Inorganica Chimica Acta, *86* (1984) 101–106

Crystal and Molecular Structure of the (2,2′-Diaminobiphenyl)-(R,R-*trans*-1,2-diaminocyclohexane)platinum(II) Complex

CECILIA A. RADLOWSKI, CHUI FAN LIU

Department of Chemistry, University of Illinois at Chicago, Chicago, Ill. 60680, U.S.A.

and MOO-JIN JUN

Department of Chemistry, Yonsei University, Seoul, Korea

Received October 25, 1983

*2,2′-Diaminobiphenyl-R,R-*trans*-1,2-diaminocyclohexaneplatinum(II) Chloride Trihydrate, [R,R-chxn)-(dabp)Pt]$Cl_2 \cdot 3H_2O$, crystallizes in the space group $P2_12_12_1$ (D_2^4, No. 19) with a = 6.219(4) Å, b = 17.633(2) Å, c = 21.523(3) Å, V = 2,360.4(8) Å^3, ρ_{calcd} = 1.739 g cm^{-3}, ρ_{measd} = 1.74 g cm^{-3}, and Z = 4. Diffraction data were collected with a Picker FACS-1 four-circle diffractometer. The structure was solved by the heavy atom method and refined by least-square calculations to residuals R = 0.0586 and weighted R = 0.0668. The 2,2′-diaminobiphenyl ligand exhibits complete stereospecificity in its coordination to platinum(II) ion with λ chiral conformation.*

Introduction

When 2,2′-diaminobiphenyl is coordinated to platinum(II) ion through the two nitrogen atoms during the synthesis of [Pt(R,R-chxn)(dabp)]$Cl_2 \cdot 3H_2O$ [1] (R,R-chxn = R,R-*trans*-1,2-diaminocyclohexane and dabp = 2,2′-diaminobiphenyl), the rings become locked into one rotomer, and as a result of the N–M–N bond angle the rings can neither be coplanar nor perpendicular to each other and the complex becomes a dissymmetric molecule. We report here details of the crystal and molecular structure of this complex.

Experimental

Preliminary X-ray examination established a four molecule orthorhombic unit cell with systematic absences h00 for h odd, 0k0 for k odd, and 001 for l odd, consistent with the choice of space group $P2_12_12_1$ (D_2^4 No. 19). Least-squares refinement of the setting angles of 12 reflections for which $39° < 2\theta < 51°$ gave the cell constants a = 6.219(4) Å, b = 17.633(2) Å, c = 21.523(3) Å, V = 2,360.4(8) Å^3, M = 618.24, ρ_{calcd} = 1.739 g cm^{-3}, ρ_{measd} = 1.74 g cm^{-3}, and Z = 4. The crystal and intensity data were collected, using a Picker FACS-1 computer-controlled four-cycle diffractometer [2], from a crystal measuring 0.12 mm × 0.39 mm × 0.10 mm from a take-off angle of 3.0° employing a 2θ scan with a scan rate 2.0°/min. A total of 4770 independent intensities for which $2\theta \leqslant 60$ (Mo K_α radiation 0.70930 Å) was collected from the (+*h*, +*k*, ±*l*) octant. Unique data used: 3279. Final number of variables: 258 independent, 100 dependent (for 'riding' hydrogens). Final error in observation of unit wt.: 1.80. Final R_F = 0.0586 Final R_{WF} = 0.0668.

Solution and Refinement of the Structure

Computer programs used in determining the structure include FORDAP (Fourier Synthesis, by A. Zalkin), LSHF (structure factor calculations with full-matrix least squares refinement, by B. G. DeBoer), STAN1 (distances and angles with their esd's, by B. G. DeBoer), PLOD (least-squares planes and lines, by B. G. DeBoer), and ORTEP (thermal ellipsoid drawings, by C. K. Johnson).

Scattering factors for neutral platinum, chlorine, oxygen, nitrogen and carbon were taken from the compilation of Cromer and Waber [3]; for hydrogen, the 'best floated spherical H atom' values of Stewart *et al.* [4] were used. Both the real ($\Delta f'$) and imaginary ($\Delta f''$) components of anomalous dispersion were included for all non-hydrogen atoms, using the values of Cromer and Liberman [5]. The function minimized in a least-square refinement is $\Sigma w(F_o - F_c)^2$.

The position of the platinum was ascertained from a three dimensional Patterson synthesis. A Fourier synthesis phased by one platinum atom revealed the positions of the two chlorines, one of which appeared as a double image due to a false mirror plane. The false symmetry was broken when three cycles of full-matrix least-squares refinement were applied to each of two trial structures. The first consisted of one platinum atom, one chlorine atom, and one of the mirror image chlorines located at (0.55, 0.26, 0.19). The resulting R factors were R_F = 19.1%, R_{WF} =

0020-1693/84/$3.00

28.2%. The second trial structure consisted of one platinum atom, one chlorine atom, and the other mirror image chlorine located at (0.55, 0.26, 0.31). The resulting R factors were R_F = 20.6%, R_{WF} = 31.3%. Accordingly, the first trial structure was used in a subsequent difference Fourier synthesis. Thus the positions of all the other twenty-five non-hydrogen atoms were revealed.

The refinement of the structure proceeded in the following manner: 1) use of a full 3310 unaveraged data set; 2) successive conversion to anisotropic thermal parameters for all 28 non-hydrogen atoms; 3) the application of the absorption correction. Each of these steps was accompanied by a reduction in the discrepancy indices. Corrections for the effects of anomalous dispersion and secondary extinction were made at all times. Values after these steps were R_F = 0.0767 and R_{WF} = 0.0948. At this point the enantiomorphic structure was refined under identical conditions to R_F and R_{WF} values of 0.0686 and 0.0878, respectively. Hamilton's significance test for the R factor ratio was applied [6]. The first model was used in subsequent calculations.

TABLE I. Positional Parameters for the Non-hydrogen Atoms.[a]

	x	*y*	*z*
Pt	-0.16030(7)	0.12301(2)	0.25207(1)
Cl1	−0.2685(7)	0.4883(2)	0.3344(2)
Cl2	−0.5842(7)	0.2583(2)	0.1881(2)
O1	−0.6129(16)	0.4083(4)	0.2544(5)
O2	−0.666(1)	0.1170(6)	0.2940(5)
O3	0.003(29)	0.3463(5)	0.2983(9)
N1	−0.085(2)	0.2032(4)	0.1876(4)
N2	−0.226(2)	0.0597(5)	0.1760(4)
N3	−0.084(2)	0.1919(5)	0.3270(5)
N4	−0.2495(19)	0.0387(5)	0.3140(4)
C1	−0.0622(26)	0.1669(6)	0.1263(6)
C2	−0.2348(24)	0.1089(6)	0.1201(5)
C3	−0.2221(29)	0.0657(7)	0.0592(5)
C4	−0.2264(32)	0.1197(7)	0.0050(6)
C5	−0.0578(31)	0.1806(8)	0.0117(6)
C6	−0.0718(28)	0.2230(7)	0.0731(6)
C7	−0.2346(24)	0.1353(6)	0.4198(5)
C8	−0.2333(26)	0.1932(6)	0.3764(6)
C9	−0.3782(25)	0.2522(7)	0.3781(7)
C10	−0.5307(31)	0.2537(9)	0.4254(9)
C11	−0.5299(28)	0.1978(9)	0.4713(8)
C12	−0.3881(30)	0.1393(8)	0.4672(7)
C13	−0.0867(25)	0.0707(7)	0.1141(6)
C14	−0.0940(24)	0.0237(6)	0.3617(6)
C15	0.0432(27)	0.0366(7)	0.3534(7)
C16	0.1975(31)	0.0514(7)	0.4002(8)
C17	−0.7944(30)	−0.0070(8)	0.4536(7)
C18	−0.9313(28)	0.0537(7)	0.4590(7)

[a] Estimated standard deviations, shown in parentheses, are right adjusted to the last digit of the preceding number.

Idealized positional parameters for 26 of the total 32 H atoms present in the molecule were computed. The positions of the 6 H atoms in the three water molecules were not computed. An sp^2 and/or an sp^3 coordination about the C and N atoms, along with an H–C bond distance of 0.95 Å and a H–N bond distance of 0.87 Å were assumed. The thermal parameter was programmed so that 1 was added to the thermal parameter of the atom to which H was bonded. Two more cycles of full-matrix least-squares including the contributions from the 26 H atoms resulted in R_F and R_{WF} values of 0.0586 and 0.0668, respectively.

In the final cycle, the largest parameter shifts were 0.010 of the estimated standard deviation for non-hydrogen atoms and 0.002 of the estimated standard deviation for the isotropic hydrogen atoms. The 'goodness of fit' defined by $[w(|F_o| - |F_c|)^2/(m - n)]^{1/2}$ was 1.80 where the number of observations (m) was 3279, the number of variables (n) was 356, and m:n = 12.72:1.

The accuracy of the determined structure was confirmed by means of a final difference Fourier synthesis. The four highest peaks (4.88, 4.62, 3.61, 3.48 e/Å^3) were all within approximately 1A of the Pt atom. All other features were less than 1 e/Å^3 in height. Final positional and thermal parameters for 28 non-hydrogen and 26 hydrogen atoms are given in Tables I through III. The final R_F and R_{WF} values were 0.0586 and 0.0668 respectively. The correct handedness of the model is confirmed because it is consistent with the known absolute configuration of R,R-*trans*-1,2-diaminocyclohexane.

Results and Discussion

Intramolecular bond distances and bond angles are given in Tables IV and V. The atom labelling scheme is shown in Fig. 1 and stereodrawing of the unit cell is seen in Fig. 2.

The conformation of the diaminobiphenyl rings are unambiguously found to be λ. The left-handedness chirality is clearly shown by the view through the diaminobiphenyl toward the Pt atom in Fig. 3. Cyclohexanediamine was also found to be in the λ conformation. This is consistent with the known absolute configuration of the levorotatory isomer of *trans*-1,2-diaminocyclohexane. The structure consists of discrete molecules of Pt(R,R-chxn)(R-dabp) where the ligator nitrogen atoms coordinate to the platinum in a square planar stereochemistry. The N1–Pt–N2 angle is 83.1(4) and the N3–Pt–N4 angle is 88.5(4). The N–Pt–N bond angles reported for the bis(ethylenediamine)platinum(II) ion are 82.8(4)° and 83.6(5)° [7]. A somewhat larger angle would be expected to accommodate the seven-membered diaminobiphenyl chelate ring. The Pt–N_4 chromophore

TABLE II. Anisotropic Thermal Parameters for Non-hydrogen Atoms with Their Estimated Standard Deviations.[a]

	B(1,1)	B(2,2)	B(3,3)	B(1,2)	B(1,3)	B(2,3)
Pt	3.05(2)	2.10(2)	2.52(2)	0.03(1)	0.03(2)	0.08(2)
Cl1	3.8(2)	4.3(1)	10.8(3)	0.1(1)	−0.4(2)	1.6(1)
Cl2	3.6(1)	4.3(1)	9.7(3)	0.1(1)	−0.5(2)	−2.3(1)
O1	5.3(5)	3.3(3)	6.1(5)	0.6(3)	−1.1(6)	−0.3(3)
O2	3.8(5)	8.2(7)	7.0(6)	0.3(6)	0.5(5)	0.7(5)
O3	6.3(8)	3.1(4)	20.1(17)	0.0(5)	4.4(9)	0.9(7)
N1	3.3(5)	1.9(3)	2.8(4)	−1.2(4)	1.1(4)	0.6(3)
N2	3.6(6)	2.4(3)	2.9(4)	−0.4(4)	−0.3(4)	−0.1(3)
N3	4.8(6)	2.6(4)	3.3(5)	1.0(5)	0.2(5)	−0.0(3)
C1	3.8(6)	2.7(5)	2.8(5)	0.7(5)	0.8(5)	−0.4(5)
C2	3.8(7)	2.8(5)	3.0(5)	−0.3(5)	−0.4(4)	0.1(4)
C3	5.1(9)	3.2(5)	3.1(5)	−0.1(6)	0.3(5)	−0.1(4)
C4	7.0(10)	5.2(7)	3.3(5)	−0.2(8)	−0.5(8)	0.2(6)
C5	4.9(8)	4.7(7)	3.7(7)	−0.5(7)	0.4(6)	0.7(5)
C6	3.8(7)	3.6(5)	3.4(6)	−0.1(6)	0.0(5)	0.9(4)
C7	4.7(7)	3.5(5)	2.2(4)	−0.7(6)	−0.2(4)	−0.3(4)
C8	4.5(7)	3.1(5)	2.9(5)	−0.54(5)	−0.7(5)	−0.8(4)
C9	3.5(7)	3.4(6)	5.1(7)	0.4(5)	0.0(6)	0.1(5)
C10	5.2(9)	4.6(7)	6.9(10)	0.2(7)	1.7(7)	−2.1(7)
C11	4.4(8)	6.6(9)	4.3(8)	−1.4(8)	1.4(6)	−1.6(7)
C12	5.9(10)	4.1(6)	4.5(7)	−2.1(7)	1.3(6)	1.1(5)
C13	3.5(7)	3.1(5)	3.2(6)	−0.5(5)	−0.1(5)	0.6(4)
C14	3.8(7)	2.4(5)	3.2(5)	−0.6(5)	0.9(5)	0.5(4)
C15	3.6(7)	3.0(5)	5.2(7)	0.0(5)	0.4(6)	0.9(5)
C16	4.6(9)	4.2(7)	7.3(9)	−0.9(7)	−0.8(8)	1.3(6)
C17	4.5(9)	5.4(7)	6.4(9)	−0.9(7)	−1.1(7)	1.4(7)
C18	4.0(7)	3.6(6)	5.1(8)	−0.7(6)	−1.6(7)	1.0(5)

[a] Anisotropic thermal parameters are in units of A^2 and enter the structure factor equation in the form: $\exp[-0.25(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

TABLE III. Interatomic Distances and Isothermal Parameters for Hydrogen Atoms.[a]

	x	y	z	B(ISO.)
HC9	−0.0768(25)	0.2907(7)	0.3472(7)	5.1267(49)
HC10	−0.6305(31)	0.2944(9)	0.4267(9)	6.8769(63)
HC11	−0.6309(28)	0.1999(9)	0.5044(8)	6.4447(56)
HC12	−0.3926(30)	0.1001(8)	0.4974(7)	5.8543(66)
HC15	0.1690(27)	−0.0543(7)	0.3330(7)	5.2080(47)
HC16	0.3406(31)	−0.0701(7)	0.3978(8)	5.9511(69)
HC17	−0.9397(30)	−0.0115(8)	0.4667(7)	6.8950(59)
HC18	−1.0760(28)	0.0338(7)	0.4705(7)	5.4124(50)
H1C1	0.0742(26)	0.1429(6)	0.1239(6)	4.3438(45)
H1C2	−0.3695(24)	0.1324(6)	0.1185(5)	4.6451(45)
H1C3	−0.0871(29)	0.0405(7)	0.0584(5)	3.9(22)
H2C3	−0.3344(29)	0.0294(7)	0.0563(5)	3.9(22)
H1C4	−0.3621(32)	0.1444(7)	0.0061(6)	2.9(19)
H2C4	−0.2112(32)	0.0934(7)	−0.0331(6)	2.9(19)
H1C5	−0.661(31)	0.2150(8)	−0.0221(6)	7.8(37)
H2C5	0.077(31)	0.1555(8)	0.0110(6)	7.8(37)
H1C6	0.0396(28)	0.2596(7)	0.0765(6)	4.7(23)
H2C6	−0.2074(28)	0.2477(7)	0.0750(6)	4.7(23)
H1N1	0.0316(20)	0.2268(4)	0.1983(4)	2.7744(35)
H2N1	−0.1907(20)	0.2355(4)	0.1851(4)	2.7744(35)
H1N2	−0.3486(20)	0.0363(5)	0.1813(4)	3.0734(49)

(Continued overleaf)

TABLE III. *(Continued)*

H1N3	0.0338(23)	0.1732(5)	0.3428(5)	3.3712(44)
H1N2	−0.1250(20)	0.0265(5)	0.1704(4)	3.0734(40)
H1N3	0.0338(23)	0.1732(5)	0.3128(5)	3.3712(44)
H1N3	−0.0635(23)	0.2368(5)	0.3136(5)	3.3712(44)
H1N4	−0.3683(19)	0.0527(5)	0.3320(4)	3.5376(40)
H1N4	−0.2719(19)	−0.0028(5)	0.2932(49)	3.5376(40)

[a]Estimated standard deviations, shown in parentheses, of positional parameters are equal to those of the attached carbon or nitrogen atom.

TABLE IV. Interatomic Bond Distances with Their Standard Deviations in Parentheses.

	Distance (Å)		Distance (Å)
Pt–N1	2.035(8)	C7–C8	1.385(17)
Pt–N2	2.023(9)	C8–C9	1.377(17)
Pt–N3	2.063(10)	C9–C10	1.391(23)
Pt–N4	2.073(9)	C10–C11	1.395(23)
N1–C1	1.474(15)	C11–C12	1.359(22)
N2–C2	1.484(14)	C12–C7	1.399(21)
N3–C8	1.408(19)	C7–C13	1.469(18)
N4–C14	1.435(18)	C13–C14	1.399(18)
C1–C2	1.488(18)	C14–C15	1.376(19)
C2–C3	1.517(16)	C15–C16	1.415(22)
C3–C4	1.504(17)	C16–C17	1.392(22)
C4–C5	1.508(20)	C17–C18	1.374(21)
C5–C6	1.522(19)	C18–C13	1.399(21)
C6–C1	1.513(17)		

TABLE V. Selected Interatomic Bond Angles with Their Estimated Standards in Parentheses.[a]

	Angle		Angle
N1–Pt–N2	83.0(4)	C8–C7–C13	120.8(12)
N2–Pt–N4	94.0(4)	C8–C7–C12	117.2(14)
N4–Pt–N3	88.4(4)	C7–C12–C11	121.9(15)
N3–Pt–N1	94.4(4)	C12–C11–C10	119.4(16)
Pt–N1–C1	109.2(6)	C11–C10–C9	120.2(16)
N1–C1–C2	107.9(11)	C10–C9–C8	118.6(15)
C2–C1–C6	110.6(12)	Pt–N4–C14	114.3(8)
C1–C2–C3	112.6(11)	N4–C14–C13	119.2(12)
C2–C3–C4	110.5(10)	C14–C13–C7	120.4(12)
C3–C4–C5	111.4(13)	C14–C13–C18	116.9(14)
C4–C5–C6	113.0(13)	C13–C18–C17	122.4(15)
C5–C6–C1	109.4(10)	C18–C17–C16	119.1(16)
Pt–N2–C2	109.8(6)	C17–C16–C15	120.5(16)
N2–C2–C1	107.7(10)	C16–C16–C14	118.0(14)
Pt–N3–C8	117.2(10)	C15–C14–C13	122.8(14)
N3–C8–C7	107.4(8)		

[a]Calculations include the effects of all elements of the positional covariance matrix as well as the uncertainties in unit cell dimensions. No corrections have been applied for the effects of thermal motion.

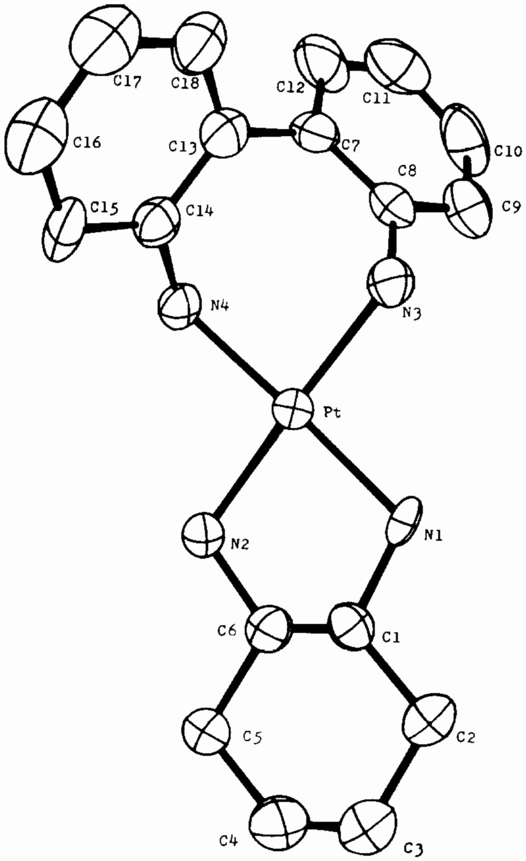

Fig. 1. The numbering of the atoms in the molecular unit.

is only slightly distorted from planarity. The Pt–N1–N2 plane is at an angle of 1.8° to the Pt–N3–N4 plane. The nitrogen atoms of the opposite rings do not eclipse each other exactly.

In the cyclohexanediamine ring C1 and C2 deviate from the Pt–N1–N2 plane by +0.366(12)° and −0.311(12)°, respectively. This conformational feature agrees with the conformation of cyclohexanediamine in Λ-(+)-[Co(λλλ)-((−)-chxn))$_3$] [8]. The dihedral angle between the normal to the N1–C1–C2 plane and the normal to the N1–C2–C1 plane defines the degree of ring puckering. The dihedral angle was found to be 51.6°. The mean dihedral angle

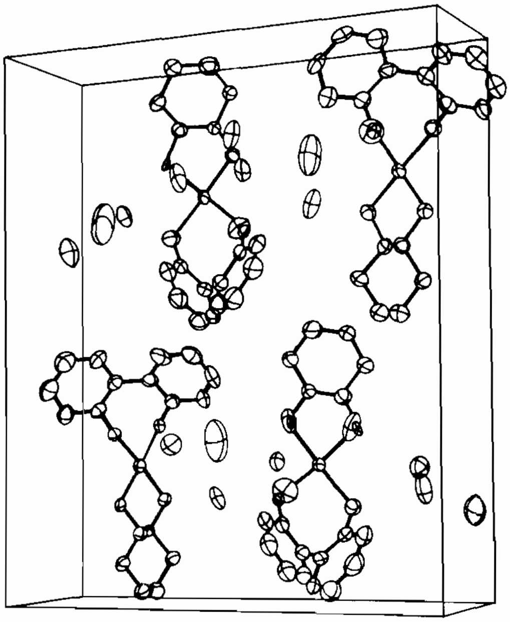

Fig. 2. Stereodrawing of the unit cell showing four molecules per unit cell.

between the N–C–C planes of (+)-cyclohexanediamine in the complex $(-)_{589}$-$[\mathrm{Co}((+)=\mathrm{chxn})_3]\mathrm{Cl}_3\cdot 5\mathrm{H}_2\mathrm{O}$ was found to be 59.3° [9]. This is almost identical with the value expected in a free *trans*-1,2-diaminocyclohexane molecule. Thus, in the platinum complex, the diaminocyclohexane seems to be somewhat strained by the formation of a chelate ring. The chair conformation is indicated for the cyclohexanediamine. All the bond angles and distances within the six-membered cyclohexanediamine are normal and comparable to those found in the tris(cyclohexanediamine)cobalt(III) complexes.

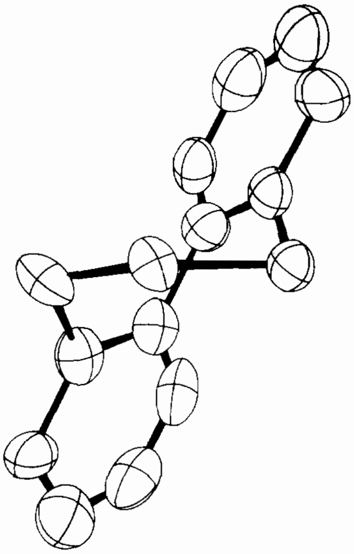

Fig. 3. View through 2,2'-diaminobiphenyl.

The bond distances and bond angles between the carbon atoms in each of the phenyl rings are typical and agree with those found in the copper(II) and

TABLE VI. Distance of Atoms from Various Planes, and Angles between Planes.

I. Plane through Pt and CHNX N's
$0.944x - 0.326y - 0.0159z + 1.73 = 0$

C1	0.366(13)
C2	−0.311(12)
N3	0.0264(12)
N4	−0.0599(10)
C12	−3.249(4)

II. Plane through Pt and DABP N's
$0.934x - 0.355y - 0.00778z + 1.746 = 0$

C8	−0.886(13)
C14	0.989(13)
N1	−0.0573(10)
N2	0.0233(10)
C15	2.168(15)
C9	−2.0994(14)
O2	−2.914(11)

III. Plane through phenyl ring,
C7–C12: $0.638x + 0.526y + 0.560z - 5.40 = 0$

Pt	−1.8529(9)
N3	−0.0177(12)
N4	−2.241(10)

IV. Plane through phenyl ring,
C13–C18: $0.264x - 0.0396y + 0.963z - 7.98 = 0$

Pt	−3.1020(4)
N3	−1.470(12)
N4	−1.903(10)

V. Plane through CHNX fragment
N1–C1–C2: $0.605x - 0.688y + 0.400z + 1.17 = 0$

VI. Plane through CHXN fragment
N2–C1–C2: $0.205x - 0.688 + 1.17 = 0$

Angles between Planes

Planes	Angles (deg.)
I–II	1.83
III–IV	46.4
V–VI	51.6

cobalt(II) complexes [10, 11]. The equations of the least square planes through selected portions of the molecule are presented in Table VI. The carbon atoms within each of the two phenyl rings are coplanar. The dihedral angle between the two phenyl rings is 46.4°.

Corey and Bailar [12] showed that the major contribution to the energy difference between the chiral conformation, $\lambda\lambda$ or $\delta\delta$ and the meso form $\lambda\delta$ in *trans*-$M(en)_2a_2$] were the interactions between the opposing NH_2 groups. In the $\lambda\lambda$ or $\delta\delta$ complex the hydrogens are staggered whereas in the meso form they are eclipsed. Models of λ-chxn and λ-dabp coordinated to platinum(II) show that the hydrogen atoms of the opposing nitrogens are staggered. Furthermore, N1–Pt–N2 plane is raised by 1.8° with respect to the N3–Pt–N4 plane to prevent complete occlusion of the opposing nitrogens. In the λ-chxn-λ-dabp model the hydrogens are very nearly eclipsed. Since λ-cyclohexanediamine coordinates stereospecifically and cannot interconvert to its enantiomer, the conformation of the incoming ligand is determined by the extent of the nonbonded interactions with cyclohexanediamine hydrogens.

From the X-ray data on the Pt(R,R-chxn)(R-dabp) system it is shown that the H–H interaction is significant. During the reaction of free 2,2′-diaminobiphenyl with [$Pt(R,R\text{-}chxn)Cl_2$] only one product was isolated, [$Pt(R,R\text{-}chxn)(R\text{-}dabp)]Cl_2 \cdot 3H_2O$. Considering the fact that there is an equal probability to form either the R-dabp or the S-dabp conformer, 2,2′-diaminobiphenyl exhibits complete stereospecificity in this reaction. The dabp was constrained to display only one conformer as a result of the nonbonded interactions resulting from the hydrogen atoms of the chxn group.

Acknowledgment

Financial support for this work from the Korea Science and Engineering Foundation (to MJJ) and from the National Institute of Health (to CFL) is gratefully acknowledged.

References

1 Submitted for publication.
2 The experimental technique has been described in the literature by M. R. Churchill and B. G. DeBoer, *Inorg. Chem., 12,* 525 (1973).
3 D. T. Cromer and J. T. Waber, *Acta Crystallogr., 18,* 104 (1965).
4 R. Stewart, E. Davidson and W. Simpson, *J. Chem. Phys., 42,* 3175 (1965).
5 D. T. Cromer and D. Lieberman, *ibid., 53,* 1891 (1970).
6 W. C. Hamilton, *Acta Crystallogr., 18,* 502 (1965).
7 W. A. Freeman, *Inorg. Chem., 15,* 2235 (1976).
8 A. Kobayashi, F. Marumo and Y. Saito, *Acta Crystallogr., B28,* 2709 (1972).
9 F. Marumo, Y. Utaumi and Y. Saito, *ibid., B26,* 1492 (1970).
10 T. Cheeseman, D. Hall and T. Waters, *J. Chem. Soc., (A),* 1396 (1966).
11 L. Pisnolet, R. Taylor and W. Horrocks, *J. Am. Chem. Soc., 91,* 5457 (1969).
12 E. J. Corey and J. C. Bailar, *ibid., 81,* 2620 (1959).