THE CRYSTAL STRUCTURE OF BIS(η^5 -CYCLOPENTADIENYL-TRICARBONYLTUNGSTEN)TIN DICHLORIDE, $[\text{Cp}(\text{CO})_3\text{W}]_2\text{SnCl}_2$

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Figure 1. Molecular structure (30 % probability ellipsoids) of $[\text{Cp}(\text{CO})_3\text{W}]_2\text{SnCl}_2$. Selected bond distances and angles: Sn(1)-Cl(1) 2.416(2), Sn(1)-Cl(2) 2.421(2), Sn(1)-W(1) 2.7965(5), Sn(1)-W(2) 2.7854(5) Å; Cl(1)-Sn(1)-Cl(2) 94.17(7), Cl(1)-Sn(1)-W(1) 104.90(5), Cl(1)-Sn(1)-W(2) 107.84(5), Cl(2)-Sn(1)-W(1) 107.76(5), Cl(2)-Sn(1)-W(2) 104.82(5), W(1)-Sn(1)-W(2) 131.24(2)°.

Comment

In context with our interest in stannasiloxanes^{1a} such as $\text{R}_2\text{Sn}(\text{OSiR}'_3)_2$ (R = alkyl, $\text{Cp}(\text{CO})_2\text{Fe}$, $\text{Cp}(\text{CO})_3\text{W}$)^{1b} we prepared the title compound, hereafter referred to as **2**, according to the procedure described by Nesmeyanov et al.² and report here its molecular structure as well as NMR data. The molecular structure of compound **2** resembles that of the dianion $\{[(\text{CO})_5\text{W}]_2\text{SnCl}_2\}^{2-}$, which appears to be the only other crystallographically characterised compound with a W_2SnCl_2 substituent pattern.^{2b}

The tin atom in **2** shows a strongly distorted tetrahedral configuration with angles between 94.17(7) (Cl-Sn-Cl) and 131.24(2)° (W-Sn-W). The Sn-Cl bonds of 2.416(2) and 2.421(2) Å are slightly shorter as compared to the corresponding bond lengths (2.473(2), 2.484(2) Å) of the dianion $\{[(\text{CO})_5\text{W}]_2\text{SnCl}_2\}^{2-}$.^{2b}

Experimental

Compound **2** was prepared as described in the literature^{2a} by bubbling dry hydrogen chloride gas through a solution of $[\text{Cp}(\text{CO})_3\text{W}]_2\text{SnPh}_2$ (**1**) in CH_2Cl_2 .

Compound 1.

 ^1H NMR (CDCl_3) δ : 7.7-7.2 ppm (10H, m; Ph), 5.16 ppm (5H, s; Cp). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3) δ : 218.6, 217.0 ppm (CO), 148.0 ($^1J(^{13}\text{C}-^{119}\text{Sn}) = 251$ Hz; Cl), 135.8 (Co), 128.7 (Cp), 127.8 (Cm), 89.4 ppm (Cp). $^{119}\text{Sn}\{^1\text{H}\}$ NMR (CDCl_3) δ : -19.0 ppm ($^1J(^{119}\text{Sn}-^{183}\text{W}) = 225$ Hz).

Compound 2.

 ^1H NMR (CDCl_3) δ : 5.69 ppm (5H, s; Cp). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3) δ : 215.9, 213.8 ppm (CO), 91.0 ppm (Cp). $^{119}\text{Sn}\{^1\text{H}\}$ NMR (CDCl_3) δ : 179.7 ppm ($^1J(^{119}\text{Sn}-^{183}\text{W}) = 453$ Hz).IR (KBr): ν_{CO} 2047, 2018, 2000, 1935, 1918, 1890 cm^{-1} .**Table 1.** Crystal data for $[\text{Cp}(\text{CO})_3\text{W}]_2\text{SnCl}_2$

Formula	$\text{C}_{16}\text{H}_{10}\text{Cl}_2\text{O}_6\text{SnW}_2$	Formula weight	855.53
cryst syst	orthorhombic	Crystal size, mm	0.25 x 0.15 x 0.15
space group	Pbca	a, Å	11.583(1)
b, Å	14.868(1)	c, Å	23.919(1)
V, Å ³	4119.2(5)	Z	8
Temperature, K	291	Diffractometer	Nonius KappaCCD
μ (Mo-K α)	12.634 mm^{-1}	Trans. factors	—
D_{calcd} , g cm^{-3}	2.759	F(000)	3088
θ_{max} , °	30.5	Refins meas.	48384
Refins unique, R_{int}	6268, 0.068	Refins with $I > 2\sigma(I)$	3207
Weighting scheme	$1/[\sigma^2(F_o^2) + (0.0244P)^2]$ with $P = (F_o^2 + 2F_c^2)/3$	R(F) ($I > 2\sigma(I)$)	0.0349
p, e Å ⁻³	0.976	$R_w(F^2)$ (all data)	0.0676
		Programs used	SHELXS97 ³ , SHELXL97 ⁴ , SHELXTL ⁵
Deposition number	CCDC 147231		

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