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# Preparation and Crystal Structure of the New Paramagnetic One-Dimensional Conductor $Co_{0.83}[Pt(C_2O_4)_2]\cdot 6H_2O$

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A new partially oxidized bis(oxalato)platinate complex has been prepared which contains paramagnetic $Co^{2+}$ counterions. The crystal and molecular structure of this new salt, $Co_{0.83}[Pt(C_2O_4)_2]\cdot 6H_2O$, has been determined by single-crystal x-ray diffraction. The complex crystallizes in the orthorhombic space group *Cccm* with cell parameters of $a = 14.379$ (2) Å, $b = 16.501$ (2) Å, $c = 5.682$ (1) Å, and $Z = 4$. Intensity data were collected on a Syntex $P2_1$ diffractometer for one octant out to a maximum $2\theta$ of 42° using Mo $K\alpha$ x radiation. The structure was solved by Patterson methods and refinement led to final discrepancy indices of $R(F_o^2) = 0.029$ and $R_w(F_o^2) = 0.045$. The structure consists of planar bis(oxalato)platinates stacked along the $c$ direction with a Pt–Pt separation of 2.841 (1) Å. The rotation angle between neighboring $Pt(C_2O_4)_2^{1.66-}$ groups is 55.0 (3)°. Each $Co^{2+}$ ion is octahedrally coordinated to six water molecules, although these sites are only 83% occupied. Furthermore, we have located a completely occupied water site which is not within the coordination sphere of the $Co^{2+}$ ion. Since in all other respects $Co_{0.83}[Pt(C_2O_4)_2]\cdot 6H_2O$ and the structure of $Mg_{0.82}[Pt(C_2O_4)_2]\cdot 5.3H_2O$ reported by Krogmann appear to be isostructural, we believe the magnesium salt may also contain this additional water site. Thermal gravimetric analysis of the cobalt salt has verified the degree of hydration is exactly 6.0.

## Introduction

Highly conducting one-dimensional inorganic compounds are mainly restricted to the partially oxidized tetracyanoplatinate (POTCP)[2a] or bis(oxalato)platinate (POBOP) complexes.[2] Although both classes of compounds consist of stacks of planar complexes with high electrical conductivity along the Pt–Pt stacking direction, the two types of complexes also have unique differences. For instance, the POTCP complexes may be either cation or anion deficient, such as $K_{1.75}[Pt(CN)_4]\cdot 1.5H_2O$[3] and $K_2[Pt(CN)_4]Br_{0.30}\cdot 3H_2O$,[4] respectively, but only cation-deficient POBOP complexes are known, such as $Mg_{0.82}[Pt(C_2O_4)_2]\cdot 5.3H_2O$[5] and $K_{1.6}[Pt(C_2O_4)_2]\cdot 1.2H_2O$.[6] Furthermore, it has not yet been possible to prepare POTCP complexes with divalent cations,[7] although hydrated $M(OH_2)_x^{2+}$ ions (M = Mg, Ca, Sr, Ba) readily form POBOP salts.[8]

The ability of POBOP complexes to crystallize with highly hydrated divalent alkali earth metal cations should permit the preparation of salts containing divalent *transition-metal* cations also. Following this line of reasoning, we have prepared a new POBOP salt containing the paramagnetic $d^7$ $Co^{2+}$ ion. The synthesis and crystal structure of this complex are presented herein.

## Experimental Section

**Preparation of $Co_{0.83}[Pt(C_2O_4)_2]\cdot 6H_2O$.** Ten milliliters of 1 M HCl was added to 5 g of $AgPt(C_2O_4)_2$ suspended in 100 mL of degassed water under an atmosphere of $N_2$. The AgCl precipitate was coagulated by heating the solution and was then filtered off to yield a clear yellow solution of $H_2Pt(C_2O_4)_2$. Two grams of $Co(NO_3)_2\cdot 6H_2O$ dissolved in 20 mL of water was added to the filtrate and the resulting solution was filtered and allowed to stand in air at 22 °C for several weeks. The solution slowly turned blue and deposited needle-shaped crystals of the product.

**Data Collection.** The crystal selected for data collection had dimensions of 0.025 × 0.075 × 0.218 mm and was sealed in a thin-walled glass capillary. With the crystal mounted in a general orientation, data were collected on a Syntex $P2_1$ diffractometer equipped with a graphite monochromator. A least-squares fit of the diffractometer angles obtained by automatically centering 25 reflections in the range $20° < 2\theta < 30°$ (Mo $K\alpha$ x radiation, $\lambda$ 0.710 69 Å) yielded cell dimensions of $a = 14.379$ (2) Å, $b = 16.501$ (2) Å, $c = 5.682$ (1) Å, and $V_c = 1348$ Å$^3$. The data revealed systematic extinctions consistent with the *C*-centered orthorhombic space groups *Ccc*2 [$C_{2v}^{13}$—No. 37][9a] and *Cccm* [$D_{2h}^{20}$—No. 66].[9a] The centrosymmetric space group *Cccm* was chosen initially and was justified by the satisfactory least-squares refinement ultimately obtained.

Intensity data were collected for one octant out to $2\theta = 42°$ using the $\theta$–$2\theta$ step–scan mode with variable scan rates of 0.49–29.30° min$^{-1}$. The data were corrected for absorption ($\mu_{calcd} = 120.34$ cm$^{-1}$) with transmission coefficients ranging from 0.58 to 0.74, and a set of $F_o^2$ values, where $F_o$ is the observed structure factor amplitude, was obtained. The variances of $F_o^2$ were calculated using the expression $\sigma^2(F_o^2) = \sigma_c^2(F_o^2) + (0.03F_o^2)^2$, where $\sigma_c^2(F_o^2)$ is determined from counting statistics and 0.03 is an added factor deduced from the 3% maximum variation of the intensities of three reference reflections which were measured periodically throughout the data collection.

**Solution and Refinement of the Structure.** The initial locations of the Pt and Co atoms were obtained from a Patterson synthesis map. The positions of the oxalate ligand atoms and the three independent oxygen atoms coordinated to the $Co^{2+}$ ion were determined by standard Fourier and least-squares refinement[11] procedures. This led to discrepancy indices[12] of $R(F_o) = 0.049$, $R(F_o^2) = 0.055$, and $R_w(F_o^2) = 0.088$ with anisotropic thermal parameters for all atoms. At this stage, a scaled difference Fourier map showed a peak centered about ($^1/_4$, $^1/_4$, $^1/_2$) with small maxima of ~2.6 e/Å$^3$ at ($^1/_4$, $y$, $^1/_2$) for $y = 0.21$ and 0.29. An oxygen atom was placed on the special position ($^1/_4$, $^1/_4$, $^1/_2$) and successfully refined anisotropically.

In order to experimentally determine the site occupancies of the Co atom, the three independent oxygen atoms in the Co coordination sphere, and the noncoordinated oxygen atom, the atom multipliers for all five atoms were allowed to vary independently. This led to site occupancies of 0.414 (3) for Co, 0.87 (2), 0.87 (1), and 0.85 (2) for the coordinated O atoms, and 1.07 (2) for the uncoordinated O atom. The last value was considered to be not significantly different from 1.0 and was reset to unity. For $Mg_{0.82}[Pt(C_2O_4)_2]\cdot 5.3H_2O$, Krogmann[5] obtained nonequivalent occupancies for the three coordinated water oxygen atoms of 1.00 (4), 0.94 (4), and 0.79 (5). For the Co salt, the three values are equivalent and, furthermore, are nearly equal to twice the Co site occupancy. These values coincide precisely with those expected if each Co atom is always hexahydrated. Therefore, using the more reliable Co atom multiplier, the multipliers for the three coordinated O atoms were set to exactly twice the Co value for final least-squares refinements.

Least-squares refinement of this model led to final discrepancy indices for all 409 independent reflections[13] of $R(F_o) = 0.036$, $R(F_o^2) = 0.029$, and $R_w(F_o^2) = 0.045$, with the standard deviation for an observation of unit weight equal to 1.08. Positional and anisotropic thermal parameters determined from the final least-squares cycle are presented in Table I, and the root-mean-square amplitudes of vibration are given in Table II.

## Discussion

A nonperspective view of the *C*-centered centrosymmetric orthorhombic unit cell with the atom-labeling scheme is shown in Figure 1. A view of two nearest-neighbor stacks of $Pt(C_2O_4)_2^{1.66-}$ units showing hydrogen-bonding interactions is presented in Figure 2. The principal distances and angles are given in Table III.

The $Pt(C_2O_4)^{1.66-}$ moieties stack in the $c$ direction with a Pt–Pt spacing of $c/2 = 2.841$ (1) Å, as compared to 2.85 Å in $Mg_{0.82}[Pt(C_2O_4)_2]\cdot 5.3H_2O$, and are crystallographically

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**Table I.** Final Positional and Thermal Parameters for $Co_{0.83}[Pt(C_2O_4)_2]\cdot 6H_2O$

| Atom | Site occupancy | $x^a$ | $y$ | $z$ |
|---|---|---|---|---|
| Pt | 1.0 | 0.0 | 0.0 | 0.0 |
| Co | 0.414 (3) | 0.0 | 0.3405 (2) | 0.25 |
| O(1) | 1.0 | 0.0502 (5) | 0.1132 (4) | 0.0 |
| O(2) | 1.0 | 0.1834 (6) | 0.1789 (4) | 0.0 |
| O(3) | 1.0 | 0.2753 (6) | 0.0299 (5) | 0.0 |
| O(4) | 1.0 | 0.1367 (6) | −0.0293 (5) | 0.0 |
| O(5) | 0.828 | −0.0626 (6) | 0.2520 (6) | 0.0 |
| O(6) | 0.828 | 0.1111 (6) | 0.3372 (6) | 0.0 |
| O(7) | 0.828 | −0.0647 (8) | 0.4308 (7) | 0.0 |
| O(8) | 1.0 | 0.25 | 0.75 | 0.0 |
| C(1) | 1.0 | 0.1391 (8) | 0.1167 (7) | 0.0 |
| C(2) | 1.0 | 0.1878 (9) | 0.0314 (8) | 0.0 |

| Atom | $\beta_{11}{}^b$ | $\beta_{22}$ | $\beta_{33}$ | $\beta_{12}$ | $\beta_{13}$ | $\beta_{23}$ |
|---|---|---|---|---|---|---|
| Pt | 1.84 (3) | 1.10 (2) | 15.5 (2) | −0.19 (5) | 0 | 0 |
| Co | 2.0 (2) | 1.4 (1) | 42 (2) | 0 | 3 (1) | 0 |
| O(1) | 2.3 (4) | 1.7 (3) | 21 (3) | −0.8 (3) | 0 | 0 |
| O(2) | 2.8 (5) | 1.3 (3) | 61 (5) | −0.9 (4) | 0 | 0 |
| O(3) | 3.0 (5) | 2.7 (4) | 29 (3) | 0.0 (3) | 0 | 0 |
| O(4) | 1.7 (5) | 2.2 (4) | 22 (3) | 0.3 (4) | 0 | 0 |
| O(5) | 1.6 (5) | 2.2 (4) | 100 (9) | 0.3 (4) | 0 | 0 |
| O(6) | 1.1 (4) | 2.8 (4) | 75 (7) | 0.3 (4) | 0 | 0 |
| O(7) | 4.0 (7) | 3.6 (6) | 135 (13) | 1.4 (5) | 0 | 0 |
| O(8) | 5 (1) | 42 (4) | 52 (10) | 6 (2) | 0 | 0 |
| C(1) | 2.3 (6) | 1.6 (6) | 22 (5) | −0.4 (5) | 0 | 0 |
| C(2) | 1.8 (8) | 2.9 (6) | 16 (5) | 1.0 (6) | 0 | 0 |

[a] $x$, $y$, and $z$ are fractional coordinates. [b] Anisotropic temperature factors are $\times 10^3$ and are of the form $\exp\{-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)\}$.

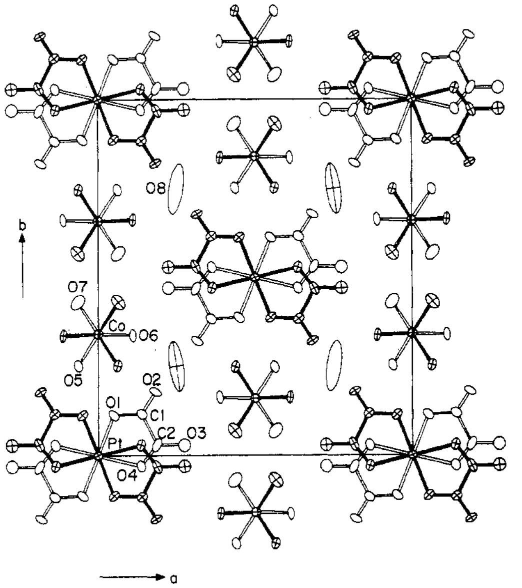

**Figure 1.** Nonperspective view of half of the unit cell of $Co_{0.83}[Pt(C_2O_4)]\cdot 6H_2O$ with $0 \leq z \leq {}^1/_2$ viewed parallel to $c$. The atoms with boundary ellipses only are at $z = 0$ and those with principal ellipses are at $z = {}^1/_2$, except for the Co atoms which are at $z = {}^1/_4$. All thermal ellipsoids enclose 50% probability.

**Table II.** Root-Mean-Square Amplitudes of Vibration (Å) for $Co_{0.83}[Pt(C_2O_4)_2]\cdot 6H_2O$

| Atom | $\mu_1$ | $\mu_2$ | $\mu_3$ |
|---|---|---|---|
| Pt | 0.119 (2) | 0.143 (2) | 0.159 (1) |
| Co | 0.134 (8) | 0.140 (6) | 0.266 (6) |
| O(1) | 0.12 (2) | 0.18 (1) | 0.19 (1) |
| O(2) | 0.11 (2) | 0.19 (1) | 0.32 (1) |
| O(3) | 0.18 (1) | 0.19 (1) | 0.22 (1) |
| O(4) | 0.13 (2) | 0.18 (2) | 0.19 (1) |
| O(5) | 0.12 (2) | 0.18 (2) | 0.40 (2) |
| O(6) | 0.10 (2) | 0.20 (2) | 0.35 (2) |
| O(7) | 0.17 (2) | 0.25 (2) | 0.47 (2) |
| O(8) | 0.21 (3) | 0.29 (3) | 0.77 (4) |
| C(1) | 0.13 (3) | 0.17 (2) | 0.19 (2) |
| C(2) | 0.12 (3) | 0.16 (2) | 0.21 (2) |

**Table III.** Distances and Angles for $Co_{0.83}[Pt(C_2O_4)_2]\cdot 6H_2O$

| | | | |
|---|---|---|---|
| | Distances, Å | | |
| Pt–Pt | 2.841 (1) | Co–O(5) | 2.227 (8) |
| Pt–O(1) | 2.003 (7) | Co–O(6) | 2.138 (7) |
| Pt–O(4) | 2.024 (8) | Co–O(7) | 2.259 (9) |
| C(1)–O(1) | 1.28 (1) | O(5)–O(1) | 2.81 (1) |
| C(1)–O(2) | 1.21 (1) | O(6)–O(3) | 2.74 (1) |
| C(2)–O(3) | 1.26 (2) | O(6)–O(2) | 2.81 (1) |
| C(2)–O(4) | 1.24 (1) | O(7)–O(3) | 2.82 (1) |
| C(1)–C(2) | 1.57 (2) | O(8)–O(5) | 2.695 (9) |
| | Angles, Deg | | |
| O(1)–Pt–O(4) | 82.7 (3) | O(3)–C(2)–O(4) | 125 (1) |
| Pt–O(1)–C(1) | 113.7 (7) | O(3)–C(2)–C(1) | 118 (1) |
| Pt–O(4)–C(2) | 112.5 (6) | O(4)–C(2)–C(1) | 117.3 (9) |
| O(1)–C(1)–O(2) | 124 (1) | O(5)–Co–O(6) | 82.0 (3) |
| O(1)–C(1)–C(2) | 114 (1) | O(5)–Co–O(7) | 82.2 (3) |
| O(2)–C(1)–C(2) | 122 (1) | O(6)–Co–O(7) | 84.6 (3) |

required to have $2/m$ symmetry. Adjacent $Pt(C_2O_4)^{1.66-}$ are staggered by 55.0 (3)° and alternate groups are in an eclipsed configuration. In $Mg_{0.82}[Pt(C_2O_4)_2]\cdot 5.3H_2O$ the rotation angle is 58°,[5] and it has been suggested that this configuration (~60° rotation)[14] or the staircase configuration (45° rotation)[15] observed for $K_{1.6}[Pt(C_2O_4)_2]\cdot 1.2H_2O$[6] is the result of favorable intermolecular back-bonding overlaps between the oxalate ligands. However, as noted by Miller[14] for $Mg_{0.82}[Pt(C_2O_4)_2]\cdot 5.3H_2O$, comparison of the intramolecular $Pt(C_2O_4)^{1.64-}$ bond distances and angles with those of the nonstacking $K_2[Pt(C_2O_4)_2]\cdot 2H_2O$ structure[16] shows no unusual differences.

Hydrogen-bonding interactions within and between the one-dimensional strands also appear to contribute to stabilization of the crystal structure, as shown in Figure 2. There are four independent O···O distances of less than 3.0 Å (see Table III) between oxalate oxygen atoms and water molecule oxygen atoms.

The $Co^{2+}$ ions are located between the bis(oxalato)platinate planes and are coordinated to six water molecules with a slightly distorted octahedral geometry. Each Co site is 41.4% occupied and each water site is 82.8% occupied. However, since it is possible for a water molecule to bond to the Co atom above or below it (relative to $c$), the $Co^{2+}$ ions are always hexahydrated. If the Co sites were 50% occupied, each chain

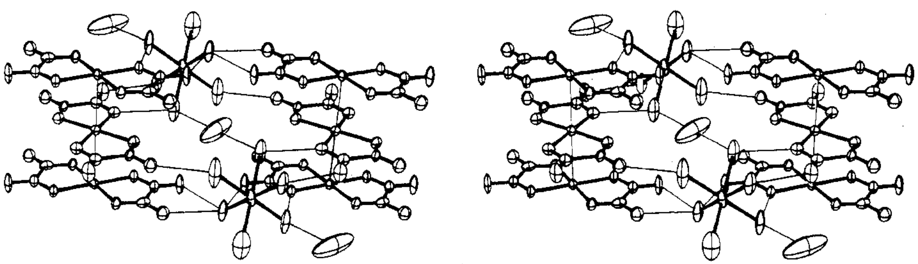

**Figure 2.** Stereoview of two nearest-neighbor $Pt(C_2O_4)^{1.66-}$ chains along the (110) direction showing possible hydrogen-bonding interactions.

of $Co(OH_2)_6^{2+}$ ions (along *c*) would be an ordered array of alternating vacant and occupied Co sites. However, with 41.4% occupancy, the sites are randomly occupied in all three dimensions.

The cobalt bis(oxalato)platinate salt, as described to this point, is essentially isostructural with the magnesium salt, $Mg_{0.82}[Pt(C_2O_4)_2]\cdot 5.3H_2O$, as reported by Krogmann.[5] However, we have located a "new" water site which is not within the $Co^{2+}$ coordination sphere but appears to be disordered about the special position ($^1/_4$, $^3/_4$, 0) as indicated by its elongated thermal ellipsoid (see Table II). This new water molecule appears to be strongly hydrogen bonded to one of the coordinated water molecules, with O(8)···O(5) = 2.695 (9) Å. Apparently it is not bonded to any of the oxalate ligand atoms. Therefore, on the basis of x-ray analysis, each cobalt bis(oxalato)platinate unit is hexahydrated. This result has been confirmed by thermal gravimetric analysis[17] of $Co_{0.83}[Pt(C_2O_4)_2]\cdot 6H_2O$. Since in all other respects the Co and Mg salts are isostructural, one may reasonably speculate that the Mg salt also contains this additional water molecule.

The introduction of the $d^7$ $Co^{2+}$ ion into the lattice of a partially oxidized one-dimensional conductor is the first example of a paramagnetic transition-metal counterion in this role. Studies of the magnetic and electrical conduction properties of $Co_{0.83}[Pt(C_2O_4)_2]\cdot 6H_2O$ are in progress.

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**Registry No.** $Co[Pt(C_2O_4)_2]\cdot 6H_2O$, 65681-92-7; $AgPt(C_2O_4)_2$, 65681-91-6.

**Supplementary Material Available:** A listing of observed and calculated structure factors (3 pages). Ordering information is given on any current masthead page.

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(10) Computer programs used in this study: data reduction and absorption corrections, DATALIB; data averaging and sort, DATASORT; least-squares refinement, ORXFLS3; Fourier summation, FORDAP; error analysis of distances and angles, ORFFE3; structure plotting, ORTEPII.

(11) All least-squares refinements were based on minimization of $\sum_i w_i|F_o^2 - S^2F_c^2|$, where *S* is the scale factor and the individual weights $w_i = 1/\sigma^2(F_o^2)$. Scattering factors are from ref 9b with Pt and Co treated for anomalous scattering.

(12) $R(F_o) = \sum||F_o| - |F_c||/\sum|F_o|$, $R(F_o^2) = \sum|F_o^2 - F_c^2|/\sum F_o^2$, and $R_w(F_o^2) = [\sum w_i|F_o^2 - F_c^2|^2/\sum w_iF_o^4]^{1/2}$.

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(17) We thank K. L. Stearley for performing the thermal gravimetric analyses.

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# Crystal and Molecular Structure of a One-Dimensionally Stacked Binuclear Rhodium(I) Complex Containing a Tetradentate Dibenzoylhydrazido Ligand

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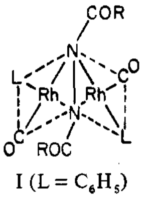
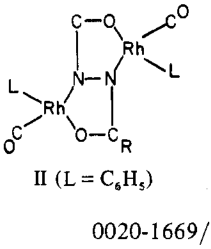
The binuclear rhodium(I) complex $[(CO)_2RhNCOC_6H_5]_2$ has been shown by a crystal structure analysis to contain a dibenzoylhydrazido group with a novel tetradentate bridging configuration. Crystals of *μ-N,N′*-dibenzoylhydrazido-(*N,N′,O,O′*)-bis(dicarbonylrhodium) are monoclinic, space group *I*2/*a*, with four molecules in a cell of dimensions *a* = 10.421 (14) Å, *b* = 14.795 (23) Å, *c* = 11.667 (17) Å, and *β* = 94.63 (3)°. The structure determination was based upon 727 diffraction maxima with 2*θ*(Mo K*α*) ≤ 45°. Full-matrix anisotropic least-squares refinement converged to a final conventional discrepancy factor of 0.046. The crystal structure is made up of centrosymmetric dimers, with the dibenzoylhydrazido ligand utilizing all four of its nitrogen and oxygen atoms to form five-membered chelate rings at each square-planar Rh atom. Bond distances within the chelate ring include Rh–N = 2.071 (10) Å, N–N = 1.398 (18) Å, N–C = 1.309 (16) Å, C–O = 1.255 (15) Å, and O–Rh = 2.028 (9) Å. This structure is believed to be a prototype for a series of related Rh(I) and Ir(I) complexes. The molecules exhibit one-dimensional stacking in the crystal, with Rh–Rh contacts of 3.347 (5) Å between adjacent dimers.

## Introduction

The reaction of *mer*-$RhCl_3L_3$ (L = $PMe_2Ph$, $PPh_3$) with 1,2-dibenzoylhydrazine was reported in 1972 by Hussein and Kasenally to yield a dimeric product, $[L(CO)RhN(COPh)]_2$, for which the N-bridged structure I was suggested.[1] This proposed structure was closely related to that of $[Rh(CO)_2Cl]_2$[2] and contained a substituted hydrazo bridging ligand similar to those which have been found in a series of dimeric bis(tricarbonyliron) species.[3-5] An alternative structure II for the rhodium dimer was proposed by Ittel and Ibers, who found the N,O-chelating configuration III for the dibenzoylhydrazido ligand in $Pt(PPh_3)_2(PhCONNCOPh)$.[6] A number of analogues of the rhodium dimer were subsequently reported by Kasenally and Hussein.[7] These workers found no definitive basis for distinction between structures I and II, though they tended to favor II.


I (L = $C_6H_5$)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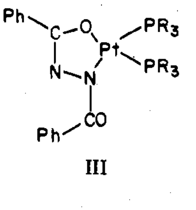
III

A potentially interesting aspect of the proposed structures is the electronic nature of the bridging ligand, which could be