= −24.4 cal K^{-1} mol^{-1}) compare with those for the $Fe(edta)^{2-}$ + cytochrome b_5(III) reaction ($\Delta H^{\ddagger}$ = 5.4 kcal mol^{-1}; $\Delta S^{\ddagger}$ = −29.2 cal K^{-1} mol^{-1}), suggesting similar influences.

Further information has been obtained from the redox-inactive complex $[Cr(en)_3]^{3+}$. Thus, with the 3+, 4+, and 5+ oxidants competitive inhibition is observed (K_{Cr} = 309 M^{-1}), and it can be concluded that either a single specific site or kinetically indistinguishable sites are involved. The accelerated effect of $[Cr(en)_3]^{3+}$ on the $[Co(edta)]^-$ reduction and fit of data suggest that $Co(edta)^-$ is able to use this same site when redox-inactive $[Cr(en)_3]^{3+}$ is present and may also use this site when $[Cr(en)_3]^{3+}$ is not present, although this is not absolutely essential. The situation is very similar to that observed previously for the oxidation of [2Fe-2S] and 2[4Fe-4S] ferredoxins by inorganic oxidants.[19]

Some concern is often expressed as to whether studies with inorganic complexes relate to natural processes involving protein-protein reactions. As a part of this work[7] it has been demonstrated that $[Cr(en)_3]^{3+}$ not only inhibits the reaction of cytochrome b_5 with the 3+, 4+, and 5+ complexes but also blocks association with cytochrome c(III), which is believed to be a natural partner for cytochrome b_5. An overlap of the site used by cytochrome c with that used by the inorganic complexes is indicated. We have no reason to believe that the reactivity here outlined will change appreciably when cytochrome b_5 is present as the 134-residue protein or when it is membrane bound, since E° values remain practically unchanged for these different situations.[4]

Much current information suggests that the exposed heme edge of a cytochrome is relevant to electron transfer. Crystal structure information is now available for cytochromes b_5,[1,3] c,[20] and c_{551},[21] a particular feature being that all three differ in the immediate environment of the heme edge. That of cytochrome b_5 has already been considered and the importance of negative residues surrounding the heme edge indicated. In cytochrome c the heme is surrounded by basic residues. Although $[Fe(CN)_6]^{3-}$ appears to react preferentially at a site close to Lys 72 (which in the normal view is to the left of the heme edge) of cytochrome c(III),[22] there is no compact positive patch such as would be needed for strong association with inorganic complexes. Thus, for reduction of cytochrome c(III) by $[Fe(CN)_6]^{4-}$ it has been demonstrated that $K < 200$ M^{-1}.[23] However NMR line-broadening studies indicate an association with $[Fe(CN)_6]^{3-}$ having K = 450 M^{-1} at I = 0.18 M.[24] This is believed to refer to a 3+/4+ charged patch in the 86-91 region which is unlikely to contribute extensively to electron transfer. The positive complex $[Co(phen)_3]^{3+}$ has been shown to react at a site close to Lys 27 on the right-hand side of the heme edge; overlap with the exposed heme edge occurs from this position as well as the site used by $[Fe(CN)_6]^{3-}$. The heme edge in cytochrome c_{551} is surrounded by hydrophobic residues, and further data illustrating the effect of these residues on reactions with inorganic complexes would be of interest. Recent studies in which "fixed-site" electron transfer from Ru(II) attached to the His 33 of cytochrome c through to the Fe(III) active site has been determined[25,26] highlight the possibility of small contributions resulting from long-distance (15 Å) electron transfer without use of the exposed heme edge.

An alternative explanation of (13) is in terms of the "dead-end" mechanism.[19] As in other studies the stance we have adopted is that discussion should proceed in terms of e.g. (11) and (12) until positive evidence for this alternative is obtained. The same numerical K values apply in both mechanisms.

The strategy described with $[Cr(en)_3]^{3+}$ as an inhibitor has possible applications with enzymes such as sulfite oxidase. The latter has cytochrome b_5 and Mo active sites,[27] and such an approach could have relevance in diagnosing which of these is used as electron "in" and "out" sites in electron-transfer processes.

Acknowledgment. We wish to thank the U.K. Science and Engineering Research Council for postdoctoral (D.M.D. and C.P.J.V.) and postgraduate (S.K.C.) support. We also thank Drs. A. G. Mauk and L. S. Reid for comments.

Registry No. Na[Co(edta)], 89196-49-6; $[Co(NH_3)_6]Cl_3$, 10534-89-1; $[Pt(NH_3)_6]Cl_4$, 16893-12-2; $[(NH_3)_5CoNH_2Co(NH_3)_5]Br_5$, 72273-61-1; $[Cr(en)_3]Cl_3$, 21510-38-3; cytochrome b_5, 9035-39-6.

Supplementary Material Available: A listing of rate constant Tables I-VII (7 pages). Ordering information is given on any current masthead page.

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Communications to the Editor

Preparation of 2-Methylenebenzocyclobutenone by the Flash Vacuum Pyrolysis of 3-((Benzoyloxy)methyl)benzofuran[1]

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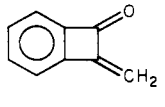
Although a few substituted methylenebenzocyclobutenones have been prepared,[2] the parent compound (**1**), which is one of the



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simple derivatives of cyclobutadiene, has not been reported. The flash vacuum pyrolysis (FVP) of furfuryl benzoate (**2**) is a convenient source of methylenecyclobutenone (**3**).[3] Several sub-

(1) Presented at the 184th Meeting of the American Chemical Society, Kansas City, MO, Sept 1982, Abstract ORGN 138.

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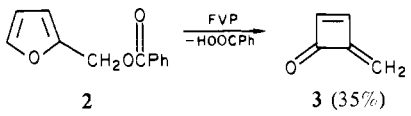
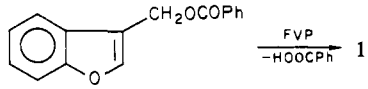


2 3 (35%)

stituted methylenecyclobutenones have been prepared by this route,[4] but this route cannot be used to prepare **1**. The precursor of **1** would have to be an isobenzofuran which would be too reactive to be pyrolyzed under FVP conditions. In this communication we report the synthesis of **1** by a route involving the FVP of 3-((benzoyloxy)methyl)benzofuran (**4**).



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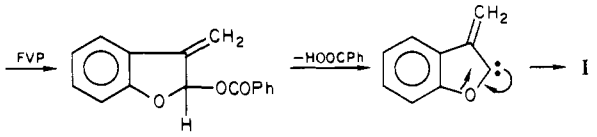
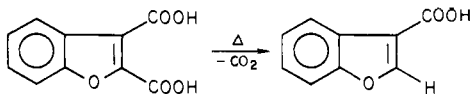
On the basis of our previous work with furans, we anticipated that under FVP conditions **4** would produce, via **5**, carbene **6**,



5 6

which would rearrange to **1** by a 1,2 shift, a rearrangement for which there is good precedent.[5]

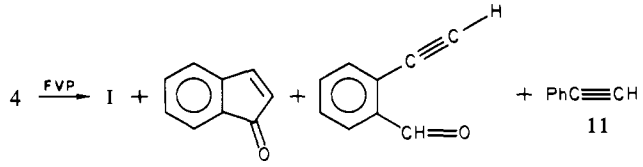
Ester **4**[6] was prepared by the lithium aluminum hydride reduction of 3-benzofurancarboxylic acid (**7**) to the alcohol, which



8 7

was esterified with benzoyl chloride. Acid **7** was prepared by a known sequence of reactions starting from ethyl phenoxyacetate.[7] The last step of this sequence is the decarboxylation of diacid **8**. The reported procedure, heating **8** to 250 °C under vacuum, gave poor yields of **7** and was difficult to scale up, but we have found that gram quantities of **8** can be converted to **7** in >80% yield by FVP[8] at 700 °C, 10^{-4} torr.

Ester **4** was pyrolyzed at several temperatures under our standard FVP conditions.[8] The desired product, **1**, was obtained along with several other products,[9] indenone (**9**),[10] *o*-ethynyl-



9 10

Table I. Products from Pyrolysis of 3-((Benzoyloxy)methyl)benzofuran (**4**) at Various Temperatures[a]

	yield, %[b]				
temp, °C	1	9	10	11	4
700–750		36.9	11.9		
600–680	24.1	21.6	3.0	<1	38.5
500–580	26.5	3.3		<1	40.0
400–480					100

[a] Sample size 300 mg; pressures ca. 10^{-4} torr; sample chamber temperature ca. 60 °C. [b] Yields were determined by ^{1}H NMR (=CH_2 of **1**, CH=O of **9**, =CHCO of **10**, and ≡CH of **11**) using diphenylmethane ($PhCH_2Ph$) as an internal standard and are based on two or more runs. Benzoic acid was also produced.

benzaldehyde (**10**),[11] and phenylacetylene (**11**).[12] The yields of these products and recovered starting material at various pyrolysis temperatures are presented in Table I.

Even at the lower temperatures, some of **9**, which is difficult to separate from **1**, is produced. Michael additions to **1** are expected to be slow because the intermediate enolate must have the anti-aromatic benzocyclobutadiene structure. Indeed, **1** was obtained in a pure form by treating the product mixture with pyrrolidine, which selectively adds to **9**. After **9** was destroyed, the amine was neutralized with acetic acid, and **1**[13] was purified by flash chromatography.[14]

Compound **1** was also obtained free of **9** by taking advantage of the more rapid Diels–Alder reaction with cyclopentadiene of **9** relative to **1**. An excess of cyclopentadiene was added to the pyrolysis product mixture in the liquid nitrogen-cooled trap. The mixture was warmed to 0 °C, and after 30 min at 0 °C, the excess cyclopentadiene was removed under reduced pressure. Pure **1** was obtained by flash chromatography.[15]

The amine–acid method of purification can be carried out more quickly but requires considerable care in measuring the amounts of reagents and in controlling the reaction time. In the cyclopentadiene method, little or none of **1** is destroyed and generally slightly higher yields are obtained by this procedure.

Reasonable pathways to **9** and **10** involve the allene–ketene formed by the electrocyclic ring opening of **1**, and decarboxylation of **10** is the likely source of **11**. Support for these pathways was obtained by pyrolyzing pure **1** (obtained as described below). FVP of **1**, dissolved in diphenyl ether to minimize polymerization during the pyrolysis experiment, at 700 °C gave recovered starting material (**1**) and **9**, **10**, and **11** in a ratio of 25:15:5:1.

(4) (a) Trahanovsky, W. S.; Alexander, D. L. *J. Am. Chem. Soc.* **1979**, *101*, 142. (b) Trahanovsky, W. S.; Cassady, T. J.; Woods, T. C. *Ibid.* **1981**, *103*, 6691.

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(6) Compound **4**: IR ($CDCl_3$) 3040, 2880, 1720, 1600, 1590, 1450, 1270 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 8.2–6.9 (m, 10 H), 5.45 (s, 2 H); molecular ion (calcd for $C_{16}H_{12}O_3$, *m/e* 252.07865) 252.07846 ± 0.8 ppm.

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(9) Products were identified by comparison of the IR and ^{1}H NMR spectra of the product mixtures to those reported for the compounds[10-12] and by ^{13}C NMR and GC-MS analysis of the product mixtures.

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(13) Compound **1**: IR ($CDCl_3$) 3010, 1770, 1740 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.5–7.3 (m, 4 H), 5.47 (d, *J* = 1.47 Hz, 1 H), 5.24 (d, *J* = 1.46 Hz, 1 H); ^{13}C NMR (from Me_4Si) δ 186.1 (C=O), 159.6, 156.4, 156.1, 135.0, 130.2, 121.6, 120.2, 102.1 (=CH_2); molecular ion (calcd for C_9H_6O, *m/e* 130.04187) 130.094191 ± 0.3 ppm. GC–MS (20 eV) *m/e* (relative abundance) 130 (100), 103 (3.2), 102 (38.6), 76 (1.2), 75 (.6); UV (EtOH) λ_{max} 250 (ε 9000), 310 nm (1500).

(14) Amine–acid procedure for preparing pure **1**: A quantity of 0.837 g (3.32 mmol) of **4** was pyrolyzed (530 °C, ca. 2.5 × 10^{-4} torr) for 3 h. To the product trap was added ca. 0.5 mL of $CDCl_3$ and a weighed amount of 1,2-dimethoxyethane as standard. ^{1}H NMR analysis of this mixture showed the presence of **1** (112.3 mg, 0.86 mmol, 26% yield) and **9** (17.3 mg, 0.13 mmol, 4% yield). The mixture was added to 1 mL of anhydrous ether. To the mixture was added 11.3 mg (0.16 mmol) of pyrrolidine in anhydrous ether. After 30 s 9.57 mg (0.16 mmol) of acetic acid in ether was added to the mixture. After 2 min 1.0 g (11.9 mmol) of solid $NaHCO_3$ was added to the mixture, and the mixture was stirred at room temperature for 2 h. The mixture was filtered and subjected to flash chromatography using silica gel 60 EM reagent (230–400 mesh) and anhydrous ether as eluent. A quantity of 55.6 mg (0.43 mmol, 13% yield) of **1**[13] was obtained as a yellow oil.

(15) Cyclopentadiene procedure for preparing pure **1**: After pyrolysis of 0.393 g (1.56 mmol) of **4**, ca. 5 mL of cyclopentadiene was added to the product trap thorugh a side arm. Nitrogen gas was introduced into the system, and the product mixture was warmed to 0 °C and held at 0 °C for 30 min. The excess cyclopentadiene was removed under reduced pressure, and **1** was purified by flash chromatography (two passes on the silica gel column described above[14] using 10% ether in hexane as eluent).

Further studies of the chemistry of **1** are in progress.

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Registry No. 1, 88180-40-9; 4, 88180-41-0; 7, 26537-68-8; 7 alcohol derivative, 4687-23-4; 8, 131-76-0; 9, 480-90-0; 10, 38846-64-9; 11, 536-74-3; ethyl phenoxyacetate, 2555-49-9.

Bifunctional Activation of CO_2: A Case Where the Basic and Acidic Sites Are Not Held in the Same Structure

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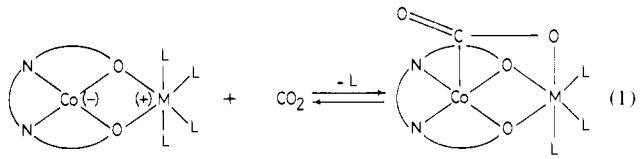
Since the discovery by C. Floriani that the bifunctional complexes Co(R-salen)M [R-salen = substituted salen ligand; salen = *N,N'*-ethylenebis(salicyldeneaminato); M = Li, Na, K, Cs] can activate CO_2 (1),[1] coordination chemistry has been pervaded by



(1)

intense efforts to find acidic–basic metal systems capable of promoting CO_2. Unfortunately, the R-salen complexes are so far unique examples of this fascinating chemistry.

We were intrigued by the possibility that CO_2 could be activated also by bifunctional metal systems that do not fulfil the limiting requirement of holding the basic and acidic centers in the same structure.

This communication presents the reactions of CO_2 with the low-valent cobalt or rhodium complexes $(np_3)CoH$ (**1**)[2] and $(np_3)RhH$ (**2**)[3] [np_3 = tris(2-(diphenylphosphino)ethyl)amine] in the presence of a solvated or complexed Lewis acid such as the sodium ion.

On bubbling of CO_2 at room temperature into a tetrahydrofuran solution of **1**, no reaction is observed even for long reaction time (24 h), the starting complex being quantitatively precipitated by addition of a solvent such as *n*-butyl ether.

By contrast, on addition of a tetrahydrofuran solution of $NaBPh_4$ to a solution of **1** under CO_2 atmosphere, a rapid reaction takes place and the original red-orange color changes to brown-green. The solution turns red-brown within 1 h, indicating the completion of the reaction. Addition of 1-butanol to the reaction mixture and partial evaporation of the solvent cause the precipitation of red crystals of the carbonyl complex $[(np_3)Co(CO)]BPh_4$ (**3**),[2] which optionaliy can be filtered off. On further concentration the precipitation of the carbonyl complex is accompanied by that of pale violet crystals of a compound that analyzes as $[(np_3{=}O)Co](BPh_4)_2$ (**4**) $[np_3{=}O = O{=}PPh_2CH_2CH_2N(CH_2CH_2PPh_2)_2]$ (μ_{eff} = 4.35 μ_B; 1140 cm^{-1} P=O stretching). Both compounds **3** and **4** can be isolated as pure samples, each of them with yields varying between 40% and 50%.

The transformation of **1** into **3** (yield 50%) is more easily obtained if the sodium ions are complexes by a crown ether like dicycloesano-18-crown-6 ($C_{20}H_{36}O_6$) before being reacted with **1** and CO_2. In this case it is sufficient to bubble CO_2 into the reaction mixture for a few minutes to have a complete reaction. Furthermore, the color of the solution changes directly from red-orange to red without assuming the initial green tinge. The sodium ions can be quantitatively collected as the crown ether complex with one tetraphenylborate counterion, whereas a minor amount of **4** (yield 5%) is formed.

It is noteworthy that CO_2 does not react at all with **1** in the presence of NBu_4BPh_4.

Analogously to **1**, a tetrahydrofuran suspension of the rhodium complex **2** reacts within a few seconds with CO_2 only in the presence of sodium ions to give the novel yellow-green diamagnetic complex $[(np_3)Rh(CO)]BPh_4$ (**5**) (yield 50%; 1990 cm^{-1} CO stretching).

In the attempt at understanding these reactions and building up a possible mechanism, the following experimental pieces of information are noteworthy. (a) The complex $(np_3)CoH$ is known to react with CO_2-like molecules such as RNCO, RNCS, and CS_2 to give the corresponding η^2-complexes $(np_3)Co(\eta^2\text{-}CXY)$ (CXY = heteroallene).[4] Analogously, the $(np_3)RhH$ complex has been found to react with CS_2 to yield an η^2-CS_2 complex.[5] (b) At present no definitive conclusions have been reached about the role played by the Co–H hydrogen in these reactions. By analogy with the isoelectronic complexes $[(np_3)NiH]BPh_4$, which can react with CO to give the nickel(0) complex $[(Hnp_3)Ni(CO)]BPh_4$,[6] and $Co(CO)_4H$, which dissociates according to the equation $Co(CO)_4H \rightleftharpoons Co(CO)_4^- + H^+$,[7] we could suggest that an equilibrium of the type $(np_3)CoH \rightleftharpoons (np_3)Co^- + H^+$ may be operating.[8] However, other conceivable pathways such as that involving a preliminary reaction between the Co–H moiety and an heteroallene molecule, cannot be excluded. (c) Dicycloesano-18-crown-6 forms sodium complexes without saturating the coordination sphere of the alkali metal. Other coligands such as water molecules can coordinate sodium.[10] (d) The formation of the complex (triphos)Ni(CO) and a [(triphos=O)Ni] species [triphos = 1,1,1-tris((diphenylphosphino)methyl)ethane] by reaction of CO_2 with the (triphos)Ni(0) moiety has been recently suggested to proceed through the intermolecular attack by a phosphorus atom from coordinated triphos on the CO_2 molecule of the intermediate species $(triphos)Ni(CO_2)$.[11]

In absence of a detailed mechanistic study the stepwise pathway (2) may be proposed for the reaction of **1** with CO_2 in the presence of sodium ions. A similar pathway can be proposed also for the reaction with the rhodium derivative, the only difference being the absence of the corresponding $np_3{=}O$ complex.

Concerning the formation of the intermediate η^1-CO_2 adduct, the presence of the sodium cations seems essential for anchoring the CO_2 molecule, which then may be thought of as being attacked by the $(np_3)Co$ fragment. It is well-known, in fact, that η^1-CO_2 coordination is attainable when the metal atom is electronically

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(8) Recent experimental and theoretical studies on the chemistry of **1** suggest that, depending on the reaction conditions, this hydride complex is a potential releaser of hydridic hydrogen, atomic hydrogen, or proton giving rise to the moieties $(np_3)Co^+$, $(np_3)Co\bullet$, and $(np_3)Co^-$, respectively.[9] The former moiety has been isolated as BPh_4^- or BF_4^- salts and does not react with CO_2 and related heteroallene molecules, whereas the radical moiety could explain the formation of the paramagnetic η^2-heteroallene complexes.[4] The d^{10} fragment $(np_3)Co^-$, which is isoelectronic with the trigonal-pyrimidal complex $(np_3)Ni$,[2] should have also the same geometry. In this case the lone pair directed toward the unoccupied site of the bipyramid could favor a C-coordination of the X=C=Y molecules (X, Y = O, S, NPh), which are electrophilic at the central carbon atom.
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