mass spectrum, m/e 344 (M^+), 267, 148, 103 (base), 80, 79, 77.

Anal. Calcd for $C_{23}H_{24}N_2O$: C, 80.20; H, 7.02; N, 8.13. Found: C, 80.42; H, 7.20; N, 7.87.

1′,3′-Diphenyl-4-azahomoadamantano[4,5-*d*]-1′,2′,4′-$\Delta^{2'}$-triazoline (15a). To a stirred mixture of **3a** (75 mg, 0.50 mmol) and (α-chlorobenzylidene)phenylhydrazine[32] (58 mg, 0.25 mmol) in benzene (3 mL) was added triethylamine (51 mg, 0.50 mmol) in benzene (3 mL) at room temperature. After the stirring was continued for 6 days, the precipitates were filtered and washed with benzene. The combined filtrate and washings were evaporated to dryness to afford an oily product which was purified on an alumina column (n-hexane–CH_2Cl_2) to give unreacted **3a** (20 mg) and **15a** as yellowish crystals (32 mg, 37.3%): mp 176–179 °C dec; IR (KBr) 3080, 3040, 1600, 1570, 1500, 1030, 750, 690 cm^{-1}; UV (MeOH) λ_{max} 220 (ϵ 11 900), 258 (8100), 346 (7800); ^1H NMR ($CDCl_3$) δ 7.7–7.0 (m, 10 H), 5.00 (d, J = 1.5 Hz, 1 H), 4.00 (br s, 1 H), 2.50 (br s, 1 H), 2.3–1.0 (m, 12 H).

Anal. Calcd for $C_{23}H_{25}N_3$: C, 80.43; H, 7.34; N, 12.23. Found: C, 80.35; H, 7.60; N, 12.06.

1′,3′-Diphenyl-5-methyl-4-azahomoadamantano[4,5-*d*]-1′,2′,4′-$\Delta^{2'}$-triazoline (15b). The reaction of **3b** (81 mg, 0.50 mmol) with (α-chlorobenzylidene)phenylhydrazine (58 mg, 0.25 mmol) as described above under the same conditions and after chromatography (alumina, n-hexane–CH_2Cl_2) afforded unreacted **3b** (26 mg) and **15b** as yellowish crystals (45 mg, 50.3%): mp 176–180 °C dec; IR (KBr) 3060, 1590, 1495, 1400, 760, 695 cm^{-1}; UV (MeOH) λ_{max} 227 nm (ϵ 11 800), 270 (7300), 347 (7600); ^1H NMR ($CDCl_3$) δ 7.7–7.0 (m, 10 H), 3.92 (br s, 1 H), 1.67 (s, 3 H), 2.8–0.8 (m, 13 H).

Anal. Calcd for $C_{24}H_{27}N_3$: C, 80.63; H, 7.61; N, 11.75. Found: C, 80.42; H, 7.90; N, 11.69.

4-Azahomoadamantano[5,4-*e*]imidazole (17). To a stirred mixture of **3a** (75 mg, 0.50 mmol) and TosMIC[29] (162 mg, 0.80 mmol) in methanol (3 mL) and dimethoxyethane (1.5 mL) was added solid potassium carbonate (137 mg, 1.00 mmol). After the stirring was continued for 12 h at room temperature, the mixture was heated under reflux for 5 h. The solvent was removed, and the residue was treated with aqueous saturated NaCl solution (6 mL) and extracted with CH_2Cl_2 (3 × 6 mL). The combined extracts were dried (Na_2SO_4) and evaporated to dryness, affording a brownish solid which gave **17** as crystals (46 mg, 48.8%) after chromatography (alumina, CH_2Cl_2): mp 107–110 °C; IR (KBr) 3080, 1610, 1490, 1240, 1212, 927, 800 cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.27 (s, 1 H), 6.67 (s, 1 H), 4.30 (br s, 1 H), 3.15 (br s, 1 H), 2.5–1.3 (m, 12 H); mass spectrum, m/e 188 (M^+, base), 173, 131, 41.

Anal. Calcd for $C_{12}H_{16}N_2$: C, 76.55; H, 8.57; N, 4.88. Found: C, 76.63; H, 8.48; N, 4.80.

The reaction of **3b** with TosMIC under the same conditions gave only recovered **3b**.

Registry No. 2a, 34197-88-1; **2b**, 65218-92-0; **2e**, 65218-95-3; **2f**, 65218-96-4; **3a**, 65218-91-9; **3b**, 65218-97-5; **3e**, 65219-00-3; **3e′**, 71302-50-6; **3e′**·HCl, 71250-91-4; **3f**, 71250-92-5; **5e**, 54530-05-1; **6**, 700-58-3; **10**, 71250-93-6; **11**, 71250-94-7; **13**, 71250-95-8; **14a**, 71250-96-9; **14b**, 71250-97-0; **14c**, 71250-98-1; **15a**, 71250-99-2; **15b**, 71251-00-8; **17**, 71251-01-9; diphenylketene, 525-06-4; phenylhydroximic acid chloride, 698-16-8; benzonitrile oxide, 873-67-6; (α-chlorobenzylidene)phenylhydrazine, 15424-14-3.

(32) For cycloadditions of diphenylnitrilimine with aldimines, see R. Huisgen, R. Grashey, H. Knupfer, R. Kunz, and M. Seidel, *Chem. Ber.*, **97**, 1085 (1964); R. Huisgen, R. Grashey, E. Aufderhaar, and R. Kunz, *ibid.*, **98**, 642 (1965).

Notes

Facile Preparation of 2- and 3-Fluoro-7,12-dimethylbenz[*a*]anthracene

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In order to elicit mutagenicity and carcinogenicity, polycyclic aromatic hydrocarbons undergo bioactivation and covalent linkage with DNA.[1] It has been shown that microsomally activated 7,12-dimethylbenz[*a*]anthracene (DMBA) binds with poly(G) to furnish products similar to the ones generated by the chemical reaction of DMBA 5,6-oxide[2,3] with poly(G). It has, however, recently been proposed that DNA binding primarily occurs[4] through the generation of a reactive diol epoxide and that the DMBA *trans*-3,4-dihydrodiol[5,6] is the most mutagenic and carcinogenic DMBA metabolite. If indeed DMBA ring A activated metabolites are primarily responsible for DNA damage and subsequent cell transformation, then the introduction of fluorine at any of positions 1, 2, 3, or 4 should considerably inhibit carcinogenicity. It has been observed that 1F- and 2F-DMBA do not produce sarcomas in long-Evans rats[7] and do not initiate tumors in mouse skin.[8] Furthermore, comparison of mutagenic and carcinogenic activities with DNA binding in Syrian hamster embryo cell culture for the 11-, 5-, and 2F analogues of DMBA showed a remarkable parallelism.[9] In connection with our research program on chemical carcinogenesis, we required relatively large amounts of the 2- and 3F-DMBA analogues for biological testing and biochemical studies.

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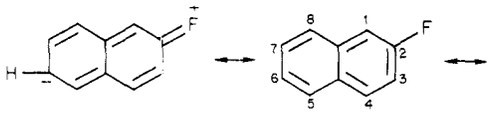
0022-3263/79/1944-3715$01.00/0

For our purposes, commercially available 2-fluoronaphthalene (1) served as starting material. Positions 1, 3, 6, and 8 of 1 are expected to undergo electrophilic attack by cations. Thus, it has been observed that the Friedel-Craft reaction[11] of β-methyltricarballylic acid anhydride with 1 in nitrobenzene occurs only in the unsubstituted ring.



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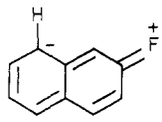
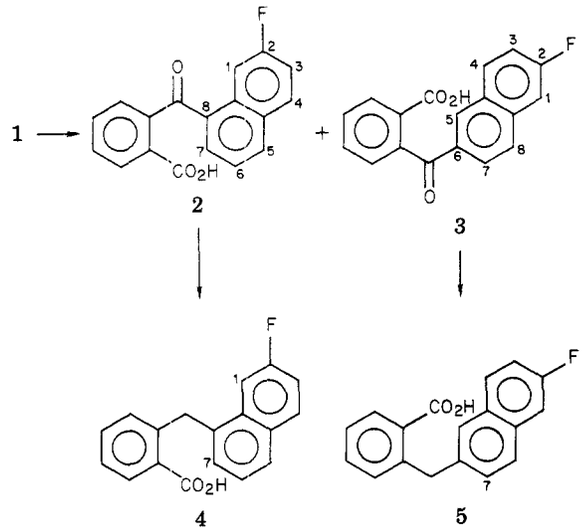


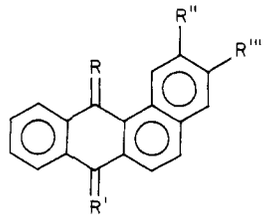
In these laboratories, Friedel-Craft's reaction of phthalic anhydride with 1 in CS_2 afforded a mixture of keto carboxylic acids **2** and **3** in a ratio of 2:1. The complex separation of **2** and **3** was avoided by reduction with Zn/NH_4OH followed by facile fractional crystallization of the resulting acids **4** and **5** from benzene, chloroform, and benzene–hexane. The separation of **4** and **5** was monitored



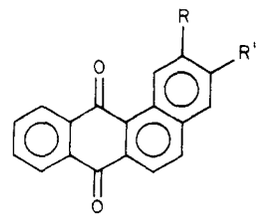
2 3 4 5

by observing their ^1H NMR benzyl–methylene resonances at δ 4.84 (**4**) and 4.60 (**5**). While the ^1H NMR spectrum for **4** depicted a complex signal at δ 8.10 (C-7 H, J_{H7-H6} = 9.0 Hz and J_{H7-H5} = 2 Hz), the ^1H NMR spectrum for **5** contained a sharp doublet at δ 8.10 (C-7 H, J_{H7-H8} = 8.0 Hz). The structures for **4** and **5** were further substantiated by comparison of their ultraviolet spectra with those of 2-fluoro-6-methylnapthalene (λ_{max} 323 nm), 2,6-dimethylnapthalene (λ_{max} 325), and 1,7-dimethylnapthalene (λ_{max} 322).[12]

Cyclization of **4** and **5** in concentrated sulfuric acid at room temperature for 1 h afforded anthrones **6** and **7**, which on subsequent oxidation provided quinones **8** and **9**. The ^1H NMR spectra for **8** and **9** exhibited a doublet of doublets at δ 9.33 (J_{H1-F2} = 13 Hz; J_{H1-H3} = 2.5 Hz) and δ 9.80 (J_{H1-H2} = 9 Hz and J_{H1-F3} = 5.2 Hz), respectively, thus confirming the assigned structures for precursor to the acids **4** and **5**.



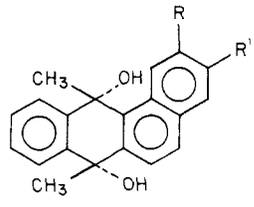
6, 7



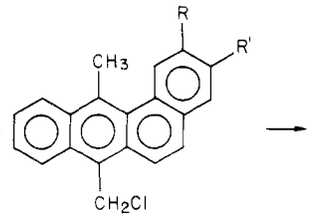
8, 9

4 → **6**, R = H_2; R′ = O; R″ = F; R‴ = H → **8**, R = F; R′ = H
5 → **7**, R = O; R′ = H_2; R″ = H; R‴ = F → **9**, R = H; R′ = F

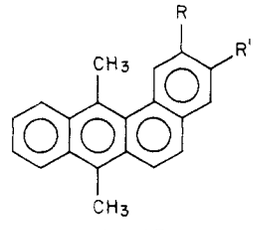
Diastereomeric diols **10** or **11**, obtained by the reaction of quinones **8** or **9** with methylmagnesium iodide, underwent reaction with HCl in ethyl acetate to furnish 7-chloromethyl-2- and 3-fluoro-7,12-dimethylbenz[*a*]anthracene derivatives **12** and **13**, respectively. Subsequent reduction of **12** and **13** with lithium aluminum hydride[13] in ether furnished 2-fluoro-7,12-dimethylbenz[*a*]anthracene[14] (**14**) (mp 93–94 °C) and the 3F analogue **15** (mp 113–14 °C). ^1H NMR spectra of **14** and **15** showed doublets at δ 8.02 (C_6H), characteristic of ring-B unsubstituted DMBA derivatives.



10, 11



12, 13



14, 15

8 → **10**, R = F; R′ = H → **12**, R = F; R′ = H → **14**, R = F; R′ = H
9 → **11**, R = H; R′ = F → **13**, R = H; R′ = F → **15**, R = H; R′ = F

Experimental Section

All melting points are uncorrected and were determined on a Thomas-Hoover capillary melting point apparatus. Ultraviolet and infrared spectra were recorded on Beckman UV-5260 and IR-4230 instruments. ^1H NMR spectra were determined on Varian A-60-A or Brucker 90 MHz instruments. All chemical shifts are in δ values. 2-Fluoronapthalene was purchased from ICN Pharmaceuticals, Inc., Plainview, N.Y. Analyses were obtained from Galbraith Laboratories Inc., Knoxville, Tenn.

2-(2-Fluoro-8-napthylmethyl)benzoic Acid (4) and 2-(2-Fluoro-6-napthylmethyl)benzoic Acid (5). To a vigorously stirred solution of 2-fluoronapthalene (1; 14.6 g; 0.1 mol) in CS_2 (200 mL) at 0 °C was added anhydrous aluminum chloride (28.0 g; 0.2 mol) in small portions over a period of 30 min. The mixture was stirred for 3 h at 0 °C and for an additional 15 h at room

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(13) We have found that $LiAlH_4$ reduction of 7-chloromethyl-7,12-dimethylbenz[*a*]anthracene derivatives is particularly useful for isotopic labeling. We have prepared a number of ^2H-labeled DMBA derivatives (fluoro, chloro, methoxy, and hydroxy) by this method. Our results shall be a subject of a future publication.
(14) ^1H NMR, UV, mp, and mmp of our 2F-DMBA compared favorably with a sample provided to us by Professor Melvin S. Newman of The Ohio State University (unpublished results). 2F-DMBA was prepared from 2-fluoro-8-bromonapthalene by known methods.

temperature. The mixture was poured onto ice and dichloromethane (500 mL) was added. The organic layer was separated, washed with water, dried over sodium sulfate, and evaporated to furnish a brown residue which was dissolved in 10% NaOH solution. The solution was extracted with ether, and the aqueous layer was acidified with 2 N HCl. The brown solid was collected and the process repeated to afford 18.0 g (61%) of a mixture of keto acids **2** and **3**. The mixture of **2** and **3** (18 g) was dissolved in NH_4OH solution (30%, 600 mL). Zinc dust (60 g) and a few crystals of $CuSO_4$ were added and the mixture vigorously stirred at reflux for 12 h. The mixture was filtered and the solid washed with 10% NaOH and H_2O. The combined filtrates were acidified with HCl, and the grey solid was collected and dried under reduced pressure affording 13 g (78%): ^1H NMR ($CDCl_3$) δ 4.60, 4.88 (s in 1:2 ratio), 6.6 to 8.4 (c), 11.8 (bs, CO_2H). The grey solid was fractionated into pure acids **4** and **5** by multiple crystallization from nitromethane, choroform, benzene, and a mixture of benzene–hexane. The progress of the separation of the two isomers **4** and **5** was monitored by ^1H NMR [**4**, 4.88 (s, 2 H); **5**, 4.60 (s, 2 H)]. Compound **4** crystallized first. The following fractions were obtained: **4** (6.5 g), **5** (2.2 g), and a mixture of **4** and **5** (4 g).

2-(2-Fluoro-8-napthylmethyl)benzoic acid (4): mp 160–162 °C (benzene, sublimes); IR (KBr) 1700 cm^{-1} (C=O); UV λ_{max} (MeOH) 320 (ϵ 1.47 × 10^3), 313 (0.73 × 10^3), 306 (0.76 × 10^3), 288 (5.88 × 10^3), 283 (6.60 × 10^3), 278 (7.35 × 10^3), 273 (4.56 × 10^3), 268 (3.80 × 10^3), 263 (4.41 × 10^3); ^1H NMR ($CDCl_3$) δ 4.84 (s, 2 H, $-CH_2-$), 7.0–7.8 (c, 9 H, aromatic), 8.1 (c, 1 H, C-7 H), 11.7 (bs, 1 H, CO_2H). Anal. Calcd for $C_{18}H_{13}FO_2$: C, 77.13; H, 4.67; F, 6.78. Found: C, 77.04, 76.92; H, 4.64, 4.65; F, 6.73, 6.74.

2-(2-Fluoro-6-napthylmethyl)benzoic acid (5): mp 153–154 °C (benzene); IR (KBr) 1700 cm^{-1} (C=O); UV λ_{max} (MeOH) 323 (ϵ 1.73 × 10^3), 315 (1.0 × 10^3), 308 (1.26 × 10^3), 302 (8.0 × 10^2), 294 (1.0 × 10^3), 280 (5.60 × 10^3), 271 (6.66 × 10^3), 263 (5.86 × 10^3); ^1H NMR ($CDCl_3$) δ 4.60 (s, 2 H, $-CH_2-$), 7.0–7.8 (c, 9 H, aromatic), 8.13 (d, 1 H, J = 8 Hz, C-7 H), 10.0–11.0 (b, 1 H). Anal. Calcd for $C_{18}H_{13}FO_2$: C, 77.13; H, 4.67; F, 6.78. Found: C, 76.91, 76.96; H, 4.83, 4.75; F, 6.77, 6.69.

2-Fluorobenz[*a*]anthraquinone (8) and 3-Fluorbenz[*a*]anthraquinone (9). A suspension of **4** (1.0 g; 3.7 mmol) in concentrated sulfuric acid (70 mL) was stirred at room temperature for 90 min. The mixture was then poured onto crushed ice. The yellow suspension was filtered through celite, washed with water, and eluted with tetrahydrofuran (500 mL). Evaporation of the THF solution, after drying over sodium sulfate, furnished a yellow solid which was oxidized in glacial acetic acid (50 mL) with potassium dichromate (7 g) at reflux for 40 min. The mixture was poured onto ice and filtered through Celite. Elution of celite with THF (300 mL) and subsequent evaporation furnished quinone **8**, 600 mg (62% from **4**). A sample for analysis was prepared by two crystallizations from acetone. A similar reaction sequence with **5** (920 mg) furnished **9** (600 mg; 66% from **5**).

2-Fluorobenz[*a*]anthraquinone (8): mp 184–185 °C (acetone); IR (KBr) 1690 cm^{-1} (C=O); UV λ_{max} (MeOH) 395 (ϵ 2.65 × 10^3), 336 (2.65 × 10^3), 278 (2.65 × 10^4), 252 (1.43 × 10^4), 246 (1.44 × 10^4), 234 (1.44 × 10^4); ^1H NMR ($CDCl_3$) δ 7.0–8.6 (c, 8 H, aromatic), 9.4 (dd, 1 H, J_{H1-F2} = 13 Hz, J_{H1-H3} = 2.5 Hz). Anal. Calcd for $C_{18}H_9FO_2$: C, 78.25; H, 3.28; F, 6.88. Found: C, 77.29, 77.47; H, 3.26, 3.24; F, 6.92.

3-Fluorobenz[*a*]anthraquinone (9): mp 203–205 °C (acetone); IR (KBr) 1690 cm^{-1} (C=O); UV λ_{max} (MeOH) 362 (ϵ 3.7 × 10^3), 330 (3.7 × 10^3), 281 (3.24 × 10^4), 252 (1.99 × 10^4), 246 (1.99 × 10^4), 232 (1.99 × 10^4); ^1H NMR ($CDCl_3$) 7.35–8.5 (c, 8 H), 9.80 (dd, 1 H, J_{H1-H2} = 9.6 Hz, J_{H1-F3} = 6.0 Hz). Anal. Calcd for $C_{18}H_9FO_2$: C, 78.25; H, 3.28; F, 6.88. Found: C, 77.99, 77.97; H, 3.55, 3.49; F, 6.66, 6.64.

2-Fluoro-7,12-dimethylbenz[*a*]anthracene (14). A solution of quinone **8** (700 mg; 2.54 mmol) in dry benzene (200 mL) was added dropwise to a Grignard prepared from magnesium turnings (1.20 g; 0.05 mol) and CH_3I (15.0 g; 0.105 mol) in ether (100 mL). The mixture was refluxed for 10 h. Saturated ammonium chloride (50 mL) and ethyl acetate (100 mL) were added. The organic layer, after drying over sodium sulfate, was evaporated to furnish a yellow gum. The gum was dissolved in dry ethyl acetate (100 mL) and dry HCl was bubbled through the solution at 0 °C for 40 min. After an additional 2 h, the solution was evaporated to dryness. The residue was dissolved in THF (20 mL) and poured into a vigorously stirred suspension of $LiAlH_4$ (200 mg) in ether (150 mL) at room temperature under N_2. After 1 h, excess $LiAlH_4$ was destroyed by addition of saturated NH_4Cl, and the ether layer was dried over sodium sulfate and evaporated to furnish a yellow gum which on column chromatography over silica gel (hexane–benzene; 1:1) furnished pure 2F-DMBA (**14**): 210 mg (30% from **8**); mp 93–94 °C (hexane); UV λ_{max} (MeOH) 293 (ϵ 6.69 × 10^4), 283 (5.95 × 10^4), 273 (3.84 × 10^4), 260 (3.80 × 10^4), 221 (4.00 × 10^4); ^1H NMR ($CDCl_3$) δ 3.00 (s, 3 H, 7-CH_3), 3.30 (s, 3 H, 12-CH_3), 7.0–7.8 (c, aromatic), 8.0 (d, 1 H, J = 9.0 Hz, C-6 H), 8.1–8.5 (c). Anal. Calcd for $C_{20}H_{15}F$: C, 87.56; H, 5.51. Found: C, 87.41; H, 5.51.

A similar reaction sequence with quinone **9** (700 mg) furnished 3F-DMBA (**15**): 170 mg (24% from **9**); mp 113–114 °C (hexane); UV λ_{max} (MeOH) 362.5 (ϵ 0.75 × 10^3), 345 (0.75 × 10^3), 305 (1.50 × 10^3), 294 (6.90 × 10^4), 284 (6.90 × 10^4), 274 (4.68 × 10^4), 262 (4.68 × 10^4), 227 (1.90 × 10^3); ^1H NMR ($CDCl_3$) δ 3.00 (s, 3 H, 7-CH_3), 3.25 (s, 3H, 12-CH_3), 7.12–7.90 (c), 8.02 (d, C-6 H), 8.2–8.45 (c). Anal. Calcd for $C_{20}H_{15}F$; C, 87.56; H, 5.51; F, 6.93. Found: C, 87.64, 87.53; H, 5.51, 5.60; F, 7.02, 6.98.

Acknowledgment. Financial assistance from Public Health Service Grant No. CA-21371 from the National Cancer Institute and Environmental Protection Agency Grant No. R 805337 is gratefully acknowledged.

Registry No. 1, 323-09-1; 2, 71276-91-0; 3, 3799-80-2; 4, 71277-74-2; 5, 71250-14-1; 8, 71250-15-2; 9, 71250-16-3; 14, 68141-56-0; 15, 71250-17-4.

Comments on "Synthesis and Nuclear Magnetic Resonance Study of Neopentyl and (Trimethylsilyl)methyl Derivatives of Phosphorus"

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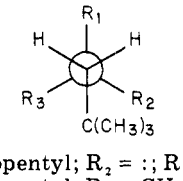
Received April 23, 1979

In a recent article, Singh and Reddy[1] deduced the preferred conformations of compounds **2**, **4**, **5**, **12**, **10**, **1**, and **11** (their numbering) by analysis of the nonequivalence of proton resonance signals of the methylene groups of neopentyl ($(CH_3)_3CCH_2$) or (trimethylsilyl)methyl ($(CH_3)_3SiCH_2$) substituents attached to phosphorus. Compounds **2**, **4**, **10**, and **12** exhibit methylene non-



2, R_1 = neopentyl; R_2 = :; R_3 = phenyl
4, R_1 = neopentyl; R_2 = CH_3; R_3 = phenyl
12, R_1 = neopentyl; R_2 = =O; R_3 = phenyl
10, R_1 = phenyl; R_2 = =O; R_3 = methyl
5, R_1 = neopentyl; R_2 = phenyl; R_3 = phenyl
1, R_1 = phenyl; R_2 = :; R_3 = phenyl
11, R_1 = phenyl; R_2 = =O; R_3 = phenyl

equivalence while compounds **1**, **5**, and **11** do not. (In the Newman projection given here, the rear atom is phosphorus; for **4** and **5** the anion is iodide.) Singh and Reddy concluded that because **2**, **4**, and **12** do not exhibit collapse of the methylene AB quartet (phosphorus coupling ig-

(1) G. Singh and G. S. Reddy, *J. Org. Chem.*, **44**, 1057 (1979).

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