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trans-Dichlorobis(tris{4-[(dimethylamino)-methyl]phenyl}phosphine)platinum(II)

Martin Lutz,[a]* Anthony L. Spek,[a] Robert Kreiter,[b] Robertus J. M. Klein Gebbink[b] and Gerard van Koten[b]

[a]Bijvoet Center for Biomolecular Research, Crystal and Structural Chemistry, Utrecht University, Padualaan 8, 3584 CH Utrecht, The Netherlands, and [b]Debye Institute, Organic Chemistry and Catalysis, Utrecht University, Padualaan 8, 3584 CH Utrecht, The Netherlands

Correspondence e-mail: m.lutz@chem.uu.nl

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.020
wR factor = 0.050
Data-to-parameter ratio = 20.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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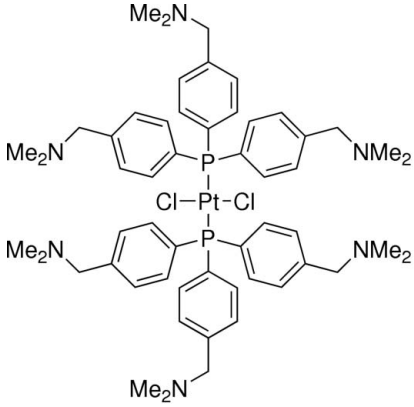
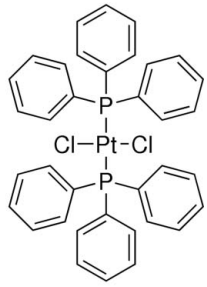
Crystals of the title compound, $[PtCl_2(C_{27}H_{36}N_3P)_2]$, as the *trans*-isomer were obtained from the originally formed *cis*-isomer after standing for more than four months in solution. In the crystal structure, the Pt atom is located on an inversion centre, which makes the molecular structure similar to the triphenylphosphine analogue. The amino groups are pyramidal and do not interact with Pt atoms or hydrogen-bond donors.

Comment

The *cis*-isomer of the title compound was formed by a reaction of the free phosphine with $[PtCl_2(COD)]$ (Kreiter *et al.*, 2005). After several months of standing in solution, the complex isomerized to the *trans*-complex (I), which is described here.



(I)



(II)

In the crystal structure, the Pt atom is located on an inversion centre and therefore has a completely planar coordination geometry. The Pt—P distance of 2.3208 (5) Å is comparable to those in the also centrosymmetric triphenyl phosphine analogue (II), with 2.3163 (11) and 2.3187 (12) Å, respectively, for two different crystals (Johansson & Otto, 2000). The same holds for the Pt—Cl distance of 2.3038 (5) Å in (I) and 2.2997 (11) Å for both crystals of (II). The Pt—P—C angles of 111.07 (7), 113.60 (7) and 117.33 (7)° in (I) are also very similar to those in (II). The orientations of the benzene rings in (I), with Pt—P—C—C torsion angles of −41.2 (2), −16.5 (2) and −71.20 (18)°, are identical to those in (II), with values of 18.0 (3), 42.3 (3) and 68.3 (3)°; the inversion of the signs is present as a space-group symmetry operation.

The *N*-methyl groups of (I) are in *trans*-conformations, with C—N—C—C torsion angles of 176.3 (2), −176.6 (2) and −177.3 (2)°. The benzyl groups have *gauche* conformations, with C—C—C—N torsion angles of 66.2 (3), 62.1 (3) and 45.6 (3)°. Because there are no interactions of the amino N

atoms with metal atoms or hydrogen-bond donors, we assume that the slight variation in these torsion angles is due only to crystal packing effects. The angles at the amino N atoms of 108.7 (2)–112.2 (2)° indicate a pyramidal geometry.

Experimental

Tris{4-[(dimethylamino)methyl]phenyl}phosphine (38.2 mg), abbreviated as $P(NC(H))_3$, was dissolved in CH_2Cl_2 (20 ml) and a solution of $[PtCl_2(COD)]$ (16.5 mg) in CH_2Cl_2 was added and the resulting solution stirred for 1 h at room temperature, followed by heating to reflux temperature for 15 min, resulting in quantitative formation of *cis*-$[PtCl_2(P(NC(H))_3)_2]$ (^{31}P NMR, J_{Pt-P} = 3677 Hz). Crystals suitable for X-ray diffraction were obtained after standing for more than 4 months in a CH_2Cl_2/hexane mixture (1:1 *v*/*v*) shielded from direct sunlight. The resulting compound showed *trans*-$[PtCl_2(P(NC(H))_3)_2]$ as the single constituent. The solution from which the crystals were taken contained no remaining Pt-complex. ^{31}P NMR of some of the crystals dissolved in $CDCl_3$ showed a resonance at 20.0 p.p.m. with a coupling constant J_{Pt-P} = 2615 Hz. Clearly, the complex has isomerized slowly to form the *trans* complex.

Crystal data

$[PtCl_2(C_{27}H_{36}N_3P)_2]$	D_x = 1.407 Mg m^{-3}
M_r = 1133.11	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 43969 reflections
a = 13.1122 (1) Å	θ = 1.0–27.5°
b = 17.6200 (1) Å	μ = 2.82 mm^{-1}
c = 11.6180 (1) Å	T = 150 (2) K
β = 94.9554 (4)°	Rod, yellow
V = 2674.16 (3) Å^3	0.27 × 0.09 × 0.09 mm
Z = 2	

Data collection

Nonius KappaCCD diffractometer	5028 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.038
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)	θ_{max} = 27.5°
T_{min} = 0.62, T_{max} = 0.78	$h = -17 \rightarrow 17$
57383 measured reflections	$k = -22 \rightarrow 22$
6131 independent reflections	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0197P)^2 + 2.3473P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.020	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.050	$\Delta\rho_{max}$ = 0.84 e Å^{-3}
S = 1.04	$\Delta\rho_{min}$ = −0.50 e Å^{-3}
6131 reflections	
301 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Pt1—Cl1	2.3038 (5)	P1—C21	1.830 (2)
Pt1—P1	2.3208 (5)	P1—C31	1.821 (2)
P1—C11	1.818 (2)		
Cl1—Pt1—P1^i	87.57 (2)	Cl1—Pt1—P1	92.43 (2)
Pt1—P1—C11—C16	−41.2 (2)	C38—N3—C37—C34	−177.3 (2)
Pt1—P1—C21—C26	−16.5 (2)	C15—C14—C17—N1	66.2 (3)
Pt1—P1—C31—C32	−71.20 (18)	C23—C24—C27—N2	62.1 (3)
C19—N1—C17—C14	176.3 (2)	C33—C34—C37—N3	45.6 (3)
C28—N2—C27—C24	−176.6 (2)		

Symmetry code: (i) $-x+1, -y+1, -z+1$.

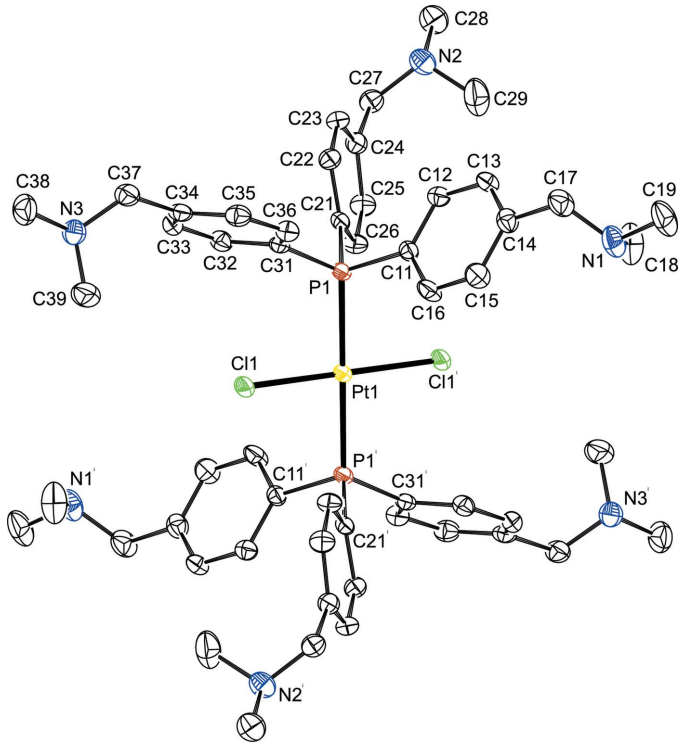


Figure 1
Displacement ellipsoid plot of (I) (50% probability level). H atoms are omitted for clarity. Primed atoms are generated by the symmetry operation $(1-x, 1-y, 1-z)$.

H atoms were introduced in geometrically idealized positions (C—H = 0.99–1.00 Å) and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl groups and $U_{iso}(H) = 1.2U_{eq}(C)$ for all other H atoms. The methyl groups were allowed to rotate but not to tip.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *HKL2000* (Otwinowski & Minor, 1997); data reduction: *HKL2000*, *DENZOX* (Farrugia, 2004) and *SADABS* (Sheldrick, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: manually editing *SHELXL97* output.

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