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The Crystal Structure of Bis(*N*-*tert*-butyldithiocarbamato)(tricyclohexylphosphine)-platinum(II) Cyclohexane Solvate

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Abstract

The crystal structure of the title compound, $Pt(C_5H_{10}NS_2)_2(C_{18}H_{33}P).C_6H_{12}$, $C_{28}H_{53}N_2PPtS_4.C_6H_{12}$, has been determined from three-dimensional intensities, measured on a computer-controlled Philips PW 1100 single-crystal diffractometer (2818 observed reflexions). The crystals are monoclinic, space group $P2_1/c$, with $a = 18·275$ (2), $b = 17·895$ (2), $c = 13·513$ (1) Å, $\beta = 111·20$ (1)° and $Z = 4$. The structure was solved by direct phase determination with *MULTAN*. The positional and thermal parameters of the atoms were refined by full-matrix least-squares calculations to a final $R = 0·048$ ($R_w = 0·036$). The Pt atom is in square-planar coordination with one P and three S atoms, while the two dithiocarbamato ligands are not equivalent, one being bidentate and the other unidentate. The Pt—S distances are in the range 2·326–2·364 Å, while in the dithiocarbamato ligands the C—S distances range between 1·68 and 1·79 Å and the C—N distances between 1·30 and 1·45 Å. In the phosphine ligand the P—C distances are 1·84, 1·86 and 1·87 Å.

Introduction

The reaction products of square-planar $[M(S—S)_2]$ complexes {where M = Pd or Pt; $(S—S)^- = (S_2PR_2)^-$, $(S_2CNR_2)^-$, $(S_2COR)^-$ or $[S_2P(OR)_2]^-$} with tertiary phosphines have attracted considerable attention in the past decade (*e.g.* Fackler, Seidel & Fetchin, 1968; Alison, Stephenson & Gould, 1971; Alison & Stephenson, 1973; Lin, Chen & Fackler, 1978). Recently, the interest was extended to the reaction products of bis(*N*-alkyldithiocarbamato)platinum(II) complexes with tertiary phosphines and some new compounds have been prepared at the Inorganic Chemistry Laboratory of the University of Thessaloniki (Katsoulos, Manoussakis & Tsipis, 1978). Spectroscopic methods indicated the coexistence of two different kinds of $RHNCS_2^-$ ligands, one acting as bidentate and the other as unidentate. To determine the true molecular structure of these complexes, it was considered advisable to carry out a detailed X-ray structure investigation of a representative member of the group, bis(*N*-*tert*-butyldithiocarbamato)(tricyclohexylphosphine)platinum(II) cyclohexane solvate (BDCPP hereafter).

Experimental

Pure, yellowish-white needles of BDCPP were kindly provided by Professor G. Manoussakis, Dr C. Tsipis and Mr G. Katsoulos of the Inorganic Chemistry Laboratory. A prism with dimensions 0·06 × 0·06 × 0·4 mm was selected and centred on our computer-controlled Philips PW 1100 four-circle single-crystal diffractometer. Accurate cell parameters were obtained by least squares from direct measurement on the diffractometer of the θ angles of 56 reflexions with large θ values. The density of the crystals was measured by flotation.

Three-dimensional intensity data were collected with a scintillation counter on the PW 1100 diffractometer in the $\theta/2\theta$ scan mode, using Mo $K\alpha$ radiation, monochromatized with a graphite monochromator. The intensities of 16 985 reflexions in the range 3–25° were examined and 6530 of these, with $I_{peak} - 2\sqrt{I_{peak}} > I_{background}$, were measured, the rest being too weak for reliable measurement. Subsequent averaging gave 2818 unique reflexions with $I > 2\sigma(I)$ (maximum h, k, l = 21, 20, ±16). These reflexions were considered as observed and included in all further computations. Integrated intensities were converted to $|F_o|$ values in the usual way, using the special measurement treatment

Table 1. *Crystal data for BDCPP*

Here and throughout this paper the e.s.d.'s are given in parentheses and refer to the last digit.

$C_{28}H_{53}N_2PPtS_4.C_6H_{12}$
Monoclinic
$a = 18·275$ (2) Å
$b = 17·895$ (2)
$c = 13·513$ (1)
$\beta = 111·20$ (1)°
$V = 4120·10$ Å^3

FW = 856·23
Space group $P2_1/c$
$Z = 4$
$F(000) = 1760$
$\rho_{calc.} = 1·38$ Mg m^{-3}
$\rho_{meas.} = 1·35$
$\mu = 3·7$ mm^{-1}
λ(Mo $K\alpha_1$) = 0·70930 Å

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program *DATRED* (Main, 1970). No absorption correction of the intensities was applied. For further calculations the programs of the *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1974) and XRAY (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) systems were used. Crystal data are given in Table 1.

Structure determination and refinement

The structure was solved by direct phase determination with the *MULTAN* system. The phases of 300 strong reflexions were determined and on the corresponding E map it was possible to recognize the square-planar group of the heavy atoms. A structure factor calculation at this stage, including these atoms with approximate individual isotropic temperature factors and using all the reflexions, gave R = 0·189. The remaining atoms were then located by successive difference Fourier syntheses. Refinement of the structure was carried out by full-matrix least-squares calculations. The atomic scattering factors for all the atoms and anomalous-dispersion corrections for Pt, S and P were taken from *International Tables for X-ray Crystallography* (1974). A single scale factor was used for the whole set of reflexion data. Six cycles of refinement with isotropic temperature factors and all the non-hydrogen atoms of the BCDPP molecule reduced R to 0·093. A difference Fourier synthesis at this stage revealed six more peaks, whose height and geometrical arrangement indicated clearly that they corresponded to the carbon atoms of a cyclohexane molecule, not directly bonded to the main BDCPP molecule. Including these atoms in the least-squares refinement and using isotropic temperature factors for all the atoms reduced the R factor to 0·085. However, the determined temperature coefficients of the cyclohexane molecule were abnormally high, about four to five times greater than the values for the carbon atoms in the main BDCPP molecule. As a result, the position of the cyclohexane molecule was ill-defined and this was indicative of disorder and/or partial occupancy of the solvate molecule. Using different values for the population parameter of the solvate molecule in the least-squares refinement, the best results were obtained for complete occupancy, and so the high temperature coefficients must be due to disorder.

Next, anisotropic temperature coefficients were introduced for the atoms of the main BDCPP molecule while for the atoms of the solvate molecule isotropic temperature coefficients were used. Also, the weighting scheme of the University of Washington, $w = 1/\{\max.\ \text{value}\ [6(F), aF]\}$ with a = 0·015, which best suited our data, was applied. The R factor reduced at the end of this stage to R = 0·057 (R_w = 0·048). Then, the positions of the hydrogen atoms in the main BDCPP molecule were calculated, assuming bond distances C—H = 1·08 and N—H = 1·05 Å. The positions found corresponded to maxima on the difference Fourier synthesis. No attempt was made to locate the H atoms of the cyclohexane molecule, because of the large thermal vibration of its C atoms.

In the final stage of refinement the located H atoms were kept fixed at their calculated positions, with their individual isotropic temperature coefficients equal to the equivalent isotropic temperature coefficients of the atoms to which they are bonded. At convergence the R factor reduced to 0·048 (R_w = 0·036) and the mean shift/error ratio to 0·06. The final positional and equivalent isotropic thermal parameters for the non-hydrogen atoms are given in Table 2 and those of the H

Table 2. *Atomic coordinates and equivalent isotropic temperature factors for the non-hydrogen atoms in BDCPP*

	x	y	z	B (Å^2)
Pt	0·7159 (0)	0·3470 (0)	0·5841 (0)	3·8
S(1)	0·6814 (2)	0·2296 (2)	0·6275 (3)	4·1
S(2)	0·5390 (2)	0·1434 (2)	0·6001 (3)	5·3
S(3)	0·6514 (2)	0·3304 (2)	0·3992 (2)	4·5
S(4)	0·7424 (2)	0·4541 (2)	0·5055 (2)	4·7
P	0·7796 (2)	0·3806 (2)	0·7567 (3)	3·7
N(1)	0·5391 (7)	0·2872 (6)	0·5431 (7)	3·7
N(2)	0·6818 (6)	0·4412 (5)	0·2902 (7)	4·9
C(1)	0·5767 (7)	0·2262 (8)	0·5852 (8)	3·9
C(2)	0·4555 (9)	0·3023 (8)	0·5032 (11)	5·0
C(3)	0·4195 (10)	0·2847 (9)	0·5850 (14)	9·1
C(4)	0·4169 (10)	0·2567 (11)	0·4001 (13)	8·7
C(5)	0·4456 (10)	0·3854 (9)	0·4733 (13)	8·2
C(6)	0·6907 (7)	0·4155 (6)	0·3837 (9)	4·3
C(7)	0·7080 (10)	0·5109 (8)	0·2606 (10)	5·5
C(8)	0·6808 (14)	0·5137 (10)	0·1414 (12)	12·5
C(9)	0·7963 (11)	0·5167 (9)	0·3089 (14)	9·0
C(10)	0·6736 (10)	0·5787 (9)	0·2985 (12)	7·9
C(11)	0·8546 (7)	0·4526 (7)	0·7647 (9)	4·1
C(12)	0·9187 (8)	0·4240 (8)	0·7285 (10)	5·1
C(13)	0·9654 (9)	0·4922 (9)	0·7100 (12)	7·2
C(14)	1·0006 (10)	0·5363 (10)	0·8132 (13)	7·4
C(15)	0·9366 (11)	0·5629 (9)	0·8522 (11)	6·5
C(16)	0·8895 (8)	0·4961 (7)	0·8685 (11)	5·4
C(17)	0·8236 (7)	0·3007 (7)	0·8475 (8)	3·4
C(18)	0·8730 (8)	0·2514 (8)	0·8063 (10)	5·0
C(19)	0·8957 (9)	0·1796 (7)	0·8755 (11)	6·5
C(20)	0·9382 (10)	0·1981 (10)	0·9901 (13)	7·3
C(21)	0·8920 (9)	0·2495 (10)	1·0303 (10)	6·3
C(22)	0·8672 (7)	0·3223 (7)	0·9625 (9)	4·5
C(23)	0·7128 (7)	0·4246 (7)	0·8136 (8)	3·4
C(24)	0·6469 (8)	0·3720 (7)	0·8116 (10)	4·9
C(25)	0·5912 (9)	0·4058 (8)	0·8599 (11)	6·0
C(26)	0·5597 (9)	0·4818 (11)	0·8095 (12)	7·1
C(27)	0·6249 (10)	0·5347 (8)	0·8128 (11)	6·3
C(28)	0·6780 (8)	0·4988 (8)	0·7597 (10)	5·0
C(29)	0·1268 (29)	0·2429 (35)	0·8324 (43)	28·1
C(30)	0·1594 (18)	0·1970 (17)	0·9342 (31)	17·9
C(31)	0·1745 (20)	0·2368 (24)	1·0332 (29)	20·4
C(32)	0·2070 (24)	0·3114 (26)	1·0435 (33)	24·1
C(33)	0·1582 (21)	0·3444 (22)	0·9425 (38)	21·9
C(34)	0·1351 (25)	0·3076 (29)	0·8340 (39)	24·1

Table 3. *Atomic coordinates (calculated) for the hydrogen atoms in BDCPP*

	x	*y*	*z*
H(N1)	0·575	0·332	0·537
H(N2)	0·651	0·406	0·227
H1(C3)	0·429	0·226	0·606
H2(C3)	0·448	0·319	0·655
H3(C3)	0·357	0·296	0·555
H1(C4)	0·442	0·273	0·342
H2(C4)	0·427	0·198	0·417
H3(C4)	0·355	0·268	0·370
H1(C5)	0·475	0·420	0·542
H2(C5)	0·470	0·397	0·413
H3(C5)	0·383	0·397	0·444
H1(C8)	0·706	0·467	0·113
H2(C8)	0·618	0·511	0·108
H3(C8)	0·700	0·566	0·119
H1(C9)	0·815	0·514	0·394
H2(C9)	0·822	0·471	0·281
H3(C9)	0·816	0·569	0·287
H1(C10)	0·610	0·576	0·263
H2(C10)	0·692	0·576	0·384
H3(C10)	0·693	0·631	0·277
H(C11)	0·816	0·489	0·704
H1(C12)	0·958	0·388	0·789
H2(C12)	0·893	0·393	0·655
H1(C13)	1·012	0·473	0·685
H2(C13)	0·926	0·528	0·649
H1(C14)	1·041	0·501	0·873
H2(C14)	1·032	0·584	0·799
H1(C15)	0·963	0·592	0·927
H2(C15)	0·898	0·600	0·794
H1(C16)	0·928	0·460	0·929
H2(C16)	0·843	0·516	0·893
H(C17)	0·771	0·271	0·842
H1(C18)	0·840	0·236	0·725
H2(C18)	0·925	0·281	0·810
H1(C19)	0·843	0·149	0·868
H2(C19)	0·933	0·145	0·848
H1(C20)	0·949	0·147	1·036
H2(C20)	0·993	0·224	0·999
H1(C21)	0·840	0·221	1·029
H2(C21)	0·927	0·265	1·111
H1(C22)	0·829	0·355	0·991
H2(C22)	0·919	0·354	0·969
H(C23)	0·750	0·435	0·895
H1(C24)	0·614	0·357	0·730
H2(C24)	0·672	0·322	0·855
H1(C25)	0·542	0·368	0·847
H2(C25)	0·622	0·413	0·944
H1(C26)	0·522	0·473	0·728
H2(C26)	0·526	0·507	0·852
H1(C27)	0·600	0·586	0·771
H2(C27)	0·660	0·547	0·895
H1(C28)	0·644	0·488	0·677
H2(C28)	0·725	0·537	0·766

atoms in Table 3. Interatomic distances and bond angles are given in Tables 4 and 5.*

Table 4. *Interatomic distances* (Å) *in BDCPP*

PtS_3P square-planar group		Tricyclohexylphosphine ligand	
Pt–S(1)	2·329 (4)	P–C(11)	1·86 (1)
Pt–S(3)	2·364 (3)	P–C(17)	1·87 (1)
Pt–S(4)	2·326 (4)	P–C(23)	1·84 (1)
Pt–P	2·278 (3)		
Unidentate *N*-dithiocarbamato ligand		Bidentate *N*-dithiocarbamato ligand	
S(1)–C(1)	1·79 (1)	S(3)–C(6)	1·73 (1)
C(1)–S(2)	1·68 (1)	S(4)–C(6)	1·72 (1)
C(1)–N(1)	1·31 (2)	C(6)–N(2)	1·30 (2)
N(1)–C(2)	1·45 (2)	N(2)–C(7)	1·44 (2)
C(2)–C(3)	1·51 (3)	C(7)–C(8)	1·51 (2)
C(2)–C(4)	1·55 (2)	C(7)–C(9)	1·51 (2)
C(2)–C(5)	1·54 (2)	C(7)–C(10)	1·54 (2)

First cyclohexyl ring: average C–C distance = 1·53 (2)
Second cyclohexyl ring: average C–C distance = 1·52 (2)
Third cyclohexyl ring: average C–C distance = 1·53 (2)

Table 5. *Bond angles* (°) *in BDCPP*

PtS_3P square-planar group		Tricyclohexylphosphine ligand	
S(1)–Pt–S(3)	94·3 (0·1)	Pt–P–C(11)	109·6 (0·4)
S(1)–Pt–P	93·7 (0·1)	Pt–P–C(17)	114·3 (0·4)
S(1)–Pt–S(4)	168·2 (0·1)	Pt–P–C(23)	111·8 (0·3)
S(3)–Pt–S(4)	74·2 (0·1)	C(11)–P–C(17)	111·1 (0·5)
S(4)–Pt–P	97·8 (0·1)	C(11)–P–C(23)	105·4 (0·6)
S(3)–Pt–P	171·8 (0·1)	C(17)–P–C(23)	104·3 (0·6)
		P–C(11)–C(12)	112·9 (0·9)
		P–C(11)–C(16)	117·5 (1·0)
		P–C(17)–C(18)	112·3 (0·9)
		P–C(17)–C(22)	114·9 (0·8)
		P–C(23)–C(24)	111·6 (0·9)
		P–C(23)–C(28)	113·3 (1·0)
Unidentate *N*-dithiocarbamato ligand		Bidentate *N*-dithiocarbamato ligand	
S(1)–C(1)–S(2)	115·5 (0·7)	S(3)–C(6)–S(4)	110·3 (0·7)
S(1)–C(1)–N(1)	116·3 (1·1)	S(3)–C(6)–N(2)	121·2 (0·8)
S(2)–C(1)–N(1)	128·1 (1·0)	S(4)–C(6)–N(2)	128·2 (0·9)
C(1)–N(1)–C(2)	129·5 (1·2)	C(6)–N(2)–C(7)	129·6 (0·9)
N(1)–C(2)–C(3)	111·6 (1·1)	N(2)–C(7)–C(8)	108·1 (1·2)
N(1)–C(2)–C(4)	108·2 (1·3)	N(2)–C(7)–C(9)	110·5 (1·1)
N(1)–C(2)–C(5)	106·7 (1·2)	N(2)–C(7)–C(10)	111·9 (1·4)
C(3)–C(2)–C(4)	111·8 (1·3)	C(8)–C(7)–C(9)	110·4 (1·7)
C(3)–C(2)–C(5)	110·7 (1·4)	C(8)–C(7)–C(10)	108·3 (1·3)
C(4)–C(2)–C(5)	107·6 (1·2)	C(9)–C(7)–C(10)	107·7 (1·2)

First cyclohexyl ring: average C–C–C angle = 109·9
Second cyclohexyl ring: average C–C–C angle = 110·8
Third cyclohexyl ring: average C–C–C angle = 111·3

Description of the structure and discussion

A projection of the asymmetric unit of BDCPP along [010] is given in Fig. 1. The characteristic feature of the molecules is that the central Pt atom is in square-planar coordination with one P and three S atoms. The same coordination has been confirmed for all the phosphine

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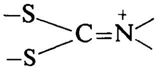
* Lists of structure factors, anisotropic thermal parameters and torsion angles of the tricyclohexylphosphine part of BDCPP have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34637 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

adducts of compounds with the general formula $M(S{-}S)_2$, where $M = Pt$ or Pd and $(S{-}S)^- = (S_2CNR_2)^-$, $(S_2COR)^-$, $[S_2P(OEt)_2]^-$ or $(S_2PR_2)^-$ (Alison & Stephenson, 1973). The two $RHNCS_2^-$ ligands in BDCPP are not equivalent; one is linked as bidentate and the other as unidentate, a situation already encountered in analogous compounds studied by spectroscopic methods (Katsoulos, Manoussakis & Tsipis, 1978). The same situation has also been established in the solid state by X-ray analysis on $[Pd(S_2PPh_2)_2PPh_3]$, as cited by Alison & Stephenson (1973).

In the PtS_3P^- group the deviation of the Pt atom from the mean plane, defined by the atoms S(1), S(3), S(4) and P, is within the average estimated standard deviation of these atoms, in distinction to bis(dithiocarbamato)nickel (Gasparri-Fava, Nardelli & Villa, 1967), where the central metal atom deviates significantly from the mean plane. As expected from other dithiocarbamate structures, the S_2CN^- groups of both ligands are strictly planar within experimental error. These two planes, which also contain essentially the Pt atom, form between them an angle of 67·4°.

The determined Pt—S bond distances range between 2·326 and 2·364 with mean value 2·340 Å, which compares well with the corresponding value 2·335 Å reported for $Pt[S_2CN(i\text{-}Bu)_2]_2(PMePh)_2$ by Lin, Chen & Fackler (1978). The distance of the dangling S(2) atom from the Pt atom is 4·928 Å. Moreover, no intermolecular Pt···S distance shorter than the sum of the corresponding van der Waals radii of the S and Pt atoms (3·52 Å, Bondi, 1964) was found. The angle S—Pt—S formed by the bidentate ligand is 74·2°, in good agreement with the corresponding mean value 73·5°, found in $Pt(Bu_2dtc)_2I_2$ (Willemse, Gras, Wijnhoven & Beurskens, 1973). The determined Pt—P distance in BDCPP is 2·278 Å, which agrees well with the corresponding value 2·280 Å, found in $Pt(S_2CO)(Ph_3P)_2$ by Lin, Chen & Fackler (1978). The mean S—C(N) bond distance in the bidentate ligand is 1·73 Å, whereas in the unidentate it is 1·79 Å. These values are in agreement with corresponding values found in other dithiocarbamate structures, *e.g.* in bis(*N*,*N*-di-*n*-propyldithiocarbamato)nickel(II) (Peyronel & Pignedoli, 1967), and lie between the usually accepted values for single and double S—C bond distances (1·81 and 1·558 Å), indicating thus some partial double-bond character. The same is true for the C(S)—N bond distances, which are 1·30 and 1·31 Å and lie between the accepted values for single- and double-bond distances (1·475 and 1·290 Å). The virtual equality of the C—N bond lengths in both dithiocarbamate ligands of BDCPP suggests that the contribution of the resonance form



is the same for both bidentate and unidentate ligands.

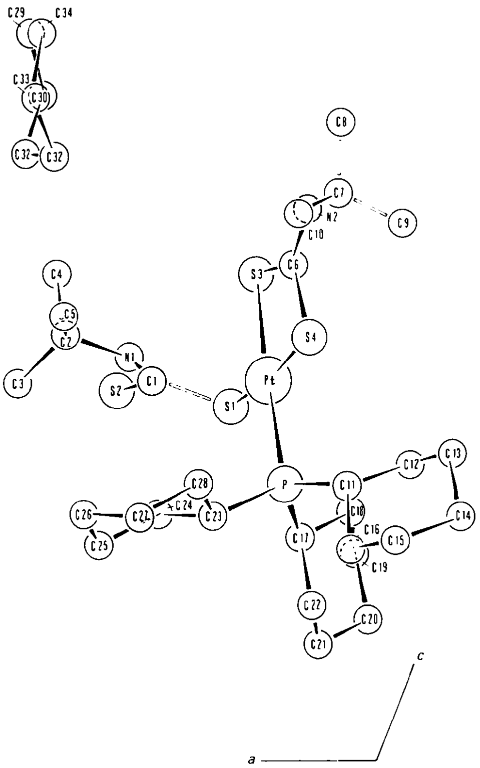


Fig. 1. Projection of the asymmetric unit of BDCPP along [010].

The geometrical features of the tricyclohexylphosphine ligand of BDCPP are normal. The P atom is tetrahedrally coordinated by the Pt and three C atoms with mean tetrahedral angle 109·4° and mean P—C bond distance 1·86 Å. These values are in excellent agreement with the corresponding mean values 109·2° and 1·86 Å, reported for $[\{Pt(SiEt_3)(\mu\text{-}H)[(C_6H_{11})_3P]\}_2]$ (Ciriano *et al.*, 1978). The mean C—C bond distances for the three cyclohexane rings are 1·53, 1·52 and 1·53 Å, respectively, while the corresponding mean C—C—C bond angles are 109·9, 110·8 and 111·3°. It may be noted that the mean torsion angles are 58·8, 56·5 and 55·1°.* These values agree well with the corresponding theoretical values (58·9, 56·6 and 55·2°) expected for regular cyclohexane rings, with valence angles equal to the observed ones.

The intermolecular contacts in BDCPP are normal, with the exception of one distance, namely that between the dangling S(2) atom of the unidentate ligand of a molecule and the N(1) atom of the bidentate ligand of an adjacent molecule $(x, \frac{1}{2}-y, -\frac{1}{2}+z)$, which is 3·29 Å, considerably shorter than the sum of the corresponding van der Waals radii (3·55 Å, Bondi, 1964). This may be due to a hydrogen-bond interaction N—H···S, which is indicated by the very short H···S distance 2·32 Å (the sum of the corresponding van der

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* See previous footnote.

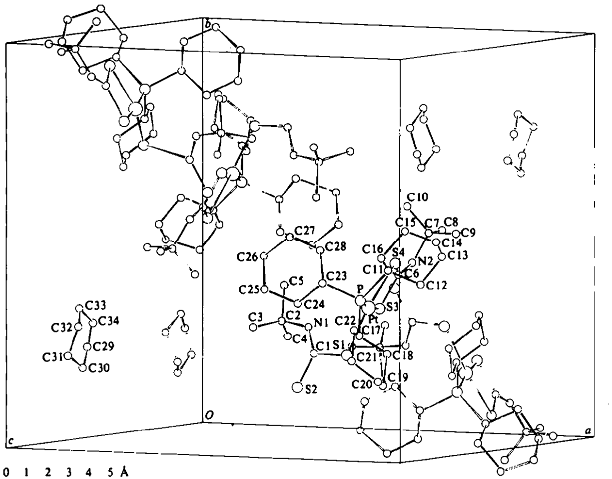

Fig. 2. Clinographic projection of BDCPP showing the molecular packing.

Waals radii is 3·0 Å) and the N—H···S angle of 155°. The molecular packing is shown in Fig. 2.

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trans-Bis(diethylphenylphosphine)dimesitylcobalt(II)

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Abstract

$C_{38}H_{52}CoP_2$, $[Co(C_9H_{11})_2(C_{10}H_{15}P)_2]$, $M_r = 629\cdot71$, monoclinic, $P2_1/c$, $a = 10\cdot695$ (8), $b = 9\cdot310$ (8), $c = 18\cdot317$ (16) Å, $\beta = 107\cdot21$ (6)°, $U = 1742$ Å^3, $Z = 2$, $D_x = 1\cdot200$ Mg m^{-3}, μ(Mo $K\alpha$) $= 0\cdot573$ mm^{-1}. The Co atom, lying on a centre of symmetry, is planar, four-coordinated by two mesityl ligands and two diethylphenylphosphine groups. All H atoms were located in this redetermination, which was refined for 296 parameters to $R = 0\cdot042$ for 2487 independent reflexions.

Introduction

An earlier crystal structure determination (Owston & Rowe, 1963) of *trans*-bis(diethylphenylphosphine)dimesitylcobalt(II) established planar four-coordination for the low-spin Co atom, the fifth and sixth positions of a potential octahedral geometry apparently being blocked by the α-methyl groups of the mesityl ligands. A highly anisotropic g tensor in which $g_x = 3\cdot72$, $g_y = 1\cdot96$, $g_z = 1\cdot74$, where the molecular frame x,y,z is oriented almost exactly parallel to Co—P, Co—mesityl and the normal to the coordination plane, respectively,

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