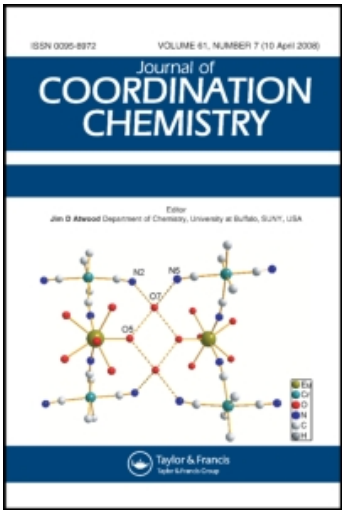

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The Crystal and Molecular Structure of Bis(tribenzylphosphine oxide)dichloro Cobalt(II)

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SHORT COMMUNICATION
The Crystal and Molecular Structure of Bis(tribenzylphosphine oxide)dichloro Cobalt(II)†

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INTRODUCTION

The crystal and molecular structure of bis(tribenzylphosphine oxide)dichlorocobalt (II) has been undertaken as part of a continuing comparative study of the tribenzyl, dibenzyl phenyl, benzyl diphenyl, and triphenyl phosphine oxide complexes in solution and solid state.[1] The purpose is the elucidation of the molecular geometry of this complex.

EXPERIMENTAL

The samples were obtained by mixing alcoholic solutions of salt and organic ligand in the proper stoechiometric proportions. The solution was concentrated and cooled in a refrigerator until crystallization. Usually the crystals were well formed blue bipiramids.

Bis(tribenzylphosphine oxide)dichlorocobalt (II) has the following cell dimensions a = 15.133(1) Å, b = 13.905(1) Å, c = 16.492(1) Å, $\alpha = \beta = \gamma = 90°$, V = 3954 Å.[3] These were obtained using a Philips automatic diffractometer PW 1100 and refined by least squares calculations. It crystallizes in the orthorhombic system. The following systematic extintions, h00 for h = odd, 0k0 for k = odd and 00ℓ for ℓ = odd were observed fixing the space group as $P2_12_12_1$. The observed density = 1.29 g/cm^3 (by flotation), calculated density = 1.293 g/cm^3 assuming Z = 4 molecules/cell.

The intensities of 2110 independent reflections were recorded using the PW 1100 Philips automatic single crystal diffractometer by courtesy of Philips Company and their Laboratories in Eindhoven. The radiation was graphite monochromatized Mokα using the $\theta-2\theta$ scan type with a scan width of 1° and a speed of 0.08°/sec. Background counts were done on each extreme of the scanning interval taking 1/2 of the scan time on both sides. The intensity of three standard reflections, (141), (230) and (330) were measured each 100 reflections and did not show any appreciable change in intensity during all data collection.

Data Reduction

The data were corrected for the Lp factor and reduced to the corresponding moduli of structure factors. All reflections in which the observed F was greater than three times the value of the corresponding standard deviation (σ(F)) were considered to be statistically above background. 2085 reflections were classified in this category. As the largest and smallest dimensions of the crystal used in data collection were respectively only of 0.5 and 0.3 mm and μ = 8.2 no absorption correction was applied.

Structure Analysis

As we intended to use direct methods, we proceeded to the determination of the scale factor and average temperature factor using the Wilson plot procedure and to the calculation of the normalized structure factors. A program written by R. Shiono[2] was used.

The program Multan[3] was used for the phasing of the 470 largest E values (E > 1.24). 16 sets of phases were generated; the corresponding E maps were

TABLE I

Non hydrogen atoms thermal and positional parameters of bis(tribenzylphosphine oxide)dichlorocobalt (II) and their estimated standard deviations[a]

ATOM	X	Y	Z	B(1,1)	B(2,2)	B(3,3)	B(1,2)	B(1,3)	B(2,3)
CO	32444(15)	14290(15)	7734(14)	676(12)	692(14)	522(9)	-33(18)	-3(22)	51(17)
P1	28100(33)	5753(32)	26562(28)	763(29)	682(19)	524(19)	1(50)	57(38)	183(39)
P2	53801(31)	17592(30)	3876(29)	662(18)	627(20)	603(18)	-33(48)	78(36)	71(39)
CL1	27144(32)	27522(31)	7065(32)	845(25)	775(18)	811(16)	171(50)	25(45)	225(45)
CL2	27365(43)	5982(39)	-2268(32)	1413(37)	1129(25)	664(18)	-383(68)	-164(57)	-334(49)
O1	29723(77)	9277(74)	18143(71)	844(65)	757(58)	738(57)	56(115)	156(120)	128(135)
O2	45118(72)	14628(72)	7278(69)	831(56)	787(58)	696(47)	49(136)	116(146)	372(120)
C1	18210(121)	9787(102)	31330(96)	856(102)	584(81)	597(68)	58(112)	316(210)	-21(146)
C2	19097(114)	18622(113)	34008(96)	737(112)	870(110)	516(70)	156(215)	173(226)	24(235)
C3	19329(119)	25766(119)	28561(97)	838(100)	910(107)	504(95)	88(236)	194(210)	42(226)
C4	20757(148)	33797(124)	30747(126)	1305(143)	725(103)	916(103)	212(210)	-37(220)	22(210)
C5	22073(144)	35256(130)	38737(139)	1147(124)	829(142)	1221(130)	54(300)	-168(215)	-591(317)
C6	22321(147)	28404(149)	44716(109)	1201(132)	1392(159)	599(109)	302(185)	-85(244)	-722(199)
C7	20545(122)	20455(122)	41909(113)	897(120)	988(116)	648(95)	-89(215)	162(218)	-416(235)
C8	27715(122)	-5562(119)	26080(101)	943(117)	865(97)	559(89)	6(210)	332(243)	219(218)
C9	19437(114)	-8316(100)	21766(98)	808(119)	554(79)	636(84)	-99(215)	30(210)	334(137)
C10	12063(121)	-3663(125)	25717(106)	771(93)	1068(103)	673(89)	-388(219)	87(213)	482(215)
C11	4447(126)	-13912(129)	21638(122)	881(114)	858(92)	1006(122)	-468(232)	118(218)	509(240)
C12	5074(136)	-15748(125)	13162(119)	1092(115)	841(115)	804(90)	-259(219)	-116(220)	-6(235)
C13	12284(128)	-13639(124)	9311(104)	1078(110)	874(103)	612(105)	0(250)	-262(238)	-197(218)
C14	19927(125)	-10434(115)	13291(102)	970(133)	814(90)	560(86)	181(218)	200(200)	190(217)
C15	36584(122)	8405(120)	33988(102)	929(114)	839(116)	565(83)	-16(270)	-47(215)	276(215)
C16	45436(106)	3753(109)	33300(99)	614(93)	763(100)	606(84)	145(220)	-177(216)	17(235)
C17	48996(118)	84(114)	40175(105)	839(110)	777(103)	685(100)	4(236)	-271(224)	417(216)
C18	56988(127)	-3774(125)	39588(115)	903(109)	907(121)	819(102)	133(240)	-362(215)	-139(234)
C19	61738(118)	-4671(130)	32705(112)	710(115)	1051(133)	762(92)	626(216)	151(216)	197(218)
C20	58227(119)	-1116(135)	25955(117)	761(114)	1148(127)	812(102)	503(236)	-282(217)	568(223)
C21	50092(125)	2979(124)	26288(113)	769(89)	928(122)	668(104)	235(232)	4(235)	122(213)
C22	53283(97)	19734(90)	-6743(92)	888(101)	779(82)	522(61)	302(204)	-58(217)	-19(133)
C23	49600(91)	28215(85)	-9357(84)	698(87)	625(72)	632(70)	-11(210)	64(225)	-108(140)
C24	40950(95)	29545(98)	-10856(82)	810(89)	679(74)	603(83)	21(214)	-60(230)	231(227)
C25	37751(99)	37213(93)	-13153(100)	767(100)	759(94)	928(87)	37(216)	-19(227)	-12(236)
C26	43183(109)	43732(99)	-13890(98)	995(113)	732(89)	831(76)	394(198)	76(196)	14(214)
C27	52121(106)	42368(90)	-12279(94)	1020(108)	605(87)	763(79)	-34(247)	27(218)	94(236)
C28	55085(90)	35054(84)	-10271(80)	762(78)	591(67)	680(72)	-73(222)	285(239)	381(146)
C29	57603(105)	26443(92)	8975(90)	1048(123)	700(80)	657(68)	151(232)	166(213)	138(137)
C30	58409(99)	25910(92)	18201(89)	874(97)	639(76)	685(104)	-217(210)	-42(198)	64(230)
C31	65643(106)	23293(107)	22254(103)	951(119)	869(89)	955(103)	147(214)	-696(157)	-293(214)
C32	65735(134)	22715(116)	30754(103)	1550(154)	964(96)	883(99)	-83(246)	-1036(246)	-176(235)
C33	58318(160)	24432(126)	35033(105)	2116(189)	1075(117)	710(103)	-383(215)	-663(210)	198(205)
C34	51347(127)	26851(124)	31244(97)	1331(138)	1194(110)	621(97)	-365(232)	-100(217)	31(216)
C35	50847(121)	27453(108)	23313(102)	1100(87)	889(66)	787(77)	87(219)	-301(197)	-75(186)
C36	61671(93)	9578(89)	4923(78)	746(126)	708(109)	544(89)	-37(236)	-2(245)	222(256)
C37	60590(94)	2153(81)	-427(81)	750(80)	520(77)	623(109)	162(135)	39(245)	-65(236)
C38	65539(123)	876(92)	-7211(103)	1448(129)	606(89)	870(93)	117(214)	519(235)	-292(219)
C39	64398(158)	-5172(114)	-12459(118)	2164(223)	795(87)	1052(108)	-342(325)	841(346)	-420(228)
C40	57932(152)	-10858(106)	-10913(115)	1979(168)	730(85)	1066(108)	295(112)	-145(248)	-739(227)
C41	52642(119)	-9914(105)	-4477(128)	1074(138)	729(114)	1503(129)	-149(245)	-231(239)	-477(247)
C42	54143(112)	-3819(95)	716(114)	1000(148)	600(97)	1240(114)	-99(237)	-499(235)	-199(195)

[a] Positional and Thermal Parameters X 10^5

calculated for the two with the best figure of merit. In those we found five well defined peaks which could be attributed to Co^{++}, Cl^- and P. At this stage we did not attempt to discriminate between chlorine and phosphorus. Structure factors were calculated for both and the solution that gave the smallest value of the agreement index R was chosen. The coordinates for the heavy atoms were in this way definitely established. Difference Fourier maps disclosed all the remaining atoms, except hydrogen. The coordinates of all atoms were then refined by the full matrix least squares refinement of $w(|F_o|-|F_o|)^2$ using the corresponding program included in the Enraf–Nonius Structure Determination Package (SDP)[4] written for use on the PDP–11 series of computers to an R value of 0.075, when all shifts were smaller than three times their standard deviations.

As the positions of the hydrogen atoms were not revealed by a difference synthesis, their coordinates were generated from the positions of the atoms to which they are bonded by assuming tetrahedral and trigonal angles and C–H distances of 1.0 Å. The full matrix least squares refinement gave a final value of R = 0.065 taking w as constant up to the end of the refinement which was considered completed when all coordinate shifts were less than twice the corresponding standard deviations. The hydrogen positional and thermal parameters were not refined. An attempt was made towards improving the refinement using weights proportional to the standard deviation of measured F's[4] and the pivot point scheme[4] with no further improvement. In all calculations the atomic scattering factors were used taking into account the anomalous scattering coefficients. Their numerical values were taken from the Vol. 4 of the International Tables for X-ray Crystallography.[8]

Structural Data

Tables I and II give the final parameters of the atoms in the asymetric unit and their estimated standard deviations in parenthesis. The intramolecular distances and angles are given in Table III. Equation for the best plane of the phenyl rings was obtained by least squares fitting. Table IV gives the equations for

TABLE II
Hydrogen positional and thermal parameters of bis(tribenzylphosphine oxide)dichlorocobalt (II) and their standard deviations[a]

ATOM	X	Y	Z	B	ATOM	X	Y	Z	B
H1	16630(0)	6224(0)	36080(0)	680000(0)	H22	49744(0)	15244(0)	-9500(0)	850000(0)
H2	13150(0)	9425(0)	27230(0)	700000(0)	H23	59545(0)	19513(0)	-9030(0)	650000(0)
H3	18122(0)	24469(0)	22601(0)	850000(0)	H24	36571(0)	24468(0)	-10396(0)	650000(0)
H4	20857(0)	38338(0)	26519(0)	850000(0)	H25	31239(0)	38359(0)	-13924(0)	850000(0)
H5	22699(0)	41242(0)	40686(0)	850000(0)	H26	41120(0)	49557(0)	-16003(0)	850000(0)
H6	24051(0)	29543(0)	50363(0)	850000(0)	H27	56597(0)	47615(0)	-12530(0)	850000(0)
H7	20323(0)	15786(0)	46063(0)	850000(0)	H28	61579(0)	33936(0)	-9165(0)	850000(0)
H8	27840(0)	-7672(0)	31777(0)	690000(0)	H29	53624(0)	31548(0)	7576(0)	650000(0)
H9	33084(0)	-7450(0)	23126(0)	720000(0)	H30	63753(0)	28127(0)	6699(0)	650000(0)
H10	10014(0)	-10001(0)	31521(0)	850000(0)	H31	71582(0)	22559(0)	18931(0)	850000(0)
H11	-1107(0)	-15082(0)	24701(0)	850000(0)	H32	71620(0)	20476(0)	33390(0)	850000(0)
H12	35(0)	-18593(0)	10203(0)	850000(0)	H33	59198(0)	23419(0)	41118(0)	850000(0)
H13	12541(0)	-14418(0)	3333(0)	850000(0)	H34	45839(0)	28615(0)	34958(0)	850000(0)
H14	25655(0)	-9322(0)	10255(0)	850000(0)	H35	44848(0)	28967(0)	20231(0)	850000(0)
H15	33941(0)	6978(0)	39445(0)	690000(0)	H36	62218(0)	7906(0)	10982(0)	650000(0)
H16	37626(0)	14452(0)	33559(0)	720000(0)	H37	67870(0)	11864(0)	3460(0)	650000(0)
H17	45559(0)	264(0)	45402(0)	850000(0)	H38	70710(0)	5251(0)	-8450(0)	850000(0)
H18	59747(0)	-5991(0)	45070(0)	850000(0)	H39	68782(0)	-5888(0)	-17339(0)	850000(0)
H19	67301(0)	-7890(0)	32483(0)	850000(0)	H40	57014(0)	-15352(0)	-15359(0)	850000(0)
H20	61601(0)	-1268(0)	20847(0)	850000(0)	H41	47424(0)	-14243(0)	-3981(0)	850000(0)
H21	47490(0)	5330(0)	21224(0)	850000(0)	H42	49720(0)	-3158(0)	5662(0)	850000(0)

[a] Positional and Thermal Parameters X 10^5

TABLE III
Bis(tribenzylphosphine oxide)dichlorocobalt (II) intramolecular distances and angles

At.1	At.2	At.3	d 12	d 23	Angle	At.1	At.2	At.3	d 12	d 23	Angle
Cl1	Co	Cl2	2.255(3)	2.249(3)	113.0(1)	C14	C9	C10	1.420(12)	1.322(13)	119.5(10)
Cl1	Co	O1	2.255(3)	1.937(7)	110.6(2)	P1	C15	C16	1.824(11)	1.534(13)	118.9(7)
Cl1	Co	O2	2.255(3)	1.920(6)	109.1(2)	C15	C16	C17	1.534(13)	1.384(12)	118.8(9)
Cl2	Co	O1	2.249(3)	1.937(7)	109.6(2)	C15	C16	C21	1.534(13)	1.360(13)	123.9(9)
Cl2	Co	O2	2.249(3)	1.920(6)	109.2(2)	C16	C17	C18	1.384(12)	1.357(14)	121.3(10)
O1	Co	O2	1.937(7)	1.920(6)	104.9(3)	C17	C18	C19	1.357(14)	1.407(14)	121.4(10)
Co	O1	P1	1.937(7)	1.517(7)	176.0(8)	C18	C19	C20	1.407(14)	1.351(13)	117.1(10)
Co	O2	P2	1.920(6)	1.507(7)	153.0(8)	C19	C20	C21	1.351(13)	1.394(14)	121.0(11)
O1	P1	C1	1.517(7)	1.808(11)	113.6(5)	C20	C21	C16	1.360(13)	1.394(14)	121.9(10)
O1	P1	C8	1.517(7)	1.819(12)	109.4(5)	C21	C16	C17	1.360(13)	1.384(12)	117.1(9)
O1	P1	C15	1.517(7)	1.824(11)	114.5(5)	P2	C22	C23	1.795(9)	1.527(12)	116.9(6)
C1	P1	C8	1.808(11)	1.819(12)	110.3(5)	C22	C23	C24	1.527(12)	1.353(12)	123.8(9)
C1	P1	C15	1.808(11)	1.824(11)	102.0(5)	C22	C23	C28	1.527(12)	1.383(11)	119.9(8)
C8	P1	C15	1.819(12)	1.824(11)	106.5(5)	C23	C24	C25	1.353(12)	1.361(12)	122.5(9)
O2	P2	C22	1.505(7)	1.795(3)	113.3(3)	C24	C25	C26	1.361(12)	1.335(13)	120.1(9)
O2	P2	C29	1.505(7)	1.748(3)	111.3(3)	C25	C26	C27	1.335(13)	1.395(14)	118.4(10)
O2	P2	C36	1.505(7)	1.756(3)	109.3(3)	C26	C27	C28	1.395(14)	1.312(12)	120.5(10)
C22	P2	C29	1.795(3)	1.748(3)	109.5(1)	C27	C28	C23	1.312(12)	1.383(11)	122.1(9)
C22	P2	C36	1.795(3)	1.756(3)	104.9(1)	C28	C23	C24	1.383(11)	1.353(12)	116.3(9)
C29	P2	C36	1.748(3)	1.756(3)	108.2(2)	P2	C29	C30	1.748(10)	1.525(12)	117.1(7)
P1	C1	C2	1.808(11)	1.479(13)	113.1(8)	C29	C30	C31	1.525(12)	1.348(13)	124.8(11)
C1	C2	C3	1.479(13)	1.449(13)	124.2(9)	C29	C30	C35	1.525(12)	1.440(14)	120.6(10)
C1	C2	C7	1.479(13)	1.356(13)	120.5(10)	C30	C31	C32	1.348(13)	1.405(15)	122.1(12)
C2	C3	C4	1.449(13)	1.345(15)	125.6(10)	C31	C32	C33	1.405(15)	1.356(18)	119.2(12)
C3	C4	C5	1.345(15)	1.353(16)	116.6(12)	C32	C33	C34	1.356(18)	1.308(17)	119.8(11)
C4	C5	C6	1.353(16)	1.470(17)	122.0(11)	C33	C34	C35	1.308(17)	1.321(14)	122.4(14)
C5	C6	C7	1.47(17)	1.370(15)	116.9(10)	C34	C35	C30	1.321(14)	1.440(14)	121.8(12)
C6	C7	C2	1.370(15)	1.356(13)	123.7(12)	C35	C30	C31	1.440(14)	1.348(13)	114.4(9)
C7	C2	C3	1.356(13)	1.449(13)	115.0(10)	P2	C36	C37	1.756(9)	1.493(11)	115.8(6)
P1	C8	C9	1.819)12)	1.529(14)	112.6(8)	C36	C37	C38	1.493(11)	1.379(12)	122.4(9)
C8	C9	C10	1.529(14)	1.322(13)	122.3(9)	C36	C37	C42	1.493(11)	1.393(13)	122.5(9)
C8	C9	C14	1.529(14)	1.420(12)	118.0(9)	C37	C38	C39	1.379(12)	1.310(14)	123.8(12)
C9	C10	C11	1.322(13)	1.432(14)	121.5(10)	C38	C39	C40	1.310(14)	1.345(17)	119.1(13)
C10	C11	C12	1.432(14)	1.429(15)	118.7(10)	C39	C40	C41	1.345(17)	1.319(17)	119.9(12)
C11	C12	C13	1.429(15)	1.306(14)	118.5(11)	C40	C41	C42	1.319(17)	1.331(14)	122.3(13)
C12	C13	C14	1.306(14)	1.424(14)	123.1(10)	C41	C42	C37	1.331(14)	1.393(13)	119.7(12)
C13	C14	C9	1.424(14)	1.420(12)	118.1(10)	C42	C37	C38	1.493(11)	1.379(12)	115.0(9)

the planes in the form Ax + By + Cz − D = 0 where A, B, C and D are constants and x, y and z are orthogonalized coordinates. Also are given the standard deviations of each atom from the mean plane. Dihedral angles between planes are given in Table V.

DISCUSSION AND DESCRIPTION OF THE MOLECULAR AND CRYSTALLINE STRUCTURE

An ORTEP[5] representation of the molecular structure of bis(tribenzylphosphine oxide)dichlorocobalt (*II*) is shown in Figure 1. The cobalt is coordinated tetrahedrally by two chlorines and two oxygens. The chlorine and oxygen atoms are close-packed, as is indicated by the fact that their interatomic distances are equal to the sum of the van der Waals radii similar to the structure of bis(N-benzyldiphenyl-phosphonic amide) dichlorocobalt (II).[6] The angles *Cl*1–Co–*Cl*2, O1–Co–O2, O1–Co–*Cl*1, O1–Co–*Cl*2, O2–Co–*Cl*1 and O2–Co–*Cl*2 are 113.01°, 104.98°, 110.60°, 109.81°, 109.10° and 109.21° respectively. The distortions in these angles from the tetrahedral value of 109° 28′ are severer for the *Cl*1–Co–*Cl*2 and O1–Co–O2. The reason for these distortions are, as pointed out by Roy and Jefferey,[5] the differences in the Co–O and Co–*Cl* bond lengths and the differences in the van der Waals radii of chlorine and oxygen. Interatomic distances and angles in the phenyl rings are normal. As we can see from Table II, there is a good agreement between the values of the interatomic distances Co–*Cl*1 and Co–*Cl*2, Co–O1 and Co–O2, O1–P1 and O2–P2 respectively within a value not larger than twice the standard deviations. There is good agreement of these bond length values and similar bond distances determined in the structure of bis(N-benzyldiphenylphosphonic amide)dichlorocobalt (II)[6] and of bis(trimethylphosphine oxide) dichlorocobalt (II).[7]

The crystal packing consists of an arrangement of

TABLE IV
Least square planes

The equations of planes are expressed in orthogonalized space as Ax+By+Cz+D=0

Plane 1 0.9821x-0.1094y-0.1532z-1.7062=0

c1	0.033	c4	0.000	c6	0.025
c2	-0.036	c5	0.006	c7	-0.028
c3	-0.001				

Plane 2 -0.3394x+0.9251y-0.1700z-2.9229=0

c8	0.007	c11	0.005	c13	0.025
c9	-0.008	c12	-0.015	c14	-0.017
c10	0.003				

Plane 3 0.4668x+0.8595y+0.2081z+4.8591=0

c15	0.019	c18	0.019	c20	0.020
c16	0.017	c19	0.021	c21	0.020
c17	0.018				

Plane 4 0.1496x-0.2316y-0.9612z+1.5678=0

c22	-0.006	c25	0.000	c27	-0.017
c23	0.006	c26	0.006	c28	0.014
c24	-0.003				

Plane 5 0.2914x+0.9543y+0.0657z+6.6754=0

c29	0.015	c32	0.019	c34	0.020
c30	-0.027	c33	0.021	c35	0.019
c31	-0.001				

Plane 6 -0.6194x+0.5603y-0.5499z-5.4039=0

c36	0.026	c39	0.013	c41	0.005
c37	-0.038	c40	0.003	c42	-0.055
c38	-0.009				

TABLE V
Dihedral angles between planes

PLANE NO.	PLANE NO.	DIHEDRAL ANGLE
1	2	-65.9
1	3	-70.6
1	4	71.4
1	5	-80.1
1	6	-54.2
2	3	-53.0
2	4	-84.2
2	5	-39.4
2	6	34.7
3	4	70.8
3	5	14.1
3	6	-85.5
4	5	76.1
4	6	72.2
5	6	-71.5

discrete molecules of the title compound. This is illustrated in the ORTEP[5] stereo view shown in Figure 2.

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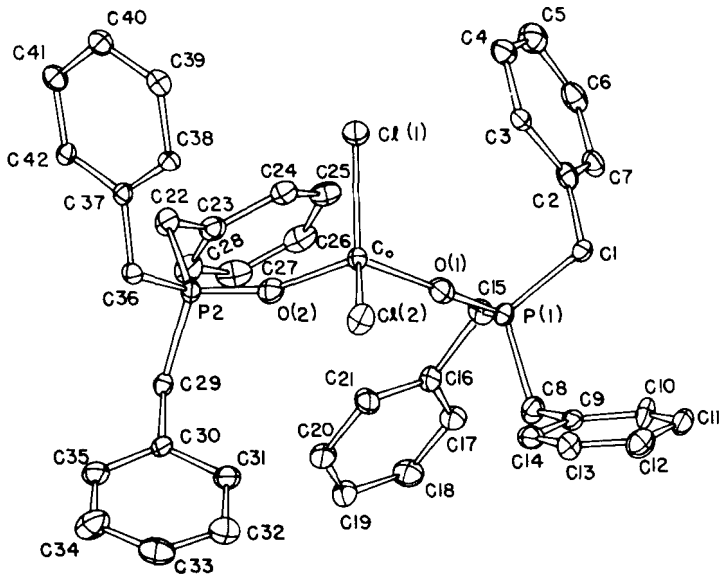

FIGURE 1 The molecular structure of bis(tribenzylphosphine oxide)dichlorocobalt(II).

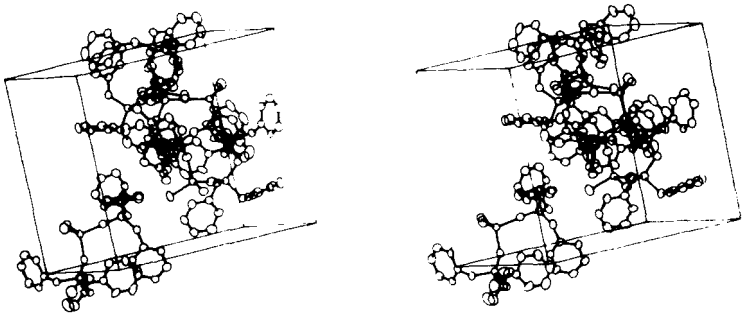

FIGURE 2 Stereo view of the molecular packing of bis(tribenzylphosphine oxide)dichlorocobalt(II)

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