In conclusion, we have shown that the use of a "chiral ligand accelerated"[1] aldehyde in the V(II)-promoted pinacol cross-coupling can lead to a highly enantioselective process. This finding makes Pedersen's innovative diol synthesis even more attractive.[24]

Experimental Section

(*S*)-2-(1-Methoxy-1-methylethyl)-1-(2-formylbenzoyl)-pyrrolidine (9). A solution of phthalic anhydride (1.48 g, 10 mmol) and (*S*)-2-(1-methoxy-1-methylethyl)pyrrolidine (1.43 g, 10 mmol), $[\alpha]^{23}_D$ −24.4 (*c* 2.3, MeOH) (lit.[13] $[\alpha]^{24}_D$ −24.5), in dry THF (50 mL) was refluxed overnight. The cooled (0 °C) solution was then treated with an ethereal solution of diazomethane to give (90% yield) the corresponding *N*-(2-carbomethoxybenzoyl)pyrrolidine after filtration through a short column of silica gel. The resulting oily material (2.75 g, 9 mmol), dissolved in refluxing *t*-BuOH (33 mL), was reduced with $NaBH_4$ (0.86 g, 22.6 mmol) by adding dropwise 6.5 mL of MeOH over a 1-h period.[25] After 1 h at reflux, H_2O (10 mL) was added to the cooled mixture, and this was extracted several times with CH_2Cl_2. Evaporation of the solvent gave the crude alcohol (86% yield) as a thick oil that was used as such. A solution of the alcohol (2.14 g, 7.7 mmol) in dry CH_2Cl_2 (20 mL) was treated with pyridinium dichromate (3.0 g, 8.0 mmol) and pulverized 4A molecular sieves (1.0 g) at room temperature for 15 h. After filtration through Celite and evaporation of the solvent the product was purified by flash chromatography with diethyl ether as eluant to give the aldehyde (1.86 g, 88% yield) as an oil; it has the following characteristics: $[\alpha]^{23}_D$ −104.6 (*c* 0.25, $CHCl_3$), IR 2940, 1690, 1590, 1380, 1080, 740 cm^{-1}; ^1H NMR δ 10.10 (s, 1 H), 7.30–7.87 (m, 4 H), 4.54 (dd, 1 H), 3.03–3.24 (m, 2 H), 3.17 (s, 3 H), 1.55–2.10 (m, 4 H), 1.15, 1.21 (2s, 6 H). $C_{16}H_{21}NO_3$ requires: C, 69.79; H, 7.69; N, 5.09. Found: C, 69.86; H, 7.76; N, 5.01.

(*S*)-2-(Diphenylhydroxymethyl)-1-(2-formylbenzoyl)-pyrrolidine (10) and (*S*)-2-[Diphenyl[(trimethylsilyl)oxy]-methyl]-1-(2-formylbenzoyl)pyrrolidine (11). Following the above described procedure, the corresponding ester, mp 177–178 °C, $[\alpha]^{23}_D$ −118.5 (*c* 1, $CHCl_3$), was prepared in 88% yield. To a stirred solution of this compound (2.075 g, 5.0 mmol) dissolved in CH_2Cl_2 (5 mL) were added TMSCl (2.5 mL, 20 mmol), triethylamine (3.5 mL, 25 mmol), and a catalytic amount of 4-(dimethylamino)pyridine in this order. The reaction was stirred at room temperature for 15 h and quenched by addition of saturated $NaHCO_3$ solution. The separated organic phase was dried and concentrated to give the crude product as a thick oil, $[\alpha]^{23}_D$ −7.5 (*c* 0.65, $CHCl_3$), that was used as such. To a stirred solution of this ester (5 mmol) in CH_2Cl_2 (10 mL) cooled at 0 °C was added a 1 M solution of diisobutylaluminium hydride in CH_2Cl_2 (15–20 mL) dropwise. After 20 min of stirring at 0 °C, the reaction was quenched by addition of a saturated NH_4Cl solution. The resulting slurry was filtered through Celite, and the organic phase was washed with water, dried, and evaporated to give the crude alcohol. This was immediately subjected to PDC oxydation (see above) to give the aldehyde. Compound **10** was obtained in 57–60% yield from the unprotected methyl ester after chromatography on silica gel with a 90:10 diethylether/hexanes mixture as eluant. It had: mp 70–72 °C; $[\alpha]^{23}_D$ −4.3 (*c* 0.3, $CHCl_3$); IR 3400, 2940, 1600, 1400, 1200, 1030, 750 cm^{-1}; ^1H NMR δ 9.87 (s, 1 H), 6.85–7.97 (m, 14 H), 6.80 (bs, 1 H), 5.27 (t, 1 H, J = 7.5 Hz), 2.65–3.05 (m, 2 H), 1.25–2.30 (m, 4 H). $C_{25}H_{23}NO_3$ requires: C, 77.90; H, 6.01; N, 3.63. Found: C, 78.03; H, 5.90; N, 3.52. Compound **11** was obtained in 50–55% overall yield from the unprotected methyl ester after short-path chromatography on Florisil with a 50:50 diethyl ether/hexanes mixture as eluant. It had: mp 56–58 °C; $[\alpha]^{23}_D$ +4.05 (*c* 0.3, $CHCl_3$); IR 2920, 1695, 1600, 1420, 1210, 1030, 750 cm^{-1}; ^1H NMR δ 9.82 (s, 1 H), 6.85–7.95 (m, 14 H), 5.30 (t, 1 H, J = 7.5 H), 2.50–3.10 (m, 2 H), 1.20–2.40 (m, 4 H), 0.05 (s, 9 H). $C_{28}H_{31}NO_3Si$ requires: C, 73.49; H, 6.83; N, 3.06. Found: C, 73.22; H, 6.71; N, 2.99.

General Procedure for the Coupling Reaction. To a stirred solution of $VCl_3(THF)_3$ (0.746 g, 2 mmol) in dry CH_2Cl_2 (5 mL) was added Zn dust (0.078 g, 1.2 mmol). After 15 min of stirring at room temperature, the solution color changed from dark red to green and the R^2CHO aldehyde (1 mmol) in CH_2Cl_2 (2 mL) was added dropwise. After 10 min of stirring at room temperature, the aromatic aldehyde (1 mmol) in CH_2Cl_2 (2 mL) was added over a 10-min period. Stirring was continued for 10–15 h, and the reaction was quenched by addition of 10 mL of a 1 N aqueous solution of HCl. When the organic layer became clear and colorless, the two phases were separated and the aqueous phase was extracted twice with CH_2Cl_2; the combined organic phases were washed with an aqueous solution of $NaHCO_3$, dried, and evaporated to give the crude diol. This was dissolved in THF (10 mL); to this solution was added PTSA (0.190 g, 1 mmol) and the mixture stirred at room temperature overnight. Solid $NaHCO_3$ was added, the mixture was filtered, and the solvent was concentrated to give the crude lactones that were purified by flash chromatography with hexanes/diethyl ether mixture as eluant. Yields, isomer ratios, and ee's are collected in Table I. Analytical and spectral data of 1(3*H*)-3-(hydroxyalkyl)isobenzofuranones **2a,b–5a,b** have been reported.[4b] Optical rotations (*c* 0.5, $CHCl_3$) of compounds of highest ee's are as follows: **2a**, −40.0 (mp 106–107 °C); **3a**, −53.0 (mp 124 °C); **4a**, −69.6 (mp 141 °C); **5a**, −49.4 (mp 133–134 °C).

Acknowledgment. Partial financial support by MURST is gratefully acknowledged.

Registry No. **1**, 131122-67-3; **2a**, 131122-72-0; **2b**, 137035-89-3; **3a**, 131122-73-1; **3b**, 137035-90-6; **4a**, 131122-75-3; **4b**, 137035-91-7; **5a**, 131122-74-2; **5b**, 137035-92-8; **7**, 118971-00-9; **9**, 137720-48-0; **10**, 137720-49-1; **11**, 137720-50-4; **12**, 89172-48-5; $R^2CHO(R^2 = Pr)$, 123-72-8; $R^2CHO(R^2 = CH_2Ph)$, 122-78-1; $R^2CHO(R^2 = Pr\text{-}i)$, 78-84-2; $R^2CHO(R^3 = C_6H_{11}\text{-}c)$, 2043-61-0; (*S*)-*N*-(2-carbomethoxybenzoyl)-2-(1-methoxy-1-methylethyl)pyrrolidine, 137720-43-5; (*S*)-*N*-[2-(hydroxymethyl)benzoyl]-2-(1-methoxy-1-methylethyl)pyrrolidine, 137720-44-6; *N*-(2-carbomethoxybenzoyl)-2-(diphenylhydroxymethyl)pyrrolidine, 137720-45-7; (*S*)-*N*-(2-carbomethoxybenzoyl)-2-[diphenyl[(trimethylsilyl)oxy]methyl]-pyrrolidine, 137720-46-8; (*S*)-2-[diphenyl[(trimethylsilyl)oxy]-methyl]-*N*-[(2-hydroxymethyl)benzoyl]pyrrolidine, 137720-47-9; phthalic anhydride, 85-44-9; 1-(2-formylbenzoyl)pyrrolidine, 84538-48-7.

(23) In this case the stereochemical purity of the alkene secures a diastereoisomerically pure 1,2-diol. The parent alkene was obtained as a 66:34 mixture of *Z*/*E* isomers.

(24) In ancilliary experiments we showed that the chiral auxiliary **8** can be used to stereocontrol the intramolecular pinacol synthesis[2] of 1,2-cyclopentane and -cyclohexane diols.

(25) Soai, K.; Oyamada, H.; Ookawa, A. *Synth. Commun.* **1982**, *12*, 463.

Attempted Synthesis of a 1,2,3,4-Tetraphenylfluoreno[1,9-*gh*]quinoline

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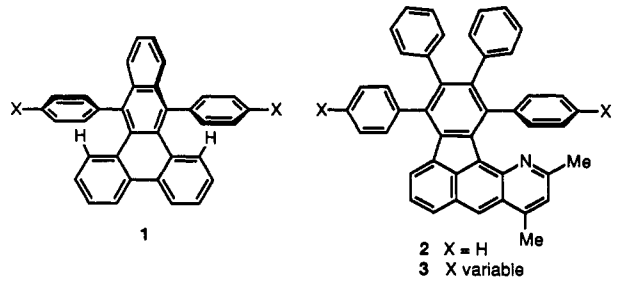
We recently described a study of the hydrogen-to-arene nonbonded interactions in the strained and twisted polycycle **1** in which the structural effects of para substitution of the phenyl groups were examined by X-ray crystallography.[1] Similar motives led us to consider close contacts between nitrogen atoms and aromatic rings, with particular interest in the structural differences resulting from the protonation state of the nitrogen and the variation of substituents on the aromatic ring.

5,7-Dimethyl-1,2,3,4-tetraphenylfluoreno[1,9-*gh*]-quinoline (**2**) was selected as a suitable framework for the juxtaposition of these functional groups, and we proposed a short synthesis (Scheme I) in which the final steps were

(1) L'Esperance, R. P.; Van Engen, D.; Dayal, R.; Pascal, R. A., Jr. *J. Org. Chem.* **1991**, *56*, 688–694.

0022-3263/92/1957-0784$03.00/0

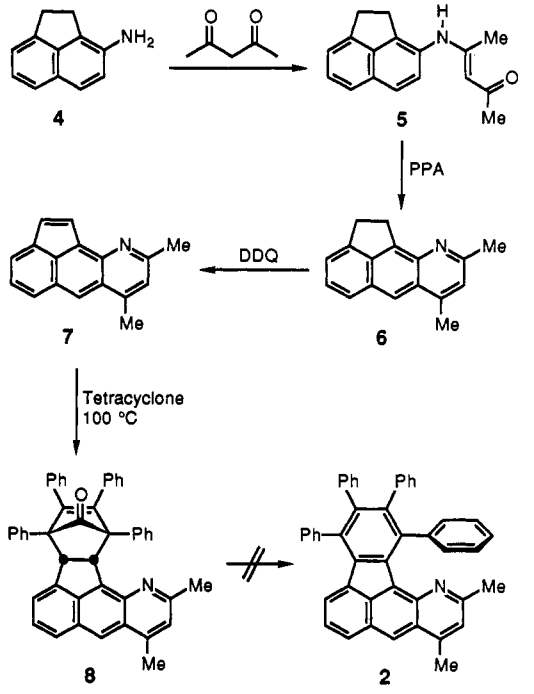


the Diels–Alder addition of 7,9-dimethyl-10-azaaceanthrylene (7) and tetracyclone followed by decarbonylation and dehydrogenation of the norbornenone 8—a frequently employed strategy for the synthesis of polycyclic aromatics via cyclopentadienones.[2] Because tetracyclone derivatives are so easily prepared, this scheme would permit the incorporation of a variety of substituents on the nitrogen-contacting phenyl ring to give a series of compounds **3**. We report here the synthesis of the desired Diels–Alder adduct **8**, our failure to convert it to **2**, the subsequent crystallographic characterization of **8**, and its structural features which frustrate this synthesis.

The azaaceanthrene **6** was assembled by a modification of the Combes quinoline synthesis.[3] For the cyclodehydration step, we found that treatment of intermediate **5** with polyphosphoric acid gave much better yields of **6** (essentially quantitative) than the more usual sulfuric acid treatment. Dehydrogenation of **6** with DDQ then gave the azaaceanthrylene **7** in moderate yield. Our samples of **7** were invariably contaminated with substantial amounts of unreacted **6**, but when excess DDQ was used for the dehydrogenation, the yield of **7** was dramatically lowered. Finally, the norbornenone **8** was formed in good yield by heating **7** and tetracyclone for several days at 80–110 °C. It was important to use recrystallized tetracyclone for this reaction since traces of base catalyzed the formation of unusual Michael adducts instead of **8**.[4]

The simplest method (though brutal) for decarbonylation and dehydrogenation of substituted norbornenones is to heat them in refluxing nitrobenzene for 24 h.[2] Typically decarbonylation is observed at ~120 °C, and the higher temperatures are required for dehydrogenation. When **8** was subjected to this treatment, only a multitude of unidentified decomposition products were observed. In the hope of separating the decarbonylation and dehydrogenation steps, we heated **8** in refluxing toluene or xylenes, but at these temperatures only a retro-Diels–Alder reaction was observed, thus yielding **7** and tetracyclone. In this regard, it is noteworthy that compound **8** dissociates upon melting (the purple color of the melt is due to tetracyclone), and in the mass spectrum of **8** the largest peaks are those due to tetracyclone (*m/z* 384) and **7** (231). In order to circumvent this problem, we attempted to dehydrogenate compound **8** to the norborna*dien*one, which would yield **2** directly by rapid decarbonylation. However, when **8** was treated with DDQ, *o*-chloranil, or palladium on carbon at relatively low temperatures no reaction occurred, and at temperatures above 100 °C the retro-Diels–Alder process was dominant. Treatment of **8** with other oxidants such as MnO_2 also failed to give the desired products, and attempted photochemical decarbonylation[5] yielded only **7**, tetracyclone, and decomposition products.

Scheme I



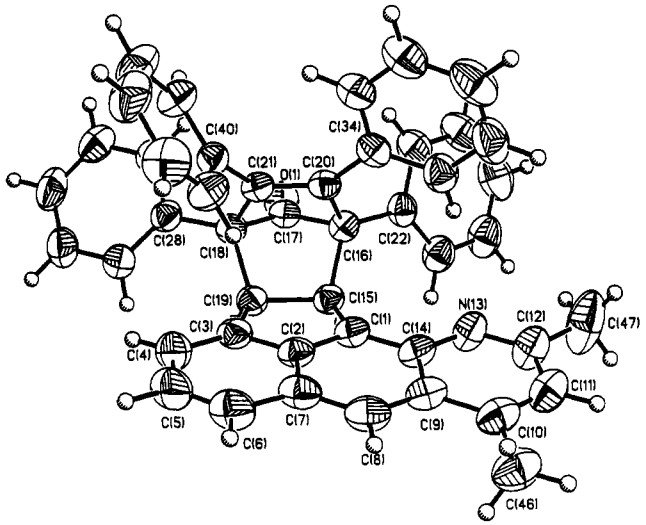
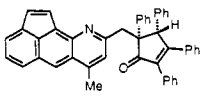


Figure 1. X-ray crystal structure of compound 8.

(2) Ogliaruso, M. A.; Romanelli, M. G.; Becker, E. I. *Chem. Rev.* **1965**, *65*, 216–367.

(3) Johnson, W. S.; Mathews, F. J. *J. Am. Chem. Soc.* **1944**, *66*, 210–215 and references cited therein.

(4) One such adduct was obtained in pure form by preparative TLC (benzene), crystallized from CH_2Cl_2, and identified as 7-methyl-9-[(1,2,3,4-tetraphenyl-5-oxocyclopent-3-enyl)methyl]-10-azaaceanthrylene by spectroscopic and X-ray analysis.



^{1}H NMR ($CDCl_3$, 270 MHz) δ 2.58 (s, 3 H, CH_3), 3.88 (d, 1 H, *J* = 12 Hz, methylene H), 4.12 (d, 1 H, *J* = 12 Hz, methylene H), 5.93 (s, 1 H, cyclopentenone methine), 6.55–7.40 (m, 21 H, aryl H), 7.58 (dd, 1 H, *J* = 8, 7 Hz, aryl H), 7.81 (d, 1 H, *J* = 7 Hz, aryl H), 7.96 (m, 2 H, aryl H), 8.48 (s, 1 H, aryl H); $^{13}C\{^1H\}$ NMR ($CDCl_3$) δ 19.5, 49.4, 58.9, 63.1, 123.3, 124.5, 126.0, 126.10, 126.13, 126.6, 126.9, 127.5, 127.6, 127.7, 127.8, 128.0, 128.2, 128.4, 128.5, 128.7, 128.8, 129.0, 129.7, 130.1, 131.4, 131.9, 134.6, 136.1, 137.6, 139.2, 140.7, 140.8, 141.4, 145.9, 160.9, 169.8, 208.4; MS *m/z* 615 (M^+, 10), 585 (M – CH_2O, 9), 384 (tetracyclone, 21), 231 (7,9-dimethyl-10-azaaceanthrylene, 100), 178 (diphenylacetylene, 24); exact mass 615.2587, calcd for $C_{46}H_{33}NO$ 615.2564. Crystal data: $C_{46}H_{33}NO$; monoclinic, space group $P2_1/a$; *a* = 11.155 (4) Å, *b* = 16.556 (6) Å, *c* = 18.742 (6) Å, β = 105.19 (3)°, *V* = 3340 (2) Å^3, *Z* = 4, D_{calc} = 1.22 g/cm^3. The collection and processing of intensity data and the solution and refinement of the structure were as described for 8. 4505 unique reflections were measured, 1906 were considered to be observed. Refinement converged at *R* = 0.099, R_w = 0.065. Full details are provided in the supplementary material.

(5) Cava, M. P.; Mangold, D. *Tetrahedron Lett.* **1964**, 1751–1754.

The X-ray crystal structure of compound 8 (Figure 1) clearly illustrates the features which oppose the formation of 2. The bonds formed in the Diels–Alder cycloaddition, C(15)–C(16) and C(18)–C(19), are quite long: 1.586 (3) and 1.571 (3) Å, respectively. While these bonds are not quite the longest ever observed in a Diels–Alder adduct,[6] compound 8 nevertheless teeters on the edge of a retro-Diels–Alder process, and the alternative reaction, the desired decarbonylation, would only force the already crowded phenyl groups closer together. It is more difficult to understand why the attempted dehydrogenation of 8 fails; however, such reactions generally require somewhat elevated temperatures, and the carbonyl and the bridgehead phenyl groups provide some steric hindrance to the approach of dehydrogenating agents to C(15) and C(19).

Experimental Section

3-Aminoacenaphthene (4) was prepared by catalytic hydrogenation of 3-nitroacenaphthene according to Friedman et al.,[7] except that 10% palladium on activated carbon was used in place of Adams' catalyst. 3-Nitroacenaphthene was prepared according to Morgan and Harrison.[8]

3-[(1-Methyl-3-oxobut-1-enyl)amino]acenaphthene (5). 3-Aminoacenaphthene (8.68 g, 51.3 mmol) and 2,4-pentanedione (13 mL, 127 mmol) were mixed and heated for 16 h at 100 °C. The mixture was cooled, and the product was recrystallized from benzene–hexanes to give compound 5 as light brown crystals (10.0 g, 39.8 mmol, 78%): mp 150–152 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.02 (s, 3 H, CH_3), 2.16 (s, 3 H, CH_3), 3.40 and 3.43 (AA'BB' system, 4 H, CH_2's), 5.26 (s, 1 H, vinyl H), 7.31 (m, 2 H, aryl H), 7.46 (t, 1 H, J = 8 Hz, aryl H), 7.61 (d, 1 H, J = 8 Hz, aryl H), 7.62 (d, 1 H, J = 8 Hz, aryl H), 12.54 (br s, 1 H, NH); MS m/z 251 (M^+, 100), 236 (M – CH_3, 50), 208 (M – $COCH_3$, 78); exact mass 251.1317, calcd for $C_{17}H_{17}NO$ 251.1310.

7,9-Dimethyl-10-azaaceanthrene (6). The enamine 5 (0.45 g, 1.79 mmol) was mixed in a Pyrex tube with polyphosphoric acid (10 g). The mixture was heated gradually to 160–170 °C with stirring. After all of the solid had dissolved (~2 h), the tube was cooled, water was added, and the reaction mixture was quenched with excess aqueous NaOH. The resulting mixture was extracted with ether (200 mL), and the extract was washed with water and dried over Na_2SO_4. Evaporation of the solvent gave the pure yellow-orange product (0.42 g, 100% yield): mp 164–166 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.75 (s, 3 H, CH_3), 2.78 (s, 3 H, CH_3), 3.56 and 3.94 (AA'BB' system, 4 H, CH_2's), 7.11 (s, 1 H, aryl H), 7.27 (d, 1 H, J = 6 Hz, aryl H), 7.47 (dd, 1 H, J = 8, 6 Hz, aryl H), 7.75 (d, 1 H, J = 8 Hz, aryl H), 8.25 (s, 1 H, aryl H); MS m/z 233 (M^+, 83), 232 (M – H, 100), 231 (M – H_2, 18), 217 (13), 189 (13); exact mass 233.1174, calcd for $C_{17}H_{15}N$ 233.1205.

7,9-Dimethyl-10-azaaceanthrylene (7). Compound 6 (0.42 g, 1.8 mmol) and DDQ (0.45 g, 2.0 mmol) were refluxed in benzene (140 mL) for 2 h. After cooling the solution was washed with 1 N NaOH followed by water. The orange organic phase was dried and concentrated to give 0.23 g of an orange solid. This material was a mixture of unreacted compound 6 (36%) and product 7 (64%), and it was used without further purification: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.78 (s, 3 H, CH_3), 2.80 (s, 3 H, CH_3), 7.10 (d, 1 H, J = 5 Hz, vinyl H), 7.11 (s, 1 H, aryl H), 7.57 (dd, 1 H, J = 8, 6 Hz, aryl H), 7.79 (d, 1 H, J = 6 Hz, aryl H), 7.85 (d, 1 H, J = 5 Hz, vinyl H), 7.95 (d, 1 H, J = 8 Hz, aryl H), 8.51 (s, 1 H, aryl H). Various runs gave product purities of 40–70% with starting material 6 as the sole contaminant; however, the use of excess DDQ gave significantly lower yields.

5,7-Dimethyl-13-oxo-1,2,3,4-tetraphenyl-4a,12c-dihydro-1,4-methanofluoreno[1,9-*gh*]quinoline (8). Compound 7 (0.44 g, 45% pure, 0.86 mmol of 7), tetracyclone (0.80 g, 2.1 mmol, recrystallized from a 1:1 mixture of 95% ethanol and toluene), and benzene (5 mL) were placed in a Pyrex tube and heated to 100 °C for 84 h. After cooling, the solution was chromatographed on a silica gel column (benzene). The first major component to elute after unreacted tetracyclone was pure Diels–Alder adduct 8 (0.43 g, 0.70 mmol, 81% based on 7), a light yellow solid. Single crystals for X-ray analysis were obtained from CH_2Cl_2–methanol: mp 191–193 °C (purple liquid); ^{1}H NMR ($CDCl_3$), 270 MHz) δ 2.55 (s, 3 H, CH_3), 2.71 (s, 3 H, CH_3), 5.02 (d, 1 H, J = 7 Hz, benzylic methine), 5.70 (d, 1 H, J = 7 Hz, benzylic methine), 5.81 (dd, 2 H, J = 7, 1 Hz, aryl H), 6.03 (dd, 2 H, J = 7, 1 Hz, aryl H), 6.29 (t, 2 H, J = 8 Hz, aryl H), 6.53 (t, 1 H, J = 7 Hz, aryl H), 6.64 (t, 2 H, J = 8 Hz, aryl H), 6.82 (t, 1 H, J = 7 Hz, aryl H), 7.02 (s, 1 H, aryl H), 7.15–7.42 (m, 6 H, aryl H), 7.50 (t, 2 H, J = 7 Hz, aryl H), 7.74 (m, 3 H, aryl H), 7.90 (dd, 2 H, J = 8, 2 Hz, aryl H), 8.26 (s, 1 H, aryl H); ^{13}C{^{1}H} NMR ($CDCl_3$) δ 19.5, 24.5, 49.0, 50.4, 67.7, 68.3, 119.9, 121.4, 121.9, 123.8, 125.9, 126.3, 126.4, 126.5, 126.6, 127.0, 127.2, 127.5, 127.8, 128.1, 128.8, 130.4, 130.5, 131.0, 131.9, 133.6, 134.5, 134.6, 135.7, 140.4, 141.6, 141.7, 142.0, 143.3, 143.7, 144.3, 158.3, 202.2; MS m/z 615 (M^+, 0.2), 587 (M – CO, 6), 384 (tetracyclone, 82), 231 (7,9-dimethyl-10-azaaceanthrylene, 100), 178 (diphenylacetylene, 83); exact mass 615.2554, calcd for $C_{46}H_{33}NO$ 615.2564.

X-ray Crystallographic Analysis of Compound 8. A single crystal of 8·CH_2Cl_2 measuring 0.04 mm × 0.29 mm × 0.70 mm was used for X-ray measurements. Crystal data: $C_{46}H_{33}NO$·CH_2Cl_2; triclinic, space group $P\bar{1}$; a = 10.778 (3) Å, b = 11.751 (3) Å, c = 15.034 (5) Å, α = 90.54 (2)°, β = 106.45 (2)°, γ = 95.83 (2)°, V = 1815.2 (9) Å^3, Z = 2, D_{calc} = 1.28 g/cm^3. Intensity measurements were made with $3° \le 2\theta \le 114°$ by using graphite-monochromated Cu Kα radiation (λ = 1.541 78 Å) at room temperature on a Nicolet R3m diffractometer. Of a total of 4556 unique reflections, 3929 were considered to be observed [$|F_o| > 3\sigma(F_o)$]. The structure was solved by direct methods and refined by using the SHELXTL software. In the final stages of refinement, all non-hydrogen atoms were refined with anisotropic temperature factors, and a riding model with idealized geometry was used for the hydrogens. Refinement converged at R = 0.054, R_w = 0.064. Full details are provided in the supplementary material.

Acknowledgment. This work was supported in part by National Science Foundation Grant CHE-9106903.

Registry No. 2, 137718-26-4; 4, 55939-13-4; 5, 137695-60-4; 6, 137695-61-5; 7, 137695-62-6; 8, 137718-28-6; $CHCOCH_2COCH_3$, 123-54-6; tetracyclone, 479-33-4; 7-methyl-9-[(1,2,3,4-tetraphenyl-5-oxocyclopent-3-enyl)methyl]-10-azaaceanthrylene, 137695-63-7.

Supplementary Material Available: NMR spectra of 5, 6, and 8 and single-crystal structure reports for compound 8 and 7-methyl-9-[(1,2,3,4-tetraphenyl-5-oxocyclopent-3-enyl)-methyl]-10-azaaceanthrylene (27 pages). This material is contained in many libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

(6) To the best of our knowledge, the current "record" is held by the Diels–Alder adduct of tetrakis(trifluoromethyl)-5-thiabicyclo[2.1.0]-pent-2-ene and tetramethylfuran, where the bond lengths of interest are 1.64 (2) and 1.63 (2) Å: Kikutani, N.; Iitaka, Y.; Kobayashi, Y.; Kumadaki, I.; Ohsawa, A.; Sekine, Y. *Acta Crystallogr., Sect. B* **1975**, *31*, 1478–1480.

(7) Friedman, O.; Gofstein, R.; Seligman, A. *J. Am. Chem. Soc.* **1949**, *71*, 3010–3012.

(8) Morgan, G.; Harrison, H. *J. Soc. Chem. Ind.* **1930**, *49*, 413T–415T.

Production of Aldehydes via Electrochemical Reduction of Acyl Halides at Mercury and Carbon Cathodes in Acetonitrile

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There have been few publications[1-4] concerning the electrochemistry of acyl halides in nonaqueous media.

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