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(η^5-1-*n*-Butyl-2,3,4,5-tetramethylcyclopentadienyl)trichloridotitanium(IV)

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Key indicators

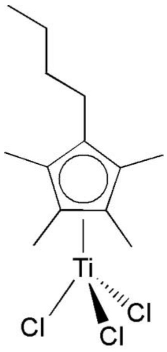
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.046
wR factor = 0.120
Data-to-parameter ratio = 23.0

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

In the title compound, [Ti($C_{13}H_{21}$)Cl_3], the titanium metal centre exhibits distorted tetrahedral coordination, with the substituted cyclopentadienyl ring bound to the $TiCl_3$ group in an η^5-coordination mode.

Comment

Group 4 metallocene complexes have attracted considerable interest owing to their application for olefin polymerization in the fields of organometallic chemistry, catalysis and polymer science (Brintzinger *et al.*, 1995; Nomura *et al.*, 2000; Ruchatz & Fink, 1998). Cyclopentadienyltitanium(IV) derivatives have been extensively studied in the past few decades as efficient catalysts for syndiotactic polymerization of styrene (Ishihara *et al.*, 1986; Foster *et al.*, 1996; Mahanthappa & Waymouth, 2001). As part of our continuing effort to explore active titanium catalysts with different substituents on the Cp ring the title compound, (I), was investigated.



(I)

As shown in Fig. 1, the coordination geometry about the Ti atom can be described as distorted tetrahedral, with the Ti—Cp(centroid) distance, 1.940 Å, less than the value of 2.032 Å reported in previous work (Chien *et al.*, 1992). The C—C distances in the Cp ring range from 1.415 (5) to 1.430 (3) Å; the C1—C2 and C1—C5 distances [1.430 (3) and 1.424 (3) Å, respectively] are longer than the other ring C—C distances. The Ti—C distances are in the range 2.345 (2)–2.364 (2) Å, similar to the observed values in the literature (Orpen *et al.*, 1989). The Ti—Cl distances range from 2.2382 (10) to 2.2471 (9) Å, and the average distance of 2.2429 (9) Å is larger than the observed value of 2.232 (1) Å in an analogous titanocene (Schneider *et al.*, 1997).

Experimental

Compound (I) was synthesized according to literature procedures (Ready *et al.*, 1996). Red crystals suitable for X-ray analysis were

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obtained by recrystallization from a mixture of dichloromethane and *n*-hexane (1:3) at room temperature (m.p. 375–377 K). Analysis calculated for $C_{13}H_{21}Cl_3Ti$: C 46.82, H 6.69%; found: C 46.65, H 6.57%.

Crystal data

$[Ti(C_{13}H_{21})Cl_3]$
$M_r = 331.55$
Orthorhombic, *Pbca*
$a = 13.341$ (2) Å
$b = 15.340$ (3) Å
$c = 15.725$ (3) Å
$V = 3218.1$ (11) Å^3
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 1.01$ mm^{-1}
$T = 295$ (2) K
0.41 × 0.30 × 0.28 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.683$, $T_{\max} = 0.762$
28366 measured reflections
3657 independent reflections
2625 reflections with I > \2s(I)
$R_{\rm int} = 0.049$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.120$
$S = 1.06$
3657 reflections
159 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.58$ e Å^{-3}
$\Delta\rho_{\min} = -0.33$ e Å^{-3}

All H atoms were positioned geometrically with C—H = 0.96–0.97 Å, and allowed to ride on their parent atoms with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ or $1.5U_{\rm eq}({\rm C})$.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC & Rigaku, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

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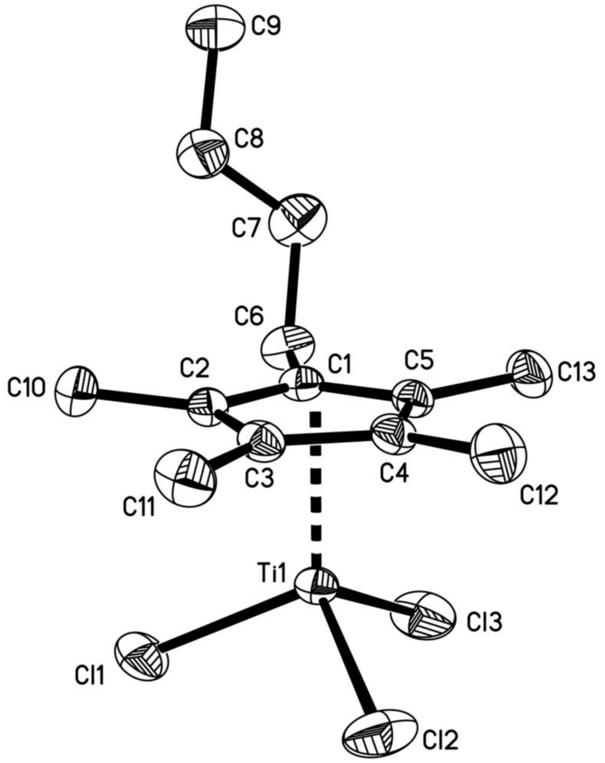

Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted.

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