Figure 1. (A) $\Delta m_s = 1$ region of the primary EPR spectrum obtained by brief irradiation of a 2-MTHF glass of **3**. The vertical bar marks 3260 G. The $\Delta m_s = 2$ transition near 1630 G was also observed but is not shown here. The triplet ZFS parameters are $|D|/(hc) = 0.020$ cm^{-1} and $|E|/(hc) = 0.007$ cm^{-1}. (B) Secondary spectrum (at reduced amplitude) obtained after prolonged irradiation of the preparation of A ($\lambda > 425$ nm). (C) Spectrum obtained by processing $3\text{-}d_4$ through the same steps used to obtain B. The triplet ZFS parameters are $|D|/(hc) = 0.0025$ cm^{-1} and $|E|/(hc) = 0.0009$ cm^{-1}.

versions of **6** are to a, a decrease of 25%, to c or d, a decrease of 62.5%, and to b or e, no change. Experiment 3 probably is not quite sensitive enough to rule out a, but it clearly eliminates c and d and is consistent with b and e.
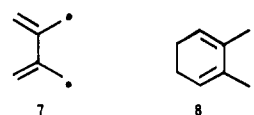
Table I displays a truth matrix that tests the compatibility of the candidates with the experimental facts. The only candidate to survive this scrutiny is the triplet biradical **2**.[7b]

A Curie study of a 2-MTHF glass of the secondary signal carrier showed the intensity to be linear with $1/T$ between 15 and 90 K ($r = 0.991$). If the slight convexity at the low-temperature end of the plot is assigned, for the sake of argument, entirely to thermal population of a triplet state from a lower energy singlet, the singlet cannot lie more than 0.024 kcal/mol below the triplet. Thus, either the singlet and triplet are almost exactly degenerate, or more plausibly, the triplet is the ground state of biradical **2**.

Triplet states of the simpler disjoint biradical system tetramethyleneethane have been observed in the parent compound[10]

7 and a derivative[11] **8**. Unfortunately, it is not known whether

these triplet species are the ground states and, if so, whether they are planar. The biradical **2** therefore represents a rare experimental test of the energetic consequences of disjoint character in a non-Kekulé compound.[12,13] Apparently in the present case, this property is insufficient to produce a low-spin ground state. Although a generalization of this finding clearly would be premature, one cannot suppress completely the conjecture that even in the absence of the degeneracy that forms the basis of the atomic Hund's Rule, a quasi-Hund's Rule may operate in non-Kekulé molecules.

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(12) For an excellent review of recent results on cyclobutadiene, a disjoint Kekulé compound, see: Borden, W. T. "Diradicals"; Wiley: New York, 1982; p 46.
(13) The assignment of a triplet ground state to the Schlenk-Brauns hydrocarbon, a non-Kekulé compound which, *if planar*, would be disjoint, rests on a Curie plot covering a narrow, high-temperature region in which curvature is difficult to detect: Schmidt, R.; Brauer, H.-D. *Angew. Chem., Int. Ed.* Engl. **1971**, *10*, 506.

Electric Field Gradient at Iron in Dicarbonyl Complexes of (Tetraphenylporphyrinato)- and (Octamethyltetrabenzoporphyrinato)iron(II)

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In diamagnetic Fe(P)L$_2$ complexes[1] the electric field gradient (EFG) at iron is due primarily to an imbalance of electron densities in the iron 3d valence orbitals,[2] and in all such species for which the sign of V_{zz}, the principal component of the EFG tensor, is known, it is positive.[3–8] This oblate charge distribution leads one to expect V_{zz} to increase with increasing π acceptance or decreasing

(7) (a) Cf. Platz, M. S.; Berson, J. A. *J. Am. Chem. Soc.* **1980**, *102*, 2358. (b) That no $\Delta m_s = 2$ transition could be observed for this species is not surprising, since the ZFS is very small, and the two-quantum transition is strongly forbidden even at low field. The total spectral width of about 40 G for the secondary signal excludes the previously discussed[8] type of triplet structure resulting from "wrong bond" photolysis, i.e., cleavage of a side bond of **3** rather than a bridge bond. This would lead to a vinyl radical, whose hyperfine splitting alone would be larger than the observed total width.[9]
(8) Rule, M.; Matlin, A. R.; Seeger, D. E.; Hilinski, E. F.; Dougherty, D. A.; Berson, J. A. *Tetrahedron* **1982**, *32*, 787.
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(1) Abbreviations: P = dianion of a porphyrin or phthalocyanine; L = nitrogen- oxygen-, or sulfur-donor ligand; TPP, OMBP, Pc = the dianions of tetraphenylporphyrin, octamethyltetrabenzoporphyrin, and phthalocyanine, respectively; pip = piperidine; py = pyridine; diox = anion of a dioxime ligand.
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5148 *J. Am. Chem. Soc., Vol. 105, No. 15, 1983*

Communications to the Editor

Table I. Mössbauer and IR Data for the Fe(P)(CO)$_2$ Complexes

P	T, K	δ,[a] mm s^{-1}	ΔE_Q,[b] mm s^{-1}	$\bar{\Gamma}$, mm s^{-1}	ν_{CO},[c] cm^{-1}	ref[d]
Pc	295	0.09	(+)0.82		2057	12
TPP	295	0.19	(+)0.27	0.26	2030	
	4.2	0.18	+0.20	0.26		
OMBP	295	0.30	(−)0.49	0.35	2025	
	4.2	0.29	−0.57	0.30		

[a] Relative to metallic iron; source and absorber at same temperature. [b] Comparative values (mm s^{-1}) at 295 K:[7,12] Fe(Pc)(pip)$_2$, 2.34; Fe(Pc)(pip)CO, 1.27; Fe(TPP)(pip)$_2$, 1.52; Fe(TPP)(pip)CO, 0.53; Fe(OMBP)(pip)$_2$, 0.89; Fe(OMBP)(pip)CO, 0.20. [c] Nujol mull. [d] This work unless otherwise noted.

σ donation by axial ligands.[7,9] In fact, substitution of an axial amine by CO (a weaker σ donor and stronger π acceptor) to give Fe(P)(CO)L, produces a large decrease in $|V_{zz}|$.[7,10–12] Similar changes have been observed for carbonylation of Fe(diox)$_2$L$_2$ complexes.[9,13] We now report Mössbauer measurements of the EFG at iron in two *trans*-dicarbonylporphyrinatoiron(II) complexes, Fe(TPP)(CO)$_2$ (**1**) and Fe(OMBP)(CO)$_2$ (**2**). In **1** V_{zz} is positive but smaller than in the corresponding monocarbonyls, while in **2** V_{zz} is negative. This is the first observation of a negative EFG in a diamagnetic iron(II) porphyrin complex.

Dicarbonylporphyrinatoiron(II) complexes have been formed in solution,[14,15] but in only a few instances have solid samples been isolated.[14] The reduction of Fe(TPP)Cl with Na$_2$S$_2$O$_4$ in toluene/1% methanol is a facile route to Fe(TPP),[16] which on addition of CO gives red-violet crystals of **1** in high yield. Nujol mulls of **1** prepared under CO display a prominent ν_{CO} band at 2030 cm^{-1}. A weaker band at 1989 cm^{-1}, which intensifies (at the expense of the 2030 cm^{-1} absorption) on exposure of the mull to vacuum or N$_2$, is consistent[14] with the presence of Fe(TPP)CO (**3**). Careful handling of **1** under CO diminishes but does not eliminate this monocarbonyl impurity.

No dicarbonyl derivatives of iron(II) benzoporphyrins have been reported. Fe(OMBP) is easily obtained by heating (180 °C) Fe(OMBP)(py)$_2$ under vacuum.[4,6] The extreme insolubility of Fe(OMBP) in noncoordinating solvents requires carbonylation to be carried out at high pressure (50 atm) and temperature (215 °C); under these conditions **2** is formed quantitatively. The second CO ligand is more tightly bound in this complex, as there is no evidence of a CO stretching frequency apart from the band at 2025 cm^{-1}.

Mössbauer spectra were recorded and analyzed as previously described;[9,17–20] parameters are given in Table I. Spectra of **2** gave no evidence of any Fe-containing impurity, but those of **1** showed additional lines consistent with the presence of **3**.[21] On the basis of areas of the absorption lines, the ratio of **1** to **3** in

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(16) We find Na$_2$S$_2$O$_4$ to be a cleaner reductant than NaBH$_4$,[14] and filtration of the reduced solution under N$_2$ using standard Schlenk techniques assures the quality of the product. The filtrate is evaporated to dryness to remove traces of methanol, and the addition of CO-saturated toluene gives **1**.

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(21) At 77 K, Mössbauer parameters of **3** are: δ = 0.17 mm s^{-1}, $|\Delta E_Q|$ = 1.13 mm s^{-1}.
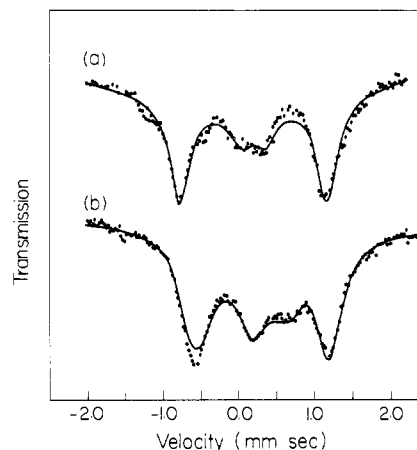

Figure 1. Mössbauer–Zeeman spectra at 4.2 K of (a) **1** in a longitudinal magnetic field of 5.8 T; (b) **2** in a longitudinal magnetic field of 4.5 T. Theoretical spectra were calculated from parameters given in Table I.

the sample used for Mössbauer–Zeeman measurements was ~4:1. Zero-field spectra of both **1** and **2** showed temperature-independent asymmetry (line intensities ~1.2:1), which we attribute to preferential crystallite orientation. This and the fact that **1** was only ~80% pure, produced some discrepancies between observed and calculated intensities in the Mössbauer–Zeeman spectra (Figure 1) but did not preclude unambiguous determinations of the signs of V_{zz}.

In the series Fe(P)(pip)$_2$ → Fe(P)(pip)CO → Fe(P)(CO)$_2$ (P = Pc, TPP, OMBP) each replacement of pip by CO increases the negative contribution to V_{zz}. For Pc and TPP these changes reduce the electronic anisotropy of the iron valence orbitals while maintaining an oblate charge distribution, while for OMBP the charge distribution changes from oblate to prolate. This reflects the weaker ligand field provided by OMBP[6,8] than by TPP and Pc. The overall changes in ΔE_Q between Fe(P)(pip)$_2$ and Fe(P)(CO)$_2$ are fairly similar in all three cases.

These results are contrary to the argument[10,13] that in metalloporphyrins replacement of a given axial ligand by one that is a weaker σ donor, stronger π acceptor, or both should increase the positive contribution to V_{zz}. This argument ignores at least two important possibilities: a significant change in axial bond length at the site of substitution and perturbations of the iron–ligand bonds both cis and trans to the site of substitution.

Although structural data are sparse for Fe(P)L(CO) and lacking for any Fe(P)(CO)$_2$ species, available X-ray results[22–25] indicate that the Fe–C(O) bond is substantially shorter than the Fe–N(amine) bond it replaces, that the remaining Fe–N(amine) bond is lengthened, and that there is a net decrease in the mean axial bond length relative to the bis(amine) complex. We have suggested[9,12] that this diminished mean axial bond length causes the dominant change in V_{zz} observed on carbonylation, although this may well be modulated by cis effects.[9] The presence of two quite short Fe–C(O) bonds can similarly explain the more negative V_{zz} values found for the dicarbonyls: the synergic nature of metal–carbonyl binding should result in a significant increase in electron density near iron along the z axis.

In going from Fe(P)(pip)CO to Fe(P)(CO)$_2$, **2** shows the largest change in V_{zz} and the smallest change in ν_{CO}; it is also the only case for which δ changes significantly. The low ν_{CO} value of **2** indicates a greater degree of Fe → CO π-electron transfer, which to be consistent with the large negative shift in V_{zz} seems to require stronger (i.e., shorter) Fe–C(O) bonds and a higher degree of localization of π density in the Fe–C internuclear regions. The increase in δ possibly reflects greater shielding of s electrons by

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d$_\pi$ density near the iron nucleus. The higher ν_{CO} values for **1** and Fe(Pc)(CO)$_2$ suggest greater competition between axial and equatorial ligands for the iron d$_\pi$ electrons, which may well result in more diffuse distributions of the π density.

Of the three macrocycles, OMBP appears to be the most effective "electron sink" in the sense that it is best able to accommodate the π-bonding demands of the axial CO ligands.

Stereoselective Formation of *cis*-Stilbene Ozonide from the Cosensitized Electron-Transfer Photooxygenation of *cis*- and *trans*-2,3-Diphenyloxiranes

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Cyano-substituted aromatic hydrocarbons such as 9,10-dicyanoanthracene (DCA) sensitize the photooxygenation of olefins, acetylenes, and sulfides.[1] A key step in this reaction is the electron-transfer quenching of singlet excited DCA by the substrate to generate DCA$^-\cdot$ and the substrate radical cation.[2] Subsequent reaction of O$_2^-\cdot$ with the radical cation affords the peroxidic product. We have, however, recently developed a method by which a compound that does not quench DCA may nevertheless be rapidly oxidized under electron-transfer photooxygenation conditions.[3] It was found that the photooxygenation of tetraphenyloxirane to yield the corresponding ozonide could be consensitized by a non-light-absorbing, chemically unreactive aromatic hydrocarbon (biphenyl, BP) in conjunction with DCA. In the absence of BP, this epoxide is relatively unreactive.[4]

The stereochemistry of this reaction has now been examined by an investigation of the DCA/BP-cosensitized photooxygenation of *cis*-2,3-diphenyloxirane (**1a**) and *trans*-2,3-diphenyloxirane (**1b**). We have observed that a *single isomer of the ozonide is formed from both epoxides*. Photooxygenation of **1a** and **1b** gives rise exlusively to *cis*-3,5-diphenyl-1,2,4-trioxolane (**2a**). This striking stereochemical result is consistent with a novel mechanism involving concerted 4 + 2 cycloaddition of singlet oxygen to an intermediate carbonyl ylide.

Photooxygenations of **1a** and **1b** were carried out in dry acetonitrile[6] with 1 × 10^{-2} M epoxide, 1 × 10^{-2} M BP, and 6 × 10^{-4} M DCA. The solutions were irradiated for 2 h at 10 °C under oxygen with a 450-W medium-pressure mercury lamp by using
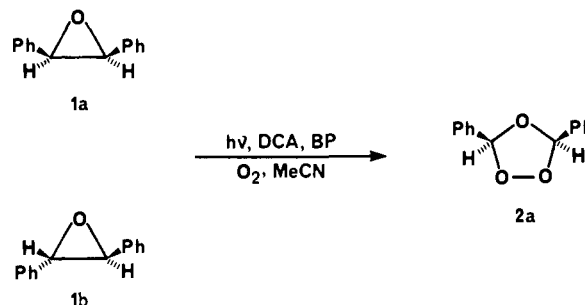
a CuSO$_4$-filter solution[7] and monitored by HPLC.[8] Yields of products were determined by removal of the solvent at 0 °C under vacuum and analysis in CDCl$_3$ by 300-MHz ^{1}H NMR. The spectra obtained from photooxygenations of **1a** and **1b** exhibited absorptions at δ 6.331 for the ring proton of *cis*-ozonide **2a** (65–70%) and at δ 10.015 for the aldehydic proton of benzaldehyde (**3**).[9] Within the limits of detection (2%) no absorption for *trans*-3,5-diphenyl-1,2,4-trioxolane (**2b**) was observed. Authentic samples of the isomeric ozonides were prepared by ozonation of stilbene. Treatment of *cis*- and *trans*-stilbene with ozone at low temperature in pentane gave a 60:40 mixture of *trans*-ozonide **2b** and *cis*-ozonide **2a**, respectively.[10] The ^{1}H NMR spectrum of the mixture in CDCl$_3$ exhibited ring proton absorptions at δ 6.360 for **2b** and 6.331 for **2a**.[11] Criegee and Korber[10b] report absorptions in CCl$_4$ at δ 6.22 and 6.18 for **2b** and **2a**, respectively. Additional evidence for the structure of *cis*-ozonide **2a** was obtained by preparative TLC of the photooxygenation products over silica gel with 10% ethyl acetate in hexane. Recrystallization of the chromatographed material from petroleum ether yielded colorless needles of **2a**, mp 98–100 °C (lit.[10b] mp 100 °C). As expected, **2a** was converted to benzaldehyde by reaction with Ph$_3$P.

As control experiments we have shown that authentic samples of ozonides **2a** and **2b** are stable under the photooxygenation conditions. No *trans*-ozonide **2b** is observed at partial conversion of **1a** or **1b** nor is any isomerization of the starting epoxides **1** found. In the absence of BP, only 7% benzaldehyde and no ozonide was obtained after 20-h irradiation. Additionally, BP is not appreciably consumed during photooxygenation.

We[3] have proposed that BP acts as a cosensitizer in photooxygenations by a process analogous to homogeneous redox catalysis for electrode reactions.[12] Epoxides **1** do not measurably quench the fluorescence of DCA in MeCN as is expected for compounds with oxidation potentials greater that 2 V vs. SCE[2] (*trans*-2,3-diphenyloxirane (**1b**), E^{ox} = 1.89 V vs. Ag/AgNO$_3$ in MeCN;[13] ~2.2 V vs. SCE in MeCN). However, BP is more easily oxidized (E_p^{ox} = 1.85 V vs. SCE in MeCN[14]) than epoxides **1** and consequently quenches ^{1}DCA* more efficiently (k_q (for BP) =

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(3) (a) Presented at the IXth IUPAC Symposium on Photochemistry, Pau, France, July 26, 1982; Abstr, ST 15. (b) Schaap, A. P.; Lopez, L.; Gagnon, S. D. *J. Am. Chem. Soc.* **1983**, *105*, 663.

(4) Ohta and co-workers have recently reported that tetraphenyloxirane does not react under their conditions with DCA alone.[5] Ozonides have, however, been obtained from more easily oxidized epoxides bearing electron-donating groups.

(5) Futamura, S.; Kusunose, S.; Ohta, H.; Kaniya, Y. *J. Chem. Soc., Chem. Commun.* **1982**, 1223.

(6) Distilled from P$_2$O$_5$ and then from CaH$_2$.

(7) The 1-cm path-length filter solution was prepared from 27 g of CuSO$_4$·5H$_2$O, 30 g of NaNO$_2$, and 50 mL of concentrated NH$_4$OH diluted with water to 1000 mL.

(8) Using a C-18 reverse-phase column and elutions with a gradient of 30–100% acetonitrile in water.

(9) The mechanism for the formation of **3** has not yet been established. Control experiments have shown that **3** is not produced photochemically from **2a** under reaction conditions.

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(11) Recrystallization (6x) of the mixture of ozonides from methanol gave authentic *cis*-ozonide **2a**, mp 97–99 °C (lit.[10b] mp 100 °C), with a ^{1}H NMR absorption at δ 6.331. The reported melting point of the *trans*-ozonide **2b** is 94 °C.[10b]

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