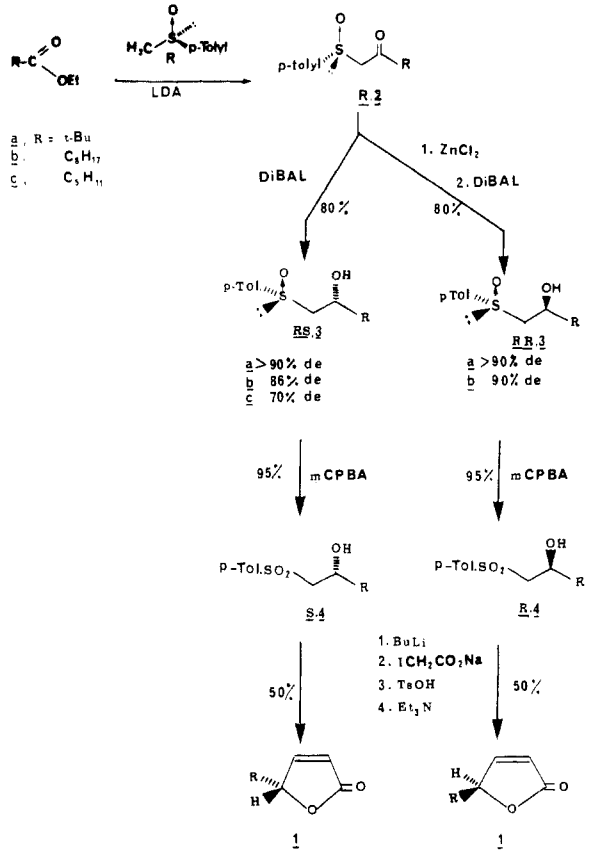
Scheme II



refluxing for 3 h in a soxhlet full of molecular sieves, the mixture was decomposed by adding a 5% aqueous solution of sodium bicarbonate (10 mL). After extraction with benzene, the organic layers were dried over sodium sulfate and concentrated to 50 mL.

(3) Triethylamine (4 equiv) was added to the solution and kept under stirring at room temperature for 15 h. The benzene solution was then washed with 10% HCl (10 mL), dried and evaporated.

The crude butenolide was finally purified by chromatography (eluent: ether/hexane 50/50). The overall yield for the three steps was around 50%.

Butenolides (*S*)-1a and (*R*)-1a. Butenolide (*S*)-**1a** was obtained from the β-hydroxy sulfone *S*-**4a** (ee > 90%) yield 50%, mp 59–61 °C; $[\alpha]_D$ +90° (dioxane, *c* 2). Anal. Calcd for $C_8H_{12}O_2$: C, 68.53; H, 8.64. Found: C, 68.37; H, 8.40. Butenolide **1a**-(*R*) was obtained from the β-hydroxy sulfone **4a**-(*R*) (ee > 90%): yield 47%, mp 59–61 °C; $[\alpha]_D$ −92° (dioxane, *c* 2); ee > 90% by RMN in presence of Eu(Tfc)$_3$ in the molar ratio (Eu)/[butenolide] = 0.4, nonequivalent *tert*-Butyl group, $\Delta\nu$ 1.5 Hz. IR ($CHCl_3$: 1740, 1160 cm^{-1}. NMR ($CDCl_3$): δ 1.0 (s, 9 H, *t*-Bu), 4.75 (m, 1 H, HC-CH=), 6.17 (dd, *J* = 5.5 Hz *J* = 2 Hz, =CHCO), 7.45 (dd, *J* = 5.5 Hz, *J* = 1.5 Hz, HC=).

Butenolides (*S*)-1b and (*R*)-1b. Butenolide (*S*)-**1b** was obtained from the β-hydroxy sulfone (*S*)-**4b** (ee = 86%); yield 45%; $[\alpha]_D$ +47° (dioxane, *c* 2).[4] There is some discrepancy with the optical rotation described in the literature probably due to the small scale experiment we performed. However, it was demonstrated in the case of butenolide **1a** that no racemization occured during the whole process.

Butenolide (*R*)-**1b** was obtained from the β-hydroxy sulfone (*R*)-**4b** (ee > 90%); yield 42%; $[\alpha]_D$ −49° (dioxane, *c* 2). IR ($CHCl_3$): 1740, 1160 cm^{-1}. NMR ($CDCl_3$): δ 0.90 (t, 3 H, CH_3), 1.20–1.85 (m, 14 H), 5.05 (m, 1 H, HCCH=), 6.12 (dd, *J* = 5.5 Hz, *J* = 2 Hz, 1 H, =HCCO), 7.45 (dd, *J* = 5.5 Hz, *J* = 1 Hz, 1 H, HC=).

Butenolide (*S*)-1c was prepared from the β-hydroxy sulfone (*S*)-**4c** (ee = 70%); yield 40%; $[\alpha]_D$ +51° (dioxane, *c* 2). Anal. Calcd for $C_9H_{14}O_2$: C, 70.08; H, 9.17. Found C, 69.41; H, 9.07. IR ($CHCl_3$): 1740, 1160 cm^{-1}. NMR ($CDCl_3$): 0.90 (t, 3 H, CH_3), 1.20–1.85 (m, 8 H), 5.05 (m, 1 H, HCCH=), 6.12 (dd, *J* = 5.5 Hz, *J* = 2 Hz, =HCCO), 7.47 (dd, *J* = 1 Hz, HC=).

Pentaphene via 1,2-Anthracyne: An Application of Repeated Aryne–Isobenzofuran Methodology

Robert Camenzind and Bruce Rickborn*

Department of Chemistry, University of California, Santa Barbara, California 93106

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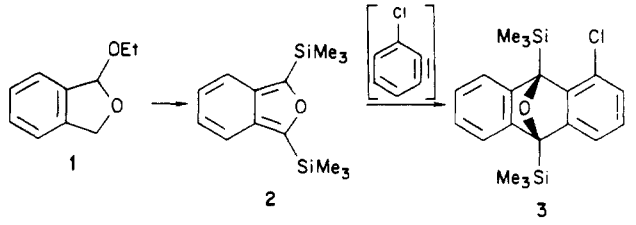
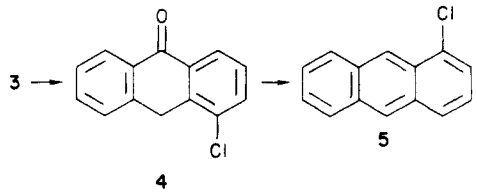
It appeared that recently developed methodology[1] which makes use of aryne cycloaddition to a protected form of isobenzofuran would allow a useful entry to the pentaphene skeleton, provided that 1,2-anthracyne could be generated. Of the various methods which in principle could be used to accomplish the formation of this reactive intermediate, strong base induced dehydrohalogenation is the only route that can avoid a potentially complex and multistep precursor synthesis. Even the 1-haloanthracenes are uncommon materials,[2] which apparently have not been examined as potential aryne-forming substrates.

A useful synthesis of 1-chloroanthracene has been developed, as outline in eq 1 and 2. By methods already described,[1] the acetal **1** was converted to 1,3-bis(trimethylsilyl)isobenzofuran (**2**); addition to the same reaction flask of *o*-dichlorobenzene and lithium tetramethylpiperidide (LTMP) generated the reactive intermediate 3-chlorobenzyne, which was efficiently trapped by **2** to give the cycloadduct **3** (62% after recrystallization).



(1)

As anticipated from the reactions of the 1-bromo and 1-methyl analogues[1b] of this material, treatment of **3** with trifluoroacetic acid (TFA) resulted in highly regioselective conversion to 4-chloroanthracen-9(10*H*)-one (**4**). This selectivity was evident from integration of the ^{1}H NMR spectrum, which exhibited a two-proton multiplet, attributed to the peri protons proximal to the carbonyl group, downfield from the major 5-proton aromatic absorption. Reduction of **4** by $LiAlH_4$ followed by acid-catalyzed dehydration gave 1-chloroanthracene (**5**) in 85% yield (based on **3**).



(2)

A separately prepared solution of **2** (1.5 equiv) was in turn treated with **5** and LTMP; that this method is suitable for the generation of 1,2-anthracyne (**6**) was shown by the isolation of the cycloadduct **7** (eq 3), as bright yellow needles (62% after recrystallization).[3]

(1) (a) Crump, S. L.; Netka, J.; Rickborn, B. *J. Org. Chem.* **1985**, *50*, 2746. (b) Netka, J.; Crump, S. L.; Rickborn, B. *Ibid.*, in press.

(2) Of the 1-haloanthracenes, only the chloride is commercially available, but the cost is prohibitive for many purposes.

(3) Anthracene itself serves well as a diene component in the Diels–Alder reaction with benzyne, and similar reaction of 1-chloroanthracene and 1,2-anthracyne is a possible competitive process in the formation of **7**. Integration of the aromatic proton region of the NMR spectrum of the crude product of eq 3 indicated that ≥70% of these absorptions are due to **7**, showing that alternative aromatics are not present in large amount.

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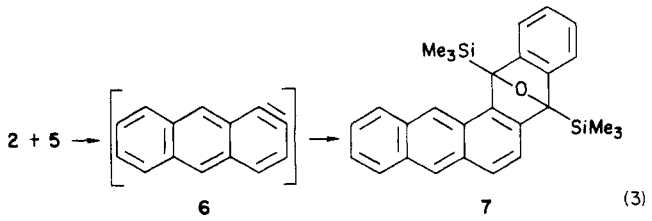
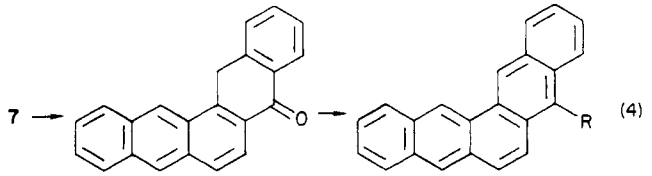


6 7 (3)

Treatment of 7 in CH_2Cl_2 solution with TFA caused facile reaction, evidenced by the rapid formation of a precipitate. Vacuum evaporation of volatiles gave a yellow solid, which was subjected to $LiAlH_4$/THF reduction followed by acidic workup. Chromatography afforded pale yellow pentacene **9** but in a disappointing 36% yield.

The reaction of 7 with TFA was repeated and the subsequent reduction carried out with $LiAlD_4$. Substantially longer times were used for both the TFA and reductions steps. These changes did not improve the yield but instead resulted in the isolation of only 13% of 5-deuteriopentaphene (**10**). The formation of **10** indicates that the hydrocarbon arises from the ketone 8, as shown in eq 4.



8 9, R=H 10, R=D (4)

Regioselective transformation of 7 to 8 was expected, on the basis of earlier work with somewhat analogous systems.[1b] It appears that the low yields of pentaphene are due to problems in this step, since the yellow solid obtained in the TFA reaction had, by ^1H NMR in THF-d_8, only a small amount (20 ± 10%) of the methylene singlet at ca. 5 ppm expected for 8. We believe that the major product of the TFA reaction is the phenolic tautomer of 8. It is not clear whether this represents an unfavorable keto/enol equilibrium position or simply lower solubility of the phenolic form. In general, we have found that ketone products are strongly favored when TFA reactions yield halocarbon-soluble materials from related substrates,[1b] in keeping with Beak's observation[4] that the keto form of anthracen-9(10*H*)-one strongly dominates the equilibrium in such solvents.

Although alternative methods to reduce the presumed pentaphenol intermediate to the hydrocarbon were considered feasible, we chose instead to focus on direct reactions of 7 and promptly observed that *Zn/HOAc treatment of 7 gives 9 in 75% yield.* Since bis(trimethylsilyl) 1,4-epoxides are the common products of this annulative procedure,[1] this finding may have wide utility in the preparation of polycyclic aromatic hydrocarbons.[5]

Pentaphene is an uncommon polycyclic aromatic hydrocarbon which has been the target of several synthetic approaches in the past.[6] The procedure outlined here compares very favorably with these. In addition, the demonstration that 1-chloroanthracene undergoes normal aryne formation on treatment with LTMP opens the way for further applications of the reactive intermediate 1,2-anthracyne.

Experimental Section

The preparation of the acetal 1 has been described previously.[7] General experimental conditions and instruments used in analyses were identical to those described in earlier work.[1] All ^1H NMR data are at 300 MHz in $CDCl_3$ solvent, unless otherwise indicated. Combustion analysis was performed by MicAnal, Tucson, AZ.

9,10-Bis(trimethylsilyl)-1-chloro-9,10-dihydro-9,10-epoxyanthracene (3). The acetal 1 (3.0 g, 18 mmol) in 50 mL of ether was treated with 0.15 mL of tetramethyliperidine and 56 mmol of *n*-butyllithium (36 mL of 1.6 M hexane solution). After 4 h, trimethylsilyl chloride (4.8 mL, 38 mmol) was added, to form a solution of **2**. This was followed within a few minutes by *o*-dichlorobenzene (3.1 mL, 27 mmol) and 37.5 mmol of LTMP (prepared separately in 25 mL of ether by addition of *n*-butyllithium to tetramethylpiperidine at 0 °C). The mixture was stirred at room temperature for 60 h and then taken up in water and extracted with ether. The combined organic phase was washed with 10% HCl and brine, dried over Na_2SO_4, and vacuum evaporated to give 6.4 g (95%) of crude product as a dark solid. This material was taken up in 135 mL of refluxing methanol, treated with decolorizing charcoal, and hot filtered. Repetition of the decolorization gave a yellow solution, which was reduced to ca. 50 mL, at which point solid began to form. After standing at room temperature and then at −10 °C for several hours, filtration gave 3.66 g (56%) of essentially pure **3** as a pale yellow solid. A second crop weighing 0.52 g (7%) was collected by reduction of the mother liquor to ca. 20 mL. A portion was recrystallized from methanol to give pure **3** as a colorless solid, mp 151.5–152.5 °C: ^1H NMR δ 0.35 (s, 9 H), 0.38 (s, 9 H), 6.84 (m, 2 H), 6.93 (m, 2 H), 7.08 (m, 1 H), 7.23 (m, 1 H), 7.33 ppm (m, 1 H); MS, *m/z* (relative intensity) 374 (4.1), 373 (3.3), 372 (9.7), 322 (19.5), 302 (35.4), 301 (39.4) 300 (100), 299 (56.5), 285 (13.9), 264 (16.9), 263 (14.6), 258 (23.3), 257 (14.6), 256 (67.3), 249 (28.1), 221 (18), 189 (13.8), 178 (30.2), 147 (51.3); calcd for $C_{20}H_{25}{}^{35}ClOSi_2$ 372.1132, found 372.1169.

1-Chloroanthracene (5). TFA (0.82 mL, 10.6 mmol) was added to a solution of 0.990 g (2.66 mmol) of **3** in 10 mL of CH_2Cl_2. A mild exotherm was noted, and no **3** was detected by TLC after 2 h. After 5 h, the volatiles were removed in vacuo, to give 616 mg (101%) of yellow solid **4**: ^1H NMR (60 MHz) δ 4.32 (s, 2 H), 7.2–7.9 (m, 5 H), 8.32 (br d, 2 H).

A solution of 99 mg (2.66 mmol) of $LiAlH_4$ in 15 mL of THF was prepared and cooled in an ice bath. Compound **4** was taken up in 5 mL of THF and added via syringe (10 min) to the stirred hydride solution. After 15 min, the excess hydride was quenched by cautious addition of 5% HCl. Concentrated HCl (ca. 5 mL) was then introduced, and the mixture was stirred for 10 min before taking up in water and extracting with CH_2Cl_2. Drying (Na_2SO_4) and rotary evaporation gave 610 mg of yellow solid; chromatography of this material (silica gel, 20% CH_2Cl_2/hexanes) gave 484 mg (85%) of very clean 1-chloroanthracene (**5**) as a pale yellow solid, mp 83–84 °C (lit.[8] mp 83.5 °C): ^1H NMR δ 7.29 (apparent t, 1 H, *J* = 7.5 Hz), 7.46 (m, 2 H), 7.52 (d, 1 H, *J* = 7 Hz), 7.85 (d, 1 *H*, *J* = 9 Hz), 7.94 (m, 1 H), 8.03 (m, 1 H), 835 (s, 1 H), 8.79 (s, 1 H); the NMR was identical with that of a commercial sample.

Further elution gave 55 mg (9%) of recovered ketone **4**; since excess hydride was employed, recovery of **4** signals (minor) competing enolate formation in the reduction step, or the presence of a small amount of the phenolic form in **4**.

5,14-Bis(trimethylsilyl)-5,14-dihydro-5,14-epoxypentaphene (7). A solution of **2** was prepared as described above, from 0.82 g (5.0 mmol) of **1**. To this was added 700 mg of **5** (3.3 mmol), followed by 6.6 mmol of LTMP. The mixture was stirred

(4) Mills, S. G.; Beak, P. *J. Org. Chem.* **1985**, *50*, 1216.

(5) The poor yield of 8 obtained in the present study suggests that 5-substituted pentaphenes would best be approached by introduction of the substituent at the stage of anthrone **4**, although we have not examined such reactions (with, e.g., Grignard reagents).

(6) (a) Clar, E. *Polycyclic Hydrocarbons*; Academic: New York, 1964; Vol. 1, p 353 and references therein. (b) Franck, H. G.; Zander, M. *Chem. Ber.* **1966**, *99*, 396. Pentaphene was obtained in ca. 2% yield by a six-step procedure starting with *o*-benzoylbenzoic acid. (c) Marsili, A.; Isola, M. *Tetrahedron Lett.* **1965**, 3023. No experimental details or yields are reported in this paper. (d) Florent'ev, V. L. *Zh. Obsch. Khim.* **1965**, *35*, 131; *Chem. Abstr.* **1965**, *62*, 13103h. An Al-Hg procedure for reduction of a bis quinone to pentaphene is described in this paper.

(7) Moss, R. J.; Rickborn, B. *J. Org. Chem.* **1982**, *47*, 5391. We thank Seid Mirsadeghi for carrying out the preparation of the acetal 1 used in the present study.

(8) *CRC Handbook of Chemistry and* Physics, 63rd ed.; Weast, R. C., Astle, M. J., Ed.; CRC: Boca Raton, FL, 1982; p C-101.

at room temperature for 20 h, and then taken up in water and extracted with ether. The organic phase was washed with 5% HCl, dried, and evaporated to give a dark solid residue (2.2 g). This was taken up in 175 mL of refluxing methanol, treated with decolorizing charcoal, and hot filtered. The resulting yellow solution was boiled down to ca. 45 mL and seeded with material obtained by evaporation of a drop of the solution. Crystals formed slowly on standing at room temperature; after several hours, filtration gave 799 mg (55%) of bright yellow **7**, mp 182-184 °C. The mother liquor was reduced to ca. 20 mL and placed in a freezer after standing at room temperature overnight to give a second crop of **7**, 107 mg, (7%).

A portion was recrystallized (methanol) to give analytically pure **7** as bright yellow needles, mp 184-185 °C: ^{1}H NMR δ 0.43 (s, 9 H), 0.55 (s, 9 H), 6.81 (m, 2 H), 7.24 (m, 1 H), 7.39 (m, 3 H), 7.55 (d, 1 H, J = 8 Hz), 7.72 (d, 1 H, J = 8 Hz), 7.89 (d, 1 H, J = 8 Hz), 7.95 (d, 1 H, J = 8 Hz), 8.38 (s, 1 H), 8.59 (s, 1 H); MS, m/z (relative intensity) 438 (18), 366 (15.2), 323 (14.5), 322 (47.5), 73 (100); MS, calcd for $C_{28}H_{30}OSi_2$ 438.1835, found 438.1845. Anal Calcd: C, 76.66; H, 6.89. Found: C, 76.44;, H, 6.89.

Pentaphene (9): (a) The TFA-LiAlH$_4$ Route. Compound **7** (109 mg, 0.249 mmol) was dissolved in 5 mL of CH_2Cl_2, in a flask equipped with a stir bar and rubber stopper, under N_2. The flask was cooled in an ice bath, and 2.1 mL of a 0.622 M solution of TFA in CH_2Cl_2 (1.31 mmol) was added dropwise. Precipitation was observed after a few minutes; stirring was maintained at room temperature for 3.5 h, and then the volatiles were removed by vacuum evaporation to give a yellow solid. A portion of this crude product (68 mg, 0.230 mmol of assumed **8**) was dissolved in 5 mL of THF and added dropwise to an ice-cooled, stirred solution of 52 mg (1.36 mmol) of $LiAlH_4$ in 5 mL of THF. A bright orange color developed. After 20 min, the excess hydride was decomposed by careful addition of 5% HCl, followed by 3 mL of concentrated HCl, with 15 min of additional stirring. The mixture was added to water, extracted with CH_2Cl_2, dried, and evaporated to afford a solid residue (80 mg). This material was chromatographed twice, first with neat CH_2Cl_2 to give 23 mg (36%) of slightly discolored pentaphene (essentially pure by NMR) and then with 40% CH_2Cl_2/hexanes, which gave a high recovery of rapidly eluted pure pentaphene **9** as yellow plates, mp 264-265 °C (lit.[6] mp 257 °C): ^{1}H NMR δ 7.57 (symmetrical m, 4 H), 7.65 (s, 2 H), 8.03 (m, 2 H), 8.15 (m, 2 H, mirror image of preceding absorption), 8.26 (s, 2 H), 9.25 (s, 2 H); the 60-MHz NMR spectrum was identical with that depicted by Martin et al.;[9] MS, m/z (relative intensity) 280 (2.7), 279 (23.8), 278 (P, 100), 277 (4.2), 276 (13.7), 275 (1.5), 274 (4.3), 139.5 (4.4), 139 (18.6), 138.5 (2.4), 138 (8.6), 137 (4.2),125 (3.6).

(b) The Zn/HOAc Method. To a 50-mL round-bottom flask equipped with a stir bar and reflux condenser were added 340 mg (0.775 mmol) of **7**, 2.0 g (31 mmol) of Zn dust, and 10 mL of glacial HOAc. The mixture was refluxed (under N_2) for 4 h and then allowed to stand at room temperature overnight (yellow crystals were observed). The mixture was then taken up in 50 mL of CH_2Cl_2 and filtered into a separatory funnel, with washings of the residual Zn and other solids. The organic phase was washed twice with water and three times with 10% KOH solution. Drying and evaporation gave 162 mg (75%) of yellow solid pentaphene (**9**), essentially pure by NMR, mp 259-260 °C. Recrystallization from HOAc returned 128 mg of golden plates, mp 263-264 °C.

The course of this reaction could not be followed by TLC because of the very similar behavior of **7** and **9**. It may be possible to improve the yield by increasing the reaction time and amount of solvent, and more thorough extraction of the solid residue (the Zn dust had coagulated into brittle pellets prior to workup).

5-Deuteriopentaphene (10). A solution of **7** (162 mg, 0.370 mmol) in 5 mL of CH_2Cl_2 was treated with 1.24 mmol of TFA in 2 mL of CH_2Cl_2. After the mixture was stirred for 15 h, the volatiles were removed in vacuo to give 110 mg of yellow solid (100% based on the formula of **8**). The ^{1}H NMR spectrum taken in THF-d_8 exhibited a small singlet at 4.98 ppm attributed to ketone **8** (estimated to account for no more than 20% of the material), a complex aromatic proton pattern (7.3-8.6 ppm), and singlets at 8.95 and 9.35 ppm. On standing (3 days) in the capped NMR tube, decomposition occurred (oxidation ?), leading to new signals in the aromatic region but no increase in the peak attributed to **8**.

A solution of 57 mg (1.36 mmol) of $LiAlD_4$ (98% D) in 15 mL of THF was treated with 97 mg of the yellow solid from the TFA reaction (dissolved in 5 mL of THF), with stirring of the bright orange solution for 4.5 h prior to workup as described above. Chromatography gave 12 mg (13%) of deuterated pentaphene **10**, mp 264-265 °C. The ^{1}H NMR of this material was identical with that of **9**, except for diminution of the singlet at 8.26 ppm to an integrated value of one H in **10**: MS, m/z (relative intensity) 281 (5.9), 280 (34.3), 279 (P, 100), 278 (5.9), 277 (13.4), 276 (3.2), 275 (4.0); the relatively high intensity of the 280 peak suggests that some dideuterated material may have been formed, although the mechanism to accomplish this is unclear.

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Registry No. **1**, 75802-19-6; **2**, 96913-93-8; **3**, 101517-29-7; **4**, 4888-02-2; **5**, 4985-70-0; **7**, 101493-04-3; **8**, 101493-05-4; **9**, 222-93-5; **10**, 101493-06-5; o-$Cl_2C_6H_4$, 95-50-1.

(9) Martin, R. H.; Defay, N.; Geerts-Evrard, F.; Delavarenne, S. Tetrahedron **1964**, *20*, 1073.

Crystal Structure of 1-(Phenoxycarbonyl)-2-(*p*-chlorophenyl)-4,5-dimethyl-1,2-dihydropyridine. Insight into the Facial Selectivity of 1,2-Dihydropyridine Diels-Alder Cycloadditions

Grant R. Krow,* Ramesh Raghavachari, and Ronald Siatkowski

Department of Chemistry, Temple University, Philadelphia, Pennsylvania 19122

Daniel F. Chodosh

Smith Kline and French Laboratories, Philadelphia, Pennsylvania 19101

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Recently, there has been considerable interest in 2-substituted-*N*-(alkoxycarbonyl)-1,2-dihydropyridines (**1**), especially as diene components in Diels-Alder reactions.[1-8] A useful stereochemical feature of such cycloadditions is

(1) For recent reviews of 1,2-dihydropyridines, see: (a) Stout, D. M.; Meyers, A. I. *Chem. Rev.* **1982**, *82*, 223-243. (b) Eisner, U.; Kuthan, J. *Ibid.* **1972**, 72, 1-42.

(2) (a) Comins, D. L.; Abdullah, A. H. *Tetrahedron Lett.* **1985**, 43-46. (b) Comins, D. L.; Abdullah, A. H.; Mantlo, N. B. *Ibid.* **1984**, 4867-4870. (c) Comins, D. L., Abdullah, A. H.; Smith, R. K. *Ibid.* 1983, 2711-2714. (d) Comins, D. L.; Abdullah, A. H. *J. Org. Chem.* **1982**, *47*, 4315-4319.

(3) (a) Natsume, M.; Utsunomiya, I.; Yamaguchi, K.; Sakai, S. I. *Tetrahedron*, **1985**, 41, 2115-2123, and references therein. (b) Natsume, M.; Ogawa, M. *Chem. Pharm. Bull.* **1982**, *30*, 3442-3445.

(4) Courtois, G.; Al-Arnaout, A.; Miginiac, L. *Tetrahedron Lett.* **1985**, 1027-1030.

(5) (a) Yamaguchi, R.; Moriyasu, M.; Yoshioka, M.; Kawanisi, M. *J. Org. Chem.* **1985**, *50*, 287-288. (b) Yamaguchi, R.; Nakazono, Y.; Kawanisi, M. *Tetradron Lett.* **1983**, 1801-1804. (c) Nakazono, Y.; Yamaguchi, R.; Kawanisi, M. *Chem. Lett.* **1984**, 1129-1132.

(6) Whitney, R. A.; Boyle, J. M. *Heterocycles* **1985**, *23*, 57-60.

(7) (a) Dubey, S. K.; Knaus, E. E. *J. Org. Chem.* **1985**, *50*, 2080-2086. (b) Knaus, E. E.; Avasthi, K.; Giam, C. S. *Can. J. Chem.* **1980**, *58*, 2447-2451. (c) Knaus, E. E.; Pasutto, R. M.; Giam, C. S.; Swinyard, E. A. *J. Heterocycl. Chem.* **1976**, 13, 481-490.

(8) (a) Krow, G. R.; Lee, Y. B.; Szczepanski, S. W.; Raghavachari, R.; Baker, A. D. *Tetrahedron Lett.* **1985**, 2617-2618. (b) Krow, G. R.; Carey, J. T.; Cannon, K. C.; Henz, K. J. *Ibid.* **1982**, 2527-2528. (c) Krow, G. R.; Carey, J. T.; Zacharias, D. E.; Knaus, E. E. *J. Org. Chem.* **1982**, *47*, 1989-1993.

0022-3263/86/1951-1916$01.50/0