δ 73.75, 86.42, 120.20, 120.85, 122.54, 127.48, 127,80, 136.84, 138.46, 140.86, 149.05, 161.79.

The **picrate**, mp 145–145.5 °C, was prepared for analysis.

Anal. Calcd for $C_{19}H_{14}N_4O_8$: C, 53.52; H, 3.29; N, 13.15. Found: C, 53.52; H, 3.26; N, 12.96.

1-(2-Pyridyl)-1-phenyl-1,3-dihydroisobenzofuran (8b) crystallized from ethyl acetate–hexane as colorless needles (68% yield): mp 98–98.5 °C; IR (KBr) 3020, 2900, 2850, 1580, 1470, 1440, 1025, 980, 770, 760, 710 cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.32 (s, 2, CH_2), 7.08–7.92 (m, 11, Ar H), 8.40–8.52 (m, 2, Ar H).

Anal. Calcd for $C_{19}H_{15}NO$: C, 83.52; H, 5.49; N, 5.13. Found: C, 83.61; H, 5.50; N, 4.95.

1-(6-Methyl-2-pyridyl)-1,3-dihydroisobenzofuran (8c) was obtained as a yellow oil in 70% yield: bp 104–109 °C (0.05 torr); IR (film) 3070, 2970, 2925, 2860, 1600, 1580, 1460, 1060, 810, 775, 750, 680, 645 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.69 (s, 3, CH_3), 5.28 (sym t (ABX), 2, CH_2), 6.23 (sym t (ABX), 1 CH), 6.86–7.70 (m, 7, Ar H); ^{13}C NMR ($CDCl_3$) δ 24.43, 73.68, 86.55, 109.16, 116.95, 120.85, 122.02, 122.80, 127.35, 127.74, 128.65, 137.10, 138.52, 141.12.

The **picrate**, mp 157–158 °C, was prepared for analysis.

Anal. Calcd for $C_{19}H_{15}NO$: C, 54.55; H, 3.64; N, 12.73. Found: C, 54.78; H, 3.61; N, 12.58.

Registry No. 2a, 1121-60-4; **2b**, 91-02-1; **2c**, 1122-62-9; **4a**, 7547-88-8; **4b**, 6634-51-1; **4c**·bromide, 74808-18-7; **4c**·perchlorate, 74808-20-1; **5**, 74824-35-4; **6a**, 74808-21-2; **6b**, 74824-36-5; **6c**, 74808-22-3; **8a**, 74808-23-4; **8a**·picrate, 74808-25-6; **8b**, 74808-26-7; **8c**, 74808-27-8; **8c**·picrate, 74808-28-9.

Convenient Preparation of (9-Fluorenyl)methanol and Its 2,7-Dihalo Derivatives

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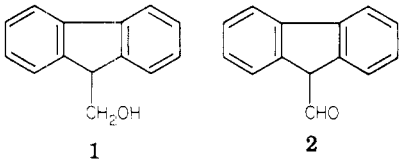
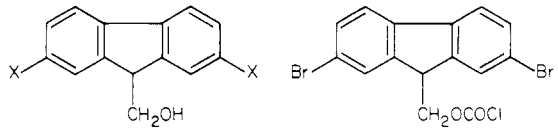
With the ever-increasing use[1-3] of the base-sensitive 9-fluorenylmethyloxycarbonyl (Fmoc) amino-protecting group there is a current need for a convenient, large-scale synthetic route to (9-fluorenyl)methanol (1), the key precursor of all Fmoc derivatives. A previously described[4] simple modification of the tedious Wislicenus–Brown[5,6] procedure, while a marked improvement over earlier methods, still suffers from the necessity to separate 1 from significant quantities of polymeric byproducts with consequent mediocre yields (50–60%). These remaining difficulties can be eliminated and 1 readily obtained in quantity by substitution of sodium borohydride for formalin in the conversion of 2 to 1. By this method 1 is obtained in 80% yield in a form pure enough for further use directly from the reaction mixture.



1 2

This simple method was not, however, satisfactory for the synthesis of the 2,7-dibromo and 2,7-dichloro derivatives of (9-fluorenyl)methanol, the corresponding Fmoc derivatives of which are of potential interest as amino-protecting groups of greater base sensitivity than the parent system. Fortunately a return to the classic procedure of Wislicenus–Brown[5,6] in the case of the dichloro derivative (3, X = Cl) and use of potassium ethoxide al-



3, X = Cl, Br 4

lowed facile formylation, and the formyl derivative was easily reduced to the alcohol. In the case of the dibromo analogue (3, X = Br) this method was not completely satisfactory because the presence of colored impurities (possibly the dibenzofulvene or products derived therefrom) made purification of the initial product exceptionally tedious. A far simpler approach to the dibromo derivative involved direct bromination of (9-fluorenyl)methanol by means of *N*-bromosuccinimide in acetic acid in the presence of hydrogen bromide. This method was developed from a related simple technique newly devised for the halogenation of fluorene. Direct bromination of (9-fluorenyl)methyl chloroformate was also noted to give the corresponding chloroformate, 4. However, the yield was not high (37%), and the chloroformate would probably be best prepared from the alcohol by reaction with phosgene. Curiously, chlorination of 1 by means of *N*-chlorosuccinimide in acetic acid by the same method as that used for the dibromo derivative gave not the alcohol but the corresponding acetate. Although this could be avoided by chlorination in dioxane–water, there was no particular advantage to this technique since the Wislicenus–Brown method proved satisfactory for the preparation of 3 (X = Cl).

That 2,7-dihalo substitution greatly accelerates base-catalyzed deblocking was shown by comparison of the ease of cleavage by piperidine and pyridine of the urethanes derived from 1 and 3 by reaction with *p*-chlorophenyl isocyanate. Deblocking rates were greatly dependent on the concentration of reactants. Under standardized conditions in dilute solution the dihalo derivatives were deblocked by a 10 molar excess of piperidine in toluene within 45–85 min, whereas the unsubstituted compound

(1) For a polymeric deblocking system for Fmoc derivatives see L. A. Carpino, J. R. Williams, and A. Lopusinski, *J. Chem. Soc., Chem. Commun.*, 450 (1978). Further work has shown that commercial polymers used as supports for piperazino deblocking agents as described in this reference are inconsistently effective even though obtained from the same suppliers (Dow Chemical Co., Rohm and Haas, Inc.). Although the deblocking step occurred readily, the scavenging of dibenzofulvene by piperazino polymers obtained from the new samples of commercial resins was more sluggish. These differences, which may have arisen because of the presence of traces of additives left over from the polymerization process, are now under study.

(2) For the unique advantages of Fmoc protection in connection with both solid-phase and solution peptide synthesis, see: (a) E. Atherton, H. Fox, D. Harkiss, C. J. Logan, R. C. Sheppard, and B. J. Williams, *J. Chem. Soc., Chem. Commun.*, 537, 539 (1978); (b) C.-D. Chang and J. Meienhofer, *Int. J. Pept. Protein Res.*, 11, 246 (1978); (c) C.-D. Chang, M. Waki, M. Ahmad, J. Meienhofer, E. O. Lundell, and J. D. Haug, *ibid.*, 15, 59 (1980); (d) M. Bodanszky, S. S. Deshmane, and J. Martinez, *J. Org. Chem.*, 44, 1622 (1979); (e) A. Bodanszky, M. Bodanszky, N. Chandramouli, J. Z. Kwei, J. Martinez, and J. C. Tolle, *ibid.* 45, 72 (1980); (f) J. Martinez, J. C. Tolle, and M. Bodanszky, *ibid.*, 44, 3596 (1979); (g) E. Atherton, C. Bury, R. C. Sheppard, and B. J. Williams, *Tetrahedron Lett.*, 3041 (1979); (h) R. Arshady, E. Atherton, and R. C. Sheppard, *ibid.*, 1521 (1979); (i) "Chemalog Hi-Lites", Chemical Dynamics Corp., South Plainfield, NJ, June 1978, Apr 1980.

(3) R. B. Merrifield and A. E. Bach, *J. Org. Chem.*, 43, 4808 (1978).

(4) (a) L. A. Carpino and G. Y. Han, *J. Am. Chem. Soc.*, 92, 5748 (1970); (b) L. A. Carpino and G. Y. Han, *J. Org. Chem.*, 37, 3404 (1972); 38, 4218 (1973); (c) L. A. Carpino, *Acc. Chem. Res.*, 6, 191 (1973).

(5) W. Wislicenus and M. Waldmuller, *Ber. Dtsch. Chem. Ges.*, 42, 785 (1909).

(6) W. G. Brown and B. A. Bluestein, *J. Am. Chem. Soc.*, 65, 1082 (1943). The conversion of 2 to 1 by means of alkaline aqueous formaldehyde was previously referred to erroneously as a cross-Cannizzaro reaction. The mechanism of this reaction was established by Burr [J. G. Burr, Jr., *J. Am. Chem. Soc.*, 73, 823 (1951)] as involving hydroxymethylation followed by base-catalyzed deformylation.

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required 5–6 days. Similarly, in pyridine as both solvent and reactant, the dihalo derivatives were deblocked in about 24 h, whereas with the unsubstituted compound the first traces of *p*-chloroaniline and dibenzofulvene were detected only after about 10 h.

Experimental Section[7]

(9-Fluorenyl)methanol. To a suspension of 6.5 g of sodium hydride (59% dispersion in mineral oil) in 100 mL of dry ether were added 8.3 g of fluorene and 9 mL of ethyl formate. The resulting mixture was refluxed with magnetic stirring for 10 h and then cooled in an ice bath, and ice chips were added until gas evolution ceased. Water (100 mL) was added and the ether layer collected in a separatory funnel and discarded. The aqueous layer was washed once with 75 mL of ligroin (bp 30–60 °C), filtered to remove a trace of brown solid, cooled in an ice bath, and acidified with 15 mL of acetic acid. The oil which separated was extracted into three 50-mL portions of methylene dichloride or ether. If an emulsion formed it could be broken by suction filtration through Whatman No. 1 filter paper. The organic extracts were washed once with 50 mL of water and once with 50 mL of sodium bicarbonate solution (1 M), dried ($MgSO_4$), and evaporated in a water bath at 50–60 °C with the aid of a water aspirator. The viscous residue was dissolved in 100 mL of methanol and treated over 2–3 min with 2 g of sodium borohydride. Gas evolution and spontaneous warming occurred. The solution was stirred at room temperature for 2 h, diluted to 400 mL with water, and treated with 15 mL of acetic acid, and the mixture was stirred magnetically at room temperature for 1.5 h. Filtration followed by washing with water gave 7.85 g (80%) of the alcohol as tiny snow-white needles, mp 98–100 °C (lit.[4b] mp 100–101 °C). The material as obtained was pure enough for further use but if desired could be recrystallized from ligroin (bp 88–98 °C; 1 g of compound/10 mL of solvent) with 94% recovery to give snow-white, papery needles, mp 98.5–100.5 °C.

2,7-Dibromofluorene.[8] To a suspension of 83.1 g of fluorene and 174 g of *N*-bromosuccinimide in 1 L of glacial HOAc was added with stirring over 3–5 min 25 mL of 48% HBr. The reactants dissolved as a new solid separated. After 3 h, 500 mL of water was added slowly, and the solid was filtered, dried in air, and recrystallized from EtOH–$EtNO_2$ (1:3) to give 88.2 g (54.4%) of the dibromofluorene: mp 166–167 °C (lit.[9] mp 164 °C); ^1H NMR ($CDCl_3$) δ 3.74 (br s, 2, CH_2), 7.2–7.6 (m, 6, aryl).

2,7-Dichlorofluorene. This was prepared as described for the corresponding dibromo analogue from 99.6 g of fluorene, 156 g of *N*-chlorosuccinimide, 750 mL of HOAc, and 48 mL of concentrated HCl added over about 10 min. Without addition of H_2O the white solid was filtered after 5 h, washed with ethanol, and recrystallized from EtOH–$EtNO_2$ (1:1) to give 35.5 g (25.2%) of the dichlorofluorene as white crystals, mp 125.5–126.5 °C (lit.[10] mp 128 °C).

(2,7-Dibromo-9-fluorenyl)methanol. A suspension of 2 g of potassium metal in 60 mL of dry ether was treated with 4.5 mL of dry EtOH, and the mixture was stirred magnetically and refluxed for 1 h after which a second 4.5 mL of dry EtOH was added. After an additional 2 h all of the potassium had dissolved. The mixture was cooled to room temperature and treated with 16.2 g of 2,7-dibromofluorene, and this mixture was refluxed for 10 min. While the mixture continued to reflux, there was added dropwise over 10 min a solution of 4.0 mL of ethyl formate in 20 mL of dry ether. The refluxing was continued for 1 h, the mixture was treated with 200 mL of water, the two layers were separated, and the aqueous layer was filtered to remove a small amount of suspended solid. The filtrate was acidified with dilute H_2SO_4 and the precipitated oil extracted with four 50-mL portions of CH_2Cl_2. The extracts were washed with two 50-mL portions of water, dried ($MgSO_4$), and evaporated in vacuo over a water bath. The residual yellow solid was dissolved in 100 mL of MeOH, and the solution was cooled in an ice bath and treated with 2 g of $NaBH_4$ over a period of 10 min with stirring. Stirring was continued at room temperature for 1 h and the mixture diluted to 400 mL with water which caused separation of a white solid. The mixture was acidified with dilute H_2SO_4, stirred at room temperature for about 10 h, filtered, washed with water, and air-dried to give 12.25 g (69.2%) of the crude alcohol as a cream-yellow powder, mp 108–133 °C. Recrystallization from 1:1 benzene–ligroin (bp 60–70 °C) gave 6.13 g (34.6%) of the alcohol as a cream-yellow powder, mp 148–153 °C. An additional recrystallization raised the melting point to 151–153 °C (lit.[11] mp 154 °C): ^1H NMR (CD_3COCD_3) δ 2.9 (br s, 1, OH), 3.9–4.3 (m, 3, $CHCH_2$), 7.5–7.9 (m, 6, aryl).

Bromination of (9-Fluorenyl)methanol. To a solution of 0.6 g of **3** (X = H) in 6 mL of acetic acid was added 1.06 g of *N*-bromosuccinimide followed by 0.2 mL of 48% HBr. The mixture was stirred for 15 min and diluted to 150 mL with water. The precipitated white powder (0.95 g, 87.7%, mp 124–140 °C) was filtered, washed with water, dried in air, and recrystallized from about 8 mL of 1:2 benzene–ligroin (bp 60–70 °C) to give 0.55 g (50.7%) of **3** (X = Br) as a flocky white solid (mp 150–152 °C) identified by mixture melting point and IR and NMR spectral comparison with an authentic sample obtained as described above.

(2,7-Dichloro-9-fluorenyl)methanol. This compound was obtained in 65–70% yield (mp 140–142 °C) by the method described for the dibromo derivative from 2,7-dichlorofluorene. The analytical sample was obtained from ligroin (bp 88–98 °C) as tiny cream-white crystals: mp 140–142 °C; ^1H NMR ($CDCl_3$) δ 1.65 (s, 1, OH), 4.2 (br s, 3, CH_2CH), 7.27–7.73 (m, 6, aryl).

Anal. Calcd for $C_{14}H_{10}Cl_2O$: C, 63.42; H, 3.80; Cl, 26.75. Found: C, 63.45; H, 4.08; Cl, 26.81.

(2,7-Dichloro-9-fluorenyl)methyl Acetate. Treatment of **3** (X = Cl) with *N*-chlorosuccinimide as described for the dibromo analogue led to isolation of the acetate rather than the alcohol. The same ester was obtained (86%) by treatment of the alcohol with Ac_2O–NaOAc. The analytical sample was obtained from ligroin (bp 88–98 °C) as snow-white crystals: mp 111.5–113.5 °C; ^1H NMR ($CDCl_3$) δ 2.13 (s, 3, CH_3CO), 4.15–4.45 (m, 3, CH_2CH), 7.25–7.7 (m, 6, aryl).

Anal. Calcd for $C_{16}H_{12}Cl_2O_2$: C, 62.56; H, 3.94. Found: C, 62.95; H, 4.29.

(2,7-Dibromo-9-fluorenyl)methyl Chloroformate (4). To a solution of 2.6 g of (9-fluorenyl)methyl chloroformate[4b] in 25 mL of $CHCl_3$ was added 1.04 g of Br_2 and 1 mL of a solution of $FeCl_3$ in $CHCl_3$ (prepared by dissolving 1 g of anhydrous $FeCl_3$ in 25 mL of $CHCl_3$ followed by filtration). The flask was covered with aluminum foil, and the solution was stirred at room temperature for about 2 days, poured into a flat dish, and allowed to evaporate. The residual solid was triturated with 4 mL of ligroin (bp 30–60 °C), filtered, and recrystallized from a 2:1 mixture of ligroin (bp 60–70 °C) and ligroin (bp 88–98 °C). Cooling gave 1.55 g (37.3%) of the chloroformate as cream-yellow crystals, mp 118–121 °C. An additional recrystallization (decolorizing carbon) gave 1.05 g (25.1%) of white crystals: mp 124–126 °C; ^1H NMR ($CDCl_3$) δ 4.15–4.6 (m, 3, CH_2CH), 7.50–7.75 (m, 6, aryl).

Anal. Calcd for $C_{15}H_9ClBr_2O_2$: C, 43.25; H, 2.18. Found: C, 42.82; H, 2.27.

(2,7-Dibromo-9-fluorenyl)methyl *N*-(*p*-Chlorophenyl)-carbamate. A solution of 0.2 g of *p*-chlorophenyl isocyanate and 0.45 g of **3** (X = Br) in 5 mL of hot benzene (filtered if cloudy) was treated with 1 drop of Et_3N. White crystals separated in a few minutes. Filtration gave 0.48 g (74.4%) of the urethane, mp 218–222 °C dec (decomposition varies greatly with the rate of heating). The same compound was obtained by treatment of **4** with *p*-chloroaniline. An analytical sample was obtained by recrystallization from $EtNO_2$; mp 221–223 °C dec.

Anal. Calcd for $C_{21}H_{14}Br_2ClNO_2$: C, 49.68; H, 2.78; N, 2.76. Found: C, 50.12; H, 3.02; N, 2.83.

(2,7-Dichloro-9-fluorenyl)methyl *N*-(*p*-Chlorophenyl)-carbamate. This compound was obtained in 53% yield as de-

(7) Melting points and boiling points are uncorrected. Infrared spectra were determined on a Perkin-Elmer 237B instrument and NMR spectra on Varian A-60 and Perkin-Elmer R-12 instruments with Me_4Si as internal standard. Elemental analyses were carried out by the University of Massachusetts Microanalytical Laboratory under the direction of Greg Dabkowski.

(8) Compare S. D. Ross, M. Finkelstein, and R. C. Peterson, *J. Am. Chem. Soc.*, **80**, 4327 (1958).

(9) A. Sieglitz, *Ber. Dtsch. Chem. Ges.*, **53**, 1236 (1920).

(10) W. R. Hodgkinson and F. E. Matthews, *J. Chem. Soc.*, **43**, 170 (1883).

(11) W. G. Brown and B. A. Bluestein, *J. Am. Chem. Soc.*, **65**, 1235 (1943).

scribed for the dibromo analogue; mp 229–232 °C dec (decomposition varies greatly with the rate of heating).

Anal. Calcd for $C_{21}H_{14}Cl_3NO_2$: C, 60.24; H, 3.34; N, 3.35. Found: C, 60.21; H, 3.41; N, 3.61.

Deblocking of 2,7-Dihalo-Substituted (9-Fluorenyl)methyl Carbanilates. A stock solution containing 42.6 mg (0.5 mequiv) of piperidine in 10 mL of toluene was prepared. To each of three 1-mL aliquots of this solution was added 0.005 mequiv of the corresponding Fmoc derivative. The reaction was followed by TLC on silica gel (Eastman Chromagram plates) with development by toluene in the case of the dihalo derivatives and ethyl acetate–toluene (10% v/v) in the case of the unsubstituted compound. Urethane consumption was complete in about 45 min in the case of the dibromo derivative and in about 85 min for the dichloro analogue. The dihalo derivatives were insoluble in toluene at the concentrations used, and part of the difference between the dibromo and dichloro compounds may have been due to this factor. In the case of the parent Fmoc derivative the first evidence for the formation of *p*-chloroaniline was observed only after about 12 h, and the urethane did not completely disappear until after 5.5 days. Similar tests in pyridine as both solvent and deblocking agent gave evidence for the appearance of *p*-chloroaniline immediately after mixing in the case of the dihalo derivatives and for complete consumption of urethane after about 24 h. In the case of the parent Fmoc system the first evidence of dibenzofulvene formation appeared only after about 10 h, and urethane was still present after 8 days, when the test was termninated.

Acknowledgment. This work was supported by a grant from the National Institutes of Health (GM-09706).

Registry No. 1, 24324-17-2; **2**, 20615-64-9; **3** (X = Br), 74316-23-7; **3** (X = Cl), 74808-81-4; **4**, 74808-82-5; fluorene, 86-73-7; ethyl formate, 109-94-4; 2,7-dibromofluorene, 16433-88-8; 2,7-dichlorofluorene, 7012-16-0; (2,7-dichloro-9-fluorenyl)methyl acetate, 74808-83-6; 9-fluorenylmethyl chloroformate, 28920-43-6; 2,7-dibromo-9-fluorenylmethyl *N*-(*p*-chlorophenyl)carbamate, 74808-84-7; *p*-chlorophenyl isocyanate, 104-12-1; *p*-chloroaniline, 106-47-8; 2,7-dichloro-9-fluorenylmethyl *N*-(*p*-chlorophenyl)carbamate, 74808-85-8.

An Improved Synthesis of Carbocyclic and Heterocyclic Arene Imines

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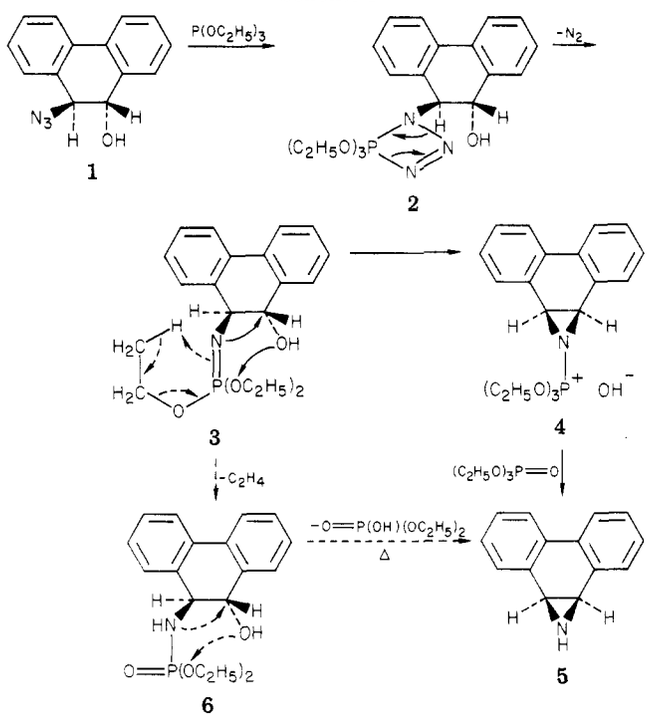
Recently we reported the synthesis of phenanthren-9,10-imine (**5**) from *trans*-10-azidodihydrophenanthren-9-ol (**1**) and a tertiary phosphine.[1] The method proved useful for the preparation of K-region imine derivatives of benz[*a*]anthracene, benzo[*a*]pyrene, and dibenz[*a*]-anthracene.[2] This synthesis has, however, two disadvantages: (i) the resulting imines decompose on prolonged treatment with excess phosphine; (ii) the separation of the polycyclic aziridines from phosphine oxides and other phosphorus-containing products is often tedious and associated with heavy losses.

We now describe an improved synthesis of polycyclic arene imines in which the tertiary phosphine is substituted by triethyl phosphite. This method does not suffer from the disadvantages and permits facile preparation of **5**, benz[*a*]anthracen-5,6-imine (**10**), and 1a,7b-dihydroazirino[5,6]benzo[1,2-*c*:3,4-*c*']dithiophene (**9**). The latter compound is the first imine of an aromatic heteropolycyclic structure.

Scheme I



When, e.g., azido alcohol **1** is treated with 1.5–2.5 equiv of $P(OC_2H_5)_3$, the exothermic reaction that takes place yields molecular nitrogen (confirmed by GLC) and 82% imine **5**. The reaction is less vigorous when the synthesis is conducted in boiling methylene chloride and the yield is essentially quantitative after 20 min. In benzene, at or below 30 °C, the interaction of **1** and triethyl phosphite leads to the formation of a phosphorus-containing compound, which is formulated as structure **6** by virtue of the elemental analysis and the spectral data. Two distinguishing bands at 3290 and 3390 cm^{-1} reveal the existence of the NH and OH groups. The 270-MHz ^1H NMR spectrum (in $CDCl_3$) shows two CH_3 triplets at 1.266 and 1.316 ppm ($J_{CH_2CH_3}$ = 6.2 Hz) and two superimposed ^{31}P-split quartets centered at 4.086 ppm. The NH resonance at 3.179 (dd, J_{NHH_9} = 10.5 Hz, J_{NHP} = 14 Hz) and the broad OH peak at 3.3 ppm[3] disappear upon addition of D_2O. The benzylic protons (α to NH and α to OH, respectively) show up at 4.353 (AB q, $J_{H_9H_{10}}$ Hz, J_{NHH_9} = 10.5 Hz) and 4.671 ppm (d, $J_{H_9H_{10}}$ = 10.0 Hz). The most indicative fragment ions in the high-resolution mass spectrum given in the experimental section are the molecular ion and its dehydration product (*m/e* 347, 329), the phosphorus containing fragments of *m/e* 302, 301, 300, 274, 255, 256, and the fluorenyl base peak which is characteristic of most 9,10-dihydrophenanthrene derivatives.[4] Fragments such as $C_{14}H_9NOP$ (*m/e* 238) and $C_4H_{11}NO_3P$ (*m/e* 152) confirm the existence of a P–N bond in **6**.

In analogy to phenanthrene-9,10-imine formation from **1** and tertiary phosphines[1] the present synthesis is assumed to follow the steps outlined in Scheme I (full arrows). The

(1) Ittah, Y.; Sasson, Y.; Shahak, I.; Tsaroom, S.; Blum, J. *J. Org. Chem.* **1978**, *43*, 4271.
(2) Blum, J.; Yona, I.; Tsaroom, S.; Sasson, Y. *J. Org. Chem.* **1979**, *44*, 4178.
(3) Owing to hydrogen bonding the location of the OH peak varies considerably when the concentration of **6** in $CDCl_3$ is changed. In our experience it appeared between 3 and 8 ppm.
(4) Eland, J. H. D.; Danby, C. T. *J. Chem. Soc.* **1965**, 5935.

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