Crystal structure of *tert*-butyl-1κ*C*-2(η^5-cyclopentadienyl)dichloro-1κ^2*Cl*-tricarbonyl-2κ^3*C*-tintungsten (*Sn—W*), *tert*-Bu[Cp(CO)$_3$W]SnCl$_2$

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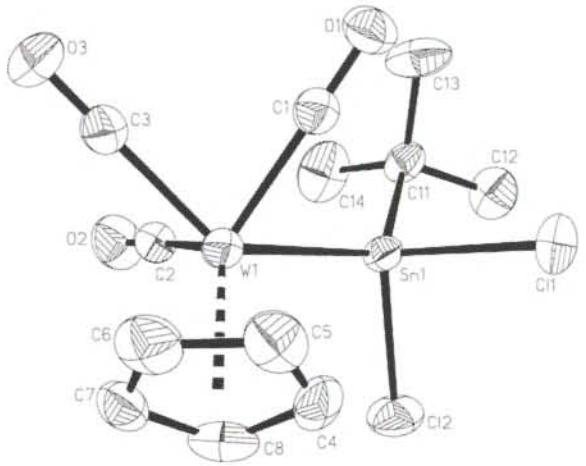

Abstract

$C_{12}H_{14}Cl_2O_3SnW$, triclinic, $P\bar{1}$ (No. 2), a = 7.590(1) Å, b = 9.083(1) Å, c = 12.786(1) Å, α = 75.448(1)°, β = 80.461(1)°, γ = 80.326(1)°, V = 834.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.040, $wR(F^2)$ = 0.099, T = 170 K.

Source of material

Crystals of the title compound were obtained from the reaction of [Cp(CO)$_3$W]^{t}BuSnPh$_2$ with gaseous HCl and recrystallisation of the crude product from CH_2Cl_2/n-pentane (1:1); mp 380 K – 382 K [1].

Discussion

The tin atom shows a significantly distorted tetrahedral coordination with angles in the range from 130.7(2)° for C(11)–Sn(1)–W(1) to 98.66(11)° for Cl(2)–Sn(1)–Cl(1). The coordination polyhedron around tungsten can be described as a square pyramid with C(1), C(2), C(3), Sn(1) in the plane. The Sn–W distance amounts to 2.7519(6) Å and fits into the range of Sn–W distances between 2.706(1) [2] and 2.897 Å [3] given in the Cambridge Structural Data Base for 28 entries.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.1 × 0.25 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	87.08 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, 714 frames via ω-rotation (Δω = 1°) and two times 5s per frame
$2\theta_{max}$:	57.32°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	21435, 4275
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2730
$N(param)_{refined}$:	178
Programs:	DENZO [4], SCALEPACK [4], SHELXS-90 [5], SHELXTL-Plus [6], SHELXL-97 [7]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(4)	2*i*	0.7286	0.4798	0.2676	0.11(2)
H(5)	2*i*	0.6813	0.2779	0.4360	0.11
H(6)	2*i*	0.5874	0.0602	0.3859	0.11
H(7)	2*i*	0.5683	0.1276	0.1875	0.11
H(8)	2*i*	0.6646	0.3848	0.1112	0.11
H(12A)	2*i*	0.0449	1.0715	0.1688	0.10(1)
H(12B)	2*i*	0.2129	1.0135	0.0933	0.10
H(12C)	2*i*	0.2299	1.0119	0.2141	0.10
H(13A)	2*i*	0.0644	0.8402	0.3649	0.10
H(13B)	2*i*	–0.0393	0.7195	0.3409	0.10
H(13C)	2*i*	–0.1146	0.8944	0.3121	0.10
H(14A)	2*i*	–0.0120	0.6947	0.1436	0.10
H(14B)	2*i*	0.0637	0.8277	0.0521	0.10
H(14C)	2*i*	–0.1179	0.8609	0.1261	0.10

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
W(1)	2*i*	0.38941(4)	0.36042(3)	0.29789(3)	0.0445(2)	0.0433(2)	0.0514(2)	–0.0069(1)	–0.0000(1)	–0.0128(1)
Sn(1)	2*i*	0.34442(7)	0.67425(5)	0.22247(4)	0.0409(3)	0.0446(3)	0.0450(3)	–0.0097(2)	–0.0005(2)	–0.0071(2)
Cl(1)	2*i*	0.4936(4)	0.7830(3)	0.3307(2)	0.081(2)	0.064(1)	0.083(2)	–0.020(1)	–0.029(1)	–0.015(1)

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Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(2)	2*i*	0.5287(4)	0.7431(3)	0.0527(2)	0.087(2)	0.095(2)	0.062(2)	–0.026(1)	0.025(1)	–0.007(1)
O(1)	2*i*	0.217(1)	0.4957(7)	0.5004(5)	0.091(5)	0.072(4)	0.053(4)	–0.013(3)	0.007(3)	–0.019(3)
O(2)	2*i*	0.130(1)	0.4143(9)	0.1213(7)	0.080(5)	0.103(6)	0.089(5)	–0.003(4)	–0.029(4)	–0.039(5)
O(3)	2*i*	0.105(1)	0.141(1)	0.4152(7)	0.115(7)	0.099(6)	0.094(6)	–0.060(5)	0.028(5)	–0.025(5)
C(1)	2*i*	0.278(1)	0.4493(9)	0.4246(7)	0.057(5)	0.050(4)	0.060(5)	–0.010(4)	–0.003(4)	–0.012(4)
C(2)	2*i*	0.221(1)	0.401(1)	0.1854(8)	0.053(5)	0.055(5)	0.065(5)	–0.009(4)	0.001(4)	–0.021(4)
C(3)	2*i*	0.206(1)	0.221(1)	0.3704(8)	0.070(6)	0.051(5)	0.069(6)	–0.007(4)	0.001(5)	–0.018(4)
C(4)	2*i*	0.693(1)	0.385(1)	0.272(1)	0.034(4)	0.067(6)	0.14(1)	–0.005(4)	–0.011(5)	–0.041(7)
C(5)	2*i*	0.666(1)	0.273(1)	0.366(1)	0.066(7)	0.086(7)	0.083(8)	0.007(5)	–0.023(5)	–0.026(6)
C(6)	2*i*	0.613(2)	0.152(1)	0.338(1)	0.069(7)	0.067(6)	0.104(9)	0.018(5)	–0.006(6)	–0.014(6)
C(7)	2*i*	0.604(1)	0.189(1)	0.2265(9)	0.054(5)	0.070(6)	0.079(7)	0.004(4)	–0.008(5)	–0.037(5)
C(8)	2*i*	0.656(1)	0.332(1)	0.1841(9)	0.056(6)	0.099(8)	0.063(6)	0.010(5)	0.014(5)	–0.009(6)
C(11)	2*i*	0.101(1)	0.8377(9)	0.2027(7)	0.049(5)	0.046(4)	0.066(5)	–0.010(3)	–0.006(4)	–0.001(4)
C(12)	2*i*	0.152(2)	0.998(1)	0.166(1)	0.083(7)	0.051(5)	0.096(8)	0.002(5)	–0.022(6)	–0.001(5)
C(13)	2*i*	–0.007(2)	0.822(1)	0.316(1)	0.059(6)	0.094(8)	0.098(9)	0.006(5)	0.022(6)	0.001(6)
C(14)	2*i*	–0.000(2)	0.802(1)	0.124(1)	0.083(9)	0.090(8)	0.13(1)	–0.006(6)	–0.050(8)	–0.008(8)

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References

1. Beckmann, J.; Jurkschat, K.; Kaltenbrunner, U.; Pieper, N.; Schürmann, M.: Strained Metallastannoxanes - Ring-Opening Polymerisation Versus Retention of Six-Membered Ring-Structure. Organometallics **18** (1999) 1586-1595.
2. Bohra, R.; Hitchcock, P. B.; Lappert, M. F.; Au-Yeung, S. C. F.; Leung, W. P.: Group 6 Metallocene(IV)-Main Group Metal Complexes: Synthesis and Structures of a Mo-B-Li Compound and of [W(η-C_5H_5)$_2${$SnCl_2CH(SiMe_3)_2$}]. J. Chem. Soc., Dalton Trans. (1995) 2999-3005.
3. Carriedo, G. A.; Hodgson, D.; Howard, J. A. K.; Marsden, K.; Stone, F. G. A.; Went, M. J.; Woodward, P.: The Synthesis and X-Ray Crystal Structures of [MoW_2(η-CR)$_2$(CO)$_4$)(η-C_5H_5)$_2$] (R=(C_6H_4Me-4), [N(PPh_3)$_2$] [W_2(η-CHR)$_2$(CO)$_7$)(η-C_5H_5)] [AuW_2(η-CHR)$_2$(CO)$_2$)(PPh_3)(η-C_5H_5)], and [W=CHR)($SnPh_3$)(CO)$_2$)(η-C_5H_5)]. J. Chem. Soc., Chem. Commun. (1982) 1006-1008.
4. Otwinowski, Z.; Minor, W.: Processing of X-Ray Diffraction Data Collected in Oscillation Mode. In Methods in Enzymology, Volume 276, Macromolecular Crystallography, Part A (Eds. Carter, C. W. & Sweet, R. M.), p. 307-326. Academic Press 1997.
5. Sheldrick, G. M.: Phase Annealing in SHELXS-90: Direct Methods for Larger Structures. Acta Crystallogr. A**46** (1990) 467-473.
6. Sheldrick, G. M.: SHELXTL-Plus. Release 4.1 Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA 1991.
7. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures, University of Göttingen, Germany 1997.