1 is presumably a consequence of coordination of four ring sulfur atoms, i.e., S1, S1′, S2, and S2′, to thorium (vide infra). The Th–S1 and Th–S2 distances are 2.768 (4) and 3.036 (3) Å, respectively. These values bracket the average Th–S bond lengths in $Th(S_2PMe_2)_4$[13] (2.90 Å), $Th(S_2CNEt_2)_4$[14] (2.87 Å), Th_2S_3[15] (2.90 Å), and ThS_2[16] (2.95 Å). The Th–S2 bond may be thought of as a dative bond while the Th–S1 bond is best described as ionic. The difference in Th–S bond lengths of 0.268 Å is comparable to the difference of 0.284 Å observed for the phosphine and phosphide systems, $Th(CH_2Ph)_4(dmpe)$[17] (where dmpe = $Me_2PCH_2CH_2PMe_2$) and $Cp^*{}_2Th(PPh_2)_2$,[18] thereby supporting coordination of S2 to thorium. Donor bonding from S2 and S2′ to the Lewis acidic Th(IV) center manifests itself in two other ring metrical parameters. The S1–Th–S1′ angle of 113.3 (1)° is 14–20° larger than X–Th–X angles previously reported in $Cp^*{}_2ThX_2$ complexes,[18,19] and the Th–S1–S2 angle at 76.9 (2)° is the smallest M–S–S angle (by 30°) observed in an MS_5 ring.[2,9] The S–S distances, on the other hand, are comparable to those found in Cp_2TiS_5.[9] The methyl-substituted $(C_5Me_{5-x}H_x)_2M$ complexes are invariably found as staggered rotamers,[2] which would also tend to stabilize the twist-boat conformation for the ThS_5 ring due to nesting of the methyl groups. Other metrical parameters associated with **1**, e.g., the Th–Cp* distance and Cp*–Th–Cp* angle, are unexceptional.

Thorium–sulfur bonding in **1** has been investigated by using extended Hückel molecular orbital calculations[20] performed on the model compound $(C_5H_5)_2ThS_5$. These calculations show a variety of bonding interactions between the Cp_2Th and S_5 fragments. Bonding between the two highest occupied molecular orbitals of $S_5{}^{2-}$ and Cp_2Th^{2+} virtual orbitals composed of 6d, 7s, and 7p produces the largest overlap populations between fragments. The HOMO of $S_5{}^{2-}$ is antisymmetric with respect to the C_2 axis while the level just beneath it in energy is symmetric. All five sulfur atoms contribute to these orbitals, but the β sulfurs have more electron density (about 0.95 electrons) than the α sulfurs (0.70) or the γ sulfur (0.35). Symmetry-adapted combinations of the p orbitals on the β sulfurs in these two orbitals point toward the plane that contains Th and the Cp centroids. The C_{2v} Cp_2Th^{2+} fragment has an empty b_1 orbital, a hybrid of xz and x metal character, that stabilizes the $S_5{}^{2-}$ HOMO. Another metal fragment empty orbital has a_1 symmetry, being a mixture of z^2 and x^2-y^2; this orbital stabilizes the second HOMO of $S_5{}^{2-}$. Both of these interactions give rise to more Th bonding to the β sulfur than to the α sulfur, but the aggregate of many other interfragment mixings (too small and numerous to discuss here) produces an overall Th–α-sulfur overlap population that exceeds the overall Th–β-sulfur overlap population.

In summary, the unusual twist-boat ThS_5 ring conformation in **1** is likely a consequence of donor–acceptor bonding between the β-sulfur atoms of the $S_5{}^{2-}$ chelate and the electron-deficient thorium(IV) center. An extension of polysulfide chemistry to other actinide ions, as well as an examination of the effect of MS_5 ring conformation on ring reactivity, are projects currently underway in this laboratory.

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Supplementary Material Available: Crystal data and tables of final atomic positional and thermal parameters, and observed and calculated structure factors for complex **1** (8 pages). Ordering information is given on any current masthead page.

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Photoinduced C–C Bond Cleavage via Electron-Transfer Reactions: Visible-Light-Mediated Scission of Tertiary Amines

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Electron-transfer quenching of potential excited acceptors including such diverse molecules as ketones, metal complexes, hydrocarbons, and dyes is a well-documented reaction.[2-10] Frequently used donors in these reactions include various tertiary amines such as triethylamine.[10-15] Depending upon the amine, the most often observed fates for the photochemically produced radical cation in solution are back electron transfer or deprotonation; deprotonation at the α-carbon of tertiary amines produces a neutral but rather strongly reducing free radical which, after electron loss, gives an iminium ion which can often be hydrolyzed to give a secondary amine and a carbonyl compound.[10,12,14-16] The deprotonation of tertiary amine radical cations involves reaction of carbon acids, which might be anticipated to be relatively slow; however, laser flash spectroscopic investigations indicate this process can occur extremely fast, on the scale of picoseconds.[14,17-19] An alternative fate of reactive amine radical cations could be cleavage of a carbon–carbon σ bond α to the nitrogen; this reaction

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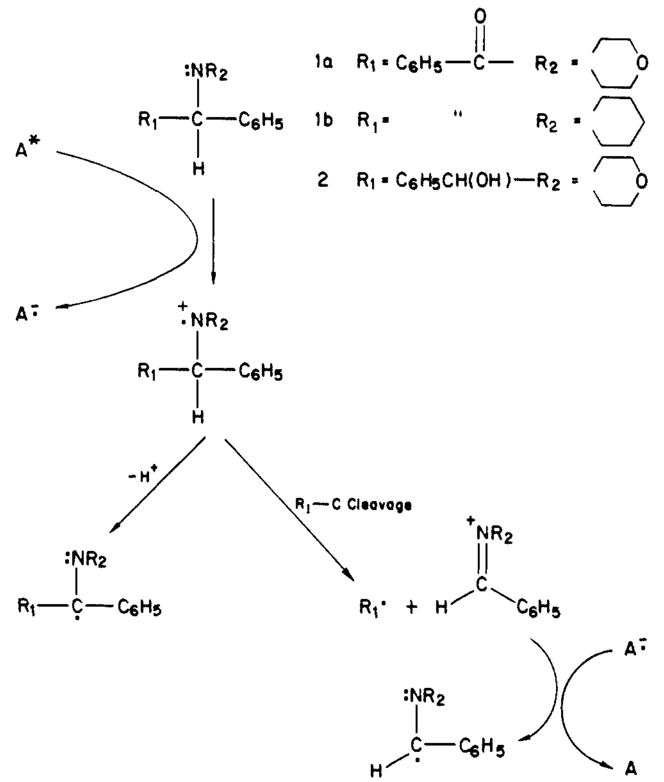
Scheme I



(Scheme I) leads to a neutral free radical and a different iminium ion from that indicated above. Related C–C bond cleavage reactions have been observed for cations generated from ethers in solution,[20] for strained or otherwise reactive hydrocarbon radical cations,[21-24] or for certain amines in the gas phase (mass spectrometer).[25] This paper reports the electron-transfer photochemistry of some substituted tertiary amines that contain potentially labile C–C bonds adjacent to the amine; we present results which indicate that photoinduced electron transfer can lead to reactions in which the net process involves use of visible light to mediate selective cleavage of C–C bonds to produce radicals.

The compounds used as amine donors in this study include the nitrogen analogues of the well-studied benzoin ethers[26-28] MDB (**1a**) and PDB (**1b**) and an alcohol analogue MDBOH (**2**). These compounds are readily prepared by substitution of desyl chloride with the appropriate secondary amine (**1a** and **1b**)[29] and subsequent reduction of the ketone with aluminum isopropoxide (**2**). The electron acceptor used in these investigations is $\mathrm{Ru[4,4'\text{-}CO_2Et(bpy)]_3(PF_6)_2(RuL_3^{2+})}$ which can be activated by light (400–500 nm) that does not excite compounds **1** or **2**. Photolyses were carried out with either a 200-W mercury lamp or a tungsten lamp. For photochemical ESR experiments the samples were irradiated directly in the cavity of a Bruker 420 ESR spectrometer; solutions in benzene were irradiated in a cylindrical tube while those in acetonitrile were irradiated in a quartz flat cell. The irradiation source was a 1000-W xenon lamp; in all of the solutions ca. 1 mM nitrosodurene (ND) or 5,5-dimethyl-1-pyrroline 1-oxide (DMPO) was employed as a spin trap. Photoproducts of acetonitrile solutions 0.1 M in amine and 10^{-4} M in $\mathrm{RuL_3^{2+}}$ were determined by HPLC and VPC analysis; the samples were irradiated in a merry-go-round reactor using a Hg lamp with Corning 5-56 and 3-73 filters (470 nm). Photopolymerization experiments were carried out by using a resin medium containing the light-absorbing compounds amine and 50% methyl methacrylate.

Irradiation of **1a** or **1b** with ultraviolet light ($\lambda \leq 330$ nm) in the presence of polymerizable monomers such as methyl methacrylate results in rapid polymerization; presumably the polymerization is initiated by cleavage of the substrates by a type I reaction to afford radicals (eq 1).[26-28,30,31] When either **1a** or

$$\mathrm{C_6H_5COCH(NR_2)Ph \xrightarrow{h\nu} C_6H_5CO\cdot + C_6H_5(NR_2)CH\cdot} \qquad (1)$$

1b is irradiated in acetonitrile alone formation of benzaldehyde ($\phi = 0.02$; chemical yield 20–25%) can be followed by UV spectroscopy, HPLC, or VPC.[30] In accord with the cleavage anticipated in eq 1, irradiation of **1a** or **1b** in an ESR spectrometer in the presence of the spin trap leads to ready detection of the trapped benzoyl radical.[32]

When solutions of camphorquinone or $\mathrm{RuL_3^{2+}}$ and **1a** or **1b** are irradiated with light absorbed only by the acceptor ($\lambda > 440$ nm) efficient polymerization of monomers such as methyl methacrylate or other resins is also observed. Irradiation of acetonitrile solutions of $\mathrm{RuL_3^{2+}}$/**1a** or $\mathrm{RuL_3^{2+}}$/**1b** with DMPO in the cavity of an ESR spectrometer leads to the trapped benzoyl radical as observed for direct irradiation of the amines at shorter wavelengths.[32] Irradiation of $\mathrm{RuL_3^{2+}}$/**1a** in acetonitrile with light absorbed only by $\mathrm{RuL_3^{2+}}$ also leads to the production of benzaldehyde in comparable quantum efficiency to the direct irradiation ($\phi = 0.033$).[33]

Irradiation of $\mathrm{RuL_3^{2+}}$ solutions with light absorbed only by the acceptor also produces efficient polymerization of various olefin monomers; in this case irradiation of **2** in the UV in the absence of acceptor results in little or no polymerization since this compound absorbs very little at $\lambda > 240$ nm (acetonitrile). Irradiation of $\mathrm{RuL_3^{2+}}$/**2** with ND present leads to a strong six-line ESR spectrum which can be attributed to the adduct of ND with the radical $\mathrm{C_6H_5CH(OH)\cdot}$. For $\mathrm{RuL_3^{2+}}$/**2** irradiation in acetonitrile without radical scavengers present leads to the production of benzaldehyde as a major product as detected by HPLC, VPC, and NMR; the quantum efficiency for formation of benzaldehyde is 0.4 and the chemical yield is comparably high.[34] The high quantum efficiency of forming benzaldehyde from $\mathrm{RuL_3^{2+}}$/**2** clearly indicates the prominence of C–C cleavage in this case; since benzoyl radicals are not detected from **2**, the most likely path of reaction is net hydrogen atom loss from the detected $\mathrm{C_6H_5CHOH\cdot}$ radical (eq 2).

$$\mathrm{C_6H_5CHOH\cdot + A \rightarrow C_6H_5CHO + AH} \qquad (2)$$

For both **1a** and **2** the observed photoreaction with $\mathrm{RuL_3^{2+}}$ originates from quenching of the luminescent triplet state. The quenching constants, $k_q = 1.3 \times 10^6$ $\mathrm{M^{-1}\,s^{-1}}$ for **1a** and $k_q = 1.4$

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(34) No products from the amine "half" of **1a** or **2** have been observed by HPLC or VPC; possible products, $\mathrm{C_6H_5CH_2NR_2}$ or $\mathrm{C_6H_5CH{=}NR_2^+}$, have been synthesized but their stability to the analysis conditions is low as is their detectability. A mixture of the diasteromers of **2** was used; both isomers react as indicated by HPLC but no quantitative analysis of differential reactivity has been attempted.

$\times 10^6$ M^{-1} for **2**, are low compared to that for RuL_3^{2+} by triethylamine ($k_q = 1.4 \times 10^8$ M^{-1} s^{-1}).[35] Although it is difficult to assess precisely the oxidation potential for alkylamines due to their electrochemical irreversibilities, an examination of comparative electron-transfer quenching constants for different metal complexes[35] suggests that the oxidation potentials of **1a** and **2** should be 1.40–1.45 V, very close to that of dibenzylamine.[35]

The results described above show that C–C bond cleavage, as outlined in Scheme I, is indeed an important path for compounds of the general type of **1** and **2**. The occurrence of C–C bond cleavage for radical ions derived from molecules such as **1** and **2** appears to be a reasonable process; the photoinduced σ-bond cleavages in solution observed in the present study are novel and potentially quite useful since relatively long wavelength light can be used to mediate selective scission of a relatively strong covalent bond. Interestingly for compounds such as **1** the electron-transfer photoreaction provides a strong wavelength extension of a reaction (eq 1) ordinarily observed only with UV; for compounds such as **2** the cleavage does not occur in an "unsensitized" process since the compounds have no carbonyl capable of mediating type I cleavage. The photoinduced electron-transfer-mediated σ-bond cleavage should be applicable for a variety of donors with appropriate substituents on the σ-bond terminal carbons. We are presently examining a number of potential substrates to determine both the scope of the reaction as well as their rates and factors controlling them.

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The Structure of Methyl Isosartortuoate, a Novel Tetracyclic Tetraterpenoid from the Soft Coral *Sarcophyton tortuosum*

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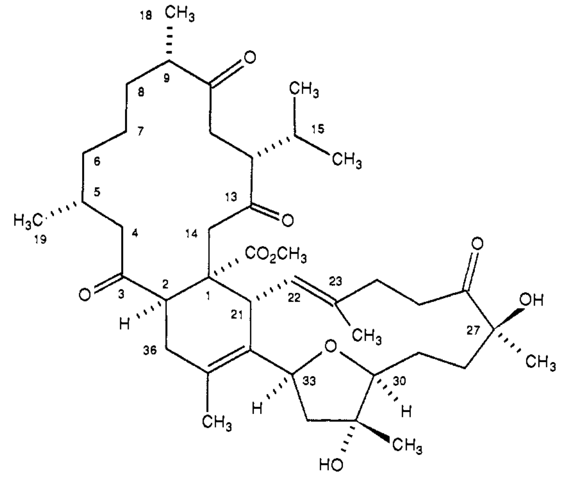
Soft corals have been an unusually productive source of chemically interesting and biologically significant secondary metabolites.[1-3] The vast majority of identified metabolites from soft corals are terpenoid compounds; among the terpenoids, the cembrene family of diterpenoids predominates.[1] In this paper we wish to describe the isolation and structure determination of an unprecedented tetracyclic tetraterpenoid, methyl isosartortuoate (**1**).

Methyl isosartortuoate was isolated from the soft coral *Sarcophyton tortuosum* Tixier-Durivault collected in the South China Sea. The fresh coral was immersed in 95% ethanol, and the alcoholic extract was concentrated and chromatographed on silica gel with acetone/petroleum ether. Compound **1** was obtained as colorless crystals, mp 194–195 °C. A molecular formula of $C_{41}H_{62}O_9$ for **1** was determined by HRMS (M^+, m/z 698.4305, calcd for $C_{41}H_{62}O_9$, 698.4389). The IR spectrum (KBr) indicated the presence of OH groups (3350 and 1105 cm^{-1}), a CO_2R group (1745 and 1215 cm^{-1}), carbonyl groups (1725 and 1712 cm^{-1}), double bonds (1640, 1658, 858, and 700 cm^{-1}), an ether (1080 cm^{-1}), and an isopropyl group (1380 and 1375 cm^{-1}).



1

The following fragments were suggested by the ^1H and ^{13}C NMR: a methyl ester (δ_C 174.4 (s), 51.2 (q); δ_H 3.55 (3 H, s)), four carbonyl groups (δ_C 215.1 (s), 213.6 (s), 213.2 (s), and 208.7 (s)), and a tri- and tetra-substituted double bond (δ_C 136.4 (s), 135.1 (s), 127.9 (d), 126.1 (s); δ_H 4.90 (1 H, d)). Since these functionalities accounted for seven degrees of unsaturation, methyl isosartortuoate had to be tetracyclic. Four carbons, not counting the methyl ester, were attached to oxygen, most plausibly as one ether (δ_C 87.1 (d), 75.16 (d); δ_H 5.06 (1 H, dd, J = 12.0, 5.0 Hz), 3.47 (1 H, dd, J = 12.5, 1.5 Hz)) and two tertiary hydroxyls. Nine methyl groups were identified, one of which was the methyl ester. The others were two olefinic (δ_C 22.21 (q), 21.23 (q); δ_H 1.85 (3 H, s), 1.64 (3 H, s)), one isopropyl group (δ_C 17.72 (q), 17.46 (q); δ_H 1.00 (3 H, d), 0.71 (3 H, d, J = 6.8 Hz), two methyls attached to quaternary oxygen bearing carbons (δ_C 25.9 (q), 23.9 (q); δ_H 1.35 (3 H, s), 1.27 (3 H, s)), and two secondary methyls (δ_C 19.7 (q), 19.0 (q); δ_H 1.13 (3 H, d, J = 7.0 Hz), 0.86 (3 H, d, J = 7.0 Hz)). The number of methyl groups combined with the parent molecular formula suggested that **1** was terpenoid in origin. The ^{13}C NMR spectrum also showed six methine groups and 12 methylene groups. Since no examples of tetracyclic tetraterpenoids were known, and the ^1H NMR and ^{13}C NMR spectra did not unambiguously suggest a structure for **1**, the structure was defined by X-ray diffraction.

Crystals of **1** formed in the tetragonal space group $P4_12_12$ with $a = b = 9.547$ (2) Å and $c = 92.722$ (7) Å. Block diagonal least-squares refinements have converged to a conventional crystallographic residual of 0.096 for the observed reflections. Additional crystallographic parameters are available and are described in the supplementary material. A computer-generated perspective drawing of the final X-ray model of **1** is given in Figure 1. Hydrogens are not shown and the absolute stereochemistry portrayed is arbitrary. There are regions of the structure where the thermal motions were large, and a poorly resolved structure resulted. This was most marked in the C5–C8 region and the methyl groups where the average isotropic thermal parameter 11.3 Å^2 vers. 7.5 Å^2 for the rest of **1**. Bond shortening, the usual consequence of large thermal motions, was quite noticeable in the C5–C8 region. However, when riding corrections[4] were introduced, the geometry was consistent with structure shown as **1**. The NMR data for methyl isosartortuoate (**1**) also supported this formulation and clearly indicated that there was no 1,2-disub-

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