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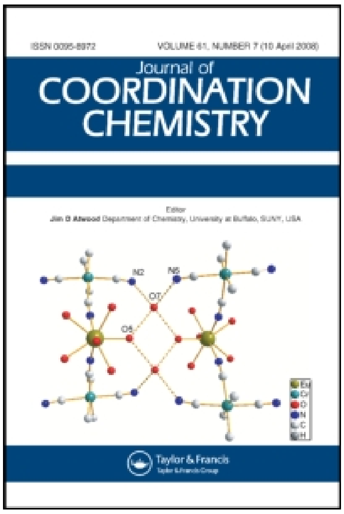

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X-RAY CRYSTAL STRUCTURE OF 6,17-DIMETHYL-8, 15-DIPHENYL-DIBENZO [b,i][1,4,8,11]TETRAAZACYCLOTETRADECINATO COPPER(II): THE MORE STERICALLY-CROWDED ISOMER

Richard P. Hotz[a]; Robert D. Bereman[b]; Suzanne T. Purrington[b]; Phirtu Singh[b]
[a] Department of Chemistry, College of Mount St. Joseph, Cincinnati, Ohio, USA [b] Department of Chemistry, North Carolina State University, Raleigh, North Carolina, USA

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X-RAY CRYSTAL STRUCTURE OF 6,17-DIMETHYL-8, 15-DIPHENYL-DIBENZO [b,i][1,4,8,11]TETRAAZACYCLOTETRADECINATO COPPER(II): THE MORE STERICALLY-CROWDED ISOMER

RICHARD P. HOTZ*

Department of Chemistry, College of Mount St. Joseph, 5701 Delhi Road, Cincinnati, Ohio 45233-1670, USA

ROBERT D. BEREMAN, SUZANNE T. PURRINGTON, and PHIRTU SINGH

Department of Chemistry, North Carolina State University, Box 8204, Raleigh, North Carolina 27695-8204, USA

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6,17-Dimethyl-8,15-diphenyldibenzo[b,i][1,4,8,11]tetraazacyclotetradecinato copper(II), [Cu(*cis*-$Me_2Ph_2Bzo_2$[14]tetraeneN_4)], was prepared by the reaction of the metal-free macrocycle with copper acetate. The compound crystallizes in the monoclinic space group $P2_1/c$ with two crystallographically independent molecules in the asymmetric unit and lattice parameters $a = 11.059(2)$Å, $b = 19.164(5)$Å, $c = 24.007(7)$Å, $\beta = 93.16°$, $V = 5080$ Å^3. Convergence to $R = 0.040$ and $R_w = 0.056$ was obtained based on 5319 unique reflections with $I > 3\sigma(I)$.

KEYWORDS: copper, macrocycle, dibenzotetraaza[14]annulene, X-ray structure

INTRODUCTION

Dibenzotetraaza[14]annulene macrocycles are synthesized by a nickel(II) template condensation between 1,2-diaminobenzene and a 1,3-diketone. The required presence of a template results in the isolation of the ligand as a metal complex. However, the nickel(II) is easily removed allowing for the isolation of the metal-free macrocycle and the preparation of other metal complexes.[1,2]

The template condensation reaction between 1,2-diaminobenzene and 1-phenyl-1,3-butanedione is regioselective. The only isomer obtained has both phenyl substituents on the same side of the molecule, the *cis* isomer. This isomer would be less favorable sterically of the two and yet, it is the only macrocyclic isomer that is produced.

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* Author for correspondence.

In order to understand the regioselectivity of this reaction, it is very important to obtain structural information on the macrocycle and its transition metal complexes. Our studies have led to the synthesis of the copper(II) complex of this ligand. In this paper, we report an X-ray structural analysis of the title compound.

EXPERIMENTAL

The title compound was prepared from the metal-free macrocycle and copper acetate by the published method[3] and crystals suitable for X-ray analysis were obtained by slow evaporation of a concentrated acetone solution. A single crystal of dimensions 0.65 × 0.40 × 0.40 mm was mounted on a thin glass fiber, secured to a goniometer head and then transferred to a Nicolet R3m/μ automated four-circle diffractometer equipped with a pulse height analyzer and a graphite monochromator. Cell constants were determined from a least-squares refinement of the setting angles of 25 reflections with 2θ values ranging from 40° to 60°. The intensity data were collected using parameters summarized in Table 1. The intensities were corrected for Lorentz and polarization effects but not for absorption. The two copper atoms in the asymmetric unit were located by the direct methods subroutine SOLV of the crystallographic computing system SHELXTL.[4] The remaining nonhydrogen atoms were located from a difference Fourier map. The structure was refined by the blocked cascade least-squares refinement method by assigning anisotropic thermal parameters to the nonhydrogen atoms. The hydrogen atoms were placed in theoretical positions 0.96Å away from the attached carbon atom and refined in the riding mode. The function minimized was $\Sigma w(|F_o|-|F_c|)^2$, where $w = 1/(\sigma^2(F) + 0.00013F^2)$. The data collection and refinement parameters are summarized in Table 1.

All calculations were performed on a Data General microeclipse desktop computer with the program package SHELXTL.[4] The X-ray atomic scattering factors and the dispersion corrections for all atoms were taken from the *International Tables X-ray Crystallography.*[5]

The final positional and equivalent isotropic thermal parameters for nonhydrogen atoms are given in Table 2. The atomic numbering scheme is shown in Figure 1.

RESULTS AND DISCUSSION

The crystal structure of 6,17-dimethyl-8,15-diphenyldibenzo [b,i][1,4,8,11]tetraazacyclotetradecinato copper(II), [Cu(*cis*-Me_2Ph_2-Bzo_2[14]tetraeneN_4)], is the first complex of the Me_2Ph_2-Bzo_2[14]tetraene ligand as well as the first copper complex of a dibenzotetraaza[14]annulene to be reported.

There are two independent molecules of the complex in the asymmetric unit. Their overall geometry, however, is quite similar as shown in Figure 2. The macrocyclic complex adopts the saddle-shape conformation that has been observed in other dibenzotetraaza[14]annulenes.[6–8] The four nitrogen atoms are coplanar and the two benzenoid rings (the rings derived from 1,2-diaminobenzene) are bent back from this plane with dihedral angles of 21.8 and 22.0° in molecule A and 24.3° and 22.7° in molecule B. The propane-1,3-diiminato chelate rings are bent to the opposite side of the N_4-plane with dihedral angles of 25.9° and 23.1° in

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Table 1 Crystal data and refinement parameters for $C_{32}H_{26}N_4Cu$.

formula	$C_{32}H_{26}N_4Cu$
formula weight	530.13
crystal size (mm)	0.65 × 0.40 × 0.40
radiation	CuKα (λ = 1.54178Å)
a(Å) = 11.059(2)	
b(Å) = 19.164(5)	
c(Å) = 24.007(7)	
β = 93.16°	
V = 5080(2)Å^3	
space group	$P2_1/c$
Z	8
Density, D_c	1.386 g cm^{-3}
F(000)	2200 e$^-$
temperature	297 K
scan type	$\omega/2$
scan range	2° + dispersion
scan speed	Variable between 4 and 29.3 deg min^{-1}
2θ range	$4 \leq 2\theta \leq 110$ deg
background	Stationary for 1/2 scan time on each side of peak
octants measured	hkl, $hk\bar{l}$
standards	2 after every 70 refl
no. measured	7206
no. used, NO	5319 with $I \geq 3\sigma(I)$
sec extinction coeff.	5.4×10^{-6}
μ	13.9 cm^{-1}
R^a	0.040
$R_w{}^b$	0.056
goodness of fit S^c	1.9
max shift/σ	1.0
no. of variables, NV	680
difference peak excursion	0.33 eÅ^{-3}

[a] $R = \Sigma(||F_0| - |F_c||)/\Sigma|F_0|$. [b] $R_w = [\Sigma_w(|F_0| - |F_c|)^2/\Sigma_w|F_0|^2]^{1/2}$. [c] $S = [\Sigma_w(|F_0| - |F_c|)^2/(NO - NV)]^{1/2}$.

molecule A and 19.4° and 23.0° in molecule B. The dihedral angle between the two diiminato planes is 48.9° (42.4° in molecule B). The Cu-N bond distances are 1.91-1.93Å and the Cu atom is displaced to the benzenoid ring side of the N_4-plane. However, the displacement in molecules A and B is only 0.061Å and 0.035Å, respectively.

Dibenzotetraaza[14]annulenes adopt a saddle-shape conformation due to congestion between substituents on the propane-1,3-diiminato framework and the benzenoid rings.[7,9] Although three benzene rings on the same side of the molecule might appear to be sterically unfavorable, the structural results indicate that the planar phenyl substituents have rotated in order to decrease interactions with the benzenoid rings (62.8° and 63.1° in molecule A and 61.1° and 64.0° in molecule B). The rotation of the phenyl rings also precludes conjugation with the propane-1,3-diiminato framework as evidenced by bond distances that are equivalent to that of single bonds. For example in molecule A, the C(26)-C(27) distance is 1.501Å and the C(7)-C(8) distance is 1.494Å. This particular configuration of the phenyl substituents relieves molecular strain but the molecule is still rather crowded with

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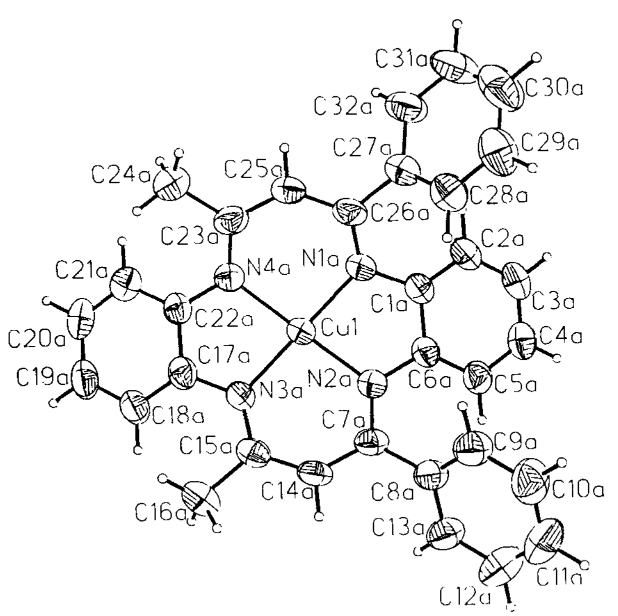

Figure 1 View of molecule A normal to the plane of atoms N(1), N(2), N(3) and N(4) showing the numbering scheme. Atoms in molecule B follow the same scheme. Thermal ellipsoids for the nonhydrogen atoms are drawn to include 50% probability density. Hydrogen atoms are shown as circles of arbitrary size.

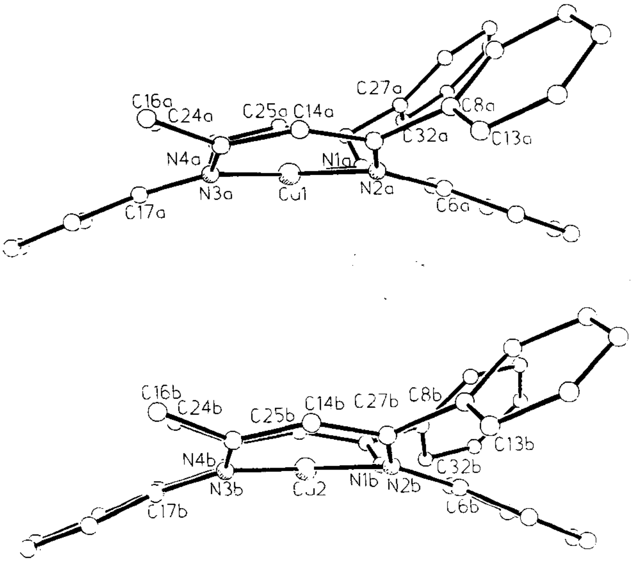

Figure 2 View of (a) molecule A, and (b) molecule B, approximately along an edge of the mean plane of the four nitrogen atoms showing the relative orientations of all the planar moities of the macrocyclic complex.

Table 2 Atomic coordinates (× 10^4) and isotropic thermal parameters ($Å^2$ × 10^3).

	x	y	z	U^a
Cu(1)	2832(1)	2854(1)	1890(1)	46(1)
N(1A)	1446(2)	2804(1)	2335(1)	49(1)
N(2A)	2527(2)	3837(1)	1902(1)	46(1)
N(3A)	4296(2)	2913(1)	1492(1)	46(1)
N(4A)	3178(2)	1865(1)	1921(1)	48(1)
C(1A)	738(2)	3415(1)	2280(1)	47(1)
C(2A)	-480(3)	3476(2)	2393(1)	53(1)
C(3A)	-1083(3)	4092(2)	2286(1)	58(1)
C(4A)	-504(3)	4646(2)	2056(1)	57(1)
C(5A)	690(3)	4595(2)	1928(1)	52(1)
C(6A)	1333(2)	3985(1)	2044(1)	45(1)
C(7A)	3421(3)	4305(1)	1863(1)	48(1)
C(8A)	3305(3)	5027(1)	2091(1)	51(1)
C(9A)	3034(3)	5130(2)	2640(1)	63(1)
C(10A)	2970(3)	5796(2)	2856(1)	78(1)
C(11A)	3165(3)	6359(2)	2525(2)	86(2)
C(12A)	3436(3)	6269(2)	1980(2)	82(1)
C(13A)	3515(3)	5606(2)	1763(1)	62(1)
C(14A)	4537(3)	4131(1)	1671(1)	51(1)
C(15A)	4967(3)	3487(2)	1494(1)	49(1)
C(16A)	6288(3)	3475(2)	1353(1)	64(1)
C(17A)	4621(2)	2254(1)	1292(1)	48(1)
C(18A)	5386(3)	2127(2)	865(1)	58(1)
C(19A)	5535(3)	1462(2)	660(1)	65(1)
C(20A)	4908(3)	917(2)	868(1)	66(1)
C(21A)	4130(3)	1023(2)	1289(1)	58(1)
C(22A)	3988(2)	1682(1)	1519(1)	47(1)
C(23A)	2845(3)	1467(1)	2340(1)	51(1)
C(24A)	3406(3)	760(2)	2461(2)	70(1)
C(25A)	2000(3)	1691(1)	2714(1)	52(1)
C(26A)	1344(2)	2312(1)	2720(1)	49(1)
C(27A)	615(3)	2425(2)	3222(1)	53(1)
C(28A)	835(3)	2994(2)	3563(1)	61(1)
C(29A)	225(3)	3079(2)	4041(1)	78(1)
C(30A)	-633(4)	2602(2)	4180(2)	96(2)
C(31A)	-867(4)	2038(2)	3842(2)	99(2)
C(32A)	-249(3)	1940(2)	3366(1)	74(1)
Cu(2)	2367(1)	1894(1)	168(1)	46(1)
N(1B)	3598(2)	1911(1)	-367(1)	45(1)
N(2B)	2575(2)	2884(1)	165(1)	46(1)
N(3B)	1104(2)	1903(1)	689(1)	48(1)
N(4B)	2089(2)	900(1)	149(1)	49(1)
C(1B)	4257(2)	2542(1)	-339(1)	45(1)
C(2B)	5423(3)	2655(2)	-512(1)	55(1)
C(3B)	5984(3)	3289(2)	-407(1)	62(1)
C(4B)	5429(3)	3812(2)	-123(1)	58(1)
C(5B)	4302(3)	3697(1)	67(1)	53(1)
C(6B)	3690(2)	3077(1)	-44(1)	44(1)
C(7B)	1688(3)	3323(1)	283(1)	47(1)
C(8B)	1691(3)	4053(1)	72(1)	50(1)
C(9B)	1819(3)	4188(2)	-483(1)	67(1)
C(10B)	1823(3)	4866(2)	-670(2)	98(2)
C(11B)	1688(3)	5409(2)	-313(2)	107(2)
C(12B)	1529(3)	5280(2)	236(2)	98(2)
C(13B)	1532(3)	4610(2)	432(2)	68(1)
C(14B)	678(3)	3110(2)	561(1)	55(1)

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Table 2 *Continued*

	x	*y*	*z*	U^a
C(15B)	383(3)	2451(2)	755(1)	52(1)
C(16B)	-847(3)	2386(2)	995(2)	77(1)
C(17B)	935(2)	1230(1)	908(1)	49(1)
C(18B)	417(3)	1089(2)	1414(1)	65(1)
C(19B)	354(3)	417(2)	1608(1)	77(1)
C(20B)	858(3)	-118(2)	1320(1)	75(1)
C(21B)	1431(3)	15(2)	834(1)	65(1)
C(22B)	1458(3)	683(1)	611(1)	50(1)
C(23B)	2381(2)	503(1)	-281(1)	50(1)
C(24B)	1857(3)	-215(2)	-383(2)	69(1)
C(25B)	3141(3)	749(1)	-685(1)	51(1)
C(26B)	3742(2)	1388(1)	-727(1)	47(1)
C(27B)	4489(3)	1477(1)	-1215(1)	48(1)
C(28B)	424(3)	2000(1)	-1603(1)	54(1)
C(29B)	4972(3)	2095(2)	-2044(1)	60(1)
C(30B)	5950(3)	1677(2)	-2100(1)	72(1)
C(31B)	6205(3)	1156(2)	-1722(1)	81(1)
C(32B)	5472(3)	1053(2)	-1286(1)	67(1)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor.

Table 3 Bond distances (Å) and selected bond angles (deg).

	Molecule A	*Molecule B*
Cu-N(1)	1.919(2)	1.924(2)
Cu-N(2)	1.915(2)	1.910(2)
Cu-N(3)	1.928(2)	1.926(2)
Cu-N(4)	1.934(2)	1.930(2)
N(1)-C(1)	1.411(3)	1.411(3)
N(1)-C(26)	1.330(3)	1.338(3)
N(2)-C(6)	1.410(4)	1.406(3)
N(2)-C(7)	1.343(3)	1.335(3)
N(3)-C(15)	1.327(4)	1.334(4)
(N(3)-C(17)	1.406(3)	1.404(4)
N(4)-C(22)	1.396(4)	1.405(4)
N(4)-C(23)	1.332(4)	1.336(4)
C(1)-C(2)	1.394(4)	1.394(4)
C(1)-C(6)	1.412(4)	1.412(4)
C(2)-C(3)	1.373(4)	1.381(4)
C(3)-C(4)	1.369(4)	1.376(4)
C(4)-C(5)	1.376(4)	1.369(4)
C(5)-C(6)	1.387(4)	1.386(4)
C(7)-C(8)	1.494(4)	1.487(4)
C(7)-C(14)	1.382(4)	1.394(4)
C(8)-C(9)	1.382(4)	1.374(4)
C(8)-C(13)	1.388(4)	1.390(4)
C(9)-C(10)	1.381(5)	1.374(5)
C(10)-C(11)	1.363(5)	1.361(6)
C(11)-C(12)	1.370(5)	1.360(6)
C(12)-C(13)	1.376(4)	1.368(4)
C(14)-C(15)	1.398(4)	1.390(4)
C(15)-C(16)	1.518(4)	1.512(4)
C(17)-C(18)	1.386(4)	1.397(4)
C(17)-C(22)	1.425(4)	1.410(4)
C(18)-C(19)	1.379(4)	1.373(5)
C(19)-C(20)	1.366(4)	1.372(5)
C(20)-C(21)	1.377(4)	1.381(5)

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Table 3 *Continued*

	Molecule A	*Molecule B*
C(21)-C(22)	1.391(4)	1.390(4)
C(23)-C(24)	1.511(4)	1.508(4)
C(23)-C(25)	1.398(4)	1.399(4)
C(25)-C(26)	1.393(4)	1.400(4)
C(26)-C(27)	1.501(4)	1.481(4)
C(27)-C(28)	1.378(4)	1.384(4)
C(27)-C(32)	1.391(4)	1.376(4)
C(28)-C(29)	1.373(5)	1.378(4)
C(29)-C(30)	1.373(6)	1.359(5)
C(30)-C(31)	1.367(6)	1.367(5)
C(31)-C(32)	1.375(5)	1.373(5)
Selected bond angles (deg)		
N(1)-Cu-N(2)	83.9(1)	83.7(1)
N(1)-Cu-N(3)	175.9(1)	178.0(1)
N(2)-Cu-N(3)	96.0(1)	94.9(1)
N(1)-Cu-N(4)	95.3(1)	96.8(1)
N(2)-Cu-N(4)	176.8(1)	177.3(1)
N(3)-Cu-N(4)	84.6(1)	84.5(1)
Cu-N(1)-C(1)	111.3(2)	111.4(2)
Cu-N(1)-C(26)	121.6(2)	122.2(2)
C(1)-N(1)-C(26)	125.8(2)	126.4(2)
Cu-N(2)-C(6)	111.7(2)	111.8(2)
Cu-N(2)-C(7)	121.7(2)	122.4(2)
C(6)-N(2)-C(7)	125.9(2)	125.3(2)
Cu-N(3)-C(15)	122.1(2)	122.9(2)
Cu-N(3)-C(17)	110.8(2)	110.5(2)
C(15)-N(3)-C(17)	126.3(2)	125.8(2)
Cu-N(4)-C(22)	110.7(2)	111.0(2)
Cu-N(4)-C(23)	121.9(2)	122.3(2)
C(22)-N(4)-C(23)	126.3(2)	126.4(2)
N(1)-C(1)-C(2)	126.2(2)	127.1(2)
N(1)-C(1)-C(6)	114.4(2)	113.8(2)
N(2)-C(6)-C(1)	113.8(2)	114.2(2)
N(2)-C(6)-C(5)	126.7(2)	125.8(2)
N(2)-C(7)-C(8)	120.9(2)	120.4(2)
N(2)-C(7)-C(14)	122.5(2)	122.3(2)
C(8)-C(7)-C(14)	116.2(2)	117.1(2)
C(7)-C(8)-C(9)	120.6(3)	120.6(3)
C(7)-C(8)-C(13)	120.9(3)	120.6(3)
C(7)-C(14)-C(15)	129.8(3)	129.5(3)
N(3)-C(15)-C(14)	122.2(3)	121.5(3)
N(3)-C(15)-C(16)	122.1(3)	122.4(3)
C(14)-C(15)-C(16)	115.4(3)	115.8(3)
N(3)-C(17)-C(18)	126.0(3)	124.8(3)
N(3)-C(17)-C(22)	114.8(2)	115.2(2)
N(4)-C(22)-C(17)	114.8(2)	114.5(2)
N(4)-C(22)-C(21)	126.4(3)	126.8(3)
N(4)-C(23)-C(24)	122.0(3)	122.5(3)
N(4)-C(23)-C(25)	122.1(2)	121.6(2)
C(24)-C(23)-C(25)	115.8(3)	115.8(3)
C(23)-C(25)-C(26)	129.5(3)	130.6(3)
N(1)-C(26)-C(25)	122.2(3)	122.3(3)
N(1)-C(26)-C(27)	121.7(2)	121.1(2)
C(25)-C(26)-C(27)	115.8(2)	116.5(2)
C926)-C(27)-C(28)	120.4(3)	121.1(2)
C(26)-C(27)-C(32)	120.7(3)	120.6(2)

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Table 4 Selected dihedral angles between mean planes.

Plane I	Plane II	Angle (deg)	
		Molecule A	Molecule B
C1,C2,C3,C4,C5,C6	C8,C9,C10,C11,C12,C13	62.8	61.1
C1,C2,C3,C4,C5,C6	C27,C28,C29,C30,C31,C32	63.1	64.0
C8,C9,C10,C11,C12,C13	C27,C28,C29,C30,C31,C32	35.7	48.6
N1,N2,N3,N4	C1,C2,C3,C4,C5,C6	21.8	24.3
N1,N2,N3,N4	C17,C18,C19,C20,C21,C22	22.0	22.7
N1,N2,N3,N4	N1,N4,C23,C25,C26	25.9	19.4
N1,N2,N3,N4	N2,N3,C7,C14,C15	23.1	23.0
N1,N4,C23,C25,C26	N2,N3,C7,C14,C15	48.9	42.4

a number of short contacts between carbon atoms in the range of 3.00Å to 3.20Å. For example, on the left-hand side of the molecule shown in Figure 1, the distance between the methyl carbon C(16) and the benzenoid carbon C(18) is 2.984Å in molecule A and 2.999Å in molecule B, and that between the methyl carbon C(24) and the benzenoid carbon C(21) is 3.009Å in molecule A and 3.018Å in molecule B. On the right-hand side of the molecule in Figure 1, the distances between C(2) and C(28) are 3.227Å and 3.123Å in molecules A and B, respectively, and those between C(9) and C(5) are 3.194Å and 3.126Å, respectively. These distances are considerably less than the normal C---C van der Waals separation of 3.40Å, or CH_3---C separation of 3.70Å.[10]

In the macrocyclic complex, the propane-1,3-diiminato chelate rings are delocalized and these rings are separated from the aromatic benzenoid rings by nominally single C-N bonds, the average bond distance being 1.407Å. The absence of complete delocalization about the macrocyclic ring is not unusual, it has been observed in other dibenzotetraaza[14]annulene ligands and complexes.[6,7,9,11]

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Supplementary data

Lists of structure factors, anisotropic thermal parameters, and hydrogen atom coordinates are available from RPH upon request.

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