to any of the clavulones. However, when ${}^{3}\text{H}-8(R)$ -HPETE¹² (19 μ M) was incubated with a coral homogenate¹¹ a product was obtained in up to 13% radiochemical yield after diazomethane esterification and HPLC¹³ separation. Although the product coeluted with 8-HPETE methyl ester by HPLC,¹³ it was clearly different since it did not undergo reduction or change upon exposure to trimethyl phosphite. The structure of this compound was established as 3 by HPLC comparison with authentic biosynthetic pre-clavulone A⁵ and, after epimerization (1,8-diazabicyclo[5.4.0]undec-7-ene, THF), with synthetic 8-epi-pre-clavulone A.5 Additionally, catalytic hydrogenation of the methylated coral product followed by epimerization provided 9-oxoprostanoic acid (4) indistinguishable from material prepared by the catalytic hydrogenation (Pd-C catalyst) of prostaglandin A₂ methyl ester acetate (1).

The data outlined herein establish the intermediacy of 8-(R)-HPETE in the biosynthesis of preclavulone A (3) from arachidonic acid. During the biosynthesis of 3, oxygen must migrate from C(8) to C(9) in a situation paralleling that which occurs in the biosynthesis of cis-jasmonic acid.⁶ It seems likely that the conversion of 8(R)-HPETE to 3 occurs via allene oxide 5 and oxidopentadienyl cation 6 intermediates,⁶ with antarafacial per-icyclic closure of 6 leading directly to $3.^{14}$ A major implication



of these results is that the biosynthesis of prostaglandin methyl ester acetate (1) in *Plexaura homomalla* may proceed in an analogous manner: arachidonic acid $\rightarrow 8,15$ -bis-HPETE \rightarrow allene oxide \rightarrow oxidopentadienyl cation \rightarrow PGA₂. Experiments to test this proposal for the biosynthesis of PG's by P. homomalla are in progress. Further, it remains to continue the study of clavulone biosynthesis using fresh (rather than frozen) coral in order to obtain more information regarding the last stages of biosynthesis.¹⁵

Insertion of Carbon Dioxide into Metal Alkoxide Bonds. Synthesis and Structure of Tungsten **Tetracarbonyl Carbonate**

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Mechanistic aspects of insertion reactions of carbon dioxide into M-R bonds (R = H, alkyl, or aryl) have received and warranted much attention.² Contrastingly, much less effort has been expended on the analogous insertion reactions of CO₂ into M-OR bonds.³ In a series of studies involving the reactivity of the recently synthesized tungsten pentacarbonyl aryl oxide complexes, $W(CO)_5OR^-$ (R = Ph, $C_6H_4CH_3$ -m), we have found that reaction with CO₂ occurs quite readily, forming the tungsten pentacarbonyl aryl carbonate species (eq 1). Hydrolysis of these

$$W(CO)_5OR^- + CO_2 \rightarrow W(CO)_5OC(O)OR^-$$
(1)

metal aryl carbonates, accompanied by loss of a carbon monoxide ligand, leads to formation of the tungsten tetracarbonyl η^2 carbonate dianion, $W(CO)_4(\eta^2 - CO_3)^2$. The assignment of the structure of this complex has been established by X-ray crystallography.

The complexes $[Et_4N][W(CO)_5OR]^4$ were prepared from the reaction of $W(CO)_5$ THF (obtained by photolysis of $W(CO)_6$ in tetrahydrofuran) with the corresponding salts, [Et₄N][OR].⁵ In solution, $W(CO)_5OR^-$ is extremely CO labile, decomposing readily to $W_4(CO)_{12}(\mu_3 - OR)_4^{4-}$ in the absence of a CO atmosphere. These tetrameric species have been independently synthesized from $W(CO)_3(CH_3CN)_3$ and $[Et_4N][OR]$ and were shown via IR and ¹³C NMR to be structurally analogous to the well-characterized chromium tetramers.^{5,6} Isolation of the mononuclear complex was only accomplished by crystallization from carbon monoxide saturated solutions. The [Et₄N][W(CO)₅OR] complexes are yellow-orange air and moisture sensitive solids.

Treatment of bright orange THF or acetone solutions of $[Et_4N][W(CO)_5OR]$ with CO_2 ($P_{CO_2} < 760$ mm) at ambient temperature or lower results in increases in the $\nu(CO)$ frequencies.⁷ This is due to CO₂ insertion into the W-OR bond, producing $W(CO)_5OC(O)OR^-$, the tungsten pentacarbonyl aryl carbonate. This complex has been characterized by IR spectroscopy and ¹H and ¹³C NMR spectroscopies.⁸ The addition of small quantities

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(7) For example, the ν (CO) bands due to W(CO)₅OPh⁻ (2057 (w), 1903 (s), and 1852 (m)) shift to 2063 (w), 1910 (s), and 1854 (m) cm⁻¹. This reaction provides enhanced yields if performed in the presence of some carbon monoxide (preventing decomposition of the tungsten pentacarbonyl alkoxide to the tetramer, which precipitates from the solution). However, large excesses of CO will cause displacement of the aryl carbonate ligand, alternatively reducing yields

(8) [Et₄N] [W(CO)₅O₂COPh]: ¹H NMR (CD₃CN) (cation) δ 3.22 (q, CH₂), 1.186 (t, CH₃), (anion) 6.51–7.23 (m, C₆H₅); ¹³C NMR (CD₃CN) (cation) δ 52.91 (CH₂), 7.55 (CH₃), (anion) δ 159.8 (O₂COPh, identified by using ¹³CO₂ in synthesis), 108.8, 116.6, 129.9, 155.9 (C₆H₅).

⁽¹²⁾ 3 H-8(R)-HPETE was prepared by incubation of commercial [5,6,8,9,11,12,14,15-3H₈]arachidonic acid (specific activity 83.6 Ci/mmol, 20 μ Ci) and unlabeled arachidonic acid (1 mg) with 50 mg of C. viridis acetone powder in 2.0 mL of 100 mM Tris buffer pH 8.0 for 1 h. The product was purified by thin-layer chromatography (ether/hexane/acetic acid, 50:50:1) at 0 °C and used immediately (the tritiated 8-HPETE was much less stable than the unlabeled 8-HPETE). This provided 8-HPETE with a specific activity of 6.08 Ci/mol. Reduction of this material with $(MeO)_3P$ followed by HPLC analysis (radioactivity detection) established that this 8-HPETE preparation was uncontaminated by pre-clavulone A. (13) A retention time of 8.5 min was found by using a Zorbaz Sil 4.6 mm

^{× 25} cm column (Du Pont), hexane-THF (15:1), flow rate 2.0 mL/min.

⁽¹⁴⁾ Studies of the biomimetic synthesis of the pre-clavulone A system via allene oxide and oxidopentadienyl cation intermediates have shown the feasibility of this chemical route to prostanoids (Ritter, K.; Yus, M.; Najera, C. Corey, E. J., unpublished results). See also: Malacria, M.; Roumestant, M. L. Tetrahedron Lett. 1977, 33, 2813.

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Figure 1. Molecular structure and labeling scheme for $[Et_4N][W-(CO)_4(CO_3)]$ ·H₂O. The formula units are assembled as they occur in the unit cell.

of water to a THF solution of $W(CO)_5OC(O)OR^-$ causes bright orange crystals to begin to preciptate from the solution. Slow layering of diethyl ether and hexane to a concentrated solution at -11 °C yields crystals of $[Et_4N]_2[W(CO)_4(\eta^2-CO_3)] \cdot H_2O$. This complex is insoluble in THF but dissolves readily in the more polar solvents acetone and acetonitrile. The ¹³C NMR shows the presence of two carbonyl groups in equal ratios at 219.5 (CO's cis to the carbonate ligand) and 230.3 ppm (CO's trans to the carbonate ligand). The carbonate carbon appears at 161.1 ppm. The structure of the tungsten tetracarbonyl carbonate was unambiguously assigned on the basis of an X-ray crystallographic determination. This chemistry is summarized schematically below.

$$[\text{ROW}(\text{CO})_3]_4^{4-} \xrightarrow[-\text{CO}]{+\text{CO}} \text{ROW}(\text{CO})_5^{-} + \text{CO}_2 \rightarrow \\ \text{ROCO}_2 \text{W}(\text{CO})_5^{-} \xrightarrow[-\text{I}/_2\text{H}_2\text{O}]{+\text{CO}_3} \text{W}(\text{CO})_4^{2-}$$

The molecular structure and atomic numbering scheme of the tungsten tetracarbonyl carbonate is shown in Figure 1.9 The overall geometry of the dianion is that of a distorted octahedron. The carbonate ligand, CO_3^{-2} , is bound in a chelating manner to the tungsten(0) center. The average W-C bond for the carbonyl groups trans to the carbonate ligand (equatorial carbonyls) is 0.0825 Å shorter than the W-C bonds that are cis to the ligand. The axial carbonyl groups (2 and 4) are bent away from the carbonate ligand, creating a C(2)-W-C(4) angle of 165.7°. Similarly, the equatorial CO groups are pushed back from the carbonate ligand with bond angles of greater than 106° for the C–W–O angles of the carbonyl carbon with the carbonate oxygen. The bond angle of the carbonate oxygen atoms with the tungsten center is very small, 59.5°. The W-O bonds are about 2.20 Å, similar to the W-O bond length of 2.207 Å in [PPN][W(C-O)₅O₂CCH₃].¹⁰ There is one water molecule per complex which is hydrogen bonded through the distal oxygen atom (O(7)) of the carbonate ligand.

The sequence of reactions leading to formation of W- $(CO)_4CO_3^{2-}$ from W(CO)₅O₂COR⁻ is assumed to involve hy-

drolysis of the aryl carbonate complex to the bicarbonate complex^{2e,3c,11} with subsequent deprotonation and loss of CO affording W(CO)₄(CO₃)²⁻. Saegusa and co-workers have presented similar processes for copper(I) *tert*-butoxide leading to formation of CuO₂COH and Cu₂CO₃ depending on the quantity of H₂O added.^{11a}

Our mechanistic studies are focusing on the CO₂ insertion process. Thus far, quantitative rate data for CO₂ reacting with $W(CO)_5OR^-$ in THF to afford $W(CO)_5O_2COR^-$ reveal this process not to be retarded in large excesses of carbon monoxide.¹² Although the $W(CO)_5OR^-$ derivatives are CO labile, the reverse reaction with CO is also a facile process; i.e., there is no accumulation of $W(CO)_4(OR)(THF)^-$ in THF solution. Therefore, the presence of an open coordination site appears unnecessary for the CO_2 insertion process to occur. Hence, the reaction is proposed to take place via a concerted insertion process, quite similar to that proposed and well documented for the insertion of CO_2 into $RW(CO)_5^{-2a,b}$ Insertion occurs more readily in this instance; i.e., the reaction of CO_2 with $W(CO)_5OPh^-$ is over within 30 min at ambient temperature and atmospheric pressure, whereas under similar reaction conditions the reaction of CO_2 with $W(CO)_5R^$ requires days. A detailed assessment of the kinetic parameters associated with CO_2 insertion into W-OR bonds is under way.

Attempts were made to induce the insertion of CO into the W-OR bond of W(CO)₅OPh⁻. A tetrahydrofuran solution of W(CO)₅OPh⁻ was pressurized with 500 psi of carbon monoxide in an in situ high-pressure cell (CIR reaction cell, Spectra Tech Inc.) and the reaction monitored by FTIR at ambient temperature. After 20 h only ν (CO) bands ascribed to the W(CO)₅OPh⁻ species were observed. Upon heating to 125 °C, displacement of phenoxide occurred with concomitant production of $W(CO)_6$. The tungsten alkoxycarbonyl species may be observed spectroscopically for short periods of time by reacting $W(CO)_6$ with $[Et_4N][OPh]$ in THF. The initial IR spectrum shows carbonyl bands at 2032, 1888, and 1865 cm^{-1} as well as a weak band at 1640 cm^{-1} (for the alkoxycarbonyl ligand). These bands gradually shift to resemble the spectrum of $W(CO)_5OPh^-$. Ultimately, the only tungsten-phenoxo species isolable from this reaction is the tetrameric $4Et_4N^+$ W₄(CO)₁₂(OPh)₄⁴⁻, which precipitates from the reaction solution.

The observations that $W(CO)_5COOPh^-$ can be seen from the reaction of OPh⁻ with $W(CO)_6$ and not from the reaction of CO with $W(CO)_5OPh^-$ (i.e., a CO insertion into the W–O bond) lend support to the formation of the alkoxycarbonyl intermediate, $CH_3OC(O)W(CO)_5^-$, for the $W(CO)_6/KOMe$ catalyzed production of methyl formate from CO and methanol via nucleophilic addition of CH_3O^- to $W(CO)_6$.¹³ Furthermore, the formation of alkoxycarbonyl complexes of iridium is proposed to proceed through an intermediate in which the original OR⁻ ligand has been displaced by CO.¹⁴

In conclusion, evidence has been shown for the insertion of CO_2 into a metal alkoxide bond. Many previously reported metal carbonate complexes have been obtained as a result of the reductive disproportionation of CO_2 (eq 2).¹⁵ In these reactions, the two electrons are supplied by oxidation of the metal center from M(0)

⁽⁹⁾ Crystallographic data for $[Et_4N]_2[W(CO)_4(CO_3)]\cdot H_2O$: An orange crystal, 0.03 × 0.20 × 0.10 mm was mounted in a glass capillary in a random orientation. The crystal system was identified as triclinic, space group, Pl. Unit cell dimensions are a = 10.513 (3) Å, b = 15.881 (10) Å, c = 8.130 (6) Å, $\alpha = 101.69$ (6)°, $\beta = 92.12$ (5)°, $\gamma = 83.25$ (5)°; V = 1319.8 Å³, Z = 2, D(calcd) = 1.596 g cm⁻³. Data was measured using Mo K α radiation ($\lambda = 0.71073$ Å) on an Enraf-Nonius CAD4 computer-controlled κ -axis diffractometer equipped with a graphite crystal, incident beam monochromator, at the Molecular Structure Corporation. A total of 4812 reflections were collected, of which 4192 were unique, and 3834 of these with $F_o \geq 3\sigma(F_o)$. Lorentz and polarization corrections were applied to the data. The structure was solved by heavy atom methods and worked up by subsequent Fourier syntheses. All hydrogen atoms were located and isotropically refined. All non-hydrogen atoms were refined with anisotropic temperature factors; $R_F = 2.92\%$; $R_{wF} = 3.01\%$; R(int) = 2.0%.

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or M(I) to M(II) or M(III), respectively.

$$2\mathrm{CO}_2 + 2\mathrm{e}^- \to \mathrm{CO}_3^{2-} + \mathrm{CO} \tag{2}$$

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Orbital Correlation Barrier between the Reaction Channels for the Combination and Disproportionation of **Radical Pairs**

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The self termination reaction of organic free radicals have received increased attention in the recent literature. Experimental studies by Fischer,^{1,2} Ruchardt,³ Golden⁴ and others have suggested that certain radicals recombine without activation. Theoretical work has suggested that recombination without activation may not be general.^{5,6} Several experimental studies confirm this.

In this paper, we present orbital correlation arguments that indicate that combination and disproportionation of radical pairs should occur by separate, nonconverting reaction paths. These paths are illustrated by AM18 calculations on ethyl radical termination reactions.

The SOMO (singly occupied molecular orbital) of the ethyl radical is illustrated in pairs oriented for recombination and disproportionation in Figure 1. The topology of this orbital is evident from the fact that it is similar to the LUMO (lowest unoccupied orbital) of the ethyl cation, which is the antibonding combination of a carbon p-orbital with the methyl group orbital of correct (π) symmetry. It is immediately apparent that the phase is inverted in passing from C_1 to C_2 on the same face of the radical. Any incoming radical that came along the path toward combination with C_1 would begin to overlap with the part of the SOMO localized on the C₁ p-orbital. Since the part of the SOMO localized on the adjacent methyl hydrogen, which would be the hydrogen abstracted in a disproportionation reaction, is of opposite sign to that on C_1 , the radical approaching C_1 would necessarily have opposite sign to that of the SOMO on this hydrogen. Clearly, the interaction of the incoming radical to the methyl hydrogen would be antibonding. Consequently, a HOMO/LUMO crossing of the interacting radical pair would be necessary for the conversion of an approach along the combination path to an approach along the disproportionation path. The converse is obviously true by analogous arguments (see Figure 1 for the two approaches).

As a probe of this phenomenon, AM1 molecular orbital calculations⁸ were performed on the approach of a hydrogen atom to the ethyl radical. The ethyl radical was, first, completely optimized using the half electron method.⁹ A test hydrogen atom





COUPLING DISPROPORTIONATION Figure 1. Ethyl radical orbitals oriented for combination and disproportionation.



Figure 2. Potential surface for the approach of a hydrogen atom to an ethyl radical showing the two reaction channels. The energy is plotted vs. distance parallel to the C-C bond in the plane of the C-H bond and the nonbonding p-orbital of the ethyl radical (zero is defined as exactly above the methylene carbon) and the distance from the C-C bond (from 2 to 3 Å) in the same plane. Each division represents 0.1 Å. The height of the surface is 3.0 kcal/mol.

was then placed at various positions with respect to the ethyl radical. The energies for the resulting radical pairs were calculated by an open-shell SCF technique and 3×3 CI. The utility of these procedures for the calculation of bond dissociations in small molecules has been previously discussed.^{10,11} It is worth noting that the artifacts reported with the MNDO techniques¹⁰ all disappear when the calculations are repeated with AM1, for the cases containing only C, H, N, and O (the only elements for which AM1¹¹ parameters were available). The results are displayed in Figure 2, which is an energy surface for the radical pair with the H atom positioned in the plane of the two carbons and perpendicular to the methylene group.

The barrier between the two paths is clearly observable in Figure 2. In addition, one can see that the downward slope of the energy with approach of the radical pair is initially greater for attack at the hydrogen (disproportionation) than for attack at carbon (combination). This seems reasonable as the methyl hydrogen protrudes in the direction of the incoming radical. Upon further approach, however, the valley leading to combination deepens more than that leading to recombination.

There is a simple experimental demonstration of this phenomenon. Upon heating, alkanes generally are thought to first break their C-C bond to form two alkyl radicals, which can then react further by disproportionation, combination, or other reactions. The activation energy is equivalent to the bond dissociation energy. If a viable path that allowed interconversion from the path of combination (the reverse of dissociation) to disproportionation were to exist, alkanes could be converted directly into disproportionation products with an activation energy significantly lower than that for bond dissociation.

The implication of these results, that once embarked along one of the two reaction paths, a radical pair can no longer be diverted to the other product, requires some rethinking of theories of radical self-reactions that depend only upon consideration of steric factors.

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