Low Symmetry in the Crystal Structure of (Octapentylphthalocyaninato)(methyl)rhodium(III)[§]

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Summary: The X-ray structure of (octa-n-pentylphthalocyaninato)(methyl)rhodium(III) is reported. Out-of-plane twisting of only one of the four isoindole groups in the molecule combined with a saddle-shaped distortion from planarity results in an unusual structure of low symmetry.

Metallophthalocyanines have been used as catalysts for a variety of oxidation and reduction processes.[1,2] Our own interest in them stems from their extreme thermal stabilities that are demonstrated for several cases by their ability to survive sublimation at temperatures as high as 500 °C.[3,4] Metallophthalocyanines are readily solubilized by substitution at the 4,7 positions of the four isoindole groups in the macrocycle, and several synthetic procedures have been devised for this purpose.[5-7] This article describes some unusual structural effects that accompany substitution at these positions. The methylrhodium complex selected for study is also of interest due to its occurrence in a methane activation process that we have been investigating.[8] The unusual structure is compared with those of related phthalocyanines and porphyrins including (octaethylporphyrinato)(methyl)rhodium(III), which is also involved in hydrocarbon activation.[9,10]

Experimental Section

Synthesis. (1,4,8,11,15,18,22,25-Octa-*n*-pentylphthalocyaninato)bis(methanol)rhodium(III) chloride[11] (870 mg, 0.68 mmol) was dissolved in $CHCl_3$ (20 mL). Removal of the solvent under vacuum left a methanol-free residue, tentatively identified as (octa-*n*-pentylphthalocyaninato)(chloro)rhodium(III). The residue was dissolved in deaerated cyclohexane (200 mL), and this solution was titrated with a diethyl ether solution of methyl lithium (1.4 M). Water (0.2 mL) was added, and the solvent was removed by vacuum distillation. The crude product was chromatographed on a basic alumina/$CHCl_3$ column. The eluent was stripped of the solvent and the product recrystallized from 1-butanol (15 mL). Yield: 580 mg. Anal. Calc for $C_{73}H_{99}N_8Rh$: C, 73.58; H, 8.37; N, 9.40; Rh, 8.64. Found: C, 74.60; H, 8.39; N, 9.42; Rh, 8.44. ^1H NMR (300 MHz, $CDCl_3$): CH_3–Rh, δ −5.45, d, J_{Rh-H} 2.1 Hz, 3H; H_{Ar}, δ 7.41, s, 8H; $H_{\alpha1}$, δ 4.378, dt, $J_{\alpha2-\alpha1}$ 14.7 Hz, $J_{\beta-\alpha1}$ 8.2 Hz, 8H; $H_{\alpha2}$, δ 3.901, dt, $J_{\beta-\alpha2}$ 6.6 Hz, 8H; H_β, δ 1.7–2.2, mult, 16H; $H_\gamma + H_\delta$, overlapping, δ 1.2–1.7, mult, 32H; H_{Me}, δ 0.891, t, J_{H-H} 7.2 Hz, 24H. Owing to changes in aggregation of the complex with molarity, the chemical shifts are slightly concentration dependent.

Table I. Crystal Data Collection and Refinement Parameters

formula	$C_{73}H_{99}N_8Rh$
fw	1191.54
color	reddish purple
lattice type	monoclinic
space group	$P2_1/n$
a, Å	14.704(3)
b, Å	25.872(5)
c, Å	17.264(3)
β, deg	108.11(0)
V, Å^3	6242.28
Z	4
d_{calc}, g cm^{-3}	1.268
μ, cm^{-1}	3.157
λ, Å	0.710 69
cryst dim, mm	0.25 × 0.30 × 0.30
collectn temp, °C	−155
2θ range, deg	6–45
scan speed, deg min^{-1}	8.0
aperture size, mm	3.0 × 4.0
no. of rflns read	10 502
no. of unique rflns obsd	8145
$R(F)$, %	6.74
$R_w(F)$, %	6.97
GOF	1.326
max Δ/σ, e Å^{-3}	0.05
structure soln	MULTAN78

X-ray Structure Determination. Sample deposited from the 1-butanol solution consisted of well-formed crystals of varying size and morphology. A typical crystal was chosen for study. This crystal was affixed to the end of a glass fiber using silicone grease, was transferred to the Picker four-circle goniometer of a diffractometer designed and constructed at the Molecular Structure Center at Indiana University, and was cooled to −155 °C for characterization and data collection. There was no apparent phase transition upon cooling. A systematic search of a limited hemisphere of reciprocal space located a set of diffraction maxima with symmetry and systematic absences corresponding to the unique monoclinic space group $P2_1/n$ (an alternative setting of $P2_1/c$). Subsequent solution and refinement confirmed this choice. Final cell parameters and other crystallographic data are summarized in Table I. Reflections were collected using a continuous θ–2θ scan with fixed background counts at each extreme of the scan. The data were corrected for Lorentz and polarization effects, and equivalent reflections were averaged to yield a unique set of intensities and estimated errors (esd's are based on counting statistics and standard propagation of errors).

The Rh atom and 20 other non-hydrogen atoms were clearly visible in a map generated using phases derived by direct methods (MULTAN78), and all other non-hydrogen atoms were located

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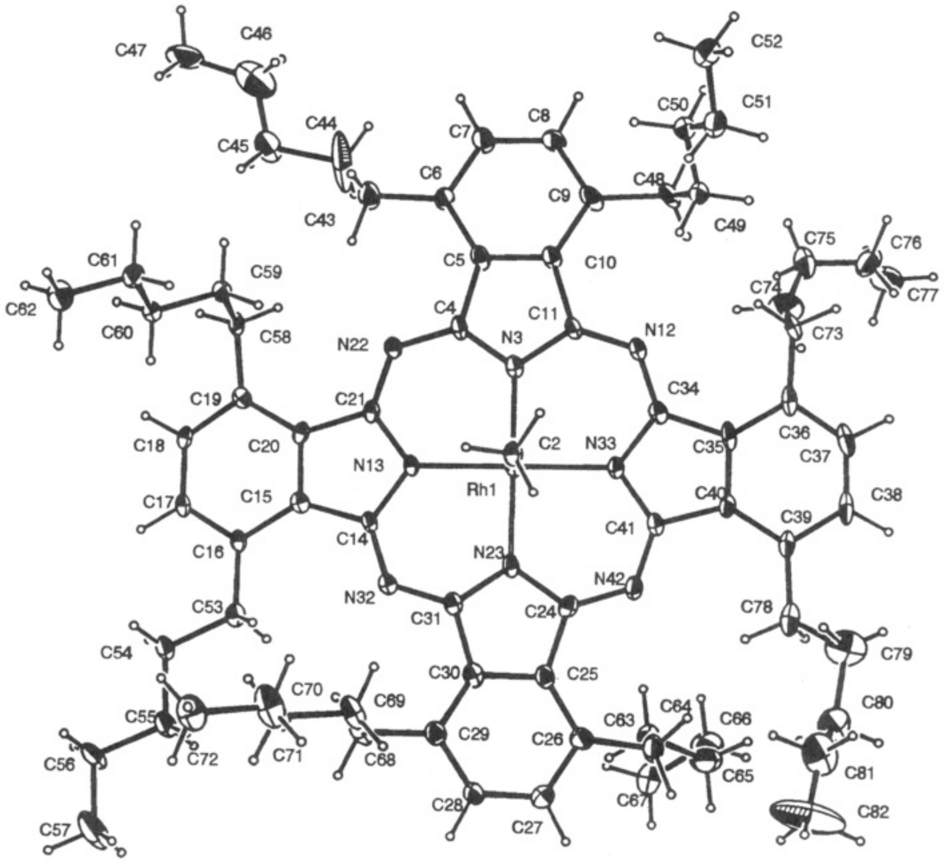

Figure 1. Ortep drawing of (octa-*n*-pentylphthalocyaninato)(methyl)rhodium(III) showing the atom numbering system.

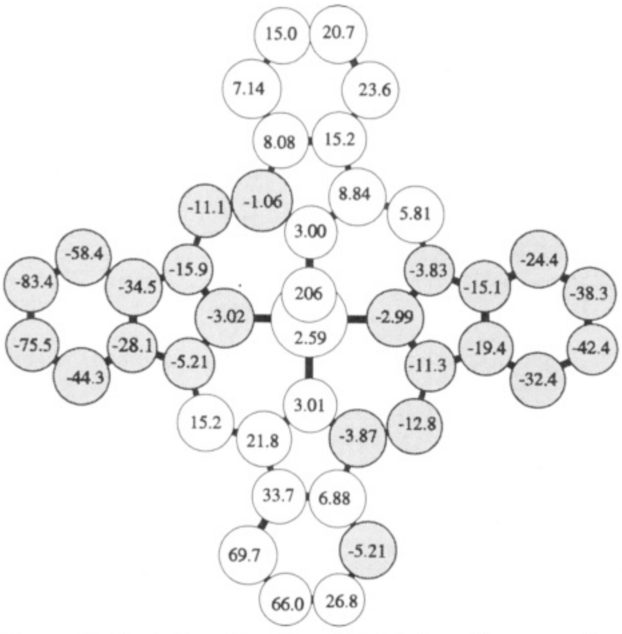

Figure 2. Deviations, in units of 0.01 Å, from the mean plane of the four inner nitrogen atoms, which is expressed by the equation: $7.312X + 22.01Y + 0.1151Z = 12.85$. Shaded atoms lie below this plane.

in successive Fourier maps. The atomic numbering system and final structure are shown in Figure 1. Two of the *n*-C_5H_{11} groups were found to be disordered, and in general, all show large thermal motion. The disorder was readily modeled by using two alternate positions for C(79) in one *n*-C_5H_{11} group and alternate positions for C(74), C(75), C(76), and C(77) in the other disordered group. Refinement of the occupancy indicated that the disorder was essentially 50:50, so the occupancies were fixed at this value for the remainder of the refinement. The three hydrogen atoms on the methyl carbon, C(2), were clearly visible in a difference Fourier map phased on the non-hydrogen atoms, and many of the other hydrogens were present. To improve the model, all hydrogen atoms were placed in idealized fixed positions for the remainder of the refinement. Hydrogen atoms with occupancies of 0.5 were calculated for both disorder models, but only one was retained (with occupancy = 1.0) if two calculated positions were within 0.3 Å of each other. The final difference Fourier map was featureless, the largest peak being 0.74 e/Å^3. Several of the larger peaks in the final map are in the vicinity of the disordered *n*-C_5H_{11} groups, indicating that the disorder may be more complex than the simple model used. The largest negative peak was −0.53 e/Å. The closest contact between rhodium and an adjacent molecule is Rh(1)···C(18) = 2.985(8) Å. The closest internuclear Rh–Rh approach is 7.343(3) Å for two molecules related by a center of inversion. The final atomic coordinates are given in Table II.

Molecular Modeling Calculations. Molecular mechanics and pi-VESCF calculations were performed using PCM386, a version of the program PCMODEL for 386 and 486 IBM compatible computers. PCMODEL was selected for use in this study because it is fully parametrized for the types of bonds present in the phthalocyanine ligand. The program was obtained from Serena Software, Bloomington, IN, and was used without modification. Structures for (octa-*n*-pentylphthalocyaninato)(methyl)rhodium(III), octa-*n*-butylphthalocyanine, and octa-*tert*-butylphthalocyanine were obtained by using the non-hydrogen atom coordinates from the X-ray structure of the methylrhodium complex in developing the starting points for the calculations. This structure was then modified by replacement of alkyl groups and removal of the rhodium center to achieve the desired phthalocyanine. Idealized geometries were used for the initial hydrogen positions in the calculations.

Results and Discussion

Bond angles and bond distances for (octapentylphthalocyaninato)(methyl)rhodium(III) are shown in Table III. Although distortions caused by the presence of the *n*-pentyl

Table II. Fractional Coordinates (×10^4) and Isotropic Thermal Parameters (×10)

atom	x	y	z	B_{iso}, Å^2	atom	x	y	z	B_{iso}, Å^2
Rh(1)	6193.0(4)	3743.8(2)	5100.2(4)	26	C(44)	4763(11)	4547(8)	8549(12)	171
C(2)	5441(6)	3072(3)	4894(5)	31	C(45)	3928(9)	4863(5)	8601(7)	77
N(3)	6319(4)	3694(2)	6286(4)	28	C(46)	3726(15)	4778(9)	9311(11)	142
C(4)	5718(5)	3910(3)	6649(5)	32	C(47)	2867(13)	5016(7)	9419(7)	108
C(5)	6011(6)	3767(4)	7513(5)	37	C(48)	8222(7)	2924(4)	8584(5)	54
C(6)	5649(7)	3888(4)	8147(5)	47	C(49)	8005(7)	2363(4)	8368(5)	48
C(7)	6147(8)	3683(5)	8911(5)	67	C(50)	7517(7)	2082(5)	8885(6)	56
C(8)	6960(8)	3386(4)	9033(5)	57	C(51)	7295(8)	1517(5)	8680(7)	61
C(9)	7326(7)	3255(4)	8411(5)	47	C(52)	6905(8)	1228(5)	9259(7)	72
C(10)	6832(6)	3461(3)	7644(5)	35	C(53)	2883(6)	4993(3)	2835(5)	31
C(11)	7028(5)	3429(3)	6853(5)	29	C(54)	2041(6)	5313(3)	2316(5)	37
N(12)	7774(4)	3195(2)	6735(4)	29	C(55)	1922(7)	5281(4)	1406(6)	46
N(13)	5016(4)	4161(2)	4919(4)	25	C(56)	1027(8)	5574(4)	889(6)	57
C(14)	4476(5)	4355(3)	4166(5)	26	C(57)	897(11)	5537(6)	8(9)	111
C(15)	3753(5)	4698(3)	4297(5)	26	C(58)	3250(5)	4941(3)	6325(5)	32
C(16)	3046(5)	5009(3)	3741(5)	27	C(59)	2607(6)	4485(3)	6406(5)	35
C(17)	2524(5)	5323(3)	4097(5)	27	C(60)	1555(6)	4560(3)	5912(5)	34
C(18)	2639(5)	5316(3)	4921(5)	27	C(61)	912(6)	4136(4)	6025(6)	43
C(19)	3277(5)	4988(3)	5472(5)	27	C(62)	−131(7)	4233(4)	5547(7)	55
C(20)	3856(5)	4689(3)	5134(5)	27	C(63)	7800(8)	3542(6)	2171(6)	81
C(21)	4652(5)	4338(3)	5504(5)	27	C(64)	8386(7)	4074(4)	2529(6)	53
N(22)	4956(4)	4211(2)	6297(4)	27	C(65)	9357(9)	4097(7)	2471(8)	97
N(23)	6060(4)	3792(2)	3923(4)	29	C(66)	9953(9)	4540(6)	2857(8)	85
C(24)	6712(6)	3609(3)	3564(5)	30	C(67)	9504(10)	5042(6)	2422(9)	91
C(25)	6325(6)	3693(3)	2684(5)	36	C(68)	3715(7)	4057(4)	1532(6)	46
C(26)	6729(7)	3617(4)	2055(5)	46	C(69)	3116(7)	3571(4)	1529(6)	55
C(27)	6097(8)	3686(5)	1266(6)	61	C(70)	2045(7)	3663(4)	1263(6)	51
C(28)	5153(7)	3822(4)	1117(5)	54	C(71)	1461(8)	3170(4)	1152(8)	76
C(29)	4756(6)	3934(3)	1728(5)	39	C(72)	391(8)	3254(4)	849(7)	64
C(30)	5390(6)	3882(3)	2524(5)	32	C(73)	9776(7)	2669(4)	7385(6)	58
C(31)	5271(5)	3972(3)	3330(5)	29	C(74)	10299(9)	3129(7)	7885(7)	100
N(32)	4553(4)	4240(2)	3442(4)	25	C(75)	10955(34)	3313(22)	8655(23)	229
N(33)	7419(4)	3361(2)	5302(4)	28	C(76)	11828(24)	3103(10)	8839(19)	90
C(34)	7986(5)	3173(3)	6041(5)	30	C(77)	12684(28)	3506(15)	8980(16)	120
C(35)	8840(6)	2941(3)	5926(6)	41	C(78)	9454(9)	2852(5)	3921(8)	75
C(36)	9643(7)	2713(4)	6490(7)	52	C(79)	9693(24)	2355(10)	3556(18)	94
C(37)	10334(9)	2548(5)	6163(8)	80	C(80)	9416(9)	2397(5)	2633(8)	73
C(38)	10270(8)	2593(5)	5361(8)	85	C(81)	8686(11)	2064(5)	2077(10)	93
C(39)	9480(7)	2813(4)	4792(7)	60	C(82)	8421(19)	2100(7)	1324(10)	187
C(40)	8759(6)	2991(3)	5098(5)	37	C(75)′	11289(14)	3122(10)	7912(15)	78
C(41)	7855(5)	3257(3)	4721(5)	32	C(76)′	11927(16)	3464(11)	8432(15)	76
N(42)	7357(5)	3373(3)	3934(4)	35	C(77)′	12151(29)	3286(20)	9385(28)	206
C(43)	4810(9)	4224(6)	8058(6)	80	C(79)′	9020(10)	2477(6)	3441(14)	50

groups are readily evident near the points of substitution, the geometry near the rhodium center is reasonably unaffected. Coordination at rhodium is almost rigorously square-based pyramidal, with the four angles formed by adjacent inner nitrogen atoms and the rhodium center averaging 90.0°. The angles formed by the inner nitrogen atoms, the rhodium center, and the methyl carbon at the apex of the pyramid are also each close to 90°. The methyl carbon-to-rhodium distance, 2.031(8) Å, is unusually short when compared with that (2.081 Å) observed in the more usual square planar Rh(III) complex[12] but identical to the value found in the structurally related (octaethylporphyrinato)(methyl)rhodium(III).[13] However, because of the smaller coordination cavity within phthalocyanines relative to that in porphyrins,[14] the Rh to coordinated N distances, which average 1.989 Å in the phthalocyanine complex, are on average 0.042 Å longer in (octaethylporphyrinato)(methyl)rhodium(III). This difference is close to that, 0.046–0.05 Å, expected on the basis of the difference in cavity sizes for typical phthalocyanines and porphyrins.[14] The Rh-to-N distances in Table III are close to those for Rh(III) complexes of the unsubstituted phthalocyanine ligand, which average 1.980 and 1.978 Å in

Table III. Selected Bond Distances and Bond Angles for (Octapentylphthalocyaninato)(methyl)rhodium(III)

A	B	distance (A–B), Å
Rh(1)	N(3)	2.001(6)
Rh(1)	N(13)	1.980(6)
Rh(1)	N(23)	1.985(6)
Rh(1)	N(33)	1.990(6)
Rh(1)	C(2)	2.031(8)

A	B	C	angle (A–B–C), deg
N(3)	Rh(1)	N(13)	89.75(25)
N(3)	Rh(1)	N(33)	89.90(25)
N(13)	Rh(1)	N(23)	90.02(24)
N(23)	Rh(1)	N(33)	90.34(25)
N(3)	Rh(1)	N(23)	179.66(25)
N(13)	Rh(1)	N(23)	176.75(26)
N(3)	Rh(1)	C(2)	89.8(3)
N(13)	Rh(1)	C(2)	92.1(3)
N(23)	Rh(1)	C(2)	90.0(3)
N(33)	Rh(1)	C(2)	91.1(3)
N(22)	Rh(1)	N(42)	175
N(12)	Rh(1)	N(32)	177.35

A	B	C	D	angle[a] (A–B–C–D), deg
N(13)	N(33)	C(26)	C(29)	−15.03
N(3)	N(23)	C(15)	C(20)	2.78
N(33)	N(13)	C(6)	C(9)	−3.29
N(23)	N(3)	C(36)	C(39)	1.65

[a] Dihedral angle, a negative sign indicates that CD is rotated counterclockwise from AB when viewed along the axis, BC, from B to C.

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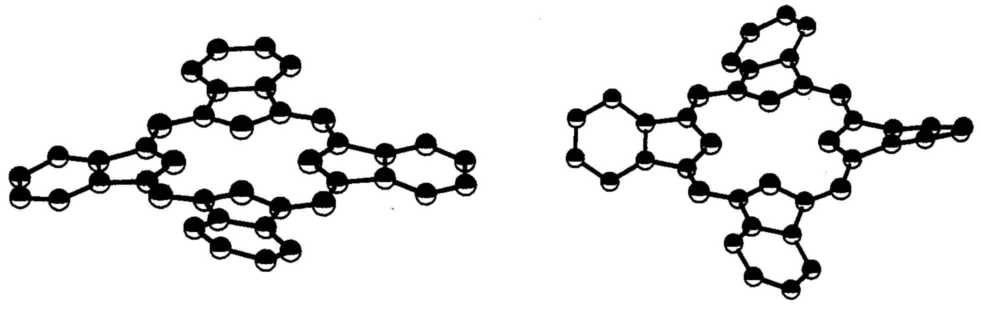

a) b)

Figure 3. (a) X-ray structure of (octa-*n*-pentylphthalocyaninato)(methyl)rhodium(III) showing only the core atoms. (b) Calculated core structure for octa-*tert*-butylphthalocyanine. Both structures are rotated 45° on a horizontal axis parallel to the plane of the paper from the orientation shown in Figures 1 and 2.

chlorodimethylaminerhodium(III) phthalocyanine and chloroaquorhodium(III) phthalocyanine, respectively.[15]

Core geometry distortion, i.e., ruffling, in metalloporphyrins leads to two conformational types, termed *ruf* and *sad*.[16] The *ruf* conformation is characterized by the angles created by the metal center and opposite methyne carbons in the porphyrin ligand. In a severely ruffled complex of the *ruf* type, these angles, normally 180°, can be as low as the 134.7° measured in (tetrapropylporphyrinato)lead(II).[17] The analogous angles in phthalocyanines are those formed by the metal center and the outer nitrogens in the ligand. As shown for the methylrhodium complex in Table III, these angles are 175 and 177°, indicating that the *ruf* type distortion is effectively absent. The *sad* conformational type distortion occurs frequently in tetraphenylporphyrin complexes and results in saddle-shaped molecules, with adjacent pyrrole moieties in the ligand forced alternately above and below the mean plane of the complex. As is evident by inspection of Figure 2, which shows the displacements of the core atoms in (octapentylphthalocyaninato)(methyl)rhodium(III) from the mean plane formed by the inner nitrogen atoms of the ligand, this molecule is distorted into the saddle conformation. In order to compare the magnitude of the distortion with that in porphyrin complexes, we computed the average of the absolute value of the deviations of the β pyrrole carbons from the mean plane formed by the metal center and 24 of the core atoms in the phthalocyanine ligand that correspond to the 24 core atoms in the porphyrin ligand. This value, 0.208 Å, is comparable to that measured in several tetraphenylporphyrin complexes, e.g., the value for bis(1-methylimidazole)(mesotetraphenylporphyrinato)iron(III) perchlorate is 0.23 Å.[18]

The most unusual feature in the structure of (octa-*n*-pentylphthalocyaninato)(methyl)rhodium(III) is that the presence of the *n*-pentyl groups causes one of the isoindole groups in the molecule (the one terminating in N(23)) to be twisted out of the plane of the macrocycle by −15.0°, while the remaining three isoindole groups are twisted by an average absolute value of only 2.6°. These values were obtained by measuring the dihedral angles between *trans* inner nitrogen atoms and the ring carbons that are attached to the alkyl groups. The appropriate atoms and the measured dihedral angles are given in Table III. The twisting seems unusual in that it does not occur more symmetrically in all four of the isoindole moieties. As shown in Figure 3a, which has all but the core atoms of the complex removed, the twisting is more apparent than the more normal distortion that leads to the saddle shape of the molecule. Presumably, the strain induced by nonbonded interactions of neighboring *n*-pentyl groups can be relieved for the entire molecule by out-of-plane twisting of only one isoindole group (in combination with *sad* distortion of the phthalocyanine core and folding of the pentyl groups away from each other). The necessity to twist only one of the isoindole groups allows the π-stabilizations of the other three to be retained as much as possible.

Table IV. Calculated Dihedral Angles and Heats of Formation of Phthalocyanines

molecule[a]	dihedral angle, deg[b]	$\Delta H_f°$, kcal/mol
A	−15.9	
	0.29	
	−2.03	
	−5.83	
B	−14.31	155
	1.23	
	−4.36	
	0.17	
C	−30.18	199
	−28.64	
	−23.45	
	−26.72	

[a] A: (octa-*n*-pentylphthalocyaninato)(methyl)rhodium(III). B: octa-*n*-butylphthalocyanine. C: octa-*tert*-butylphthalocyanine. [b] The dihedral angles for each structure are presented in the same order as those in Table III.

The molecular modeling program, PCMODEL, is fully parameterized for the various types of bonds in the phthalocyanine ligand and reproduces the twisting of the isoindole groups in (octa-*n*-pentylphthalocyaninato)-(methyl)rhodium(III) with fair accuracy. From Table IV, the calculated values for the large angle, −15.9°, and the average of the absolute values of the smaller angles, 2.7°, agree well with the aforementioned X-ray results. The *sad* distortion was also, at least qualitatively, reproduced, although this latter feature was not quantified for the calculated structure.

Also shown in Table IV are the results of calculations for the isomeric unmetalated phthalocyanines, octa-*n*-butylphthalocyanine and octa-*tert*-butylphthalocyanine. The *n*-butyl isomer has unsymmetrical twisting of the isoindole groups similar to that in the *n*-pentyl substituted

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rhodium complex. However, the *tert*-butyl derivative, shown in Figure 3b, has a more symmetrical propeller-shaped structure, with all four of the isoindole moieties twisted at large and more nearly equal angles, averaging 27.2°. Unlike the *n*-butyl and *n*-pentyl groups, the *tert*-butyl groups are not flexible enough to be bent away from each other; thus, large twists in the isoindole groups are required to accommodate them. The large twisting leads to considerable loss in stability, and the *tert*-butyl derivative is calculated to be 44 kcal/mol less stable than the *n*-butyl isomer (Table IV). Consistent with this large energy difference, octasubstituted phthalocyanines with branched substituents in the 4,7 isoindole positions that easily accommodate linear chains have never been prepared.

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Supplementary Material Available: Tables of calculated hydrogen atom positions, anisotropic thermal parameters, and bond distances and bond angles (10 pages). Ordering information is given on any current masthead page.

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