P1—Ru1—Ru2	90.03 (2)	P2—Ru2—Ru1	94.28 (2)
C3—Ru1—Ru3	109.18 (10)	Ru3—Ru2—Ru1	60.618 (7)
C1—Ru1—Ru3	90.82 (8)	C8—Ru3—Ru2	88.70 (12)
C2—Ru1—Ru3	83.26 (9)	C7—Ru3—Ru2	94.86 (9)
P1—Ru1—Ru3	148.94 (2)	C9—Ru3—Ru2	86.35 (10)
Ru2—Ru1—Ru3	59.619 (9)	P3—Ru3—Ru2	170.91 (2)
C5—Ru2—P2	102.19 (11)	C8—Ru3—Ru1	148.07 (12)
C4—Ru2—P2	94.06 (9)	C7—Ru3—Ru1	82.59 (9)
C6—Ru2—P2	91.38 (8)	C9—Ru3—Ru1	92.52 (10)
C5—Ru2—Ru3	104.27 (11)	P3—Ru3—Ru1	111.76 (2)
C4—Ru2—Ru3	92.80 (9)	Ru2—Ru3—Ru1	59.762 (9)
C6—Ru2—Ru3	81.57 (8)	P1—C10—P2	115.25 (12)
P2—Ru2—Ru3	152.44 (2)		

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93. Program used for geometric calculations: *PARST* (Nardelli, 1983).

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A Novel Cyclotriphosphazene Complex with a Short Transannular P···P Contact

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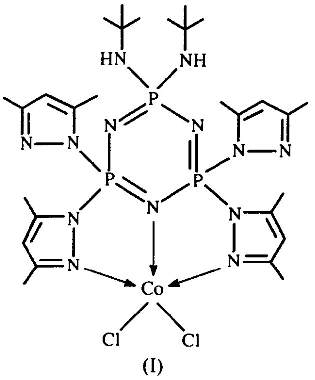
Abstract

The structure of dichloro[2,2-di-*tert*-butylamino-4,4,6,6-tetrakis(3,5-dimethylpyrazolyl)cyclotriphosphazene]cobalt(II), [CoCl$_2$(C$_{28}$H$_{48}$N$_{13}$P$_3$)], exhibits an unusual transannular P···P interaction.

Comment

Interest in heterocycles such as cyclotriphosphazenes results from their novel reactivity towards various organometallic reagents and transition metal ions, and their industrial applications (Allcock *et al.*, 1987; Chandrasekhar & Thomas, 1993). Pyrazolyl-substituted cyclotriphosphazenes have been shown to possess interesting coordination chemistry (Thomas *et al.*, 1993, 1994; Chandrasekaran *et al.*, 1994; Byun *et al.*, 1996). In an attempt to study the electronic and steric effect exerted by the substituents on the coordination behaviour of the cyclotriphosphazene moiety, we have synthesized the title compound, (I), which includes two bulky *tert*-butylamino groups.



(I)

In (I), the Co^{II} atom assumes a distorted trigonal-bipyramidal geometry comprising of two pyrazolyl N atoms, a cyclophosphazene N atom and two Cl^- ions. The trigonal plane contains two Cl^- ions and a cyclophosphazene N atom. The Co—Cl distances are simi-

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lar to those observed in $N_3P_3Ph_2Pz_4.CoCl_2$ (Thomas *et al.*, 1995). However, the cyclotriphosphazene interaction with the Co atom [2.199 (2) Å] is stronger and closer to that from the pyrazolyl N atoms [average 2.109 (2) Å]. This is in contrast to the observation made for related complexes derived from pyrazolyl cyclotriphosphazenes where the metal–cyclophosphazene-ring N-atom distance is appreciably longer than the metal–pyrazolyl N-atom bond length. This clearly illustrates that the electron-releasing effect of the *tert*-butylamino groups play an important role in the coordination ability of the cyclophosphazene-ring N atom through extended π bonding.

The cyclophosphazene ring is severely distorted from planarity. There are three sets of P—N bonds within the cyclotriphosphazene ring. The shortest distances [average 1.547 (2) Å] are associated with the P—N bonds adjacent to the P2—N2—P1 fragment which comprises the coordinated cyclophosphazene N atom. The exocyclic and endocyclic P—N bonds originating from the P atom bearing the *tert*-butylamino group are identical; this may be due to the electron flow from the *tert*-butylamino groups into the cyclophosphazene ring (Begley *et al.*, 1979). Another interesting feature in the structure is the short contact between the P atoms, P1 and P2. This is clearly the result of coordination by N2, which induces distortion in the ring and brings P1 and P2 closer. The short interaction between P1 and P2 is mainly due to the *ansa* loop arising because of the nongeminal coordination from atoms N5 and N9 to the Cu atom. This will induce substantial compression in the inorganic N_3P_3 ring. In the classical cyclophosphazene compound $N_3P_3Cl_6$, the transannular P···P distance is 2.746 (2) Å (Bullen, 1971). In the present compound, the P1···P2 distance is appreciably shorter [2.699 (2) Å]. However, it should be noted that the mean P···P interaction is 2.745 (2) Å, which is not different from that observed from $N_3P_3Cl_6$. Recently, a short P...P interaction [2.675 (1) Å] has been reported for an *ansa* compound derived from a difunctional nucleophilic reagent (Bosscher *et al.*, 1997).

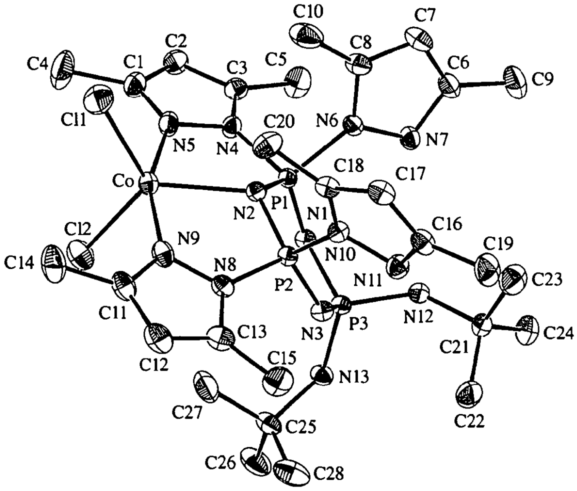

Fig. 1. The structure of (I) showing 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

The amido H12 atom participates in a stronger intramolecular hydrogen bond with an uncoordinated pyrazolyl pyridinic N atom (N7) [H12···N7 2.28 (3) Å]. Also, the C5 methyl proton H5C is involved in a bifurcated hydrogen bond with the acceptor atoms N7 and N6.

All other distances and angles are generally as expected. They correspond well with values found in the related metal complexes obtained from pyrazolyl-linked cyclotriphosphazene ligands (Thomas *et al.*, 1993, 1994).

Experimental

The reaction of cobaltous chloride with 2,2-di-*tert*-butylamino-4,4,6,6-tetrakis(3,5-dimethylpyrazolyl)cyclotriphosphazene in dichloromethane in a 1:1 molar ratio produces the title compound in nearly quantitative yield.

Crystal data

$[CoCl_2(C_{28}H_{48}N_{13}P_3)]$
$M_r = 789.53$
Monoclinic
$P2_1/c$
$a = 11.503\ (5)$ Å
$b = 17.843\ (5)$ Å
$c = 19.132\ (9)$ Å
$\beta = 106.51\ (3)°$
$V = 3765\ (3)$ Å^3
$Z = 4$
$D_x = 1.393$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11.0–18.0°$
$\mu = 0.765$ mm^{-1}
$T = 293\ (2)$ K
Prism
0.44 × 0.28 × 0.24 mm
Blue

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega–2\theta$ scans
Absorption correction: none
7210 measured reflections
6609 independent reflections
5393 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.018$
$\theta_{\rm max} = 24.99°$
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 21$
$l = 0 \rightarrow 22$
2 standard reflections every 100 reflections intensity decay: 0.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.089$
$S = 1.088$
6605 reflections
589 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2 + 2.5753P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = -0.004$
$\Delta\rho_{\rm max} = 0.286$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.288$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Co—N9	2.104 (2)	P1···P2	2.6992 (10)
Co—N5	2.113 (2)	P2—N3	1.546 (2)
Co—N2	2.199 (2)	P2—N2	1.617 (2)
Co—Cl1	2.2788 (12)	P2—N10	1.693 (2)
Co—Cl2	2.2893 (12)	P2—N8	1.701 (2)
P1—N1	1.548 (2)	P3—N3	1.615 (2)
P1—N2	1.614 (2)	P3—N13	1.618 (2)
P1—N4	1.688 (2)	P3—N12	1.620 (2)
P1—N6	1.698 (2)	P3—N1	1.634 (2)
N9—Co—N5	159.31 (8)	N1—P1—N2	118.04 (10)
N9—Co—N2	79.66 (7)	N4—P1—N6	102.30 (10)
N5—Co—N2	80.69 (7)	N3—P2—N2	118.80 (10)
N9—Co—Cl1	98.86 (6)	N10—P2—N8	101.25 (10)
N5—Co—Cl1	96.40 (7)	N13—P3—N12	105.71 (12)
N2—Co—Cl1	119.44 (6)	N3—P3—N1	110.90 (10)
N9—Co—Cl2	92.30 (6)	P1—N1—P3	119.60 (11)
N5—Co—Cl2	93.11 (6)	P1—N2—P2	113.32 (11)
N2—Co—Cl2	123.44 (6)	P2—N3—P3	125.74 (12)
Cl1—Co—Cl2	117.12 (4)		

Most of the H atoms were located from a difference Fourier map and the remainder were fixed at calculated positions. All H atoms were refined isotropically. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990) and refined on F^2 by full-matrix least-squares methods using *SHELXL*93 (Sheldrick, 1993).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *HELENA* (Spek, 1990*b*). Molecular graphics: *PLATON* (Spek, 1990*a*). Software used to prepare material for publication: *SHELXL*93.

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cis-Bis[(2-diphenylphosphinoethyl)dimethylstannyl]platinum(II)

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Abstract

In the spirocyclic title complex, $[\mathrm{PtSn_2(CH_3)_4(C_{14}H_{14}P)_2}]$, the Pt atom is situated at the centre of a slightly distorted square-planar coordination polyhedron. The Pt—P [2.283 (1) and 2.286 (1) Å] and Pt—Sn [2.5973 (5) and 2.6047 (5) Å] distances agree well with the corresponding distances in related compounds.

Comment

Bis(stannyl)bis(phosphine)palladium(II) and -platinum(II) complexes are of interest as intermediates in the metal-catalysed double stannylation of 1,3-dienes, alkynes and allenes; the oxidative addition of organodistannanes to palladium(0) and platinum(0) complexes, *e.g.* $[M^0(\mathrm{CH_2{=}CH_2})(\mathrm{PPh_3})_2]$ and $[M^0(\mathrm{PPh_3})_4]$ (M = Pd, Pt) is believed to be a crucial step in the catalytic cycle of this stannylation reaction. At present, there are only two fully characterized examples of this type of complex, namely *cis*-$[\mathrm{Pt(SnMe_3)_2\{P(C_6H_4Me\text{-}4)_3\}_2}]$ (Obora *et al.*, 1996) and *cis*-$[\mathrm{Pt\{1,1'\text{-}(SnMe_2)_2Fc\}(PPh_3)_2}]$ (Fc = $\mathrm{FeC_{10}H_8}$) (Herberhold *et al.*, 1997). We report here the crystal structure of the related complex *cis*-bis[(2-diphenylphosphinoethyl)dimethylstannyl]platinum(II), (I). Its synthesis and structural characterization by NMR studies have been published previously (Weichmann, 1982).