroleum ether (bp 60–80 °C), yielded 1.8 g of a low-melting solid. Further crystallization from methanol/water produced 0.8 g (46% yield) of benzophenone, mp 47–48 °C (lit.[10]a mp 48 °C). Its identity was confirmed by infrared spectroscopy[11] and the 2,4-dinitrophenylhydrazone derivative, mp 238–239 °C (lit.[10a] mp 238 °C).

Carbon dioxide (as $BaCO_3$), accounting for an approximately 20% yield based on loss of one phenyl group, was isolated from 4-h, biphasic reactions of **5a**. For these reactions, hypochlorite solutions containing tetra-*n*-butylammonium hydroxide as catalyst were saturated with $Ba(OH)_2$, and the $BaCO_3$ formed from CO_2 in the bleach was filtered prior to use.

Oxidation of 1,1-Diphenylethanol (5b). The alcohol (1.0 g, 0.005 mol) and 0.11 g of TBAHS in 50 mL of dichloromethane were stirred magnetically with 100 mL of aqueous hypochlorite set at pH 9.5. After 17 h, the pH had dropped to 8.2, and GC analysis indicated almost complete consumption of the alcohol. After raising the pH to 12 and stirring with the dichloromethane layer, the aqueous hypochlorite layer was separated. Excess hypochlorite was decomposed by the addition of powdered sodium bisulfite until the pH was reduced to 5. (The solution warmed, and considerable effervescence occurred.) Upon lowering the pH to 1 with 6 M HCl, 0.16 g of a white precipitate, mp 119–120 °C (lit.[10]b mp for benzoic acid 122 °C), was isolated. Diethyl ether extracts of the aqueous solution provided an additional 0.17 g of crude benzoic acid for a total yield of approximately 50%.

Acetophenone and benzophenone were detected (10–31% and 3–7%, respectively) by GC and LC analysis of 2–4-h, phase transfer catalyzed reactions of **5b** with hypochlorite. Acidification of 3.5-h reaction mixtures blanketed and flushed with nitrogen resulted in isolation of carbon dioxide, as $BaCO_3$ from $Ba(OH)_2$ traps, in yields of approximately 40%, based on loss of one phenyl group.

Oxidation of 2-Phenyl-2-propanol (5c). Procedures employed strictly were analogous to those used in phase transfer catalyzed hypochlorite reactions of alcohol **5b**. LC and GC analyses showed alcohol **5c** to be converted to >50% yield to acetophenone in 4 h. Benzoic acid was isolated in 27–37% yield upon acidification of reaction mixtures maintained at pH 8–9 for 24 h.

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Registry No. **2**, 611-73-4; **4a**, 595-91-5; **4b**, 5558-66-7; **5a**, 76-84-6; **5b**, 599-67-7; **5c**, 617-94-7; **6a**, 93-99-2; **6b**, 98-86-2; **7**, 117-34-0; benzoic acid, 65-85-0.

(10) (a) Pavia, D. L.; Lampman, G. M.; Kritz, G. S. *Introduction to Organic Laboratory Techniques*, 2nd ed.; Saunders: Philadelphia, 1982; p 637; (b) *Ibid.* p 638.

(11) *Sadtler Standard Spectra*, Midget Edition; Sadtler Research Laboratories: Philadelphia, 1962; Spectrum No. 14800.

Derivatives of the Thebaine Anion. 2. 5-Methylmorphine, 5-Methylcodeine, 5-Methylheroin, and Some Related Compounds

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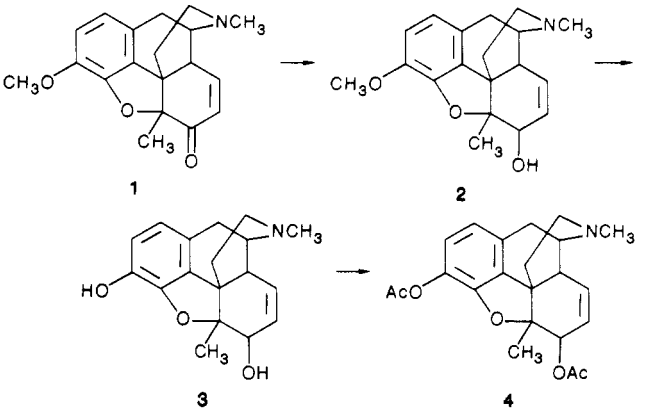
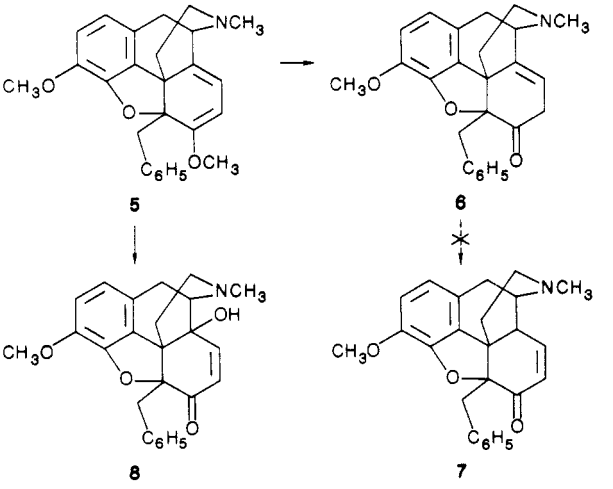
Simple direct routes to 5-methylmorphine, 5-methylcodeine, and 5-methylheroin from the recently described[1] thebaine anion are herein described.

(1) Boden, R. M.; Gates, M.; Ho, S. P.; Sundararaman, P. *J. Org. Chem.* **1982**, *47*, 1347.

5-Methylcodeinone (**1**)[1] on reduction with sodium borohydride[2] gives 5-methylcodeine (**2**) in high yield. Cleavage of the phenolic methoxyl group of **2** with sodium ethanethiolate gives 5-methylmorphine (**3**) likewise in excellent yield, and acetylation of **3** provides 5-methyldiacetylmorphine (5-methylheroin) (**4**). That **2**, **3**, and **4** have the natural codeine configuration at C_6 was shown conclusively by an X-ray crystallographic structure determination carried out on **4** (5-methylheroin) by Professor William D. Jones of these laboratories.[3]



The alkylation of the thebaine anion with benzyl chloride yields 5-benzylthebaine (**5**), but application to **5** of the procedure of Dauben, Baskin, and von Riel[4] for the conversion of thebaine to codeinone yields only the β,γ-unsaturated ketone, 5-benzylneopinone (**6**) and all attempts to isomerize this to the α,β-isomer, 5-benzylcodeinone (**7**),



failed and with it our projected preparation of 5-benzylmorphine. 5-Benzylthebaine is, however, readily converted into 5-benzyl-14-hydroxycodeinone (**8**) with hydrogen peroxide in formic acid. 5-Carbethoxythebaine (**9**), prepared by the action of ethyl chloroformate on the thebaine anion, is readily transformed into 5-carbethoxycodeinone (**10**) from which 5-(hydroxymethyl)codeine (**11**) is produced by reduction with sodium borohydride.

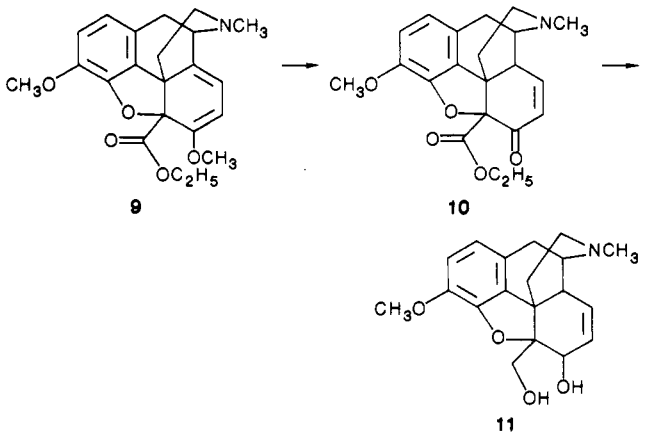
The results of screening for antinociceptive activity are tabulated in Table I. These tests were carried out for us

(2) Gates, M. *J. Am. Chem. Soc.* **1953**, *75*, 4340.

(3) A 3-dimensional ORTEP drawing of 5-methylheroin (**4**), tables of fractional atomic coordinates, anisotropic thermal parameters, bond angles and distances, and observed and calculated structure factors will be made available on request.

(4) Dauben, W. G.; Baskin, C. P.; van Riel, H. C. A. *J. Org. Chem.* **1979**, *44*, 1567.

0022-3263/89/1954-0972$01.50/0



at the National Institute on Drug Abuse through the courtesy of Dr. Arthur Jacobson of the Section on Drug Design and Synthesis.

Experimental Section

NMR spectra were recorded either on a Bruker WH 400 high-resolution pulse Fourier transform spectrometer, a Nicolet GE 300 spectrometer, or a Varian EM 390 spectrometer in $CDCl_3$. Infrared spectra were recorded on a Beckman Acculab B IR spectrometer. Optical rotations were observed on a Rudolph Model 80 polarimeter. Melting points are uncorrected.

Reduction of 5-Methylcodeinone (1) with Sodium Borohydride. 5-Methylcodeine (2). A solution of 5-methylcodeinone[1] (1.36 g, 4.37 mmol) in 40 mL of methanol was treated with 2 g of sodium borohydride and stirred at room temperature for 4 h, concentrated to about half the original volume, and diluted with 25 mL of 10% sodium hydroxide. The clear colorless solution was heated momentarily to boiling, diluted with water, and extracted four times with chloroform. The washed, dried, and filtered chloroform extract on concentration left 1.59 g of foam, the TLC of which showed it to be a mixture of three products. Medium-pressure liquid chromatography on silica gel (MPLC) with chloroform–methanol (9:1) as eluent gave 5-methylcodeine (**2**, 1.11 g, 81%) as a foam. Crystallization from benzene–hexanes gave colorless leaves (1.02 g, 75%), mp 88–96 °C (hydrate). Attempts to obtain anhydrous crystalline material either by crystallization or sublimation at 10^{-4} mm gave crystalline material only after rehydration: IR ν 3450, 2890, 1630, 1600, 1400, 1280, 1100, and 910 cm^{-1}; NMR δ 1.66 (s, 3 H, C_5-Me), 2.43 (s, 3 H, N-Me), 3.80 (s, 3 H, C_3-OCH_3) 5.22 (br d, 1 H, J = 9 Hz, C_7-H), 5.65 (br d, 1 H_1, J = 9 Hz, C_8-H), 6.58 (m, 2 H, aromatic H); ^{13}C NMR δ 46.353, 56.060, 59.335, 71.106, 75.717 (C_5-Me), 76.991 (N-Me), 79.204 (C_3-OMe), 111.995, 119.337, 127.467, 133.355; $[\alpha]^{24}_D$ −60.6° (*c* 0.94, 95% alcohol). Anal. Calcd for $C_{19}H_{23}NO_3 \cdot H_2O$: C, 68.86; H, 7.60; N, 4.23. Found: C, 69.06; H, 7.10; N, 4.13.

The minor fractions (containing two more polar components of very close R_f on TLC) were pooled to give 198 mg (15%) of amorphous material, which appears to be a mixture of the epimers at C_6 of 5-methyldihydrocodeine in the ratio 2:1 resulting from 1,4-hydride addition followed by reduction: IR ν 3590, 2850, 1636, 1610, 1490, 1335, 1150, and 980 cm^{-1}; NMR δ 0.9–3.8 (complex, 23 H, with spikes at 1.5 for C_5–Me, 2.34, 2.43 for N-Me, 3.79, 3.82 for C_3-OMe) 6.62 (m, 2 H, aromatic H).

In another experiment, 104 mg of the crude sodium borohydride reduction product of 5-methylcodeinone was acylated with acetic anhydride in pyridine at room temperature. Processing gave a syrup, preparative TLC separation of which yielded the following three products:

(a) 6-*O*-Acetyl-5-methylcodeine (90 mg, mp 153–154 °C): IR ν 2910, 1730, 1635, 1600, 1440, 1370, 1025, 910, and 870 cm^{-1}; NMR δ 1.67 (s, 3 H, C_5-Me), 2.0 (s, 3 H, OCOMe), 2.3 (s, 3 H, N-Me), 3.83 (s, 3 H, C_3-OMe), 5.1 (m, 1 H, C_6-H), 5.53 (br s, 2 H, C_7- and C_8-H), 6.51, 6.64 (AB, 2 H, J = 8 Hz, aromatic H). Anal. Calcd for $C_{21}H_{25}NO_4$: C, 70.96; H, 7.09; N, 3.94. Found: C, 69.37; H, 7.21; N, 3.74.

(b) 6α-*O*-Acetyl-5-methyldihydrocodeine (9 mg): mp 115–118 °C; IR ν 2930, 1730, 1440, 1120, 1070, and 870 cm^{-1}; NMR δ 1.58 (s, 3 H, C_5-OMe), 1.63 (s, 3 H, 6-*O*-α-acetylmethyl), 2.49 (s, 3 H, N-CH_3), 3.93 (s, 3 H, C_3-OMe), 5.1 (m, 1 H, C_6-H), 6.63 (m, 2 H, aromatic H); ^{13}C NMR δ 20.264, 21.052, 31.549, 40.649, 42.955.

(c) 6β-*O*-Acetyl-5-methyldihydrocodeine (5 mg): IR ν 1730 cm^{-1}; NMR δ 1.55 (s, 3 H, C_5-Me), 2.05 (s, 3 H, C_6-*O*-acetylmethyl), 2.40 (s, 3 H, N-CH_3), 3.90 (s, 3 H, C_3-OMe), 4.66 (m, 1 H, C_6-H), 6.53 (m, 2 H, aromatic H).

5-Methylmorphine (3). To a suspension of sodium hydride (250 mg, 50% oil dispersion previously washed three times with pentane, 5 mmol) in dry dimethylformamide was added 5-methylcodeine (**2**, 157 mg, 0.5 mmol) followed by ethanethiol (310 mg, 5 mmol) under nitrogen. After being stirred at room temperature for 15 min, the mixture was heated to 100 °C for 15–20 h. After being cooled the reaction mixture was quenched by addition of saturated ammonium chloride solution. Extractive workup with chloroform gave a brown syrup, which on chromatography on silica gel with 10% CH_3OH in $CHCl_3$ gave 135 mg (90%) of hydrated **3**. After crystallization from chloroform–ether it had mp 139–150 °C: IR ν 3580, 3300, 2940, 1630, 1600, 1450, 1130, 1060, 960, 900, and 860 cm^{-1}; NMR δ 1.57 (s, 3 H, C_5-Me), 2.43 (s, 3 H, N-Me), 5.2 (br m, 3 H, collapses into a d, J = 9 Hz, of 1 H intensity on D_2O exchange, 2 exchangeable protons and C_7-H), 5.62 (d, 1 H, J = 9 Hz, C_8-H), 6.42, 6.58 (AB, 2 H, J = 9 Hz, aromatic H); $[\alpha]^{23}_D$ −59° (*c* 0.88, 95% alcohol). Anal. Calcd for $C_{18}H_{21}NO_3 \cdot H_2O$: C, 68.12; H, 7.31; N, 4.41; H_2O, 6.02. Found: C, 68.03; H, 7.27; N, 4.40; H_2O, 6.41. We have not obtained this substance in anhydrous crystalline form, even after sublimation at 10^{-4} mmHg.

5-Methylheroin (4). 5-Methylmorphine (**3**, 100 mg, 0.33 mmol) was acetylated with acetic anhydride in pyridine at room temperature for 12 h. Extractive workup (chloroform–water) and purification by chromatography on silica with 2% methanol in chloroform as eluent gave 108 mg (84.4%) of **4**, which slowly solidified, mp 136.5–142 °C. Recrystallization from ethyl acetate–cyclohexane gave needles: mp 143–144 °C; IR ν 2940, 1760, 1740, 1620, 1440, 1570, 1100, and 890 cm^{-1}; NMR δ 165 (s, 3 H, C_5-Me), 2.06 (s, 3 H, acetylmethyl), 2.22 (s, 3 H, ester methyl), 2.32 (s, 3 H, N-CH_3), 5.08 (m, 1 H, C_6-H), 5.5 (br s, 1 H), 6.51, 6.73 (AB, 2 H, J = 8 Hz, aromatic protons); $[\alpha]^{23}_D$ −30° (*c* 0.83, 95% alcohol). Anal. Calcd for $C_{22}H_{25}NO_5$: C, 68.91; H, 6.57; N, 3.65. Found: C, 68.75; H, 6.63; N, 3.56.

5-Benzylthebaine (5). To a solution of the thebaine anion, prepared as reported earlier[1] from 1.533 g of thebaine, was added freshly distilled benzyl chloride (0.7 mL, 0.76 g, 6 mmol). Only a slight change in color was observed. After being stirred a further 20 min at −78 °C, the solution was allowed to come to room temperature over 2 h, during with the color changed to orange-yellow. Water (5 mL) was added, and most of the solvent was removed under diminished pressure. The yellow-brown residue was taken into chloroform, washed twice with water, filtered through anhydrous Na_2SO_4, and concentrated. The residue was chromatographed on silica with 5% methanol in chloroform as eluent (MPLC); 990 mg of 5-benzylthebaine (**5**) was obtained as a pale yellow foam, and 693 mg of unreacted thebaine also was recovered. The yield of 5-benzylthebaine based on thebaine reacted was 90.5%. A sample crystallized from anhydrous ether for analysis had mp 124 °C: IR ν 2910, 2860, 1601, 1450, 1110, 1080, and 907 cm^{-1}; NMR δ 2.44 (s, 3 H, N-CH_3), 2.45–2.9 (m, 5 H), 3.2–3.3 (m, 1 H), 3.36 (s, 3 H, C_6-OMe), 3.58 (d, 1 H, J = 15 Hz), 3.66 (d, 1 H, J = 7 Hz), 3.79 (d, 1 H, J = 15 Hz), 3.86 (s, 3 H, C_3-OMe), 4.94 (d, 1 H, J = 6.5 Hz, C_8-H), 5.52 (d, 1 H, J = 6.5 Hz, C_7-H), 6.6, 6.66 (AB, 2 H, J = 8 Hz, aromtic H), 7.22 (m, 5 H, aromatic H); $[\alpha]^{23}_D$ −527° (*c* 1.24, 95% alcohol). Anal. Calcd for $C_{26}H_{27}NO_3$: C, 77.78; H, 6.78; N, 3.49. Found: C, 77.66; H, 6.83; N, 3.46.

Attempts to convert **5** into 5-benzylcodeinone with mercuric acetate and formic acid[4] yielded material whose IR absorption

Table I

	ED/50, mice, subcutaneous, mg/kg (95% SE limit)	
	hot plate	Nilsen
5-methylmorphine (**3**)	5.3 (4.0–7.1)	11.5 (8.5–15.6)
5-methylcodeine (**2**)	14.6 (10.9–19.6)	(poor dose response)
5-methyldiacetylmorphine (**4**)	1.1 (0.83–1.5)	2.6 (1.9–3.5)
morphine sulfate	0.98 (0.83–1.1)	1.3 (1.0–1.7)
codeine phosphate	6.8 (4.5–10.2)	7.4 (4.9–11.0)

at 1720 cm^{-1} suggested the presence of 5-benzylneopinone rather than 5-benzylcodeinone.

5-Benzyl-14-hydroxycodeinone (8). 5-Benzylthebaine (**5**, 200 mg, 0.5 mmol) in 97% formic acid (2 mL) was treated with 30% H_2O_2 (0.1 mL) at 0 °C. After 30 min the mixture was heated at 55 °C for 45 min, cooled, poured into ice, and neutralized with ammonia. Extractive workup (chloroform–water) gave a crude brown gum, which was chromatographed on silica, eluting with 5% MeOH in chloroform to yield 5-benzyl-14-hydroxycodeinone (**8**, 48 mg, 24%). A sample crystallized from ethyl acetate for analysis had mp 178–179 °C: IR ν 3330, 2910, 2840, 1690, 1630, 1600, 1450, 1360, 1130, 1100, 1000, and 950 cm^{-1}; NMR δ 1.39–1.45 (m, 1 H), 2.1–2.2 (m, 1 H), 2.35 (s, 3 H, N-Me), 2.36–2.5 (m, 3 H), 2.85 (m, 1 H), 3.11 (d, 1 H, J = 18.5 Hz), 3.21, 3.53 (AB, 2 H, J = 14.5 Hz), 6.01 (d, 1 H, J = 10 Hz, C_8-H), 6.44 (d, 1 H, J = 10 Hz, C_7-H), 6.50, 6.58 (AB q, 2 H, J = 8 Hz, aromatic H), 7.2 (m, 5 H, aromatic H); $[\alpha]^{23}_D$ −233° (c 0.66, 95% alcohol). Anal. Calcd for $C_{25}H_{25}NO_4$: C, 74.42; H, 6.25; N, 3.47. Found: C, 74.42; H, 6.64; N, 3.56.

5-Carbethoxythebaine (9). To a solution of the thebaine anion, prepared as reported earlier[1] from 1.533 g of thebaine, was added 0.75 g of ethyl chloroformate (distilled and stored over $CaCO_3$). Only a slight change in color was observed. After being stirred a further 20 min at −78 °C, the solution was allowed to come to room temperature over 2 h, during which the color changed to orange-yellow. Water (5 mL) was added, and most of the solvent was removed at diminished pressure. The yellow-brown residue was taken up in chloroform, washed twice with water, filtered through anhydrous sodium sulfate, and concentrated. The residue was subjected to MPLC on silica gel (elution with 2% methanol in chloroform) to give 1.32 g, 86% based on unrecovered thebaine, of 5-carbethoxythebaine (**9**) as a pale yellow foam; 386 mg of unreacted thebaine was also recovered. Attempts to crystallize **9** were unsuccessful: IR ν 2950, 1740, 1600, 1430, 1220, 1150, 1100, 980, and 900 cm^{-1}; NMR δ 1.27 (t, 3 H, J = 7 Hz, ester methyl), 2.31 (s, 3 H, N-Me), 3.52 (s, 3 H, C_6-OMe), 3.81 (s, 3 H, C_3-OMe), 3.82 (q, 2 H, J = 7 Hz, ester methylene), 5.1 (d, 1 H, J = 6 Hz, C_8-H), 5.53 (d, 1 H, J = 6 Hz, C_7-H), 6.58 (m, 2 H, aromatic H); $[\alpha]^{23}_D$ −395° (c 0.63, 95% alcohol). Anal. Calcd for $C_{22}H_{25}NO_5$: C, 68.91; H, 6.57; N, 3.65. Found: C, 68.67; H, 6.74; N, 3.55.

5-Carbethoxycodeinone (10). 5-Carbethoxythebaine (686 mg, 1.79 mmol) in 50 mL of 3 M formic acid was treated with mercuric acetate (55 mg) under N_2 at room temperature. Stirring at room temperature continued for 7 days, with 25 mg of fresh mercuric acetate added at the end of each 24-h period. Saturated potassium carbonate solution (60 mL) was added slowly, and the mixture was extracted with chloroform four times. The combined chloroform extracts were washed with water, filtered through anhydrous sodium sulfate, and concentrated to yield a reddish brown gum. This was chromatographed on silica (elution with 5% methanol in chloroform) to give 348 mg, 52.7%, of 5-carbethoxycodeinone (**10**) as a pale yellow solid after crystallization from anhydrous ether at −5 °C: mp 152–154 °C; IR ν 1740 (ester carbonyl) and 1690 (enone) cm^{-1}; NMR δ 1.3 (t, 3 H, J = 7 Hz, ester methyl), 2.32 (s, 3 H, N-CH_3), 3.83 (s, 3 H, C_3-OMe), 4.33 (q, 2 H, J = 7 Hz, ester methylene), 6.02 (d of d, 1 H, J_{14} = 3 Hz, J_8 = 11 Hz, C_7-H), 6.69 (m, 3 H, C_8-H and aromatic protons); $[\alpha]^{23}_D$ −255° (c 0.60, 95% alcohol). Anal. Calcd for $C_{21}H_{23}NO_5$: C, 68.28; H, 6.29; N, 3.79. Found: C, 68.15; H, 6.40; N, 3.62.

Reduction of 5-Carbethoxycodeinone with Sodium Borohydride (11). 5-Carbethoxycodeinone (**10**, 348 mg, 0.943 mmol) in 5 mL of methanol was treated with sodium borohydride (180 mg) at −5 °C. TLC indicated complete disappearance of starting material and formation of several products. The reaction was quenched by aqueous ammonium chloride and extracted with chloroform. Workup gave 280 mg of a pale yellow foam, the TLC of which showed it to be a complex mixture of products. This complex mixture was subjected to preparative TLC separation (30% MeOH in $CHCl_3$), and only one product could be obtained in pure crystalline form (ethyl acetate–hexanes), which was identified as 5-(hydroxymethyl)codeine (**11**) from its spectral properties. Its yield was 60 mg (19.3%): mp 180–182 °C; IR ν 3450, 2950, 2860, 1640, 1610, 1450, 1140, 1100, 970, and 870 cm^{-1}; NMR δ 1.6 (br, 1 H, exchangeable with D_2O), 2.05–2.4 (m, 4 H), 2.42 (s, 3 H, N-Me), 2.50–3.02 (m, 4 H, with a broad bump at δ 2.8, which vanishes on D_2O exchange), 3.31 (m, 1 H), 3.82 (s, 3 H, C_3-OMe), 4.12 (q, 2 H, J = 11.5 Hz, C_5-hydroxymethyl), 4.22 (br s, 1 H, C_6-H), 5.11 (complex d, 1 H, J = 9 Hz, C_8-H), 5.71 (complex d, 1 H, J = 9 Hz, C_7-H), 6.12, 6.62 (AB, 2 H, J = 8 Hz, aromatic H). Anal. Calcd for $C_{19}H_{23}NO_4$: C, 69.28; H, 7.04; N, 4.25. Found: C, 69.06; H, 7.24; N, 4.08.

Acknowledgment. We are indebted to Dr. Arthur Jacobson of the Section on Drug Design and Synthesis, LN, NIDDK, NIH, for arranging for screening tests, and to the National Institute on Drug Abuse for generous financial support under Grant No. 5 RO1DA0 2469. We are also greatly indebted to Professor William D. Jones of these laboratories for carrying out the X-ray crystallographic structural determination of 5-methylheroin (**4**) for us.

Registry No. **1**, 118112-52-0; **2**, 118112-53-1; **3**, 118142-13-5; **4**, 118142-14-6; **5**, 118112-54-2; **6**, 118142-15-7; **8**, 118112-55-3; **9**, 118112-56-4; **10**, 118112-57-5; **11**, 118112-58-6; 5-lithiothebaine, 80583-33-1; 5-methyldihydrocodeine, 118112-59-7; 5-methyldihydroisocodeine, 118112-60-0; 6-*O*-acetyl-5-methylcodeine, 118112-61-1; 6α-*O*-acetyl-5-methyldihydrocodeine, 118112-62-2; 6β-*O*-acetyl-5-methyldihydrocodeine, 118112-63-3.

New Synthesis of Cyclobutane Annelated Compounds by the Use of a (1-Cyclobutenyl)triphenylphosphonium Salt

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Many methods for the synthesis of cyclobutane fused compounds by means of both photochemical[1] and thermochemical cycloaddition reactions[2] have been well-studied. We have recently reported a new synthesis of heterocyclic fused cyclobutanes by the use of the (1-cyclobutenyl)triphenylphosphonium salt **1**.[3] On the other hand,

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(4) Schweizer, E. E.; O'Neill, G. J. *J. Org. Chem.* **1965**, *30*, 2082.

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