Crystal structure of chlorobis(η^5-pentamethylcyclopentadienyl)(η^1-2-propenyl)zirconium, $C_{23}H_{35}ClZr$

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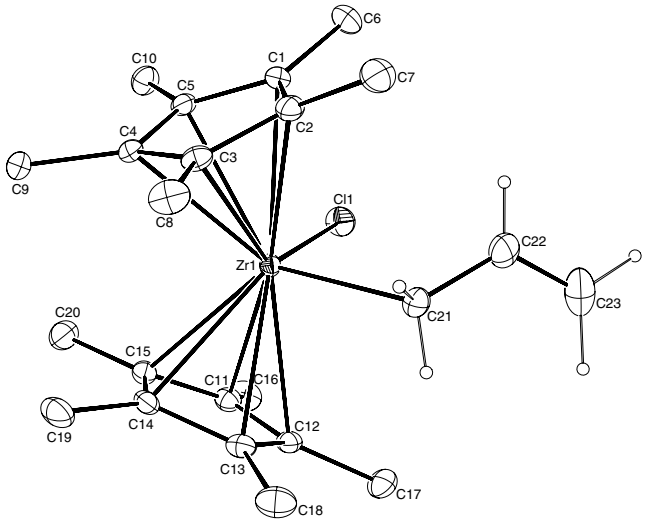

Abstract

$C_{23}H_{35}ClZr$, monoclinic, $P12_1/n1$ (No. 14), $a = 8.6485(1)$ Å, $b = 26.9339(5)$ Å, $c = 9.9577(2)$ Å, $\beta = 112.3270(9)°$, $V = 2145.7$ Å^3, $Z = 4$, $R_{gt}(F) = 0.031$, $wR_{ref}(F^2) = 0.067$, $T = 150$ K.

Source of material

The compound was synthesized as described previously [1] and was crystallized by evaporation of a saturated hexane solution.

Discussion

The structural result reveals that the compound is a 16 electron σ allyl, rather than an 18 electron π allyl, complex. The average Zr—C distance for the pentamethylcyclopentadienyl ligands is 2.559(7) Å, although the distances for the carbon atoms close to the Cl or allyl ligands are longer than the others. The Zr—C21 and Zr—Cl distances are shorter, at 2.325(2) Å and 2.4362(5) Å, respectively. As is normal, the methyl substituents tilt out of the cyclopentadienyl planes by an average of 7.6°, away from the zirconium center.

Table 1. Data collection and handling.

Crystal:	yellow plate, size 0.08 × 0.13 × 0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	6.40 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, φ/ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7788, 4753
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3934
$N(param)_{refined}$:	366
Programs:	SIR97 [2], SHELXL-97 [3]

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

Atom	Site	x	y	z	U_{iso}
H(6A)	4*e*	0.064(4)	0.045(1)	0.038(3)	0.059(9)
H(6B)	4*e*	0.030(3)	0.094(1)	–0.065(3)	0.056(8)
H(6C)	4*e*	0.068(4)	0.040(1)	–0.109(3)	0.060(9)
H(7A)	4*e*	0.442(3)	–0.032(1)	0.128(3)	0.059(9)
H(7B)	4*e*	0.279(5)	–0.016(1)	0.155(4)	0.08(1)
H(7C)	4*e*	0.458(3)	–0.014(1)	0.275(3)	0.052(8)
H(8A)	4*e*	0.739(3)	0.011(1)	0.185(3)	0.047(8)
H(8B)	4*e*	0.758(3)	0.035(1)	0.322(3)	0.046(8)
H(8C)	4*e*	0.823(3)	0.061(1)	0.215(3)	0.044(8)
H(9A)	4*e*	0.658(3)	0.1215(9)	–0.082(3)	0.036(7)
H(9B)	4*e*	0.763(4)	0.134(1)	0.077(3)	0.055(9)
H(9C)	4*e*	0.631(3)	0.170(1)	–0.016(3)	0.053(8)
H(10A)	4*e*	0.223(4)	0.139(1)	–0.216(3)	0.059(9)
H(10B)	4*e*	0.167(4)	0.164(1)	–0.106(3)	0.053(8)
H(10C)	4*e*	0.338(3)	0.180(1)	–0.123(3)	0.046(7)
H(16A)	4*e*	0.241(3)	0.243(1)	0.361(3)	0.048(8)
H(16B)	4*e*	0.284(3)	0.263(1)	0.232(3)	0.050(8)
H(16C)	4*e*	0.374(4)	0.284(1)	0.387(3)	0.058(9)
H(17A)	4*e*	0.486(3)	0.154(1)	0.663(3)	0.047(8)
H(17B)	4*e*	0.326(3)	0.1719(9)	0.551(3)	0.037(7)
H(17C)	4*e*	0.452(3)	0.211(1)	0.646(3)	0.039(7)
H(18A)	4*e*	0.721(4)	0.114(1)	0.661(4)	0.08(1)
H(18B)	4*e*	0.802(4)	0.095(1)	0.573(3)	0.08(1)
H(18C)	4*e*	0.877(5)	0.140(1)	0.664(4)	0.10(1)
H(19A)	4*e*	0.890(4)	0.135(1)	0.349(3)	0.06(1)
H(19B)	4*e*	0.956(4)	0.180(1)	0.433(3)	0.067(9)
H(19C)	4*e*	0.869(4)	0.186(1)	0.267(4)	0.08(1)
H(20A)	4*e*	0.666(4)	0.237(1)	0.146(3)	0.055(9)
H(20B)	4*e*	0.475(4)	0.235(1)	0.078(3)	0.062(9)
H(20C)	4*e*	0.568(3)	0.277(1)	0.187(3)	0.054(8)
H(21A)	4*e*	0.507(3)	0.045(1)	0.424(3)	0.044(7)
H(21B)	4*e*	0.470(3)	0.0848(9)	0.521(3)	0.036(7)
H(22)	4*e*	0.208(3)	0.032(1)	0.325(3)	0.051(8)
H(23A)	4*e*	0.059(4)	0.043(1)	0.477(3)	0.063(9)
H(23B)	4*e*	0.236(4)	0.074(1)	0.595(3)	0.07(1)

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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}
Zr(1)	4*e*	0.42750(2)	0.127924(7)	0.25744(2)	0.0154(1)	0.0179(1)	0.0173(1)	–0.00074(8)	0.00710(8)	0.00058(8)
Cl(1)	4*e*	0.14086(6)	0.15763(2)	0.18191(6)	0.0180(3)	0.0404(3)	0.0351(3)	0.0027(2)	0.0106(2)	–0.0039(2)
C(1)	4*e*	0.2810(3)	0.07364(8)	0.0322(2)	0.022(1)	0.023(1)	0.020(1)	–0.0054(8)	0.0078(9)	–0.0050(8)
C(2)	4*e*	0.4104(3)	0.04351(8)	0.1267(2)	0.032(1)	0.019(1)	0.022(1)	–0.0017(9)	0.011(1)	–0.0021(9)
C(3)	4*e*	0.5658(3)	0.06580(8)	0.1444(2)	0.024(1)	0.023(1)	0.019(1)	0.0053(9)	0.0072(9)	–0.0026(8)
C(4)	4*e*	0.5318(2)	0.10865(7)	0.0543(2)	0.022(1)	0.020(1)	0.017(1)	–0.0005(8)	0.0092(9)	–0.0027(8)
C(5)	4*e*	0.3558(2)	0.11415(8)	–0.0115(2)	0.023(1)	0.022(1)	0.015(1)	–0.0004(8)	0.0067(9)	–0.0015(8)
C(6)	4*e*	0.0970(3)	0.0621(1)	–0.0274(3)	0.026(1)	0.037(2)	0.034(2)	–0.011(1)	0.008(1)	–0.005(1)
C(7)	4*e*	0.3932(4)	–0.00809(9)	0.1756(3)	0.051(2)	0.021(1)	0.035(2)	–0.002(1)	0.017(1)	0.001(1)
C(8)	4*e*	0.7332(3)	0.0418(1)	0.2230(3)	0.029(1)	0.033(1)	0.030(1)	0.012(1)	0.007(1)	–0.001(1)
C(9)	4*e*	0.6554(3)	0.13514(9)	0.0066(3)	0.027(1)	0.033(1)	0.025(1)	–0.006(1)	0.015(1)	–0.005(1)
C(10)	4*e*	0.2634(3)	0.15283(9)	–0.1211(3)	0.029(1)	0.031(1)	0.023(1)	0.003(1)	0.009(1)	0.006(1)
C(11)	4*e*	0.4654(3)	0.21617(8)	0.3643(2)	0.023(1)	0.021(1)	0.025(1)	–0.0003(8)	0.0090(9)	–0.0042(9)
C(12)	4*e*	0.5177(3)	0.18349(8)	0.4842(2)	0.020(1)	0.026(1)	0.020(1)	–0.0033(8)	0.0079(9)	–0.0070(9)
C(13)	4*e*	0.6642(3)	0.15850(8)	0.4868(2)	0.019(1)	0.025(1)	0.022(1)	0.0005(8)	0.0043(9)	–0.0052(9)
C(14)	4*e*	0.7009(3)	0.17585(8)	0.3676(2)	0.019(1)	0.027(1)	0.027(1)	–0.0076(9)	0.0114(9)	–0.0108(9)
C(15)	4*e*	0.5739(3)	0.21066(8)	0.2893(2)	0.030(1)	0.022(1)	0.025(1)	–0.0083(9)	0.014(1)	–0.0069(9)
C(16)	4*e*	0.3313(3)	0.2547(1)	0.3337(3)	0.038(2)	0.026(1)	0.044(2)	0.008(1)	0.012(1)	–0.003(1)
C(17)	4*e*	0.4388(3)	0.1804(1)	0.5942(3)	0.034(1)	0.042(2)	0.028(1)	–0.001(1)	0.017(1)	–0.007(1)
C(18)	4*e*	0.7764(3)	0.1251(1)	0.6040(3)	0.027(1)	0.039(2)	0.031(1)	0.005(1)	0.000(1)	–0.001(1)
C(19)	4*e*	0.8645(3)	0.1678(1)	0.3523(3)	0.023(1)	0.054(2)	0.045(2)	–0.011(1)	0.017(1)	–0.023(2)
C(20)	4*e*	0.5672(4)	0.2428(1)	0.1634(3)	0.061(2)	0.026(1)	0.033(2)	–0.014(1)	0.025(2)	–0.005(1)
C(21)	4*e*	0.4230(3)	0.07010(9)	0.4292(3)	0.032(1)	0.027(1)	0.023(1)	–0.002(1)	0.011(1)	0.003(1)
C(22)	4*e*	0.2581(3)	0.0501(1)	0.4076(3)	0.036(1)	0.043(2)	0.027(1)	–0.002(1)	0.007(1)	0.012(1)
C(23)	4*e*	0.1761(4)	0.0555(1)	0.4955(4)	0.036(2)	0.055(2)	0.060(2)	0.004(1)	0.022(2)	0.025(2)

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