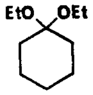
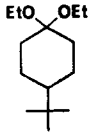
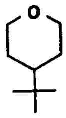
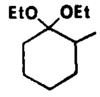
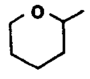
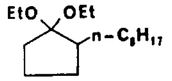
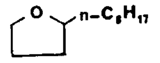
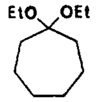
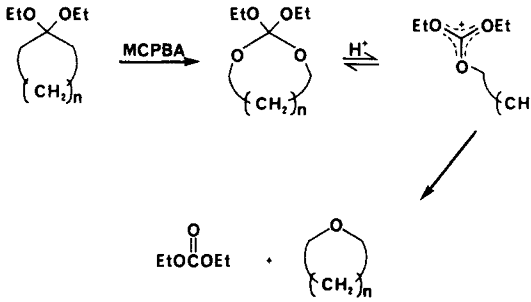
Table I. Oxidation of Ketals Derived from Five- and Six-Membered Ring Ketones[a]

entry	ketal	ether	yield,[b,c] %
1			28
2			63 (40)
3			20
4			44
5			6.4 (5.2)
6			0

[a] Oxidations were conducted as described in ref 2 (3-4 molar equiv MCPBA in CH_2Cl_2). Following complete consumption of the ketal, the product mixture was stirred for several hours at reflux in the presence of the *m*-chlorobenzoic acid generated from MCPBA. [b] Absolute yields determined either by GLC or ^1H NMR analysis using internal standards. [c] Isolated yield of purified material in parentheses.

Scheme I



to internal Me_4Si and peak multiplicities are reported for off-resonance, proton-decoupled spectra. Analytical gas-liquid chromatography (GLC) was effected with a Perkin-Elmer Model 3920-B instrument fitted with a 10-ft, 10% FFAP on Chromosorb W (60/80 mesh) column. Area ratios were determined with a Linear Instrument Model 282 recording integrator, and all yields determined by GLC (Table I) have been corrected for detector response under the conditions of the analysis by using weighed samples of pure product and hydrocarbon standard. Microanalyses were performed by Galbraith Laboratories, Inc., Knoxville, TN.

Methylene chloride was distilled from P_2O_5 prior to use, and commercial (80-85%) *m*-chloroperoxybenzoic acid (Aldrich) was employed in the oxidations. Commerical samples of cyclopentanone, cyclohexanone, cycloheptanone, 2-methylcyclohexanone, and 4-*tert*-butylcyclohexanone were purified before use, and 2-*n*-octylcyclopentanone was prepared as previously described.[5] Diethyl ketals were prepared by a standard method[6] from equimolar amounts of ketone and triethyl orthoformate dissolved in excess anhydrous ethanol containing a catalytic quantity of anhydrous HCl generated by addition of acetyl chloride to the dry ethanol. With the exception of 4-*tert*-butyltetrahydropyran, the cyclic ethers generated in the oxidations (Table I) are known compounds and structures were established by comparison of the physical and spectroscopic properties of isolated material with those reported for 2-*n*-octyltetrahydrofuran[7] or available from authentic samples of tetrahydrofuran, tetrahydropyran, and 2-methyltetrahydropyran.

Oxidations were conducted using 3-4 molar equiv of *m*-chloroperoxybenzoic acid as previously described,[2] and, following complete consumption of the ketal, the product mixture was stirred for several hours at reflux in the presence of the *m*-chlorobenzoic acid generated from the peroxy acid. The following preparation is illustrative of the general procedure.

4-*tert*-Butyltetrahydropyran. A flame-dried, three-necked, round-bottomed flask fitted with a reflux condenser, addition funnel, efficient stirrer, and argon inlet was charged with 40.6 g of 80-85% *m*-chloroperoxybenzoic acid (180-220 mmol) in 480 mL of dry CH_2Cl_2. The suspension was rapidly stirred under argon, and 11.40 g (50 mmol) of 1,1-diethoxy-4-*tert*-butylcyclohexanone was added over a 10-min period. The stirred mixture was heated at gentle reflux for 8.5 h and then rapidly poured into 500 mL of vigorously stirred, ice-cold 2.5% aqueous NaOH solution. The organic phase was separated and the aqueous phase was extracted with two 250-mL portions of diethyl ether. The combined organic extracts were washed successively with two 150-mL portions of 15% aqueous Na_2SO_3 and 150 mL of brine, dried (K_2CO_3), and concentrated by rotary evaporation. Distillation of the residue through a short, vacuum-jacketed Vigreux column afforded 2.81 g (40%) of product: bp 70-72 °C (24 mm); ^1H NMR ($CDCl_3$) δ 0.88 (s, 9 H), 0.97-1.75 (m, 5 H), 2.92-3.52 (m, 2 H), 3.65-4.12 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 27.00 (q, CH_3), 27.72 (t, C(3,5)), 32.15 (s, $C(CH_3)_3$), 45.79 (d, C(4)), 68.67 (t, C(2,6)). Anal. Calcd for $C_9H_{18}O$: C, 76.00; H, 12.75. Found: C, 76.13; H, 13.14.

Acknowledgment. This work was supported by a grant from the Humphrey Chemical Company, North Haven, CT.

Registry No. **1** (ketal), 1670-47-9; **1** (ether), 142-68-7; **2** (ketal), 1900-58-9; **2** (ether), 96964-38-4; **3** (ketal), 1528-18-3; **3** (ether), 10141-72-7; **4** (ketal), 23786-93-8; **4** (ether), 109-99-9; **5** (ketal), 96964-37-3; **5** (ether), 5921-92-6; **6** (ketal), 1130-34-3; **6** (ether), 592-90-5; methylene chloride, 75-09-2; *m*-chloroperoxybenzoic acid, 937-14-4; cyclopentanone, 120-92-3; cyclohexanone, 108-94-1; cycloheptanone, 502-42-1; 2-methoxycyclohexanone, 583-60-8; 4-*tert*-butylcyclohexanone, 98-53-3; 2-*n*-octylcyclopentanone, 40566-23-2; triethyl orthoformate, 122-51-0; ethanol, 64-17-5; acetyl chloride, 75-36-5.

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(5) Hajek, M.; Malek, J. *Collect. Czech. Chem. Commun.* **1976**, *41*, 746.
(6) Carswell, J. E.; Adkins, J. *J. Am. Chem. Soc.* **1928**, *50*, 235.
(7) Elad, D.; Youssefyeh, R. D. *J. Org. Chem.* **1964**, *29*, 2031.

Electroreduction of Cyclobutadienopleiadiene. Cathodic Hydrogenation of a Fused Cyclobutene Ring

Ronald L. Blankespoor

Department of Chemistry, Calvin College, Grand Rapids, Michigan 49506

Received January 21, 1985

With the availability of numerous chemical reducing agents, the electrochemical reduction of unsaturated hydrocarbons to their corresponding dihydro derivatives has traditionally played only a minor role as a synthetic tool. One exception to this, however, is the Birch reduction of appropriately substituted aromatic compounds to 1,4-cyclohexadienes, which can be accomplished in high yield at a mercury electrode in aqueous electrolyte[1,2] and thereby

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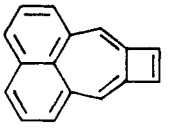
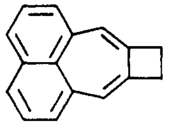
(1) Coleman, J. P.; Wagenknecht, J. H. *J. Electrochem. Soc.* **1981**, *128*, 322, and references cited therein.

serves as an alternative to the chemical method.[3] Since the two reduction methods produce the same dihydro products, it has been suggested that the mechanism for the conversion of the aromatic ring to 1,4-cyclohexadiene is the same regardless of the electron source.[2] Recently, Farnia and co-workers examined the reduction of some phenyl substituted indenes at a mercury electrode.[4] They observed two different $2e^-/2H^+$ processes, depending upon the presence of more or less acidic proton donors: protonation of the radical anion formed in the heterogeneous electron transfer and protonation of the dianion formed by disproportionation of the radical anion.

It was our aim in this work to study the electrochemical reduction of an unsaturated hydrocarbon to its dihydro derivative to gain some insight into the mechanism for this "cathodic hydrogenation". Cyclobutadienopleiadiene (1)

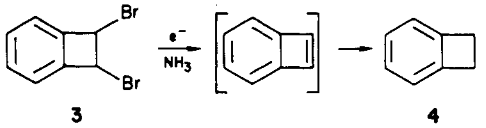


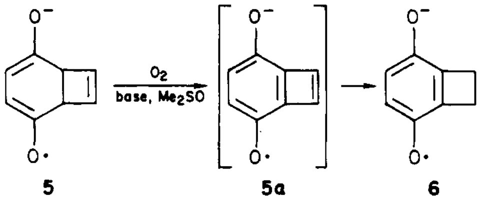
1



2

was selected as the substrate for several reasons. First, its dihydro derivative, cyclobutenopleiadiene (2), undergoes a one-electron reduction to its radical anion $2^{-\cdot}$ at a half-wave potential ($E_{1/2}$) of −1.91 V (SCE).[5] Since 1 has a more extended π system than 2, its $E_{1/2}$ would be expected to be slightly more positive than −1.91 V, which would allow the reduction of 1 to be carried out at potentials where electrolytes such as $LiClO_4$ and tetraalkylammonium salts are not electroactive. Studies of low-lying electronic states of hydrocarbons derived from pleiadiene support this expectation.[6] Second, there are several reports in the literature describing the rapid reduction of a fused cyclobutene, such as is found in 1, to a fused cyclobutane in basic, nonaqueous media. Thus, treatment of 1,2-dibromobenzocyclobutene (3) with solvated electrons in ammonia produces benzocyclobutene (4) via the proposed intermediate benzocyclobutadiene.[7] Another rather intriguing example of this transformation is the air oxidation of 5 to intermediate 5a, which subsequently yields 6.[8]





Other examples of semidiones undergoing facile reduction to relieve strain have also been reported.[9,10]

In this paper we report the electrochemical reduction of 1 using electron spin resonance (ESR) spectroscopy and cyclic voltammetry (CV). Facile reduction of 1 to 2 occurs even at relatively low temperatures.

Experimental Section

Electrochemical Measurements. Electrochemical experiments were performed with a Princeton Applied Research (PAR) potentiostat, Model 173, in conjunction with a PAR 175 universal programmer. Voltammograms were recorded on a Houston Omnigraphic 2000 x–y recorder. All potential measurements were referred to a saturated calomel electrode (SCE). It was necessary to separate the SCE from the DMF test solutions to prevent leakage of aqueous solution into the nonaqueous medium. This was accomplished by using in sequence a course glass frit, a 10-cm tube (0.5-cm diameter) containing a DMF (0.50 M $LiClO_4$)/methyl cellulose gel, and then directly an aqueous agar (1.0 M NaCl), and a saturated KCl solution in which the SCE was suspended. A one-compartment cell was used in the CV experiments. The working electrode was a glassy carbon disk (A = 0.090 cm^2) set in a Teflon tube. Prior to measurements on each solution this electrode was cleaned and polished with 0.30- and 0.050-μm α alumina (Buehler), wiped with a tissue, sonicated for 3–5 min, and wiped dry with a tissue. A graphite rod served as a counter electrode. All measurements were made under N_2.

Chemicals. Tetra-n-butylammonium perchlorate (TBAP) was purified by a literature method.[11] Tetra-n-butylammonium tetrafluoroborate was recrystallized twice from ethyl acetate–pentane (90:10). Reagent DMF (Fisher) was heated at 60 °C over CaH_2 for 6–10 h before distillation at 45–50 °C under reduced pressure. Further drying was accomplished by stirring the distillate over neutral Al_2O_3 (dried under vacuum at 160–170 °C) for several hours prior to redistillation at 45–50 °C and repeating the procedure. The DMF was stored over molecular sieves (4 Å) and under N_2.

Cyclobutadienopleiadiene (1) was prepared by the literature method.[12] Final purification was accomplished by recrystallizing 1, which is air sensitive, from pentane at −30 to −50 °C in a glovebag under N_2. The resulting yellow plates of 1, which decompose at ~70 °C to form a red, apparently polymeric material as previously described,[12] were stored under N_2 at −10 °C: ^1H NMR ($CDCl_3$, 60 MHz) δ 6.27 (s, 2 H), 7.07 (s, 2 H), 7.31–7.66 (ABC m, 6 H).

ESR Measurements. ESR spectra were recorded on a Varian Associates V-4502 spectrometer. Compound 1 was reduced in the ESR cavity in DMF (0.10 M TBAP) using a Varian flat-fused silica electrolytic cell with a mercury cathode and platinum anode. The emf across the electrodes was slowly increased until an ESR signal was obtained.

In the sodium reductions a glass apparatus, consisting of a long tube (3-mm diameter) with two side compartments at the top, was attached to a vacuum line. One compartment contained compound 1 and sodium was deposited in the other as a mirror. Dimethoxyethane, stored over Na/K, was distilled under vacuum into the sample compartment cooled in a dry ice/acetone bath. The glass apparatus was removed from the vacuum line using a blow torch and the solution of 1 was transferred to the sodium compartment. The reaction mixture was passed through a glass filter into the long tube, which was immediately inserted into the ESR cavity.

Results and Discussion

ESR Studies. Exposure of a dimethoxyethane solution of 1 to a sodium mirror at 25 °C gave the radical anion $2^{-\cdot}$ as evidenced by its ESR spectrum with hyperfine splitting constants (in G) of 0.66 (2 H), 1.67 (2 H), 5.96 (2 H), and 6.57 (4 H), in reasonable agreement with the values reported for $2^{-\cdot}$ in the literature.[5] Electrolytic reduction of 1 at mercury (−55 °C) in DMF (0.10 M TBAP) in an ESR spectrometer generated a weak, poorly resolved spectrum

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(2) Kariv-Miller, E.; Swenson, K. E.; Zemach, D. *J. Org. Chem.* **1983**, *48*, 4210.

(3) Birch, A. J.; Rao, G. S. *Adv. Org. Chem.* **1972**, 8, 1–65.

(4) Farnia, G.; Marcuzzi, F.; Melloni, G.; Sandona, G. *J. Am. Chem. Soc.* **1984**, *106*, 6503.

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(7) Rieke, R. D.; Hudnall, P. M. *J. Am. Chem. Soc.* **1973**, *95*, 2646.

(8) Russell, G. A.; Dodd, J. R.; Ku, T.; Tanger, C.; Chung, C. S. C. *J. Am. Chem. Soc.* **1974**, *96*, 7255.

(9) Russell, G. A.; Whittle, P. R. *J. Am. Chem. Soc.* **1967**, *89*, 6781.

(10) Russell, G. A.; Whittle, P. R.; Keske, R. G.; Holland, G.; Aubuchon, C. *J. Am. Chem. Soc.* **1972**, *94*, 1693.

(11) Lagu, A.; Mark, H. B., Jr.; Jezorek, J. R. *J. Org. Chem.* **1977**, *42*, 1063.

(12) Roberts, B. W.; Wissner, A. *J. Am. Chem. Soc.* **1972**, *94*, 7168.

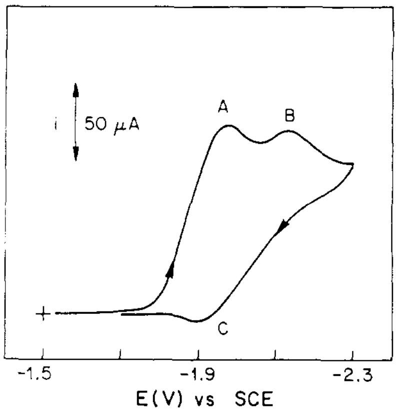

Figure 1. Cyclic voltammogram of 4.78 mM 1 in DMF (0.10 M $LiClO_4$) at glassy carbon with ν = 100 mV s^{-1}.

of $2^{-\cdot}$ as the first observable paramagnetic species. Upon warming to −25 °C, the ESR spectrum of $2^{-\cdot}$ intensified and became better resolved. There was no evidence of a second ESR signal that might have originated from $1^{-\cdot}$ or some other radical species.

These results, qualitative as they are, show that some 1 is reduced to $2^{-\cdot}$ via a sequence of reactions involving $3e^-$ and $2H^+$. It is reasonable to conclude that the radical anion of 1, which presumably forms in the first step of the sequence, has a very short lifetime under these conditions and thereby escapes ESR detection. Likely modes of reaction for $1^{-\cdot}$ would be protonation by trace amounts of water or the electrolyte or rapid reduction to 1^{2-}.

Cyclic Voltammetry Studies. A cyclic voltammogram (CV) of 4.78 mM 1 in DMF containing 0.10 M $LiClO_4$ as electrolyte at a freshly polished glassy carbon (GC) electrode is shown in Figure 1. At a scan rate (ν) of 100 mV s^{-1} two cathodic waves (A and B) at −1.972 and −2.130 V (SCE) and one anodic wave (C) at −1.900 V are observed. Similar CVs and peak potentials (E_p) were recorded when tetra-*n*-butylammonium perchlorate (TBAP) and tetra-*n*-butylammonium tetrafluoroborate were used as supporting electrolytes.

Electrode passivation was a problem when 1 was reduced at GC, Pt, or Au especially with $LiClO_4$ or TBAP as electrolyte (DMF). Although the first several cycles between −1.0 and −2.3 V were fairly reproducible, continued cycling resulted in a substantial reduction in current to the extent that no well-defined waves were present after ca. 10 cycles. Passivation was less of a problem, however, when tetra-*n*-butylammonium tetrafluoroborate was employed as the electrolyte (DMF). When a −1.95-V potential was applied to a GC electrode in the presence of a stirred solution of 1 (1–3 mM) in this electrolyte, the current did not decrease appreciably after the first several minutes.

The origin of this passivation is likely the formation of a polymer at the electrode surface. After electrolysis periods of 15–30 min at −1.95 V, a small amount of an acetone-insoluble material could be scraped from the GC surface. This material was not identified. To minimize passivation and its possible effects upon the electrochemistry of 1, the GC electrode was polished prior to each CV scan.

The Reduction Process at Wave A. The reduction process at wave A in Figure 1 is diffusion controlled as shown by plots of $\nu^{1/2}$ vs. i_p (peak current) that are linear. E_p does not change with varying concentrations of 1 (0.91–4.8 mM), which is consistent with a rate-determining step that is first order in 1. At scan rates of 20–500 mV s^{-1} and without IR compensation, a plot of E_p vs. log ν is nearly linear with a slope of −70 mV/decade (r = 0.987) which suggests that the reduction is an irreversible process.[13] Attempts to isolate and identify the product(s) from the reduction of 1 at wave A were unsuccessful due to electrode passivation. The question then of the n value for the reduction of 1 at wave A remains. Ordinarily, this would best be determined by coulometry experiments. The tendency of 1 to electropolymerize after long reduction times, however, would make any measured n value from this technique suspect. It was necessary, therefore, to use an alternative, approximate method.

With the assumption that the diffusion coefficients for a given pair of compounds are similar, which is often observed in a given solvent and electrolyte provided that their polarities and sizes are comparable,[14] an approximate n value for one compound can be determined if the n value for the other compound is known by using eq 1.[15] CVs

$$\frac{\text{slope}_{\text{rev}}}{\text{slope}_{\text{irrev}}} = \frac{0.90n^{3/2}}{n'\alpha^{1/2}} \qquad (1)$$

of a 2.0 mM solution of 2-methyl-9,10-anthraquinone (7) in DMF (0.10 M $LiClO_4$) were recorded at a variety of scan rates (20–500 mV s^{-1}) using a GC electrode. Compound 7 undergoes a one-electron reversible reduction in this electrolyte at −0.90 V (SCE). A plot of i_p vs. $\nu^{1/2}$ gives a straight line with a slope ($\text{slope}_{\text{rev}}$) of 134 μA s V^{-1}. The same experiment was conducted with 2.0 mM 1 in DMF (0.10 M $LiClO_4$) except that the GC electrode was polished prior to each scan. After correcting for an estimated 10% contribution of wave B to the peak current of wave A, a slope ($\text{slope}_{\text{irrev}}$) of 221 μA s V^{-1} is obtained. From eq 1 ($n^{3/2}$ = 1 for 7 and an experimental α value of 0.55[16]) n' for 1 is 1.81.

Ferrocene, which undergoes a reversble one-electron oxidation at +0.44 V in the above DMF electrolyte, was used to obtain a second estimate of n' for 1. A plot of i_p vs. $\nu^{1/2}$ from CVs of ferrocene (2.0 mM) generated a slope of 139 μA s V^{-1} from which an n' value of 1.75 is calculated.

If the diffusion coefficients for 1, 7, and ferrocene are similar, then the above experiments show that the reduction of 1 at wave A is a two-electron process. If, on the other hand, wave A is actually a one-electron process, then the diffusion coefficients of both 7 and ferrocene would have to be approximately 3 times less than that for 1. This seems unlikely.

The ESR experiments show that the reduction product of 1 is rapidly protonated in DMF, even at low temperatures, ultimately giving $2^{-\cdot}$. To probe the involvement of protonation in the reduction process at wave A, CVs of a 1.24 mM of 1 in DMF (0.10 M tetra-*n*-butylammonium tetrafluoroborate) and in the DMF electrolyte containing 2.64 mM *tert*-butyl alcohol were recorded. The presence of the alcohol proton donor affected wave A to a greater extent than waves B and C. Its E_p was shifted anodically by ca. 10 mV and its i_p increased from 17.2 to 21.8 μA. The increase in i_p for wave A was accompanied by a decrease in the peak width. These qualitative results support the involvement of protonation in the reduction process at wave A.

Electrode Processes at Waves B and C. The oxidation process at wave C does not appear to be coupled with the reduction process at wave A. As mentioned earlier,

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(13) Bard, A. J.; Faulkner, L. R. "Electrochemical Methods, Fundamentals and Applications"; Wiley: New York, 1980; pp 223, 455.

(14) Adams, R. N., "Electrochemistry at Solid Electrodes"; Marcel Dekker: New York, 1969; pp 220–222.

(15) Derived from the peak current expressions for reversible (ref 13, p 218) and irreversible (ref 13, p 222) processes.

(16) Reference 13, p 223.

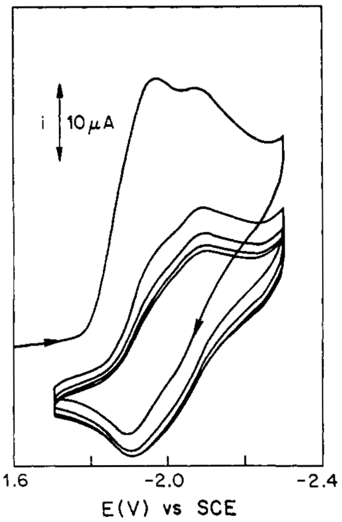

Figure 2. Cyclic voltammogram of 1.0 mM 1 in DMF (0.10 M tetra-*n*-butylammonium tetrafluoroborate) at glassy carbon with ν = 200 mV s^{-1}.

wave A shifts cathodically with increasing ν (−70 mV/decade). In contrast, wave C shifts only 30 mV/decade in the anodic direction with increasing ν. Both A and C would be expected to shift in opposite directions by the same amount if they represented a quasi-reversible couple. More compelling evidence for the postulate that waves A and C are not coupled is shown in Figure 2. A CV of 1 (1.0 mM) in DMF (0.10 M tetra-*n*-butylammonium tetrafluoroborate) at a scan rate of ν = 200 mV s^{-1} shows that repetitive scans between −1.7 and −2.3 V rapidly deplete the concentration of 1 at the electrode surface resulting in a large decrease in current for wave A. In contrast, the currents at waves B and C are less affected and tend to remain constant after several cycles. Thus, it is evident that wave C is not coupled to wave A, but is coupled to wave B.

Conclusion

One possible picture that emerges from the ESR and electrochemical data is summarized in eq 2. Compound

$$1 \xrightarrow[\text{(wave A)}]{2e^-/2H^+} 2 \underset{-e^-\text{ (wave C)}}{\overset{e^-\text{ (wave B)}}{\rightleftharpoons}} 2^{-}\cdot \qquad (2)$$

1 is reduced irreversibly at wave A to its dihydro derivative 2. Although a DISP2 mechanism[17] for this first-order $2e^-/2H^+$ process can be eliminated, the ECEC, EECC, and DISP1 mechanisms cannot be distinguished. Compound 2 is then reduced to its relative stable radical anion ($2^-\cdot$), which gives the quasi-reversible couple at waves B and C, in reasonable agreement with the $E_{1/2}$ of −1.91 V (SCE) reported for $2^-\cdot$ (Me_2SO) in the literature.[5]

Acknowledgment. This work was supported by the Research Corporation and the National Science Foundation (PRM 8104443). The author is also grateful to Professor Larry L. Miller for making it possible for him to spend a sabbatical leave at the University of Minnesota where this manuscript was written.

Registry No. 1, 30735-61-6; $1^-\cdot$, 96845-63-5; 2, 41232-69-3; 7, 84-54-8.

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(17) Amatore, C.; Saveant, J. M. *J. Electroanal. Chem.* 1977, *85*, 27.

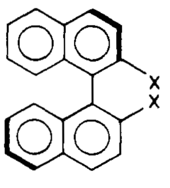
Enantioselective Reduction of Ketones with Reagents from Lithium Aluminum Hydride and Axially Chiral 2,2′-Diamino-1,1′-binaphthyls

Kuninobu Kabuto,† Takashi Yoshida,‡ Shozo Yamaguchi,*† Sotaro Miyano,‡ and Harukichi Hashimoto*‡

Department of Chemistry, College of General Education, Tohoku University, Kawauchi, Sendai 980, Japan, and Department of Applied Chemistry, Faculty of Engineering, Tohoku University, Aramaki-Aoba, Sendai 980, Japan

Received August 30, 1984

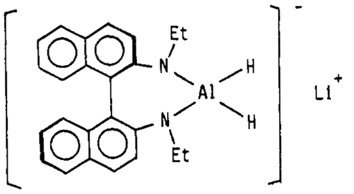
The enantioselective reduction of an achiral carbonyl compounds by a chiral hydride reagent has been the subject of much study. So far, various types of chiral ligands with centro chirality have been developed.[1] Axially chiral 2,2′-dihydroxy-1,1′-binaphthyl (**1f**) has been used for the formation of a chiral $LiAlH_4$ complex having an excellent enantioselectivity in the reduction of phenyl alkyl ketones,[2] whereas similar types of diamino analogue, 2,2′-diamino-6,6′-dimethyl-1,1′-biphenyl, afforded a moderate one.[3]



1a, X = NH_2
b, X = NHMe
c, X = NHEt
d, X = NH-*i*-Bu
e, X = $NHCH_2Ph$
f, X = OH

Recently, we have synthesized various 2,2′-bis(alkylamino)-1,1′-binaphthyls **1b–e** with axial chirality in order to examine their usefulness as a chiral aminophosphine ligand of homogeneous Rh catalyst for the asymmetric hydrogenation of prochiral olefins.[4] In this paper, we wish to report an enantioselective reduction of various ketones with reagents from $LiAlH_4$ and (*R*)-**1a–e**. Compounds **1b–e** were prepared from homochiral (*R*)-**1a** by acylation and subsequent $LiAlH_4$ reduction.

A typical example for the preparation of a chiral lithium aluminum hydride reagent is as follows. To a stirred, standardized solution of $LiAlH_4$ in THF[5] was added equimolar amount of homochiral *R* diamine **1c** in THF at 0 °C; then the solution was kept at 60 °C for 40 min. Two molar equivalents of hydrogen evolved to give a homogeneous solution suggesting that the active species in the hydride solution is **2**. The complex thus formed does not separate even at −100 °C.



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We have now carried out experiments designed to explore the effects of steric bulk of the substituents in the chiral amine ligands, temperature, solvents, additive, and various substrates upon the selectivity of this reduction.

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† Department of Chemistry, College of General Education.
‡ Department of Applied Chemistry, Faculty of Engineering.