formed in similar yield when toluene solutions of **1** are treated with 1 equiv of tri-*n*-butylphosphine (PBu_3) at 70 °C for 2 h followed by evaporation and trituration with aqueous methanol. This unusual species can be easily crystallized from cold hexane. The ^1H NMR spectrum of **2** shows three nonequivalent Cp* ligands (1:1:2) and its structure is suggested to resemble that for $Cp_4Fe_4S_2(S_2)_2$.[7] The conversion of **1** into a larger cluster is reminiscent of our previous observation that $(MeCp)_2V_2S_4$ reacts with PBu_3 to give $(MeCp)_4V_4S_4$.[8]

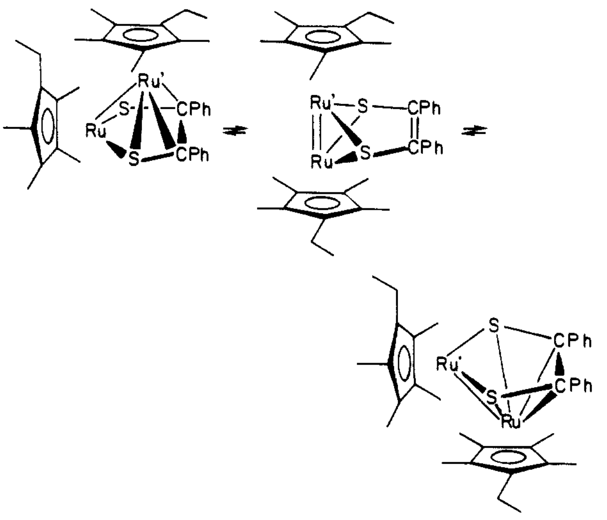
When toluene solutions of **1** are treated with PBu_3 (2 equiv) *in the presence* of diphenylacetylene (1 equiv, 70 °C, 2 h) one obtains, after solvent evaporation and trituration with methanol, $Cp^*_2Ru_2S_2C_2Ph_2$ (**3**) as red brown crystals from pentane in ca. 70% yield (eq 1).[9] It is important to note that compound **1** does not react with Ph_2C_2 (2 equiv, 70 °C, 2 weeks). Furthermore compound **2** is not an intermediate in the dithiolene synthesis since it can be recovered in good yield after attempted reaction with Ph_2C_2 (2 equiv), alone or in the presence of PBu_3 (2 equiv). The dithiolene synthesis also works well for acetylene and phenylacetylene.[10] The reaction of **1**, PBu_3 (*1 equiv*), and C_2H_2 (2 atm) gave ~25% yield of $Cp^*_2Ru_2S_2C_2H_2$;[10] chromatographic workup of the products returned ~35% yield of **1**.

$$Cp^*_2Ru_2S_4 + R_2C_2 + 2PBu_3 \rightarrow Cp^*_2Ru_2S_2C_2R_2 + 2SPBu_3 \quad (1)$$

$$R_2 = Ph_2\ (\mathbf{3}),\ PhH,\ H_2$$

An X-ray diffraction study shows that compound **3** is a square-pyramidal, nido cluster (Figure 2).[11] The most distinctive structural feature of **3** is the bridging dithiolene ligand which is folded over so as to bind to one metal in an η^4 manner. The dithiolene and the Cp* ring carbon atoms are nearly equidistant (±0.02 Å) from Ru(2). Bridging 1,2-dithiolene ligands are common[12] but the present μ-η^2,η^4 form is unique.[13] Structurally, **3** is related to certain diazabutadiene complexes, e.g., $Mn(_2(\mu$-η^2,η^4-$CH_3NCHCHNCH_3)(CO)_6$,[14] and to the binuclear ferroles $Fe_2(C_4R_4)(CO)_6$.[15] If the μ-η^2,η^4-$R_2C_2S_2$ ligand is viewed as a neutral 8e$^-$ donor, **3** is seen to be an electron-precise 34e$^-$ species. The Ru–Ru distance in **3** is 2.980 (1) Å whereas the Ru–Ru distance in **1** is nonbonding at 3.749 (1) Å.

The ^1H NMR spectrum of **3** consists of four methyl singlets and two methyl triplets showing that the C_5Me_4Et ligands are nonequivalent but suggesting that both lie on a plane of symmetry. At 150 °C in $C_6D_5NO_2$ solution, the separation of the closer pair of methyl singlets in its ^1H NMR spectrum ($\Delta\delta$ = 5.5 Hz at 40 °C) narrows to 1.5 Hz. Therefore ΔG^* for the equivalencing of the two Ru centers is somewhat greater than 95 kJ/mol. We propose that a dynamic equilibrium of the type shown in eq 2 is involved. Conceivably, related equilibria but with different energy minima apply to other dithiolene bridged complexes.



(2)

The mechanism of the conversion of **1** into **3** and the generality of the structural motif illustrated by **3** are of further interest.

Acknowledgment. This research was funded by the National Science Foundation and, in part, the Illinois Coal Research Board. We also acknowledge the support of the donors of the Petroleum Research Fund, administered by the American Chemical Society. T.B.R. is grateful to the Camille and Henry Dreyfus Foundation for a Teacher–Scholar Fellowship.

Supplementary Material Available: Tables of bond distances, bond angles, thermal parameters, fractional coordinates, and structure factor tables for $Cp^*_2Ru_2S_2C_2Ph_2$ and $Cp^*_2Ru_2S_4$ (43 pages). Ordering information is given on any current masthead page.

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(8) Bolinger, C. M. Ph.D. Thesis, University of Illinois at Urbana—Champaign, 1984.

(9) Anal C, H, S. FABMS (*m/z*) 744 (P^+), 566 ($P^+ - Ph_2C_2$); ^1H NMR (C_6D_6) 7.33 and 6.96 (m, 10 H, C_6H_5), 2.50 (q, 2 H, 7.5), 2.20 (q, 2 H, 7.7), 2.04 (s, 6 H), 2.01 (s, 6 H), 1.71 (s, 6 H), 1.66 (s, 6 H), 1.07 (t, 3 H, 7.5); 0.85 (t, 3 H, 7.5).

(10) Characterized by ^1H NMR and FAB mass spectrometry.

(11) Compound **3** crystallized from pentane in the space group $P2_1/n$, with cell dimensions a = 13.222 (3) Å, b = 16.312 (4) Å, c = 15.797 (2) Å, V = 3299 (2) Å^3, β = 104.48 (2)°, Z = 4, ρ_{exp} = 1.47 g cm^{-3}. for $\pm h,-k,+l$ in the range $2.0° < 2\theta < 46.0°$. These were averaged to (R_{av} = 0.017). The structure, 4576 independent reflections, was solved by direct methods (SHELX), refined with use of 3489 intensities ($I > 2.58\sigma(I)$) to R = 0.033 and R_w = 0.046.

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Selective Deoxygenation of Secondary Alcohols by Photosensitized Electron-Transfer Reaction. A General Procedure for Deoxygenation of Ribonucleosides[1]

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Received December 23, 1985

While photosensitized electron-transfer reactions are currently attracting considerable mechanistic interest in organic photochemistry,[2] synthetic methodology based on these reactions has been rather limited.[3] We report a general and practically useful

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0002-7863/86/1508-3115$01.50/0

Scheme I

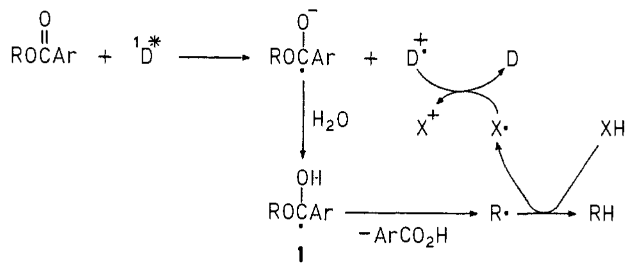


Table I. Photoreduction of the Esters **2a–c** in the Presence of MCZ and Calculated ΔG Values[a]

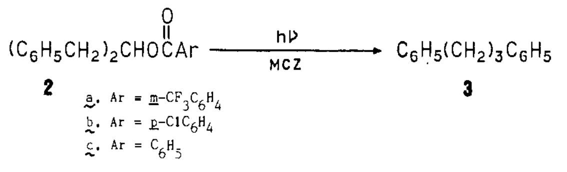
Ar	irradiatn time, h	conversn, %	**3**,[b] %	recovery of MCZ, %[b]	ΔG,[c] kcal mol^{-1}
m-$CF_3C_6H_4$	6	100	91	98	−20.1
p-ClC_6H_4	10	100	68	98	−19.7
C_6H_5	10	67	82	85	−6.5

[a] In THF–water (10:1) containing **2** (1.9 mM) and MCZ (1.9 mM). [b] Isolated yield. [c] Calculated values, using Rehm–Weller equation[12] and the thermodynamic data: $E_{1/2}^{\mathrm{red}}$(**2a**–c) and $E_{1/2}^{\mathrm{ox}}$(MCZ) were measured by cyclic voltammetry in dioxane–water (10:1) and acetonitrile, respectively. Details will be reported elsewhere.

method for selective deoxygenation of secondary alcohols via benzoates by use of photosensitized electron-transfer reaction. Among a variety of methods for alcohol deoxygenation,[4] tin radical cleavage of thiono esters developed by Barton and co-workers[5] has been widely used and was particularly effective for the conversion of ribonucleosides to 2′-deoxyribonucleosides.[6,7] However, for practical alcohol deoxygenation a more convenient and selective method that may be accomplished in neutral aqueous solvents under milder conditions avoiding the consumption of expensive reagents such as tri-*n*-butyltin hydride and thiocarbonyl reagents is clearly desirable. The present photosensitized deoxygenation utilizing simple benzoate esters occurs readily at or below room temperature in aqueous organic solvents and has high selectivity toward secondary alcohols in preference to primary alcohols. Dideoxygenation of 1,2-diols can be readily accomplished by this procedure.[8]

We envisioned the possibility that photosensitized electron transfer from a suitable electron-donor sensitizer (D) to an aromatic ester might generate the tertiary radical **1** by protonation of the resulting anion radical in aqueous solvent (Scheme I).[4,9] If the ester of a secondary or teriary alcohol is used, the radical **1** would undergo β-scission to produce stabilized secondary or tertiary alkyl radical which may abstract hydrogen from hydrogen-donating solvents to furnish a deoxygenated product. Previously, nonselective deoxygenation of alcohols by irradiation of the esters with 254-nm light in hexamethylphosphoric triamide (HMPTA) has been reported by Portella et al.[10] The photoreaction has been proposed to proceed via one electron transfer from a photoexcited HMPTA to esters.[10c] In such a system, however, complications occur when compounds having other chromophores in addition to ester groups are used.

Irradiation of a THF–water (10:1) solution of *m*-(trifluoromethyl)benzoate **2a** and *N*-methylcarbazole (MCZ)[11] through a Pyrex filter with a 100-W high-pressure mercury lamp under nitrogen gave 1,3-diphenylpropane (**3**) in 91% yield along with *m*-(trifluoromethyl)benzoic acid (95%) with the recovery of MCZ (98%). Polymerized product derived from THF was also detectable.

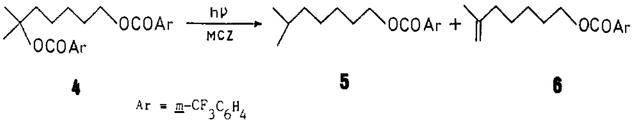


$(C_6H_5CH_2)_2CHOC(=O)Ar \xrightarrow[\text{MCZ}]{h\nu} C_6H_5(CH_2)_3C_6H_5$

2 **3**

a, Ar = *m*-$CF_3C_6H_4$
b, Ar = *p*-ClC_6H_4
c, Ar = C_6H_5

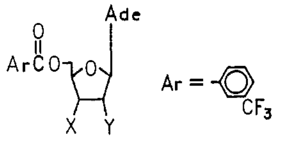
In control runs it was confirmed that no reaction occurs in the dark or in the absence of MCZ. Irradiation of other esters (**2b,c**) also produced **3** but in lower yields. In Table I are listed the results and the calculated values of the free energy change (ΔG) associated with the electron-transfer process, using the Rehm–Weller equation.[12] The near-diffusion-controlled rate (k_q = 4.5 × 10^9 M^{-1} s^{-1} in THF–water) for quenching of MCZ fluorescence by **2a** and the large negative value of ΔG suggest an electron transfer from singlet MCZ to **2a**. By contrast, irradiation of diester **4** (2.9 mM) and MCZ (1 equiv) in isopropyl alcohol–water (10:1) led to the selective reduction of the ester of a tertiary alcohol to give a 1:1 mixture of **5** and **6** in 85% yield



Ar = *m*-$CF_3C_6H_4$

probably via the disproportionation of the resulting tertiary alkyl radical,[9] indicating the inertness of the ester of a primary alcohol toward the photosensitized irradiation.

Irradiation of 2′,3′,5′-tri-*O*-[*m*-(trifluoromethyl)benzoyl]adenosine (**7**) in the presence of MCZ (1.1 equiv) in isopropyl



7 X = Y = OCOAr

8 X = Y = H

alcohol–water (10:1) gave the 2′,3′-dideoxyadenosine **8** (73%) and the corresponding acid (96%) with the recovery of MCZ (67%).[13] The result also indicates a higher reactivity of the C2′ and C3′ esters compared to the C5′ ester. Irradiation of 2′-*O*-[*m*-(trifluoromethyl)benzoyl]-3′,5′-*O*-TPDS-uridine (**9**, 1.0 mM) and MCZ (1 equiv) in isopropyl alcohol–water under similar conditions for 3 h gave the protected 2′-deoxyuridine **10** (85%), providing an alternative to the tin radical 2′-deoxygenation of ribonucleosides.[6] When the same photoreaction was conducted in CD_3CD_2OD–water (8:1) and the resulting 2′-deuterio-2′-deoxyuridine epimers (**11**) were purified by silica gel TLC, the ratio of ribo (α)/arabino (β) deuterium incorporation as determined by 400-MHz ^1H NMR was 86:14. The epimer ratio was very similar to the value reported for the deoxygenation of a protected adenosine with tri-*n*-butyltin deuteride:[6c] no deuterium incorporation from ethanol–D_2O was observed. These results strongly

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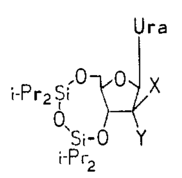
(11) Other electron-donor sensitizers such as 1,2,4,5-tetramethoxybenzene, 1,5-dimethoxynaphthalene, and 1-(dimethylamino)naphthalene were also effective. However, prolonged irradiation is necessary and the sensitizers are consumed considerably during irradiation in these cases.

(12) Rehm, D.; Weller, A. *Isr. J. Chem.* **1970**, *8*, 259.

(13) A typical procedure is as follows. A solution of **7** (450 mg, 0.58 mmol) and MCZ (114 mg, 0.63 mmol) in isopropyl alcohol–water (10:1, 500 mL) was irradiated with a 400-W high-pressure mercury lamp through Pyrex filter under nitrogen at room temperature. After confirmation of complete disappearance of **7** (~15 h), the residue was treated with a mixture of ethyl acetate and saturated $NaHCO_3$ solution. Extractive workup followed by preparative TLC (silica gel, $CHCl_3$–methanol 10:1) gave **8** (171 mg, 73%) and MCZ (78 mg). From the aqueous layer *m*-(trifluoromethyl)benzoic acid (210 mg, 96%) was recovered.

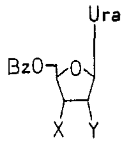
J. Am. Chem. Soc. **1986**, *108*, 3117-3118



9 $X = H$, $Y = OCOC_6H_4$-*m*-CF_3

10 $X = Y = H$

11 $X = H(D)$, $Y = D(H)$ (~86%)



12 $X = Y = OBz$

13 $X = Y = H$

suggest the intervention of a planer sp^2 radical on the C2′ which abstracts hydrogen on the less hindered α (ribo) face.

A more convenient deoxygenation method is to use less expensive benzoyl esters. Photoreaction of tri-*O*-benzoyluridine **12** (1.4 mM) and MCZ (1 equiv) in isopropyl alcohol–water proceeded only slowly to give a poor yield of **13** even after a 24- h irradiation. Interestingly, addition of magnesium perchlorate (1.4 mM)[14] to the reaction system remarkably accelerated the photoreduction to produce a high yield (85%) of **13** at 18 h of irradiation with the recovery of MCZ (78%).[15]

The present method provides access to the generation of free radicals selectively from secondary hydroxyl groups of complex molecules under mild aqueous conditions, thus broadening the application of free radical reactions in organic synthesis. Further applications of this photosensitized electron-transfer reaction as well as the mechanistic study are under investigation.

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(15) Throughout this photosensitized irradiation, sensitizer MCZ was mostly recovered, indicating a recycling of the sensitizer probably via electron transfer from solvent radical (X$^\bullet$) to the sensitizer radical cation (D$^{\bullet+}$) as depicted in Scheme I.

The Least Coordinating Anion

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Attention has been drawn recently to the myth of the noncoordinating anion.[1-4] The common spectator anions of polar solution (ClO_4^-, PF_6^-, BF_4^-, $CF_3SO_3^-$, etc.) frequently become coordinated ligands in low dielectric media, particularly when attempting to synthesize coordinatively unsaturated cations. While there is probably no such thing as a truly noncoordinating anion, the search for the *least* coordinating anion has as its reward the opportunity to synthesize the most reactive, coordinatively unsaturated cations. It has also been pointed out recently that the structural parameters of complexes with so-called weakly binding anions may in fact reflect quite strong binding.[1,2,5] Short metal–anion bond distances are frequently observed. A relatively unperturbed structure of the coordinated vs. uncoordinated anion is taken to reflect ionicity in the bonding.[1] In such complexes it is often the kinetic criterion of substitutional lability that earns the anion its designation as weakly coordinating.[6,7] These observations prompt us to report the use of the carborane anion $B_{11}CH_{12}^-$ as a novel candidate for the least coordinating anion. The structure of the complex formed by pairing $B_{11}CH_{12}^-$ with the formal cation $[Fe^{III}(TPP)]^+$ (TPP = tetraphenylporphyrinate) provides ready structural criteria for illustrating weak coordinative binding.

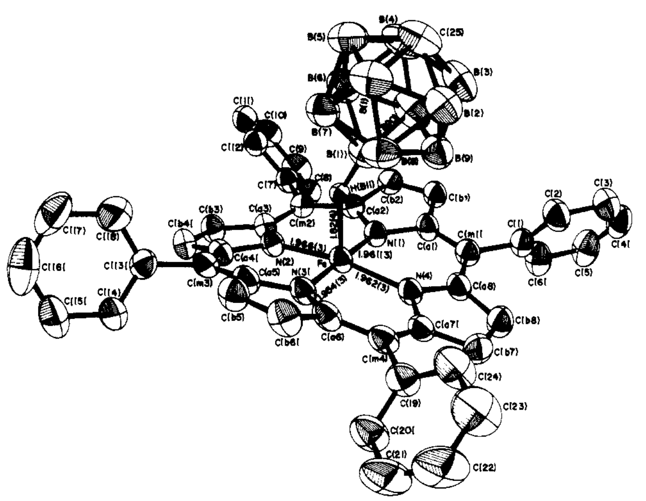

Figure 1. ORTEP drawing of the Fe(TPP)($B_{11}CH_{12}$) molecule. Individual bond distances in the coordination group are displayed. Vibrational ellipsoids are contoured to enclose 50% of the electron density. For clarity, the only hydrogen atom shown is that involved in the Fe–H–B bonding.

In our search for the most weakly coordinating anion we have chosen $B_{11}CH_{12}^-$ for its large size, nearly spherical shape, and surprising chemical stability.[8] An unusual, η^1 mode of benzene coordination observed in the X-ray crystal structure of its silver salt[9] gave the first indication that $B_{11}CH_{12}^-$ is an unusually poor electron donor.[10] We have chosen $[Fe(TPP)]^+$ as the test case cation for several reasons. The square-planar cation with two vacant coordination sites is sterically wide open for coordination and thus offers a severe test of the coordinative passivity of an anion. In fact, this cation has never been isolated. The perchlorate "salt" has a notably short Fe–O bond[4] and, contrary to an earlier suggestion,[11] hexafluoroantimonate is also a coordinating anion.[3] The structural parameters in Fe(TPP)X systems are particularly sensitive to the nature of X^- and offer a number of criteria for measuring the strength of the cation/anion interaction. Large out-of-plane displacements of the iron atom toward the axial ligand X are observed for strongly binding halides (~0.5 Å) but these displacements progressively decrease $I^- > ClO_4^- > SbF_6^-$ as the interaction weakens.[12] Similarly, as the axial anion interaction decreases the equatorial charge attraction of the iron atom for the porphyrinato nitrogen atoms should increase. Thus, the complex with the least coordinating anion is expected to have the shortest Fe-N bond lengths.

Fe(TPP)($B_{11}CH_{12}$) is conveniently prepared by stoichiometric reaction of Fe(TPP)Br with $AgB_{11}CH_{12}$[9] in dry toluene.[13] Coordination of the carborane anion is indicated[9] by splitting of the strong B-H absorption in the IR spectrum (2550 (s), 2380 (m) cm^{-1}, KBr disk). Consistent with weak coordination of the anion,

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(10) η^1-benzene coordination in $Ag(B_{11}CH_{12})\cdot(C_6H_6)_2$ reflects arene-to-silver σ donation rather than silver-to-arene π back-donation which in turn reflects poor σ donation from the carborane anion.[9]

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(13) λ_{max}(toluene) 406, 500, 675 nm.

0002-7863/86/1508-3117$01.50/0