Compound **2** is an air-stable green solid; crystals suitable for a single-crystal X-ray structure analysis were grown by vapor diffusion of diethyl ether into a dichloromethane solution.

The molecular arrangement detrmined for **2** is shown in Figure 1 and that of the $\{Ru_3H_2(CH_2Cl_2)\}$ fragment is depicted in Figure 2. The analysis reveals a triangle of ruthenium atoms with two of the metals in coordination with two hexamethylbenzene ligands; the third ruthenium atom forms part of a $\{2,3\text{-}(OEt)_2\text{-}isocloso\text{-}1\text{-}RuB_{10}H_8\}$ subcluster. The CH_2Cl_2 occupies a position on one side of the $\{Ru_3\}$ triangle such that each Cl forms an asymmetric bridge between a pair of ruthenium atoms, notionally replacing the H(1,2) and H(1,3) hydride bridges of the parent molecule.[4] The ruthenium atom involved in the metallaundecaborane moiety is associated with the longer Ru–Cl distances of 2.403 (3) and 2.396 (2) Å for Ru(1)–Cl(1) and Ru(1)–Cl(2), respectively. These may be compared with the more normal[5] distances for Ru(2)–Cl(2) and Ru(3)–Cl(1) of 2.315 (3) and 2.319 (3) Å, respectively. Two bridging hydride atoms have been located: H(12A) is approximately equidistant from Ru(2) and Ru(3), and H(12B) is approximately equidistant from all three metal atoms but at a significantly longer distance. The dimensions of the *isocloso*-1-metallaundecaborane are similar to those in **1**[4] and in related compounds of ruthenium[6-8] and osmium.[9] The Ru(1) atom center may be regarded as being formally ruthenium(II) if the hypercloso view[10-12] is adopted, or ruthenium(IV) for the isocloso view.[6,8,9,13,14] Whichever view is taken, the two hexamethylbenzene ligands contribute six electrons each, the formal borane ligand four, the two bridging hydrogen atoms one each, and the three ruthenium atoms eight each. The chlorine–ruthenium bond lengths suggest that both chlorine atoms are donating two lone pairs to the cluster, thus giving a total cluster electron count of 50, two greater than the 48 usually[15] associated with triangular clusters. The greater thermal stability of **2**, which is stable in refluxing acetonitrile, in comparison with $Ag_2(CH_2Cl_2)_4Pd(OTeF_5)_4$,[1] which is stable only below −20 °C, may reflect the stronger interaction of the dichloromethane in **2**, where both lone pairs of electrons on each chlorine atom are involved in cluster bonding.

Refluxing **1** in dichloromethane alone, even over an extended period, was shown to effect no change, indicating a role for phenylacetylene in the formation of **2**. A second product, isolated from the reaction in 69% yield (based on Ru), has been characterized as the μ-η^2-alkenyl compound[16] $[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_3(\mu\text{-}\eta^2\text{-}HC{=}CHPh)\}RuB_{10}H_8(OEt)_2]$. This latter compound may suggest that **2** is formed via a μ-η^2-alkenyl complex, with dichloromethane displacing the μ-η^2-alkene ligand as styrene.

Acknowledgment. We thank the Massey University Research Fund for a postdoctoral fellowship (M.B) and the NZ University Grants Committee for funding toward equipment. We are grateful to Drs. J. D. Kennedy and X. L. R. Fontaine of the University of Leeds for NMR spectra.

Supplementary Material Available: Tables of atomic coordinates and thermal parameters for all atoms and bond distances and angles (10 pages); listing of observed and calculated structure factors for $[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_2(CH_2Cl_2)\}RuB_{10}H_8(OEt)_2]$ (11 pages). Ordering information is given on any current masthead page.

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Ultraviolet Resonance Raman Spectra of Bacteriorhodopsin in the Light-Adapted and Dark-Adapted States

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Received June 30, 1989

Ultraviolet resonance Raman (UVRR) spectra of bacteriorhodopsin (bR) in the light-adapted (LA) and dark-adapted (DA) states are reported for the first time. The spectra have provided key information on the structures and environments of aromatic amino acid side chains, in particular Trp and Tyr. Conclusions derived are that (1) some Trp side chains in bR_{568} are located in hydrophobic environments, and the hydrophobicity of the Trp side chains or the number of such Trp side chains increases in bR_{548}; (2) the C_β–C_3 torsion angles of most Trp side chains are about +102° or −102° in both bR_{568} and bR_{548}; (3) the indole N_1H sites of some Trp in bR_{568} and bR_{548} are strongly H-bonded; and (4) at least one Tyr is present as the anionic form (Tyr^-) in bR_{568}, and the number of Tyr^- decreases in bR_{548}.

LA-bR consists solely of bR_{568} with *all-trans*-retinal as the visible chromophore while DA-bR is a mixture of bR_{568} and bR_{548}, the latter containing 13-*cis*,15-*cis*-retinal.[1] Figure 1 shows the UVRR spectra of LA-bR and DA-bR excited at 240 nm with an H_2-Raman-shifted pulsed Nd:YAG laser. The spectra are dominated by the bands arising from 8 Trp and 11 Tyr side chains among which the band at 1617 cm^{-1} is an overlap of those of tyrosyl ν_{8a} and tryptophyl W1.[4] Parts a and b of Figure 2 are 240-nm excited spectra (1675–1500-cm^{-1} region) of bR-Tyr-*d* containing ring-deuteriated Tyr (Tyr-*d*, deuteriation being 97% at ϵ and 60% at δ), where W1 (1620 cm^{-1}) is separated from the downshifted ν_{8a} of Tyr-*d* at 1598 cm^{-1}. Figure 2c is a 240-nm spectrum of aqueous Trp. Parts d–g of Figure 2 are 253-nm excited spectra of LA-bR, DA-bR, and aqueous Trp and Tyr^-.

The UVRR spectra of individual aqueous aromatic amino acids have now been established.[8-10] A pair of Trp bands around 1360

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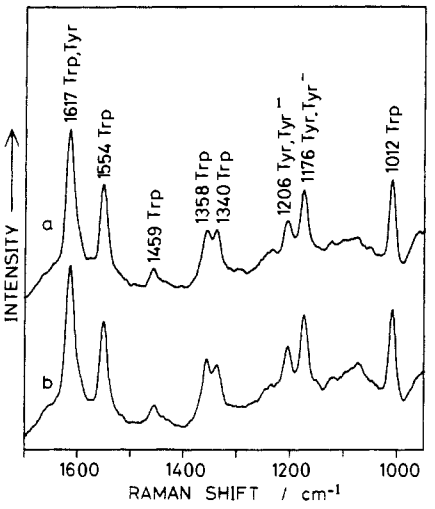

Figure 1. UVRR spectra of LA-bR (a) and DA-bR (b) (0.23 mM suspension in 10 mM HEPES buffer, pH 7.9) in a spinning cell, obtained with 240-nm excitation (1 mW) from an H_2-Raman-shifted pulsed (30 Hz) Nd:YAG laser using a multichannel spectrometer.[2] The beam spot size was 100–150 μm, and the typical power density was 0.4–0.2 J cm^{-2}/pulse. About one-tenth of the chromophores around 240–253 nm (8 Trp and 11 Tyr per molecule) are being hit at these conditions. Practically the same spectra were obtained with a lower laser power. Bacteriorhodopsin was obtained by the standard method.[3] LA-bR was prepared by light adaptation of bR with a slide projector lamp for 20 min. Spectrum a was obtained by repeated accumulation of Raman scattering for 7 min after lamp irradiation for 10 min. DA-bR was prepared by dark adaptation of bR for 24 h. Spectrum b was obtained by repeated accumulation of Raman scattering for 7 min after dark adaptation for 30 min.

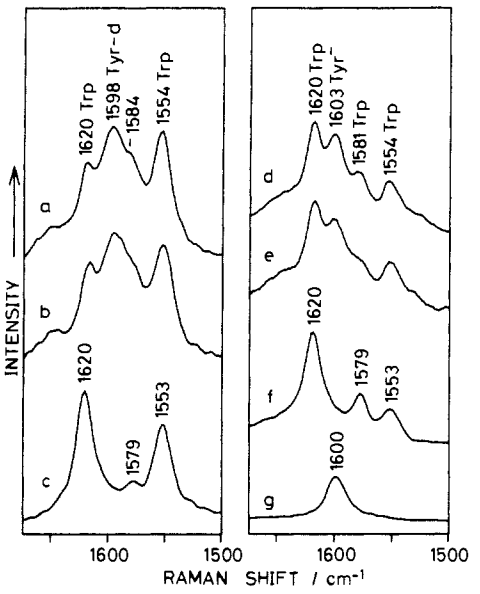

Figure 2. UVRR spectra in the 1675–1500-cm^{-1} region with 240-nm and 253-nm excitation. Parts a–c are those of aqueous LA-bR-Tyr-*d* (a), DA-bR-Tyr-*d* (b), and Trp (c) with 240-nm excitation (1 mW). bR-Tyr-$d^{5,6}$ was obtained by incubation of *Halobacteria halobium* S9 in a culture medium containing Tyr-d^7 and purified by the standard method. The bands of Tyr-*d* are twice as broad than the other bands because of imperfect deuteriation. A shoulder at 1584 cm^{-1} in part a may be assigned to ν_{8a} of Tyr-d^-. Parts d–g are the spectra of aqueous LA-bR (d), DA-bR (e), Trp (f), and Tyr^- (g) with 253-nm excitation (1 mW). Intensities of the spectra of Trp and Tyr^- are scaled to a possible proportion of 8:1.

and 1340 cm^{-1} are known to arise from Fermi resonance between a fundamental and one or two combination bands of the indole ring, and the intensity ratio $I(1360)/I(1340)$ in the visible Raman spectra is sensitive to the ring environment: it is especially large for a Trp whose indole ring is closely surrounded by aliphatic groups.[11,12] In order to see if such correlation applies to the ratio in resonance Raman spectra,[8] we studied solvent effects for indole, which was one of the model compounds examined previously.[11] Although the intensity ratio was different (e.g., 0.65 for cyclohexane solution with 240-nm excitation vs 1.32 with 488-nm excitation), the tendency of the ratio to be higher in hydrophobic environments than in hydrophilic ones paralleled that observed in the visible Raman spectra. In fact, the ratio in Figure 1a (1.0) is much larger than that for aqueous amino acid Trp (0.67), which indicates that the environments of some Trp side chains are hydrophobic. In Figure 1b, the intensity ratio further increases (1.2), suggesting that the environments of such Trp become more hydrophobic or the number of Trp in the hydrophobic environments increases in bR_{548}.

Recently, a relation between the Trp W3 frequency near 1550 cm^{-1} and the absolute value of the torsion angle $\chi^{2,1}$ around the C_β–C_3 bond has been established. The frequency increases monotonically from 1542 cm^{-1} for a small $|\chi^{2,1}|$ angle of 61° to 1557 cm^{-1} for 117°.[13] The W3 peak position (1554 cm^{-1}) and the band width (16 cm^{-1}, fwhm) in Figure 1a are not affected by dark adaptation (Figure 1b), indicating that the C_β–C_3 torsion angles are within a few degrees around ±102° in bR_{568} and remain practically unchanged in bR_{548} even though the environments of some indole rings are altered.

The peak intensity ratio of W3 and W1, $I(W3)/I(W1)$, with 240-nm excitation can be a good test for a red shift of the strong $B_{a,b}$ absorption (normally at 218 nm) of Trp, because it is significantly different between 218-nm (2.5)[8] and 240-nm (0.5,[8] 0.6 in this work (Figure 2c)) excitation for aqueous Trp, and a red shift of the absorption must be reflected sensitively in its increase in the 240-nm spectrum. According to our preliminary study on skatole (a model compound for Trp), the corresponding ratio was observed to be 0.6 in cyclohexane, 0.9 in diethyl ether, and 1.2 in methanol, concomitant with a small red shift (2 nm) of the $B_{a,b}$ absorption for the latter two compared with the former, presumably due to H-bonding from skatole NH to the solvents. The ratio is about 1.6 for LA-bR and 1.3 for DA-bR (Figure 2a,b), both of which are very much larger than that for aqueous amino acid Trp. These observations suggest that some Trp side chains are strongly H-bonded. Furthermore, the small but significant difference in the ratio between LA-bR and DA-bR may reflect the changes in the Trp environments.

With 253-nm excitation, the scattering from Tyr is negligibly weak as compared with that from Trp, while that from Tyr^- is significant (Figure 2f,g). Accordingly, only scattering from Trp (W1, W2, and W3)[4] is expected to be observed in the 1675–1500-cm^{-1} region if Tyr^- is absent. An intense peak at 1603 cm^{-1} in Figure 2d, which becomes weaker in Figure 2e, is certainly assigned to ν_{8a} of Tyr^-. Estimation of the number of Tyr^- from the intensities, however, is not straightforward because any Tyr^- in bR at neutral pH must be located in the interior of the protein and could give a 1603-cm^{-1} intensity much different from that of aqueous Tyr^-. However, it can be safely said that at least one Tyr^- is present in bR_{568} and the number of Tyr^- decreases in bR_{548}. The present observation is in agreement with earlier results by FTIR and UV absorption studies[14] but in conflict with a recent solid-state NMR report.[15]

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A merit of UVRR spectroscopy is that it is possible to investigate the opsin molecule, but not the retinal chromophore, in detail under physiological conditions. The present study has shown some characteristics of LA-bR and DA-bR semiquantitatively. More studies are in progress in our laboratory, including time-resolved spectroscopy on the opsin molecule in the photocycle.

Acknowledgment. We are indebted to Professor Fumio Tokunaga for the gift of *Halobacteria halobium* strain S9. Financial support from the Ministry of Education, Science and Culture of Japan (a Grant-in-Aid for General Scientific Research (62430004)) is gratefully acknowledged.

Registry No. H-Trp-OH, 73-22-3; H-Tyr-OH, 60-18-4.

Crystal Structure of a Side-On Superoxo Complex of Cobalt and Hydrogen Abstraction by a Reactive Terminal Oxo Ligand

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Received December 6, 1989

The activation of dioxygen by coordination to a metal center is a venerable and yet elusive goal of research in oxidation catalysis.[1] Most available evidence suggests that bound dioxygen itself is not very reactive, while metal oxo species derived therefrom often are.[2] Thus an obvious and appealing approach to O_2 activation is shown in eq 1. Much precedent exists for all of the

$$2M + O_2 \rightarrow MO_2 + M \rightarrow MOOM \rightarrow 2M{=}O \quad (1)$$

intermediate species depicted, but there are few examples of the whole sequence of conversions.[3] Herein we report on a cobalt complex that binds dioxygen in an unprecedented manner and eventually yields products implying a reactive cobalt oxo intermediate.

Magnesium reduction of the readily available cobalt halides Tp'Co^{II}X (Tp' = hydridotris(3-*tert*-butyl-5-methylpyrazolyl)borate, X = Cl, I; THF solvent)[4] in a nitrogen atmosphere yielded the dinitrogen complex Tp'Co(N_2) in 55% isolated yield (see Scheme I). The IR spectrum of this compound exhibited ν_{NN} at 2046 cm^{-1} (KBr), and dissolution in degassed CH_2Cl_2 resulted in the release of 1.0 equiv of N_2 (measured with a Toepler pump) and quantitative reisolation of Tp'CoCl. The ^{1}H NMR spectrum

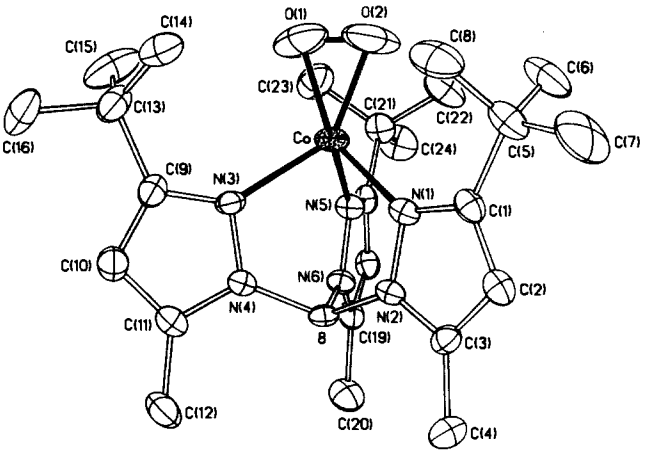
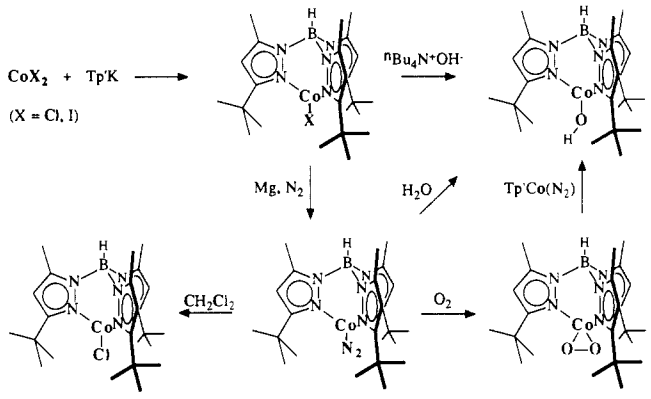


Figure 1. The molecular structure of Tp'Co(O_2). Selected bond distances: Co–O(1), 1.816 (5) Å; Co–O(2), 1.799 (6) Å; O(1)–O(2), 1.262 (8) Å; Co–N(1), 2.008 (4) Å; Co–N(3), 2.042 (4) Å; Co–N(5), 2.069 (4) Å. Interatomic angles: O(1)–Co–O(2), 40.9 (3)°; N(1)–Co–N(3), 91.6 (2)°; N(1)–Co–N(5), 92.1 (2)°; N(3)–Co–N(5), 92.9 (1)°.

Scheme I



of Tp'Co(N_2) exhibited isotropically shifted resonances expected of the tris(pyrazolyl)borate ligand ($\delta(C_6D_6)$: −7.3 (27 H), 16.7 (9 H), 27.5 (1 H), 39.8 (3 H) ppm), and the magnetic susceptibility showed Curie behavior of a simple paramagnet with an effective magnetic moment of 3.87 μ_B at room temperature.[5] This is consistent with the two unpaired electrons of a tetrahedral Co(I) complex augmented by a significant orbital contribution.

Exposure of a pentane suspension of Tp'Co(N_2) to an excess of dioxygen immediately yielded the dioxygen complex Tp'Co(O_2), which was purified by filtration through Florisil and recrystallization from hot toluene (44% isolated yield).[6] The IR spectrum of this compound exhibited a new band (assigned as ν_{OO}) at 961 cm^{-1}. Whereas the O–O stretching vibration is generally used to classify dioxygen complexes into superoxo-containing (1200–1070 cm^{-1}) or peroxo-containing (930–740 cm^{-1}) species,[7] this criterion obviously fails in the case at hand. Therefore the molecular structure of Tp'Co(O_2) was determined by X-ray diffraction (see Figure 1).[8]

The crystal consists of isolated molecules featuring cobalt in the embrace of the sterically hindered tridentate nitrogen ligand. The fourth and last coordination site left by this "tetrahedral

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(5) The susceptibility data was fitted with a Curie–Weiss expression ($\chi_m = [C/(T-\theta)] + TIP$). C = 1.59 emu K/mol, θ = 2.1 K, TIP = 6.91 × 10^{-4} emu.

(6) Tp'Co(O_2): ^{1}H NMR (C_6D_6) δ −2.1 (s, 27 H), 9.5 (br s, 1 H), 18.2 (s, 9 H), 33.6 (s, 3 H); IR (KBr) 2543, 2523 (ν_{B-H}), 961 (ν_{O-O}) cm^{-1}; UV–vis (THF) 321 (ϵ = 1286), 365 (1033) nm; mp 206–208 °C dec. Anal. Calcd for $C_{24}H_{40}BCoN_6O_2$: C, 56.02; H, 7.85; N, 16.34. Found: C, 56.52; H, 7.74; N, 16.40.

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