

lactatobis(*N*-isopropyl-2-methyl-1,2-propanediamine)-copper(II) L-lactate monohydrate (Ahlgren & Hämäläinen, 1978), the Cu atom has normal 4 + 1 coordination. The trigonal-bipyramidal configuration can be considered a distortion of the square-planar arrangement through movement of the two *trans* N atoms [N(2) and N(4)] within the plane containing them, Cu, and the atom at the apex of the square pyramid [N(5)] to positions such that the angles made with the Cu and the apical atom are about 120°. In this structure N(2)—Cu—N(5) is 133.8 (15)° and N(4)—Cu—N(5) is 127.1 (14)°.

Each of the five-membered Cu—diamine rings is in an unsymmetrical *gauche* configuration. The complex cation has approximate C₂ symmetry, the axis passing through N(5) and Cu. All interionic contacts are normal, the shortest being 2.93 (2) Å between N(2) and O(3).

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Bis(tri-*tert*-butylphosphine)platinum(0)

BY KELLY J. MOYNIHAN AND CHUNG CHIEH

Guelph–Waterloo Centre for Graduate Work in Chemistry, Department of Chemistry, University of Waterloo, Waterloo, Ontario, Canada N2L 3G1

AND RAM G. GOEL

Guelph–Waterloo Centre for Graduate Work in Chemistry, Department of Chemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1

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Abstract. C₂₄H₅₄P₂Pt, [Pt(C₁₂H₂₇P)₂], monoclinic, P2₁/n, *a* = 8.630 (2), *b* = 13.733 (7), *c* = 12.049 (6) Å, β = 93.74 (3)°, *Z* = 2, *D_m* = 1.40, *D_x* = 1.396 Mg m⁻³, μ(Mo Kα) = 5.27 mm⁻¹, λ(Mo Kα) = 0.71069 Å. The structure was refined from 1804 observed reflections measured on a diffractometer; *R* = 0.046. The Pt—P distance is 2.249 (3) Å.

Introduction. Although crystal structures of two-coordinated complexes of zerovalent Pt or Pd with phosphines have been reported (Otsuka, Yoshida, Matsumoto & Nakatsu, 1976; Immirzi, Musco, Zambelli & Carturan, 1975), no data on this type of

compound involving the bulky tri-*tert*-butylphosphine group were available. Therefore, a structural determination was carried out for the title compound, Pt[P(*tert*-Bu)₃]₂.

The space group was determined by photographic methods. The crystal used, which was approximately cubic with edges of 0.11 mm, was mounted on a Syntex P2₁ automatic diffractometer. 15 automatically centred reflections were used in a least-squares refinement to give the cell constants and the orientation matrix. Two reflections (011 and 110) were measured after every 48 reflections to monitor the stability of the data collection; the intensities of these checking

Table 1. Atomic coordinates ($\times 10^4$) and temperature factors ($\times 10^3$) with *e.s.d.*'s in parentheses

	x	y	z	U_{eq} (\AA^2)*
Pt	0	0	0	46
P	-525 (3)	1071 (2)	1337 (2)	38
C(11)	-2734 (13)	1200 (10)	1416 (11)	63
C(12)	-3479 (15)	1276 (13)	216 (13)	94
C(13)	-3346 (17)	273 (11)	1929 (16)	92
C(14)	-3283 (17)	2070 (13)	2110 (15)	100
C(21)	393 (19)	641 (11)	2752 (10)	71
C(22)	-240 (21)	1140 (13)	3795 (15)	94
C(23)	58 (21)	-489 (11)	2824 (12)	80
C(24)	2166 (16)	744 (13)	2756 (12)	85
C(31)	332 (14)	2334 (8)	1047 (10)	58
C(32)	1966 (18)	2171 (12)	609 (15)	90
C(33)	-659 (23)	2791 (12)	37 (15)	104
C(34)	441 (20)	3050 (10)	2022 (13)	84

$$* U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{13} \cos \beta).$$

reflections fluctuated within $\pm 4\%$. Of 2534 reflections with $2\theta < 50^\circ$, 1804 had net intensities greater than $\sigma(I)$ and were considered to be observed. Lorentz and polarization corrections were applied to derive the structure amplitudes. No absorption correction was made, $\mu R = 0.53$.

The measured density indicated that there were two molecules per unit cell and the Patterson map showed a partial image of the molecule. A least-squares refinement based on isotropic temperature factors for all non-hydrogen atoms gave an R ($= \sum |F_o| - |F_c| / \sum |F_o|$) factor of 0.08 for all observed reflections. The unobserved reflections were given zero weight in the refinement. When anisotropic temperature factors were introduced, the R and R_w ($= [\sum w(|F_o| - |F_c|)^2 / \sum w|F_o|^2]^{1/2}$) were reduced to 0.046 and 0.055 respectively, where $w^{-1} = (80 - F_o + 0.0125 F_o^2)$. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$. Atomic scattering factors from Cromer & Waber (1965) with anomalous dispersion for Pt (Cromer, 1965) were used. The R factor including all reflections was 0.065. The difference Fourier map (max. 1.3 e \AA^{-3}) at this stage did not lead us to feel confident about the locations of the H atoms, which were therefore not included in the structure factor calculations. Atomic coordinates are given in Table 1.*

Discussion. The structure consists of individual molecules packed in the crystal *via* van der Waals forces. The molecule has a centre of symmetry on the Pt atom and, therefore, the substituents on the P atoms exist in a staggered conformation. The molecular centre of symmetry coincides with that of the unit cell. The

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34766 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

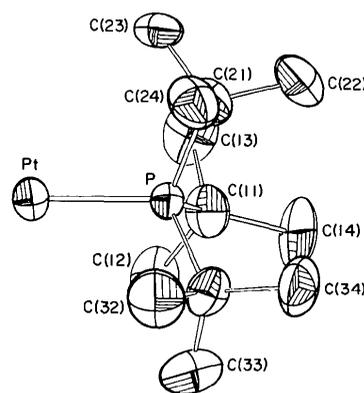


Fig. 1. An ORTEP drawing (Johnson, 1965) of half a molecule with the atomic numbering.

Table 2. Bond lengths (\AA) and angles ($^\circ$) with *e.s.d.*'s

Pt-P	2.249 (3)	P-Pt-P	180 (0)
P-C(11)	1.92 (1)	Pt-P-C(11)	110.0 (3)
P-C(21)	1.93 (1)	Pt-P-C(21)	110.1 (4)
P-C(31)	1.93 (1)	Pt-P-C(31)	111.2 (3)
		C(11)-P-C(21)	109.7 (5)
		C(11)-P-C(31)	108.6 (5)
		C(21)-P-C(31)	107.2 (5)
C(11)-C(12)	1.55 (2)	P-C(11)-C(12)	108.3 (9)
C(11)-C(13)	1.52 (2)	P-C(11)-C(13)	108.3 (9)
C(11)-C(14)	1.55 (2)	P-C(11)-C(14)	115.9 (10)
		C(12)-C(11)-C(13)	107.5 (11)
		C(12)-C(11)-C(14)	109.2 (11)
		C(13)-C(11)-C(14)	107.4 (12)
C(21)-C(22)	1.56 (2)	P-C(21)-C(22)	115.6 (10)
C(21)-C(23)	1.58 (2)	P-C(21)-C(23)	106.4 (10)
C(21)-C(24)	1.54 (2)	P-C(21)-C(24)	109.1 (10)
		C(22)-C(21)-C(23)	108.2 (12)
		C(22)-C(21)-C(24)	110.9 (12)
		C(23)-C(21)-C(24)	106.0 (12)
C(31)-C(32)	1.55 (2)	P-C(31)-C(32)	107.4 (9)
C(31)-C(33)	1.57 (2)	P-C(31)-C(33)	107.7 (9)
C(31)-C(34)	1.53 (2)	P-C(31)-C(34)	116.4 (9)
		C(32)-C(31)-C(33)	104.8 (11)
		C(32)-C(31)-C(34)	110.0 (11)
		C(33)-C(31)-C(34)	109.9 (11)

linear arrangement is a noteworthy structural feature when compared with $\text{Pt}[\text{P}(\text{C}_6\text{H}_{11})_3]_2$ and $\text{Pt}[\text{P}(\text{C}_6\text{H}_5)(\text{tert-Bu})_2]_2$. Both the latter two compounds have a bent P-Pt-P moiety with angles of 160.5 (2) and 177.0 (1) $^\circ$ respectively (Immirzi *et al.*, 1975; Otsuka *et al.*, 1976). The Pt-P bond lengths are 2.249 (3), 2.231 (4) and 2.252 (1) \AA respectively for the aforementioned compounds. These data support the claim that $\text{P}(\text{tert-Bu})_3$ is a bulky phosphine ligand which has a cone angle of 182 (2) $^\circ$ (Tolman, 1970). In contrast, the less bulky phosphines, $\text{P}(\text{C}_6\text{H}_5)(\text{tert-Bu})_2$ [cone angle 170 (2) $^\circ$], in the Pt complex adopt an eclipsed conformation.

An ORTEP drawing of half the molecule is given in Fig. 1, which shows a view from a direction perpen-

dicular to the P–Pt–P moiety. Selected bond lengths and angles are given in Table 2. Values of 115.9 (10), 115.6 (10) and 116.4 (9)° are found for \angle PC(11)–C(14), \angle PC(21)C(22) and \angle PC(31)C(34) respectively. All these angles involve a C atom pointing away from the Pt. The mean value for the remaining \angle PCC angles is 107.9 (9)°.

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Structure of Aquachlorobis[*N*-(2-pyridylmethylene)aniline]cobalt(II) Nitrate Monohydrate

BY A. MONGE

Instituto de Química Inorgánica 'Elhuyar', Serrano 113, Madrid-6, Spain

M. MARTÍNEZ-RIPOLL

Departamento de Rayos X, Instituto de Química-Física 'Rocasolano', Serrano 119, Madrid-6, Spain

E. GUTIÉRREZ-PUEBLA

Instituto de Química Inorgánica 'Elhuyar', Serrano 113, Madrid-6, Spain

AND S. GARCÍA-BLANCO

Departamento de Rayos X, Instituto de Química-Física 'Rocasolano', Serrano 119, Madrid-6, Spain

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Abstract. $[\text{Co}(\text{C}_{12}\text{H}_{10}\text{N}_2)_2\text{Cl}(\text{H}_2\text{O})]\text{NO}_3 \cdot \text{H}_2\text{O}$, $\text{C}_{24}\text{H}_{22}\text{ClCoN}_4\text{O}^+ \cdot \text{NO}_3^- \cdot \text{H}_2\text{O}$, $M_r = 556.38$, triclinic, $P1$, $a = 9.977$ (1), $b = 14.171$ (3), $c = 9.185$ (1) Å, $\alpha = 103.68$ (1), $\beta = 89.12$ (1), $\gamma = 98.27$ (1)°, $V = 1248.4$ (2) Å³, $Z = 2$, $D_c = 1.48$ Mg m⁻³. Final $R = 0.051$ for 1958 observed reflexions. The Co atom presents distorted octahedral coordination. The free water molecule connects the NO₃ group to the Cl atom and to the coordinated water molecule through hydrogen bonds.

Introduction. The crystals were provided by the Departamento de Química Analítica of the University of Granada (Spain). An orange prismatic single crystal was used to collect the intensities of 3455 reflexions up to $\theta = 25^\circ$ at 295 K on a PW 1100 four-circle diffractometer. An $\omega/2\theta$ scan technique and graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å) were used.

Two reflexions were monitored periodically and showed no crystal decomposition. The intensities were corrected for Lorentz and polarization effects and 1958 of them were considered as observed by the criterion $I > 2\sigma(I)$. The absorption effects ($\mu = 0.813$ mm⁻¹) were not corrected. Scattering factors for neutral atoms and anomalous-dispersion corrections for Co and Cl were taken from *International Tables for X-ray Crystallography* (1974).

The structure was solved by *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977). The best E map revealed ~20% of the non-hydrogen atoms. The remaining non-hydrogen atoms appeared in successive Fourier syntheses. Anisotropic full-matrix least-squares refinement with unit weights led to $R = 0.060$. A difference synthesis calculated with those reflexions within $\sin \theta/\lambda < 0.5$ Å⁻¹ showed all H atoms. In order to prevent bias on ΔF vs F_o or $\sin \theta/\lambda$, the last steps of the refinement were carried out with weights w