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Crystallographic report

Bis(3-methyl-2-cyclopenten-1-one)dichloro-oxovanadium(IV)

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The crystal structure of the oxovanadium(IV) complex $(CH_3C_5H_5O)_2VOCl_2$ was determined. The molecule has trigonal bipyramidal geometry, with oxygen atoms of cyclopentenones in axial positions and oxygen and two chlorine atoms in equatorial positions. Copyright © 2005 John Wiley & Sons, Ltd.

KEYWORDS: vanadyl(IV); crystal structure

COMMENT

The title compound $(CH_3C_5H_5O)_2VOCl_2$ has rigorous C_2 symmetry (Figure 1). The coordination at the vanadium centre approaches the trigonal bipyramidal geometry, with oxygen atoms of cyclopentenones in axial positions and two chlorine atoms and oxygen in equatorial positions. The bond angle O2–V1–O2′ (162.9°) is much larger than in compounds $[(CH_3)_2NCOCH_3]_2VOCl_2$ (156.6°),[1] $\{[(CH_3)_2N]_2CO\}_2VOCl_2$ (150.9°),[2] $(H_2O)_2VOCl_2$ (153.2–153.8°)[3] and $(Ph_3PO)_2VOCl_2$ (155.4°).[4] The bond lengths V=O (1.580 Å) and V–Cl (2.290 Å) are in the range of pentacoordinated dichloro-oxovanadium(IV) compounds (V=O, 1.55–1.65 Å; V–Cl, 2.25–2.50 Å).[1–7] The coordinated carbonyl group has longer C=O bond length (1.251 Å) than is usual in free ketones (1.22 Å).[8]

EXPERIMENTAL

Synthesis

To a solution of 0.002 mol of $VOCl_2$ in 100 ml of THF (prepared by a literature procedure)[9] was added 0.39 g (0.004 mol) of 3-methyl-2-cyclopenten-1-one (Aldrich), and the solution was stirred overnight. Then, the solvent was evaporated and the product was recrystallized in CH_2Cl_2. Yield = 0.54 g (82%). The crystals suitable for X-ray analysis were obtained by careful layering of the CH_2Cl_2 solution with a double volume of hexane. Electron paramagnetic resonance (CH_2Cl_2): $A_{iso} = 99.0 \times 10^{-4}$ cm^{-1}, $g_{iso} = 1.9746$.

Crystallography

Crystal data for $C_{12}H_{16}Cl_2O_3V$: $M_r = 330.09$, $0.2 \times 0.17 \times 0.05$ mm^3, monoclinic, $C2/c$, $a = 11.2680(6)$ Å, $b = 9.3150(3)$ Å, $c = 13.9330(8)$ Å, $\beta = 103.012(2)°$, $V = 1424.88(12)$ Å^3, $Z =$

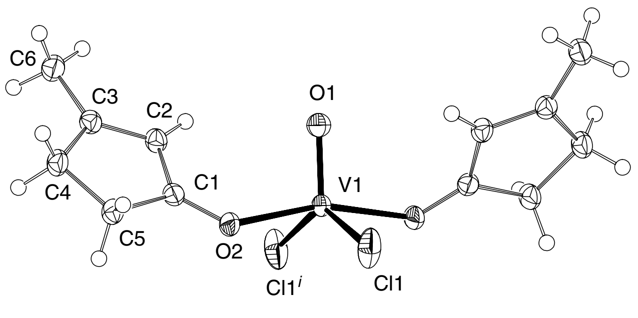

Figure 1. ORTEP drawing of molecular structure of $(CH_3C_5H_5O)_2VOCl_2$ (ellipsoids: 50% probability). Important bond distances (in Å) and angles (in deg): V1–O1 = 1.580(2), V1–O2 = 2.0431(14), V1–Cl1 = 2.2904(6), O2–V1–O2^i = 162.88(8), O1–V1–Cl1 = 111.72(2), Cl1–V1–Cl1^i = 136.57 (4), V1–O2–C1 = 130.20(13), C1–O2 = 1.251(2), C1–C2 = 1.436(3), C2–C3 = 1.349(3), C3–C4 = 1.499(3), C4–C5 = 1.523(3), C1–C5 = 1.504(3), C3–C6 = 1.479(3). Symmetry code *i*: $1 - x, y, 0.5 - z$.

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4, $D_x = 1.539$ g cm^{-3}. Nonius KappaCCD diffractometer, $T = 150(2)$ K, $\theta_{\max} = 27.45°$, $\mu(\text{Mo K}\alpha) = 1.067$ mm^{-1}, $\lambda = 0.71073$ Å, 10026 measured reflections, 1636 independent, $R_{int} = 0.035$, 1419 reflections with $I > 2\sigma(I)$, $R = 0.0340$ for observed diffractions, $wR(F^2) = 0.0866$ for all diffractions. Programs used: DENZO-SMN,[10] Sir92,[11] SHELXL97[12] and PLATON.[13] CCDC deposition number: 232 818.

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