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Bis(η^5-methylcyclopentadienyl)(5-nitrosalicylato-κ^2O^1,O^2)titanium(IV)

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In the title compound, [Ti(C_6H_7)$_2$($C_7H_3NO_5$)], the Ti atom is four-coordinate. The 5-nitrosalicylate ligand chelates it, forming a six-membered ring.

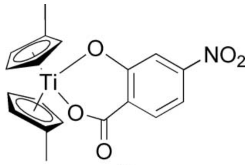
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.034
wR factor = 0.091
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

In the title compound, (I), the Ti atom exists in a four-coordinate distorted tetrahedral environment, with the 5-nitrosalicylate group acting as a bidentate chelate (Fig. 1 and Table 1). The structure is similar to that of the 3,5-nitrosalicylate, which is reported in the preceeding paper (Xu *et al.*, 2007).



(I)

Experimental

The methyl-substituted titanocene dichloride (η^5-$CH_3C_5H_4$)$_2$$TiCl_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 5-nitrosalicylic acid (2.2 mmol, 0.852 g) dissolved in chloroform–diethyl ether (20 ml, 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 give a product that was purified by recrystallization from a 1:1 mixture of dichloromethane and n-hexane. The crystals were allowed to grow at below room temperature. Dark-red acicular crystals of (I) were obtained after about one month. Analysis calculated for $C_{19}H_{17}NO_5Ti$: C 58.94, H 4.43, N 3.62%; found: C 59.50, H 3.39, N 3.25%.

Crystal data

[Ti(C_6H_7)$_2$($C_7H_3NO_5$)]
M_r = 387.21
Triclinic, $P\bar{1}$
a = 7.825 (3) Å
b = 7.980 (3) Å
c = 14.706 (5) Å
α = 95.731 (6)°
β = 99.083 (5)°
γ = 112.056 (5)°
V = 827.6 (5) Å^3
Z = 2
D_x = 1.554 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.55 mm^{-1}
T = 298 (2) K
Needle, dark red
0.39 × 0.13 × 0.09 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.814, T_{max} = 0.952
4416 measured reflections
2900 independent reflections
2412 reflections with $I > 2\sigma(I)$
R_{int} = 0.013
θ_{max} = 25.0°

doi:10.1107/S160053680700219X

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.092$
$S = 1.06$
2900 reflections
235 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 0.3329P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ti1—O3	1.9279 (16)	Ti1—C18	2.371 (3)
Ti1—O1	1.9458 (16)	Ti1—C15	2.386 (3)
Ti1—C16	2.339 (3)	Ti1—C10	2.391 (3)
Ti1—C11	2.348 (2)	Ti1—C9	2.413 (2)
Ti1—C17	2.351 (3)	Ti1—C8	2.416 (2)
Ti1—C12	2.354 (2)	Ti1—C14	2.445 (2)
O3—Ti1—O1	87.56 (7)	C3—O3—Ti1	130.47 (14)
C1—O1—Ti1	132.83 (15)		

There is methyl C—H rotational disorder in compound (I). The three H atoms attached to C13 are each disordered over two positions with equal occupancy; likewise for the three H atoms attached to C19. All H atoms were placed in calculated positions and treated as riding, with C—H = 0.93–0.96 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: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

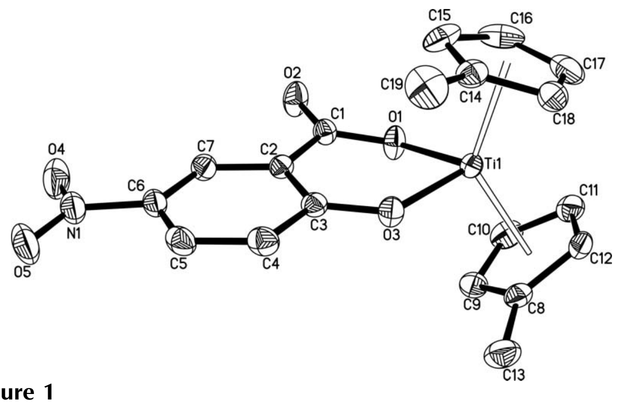


Figure 1
The molecular structure of (I), showing the atomic numbering. Displacement ellipsoids are drawn at the 30% probability level. H atoms and minor disorder components have been omitted.

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