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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1259). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Trichloro[η^5-(trimethylstannyl)cyclopentadienyl]titanium

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Abstract

The title compound, $[TiCl_3\{(CH_3)_3Sn(C_5H_4)\}]$, is the first structurally characterized complex of an early transition metal with a cyclopentadienyl ligand-bearing stannyl moiety. The Ti—*Cp* distance of 1.986 (4) Å [where *Cp* is the centroid of the cyclopentadienyl (Cp) ring] is the shortest among the complexes containing the $CpTiCl_3$ moiety.

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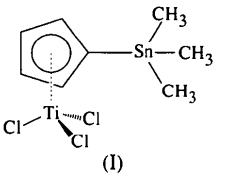
Comment

The structure of the title compound, (I), has been established as part of a study on ring-substituted monocyclopentadienyl complexes of titanium (Churakov, Lemenovskii & Kuz'mina, 1995; Rufanov, Churakov, Kazennova, Brusova, Lemenovskii & Kuz'mina, 1995).



(I)

The molecular structure of (I) is shown in Fig. 1. The Ti—C distances are quite regular [2.299 (7)–2.337 (8) Å], the shortest being to the C(1) atom which is bonded to Sn. The displacement of the Ti atom from the least-squares plane of the cyclopentadienyl ring is 1.986 (4) Å. A search of the Cambridge Structural Database (Version 5.10 of October 1995; Allen *et al.*, 1991) shows that this is the shortest reported value among ring-substituted complexes containing the $CpTiCl_3$ moiety (2.003–2.022 Å).

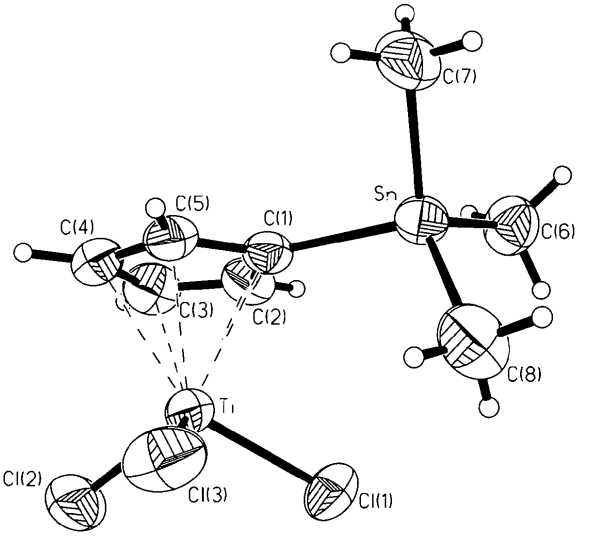


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids.

The coordination at the Sn atom is tetrahedral, with C—Sn—C angles ranging from 103.3 (4) to 113.5 (4)°. The deviation of the Sn atom from the Cp ring plane is 0.093 (13) Å. The Sn—C_{Cp} bond distance [2.169 (8) Å] is significantly longer than those found in ferrocenyls [2.146 (3) and 2.125 (4) Å (Clearfield, Simmons, Withers & Seyferth, 1983), and 2.103 (5) Å (Dong, Hwang, Wen & Hwang, 1990)] and cemantrenylstannanes [2.109 (4) Å (Bokii & Struchkov, 1978)].

Experimental

The synthesis of (I) was carried out by reaction of bis(trimethylstannyl)cyclopentadiene with $TiCl_4$ in toluene at room temperature (molar ratio 1:1.1) in an evacuated glass vessel. The product was recrystallized from hexane.

Crystal data

$[TiCl_3(C_8H_{13}Sn)]$
M_r = 382.12
Orthorhombic
$Pna2_1$
a = 16.164 (8) Å
b = 7.194 (5) Å
c = 11.923 (5) Å
V = 1386.5 (13) Å^3
Z = 4
D_x = 1.831 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 11–13°
μ = 2.915 mm^{-1}
T = 293 (2) K
Block
0.5 × 0.3 × 0.3 mm
Dark yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
1856 measured reflections
1856 independent reflections
1244 observed reflections [$I > 2\sigma(I)$]
θ_{max} = 29.96°
$h = 0 \rightarrow 22$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 16$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R(F)$ = 0.0351
$wR(F^2)$ = 0.0913
S = 1.043
1856 reflections
122 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = −0.001
$\Delta\rho_{max}$ = 0.632 e Å^{-3}
$\Delta\rho_{min}$ = −0.925 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0009 (4)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = 0.12 (6)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Sn	−0.23951 (3)	−0.08689 (7)	0.00040 (6)	0.0546 (2)
Ti	−0.00396 (9)	−0.0047 (2)	0.06976 (14)	0.0536 (3)
Cl(1)	−0.0486 (2)	−0.2142 (3)	0.1936 (2)	0.0723 (6)
Cl(2)	0.1191 (2)	0.0758 (4)	0.1413 (3)	0.1018 (10)
Cl(3)	0.0291 (2)	−0.1804 (4)	−0.0765 (2)	0.0982 (9)
C(1)	−0.1350 (5)	0.0972 (10)	0.0268 (6)	0.052 (2)
C(2)	−0.1116 (5)	0.1836 (10)	0.1302 (8)	0.059 (2)
C(3)	−0.0451 (6)	0.2953 (12)	0.1119 (9)	0.071 (2)
C(4)	−0.0254 (5)	0.2929 (11)	−0.0034 (13)	0.076 (2)
C(5)	−0.0823 (5)	0.1695 (13)	−0.0541 (8)	0.065 (2)
C(6)	−0.3068 (7)	−0.0779 (14)	0.1549 (9)	0.078 (3)
C(7)	−0.3032 (7)	0.0379 (18)	−0.1357 (10)	0.092 (4)
C(8)	−0.1993 (7)	−0.3589 (15)	−0.0362 (12)	0.097 (4)

Table 2. *Selected geometric parameters (Å, °)*

Sn—C(8)	2.107 (10)	Ti—C(3)	2.313 (8)
Sn—C(7)	2.121 (10)	Ti—C(2)	2.320 (8)
Sn—C(6)	2.140 (10)	Ti—C(4)	2.337 (8)
Sn—C(1)	2.169 (8)	C(1)—C(5)	1.388 (11)
Ti—Cl(3)	2.219 (3)	C(1)—C(2)	1.432 (11)
Ti—Cl(1)	2.230 (3)	C(2)—C(3)	1.360 (12)
Ti—Cl(2)	2.241 (3)	C(3)—C(4)	1.41 (2)
Ti—C(1)	2.299 (7)	C(4)—C(5)	1.414 (13)
Ti—C(5)	2.314 (9)		
C(8)—Sn—C(7)	112.6 (5)	Cl(1)—Ti—Cl(2)	102.11 (13)
C(8)—Sn—C(6)	111.3 (5)	C(5)—C(1)—C(2)	105.9 (7)
C(7)—Sn—C(6)	113.5 (4)	C(5)—C(1)—Sn	127.3 (6)
C(8)—Sn—C(1)	110.9 (4)	C(2)—C(1)—Sn	126.5 (6)
C(7)—Sn—C(1)	103.3 (4)	C(3)—C(2)—C(1)	109.1 (8)
C(6)—Sn—C(1)	104.6 (4)	C(2)—C(3)—C(4)	109.2 (8)
Cl(3)—Ti—Cl(1)	102.30 (13)	C(5)—C(4)—C(3)	106.1 (8)
Cl(3)—Ti—Cl(2)	103.45 (14)	C(1)—C(5)—C(4)	109.7 (9)

All H atoms were placed in calculated positions (C—H = 0.96 Å) and assigned isotropic displacement parameters 1.2U_{eq} of their parent C atoms. An absorption correction was applied using *DIFABS* (Walker & Stuart, 1983), but no changes to the geometric data resulted. The structural parameters given in this paper were refined without absorption corrections.

Data collection: *CAD-4 Software* (Schagen, Strauer, van Meurs & Williams, 1988). Cell refinement: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXTL-Plus*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SK1054). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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