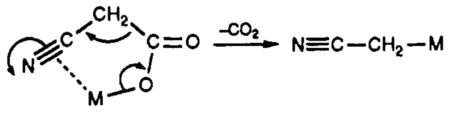
Confirmation of the structure of the product (**1**) was accomplished by single-crystal X-ray analysis.[10] **1** crystallizes from a hexane/THF mixture in space group *C*2/*c* with four molecules per unit cell. **1** sits about the crystallographic 2-fold axis with Cu on the special position ($^1/_2$, 0.7021, $^1/_4$). The complex involves a highly distorted tetrahedral environment of two phosphorus and two nitrogen atoms about a Cu(I) center (Figure 1). The most striking feature of the structure is that it contains two cyanoacetate units, both bonded to Cu(I) via the cyano moiety as opposed to the carboxylate functionality, with strong intermolecular hydrogen bonding between molecular units (Figure 2). The Cu-N(1)-C(1) angle is nonlinear, being 153.3 (3)°. Because of the large steric requirements of the triphenylphosphine ligands, the P-Cu-P angle is obtuse at 123.1 (1)°; correspondingly, the N-Cu-N angle is acute at 86.2 (2)°. The P-Cu-P angle in triphenylphosphine derivatives of Cu(I) generally differ greatly from the tetrahedral value of 109.5°, ranging 120-133°.[8,11] The Cu-P and Cu-N bond distances, 2.264 (2) and 2.083 (4) Å, respectively, are similar to those reported for related derivatives.[8,11,12]

The carboxylate groups are all equivalent, where the C(3)-O(1)H distance is 1.272 (6) Å, with the C(3)-O(2) bond length being 1.216 (9) Å. The O···H···O distance at 2.487 Å is considerably less than the sum of the van der Waals radii of the two oxygen atoms.[13] This is consistent with related carboxylate species, which contain strong symmetric hydrogen bonds; e.g., in $KH(CF_3CO_2)_2$ the hydrogen's position was found centered between the two oxygen atoms by both neutron and X-ray diffraction data, with an O···H···O distance of 2.435 Å.[14] These very strong centered hydrogen bonds generally have associated bond energies greater than 50 kJ/mol.

The solution structure of **1** is compatible with what would be anticipated upon dissolution on the basis of its solid-state structure; i.e., it possesses both an acid (-COOH) and a carboxylate ($-CO_2^-$) functionality. For example, free cyanoacetic acid exhibits a C=O stretching vibration at 1746 cm^{-1} in THF solution, whereas complex **1** in THF displays C=O vibrations at 1746 and 1609 cm^{-1}. This latter frequency corresponds to the asymmetrical stretching band of the carboxylate ion. On the other hand, the solid-state infrared spectrum of **1** exhibits only a -COOH vibrational mode shifted slightly to lower frequency at 1735 cm^{-1} due to the observed intermolecular hydrogen bonding. Concomitantly, the -C≡N stretch in THF solution in the free acid at 2261 cm^{-1} is shifted to lower frequency at 2254 cm^{-1} in the solution spectrum of **1**. This latter observation is consistent with both cyanoacetate ligands being bound through the nitrogen donor and is most likely indicative of a small degree of π back-bonding from the Cu(I) into the π^* orbitals of the nitrile group.[15]

Present studies are underway to assess the possible role of species containing cyanoacetate ligands bound as in complex **1** in the decarboxylation reaction of cyanoacetic acid with copper(I) phosphine derivatives. Additionally, we are pursuing investigations aimed at determining the conditions for affording copper(I) phosphine complexes containing the cyanoacetate ligand bound via the carboxylate functionality. Indeed, the role of both types of interaction at the metal center (**2**) has been suggested to be of general importance in decarboxylation processes.[16]



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Acknowledgment. Financial support from the National Science Foundation (Grant 88-17873) is gratefully acknowledged.

Supplementary Material Available: Listings of atomic coordinates and isotropic thermal parameters, anisotropic thermal parameters, H atom coordinates and isotropic displacement parameters, and all bond distances and bond angles (4 pages); a listing of observed and calculated structure factors (14 pages). Ordering information is given on any current masthead page.

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(10) Crystal data for **1** ($C_{42}H_{35}N_2O_4P_2Cu$): $M_r = 757.2$; space group *C*2/*c* (No. 15); $a = 17.377$ (11), $b = 23.962$ (14), $c = 11.363$ (3) Å; $\beta = 117.44$ (4)°; $V = 4199$ (4) Å^3; $Z = 4$; $d_{calc} = 1.198$ g cm^{-3}; Nicolet R3m instrument; 193 K; Mo Kα ($\lambda = 0.71073$ Å); scan method ω (Wyckoff); data collection range $4 < 2\theta < 50°$; number of unique data 3726; number of total data used 2953 ($I > 2\sigma(I)$); solution by heavy-atom methods (Patterson and Fourier) with full-matrix least-squares refinement to convergence (number of refined parameters 231); $R = 0.054$; $R_w = 0.067$, $S = 2.86$.

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Comments on the Substitutional Lability of the Dimetal Carboxylates of Molybdenum and Rhodium. Effects of M-M MO Configuration

The substitutional lability of mononuclear transition-metal ions is greatly influenced by electronic configuration. For example, the rate of H_2O exchange in the aquo ions $M(H_2O)_6^{n+}$ spans a range of 10^{16} at room temperature and the effects of electronic configuration far outweigh the combined effects of the charge and size of the ion; cf. $k_{exch}(H_2O) = 10^9$ s^{-1} for $Cr(H_2O)_6^{2+}$ and 10^{-6} s^{-1} for $Cr(H_2O)_6^{3+}$.[1] Substitutionally inert octahedral ions are associated with the t_{2g}^6 (e.g. Co^{III}, Rh^{III}) and t_{2g}^3 (Cr^{III}) configurations, while *super labile* ions are found for the configurations $t_{2g}^3e_g^1$ (Cr^{2+}) and $t_{2g}^6e_g^3$ (Cu^{2+}) that contain an odd number of electrons in the destabilized e_g orbitals (M-L σ^*) and are subject to pronounced Jahn-Teller distortions. These ground-state effects underscore the kinetic applications of crystal field theory. We wish here to note that the substitutional lability of closely related dinuclear complexes is likewise influenced by electronic structure and, in particular, the M-M MO configuration.

The ligand redistribution reaction shown in eq 1 has been followed by NMR spectroscopy. For M = Mo there is rapid

$$M_2(O_2CR)_4 + M_2(O_2CR')_4 \xrightleftharpoons[\text{benzene-}d_6]{T\ ^\circ\text{C}} M_2(O_2CR)_n(O_2CR')_{4-n} \tag{1}$$

scrambling ($T = 22$ °C) with the formation of all possible $Mo_2(O_2CR)_n(O_2CR')_{4-n}$ (R = *t*-Bu, R′ = CH$_2$-*t*-Bu or *p*-*t*-BuC$_6$H$_4$). No similar exchange ($T = 60$ °C) occurs for rhodium (R = *t*-Bu, R′ = CH$_2$-*t*-Bu).

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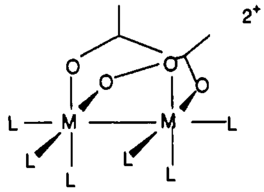
The $M_2(O_2CR)_4$ compounds are known to react with trialkyloxonium salts in CH_3CN to give solvated cations $M_2(O_2CR)_2^{2+}$.[2] When $M_2(O_2CR)_4$ and the $M_2(O_2CR)_2^{2+}$-containing salts are mixed in acetonitrile as solvent, the equilibrium (2) is readily established for M = Mo and R = *t*-Bu (T = 22 °C, 24 h), but no reaction is observed for M = Rh and R = Me even under reflux (T = 82 °C, 24 h).

$$M_2(O_2CR)_4 + M_2(O_2CR)_2^{2+} \underset{CH_3CN}{\overset{T\,°C}{\rightleftharpoons}} 2M_2(O_2CR)_3^{+} \quad (2)$$

The solvated cation $Rh_2(O_2CMe)_3^+$ can be prepared from a reaction involving $Rh_2(O_2CMe)_4$ and $Me_3O^+BF_4^-$ (1.0 equiv) in CH_3CN. This generates a mixture of $Rh_2(O_2CMe)_2^{2+}$, $Rh_2(O_2CMe)_3^+$, and unreacted $Rh_2(O_2CMe)_4$, as determined by NMR spectroscopy. The $Rh_2(O_2CMe)_3^+$ cation has been prepared separately from the reaction between $Rh_2(O_2CMe)_2^{2+}$ and O_2CMe^- in CH_3CN.

The solvated cations $M_2(O_2CMe)_2(CH_3CN)_6^{2+}$ have been shown to share a common geometry, depicted by A, having two cis-bridging acetate ligands. The CH_3CN ligands fall into two types—those lying along the M–M axis and those oriented perpendicular to the M–M bond, referred to hereafter as axial and equatorial MeCN ligands, respectively.



A

M = Mo, Rh; L = CH_3CN

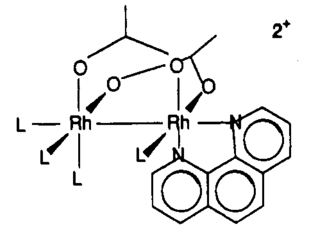
The axial M–N distances are notably longer than the M–N equatorial distances (Δ = 0.5 Å for M = Mo and 0.25 Å for M = Rh), and upon dissolution of the $M_2(O_2CR)_2(CH_3CN)_6^{2+}$ ions in CD_3CN the axial CH_3CN ligands undergo facile exchange with CD_3CN. The weakly bound and substitutionally labile axial CH_3CN ligands are understandable in terms of the high trans influence and trans effect of the M–M bond. However, the substitutional lability of the four equatorial CH_3CN ligands in the solvated $M_2(O_2CR)_2^{2+}$ cations is markedly different for M = Mo and Rh (eq 3).

$$M_2(O_2CR)_2(CH_3CN)_4^{2+} + CD_3CN\ (\text{solvent}) \xrightarrow{T\,°C} M_2(O_2CR)_2(CD_3CN)_4^{2+} + 4CH_3CN \quad (3)$$

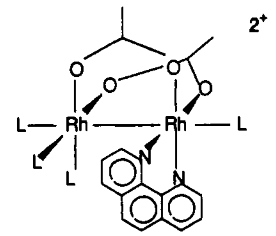
For M = Mo, the exchange of CH_3CN for CD_3CN occurs rapidly upon dissolution of the salt at −45 °C, but for M = Rh, the $t_{1/2}$ for exchange is ca. 4 h at 100 °C. From studies of reaction 3 for M = Rh in the temperature range +93 to +124 °C we have determined the activation parameters for CH_3CN/CD_3CN solvent exchange to be ΔH^{*} = 33 (1) kcal mol^{-1} and ΔS^{*} = +11 (3) eu,[3] suggestive of bond breaking in the transition state.

We propose that the relative substitutional lability of the Mo_2^{4+} and Rh_2^{4+} carboxylates can be correlated with the M–M MO configuration.[4] The $\sigma^2\pi^4\delta^2$ configuration has low-lying, empty, metal-centered orbitals δ^* and π^*, and the molybdenum species can therefore react via an associative or I_a mechanism. The Rh_2^{4+} complexes have M–M single bonds as a result of the M–M MO configuration $\sigma^2\pi^4\delta^2\delta^{*2}\pi^{*4}$ and are thus substitutionally inert to the associative or I_a mechanism in a manner akin to octahedral t_{2g}^6 ML_6 complexes. The activation parameters for CH_3CN/CD_3CN exchange for rhodium are consistent with a transition state involving bond breaking by the I_d mechanism.

Although the $Rh_2(O_2CMe)_2(CH_3CN)_4^{2+}$ cation is inert to exchange with the solvent CD_3CN at room temperature, the reaction between this cation and chelating ligands such as acetate, α,α'-bipyridine and phenanthroline do proceed at a reasonable rate at room temperature. For example in studies of the reaction with phenanthroline (phen) in CD_3CN at 22 °C formation of B is detectable after 20 min by ^{1}H NMR spectroscopy. After 48 h the conversion to C is essentially complete. On the basis of the ^{1}H NMR data,[3] we formulate the $Rh_2(O_2CMe)_2(phen)(CH_3CN)_4^{2+}$ isomers as

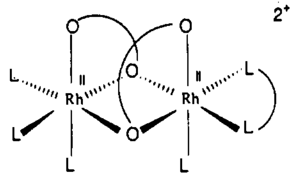


B



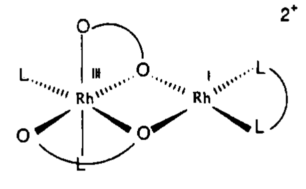
C

What is perhaps most remarkable in studies of the reactions between $Rh_2(OAc)_2(CH_3CN)_4^{2+}$ and the chelating ligands O_2CMe^-, phen, and α,α'-bipyridine in CD_3CN is that even before formation of B and C there is CH_3CN for CD_3CN exchange in the starting material. Thus, there must be a chemically more rapid and reversible reaction between $Rh_2(O_2CMe)_2(CH_3CN)_4^{2+}$ and phen to give a substitutionally labile activated complex wherein CH_3CN for CD_3CN occurs. Why this should yield facile



$t_{2g}^6 e_g^1$ $t_{2g}^6 e_g^1$

D



t_{2g}^6 d^8

E

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(2) Garner, C. D.; Clegg, W.; Pimblett, G. *J. Chem. Soc., Dalton Trans.* **1986**, 1257. These authors also reported the X-ray structures of the compounds $M_2(OCR)_2(CH_3CN)_6^{2+}(BF_4^-)_2$ for M = Mo and Rh and mentioned that the ^{1}H NMR spectra of these compounds when recorded in CD_3CN indicated the exchange described in detail in this work. The preparation and structure of $Mo_2(OCR)_2(CH_3CN)_6^{2+}(BF_4^-)_2$ was independently reported in the previous year: Cotton, F. A.; Reid, A. H., Jr.; Schwotzer, W. *Inorg. Chem.* **1985**, *24*, 3965.

(3) Studies of the CH_3CN for CD_3CN exchange involving $Rh_2(O_2CMe)_2(CH_3CN)_6^{2+}(BF_4^-)_2$ dissolved in CD_3CN as solvent were followed by ^{1}H NMR spectroscopy. Typical samples involved ca. 22 mg of metal complex dissolved in 0.6 mL of CD_3CN, approximately 10 mM solutions. Spectra were recorded at room temperature, but samples were heated in an oil bath, 93–124 °C, to induce the CH_3CN/CD_3CN exchange. The rate of reaction was followed by integration of the coordinated CH_3CN (equatorial) signal relative to the acetate signal. The latter provided an internal standard, being invariant with time. Plots of ln $[CH_3CN]/[CH_3CO_2]$ versus time gave a straight line of slope k; plots of $-\ln k$ versus $1/T$ gave a straight line where slope = E_{Act}; plots of $(-\ln k)/T$ versus $1/T$ gave slope = ΔH^{*} and intercept = ΔS^{*}. Errors were determined according to procedures described by: Mortimer, R. G. In *Mathematics for Physical Chemistry*; MacMillan Publishing Co., Inc.: New York, 1981; pp 266–309. Characteristic of NMR data for B is the presence of two acetate signals in the ratio 1:1 and three CH_3CN (equatorial) signals in the ratio 1:1:1. Also the chelating ligand occupies two different coordination sites in B whereas in C the chelating ligand occupies two equivalent equatorial sites. In C there is only one acetate signal (6 H) and one CH_3CN (equatorial) signal (6 H). NMR (at 22 °C, 300 MHz, CD_3CN as solvent): for B where L–L = 1,10-phenanthroline, $\delta(O_2CMe)$ = 1.66 (3 H) s and 2.33 (3 H) s, $\delta(CH_3CN_{eq})$ = 2.16 (3 H) s, 2.41 (3 H) s, and 2.61 (3 H) s, $\delta(CH_3CN_{ax})$ = 1.955 (6 H) s, δ(phen) = 8.03 (m), 8.21 (m), 8.25 (d), 8.30 (d), 8.78 (m), and 8.92 (m) (all of 1 H) and 9.26 (m) (2 H); for C where L–L = 1,10-phenanthroline, $\delta(O_2CCH_3)$ = 2.31 (6 H) s, $\delta(CH_3CN_{eq})$ = 1.73 (6 H) s, $\delta(CH_3CN_{ax})$ = 1.955 (6 H) s, δ(phen) = 8.07 (m), 8.27 (s), 8.805 (m), and 8.89 (m) (all of intensity 2 H).

(4) For a description of the electronic structure, see: Cotton, F. A.; Walton, R. A. In *Multiple Bonds Between Metal Atoms*; 1982, John Wiley & Sons: New York, 1982. There is no reason to believe that Mo–O and Mo–N bonds are significantly weaker than the Rh–ligand bonds in these complexes. Indeed, one might well expect the reverse to be true.

CD_3CN/CH_3CN exchange but less rapid formation of B is not obvious. However, in Rh_2^{4+} carboxylates the weakest bond is probably the M-M bond and it is not unreasonable that reaction with a chelate could lead to a change in M-M MO configuration and thereby render the metal ions substitutionally labile. For example, edge-shared bioctahedra are common for M_2L_{10} complexes and reversible formation of an activated complex of type D (Rh^{II}-Rh^{II}) or E (Rh^{III}-Rh^{I}) with a chelating ligand L-L would generate substitutional labile metal centers Rh^{II} $t_{2g}^6e_g^1$ and square-planar Rh^I d^8.

Detailed studies of substitution reactions at dinuclear metal centers are clearly warranted.

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Articles

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Thermal Decomposition of Energetic Materials. 48. Structures and Decomposition Mechanisms of Copper(II) Complexes of Furazans (1,2,5-Oxadiazoles)

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The thermal decomposition of aminofurazan (furazan = 1,2,5-oxadiazole) complexes of Cu(II)-nitrate $[Cu(diaminofurazan)_2(H_2O)_2](NO_3)_2$ (**1**), $[Cu(furazanopiperazine)_2(CH_3CN)_2](NO_3)_2$ (**2**), and $[Cu(furazanopiperazine)_2(H_2O)_2](NO_3)_2$ (**3**) is initiated by partial displacement of H_2O or CH_3CN from the coordination sphere by NO_3^-. Proton transfer occurs forming HNO_3, some of which vaporizes. The thermal decomposition products of the complexed furazan molecule differ from the uncomplexed molecule largely because of this proton-transfer reaction. The thermal decomposition of $[Cu(furazanopiperazine)_4(H_2O)_2](ClO_4)_2{\cdot}CH_3NO_2$ (**4**) is more extensive in the early stage than is the case for the analogous nitrate salts. The crystal structures of **1** and **4** show that the furazan ligand and Cu(II) bond through a ring nitrogen atom. Crystal data for **1**: monoclinic, $P2_1/c$, $a = 6.382$ (1) Å, $b = 16.294$ (4) Å, $c = 7.232$ (2) Å, $\beta = 109.32$ (2)°, $V = 709.8$ (3) Å,^{3} $Z = 2$. For **4**: orthorhombic, *Pnnm*, $a = 6.596$ (2) Å, $b = 18.362$ (8) Å, $c = 14.204$ (5) Å, $V = 1720$ (1) Å^3, $Z = 2$.

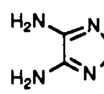
Introduction

The coordination chemistry of simple aminofurazans (furazan = 1,2,5-oxadiazole) is unknown. A potential ambiguity exists regarding the ligand donor site: does the amine nitrogen atom or the ring nitrogen atom or the oxygen atom serve as the Lewis base toward a transition-metal atom?

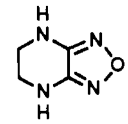
A practical aspect of aminofurazans is the fact that diaminofurazan (3,4-diamino-1,2,5-oxadiazole) suppresses the burn rate and changes the pressure dependence of combustion of rocket propellants containing NH_4ClO_4 oxidizer.[1] Several mechanisms for this effect have recently been proposed on the basis of the thermal decomposition products of diaminofurazan at high heating rates.[2] Among the products are highly thermally stable cyclic azines, such as melamine and melon. These thermally stable solids may suppress the heat and mass transfer at the burning surface.[2] The precursor compounds to the azines are NH_2CN and $[NH_4]N(CN)_2$.[2] Another mechanism is the formation of NH_3, which is known to impede the decomposition of NH_4ClO_4.[3,4]

A potential useful variation on furazan-type burn-rate modifiers might be gained by the use of metal complexes of furazans. Upon rapid decomposition of the complex, the metal center could become coordinatively unsaturated and be able to catalyze reactions in the condensed phase. At the same time the products from the furazan ligands might alter the heat and mass transfer during the surface thermolysis process. The only previously reported transition-metal complexes involving the simple furazan ligand are $ML_3(SbCl_5)_2$ salts, where L = 1,2,5-oxadiazole.[5] On the basis of electronic spectra, it was suggested that L bridges the metal atoms through its N atoms, giving octahedral coordination about M.[5] However, no crystal structures have been determined to confirm the bonding. Quadradentate chelate ligands containing diaminofurazan have recently been found to complex first-row transition-metal ions,[6-8] but these compounds are only remotely related to the ones of interest here.

Four new Cu(II) complexes of the neutral furazan ligands, diaminofurazan (DAF) and furazanopiperazine (FP), were prepared and several crystal structures were determined. DAF could bond to Cu(II) through the NH_2 groups or the ring nitrogen atom. The complexes described are $[Cu(DAF)_2(H_2O)_2](NO_3)_2$ (**1**), $[Cu(FP)_2(CH_3CN)_2](NO_3)_2$ (**2**), $[Cu(FP)_2(H_2O)_2](NO_3)_2$ (**3**), and $[Cu(FP)_4(H_2O)_2](ClO_4)_2{\cdot}CH_3NO_2$ (**4**). The crystal structures of **1** and **4** and the thermal decomposition of all of the salts at slow (5 °C/min) and fast (>90 °C/s) heating rates were determined. The IR-active gas products reveal important features of the thermal decomposition mechanisms of these complexes.



DAF



FP

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