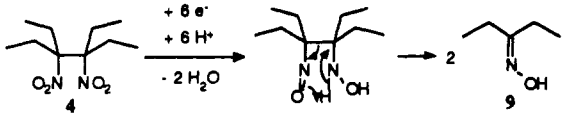
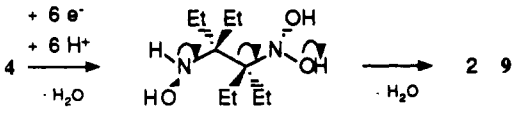
Scheme II

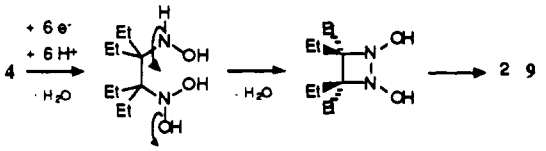
a) pericyclic rearrangement after 6-electron reduction



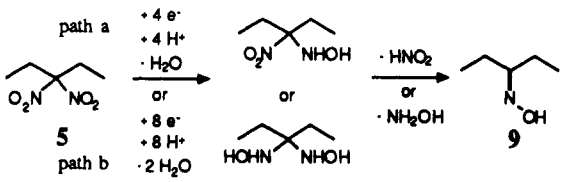
b) *trans*-elimination after 6-electron reduction



c) cleavage of diazetine after 6-electron reduction



Scheme III



week period. These results suggest that the oxime can form from the geminal dinitro compound **5** as well as from **4** during the reduction step. Accordingly, reduction of pure **5** under these conditions did give **9** in 85% yield.

Decomposition during reduction of **4** to **8** may result from an increase in the reduction potential, as electron-donating alkyl groups are added to the vicinal dinitro backbone and/or from an increased tendency for intramolecular decomposition imposed by the rotational rigidity of **4**. Scheme II shows mechanisms by which oxime **9** might form during reduction of the vicinal dinitro compound **4**. A six-electron reduction of **4** could produce the nitrosohydroxylamine, and then pericyclic rearrangement with cleavage of the central carbon–carbon bond would give 2 equiv of **9** (a). Both nitroso species and hydroxylamines have been proposed as intermediates during metal–halide reductions of nitro compounds.[3] A six-electron reduction of **4** to give the (hydroxylamino)dihydroxyamine followed by trans elimination of water gives 2 equiv of **9** (b). A third possibility is rearrangement of the six-electron reduction product to give a 1,2-diazetidine, which then cleaves to give **9** (c). Based on the work of Shustov and co-workers on oxidative cleavage of vicinal bis(hydroxylamines),[19] the trans elimination (b) appears most probable for reductive cleavage of vicinal dinitro compounds.

Scheme III shows two routes by which oxime **9** might form from the *gem*-dinitro compound **5**. Either a four-electron reduction of **5** followed by elimination of nitrous acid (path a) or an eight-electron reduction followed by loss of hydroxylamine (path b) gives **9**, which is apparently inert to further reduction by tin in strong acid.[20]

Conclusion

Tetraalkyl-substituted primary vicinal diamines are accessible from nitroalkyls via dimerization followed by reduction of the resultant vicinal dinitroalkyls. The geminal dinitroalkyl appears to be an intermediate in formation of the tetraethyl-substituted vicinal dinitro compound; further mechanistic studies are necessary to determine the generality of geminal dinitro intermediates in the Shechter reaction. Both inductive effects and rotational rigidity may make reduction of the tetraethyl-substituted dinitro compound subject to side reactions. The oxime, isolated as a side product from this reduction, is not a reaction intermediate; however, its formation supports the intermediacy of hydroxylamines in the reduction of nitro compounds by metals in mineral acids.

(19) Shustov, G. V.; Tavakalyan, N. B.; Shustova, L. L.; Pleshkova, N. B.; Kostyanovskii, R. G. *Bull. Acad. Sci. USSR Div. Chem. Sci.* **1982**, *31*, 330.

(20) It is unknown whether the trace 3-amino-3,4-diethyl-4-hexene that was also isolated from the supernatant of **10** resulted from elimination of an ammonium cation during reduction of the vicinal dinitro compound or whether it represents the reduction product of a trace quantity of 3,4-diethyl-3-nitro-4-hexene in the starting dinitro mixture.

Synthesis of Optically Active 3-(1-Hydroxyalkyl)phthalides by Stereoselective Pinacol Cross-Coupling

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Received July 19, 1991

The vanadium(II)-promoted reaction between an aliphatic aldehyde and a "ligand accelerated" aliphatic or aromatic aldehyde recently reported by Pedersen et al.[1] represents the first synthetically useful approach to 1,2-diols via intermolecular[2] pinacol cross-coupling.[3] By the use of a chiral, nonracemic amide function as in **1** we were able to establish an enantioselective version of this extremely convenient process that allowed the synthesis of 3-(1-hydroxylalkyl)phthalides **2a,b–5a,b** in 75–84% enantiomeric excess (ee) and up to 91:9 syn/anti diastereoisomeric ratio[4] via the corresponding dihydroxyamides.

However, for the pinacol coupling to be a successful competitor with other methods to produce optically active 1,2-diols,[5-11] a higher level of enantiocontrol was required

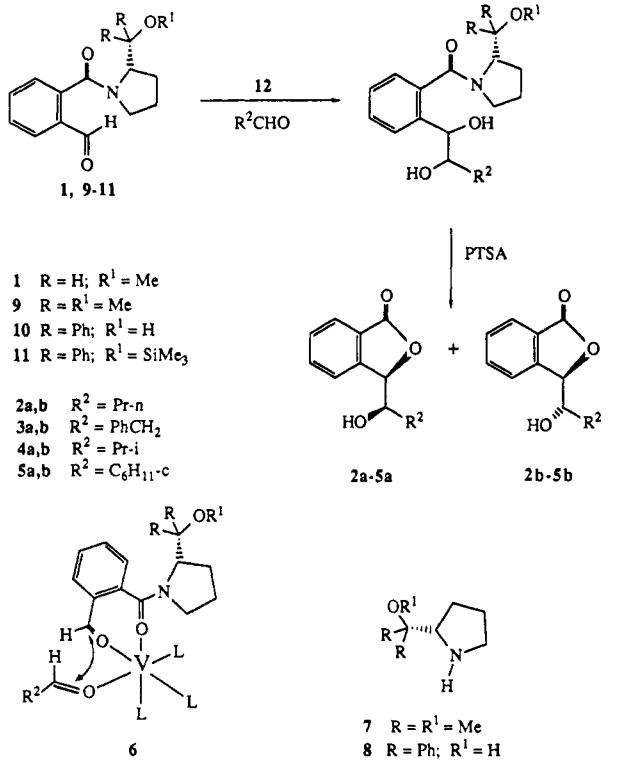
(1) (a) Freudenberger, J. H.; Konradi, A. W.; Pedersen, S. F. *J. Am. Chem. Soc.* **1989**, *111*, 8014. (b) Takahara, P. M.; Freudenberger, J. H.; Konradi, A. W.; Pedersen, S. F. *Tetrahedron Lett.* **1989**, 7177. (c) Park, J.; Pedersen, S. F. *J. Org. chem.* **1990**, *55*, 5924. (d) Konradi, A. W.; Pedersen, S. F. *J. Org. Chem.* **1990**, *55*, 4506.

(2) For an intramolecular version of this reaction see: Raw, A. S.; Pedersen, S. F. *J. Org. Chem.* **1991**, *56*, 830.

(3) Reviews: McMurry, J. E. *Chem. Rev.* **1989**, *89*, 1513. Pons, J. M.; Santelli, M. *Tetrahedron* **1988**, *44*, 4295.

(4) (a) Annunziata, R.; Cinquini, M.; Cozzi, F.; Giaroni, P. *Tetrahedron: Asymmetry* **1990**, *1*, 355. (b) Annunziata, R.; Cinquini, M.; Cozzi, F.; Giaroni, P.; Benaglia, M. *Tetrahedron* **1991**, *47*, 5737. In the original model the attack of the ketyl radical of the aliphatic aldehyde on **1** was hypothesized. However, as pointed out by one of the reviewers, attack of the ketyl radical of **1** on the aliphatic aldehyde could take place as well.

0022-3263/92/1957-0782$03.00/0



in this reaction. On the basis of the tentative model of stereoselection **6** that we proposed[4b] to rationalize our results, we reasoned that a pyrrolidine bearing a bulkier residue at C-2 should shield more efficiently the *re* face of the aromatic aldehyde **1**, thus leading to phthalides of high enantiomeric purity.[12] (*S*)-2-(1-Methoxy-1-methylethyl)pyrrolidine (**7**), easily obtained from proline,[13] and commercially available (*S*)-2-(diphenylhydroxymethyl)-pyrrolidine (**8**)[14] were selected and converted into aldehydes **9**, **10**, and **11**. These were coupled[1,4] with a series of aliphatic aldehydes in the presence of the vanadium(II) species $[V_2Cl_3(THF)_6]_2[Zn_2Cl_6]$ (**12**),[15] prepared in situ[1] from $VCl_3(THF)_3$[16] and Zn dust,[17] to give, after acidic workup,[18] phthalides **2a,b–5a,b** as mixtures of diastereoisomers. Yields, diastereoisomeric ratios (as determined by 300-MHz ^{1}H NMR spectroscopy), and ee's (evaluated by LSR techniques)[19] are collected in Table I and compared with those obtained[4] starting from **1**.[20]

Table I. Stereoselective Synthesis of Lactones 2a,b–5a,b by Coupling Amides 1, 9–11 with R²CHO

amides	R^2	product	yield[a] (%)	diastereoisomeric ratio a:b[b]	ee[c]
1	n-Pr	**2a,b**	62	80:20	77
9	n-Pr	**2a,b**	61	90:10	93
10	n-Pr	**2a,b**	61	86:14	92
11	n-Pr	**2a,b**	67	85:15	96
1	$PhCH_2$	**3a,b**	70	91:9	75
9	$PhCH_2$	**3a,b**	72	92:8	95
10	$PhCH_2$	**3a,b**	71	93:7	91
11	$PhCH_2$	**3a,b**	73	90:10	94
1	i-Pr	**4a,b**	62	89:11	84
9	i-Pr	**4a,b**	60	91:9	93
10	i-Pr	**4a,b**	62	86:14	96
11	i-Pr	**4a,b**	61	85:15	94
1	c-C_6H_{11}	**5a,b**	55	88:12	83
9	c-C_6H_{11}	**5a,b**	58	81:19	88
10	c-C_6H_{11}	**5a,b**	83	85:15	92
11	c-C_6H_{11}	**5a,b**	70	85:15	92

[a] Overall yield from 1, 9–11. [b] As determined by 300-MHz ^{1}H NMR spectroscopy. [c] Of major isomers. In some cases the ee of the minor isomers was determined and found to range from 86 to ≥96%.

As can be seen from the reported data the change in the chiral auxiliary led to the expected increase in the ee's to values generally higher than 90%, while the diastereoselectivity was affected at a lower extent. Both of these effects were predictable on the basis of model **6**, in which an increase of the bulkiness of the substituent at C-2 on the pyrrolidine moiety does not seem capable of influencing the mode of attack on the aromatic aldehyde. It must be noted that the similar results obtained with amides **10** and **11** seem to indicate that the very sterically hindered oxygenated group of these chiral auxiliaries exerts a negligeable effect on the stereoselection.[22] Worth of mention is the fact that compound **2a** can be obtained by this approach with an ee (≥96%) that is superior to that (80%) observed for the same compound prepared by stoichiometric osmylation of the corresponding (*E*)-alkene[20] in the presence of acetyldihydroquinidine.[6,23]

(5) These methods include osmylation,[6] epoxide opening,[7] reduction and alkylation of α-alkoxycarbonyls,[8] addition of α-alkoxyanions to aldehydes,[9] and oxidative elaboration of α-alkoxyborates[10] and α-alkoxysilyl derivatives.[11] The first two approaches require alkene geometry control, while the others need enantiomerically pure substrates or reagents.

(6) Reviews: Nakajima, M.; Tomioka, K.; Koga, K. *J. Synth. Org. Chem. Jpn.* **1989**, *47*, 878. Recent reports: Corey, E. J.; Da Silva Jardine, P.; Virgil, S.; Yuen, P.-W.; Connell, R. D. *J. Am. Chem. Soc.* **1989**, *111*, 9243. Oishi, T.; Hirama, M. *J. Org. Chem.* **1989**, *54*, 5834. Tomioka, K.; Nakajima, M.; Koga, K. *Tetrahedron Lett.* **1990**, 1741. Kuong, H.-L.; Sorato, C.; Ogino, Y.; Chen, H.; Sharpless, K. B. *Tetrahedron Lett.* **1990**, 2999. Kim, B. M.; Sharpless, K. B. *Tetrahedron Lett.* **1990**, 3003.

(7) (a) Review: Ko, S. Y.; Lee, A. W. M.; Masamune, S.; Reed, L. A., III; Sharpless, K. B.; Walker, F. J. *Science* **1983**, *220*, 949. Recent reports: (b) Beherens, C. H.; Ko, S. Y.; Sharpless, K. B.; Walker, F. J. *J. Org. Chem.* **1985**, *50*, 5687. (c) Matsumoto, T.; Kitano, Y.; Sato, F. *Tetrahedron Lett.* **1988**, 5685. (d) Prestwich, G. D.; Graham, S. McG.; Konig, W. A. *J. Chem. Soc., Chem. Commun.* **1989**, 575.

(8) (a) Eliel, E. L. *Asymmetric Synthesis*; Morrison, J. H., Ed.; Academic Press: New York, 1985; Vol. 2, p 125. (b) Reetz, M. T. *Angew. Chem., Int. Ed. Engl.* **1984**, *23*, 556.

(9) Review: Seebach, D. *Angew. Chem., Int. Ed. Engl.* **1979**, *18*, 239. Recent reports: Hoppe, D.; Hintze, F.; Tebben, P. *Angew. Chem., Int. Ed. Engl.* **1990**, *29*, 1422. Marshall, J. A.; Welmaker, G. S.; Gung, B. W. *J. Am. Chem. Soc.* **1991**, *113*, 647.

(10) Review: Matteson, D. S. *Acc. Chem. Res.* **1988**, *21*, 294. Recent reports: Matteson, D. S.; Tripatny, P. B.; Sarkar, A.; Sadhu, K. M. *J. Am. Chem. Soc.* **1989**, *111*, 4399.

(11) Enders, D.; Makai, S. *Helv. Chim. Acta* **1990**, *73*, 1833.

(12) For recent enantioselective syntheses of phthalides see: (a) Commerçon, M.; Mangeney, P.; Tejero, T.; Alexakis, A. *Tetrahedron: Asymmetry* **1990**, *1*, 207. (b) Soai, K.; Hori, H.; Kawahara, M. *Tetrahedron: Asymmetry* **1991**, *2*, 253.

(13) Nakayama, K.; Thompson, W. J. *J. Am. Chem. Soc.* **1990**, *112*, 6936.

(14) For the synthesis of a series of (*S*)-2-(diarylhydroxymethyl)-pyrrolidines see: Mathre, D. J.; Jones, T. K.; Xavier, L. C.; Blacklock, T. J.; Reamer, R. A.; Mohan, J. J.; Turner Jones, E. T.; Hoogsteen, K.; Baum, M. W.; Grabowski, E. J. J. *J. Org. Chem.* **1991**, *56*, 751.

(15) Cotton, F. A.; Duraj, S. A.; Roth, W. J. *Inorg. Chem.* **1985**, *24*, 913 and references cited therein.

(16) Manzer, L. E. *Inorg. Synth.* **1982**, *21*, 135.

(17) We also tested the complex $[V_2Cl_3(THF)_6][AlCl_2Et_2]$ prepared from $VCl_3(THF)_3$ and $AlEt_2(OEt)$ as described by: Cotton, F. A.; Duraj; S. A.; Manzer, L. E.; Roth, W. J. *J. Am. Chem. Soc.* **1985**, *117*, 3850. This complex, however, gave lower yields in the coupling reaction.

(18) We showed[4] that this procedure did not alter the stereochemical result: indeed, in some cases the mixtures of diols were isolated and their diastereoisomeric ratios determined by 300-MHz ^{1}H NMR spectroscopy at 60 °C (to avoid problems related to slow rotation around the amide bond); the ratios agreed well with those determined for the corresponding lactones. The lactone formation allowed recovery of the chiral auxiliary in good yield. For instance, in the synthesis of **2a,b** from **10**, **8** was recovered in 70% yield.

(19) Racemic samples were prepared from 1-(2-formylbenzoyl)-pyrrolidine and R²–CHO in the presence of **12**.

(20) The relative and absolute stereochemistry of **2a** has been established by chemical correlation:[4] the syn configuration by osmylation of the corresponding (*E*)-alkene; the *R,R* configuration by converting **2a** into (*S*)-3-butylphthalide.[21] Chemical shift trend considerations and analogy in optical rotation signs strongly suggested the indicated structure for **3a,b–5a,b**.[4b]

(21) Asami, M.; Mukaiyama, T. *Chem. Lett.* **1980**, 17.

(22) The possibility that the O–Si bond in **11** can be cleaved by the Lewis acidic **12** before coupling takes place cannot be ruled out.

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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Received September 16, 1991

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