

C5A	-0.0193 (3)	0.1411 (7)	-0.1885 (9)	0.054 (2)
O1B	0.2474 (2)	0.3995 (3)	0.2618 (5)	0.0405 (10)
O2B	0.2578 (2)	0.4444 (3)	0.5343 (4)	0.0337 (9)
C7B	0.2563 (2)	0.4663 (4)	0.3818 (6)	0.0248 (11)
C1B	0.2638 (3)	0.5840 (4)	0.3365 (6)	0.0264 (11)
C2B	0.2178 (3)	0.6436 (5)	0.2512 (7)	0.0362 (14)
F2B	0.1661 (2)	0.5908 (4)	0.2015 (5)	0.0590 (11)
C3B	0.2222 (4)	0.7515 (6)	0.2189 (10)	0.058 (2)
C4B	0.2750 (5)	0.8052 (6)	0.2742 (10)	0.063 (2)
C5B	0.3214 (4)	0.7513 (6)	0.3572 (10)	0.059 (2)
C6B	0.3149 (3)	0.6415 (5)	0.3866 (7)	0.0383 (14)
F6B	0.3601 (2)	0.5875 (4)	0.4671 (5)	0.0625 (12)
O1C	0.3123 (2)	0.2287 (3)	0.5214 (5)	0.0297 (9)
O2C	0.3470 (2)	0.1136 (4)	0.3384 (5)	0.0548 (11)
C7C	0.3537 (3)	0.1639 (5)	0.4723 (7)	0.0316 (13)
C1C	0.4109 (3)	0.1484 (5)	0.5767 (7)	0.0308 (12)
C2C	0.4302 (3)	0.0478 (6)	0.6316 (9)	0.048 (2)
F2C	0.3934 (2)	-0.0377 (4)	0.5988 (6)	0.0695 (13)
C3C	0.4851 (4)	0.0325 (8)	0.7167 (11)	0.071 (3)
C4C	0.5237 (4)	0.1180 (10)	0.7459 (11)	0.079 (3)
C5C	0.5070 (4)	0.2196 (8)	0.6940 (11)	0.068 (2)
C6C	0.4509 (3)	0.2344 (6)	0.6148 (9)	0.047 (2)
F6C	0.4343 (2)	0.3331 (3)	0.5574 (7)	0.0680 (13)

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *XPLOR* and *ZORTEP* (Zsolnai, 1995).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1180). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 2. Selected geometric parameters (Å, °)

Pr—O2B ⁱ	2.405 (4)	O1B—C7B	1.265 (6)
Pr—O1C ⁱ	2.459 (4)	O2B—C7B	1.234 (6)
Pr—O2C	2.497 (4)	O2B—Pr ⁱⁱ	2.405 (4)
Pr—O1B	2.501 (4)	C7B—C1B	1.503 (7)
Pr—O1A ⁱⁱ	2.517 (4)	C2B—F2B	1.347 (8)
Pr—O1W	2.525 (4)	C6B—F6B	1.331 (8)
Pr—O2A	2.526 (4)	O1C—C7C	1.277 (7)
Pr—O1A	2.578 (4)	O1C—Pr ⁱⁱ	2.459 (4)
Pr—O1C	2.612 (4)	O2C—C7C	1.231 (7)
O1A—C7A	1.268 (6)	C7C—C1C	1.480 (7)
O1A—Pr ⁱ	2.517 (4)	C2C—F2C	1.344 (8)
C7A—O2A	1.243 (6)	C6C—F6C	1.343 (8)
C7A—C1A	1.508 (7)		
O2B ⁱ —Pr—O1C ⁱ	73.60 (13)	C7A—O1A—Pr ⁱ	147.3 (3)
O2B ⁱ —Pr—O2C	78.91 (14)	C7A—O1A—Pr	92.2 (3)
O1C ⁱ —Pr—O2C	81.5 (2)	Pr ⁱ —O1A—Pr	109.47 (14)
O2B ⁱ —Pr—O1B	140.01 (13)	O2A—C7A—O1A	121.1 (5)
O1C ⁱ —Pr—O1B	71.00 (14)	O2A—C7A—C1A	119.4 (5)
O2C—Pr—O1B	113.1 (2)	O1A—C7A—C1A	119.4 (5)
O2B ⁱ —Pr—O1A ⁱⁱ	139.19 (14)	C7A—O2A—Pr	95.3 (3)
O1C ⁱ —Pr—O1A ⁱⁱ	144.48 (13)	F2A—C2A—C1A	118.7 (5)
O2C—Pr—O1A ⁱⁱ	113.00 (13)	F2A—C2A—C3A	118.2 (6)
O1B—Pr—O1A ⁱⁱ	73.48 (14)	F6A—C6A—C5A	118.9 (6)
O2B ⁱ —Pr—O1W	80.95 (13)	F6A—C6A—C1A	118.3 (5)
O1C ⁱ —Pr—O1W	147.25 (14)	C7B—O1B—Pr	134.2 (3)
O2C—Pr—O1W	73.5 (2)	C7B—O2B—Pr ⁱⁱ	145.4 (4)
O1B—Pr—O1W	138.54 (13)	O2B—C7B—O1B	125.9 (5)
O1A ⁱⁱ —Pr—O1W	66.91 (13)	O2B—C7B—C1B	116.4 (4)
O2B ⁱ —Pr—O2A	73.99 (14)	O1B—C7B—C1B	117.7 (4)
O1C ⁱ —Pr—O2A	118.07 (12)	C6B—C1B—C2B	115.5 (6)
O2C—Pr—O2A	139.2 (2)	C6B—C1B—C7B	121.7 (5)
O1B—Pr—O2A	107.3 (2)	C2B—C1B—C7B	122.6 (5)
O1A ⁱⁱ —Pr—O2A	72.85 (12)	F2B—C2B—C3B	118.8 (6)
O1W—Pr—O2A	72.56 (14)	F2B—C2B—C1B	117.8 (6)
O2B ⁱ —Pr—O1A	74.17 (13)	F6B—C6B—C1B	117.4 (5)
O1C ⁱ —Pr—O1A	70.24 (12)	F6B—C6B—C5B	119.4 (7)
O2C—Pr—O1A	145.31 (14)	C1B—C6B—C5B	123.2 (7)
O1B—Pr—O1A	76.85 (13)	C7C—O1C—Pr ⁱⁱ	151.7 (3)
O1A ⁱⁱ —Pr—O1A	101.68 (12)	C7C—O1C—Pr	91.0 (3)
O1W—Pr—O1A	122.16 (14)	Pr ⁱⁱ —O1C—Pr	110.23 (14)
O2A—Pr—O1A	50.74 (12)	C7C—O2C—Pr	97.6 (4)
O2B ⁱ —Pr—O1C	128.48 (13)	O2C—C7C—O1C	120.8 (5)
O1C ⁱ —Pr—O1C	103.52 (13)	O2C—C7C—C1C	119.1 (5)
O2C—Pr—O1C	50.46 (13)	O1C—C7C—C1C	120.1 (5)
O1B—Pr—O1C	78.27 (13)	F2C—C2C—C3C	119.6 (7)
O1A ⁱⁱ —Pr—O1C	68.81 (12)	F2C—C2C—C1C	118.1 (6)
O1W—Pr—O1C	76.58 (14)	F6C—C6C—C5C	120.1 (7)
O2A—Pr—O1C	137.74 (12)	F6C—C6C—C1C	117.0 (5)
O1A—Pr—O1C	155.03 (12)		

Symmetry codes: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $x, \frac{1}{2} - y, \frac{1}{2} + z$.

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Dichloro[*N,N'*-ethylenebis(3-*tert*-butyl-5-methylsalicylideneiminato)-*N,N',O,O'*]-titanium(IV), [TiCl₂(C₂₆H₃₄N₂O₂)₂]

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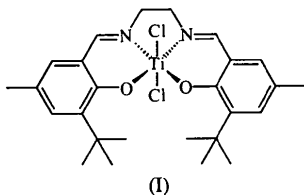
Abstract

The synthetic route to obtain single crystals of dichloro{6,6'-di-*tert*-butyl-2,2'-[1,2-ethanediylbis(nitrilomethylidyne-*N*)]-4,4'-dimethyldiphenolato-*O,O'*}titanium(IV) is described. The X-ray analysis reveals that the title compound is a monomer with four molecules in the unit cell. There are only weak van der Waals-type forces between neighboring molecules. The compound

crystallizes in the monoclinic space group $P2_1/c$ (No. 14). The Ti atom has a distorted octahedral coordination, with chloride ions in *trans* positions.

Comment

The chemical properties of titanium(IV)–salen or salen-type complexes [salen is *N,N'*-ethylenebis(salicylideneiminato)] where the ligand has a pseudo-planar configuration and the chloride ligands are in *trans* positions have been investigated, for example, by arylation or alkylation reactions (Solari, Floriani, Chiesi-Villa & Rizzoli, 1992) and also by the reactions with α -hydroxy carboxylic acids (Carroll, Schwartz & Ho, 1994). The catalytic properties of titanium–salen-type complexes have also been studied, for example, titanium(IV)–Schiff base complexes derived from salicylaldehydes and chiral 1,2-diamines have been used as catalysts for the oxidation of aryl alkyl sulfides to sulfoxides (Colombo, Marturano & Pasini, 1986; Sasaki, Nakajima, Kojima & Fujita, 1991). Zirconium–salen complexes have similar catalytic properties to the titanium complexes and in addition they are active in the polymerization of ethylene (Repo *et al.*, 1996). The structure of dichloro{6,6'-di-*tert*-butyl-2,2'-[1,2-ethanediy]bis(nitrilomethylidene-*N*)-4,4'-dimethyldiphenolato-*O,O'*}titanium(IV), (I), has been determined.



The structure of (I) (Fig. 1) consists of discrete monomeric $[\text{TiCl}_2(\text{C}_{26}\text{H}_{34}\text{N}_2\text{O}_2)]$ molecules with a pseudo-mirror plane through the Cl–Ti–Cl atoms, except that the C8 and C9 atoms in the ethylene bridge are not mirror images. The substituted salen ligand occupies the equatorial positions, while the Cl ions occupy the axial *trans* positions. The environment of the Ti atom is distorted octahedral, as indicated by the angles around the Ti atom which vary from 76.7 (2) (N1–Ti–N2) to 112.0 (2)° (O1–Ti–O2). The Ti–Cl bonds [2.345 (3) and 2.353 (3) Å] and the Cl–Ti–Cl angle [169.18 (10)°] are very close to those found in $[\text{TiCl}_2(\text{salen})]\cdot\text{THF}$ (THF is tetrahydrofuran) (Gilli, Cruickshank, Beddoes & Mills, 1972). The Ti–O bond lengths are 1.816 (4) and 1.820 (5) Å, but the Ti–N bond lengths are considerably longer at 2.110 (6) and 2.136 (5) Å. The difference between the Ti–N and Ti–O bond lengths has been rationalized according to the principles of hard and soft acids and bases of Schlemper (1967). An analogous difference in bond lengths has been found, however, even when both donor

atoms are oxygen. In a six-coordinate titanium dichloro-benzaldehyde derivative complex $[\text{Ti}(\text{C}_{12}\text{H}_{15}\text{O}_2)\text{Cl}_2]$, a negative phenolic O atom is σ -coordinated to titanium, with a mean Ti–O bond length of 1.866 (10) Å, and a neutral aldehyde O atom is π -coordinated using its free electron pairs, with a mean Ti–O bond length of 2.125 (10) Å (Matilainen, Klinga & Leskelä, 1996). We therefore assume that the reason for the difference in Ti–N and Ti–O bond lengths in the title compound is analogous to that for the difference in Ti–O bond lengths in the $[\text{Ti}(\text{C}_{12}\text{H}_{15}\text{O}_2)\text{Cl}_2]$ complex.

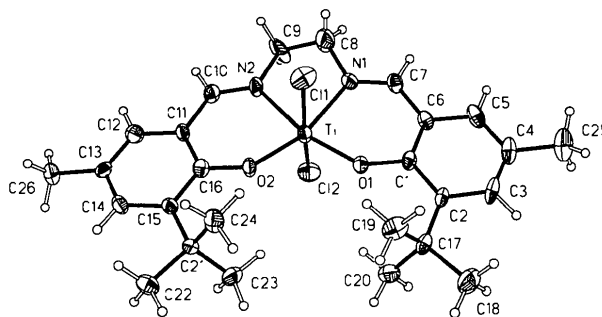


Fig. 1. View of $[\text{TiCl}_2(\text{C}_{26}\text{H}_{34}\text{N}_2\text{O}_2)]$ with the atom labels and displacement ellipsoids drawn at the 30% probability level.

The Ti atom and its equatorial donor atoms are practically coplanar. The Ti atom is only 0.003 (2) Å out of the TiN_2O_2 mean plane. The largest deviations of the donor atoms themselves from the plane are about 0.04 Å. The salicylideneiminato frame of nine atoms (O1–C1...C7–N1) and its equivalent (O2–C16...C10–N2) are also fairly planar, the largest deviations of the atoms from their corresponding least-squares planes being only about 0.02 and 0.06 Å, respectively. The dihedral angle between these nine-atom planes is 13.15 (17)°. The ethylene bridge C atoms are on different sides of the TiN_2 plane and a *gauche* conformation is established.

Experimental

Melting points were measured with an Electrothermal apparatus. The ^1H NMR spectrum was recorded on a Varian Gemini 200 MHz spectrometer. The mass spectrum were recorded on a Jeol JMS-SX102 mass spectrometer. Preparation of the titanium complex was carried out using a purified argon atmosphere and standard Schlenk techniques. THF and toluene were purified by distillation over LiAlH_4 . For the preparation of *N,N'*-bis(3-*tert*-butyl-5-methylsalicylidene)ethylenediamine, ethylenediamine (0.99 ml, 14.9 mmol) and 3-*tert*-butyl-5-methylsalicylaldehyde (5.7 g, 29.6 mmol) were refluxed in absolute ethanol (75 ml) for 1 h. A small amount of water was added and the mixture was cooled at 277 K for 2 h to give yellow crystals. Yield 5.5 g (91%); m.p. (corrected, recrystallization from EtOH): 431–432 K; ^1H NMR (200 MHz, CDCl_3 , TMS): δ 1.42 (18H, s, 'Bu), 2.26 (6H, s, CH_3),

3.91 (4H, *s*, CH₂N), 6.88 (2H, *d*, *J* = 2.0 Hz, HAr), 7.11 (2H, *d*, *J* = 2.1 Hz, HAr), 8.33 (2H, *s*, CH=N), 13.4 (2H, *brs*, OH); ¹³C NMR (CDCl₃): δ 167.5, 158.4, 137.4, 130.9, 126.9, 118.6, 59.9, 35.1, 29.7, 21.0; HRMS (EI, 70 eV): calculated for C₂₆H₃₆N₂O₂ 408.2777, found 408.2763. For the preparation of (I), complexation was carried out following the synthetic route described earlier by Dell'Amico, Marchetti & Floriani (1982) for dichloro[*N,N'*-ethylenebis(salicylidene-iminiato)]titanium(IV). *N,N'*-Bis(3-*tert*-butyl-5-methylsalicylidene)ethylenediamine (2.84 g, 6.96 mmol) was dissolved in THF (30 ml) and cooled to 195 K. A solution of TiCl₄ (1.32 g, 0.76 ml, 6.96 mmol) in toluene (15 ml) was added dropwise to the cool THF solution. The temperature of the reaction mixture was allowed to rise slowly to ambient temperature and reflux was carried out for 1 h. The reaction mixture was then dried by evaporation in a vacuum, washed with a water/acetone mixture (1:1), filtered and washed again with acetone. After careful drying in a vacuum, a dark-red powder (2.75 g, 5.23 mmol, 76%) was obtained. ¹H NMR (200 MHz, CDCl₃, TMS): δ 1.52 (18H, *s*, 'Bu), 2.33 (6H, *s*, CH₃), 4.17 (4H, *s*, CH₂N), 7.12 (2H, *d*, HAr), 7.36 (2H, *d*, HAr), 8.28 (2H, *s*, CH=N); MS (EI, 70 eV): 525 (*M*⁺), 489 (*M*⁺—Cl).

Crystal data

[TiCl₂(C₂₆H₃₄N₂O₂)]

M_r = 525.35

Monoclinic

*P*₂₁/*c*

a = 10.608 (7) Å

b = 9.782 (9) Å

c = 25.999 (7) Å

β = 90.44 (4)°

V = 2698 (3) Å³

Z = 4

D_x = 1.293 Mg m⁻³

D_m = 1.282 Mg m⁻³

D_m measured by flotation

Data collection

Rigaku AFC-7S diffractometer

ω scans

Absorption correction:

ψ scan (North, Phillips & Mathews, 1968)

T_{min} = 0.86, *T_{max}* = 0.98

4666 measured reflections

4273 independent reflections

1900 observed reflections

[*I* > 2σ(*I*)]

Refinement

Refinement on *F*²

R(*F*) = 0.0957

wR(*F*²) = 0.1591

S = 0.978

4235 reflections

300 parameters

H atoms: riding model

w = 1/[σ²(*F_o*²) + (0.0367*P*)²]

where *P* = (*F_o*² + 2*F_c*²)/3

Mo Kα radiation

λ = 0.71073 Å

Cell parameters from 20 reflections

θ = 7.36–12.56°

μ = 0.540 mm⁻¹

T = 193 (2) K

Plate

0.42 × 0.32 × 0.03 mm

Red

R_{int} = 0.0657

θ_{max} = 26.5°

h = 0 → 13

k = 0 → 12

l = -32 → 32

3 standard reflections

monitored every 200

reflections

intensity decay: <2%

(Δ/σ)_{max} = 0.009

Δρ_{max} = 0.338 e Å⁻³

Δρ_{min} = -0.377 e Å⁻³

Extinction correction: none

Atomic scattering factors from *International Tables for Crystallography* (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Ti	0.09758 (12)	0.63423 (15)	-0.16614 (5)	0.0350 (4)
Cl1	-0.0024 (2)	0.4221 (2)	-0.15647 (8)	0.0616 (7)
Cl2	0.1604 (2)	0.8613 (2)	-0.18128 (8)	0.0590 (7)
O1	0.1143 (4)	0.6588 (5)	-0.0972 (2)	0.0383 (13)
O2	0.2382 (4)	0.5591 (5)	-0.1942 (2)	0.0418 (14)
N1	-0.0867 (5)	0.7118 (6)	-0.1576 (2)	0.043 (2)
N2	0.0268 (5)	0.6397 (7)	-0.2433 (2)	0.040 (2)
C1	0.0512 (7)	0.7150 (7)	-0.0578 (3)	0.036 (2)
C2	0.1073 (7)	0.7180 (7)	-0.0089 (3)	0.037 (2)
C3	0.0377 (9)	0.7763 (8)	0.0304 (3)	0.053 (3)
C4	-0.0825 (9)	0.8327 (8)	0.0230 (3)	0.055 (3)
C5	-0.1363 (7)	0.8254 (8)	-0.0251 (3)	0.051 (2)
C6	-0.0694 (7)	0.7662 (7)	-0.0667 (3)	0.037 (2)
C7	-0.1331 (7)	0.7594 (8)	-0.1157 (3)	0.041 (2)
C8	-0.1670 (8)	0.6996 (12)	-0.2030 (3)	0.091 (4)
C9	-0.0988 (7)	0.6978 (11)	-0.2478 (3)	0.086 (4)
C10	0.0903 (4)	0.6102 (4)	-0.2832 (2)	0.042 (2)
C11	0.2196 (4)	0.5614 (4)	-0.2844 (2)	0.034 (2)
C12	0.2735 (4)	0.5403 (4)	-0.3323 (2)	0.048 (2)
C13	0.3947 (7)	0.4901 (8)	-0.3361 (3)	0.040 (2)
C14	0.4593 (7)	0.4556 (7)	-0.2918 (3)	0.045 (2)
C15	0.4110 (7)	0.4743 (8)	-0.2420 (3)	0.035 (2)
C16	0.2899 (7)	0.5312 (8)	-0.2398 (3)	0.037 (2)
C17	0.2385 (7)	0.6609 (8)	0.0012 (3)	0.043 (2)
C18	0.2798 (8)	0.6730 (10)	0.0581 (3)	0.078 (3)
C19	0.2439 (8)	0.5100 (8)	-0.0135 (3)	0.059 (3)
C20	0.3354 (7)	0.7409 (9)	-0.0301 (3)	0.063 (3)
C21	0.4833 (6)	0.4367 (8)	-0.1935 (3)	0.037 (2)
C22	0.6102 (6)	0.3709 (9)	-0.2051 (3)	0.059 (2)
C23	0.5072 (7)	0.5674 (8)	-0.1608 (3)	0.058 (3)
C24	0.4075 (7)	0.3324 (8)	-0.1619 (3)	0.053 (2)
C25	-0.1571 (8)	0.8906 (9)	0.0683 (3)	0.085 (3)
C26	0.4598 (7)	0.4684 (8)	-0.3878 (3)	0.056 (3)

Table 2. Selected geometric parameters (Å, °)

Ti—O1	1.816 (4)	O1—C1	1.345 (8)
Ti—O2	1.820 (5)	O2—C16	1.337 (8)
Ti—N1	2.110 (6)	N1—C7	1.286 (8)
Ti—N2	2.136 (5)	N1—C8	1.454 (9)
Ti—Cl1	2.345 (3)	N2—C10	1.276 (6)
Ti—Cl2	2.353 (3)	N2—C9	1.453 (8)
O1—Ti—O2	112.0 (2)	N1—Ti—Cl1	83.5 (2)
O1—Ti—N1	86.1 (2)	N2—Ti—Cl1	88.1 (2)
O2—Ti—N1	161.7 (2)	O1—Ti—Cl2	90.8 (2)
O1—Ti—N2	162.6 (2)	O2—Ti—Cl2	94.6 (2)
O2—Ti—N2	85.2 (2)	N1—Ti—Cl2	86.7 (2)
N1—Ti—N2	76.7 (2)	N2—Ti—Cl2	85.3 (2)
O1—Ti—Cl1	93.0 (2)	Cl1—Ti—Cl2	169.18 (10)
O2—Ti—Cl1	93.3 (2)		

The intensity data were corrected for Lorentz and polarization effects and also for absorption. All non-H atoms were refined anisotropically. H atoms were refined on calculated positions with displacement parameters 1.3 times those of the host atom (riding model). The high value of *R* is due to the poor quality of the crystal used for analysis.

Data collection: *TEXSAN* (Molecular Structure Corporation, 1993). Cell refinement: *TEXSAN*. Data reduction: *TEXSAN*. Program(s) used to solve structure: *SIR92* (Burla *et al.*, 1992). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: NA1254). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dicyano(2,9-dimethyl-1,10-phenanthroline-*N,N'*)(η^2 -ethene)platinum(II)

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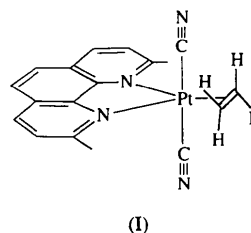
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Abstract

In the title compound, $[\text{Pt}(\text{CN})_2(\text{C}_2\text{H}_4)(\text{C}_{14}\text{H}_{12}\text{N}_2)]$, the Pt atom has a trigonal-bipyramidal coordination geometry, with the phenanthroline and ethene ligands in the equatorial plane and the cyano ligands axial. A *trans* configuration of cyano ligands in such five-coordinate Pt^{II} complexes is unprecedented.

Comment

A recent report of the synthesis of coordinatively saturated Pt^{II} complexes suitable as precursors of organometallic polymers (Giordano, Panunzi, Roviello & Ruffo, 1995) included the title compound, (I), or the oxidative addition of $(\text{CN})_2$ to $(\text{dmphen})\text{Pt}(\text{C}_2\text{H}_4)$ (where dmphen is 2,9-dimethyl-1,10-phenanthroline). Analytical and spectroscopic data indicated the formula $[(\text{dmphen})\text{Pt}(\text{C}_2\text{H}_4)(\text{CN})_2]$ and a trigonal-bipyramidal (TBP) geometry, with axial cyano ligands attached through their C atoms and ethene being considered as occupying a single equatorial site. Such an axial arrangement of the cyano ligands is unprecedented for 18-electron TBP- Pt^{II} complexes and provides potential for the linking of metal ions. We have confirmed the proposed structure of (I) by X-ray crystallographic analysis.



The Pt coordination geometry is indeed TBP, with equatorial chelating dmphen and ethene ligands and with axial cyanides (Fig. 1). The Pt atom lies on a twofold rotation axis bisecting the ethene ligand and the overall molecular symmetry is approximately C_{2v} (*mm2*). The N—C—Pt—C—N unit is essentially linear, with angles of 177.6(1) at platinum and 178.9(4)° at carbon. The Pt—C [2.007(5) Å] and C—N [1.130(7) Å] bond lengths are comparable to corresponding values in square-planar complexes having *trans*- CN^- ligands (Ebihara, Imai & Kawamura, 1995; Staples, Khan, Wang & Fackler, 1992). The dmphen ligand is essentially planar, with a maximum deviation of 0.039(5) Å for

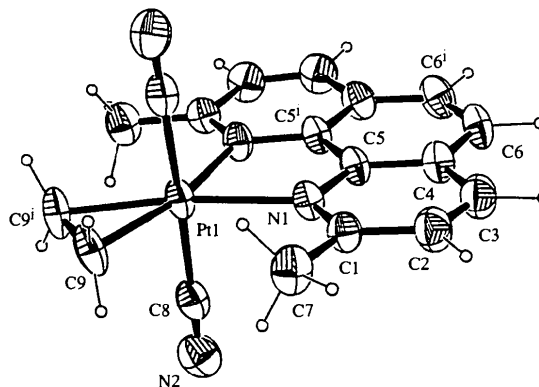


Fig. 1. The molecular structure of (I) with ellipsoids at the 50% probability level. H atoms are drawn as small circles of arbitrary radii.