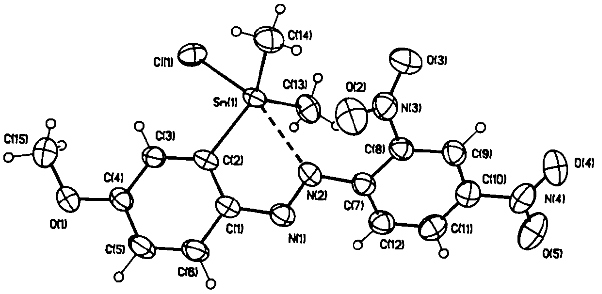

Fig. 1. General view (*SHELXTL-Plus*; Sheldrick, 1987) of the molecule, showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries.

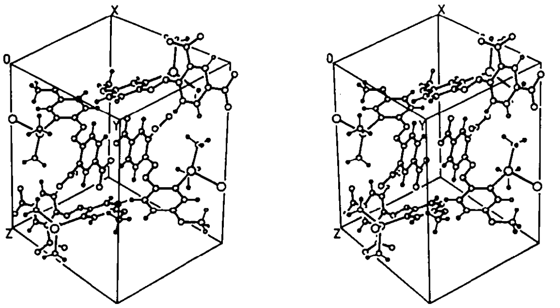

Fig. 2. Stereoscopic view (*SHELXTL-Plus*; Sheldrick, 1987) of part of the crystal structure with the boundaries of the unit cell.

dinitrobenzene tetrafluoroborate in acetonitrile at 296 K, and subsequent workup with column chromatography. Surprisingly, a tin methyl bond has been split instead of the tin phenyl bond, whereas the reactivity is normally the inverse (Al-Allaf, Kobs & Neumann, 1989). This is one of the first cases of complexation of a triorganotin halide by an azo compound forming intramolecular pentacoordination at the Sn atom (Brianso, Solans & Vicente, 1983; Vicente, Chicote, Carreno & del Carmen Ramirez-de-Arellano, 1989; Vicente, Chicote, del Carmen Ramirez-de-Arellano, Pelizzi & Vitali, 1990; Vicente, Chicote, del Carmen Ramirez-de-Arellano & Jones, 1990).

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Structure of Trichloro(η^5-methylcyclopentadienyl)titanium(IV)

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Abstract. [$TiCl_3(CH_3C_5H_4)$], $M_r = 233{\cdot}38$, triclinic, $P\bar{1}$, $a = 6{\cdot}862\,(1)$, $b = 6{\cdot}923\,(2)$, $c = 11{\cdot}215\,(2)$ Å, $\alpha = 82{\cdot}65\,(1)$, $\beta = 83{\cdot}27\,(1)$, $\gamma = 61{\cdot}30\,(1)°$, $V = 462\,(3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}68$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 17{\cdot}2$ cm^{-1}, $F(000) = 232$, $T = 294\,(1)$ K, $R = 0{\cdot}036$ for 1568 unique reflections with $F_o^2 > 3\sigma(F_o^2)$. The five ring C atoms of the methylcyclopentadienyl ligand and the three Cl atoms form a piano-stool coordination about the Ti atom with a dihedral angle between the methylcyclopentadienyl plane and the Cl_3 plane of 2·7°. The Ti—Cl distances range from 2·2229 (9) to 2·228 (1) Å, the Ti—C* distance is 2·004 Å (C* = centroid of the cyclopentadienyl ring) and the Ti—C distances range from 2·301 (3) to 2·379 (3) Å. The torsion angle Cl(2)—Ti—C(5)—C(6), with a value of −0·9 (3)°, shows the

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eclipsed configuration of the methyl group and the Cl(2) atom.

Experimental. The title compound was obtained by reaction of $(CH_3C_5H_4)_2TiCl_2$ with $TiCl_4$ in xylene by the procedure of Gorsich (1958); the method of Wilkinson & Birmingham (1954) was used to prepare $(CH_3C_5H_4)_2TiCl_2$. Orange columnar crystals of $(CH_3C_5H_4)TiCl_3$ suitable for X-ray diffractometry were obtained from a saturated solution of dichloromethane/hexane (1:1) at 243 K. Crystal 0·45 × 0·20 × 0·20 mm; Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation; ω–2θ scan technique; $2\theta_{\rm max}$ = 52·0°, *hkl* range $0 \le h \le 8$, $-7 \le k \le 8$, $-13 \le l \le 13$; cell dimensions obtained from least-squares refinement on 25 reflections ($10 < \theta < 14°$). Lorentz and polarization corrections, three standard reflections indicated a total loss in intensity of 0·6%, anisotropic decay correction applied (from 0·975 to 1·008 on I), empirical absorption correction (from 0·869 to 0·998 on I). 1982 reflections measured, 1823 unique (four reflections rejected from the averaging process because their intensities differed significantly from the average, $R_{\rm int}$ = 0·013 based on I), 1568 reflections with $F_o^2 > 3{\cdot}0\sigma(F_o^2)$. Solved by direct methods and difference Fourier syntheses; refined by full-matrix least squares minimizing $\sum w(|F_o| - |F_c|)^2$, $w = 4F_o^2/\sigma^2(F_o^2)$; H atoms located, positions and isotropic thermal parameters refined, anisotropic thermal parameters for non-H atoms. 119 parameters, R = 0·036, wR = 0·050, S = 2·79, final $(\Delta/\sigma)_{\rm max} \le 0{\cdot}01$; $(\Delta\rho)_{\rm max}$ = 0·51 (6), $(\Delta\rho)_{\rm min}$ = −0·40 (6) e Å^{-3}. Atomic scattering factors and values of f' and f'' were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2, pp. 71–102; Table 2.3.1., pp. 149–150). Calculations on a VAX11/750 computer using *SDP*/*VAX* (Frenz, 1978), figure produced with *ORTEP* (Johnson, 1971). The atom-numbering scheme is shown in Fig. 1, Table 1 gives the atomic coordinates, while selected bond lengths and angles are listed in Table 2.*

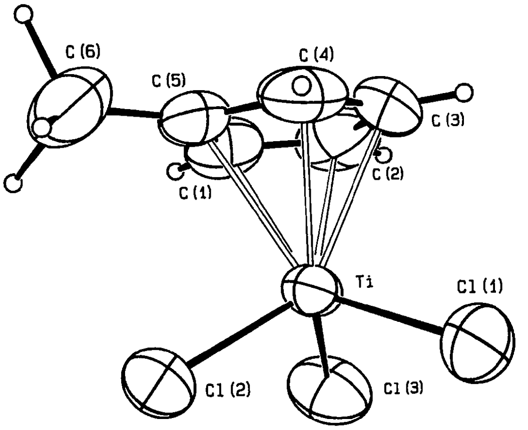

Fig. 1. *ORTEP* (Johnson, 1971) drawing and numbering scheme for trichloro(η^5-methylcyclopentadienyl)titanium(IV) with 50% probability ellipsoids.

Table 1. *Positional parameters and their e.s.d.'s*

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $(8\pi^2/3)$ × trace of the orthogonalized U tensor. C* is the centroid of the C(1)–C(5) ring.

	x	*y*	*z*	*B*(Å^2)
Ti	0·35273 (7)	0·04280 (7)	0·27021 (4)	3·66 (1)
Cl(1)	0·6300 (1)	0·1268 (1)	0·25409 (9)	6·55 (2)
Cl(2)	0·1599 (1)	0·2272 (1)	0·11044 (7)	6·07 (2)
Cl(3)	0·1339 (2)	0·2384 (2)	0·42028 (8)	6·90 (3)
C(1)	0·2559 (5)	−0·2369 (5)	0·3190 (3)	5·58 (8)
C(2)	0·4163 (6)	−0·2689 (5)	0·3959 (3)	6·9 (1)
C(3)	0·6080 (5)	−0·3152 (5)	0·3264 (4)	7·08 (9)
C(4)	0·5710 (5)	−0·3103 (5)	0·2084 (4)	5·96 (9)
C(5)	0·3505 (4)	−0·2683 (4)	0·2031 (3)	4·89 (7)
C(6)	0·2419 (7)	−0·2644 (6)	0·0959 (4)	8·6 (1)
H(1)	0·103 (4)	−0·196 (5)	0·345 (3)	6·7 (8)†
H(2)	0·408 (5)	−0·254 (6)	0·479 (3)	9 (1)†
H(3)	0·737 (5)	−0·345 (5)	0·354 (3)	8 (1)†
H(4)	0·636 (4)	−0·314 (5)	0·149 (3)	5·9 (7)†
H(5)	0·253 (6)	−0·428 (7)	0·086 (4)	12 (1)†
H(6)	0·277 (7)	−0·218 (8)	0·020 (5)	14 (1)†
H(7)	0·034 (6)	−0·140 (8)	0·120 (4)	14 (2)†
C*	0·4403	−0·2799	0·2906	

† H atoms were refined isotropically.

Table 2. *Selected bond distances* (Å) *and angles* (°) *with e.s.d.'s*

C* is the centroid of the C(1)–C(5) ring.

Ti	Cl(1)	2·224 (1)	Ti	C(3)	2·301 (3)
Ti	Cl(2)	2·2229 (9)	Ti	C(4)	2·319 (3)
Ti	Cl(3)	2·228 (1)	Ti	C(5)	2·379 (3)
Ti	C(1)	2·316 (4)	Ti	C*	2·004
Ti	C(2)	2·301 (3)			

Cl(1)	Ti	Cl(2)	103·95 (4)	Cl(1)	Ti	C*	116·1
Cl(1)	Ti	Cl(3)	102·29 (5)	Cl(2)	Ti	C*	115·0
Cl(2)	Ti	Cl(3)	102·36 (3)	Cl(3)	Ti	C*	115·3

Cl(2)	Ti	C(5)	C(6)	−0·9 (3)

Related literature. The title compound is a common starting material for the synthesis of titanium organometallic compounds (*e.g.* Verkouw & van Oven, 1973; Petersen, 1980; Cesarotti, Ugo, Vitiello, 1981; Zank, Jones, Rauchfuss & Rheingold, 1986; Klapötke, Laskowski & Köpf, 1987; Paquette, Moriarty, Meunier, Gautheron & Crocq, 1988;

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* Lists of structure factors, anisotropic thermal parameters and all bond distances and angles including torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54198 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Paquette, Moriarty, Meunier, Gautheron, Sornay, Rogers & Rheingold, 1989). For reviews on titanium organometallic chemistry see Cozak & Melnik (1986) and Bottrill, Gavens, Kelland & McMeeking (1982). The methylcyclopentadienyl ligand offers several advantages over the unsubstituted ligand: increased solubility, elements of molecular symmetry from NMR spectra and most importantly increased crystallizability (Darkwa, Giolando, Jones Murphy & Rauchfuss, 1990). The coordination geometry of the title compound is similar to that of $(C_5H_5)TiCl_3$ which was published with some space group ambiguity (Allegra, Ganis, Porri & Corradini, 1961; Ganis & Allegra, 1962; Engelhardt, Papasergio, Raston & White, 1984). Further single-crystal studies of related transition-metal complexes include $[(C_5H_5)_2Co][(C_5H_5)TiCl_3]$ (Hughes, Jimenez-Tenorio & Leigh, 1989), $(CH_3C_5H_4)VCl_3$ (Morse, Hendrickson, Rauchfuss & Wilson, 1988), $[C_5(CH_3)_4(C_2H_5)]VCl_3$ (Hammer & Messerle, 1990), $(C_5H_5)ZrCl_3$ (Engelhardt, Papasergio, Raston & White, 1984) and $[Li(C_5H_5)CrCl_3.2C_4H_8O]_2.C_4H_8O_2$ (Müller & Krausse, 1972).

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7-Methoxy-2-naphthol

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Abstract. $C_{11}H_{10}O_2$, $M_r = 174{\cdot}2$, monoclinic, $P2_1/c$, $a = 17{\cdot}662\,(3)$, $b = 5{\cdot}9287\,(7)$, $c = 8{\cdot}4003\,(10)$ Å, $\beta = 99{\cdot}857\,(13)°$, $V = 866{\cdot}6\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}335$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54184$ Å, $\mu = 7{\cdot}01$ cm^{-1}, $F(000) = 368$, $T = 300$ K, $R = 0{\cdot}045$ for 1137 observations (of 1708 unique data). The average deviation from planarity is 0·010 Å with a maximum of 0·022 (2) Å for the fused rings. The methoxy group adopts a conformation with the methyl group *syn* to the neighboring α-carbon of the ring, with a C—C—O—C torsion angle of 3·8 (3)°. Steric interaction between the methyl group and the adjacent

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