which is soluble in common organic solvents to produce dark green solutions. Chlorinated solvents, such as $CH_2Cl_2$, convert **1** cleanly to $CpCr(NO)(PPh_3)Cl$ within 1 week at ambient temperatures. As a solid, **1** is moderately stable in air at room temperature for short periods of time and is stable indefinitely under an inert atmosphere.

A single-crystal X-ray crystallographic analysis of **1**[11] confirmed its monomeric nature and revealed its "three-legged piano-stool" molecular structure (Scheme I). The most interesting feature of this structure is that there are no unusual bond distances or angles, the intramolecular dimensions[12] being comparable to those exhibited by related 18-electron cyclopentadienylchromium nitrosyl complexes.[13-17] This feature contrasts with that exhibited by most other structurally characterized 17-electron, metal-centered organometallic radicals which generally display distortions in the metal's coordination-sphere geometry consistent with the unpaired electron being localized primarily at one site.[5] The relatively complicated ESR spectrum of **1** in hexanes (Figure 1) also indicates that there is considerable delocalization of the unpaired electron throughout the molecule. Thus, the spectrum exhibits signals manifesting couplings of the unpaired electron to the five Cp ring protons, to the $^{31}P$ nucleus, to the $^{14}N$ nucleus,[18] to both of the diastereotopic methylene protons, and to $^{53}Cr$. This spectrum contrasts with the ESR spectra exhibited by the $CpCr(NO)(PPh_3)X$ (X = Cl, Br, I) precursor complexes, all of which display features indicative of more localization of their unpaired electrons.[8]

Complex **1**, like most odd-electron metal complexes, is substitutionally labile.[5b] However, unlike these other complexes, its lability is not restricted solely to the replacement of two-electron ligands. Hence, with the sagacious choice of reagents, this property of **1** can be exploited to produce novel diamagnetic complexes (Scheme I). For instance, reaction of **1** with the NO radical results in replacement of $PPh_3$ to produce the known alkyl dinitrosyl complex $CpCr(NO)_2(CH_2SiMe_3)$ (**2**)[19] in good yield. In contrast, exposure of **1** to $NOPF_6$ in $CH_2Cl_2$ results in the loss of the alkyl ligand as $Me_4Si$ (established by GC) and the formation of $[CpCr(NO)_2(PPh_3)]PF_6$ (**3**),[20] a previously inaccessible salt.[21] Both of these conversions could conceivably proceed through an intermediate dinitrosyl species. Finally, treatment of **1** with 2 equiv of $HSnPh_3$ results in loss of the alkyl group as $Me_4Si$ and subsequent addition of the Sn–H bond to the Cr center, thereby producing $CpCr(NO)(PPh_3)(H)(SnPh_3)$ (**4**),[22] and $Sn_2Ph_6$.

Significantly, the synthesis of complex **3** means that it should be possible to incorporate other, more useful, ligands onto the electrophilic chromium dinitrosyl center. Furthermore, complex **4** is the first hydrido nitrosyl complex of chromium to have been isolated,[23] and its chemistry remains to be explored. Nevertheless, its manner of formation suggests that this reactivity of $CpCr(NO)(PPh_3)(CH_2SiMe_3)$ could be exploited for the activation of other main group–element bonds. Efforts to realize both of these synthetic goals are currently underway.

**Acknowledgment.** We are grateful to the Natural Sciences and Engineering Research Council of Canada for support of this work in the form of grants to F.G.H., P.L., and F.W.B.E. and scholarships to M.J.S. and W.S.M.

**Supplementary Material Available:** The simulated ESR spectrum of $CpCr(NO)(PPh_3)(CH_2SiMe_3)$ (**1**) and a table of coupling constants and tables of fractional coordinates, equivalent isotropic thermal parameters, and selected bond distances and bond angles for the non-hydrogen atoms of $CpCr(NO)(PPh_3)(CH_2SiMe_3)$ (**1**) (6 pages). Ordering information is given on any current masthead page.

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(10) Measured on a solid sample with a Johnson Matthey magnetic susceptibility balance.

(11) Crystals of **1** are monoclinic; space group $P2_1/c$; $a = 7.962$ (3) Å, $b = 15.440$ (4) Å, $c = 20.883$ (6) Å, $\beta = 90.31$ (3)°; $V = 2567.0$ Å$^3$; $Z = 4$; absorption coefficient = 5.6 cm$^{-1}$; diffractometer, Enraf Nonius CAD-4F; radiation, Mo K$\alpha$ ($\lambda = 0.71069$ Å); $2\theta_{max} = 46°$; $T = 200$ K; reflections = 2128 with $I > 2.5\sigma(I)$; variable parameters = 293; goodness of fit = 1.36; $R_F = 0.048$; $R_{wF} = 0.052$. The non-hydrogen atoms were refined anisotropically, and hydrogen atom coordinate shifts were linked with those of the carbon atoms to which they were bonded.

(12) Selected bond lengths (Å) and angles (deg) for **1** are Cr–P = 2.386 (2), Cr–N = 1.678 (5), Cr–C(1) = 2.096 (5), Cr–CP = 1.898, N–O = 1.216 (6), CP–Cr–P = 121.4, CP–Cr–N = 126.4, CP–Cr–C(1) = 119.0, N–Cr–P = 91.3 (2), C(1)–Cr–P = 91.2 (2), C(1)–Cr–N = 99.2 (2) where CP represents the center of mass of the cyclopentadienyl ring.

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(18) The couplings to the $^{14}N$ and the $^{31}P$ nuclei are similar in magnitude to those observed previously in related systems.[8]

(19) Legzdins, P.; Richter-Addo, G. B.; Wassink, B.; Einstein, F. W. B.; Jones, R. H.; Willis, A. C. *J. Am. Chem. Soc.* **1989**, *111*, 2097.

(20) Isolated in 60% yield. Anal. Calcd for $C_{23}H_{20}N_2O_2F_6P_2Cr$: C, 47.27; H, 3.44; N, 4.79. Found: C, 47.11; H, 3.47; N, 4.70. IR (Nujol mull) $\nu_{NO}$ 1826, 1730 cm$^{-1}$. FAB-MS *m/z* 439 [$P^+ - PF_6$]. $^1H$ NMR ($CD_2Cl_2$) $\delta$ 7.90–7.10 (m, 15 H, $P(C_6H_5)_3$), 5.70 (s, $C_5H_5$). $^{31}P\{^1H\}$ NMR ($CD_2Cl_2$) $\delta$ 68.46 (s, *P*$Ph_3$), −31.07 (septet, $PF_6^-$, $J$(P–F) = 710 Hz).

(21) Regina, F. J., Wojcicki, A. *Inorg. Chem.* **1980**, *19*, 3803.

(22) Anal. Calcd for $C_{41}H_{26}NOPCrSn$: C, 64.88; H, 4.74; N, 1.84. Found: C, 64.90; H, 4.82; N, 1.90. IR (Nujol mull) $\nu_{NO}$ 1620 cm$^{-1}$. $^1H$ NMR ($C_6D_6$) $\delta$ 8.09–7.43 (m, 15 H, $Sn(C_6H_5)_3$), 7.40–6.90 (m, 15 H, $P(C_6H_5)_3$), 4.56 (d, $C_5H_5$, $J$(P–H) = 3 Hz), −2.47 (d, Cr–H, $J$(P–H) = 90.3 Hz, $J$(Sn–H) = 23.7 Hz). $^{31}P\{^1H\}$ NMR ($C_6D_6$) $\delta$ 87.94 (s, *P*$Ph_3$, $J$(Sn–P) = 38.6 Hz).

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## Synthesis and Structure of the First Base-Free Diphosphadigalletane

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*Received May 23, 1991*

Much of the current interest in the chemistry of the heavier group 13 and 15 elements has been spurred on by the desire to develop alternative compound semiconductor precursors.[1] In the context of single source precursors, most of the emphasis has been placed on the chemistry of compounds of empirical formula $R_2MER'_2$ (M = Ga, In; E = P, As, Sb).[1,2] Information concerning less ligated species of the general type $(RMER')_n$ is much more sparse and base-free examples are confined to the cubanes $[t\text{-BuAl}(\mu_3\text{-PSiPh}_3)]_4$ and $[t\text{-BuGa}(\mu_3\text{-PSiPh}_3)]_4$.[3,4] In principle, less aggregated species should be obtainable by increasing the steric demands of the M and/or E substituents. We report (i) the

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(1) For reviews, see: Cowley, A. H.; Jones, R. A. *Angew. Chem., Int. Ed. Engl.* **1989**, *28*, 1208. Zanella, P.; Rossetto, G.; Brianese, N.; Ossola, F.; Porchia, M.; Williams, J. O. *Chem. Mater.* **1991**, *3*, 225.

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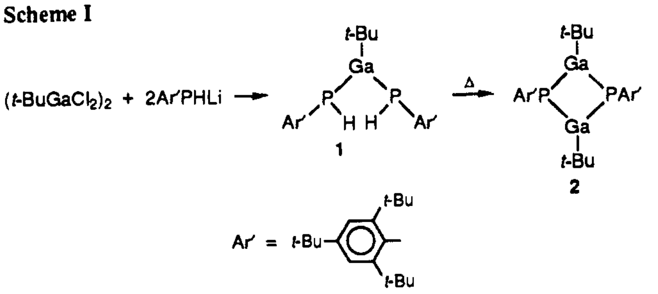
(4) A novel cluster, $[Ga_4(Trip)\{P(1\text{-Ad})_4\}P(H)(1\text{-Ad})]$ (1-Ad = 1-adamantyl, Trip = 2,4,6-$(i\text{-Pr})_3C_6H_2$), which has an overall ratio of substituents to main-group elements of unity, has been reported very recently: Waggoner, K. M.; Parkin, S.; Pestana, D. C.; Hope, H.; Power, P. P. *J. Am. Chem. Soc.* **1991**, *113*, 3597.

0002-7863/91/1513-7050$02.50/0

**Scheme I**



synthesis and structure of $[t\text{-BuGaPAr}']_2$, the first base-free diphosphadigalletane, (ii) a new approach to the synthesis of group 13–15 compounds, and (iii) an unusually short Ga–P bond distance.

The synthesis of the bis(phosphido)gallane **1** (Scheme I) was accomplished by treatment of $(t\text{-BuGaCl}_2)_2$ with Ar′PHLi (Ar′ = 2,4,6-$t$-$\text{Bu}_3\text{C}_6\text{H}_2$) at low temperature.[5] The $^{31}$P chemical shift and $^{31}$P–H coupling constant of **1** were indicative of the presence of Ar′PH moieties and the $^1$H integration indicated that bis(phosphido) substitution had occurred.[6] Several monomeric group 13/15 compounds with 1:1 and 1:3 stoichiometry have been structurally characterized;[1,7] however, no structural data are available for a monomeric 1:2 compound. An X-ray analysis of **1** was therefore undertaken.[8] The geometry at Ga (Figure 1) is essentially trigonal planar; however, the steric demands of one of the Ar′ groups causes considerable departure from the ideal angles. Likewise, the bulk of the Ar′ moieties causes the trans disposition of the two P–H groups. The Ga–P bond distances are identical within experimental error (2.324 (5) Å) and over 0.1 Å shorter that those found in dimers or trimers of the type $(\text{RR}'\text{GaPR}''\text{R}''')_n$.[1]

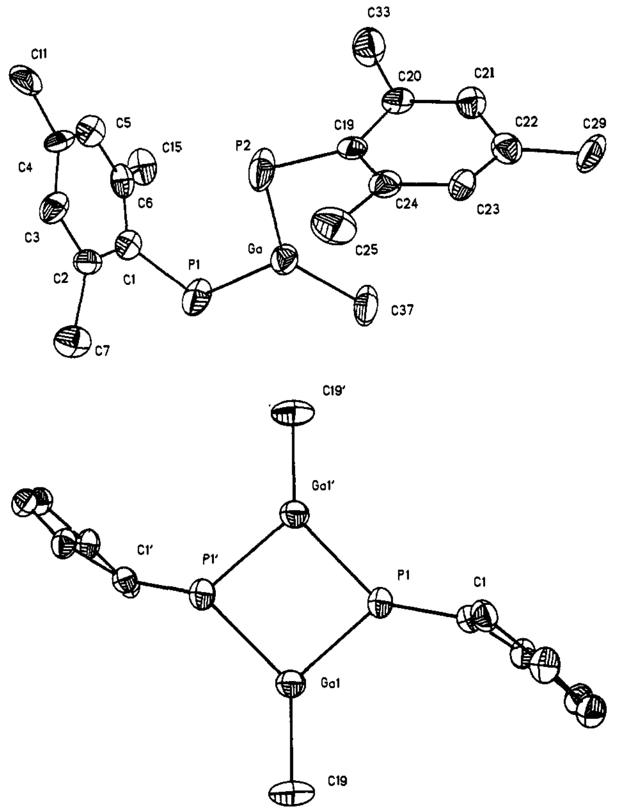


**Figure 1.** (a, top) ORTEP drawing of **1** ($t$-Bu methyls omitted for clarity). Important bond distances (Å) and angles (deg): Ga–P(1) 2.326 (4), Ga–P(2) 2.323 (5), Ga–C(37) 2.05 (2), C(37)–Ga–P(1) 112.4 (5), P(1)–Ga–P(2) 116.4 (2), C(37)–Ga–P(2) 131.3 (5). (b, bottom) ORTEP drawing of **2** (ring $t$-Bu's and Ga-$t$-Bu methyls omitted for clarity). Important bond distances (Å) and angles (deg): Ga(1)–P(1) 2.274 (4), Ga(1)–C(19) 2.00 (1), P(1)–C(1) 1.85 (1), P(1)–Ga(1)–P(1)′ 93.1(4), P(1)–Ga(1)–C(19) 131.2 (6), Ga(1)–P(1)–Ga(1)′ 86.9 (4), Ga(1)–P(1)–C(1) 113.9 (4).

Initial attempts to prepare **2** focused on the metalation of **1** followed by reaction with a gallium dihalide. However, the dilithium and dipotassium salts of **1** are not stable hence it was necessary to develop an alternative synthetic method. A better approach involved the thermolysis of **1** (Scheme I) which resulted in the elimination of $\text{Ar}'\text{PH}_2$ and the formation of **2** in 55% yield.[5] The proposed formula for **1** is in accord with the detection of a mass spectral parent peak at $m/e$ 806. Moreover, the $^{31}$P NMR chemical shift of $\delta$ −70.0 is consistent with the presence of a four-membered ring. Equal abundances of $t$-Bu and Ar′ substituents were apparent from integration of the respective $^1$H resonances.[6]

In principle, two canonical forms, A and B, can be written for **2**. From A is the cyclobutadiene-analogous structure in which the Ga and P centers are trigonal planar, while in the single-bonded form, B, the P atoms adopt a pyramidal geometry. An X-ray structure of **2** was undertaken[8] to clarify the nature of the bonding. The solid state of **2** consists of individual molecules with no short intermolecular contacts (Figure 1). The $\text{Ga}_2\text{P}_2$ rhombus resides on a center of symmetry and the Ga–P bond distance of 2.274 (4) Å is the shortest of which we are aware. The internal bond angles at Ga are >6° larger than those at P. Interestingly, this is the opposite trend to that observed in more highly ligated ring systems of the type $(\text{RR}'\text{MER}''\text{R}''')_2$ (M = Ga, In; E = P, As).[1]

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(5) **1**: A solution of LiP(H)Ar′ (Ar′ = 2,4,6-$t$-$\text{Bu}_3\text{C}_6\text{H}_2$) was prepared by addition of 7.2 mL of a 1.4 M solution of MeLi in $\text{Et}_2\text{O}$ to a cooled (−78 °C) suspension of $\text{Ar}'\text{PH}_2$ (2.82 g, 10.1 mmol) in 40 mL in $\text{Et}_2\text{O}$. After being warmed to 25 °C, the resulting yellow solution was stirred for 1 h, following which it was added via cannula to a cooled (−78 °C) suspension of $t$-$\text{BuGaCl}_2$ (1.0 g, 5.1 mmol) in 30 mL of $\text{Et}_2\text{O}$. The reaction mixture was allowed to warm to 25 °C and was stirred for an additional 4 h. After removal of the solvent and volatiles under vacuum the resulting yellow residue was extracted with 60 mL of hexane. Filtration and concentration of this solution afford yellow, crystalline **1** (mp 125–128 °C) in 75% yield. **2**: A solution of **1** (1.0 g, 1.47 mmol) in 20 mL of toluene was refluxed for 12 h, following which the solvent and volatiles were removed under vacuum. The resulting residue was sealed in an evacuated tube and placed in a thermal gradient sublimator (maximum temperature = 110 °C). A band of white crystals of $\text{Ar}'\text{PH}_2$ was formed and a yellow residue of **2** remained unsublimed. Crude **2** was purified by recrystallization from toluene at −20 °C. Yellow crystalline **2** (mp 208–211 °C) was obtained in 55% yield.

(6) **1**: $^1$H NMR (300.15 MHz, $\text{C}_6\text{D}_6$, 295 K, TMS ext.) $\delta$ 0.77 (s, 9 H, $(\text{C}H_3)_3$CGa), 1.33 (s, 18 H, $p$-$(\text{C}H_3)_3$C), 1.64 (s, 36 H, $o$-$(\text{C}H_3)_3$C), 4.50 (d, 2 H, $H$-P, $^1J_{\text{PH}}$ = 205 Hz), 7.46 (s, 4 H, H-aryl). $^{13}\text{C}\{^1\text{H}\}$ NMR (75.47 MHz, $\text{C}_6\text{D}_6$, 295 K, TMS ext.) $\delta$ 29.62 (d, C-Ga, $^2J_{\text{PC}}$ = 32 Hz), 31.58 (d, $(C\text{H}_3)_3$CGa, $^3J_{\text{PC}}$ = 6 Hz), 33.17 (br s, $o$-$(C\text{H}_3)_3$C), 34.84 (s, $o$-$C(\text{CH}_3)_3$), 36.71 (s, $p$-$C(\text{CH}_3)_3$), 38.15 (s, $p$-$(C\text{H}_3)_3$C), 121.91 (s, $o$-C aryl), 122.09 (s, $m$-C aryl), 148.01 (s, $p$-C aryl), 153.68 (br s, $C$-P). $^{31}$P NMR (121.5 MHz, 295 K, $\text{C}_6\text{H}_6$, 85% $\text{H}_3\text{PO}_4$ ext.) $\delta$ −113.05 (d, $^1J_{\text{PH}}$ = 203 Hz). MS (El, 70 eV) 679 ($\text{M}^+$ − H), 622 ($\text{M}^+$ − H-$t$-Bu), 553 ($(\text{Ar}'\text{PH})_2^+$), 497 (($\text{Ar}'\text{PH}_2^+$-$t$-Bu), 403 ($\text{M}^+$ − Ar′PH), 277 ($\text{ArPH}^+$), 220 (Ar′PH-$t$-Bu). **2**: $^1$H NMR (300.15 mHz, $\text{C}_6\text{D}_6$, 295 K, TMS ext.) $\delta$ 1.15 (s, 18 H $(\text{C}H_3)_3$CGa), 1.28 (s, 18 H, $p$-$(\text{C}H_3)_3$C), 1.90 (36 H, $o$-$(\text{C}H_3)_3$C) 7.53 (s, 4 H, H-aryl). $^{13}\text{C}\{^1\text{H}\}$ NMR (75.47 MHz, $\text{C}_6\text{H}_6$, 295 K, TMS ext.) $\delta$ 29.77 (s, $(C\text{H}_3)_3$CGa), 31.43 (s, $p$-$(C\text{H}_3)_3$C), 34.21 (t, $o$-$(C\text{H}_3)_3$C, $^4J_{\text{PC}}$ = 3.3 Hz), 34.95 (s, $p$-$C(\text{CH}_3)_3$), 36.37 (t, C-Ga, $^2J_{\text{PC}}$ = 8.2 Hz), 39.07 (s, $o$-$C(\text{CH}_3)_3$), 121.88 (t, $m$-phenyl C, $^3J_{\text{PC}}$ = 3 Hz), 133.48 (t, C-P, $^1J_{\text{PC}}$ = 108 Hz), 149.07 (s, $m$-phenyl C), 156.85 (t, $o$-phenyl C, $^2J_{\text{PC}}$ = 6 Hz), $^{31}$P NMR (121.5 MHz, 295 K, $\text{C}_6\text{H}_6$, 85% $\text{H}_3\text{PO}_4$ ext.) $\delta$ −70.0 MS (El, 70 eV) 806 ($\text{M}^+$), 472 ($\text{M}^+/2$ + Ga), 345 ($\text{M}^+/2$ − $t$-Bu), 316 ($\text{M}^+$ − 2$t$-$\text{Bu}_3\text{C}_6\text{H}_2$), 277 ($\text{Ar}'\text{PH}^+$).

(7) Higa, K. T.; George, C. *Organometallics* **1990**, *9*, 275.

(8) Crystal data for **1** ($\text{C}_{40}\text{H}_{67}\text{P}_2\text{Ga}$): monoclinic, space group $P2_1/a$ (No. 14) with $a$ = 21.05 (3) Å, $b$ = 9.514 (3) Å, $c$ = 21.770 (7) Å, $\beta$ = 102.89 (5)°, $V$ = 4250 (5) Å$^3$, $Z$ = 4, $d_{\text{calcd}}$ = 1.065 g cm$^{-3}$, $\mu$(Mo K$\alpha$) = 7.42 cm$^{-1}$. Crysıal data for **2** ($\text{C}_{44}\text{H}_{76}\text{P}_2\text{Ga}_2$): monoclinic, space group $P2_1/n$ (No. 14) with $a$ = 16.473 (2) Å, $b$ = 9.217 (1) Å, $c$ = 17.003 (1) Å, $\beta$ = 112.72 (9)°, $V$ = 2381 (9) Å$^3$, $Z$ = 2, $d_{\text{calcd}}$ = 1.128 g cm$^{-3}$, $\mu$(Mo K$\alpha$) = 12.22 cm$^{-1}$. Both data sets were collected on an Enraf Nonius CAD-4 diffractometer at 25 °C using graphite monochromated Mo K$\alpha$ radiation ($\lambda$ = 0.71069 Å). Totals of 3376 and 5396 reflections were collected for **1** and **2**, respectively, using the $\theta/2\theta$ scan mode. Of these 2076 and 2186 were considered observed ($I > 6.0\sigma(I)$) and were used to solve (Patterson) and refine (full matrix, least squares) the structures of **1** and **2**, respectively. The final $R$ values were 0.0700 for **1** and 0.0742 for **2**.

While the short Ga-P bond distance and the yellow color of **2** are suggestive of a $p_\pi$-$p_\pi$ interaction, this cannot be extensive since the P atoms remain pyramidal (sum of angles = 314.7°). Interestingly, the Ga atoms are also slightly nonplanar. Overall, the bonding description for **2** is closer to form B than to form A.

**Acknowledgment.** We thank the National Science Foundation, the Robert A. Welch Foundation, and the U.S. Army Research Office (Durham) for generous financial support.

**Note Added in Proof.** The trimer $[(2,4,6\text{-}Ph_3C_6H_2)GaP(C_6H_{11})]_3$ has been reported very recently by Power et al. (Hope, H.; Pestana, D. C.; Power, P. P. *Angew. Chem., Int. Ed. Engl.* **1991**, *30*, 691). The $Ga_3P_3$ system is not planar and little delocalization is apparent.

**Supplementary Material Available:** Tables of bond distances, bond angles, atomic coordinates, and thermal parameters for **1** and **2** (8 pages); listings of observed and calculated structure factors for **1** and **2** (26 pages). Ordering information is given on any current masthead page.

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# Iron-Cyclam Complexes as Catalysts for the Epoxidation of Olefins by 30% Aqueous Hydrogen Peroxide in Acetonitrile and Methanol

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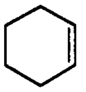
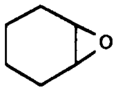
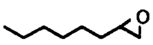
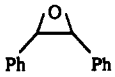
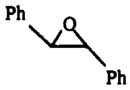
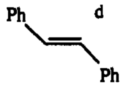
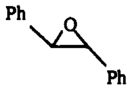
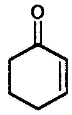
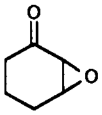
In recent years, several non-porphyrin iron-containing catalysts of alkane hydroxylation by dioxygen and/or peroxides have been reported that appear to react via nonradical pathways.[1-7] In this respect, their reactions resemble those of non-heme iron containing monooxygenase enzymes and iron bleomycin, but unlike the biological systems,[8-11] only a few of these non-porphyrin iron complexes are reported to catalyze olefin epoxidation.[4a,b,5a,12] Here we report that iron complexes of cyclam (1,4,8,11-tetraazacyclotetradecane) and related ligands are highly effective catalysts for olefin epoxidations by aqueous 30% hydrogen peroxide in acetonitrile or methanol and that, unlike previously reported systems, we observe (1) high turnovers based on the iron complex, (2) high percent yields based on $H_2O_2$, (3) tolerance for at least small amounts of water, (4) stereospecificity, and (5) only small amounts of allylic oxidation products.

**Table I.** Products Obtained from Reaction of $H_2O_2$ with Olefins in the Presence of $Fe(cyclam)(CF_3SO_3)_2$[a]

| Substrate | Products | Yield (mmol) | Turnover[b] |
|---|---|---|---|
|  |  | 0.40[c] | 20 |
|  |  | 0.14 | 7 |
|  |  | 0.26 | 13 |
|  |  | 0.017 |  |
|  | PhCHO | 0.039 |  |
|  d |  | 0.36 | 18 |
|  | PhCHO | 0.02 |  |
|  |  | trace |  |

[a] Reactions were quenched after 8 min for cyclohexene and 20 min for other substrates. See text for experimental conditions. [b] (Millimoles of epoxide)/(millimoles of iron catalyst). [c] See Table II for amounts of allylic oxidation products. [d] Reaction was run in the solvent mixture of acetonitrile (4 mL) and toluene (1 mL).

Hydrogen peroxide (1 mmol, 30% aqueous) was slowly added under an inert atmosphere over a period of 1 min to a solution of the Fe$^{II}$ complexes of ligands **1**–**6** (0.02 mmol) and cyclohexene (1 mmol) in 5 mL of acetonitrile.[13] The reaction mixture was quenched after 8 min by addition of sodium sulfite, filtered through a 0.45-$\mu$m filter, and then analyzed by GC/MS or HPLC. The results were as follows: Fe(**1**)$^{2+}$ (ferrous cyclam) gave 0.40 mmol of cyclohexene oxide (40% yield based on $H_2O_2$, 20 turnovers based on Fe). Fe(**2**)$^{2+}$ gave 0.20 mmol, Fe(**3**)$^{2+}$ gave 0.10 mmol, and Fe(**4**)$^{2+}$ gave 0.04 mmol of cyclohexene oxide. The Fe$^{II}$ complexes of **5** and **6** gave no epoxidation of cyclohexene. Only small amounts of allylic oxidation products were observed in any case, suggesting that typical radical reactions were not involved. Fe(**1**)$^{2+}$ in methanol under the same conditions but at 4 °C[17] gave the same

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[13] The complexes $Fe(\mathbf{2})(CF_3SO_3)_2$[14] and $Fe(\mathbf{4})(PF_6)_2$[15] and the ligand **5**[16] were prepared by literature methods. Ligands **1**, **3**, and **6** were purchased from Aldrich. The complexes $FeL(CF_3SO_3)_2$, L = **1**, **3**, **5**, **6**, were prepared by stirring equimolar quantities of $Fe(CF_3SO_3)_2$ and ligand in anhydrous acetonitrile followed by filtration, reduction of volume by evaporation, and addition of $Et_2O$ to induce crystallization. The resulting solid was filtered, washed with $Et_2O$, and dried in vacuo. All manipulations were carried out under an inert atmosphere. Satisfactory elemental analyses were obtained for each complex.

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