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(3,5-Dinitrosalicylato-κ^2O^1,O^2)bis(η^5-methylcyclopentadienyl)titanium(IV)

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The Ti atom in the title compound, $[Ti(C_6H_7)_2(C_7H_2N_2O_7)]$, is four-coordinate; the 3,5-dinitrosalicylate, acting as bidentate ligand, chelates it *via* the hydroxy O atom and one carboxylate O atom, forming a six-membered ring.

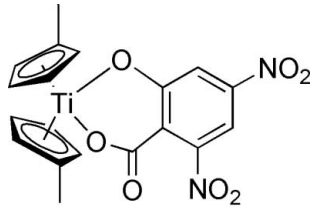
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.036
wR factor = 0.098
Data-to-parameter ratio = 12.3

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

Comment

Titanocene derivatives have attracted considerable interest in recent years, partly due to the fact that they act as catalysts in organic synthesis (Maldanis *et al.*, 2000), as antitumor activity drugs (Mokdis & Harding, 1998) and even as antioxidative agents (Zeng & Xie, 2000), and also for the versatile molecular structures exhibited by these complexes (Radim *et al.*, 2004). Salicylic acid derivatives are of importantance in plant disease resistance owing to their antibiotic function of diminishing inflammation (McMahon & Kelly, 1998). Subsituted salicylate titanocene derivatives are of potential use as anticancer medicines. These two structural units are combined in the title bis(methylcyclopentadienyl)titanium compound, (I).



(I)

Compound (I) exhibits a tetrahedral geometry for the Ti atom. The 3,5-dinitrosalicylate acts as a bidentate ligand and coordinates to the metal centre *via* the hydroxy O atom and one carboxylate O atom (Fig. 1 and Table 1).

Experimental

The methyl-substituted titanocene dichloride $(\eta^5\text{-}CH_3C_5H_4)_2TiCl_2$ (2.0 mmol, 0.554 g) and acetylacetone (2.0 mmol) were dissolved in water (20 ml). The solution was added to a solution of 3,5-dinitrosalicylic acid (2.2 mmol, 0.950 g) dissolved in 20 ml chloroform–diethyl ether (3:1). The mixture was stirred for about 30 min. The organic phase was then separated, washed with saturated Na_2CO_3 and distilled water, and finally dried over anhydrous $MgSO_4$. Removal of the solvent gave a product that was purified by recrystallization from a 1:1 mixture of dichloromethane and *n*-hexane. Crystals were allowed to grow at below room temperature. Red prismatic crystals of (I) were obtained after about one month. Analysis calculated for $C_{19}H_{16}N_2O_7Ti$: C 52.80, H 3.73, N 6.48%; found: C 53.04, H 3.95, N 6.26%.

Crystal data

$[Ti(C_6H_7)_2(C_7H_2N_2O_7)]$
M_r = 432.21
Monoclinic, $P2_1/c$
a = 7.9457 (9) Å
b = 8.1111 (9) Å
c = 28.409 (3) Å
β = 95.3940 (10)°
V = 1822.8 (4) Å^3
Z = 4
D_x = 1.575 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.52 mm^{-1}
T = 296 (2) K
Prism, red
0.38 × 0.28 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.827, T_{max} = 0.920
8884 measured reflections
3254 independent reflections
2837 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 25.1°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.098
S = 1.03
3254 reflections
265 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 0.7525P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0093 (10)

Table 1
Selected geometric parameters (Å, °).

Ti1—O6	1.9520 (13)	Ti1—C18	2.374 (2)
Ti1—O7	1.9622 (14)	Ti1—C16	2.385 (2)
Ti1—C12	2.339 (2)	Ti1—C8	2.393 (2)
Ti1—C15	2.352 (2)	Ti1—C9	2.409 (2)
Ti1—C14	2.357 (2)	Ti1—C17	2.412 (2)
Ti1—C11	2.364 (2)	Ti1—C10	2.416 (2)
O6—Ti1—O7	86.45 (6)	C2—O7—Ti1	127.25 (12)
C7—O6—Ti1	131.53 (12)		

All H atoms were placed in calculated positions and treated as riding, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C).

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SHELXTL* (Sheldrick, 1997*b*) and *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

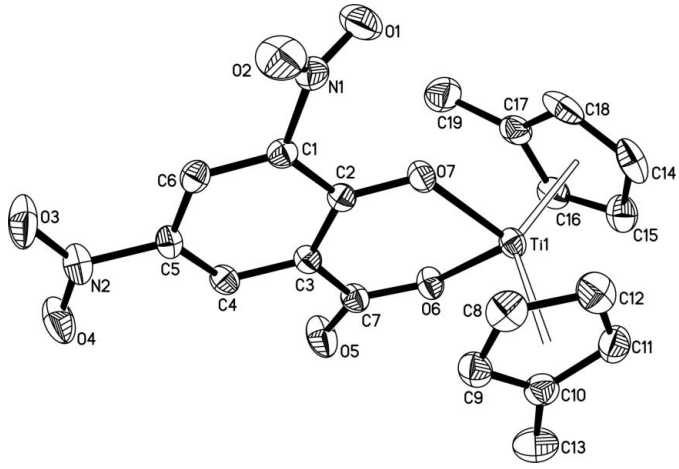


Figure 1
The molecular structure of (I), showing the atomic numbering. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted.

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