in good agreement with those in $(CH_2)_6N_4$[21] and in $(CH_2)_6N_4O$.[15]

As a consequence of the large cationic size in the present structure, the I_8^{2-} aggregates are well separated from one another. The observed nonbonded I···I contact of 4.28 Å is the second largest among discrete higher polyiodides and is appreciably longer than the corresponding interionic distance in Cs_2I_8 (Table I).

Registry No. $[(CH_2)_6N_4CH_3]_2I_8$, 70955-00-9; $(CH_2)_6N_4O$, 62190-92-5; $(CH_2)_6N_4$, 100-97-0; CH_3I, 74-88-4; I_2, 7553-56-2.

Supplementary Material Available: Thermal parameters (Table IV), positions of generated methylene H atoms (Table V), comparison of observed and calculated structure factors (Table VI), and a stereoview of the molecular packing (Figure 3) (43 pages). Ordering information is given on any current masthead page.

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Dimeric (Pentamethylcyclopentadienyl)rhodium and -iridium Complexes. 6.[1] Crystal Structure of $[(\eta^5\text{-}C_5Me_5)RhI]_2(\mu\text{-}I)_2\cdot 2C_6H_5Me$ and Systematics in the Series $[(\eta^5\text{-}C_5Me_5)RhX]_2$ (X = Cl, Br, I)

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Received April 3, 1979

In conjunction with studies on molecules containing bridging hydride ligands, e.g., $[(\eta^5\text{-}C_5Me_5)RhCl]_2(\mu\text{-}H)(\mu\text{-}Cl)$,[2] we have undertaken single-crystal X-ray diffraction studies on the parent rhodium(III) dihalides, $[(\eta^5\text{-}C_5Me_5)RhX]_2(\mu\text{-}X)_2$ (X = Cl, Br, I). Details concerning the chloride[3] and bromide[4] species have appeared previously; for completeness, we now report the results of a structural study on the final member of this series, $[(\eta^5\text{-}C_5Me_5)RhI]_2(\mu\text{-}I)_2$.

Table I. Crystal Data for $[(\eta^5\text{-}C_5Me_5)RhI]_2(\mu\text{-}I)_2\cdot 2C_6H_5Me$

A. Cell Parameters at 24 °C[a]

crystal system: monoclinic	$a = 13.9414\ (18)$ Å
space group: $C2/m\,[C_{2h}^3$; No. 12]	$b = 11.2421\ (14)$ Å
$Z = 2$	$c = 11.9675\ (15)$ Å
mol wt 1168.18	$\beta = 90.406\ (10)°$
ρ(calcd) = 2.068 g cm^{-3}	$V = 1875.6\ (4)$ Å^3

B. Collection of Intensity Data

diffractometer: Syntex $P2_1$
radiation: Mo Kα (λ 0.710 73 Å)
monochromator: pyrolitic graphite; equatorial
reflctns measd: $+h,\pm k,\pm l$ (two forms)
2θ range: 4.0–45.0°
scan type: coupled θ(crystal)–2θ(counter)
scan speed: 3.0 °/min in 2θ
scan range: $[2\theta(K\alpha_1) - 1.0]°$ to $[2\theta(K\alpha_2) + 1.0]°$
bkgd: stationary crystal and counter at beginning and end of 2θ scan; each for half of total scan time
standards: 3 every 97 reflections; no measurable decay
reflctns collected: 2589 total, yielding 1311 independent
data averaging: $R(I) = 2.27\%$ (after correction for absorption) for 1174 data having two or more contributions
absn coeff: $\mu = 41.3$ cm^{-1}
absn correction: empirical, based on ψ scans of 8 reflections[b]

[a] Based on 24 reflections with $27° < 2\theta < 29°$, well dispersed in reciprocal space. [b] Reflections used, their 2θ values, and their maximum:minimum transmission ratios were as follows: $3\bar{5}1$ and $\bar{3}5\bar{1}$, 20.5°, 1.12:1; 371 and $\bar{3}7\bar{1}$, 27.3°, 1.15:1; 591 and $\bar{5}9\bar{1}$, 36.4°, 1.14:1; $6,\overline{10},1$ and $\bar{6},10,\bar{1}$, 41.2°, 1.14:1.

Experimental Section

The complex was prepared by metathesis of $[(\eta^5\text{-}C_5Me_5)RhCl]_2(\mu\text{-}Cl)_2$ with sodium iodide in acetone. Attempts to obtain crystals from acetone solution yielded mainly thin plates; the material was finally recrystallized from toluene. A crystal of approximate dimensions 0.3 × 0.2 × 0.15 mm was mounted along its extended dimension (*b*) and transferred to our Syntex $P2_1$ diffractometer. Crystal alignment, determination of cell parameters, and data collection were carried out as described previously.[5] Details are given in Table I.

All computations were performed by using our Syntex XTL system.[6] The structure was solved via the multiple tangent formula method, using MULTAN,[7] which revealed the $Rh_2I_2(\mu\text{-}I)_2$ core of the molecule. A difference Fourier synthesis revealed all nonhydrogen atoms (including a toluene of crystallization). Refinement and a further difference Fourier synthesis led to the location of all hydrogen atoms in the structure. Continued full-matrix least-squares refinement of positional parameters for all atoms, anisotropic thermal parameters for nonhydrogen atoms, and isotropic thermal parameters for the hydrogen atoms led smoothly to convergence with $R_F = 2.0\%$, $R_{wF} = 2.2\%$, and GOF = 1.074 for all 1311 independent reflections ($R_F = 1.8\%$ and $R_{wF} = 2.1\%$ for those 1221 reflections with $|F_o| > 3\sigma(F_o)$). The secondary extinction parameter, *k*, was 3.327×10^{-5}; this modifies $|F_o|$ as shown in eq 1. The greatest feature on a final difference Fourier

$$|F_o|^{\mathrm{cor}} = |F_o|^{\mathrm{uncor}}(1.0 + kI_o) \qquad (1)$$

synthesis was of height 0.46 e Å^{-3} and was close to a heavy-atom position; the structure is thus complete. Final positional and thermal parameters are collected in Table II.

Discussion

The $[(\eta^5\text{-}C_5Me_5)RhI]_2(\mu\text{-}I)_2$ molecule is centered on the position (1/2, 0, 0) and has crystallographically imposed C_{2h} $(2/m)$ symmetry. Atoms I(T), Rh, C(1), C(4), and H(4B)

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Table II. Final Positional and Thermal Parameters for $[(\eta^5\text{-}C_5Me_5)RhI]_2(\mu\text{-}I)_2\cdot 2C_6H_5Me$

atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Rh	0.56258 (2)	0.00000 (0)	0.15700 (3)	2.77 (2)	2.33 (2)	1.81 (2)	0.0000 (0)	−0.14 (1)	0.0000 (0)
I(T)	0.39340 (2)	0.00000 (0)	0.26641 (3)	3.28 (2)	5.04 (2)	3.02 (2)	0.0000 (0)	0.51 (1)	0.0000 (0)
I(B)	0.50000 (0)	0.16030 (3)	0.00000 (0)	4.68 (2)	2.33 (1)	2.47 (1)	0.0000 (0)	−0.89 (1)	0.0000 (0)
C(1)	0.7164 (3)	0.00000 (0)	0.1340 (4)	2.9 (2)	3.7 (2)	2.9 (2)	0.0000 (0)	−0.1 (2)	0.0000 (0)
C(2)	0.6908 (2)	−0.1023 (3)	0.1976 (3)	3.0 (1)	3.2 (1)	3.0 (1)	0.7 (1)	−0.7 (1)	−0.1 (1)
C(3)	0.6458 (2)	−0.0635 (3)	0.2982 (3)	3.0 (1)	3.3 (1)	2.5 (1)	0.0 (1)	−0.6 (1)	0.3 (1)
C(4)	0.7703 (5)	0.00000 (0)	0.0258 (6)	4.3 (3)	6.9 (4)	3.4 (3)	0.0000 (0)	1.0 (2)	0.0000 (0)
C(5)	0.7111 (4)	−0.2292 (4)	0.1683 (5)	5.3 (2)	3.6 (2)	5.3 (2)	1.5 (2)	−1.1 (2)	−0.9 (2)
C(6)	0.6152 (4)	−0.1417 (5)	0.3921 (4)	5.4 (2)	5.0 (2)	3.1 (2)	−0.5 (2)	−0.4 (2)	1.4 (2)
C(7)	0.3849 (4)	0.50000 (0)	0.4212 (5)	4.2 (3)	6.0 (3)	5.1 (3)	0.0000 (0)	−1.4 (2)	0.0000 (0)
C(8)	0.4061 (3)	0.3941 (5)	0.3673 (4)	5.4 (2)	4.7 (2)	6.5 (3)	0.3 (2)	−1.6 (2)	−0.4 (2)
C(9)	0.4467 (4)	0.3944 (6)	0.2658 (5)	6.4 (3)	7.0 (3)	6.4 (3)	1.2 (3)	−1.4 (2)	−1.4 (3)
C(10)	0.4696 (6)	0.50000 (0)	0.2136 (7)	6.2 (4)	11.0 (7)	5.5 (4)	0.0000 (0)	−1.3 (3)	0.0000 (0)
C(11)	0.3429 (6)	0.50000 (0)	0.5355 (7)	5.6 (4)	9.0 (5)	6.0 (4)	0.0000 (0)	0.2 (3)	0.0000 (0)

atom	x	y	z	B, Å^2	atom	x	y	z	B, Å^2
H(4A)	0.756 (5)	0.054 (5)	−0.012 (6)	10.4 (21)	H(6C)	0.575 (4)	−0.116 (5)	0.439 (5)	8.3 (17)
H(4B)	0.838 (8)	0.0000 (0)	0.042 (9)	12.0 (32)	H(8)	0.379 (3)	0.323 (4)	0.393 (4)	5.9 (11)
H(5A)	0.665 (3)	−0.287 (4)	0.195 (4)	5.3 (10)	H(9)	0.466 (3)	0.328 (4)	0.236 (4)	6.5 (13)
H(5B)	0.769 (3)	−0.253 (4)	0.195 (4)	6.4 (12)	H(10)	0.512 (3)	0.50000 (0)	0.141 (4)	2.8 (9)
H(5C)	0.703 (4)	−0.249 (5)	0.094 (5)	7.6 (14)	H(11A)	0.305 (6)	0.425 (8)	0.531 (8)	17.3 (32)
H(6A)	0.675 (7)	−0.192 (8)	0.424 (7)	16.9 (30)	H(11B)	0.397 (14)	0.50000 (0)	0.582 (16)	24.7 (79)
H(6B)	0.590 (4)	−0.203 (6)	0.369 (5)	9.0 (18)					

[a] The anisotropic thermal parameters have units of Å^2 and enter the expression for the calculated structure factor in the form $\exp[-0.25(h^2a^{*2}B_{11} + \ldots + 2hka^*b^*B_{12} + \ldots)]$.

Table III. Selected Distances (in Å)

A. Within Dimeric Molecule			
Rh···Rh′	4.132 (0)	Rh-I(T)	2.706 (0)
I(B)···I(B)′	3.604 (0)	Rh-I(B)	2.741 (0)
Rh-C(1)	2.164 (5)	Rh···C(4)	3.304 (7)
Rh-C(2)	2.177 (3)	Rh···C(5)	3.307 (5)
Rh-C(3)	2.164 (3)	Rh···C(5)	3.311 (5)
C(1)-C(2)	1.426 (4)	C(1)-C(4)	1.502 (9)
C(2)-C(3)	1.429 (4)	C(2)-C(5)	1.497 (6)
C(3)-C(3)′	1.428 (5)	C(3)-C(6)	1.492 (6)
B. Within Toluene Molecule			
C(7)-C(8)	1.386 (6)	C(7)-C(11)	1.491 (11)
C(8)-C(9)	1.344 (8)		
C(9)-C(10)	1.380 (8)		

Table IV. Selected Angles (in deg)

A. Within Dimeric Molecule			
Rh-I(B)-Rh′	97.80 (1)	Rh′···Rh-I(B)	41.10 (1)
I(B)-Rh-I(B)′	82.20 (1)	I(B)-Rh-I(T)	93.29 (1)
C(2)-C(1)-C(2)′	107.5 (3)	C(2)-C(1)-C(4)	126.1 (4)
C(1)-C(2)-C(3)	108.5 (3)	C(1)-C(2)-C(5)	126.6 (4)
C(2)-C(3)-C(3)′	107.8 (3)	C(3)-C(2)-C(5)	124.9 (3)
		C(2)-C(3)-C(6)	125.8 (3)
		C(3)′-C(3)-C(6)	126.1 (3)
B. Within Toluene Molecule			
C(8)′-C(7)-C(8)	118.3 (5)	C(8)-C(7)-C(11)	120.9 (5)
C(7)-C(8)-C(9)	120.8 (5)		
C(8)-C(9)-C(10)	120.7 (6)		
C(9)-C(10)-C(9)′	118.7 (6)		

lie in the mirror plane at $y = 0$, while atoms I(B) and the related I(B)′ lie on the twofold axis at $x = {}^1/_2$ and $z = 0$. The toluene of crystallization is also bisected by a mirror plane, with atoms C(7), C(10), C(11), H(10), and H(11B) lying on the mirror plane at $y = {}^1/_2$. (There is, possibly, some slight disorder or high thermal libration of the methyl hydrogens on C(11), as is evidenced by their large thermal parameters.) The overall stoichiometry is therefore $[(\eta^5\text{-}C_5Me_5)RhI]_2(\mu\text{-}I)_2\cdot 2C_6H_5Me$.

Distances, angles, and planes are given in Tables III, IV, and V, respectively. All important dimensional trends in the series $[(\eta^5\text{-}C_5Me_5)RhX]_2(\mu\text{-}X)_2$ (X = Cl → Br → I) are indicated in Table VI. Noteworthy are the following points.

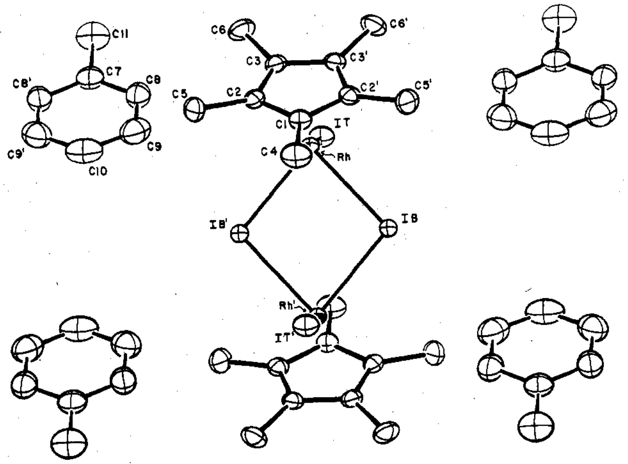

Figure 1. A portion of the crystal structure, showing the $[(\eta^5\text{-}C_5Me_5)RhI]_2(\mu\text{-}I)_2$ molecule surrounded by toluene molecules of crystallization. Hydrogen atoms have been omitted for the sake of clarity (ORTEP-II diagram).

Table V. Planes within the Dimeric Molecule

atom	dev, Å	atom	dev, Å
I. Rh(μ-I)$_2$Rh Plane ($0.9094X - 0.4159Z = 6.3393$)[a]			
I(T)	−2.6979 (3)		
II. Cyclopentadienyl Plane ($0.8876X + 0.0235Y + 0.4600Z = 9.5933$)[a]			
Rh	+1.7792 (3)	C(4)	−0.079 (8)
		C(5)	−0.059 (5)
		C(6)	−0.111 (5)
Dihedral Angle: I/II = 128.0°			

[a] Cartesian (Å) coordinates.

(1) The rhodium···rhodium distance increases incrementally, as expected for an "electron precise" dimeric molecule. These values contrast markedly with the species $[(\eta^5\text{-}C_5Me_5)RhCl]_2(\mu\text{-}H)(\mu\text{-}Cl)$ in which the (bonding) rhodium–rhodium distance is reduced to 2.906 (1) Å.[2]

(2) The bonding of the $\eta^5\text{-}C_5Me_5$ ligand to the rhodium(III) atom is strongest for the chloride, intermediate for the bromide, and weakest for the iodide—as indicated by Rh–C(ring), Rh···C(Me), and Rh–Cp distances.

Table VI. Important Intramolecular Distances (Å) and Angles (deg) with Esd's[a] for the Species $[(\eta^5\text{-}C_5Me_5)RhX]_2(\mu\text{-}X)_2$ (X = Cl, Br, I)

	$[RhCl]_2$-$(\mu\text{-}Cl)_2$[b]	$[RhBr]_2$-$(\mu\text{-}Br)_2$[c]	$[RhI]_2$-$(\mu\text{-}I)_2$
Rh···Rh	3.719 (1)	3.848 [9]	4.132 (0)
Rh–X(T)	2.397 (1)	2.528 [6]	2.706 (0)
Rh–X(B)	2.459 [9]	2.576 [9]	2.741 (0)
X···X	3.217 (2)	3.427 [15]	3.604 (0)
Rh–X(B)–Rh′	98.29 (3)	96.62 [11]	97.80 (1)
X(B)–Rh–X(B)′	81.71 (3)	83.38 (3)	82.20 (1)
X(B)–Rh–X(T)	91.51 [111]	91.16 [88]	93.29 (1)
Rh–C(ring)	2.126 [11]	2.146 [15]	2.168 [8]
Rh–Cp[d]	1.7558 (3)	1.7691 [3]	1.7792 (3)
Rh···C(Me)	3.256 [24]	3.280 [24]	3.307 [4]
r(X)[e]	0.99	1.14	1.33

[a] Esd's on individual distances or angles are enclosed in parentheses. Esd's on average distances, enclosed in brackets, are calculated by using the "scatter formula" $[\sigma] = [\Sigma(d_i - \bar{d})^2/(N-1)]^{1/2}$. Here d_i is the ith of N equivalent distances and $\bar{d}$ is the average distance. Note that this result provides the "scatter" of values about the average value–i.e., it is an external estimate of the esd on an individual value. The precision of determination of the average value is given by $[\Sigma(d_i - \bar{d})^2/N(N-1)]^{1/2}$. [b] See ref 3. [c] See ref 4. [d] Rh–Cp is the perpendicular distance from the rhodium atom to the pentaatomic carbocyclic ring. [e] These are the covalent radii, taken from: Pauling, L. "The Nature of the Chemical Bond", 3rd ed.; Cornell University Press: Ithaca, N.Y., 1960; p 224.

(3) The suggested covalent radius for Rh(III), as a mean of $\{d(\text{Rh–X(T)}) - r(\text{X})\}$ is 1.39 Å.

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Registry No. $[(\eta^5\text{-}C_5Me_5)RhI]_2(\mu\text{-}I)_2 \cdot 2C_6H_5Me$, 71049-91-7.

Supplementary Material Available: A table of data-processing formulas and a list of structure factor amplitudes (7 pages). Ordering information is given on any current masthead page.

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Macrocyclic Ligand Synthesis. Isolation of a Dicarbinolamine Complex from a Zinc(II)-Promoted Cyclization Reaction

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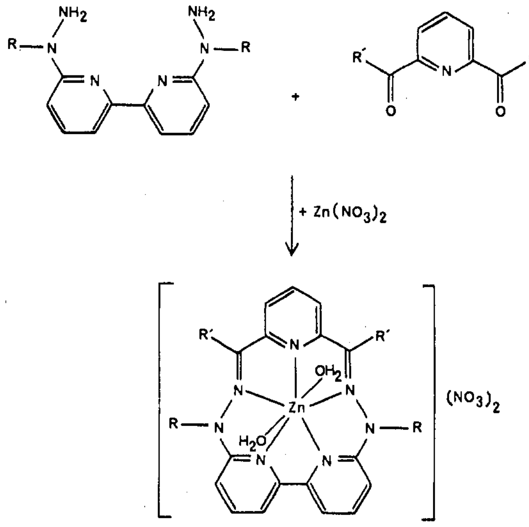
Metal ion "template" assistance in the synthesis of macrocyclic ligands is well documented.[1] Many such syntheses involve the condensation of primary amines with carbonyl-containing functional groups to give macrocyclic imines in which the "template" ion is found coordinated to the imine. Such reactions are presumed to proceed via carbinolamine intermediates,[2] but little is known of the mode of coordination of such intermediates with the metal ion "template".

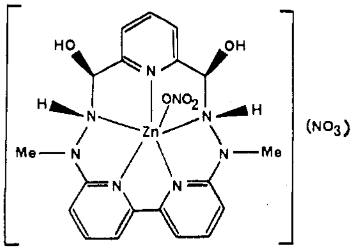
We report here the isolation of a zinc complex of a dicarbinolamine which appears to be an intermediate in the formation of the macrocyclic complex **1a**. It has been demonstrated previously[3] that the macrocycle in **1b** presents a very nearly planar "N_5" donor set to the complexed zinc(II) ion. NMR data[4] and anomalous N–N bond lengths for this compound suggested extensive π delocalization in the macrocycle and led us to attempt the structure determination of the N-methylated compound **1a**. An attempt to grow crystals of **1a** by allowing the reaction to proceed in a large volume of ethanol at room temperature resulted in yellow prisms of {2,6-dihydroxy-8,15-dimethyltripyrido[*c*,*d*:*i*,*j*:*l*,*m*][1,4,7,8,10,13,15]heptaaza[1,2,6,7,8,15]hexahydrocyclopentadecine-$N^1,N^{2b},N^7,N^{8b},N^{12b}$}nitratozinc(II) nitrate (**2**). Longer reaction times and reducing the volume of solvent give in addition to **2** a second product which is assumed to be **1a** on the basis of analytical data[5] and a comparison of IR spectral data with those of the previously characterized complexes **1b** and **1c**.



(1a) R = Me R′ = H

(1b) R = H R′ = Me

(1c) R = H R′ = H



(2)

An X-ray structure determination of **2** was accomplished by using 3185 independent reflections with $I/\sigma(I) \geq 3$ and $3 < \theta < 27°$ obtained on a Philips PW1100 automatic diffractometer with graphite-monochromatized Mo Kα radiation (λ 0.71069 Å). Crystals of **2** ($ZnC_{19}H_{21}N_9O_8$, mol wt 568.8) were triclinic with space group $P\bar{1}$, a = 12.015 (3) Å, b = 10.646 (2) Å, c = 10.434 (2) Å, α = 108.23 (2)°, β = 113.04 (2)°, γ = 78.92 (2)°, U = 1163 Å^3, D_m = 1.60 g cm^{-3}, D_c = 1.62 g cm^{-3}, and Z = 2. Full-matrix least-squares refinement[6] of atomic parameters (Zn, nitrate, and coordinated N atoms anisotropic) gave R_1 = 0.0580 and R_2 = 0.0584.