Crystal structure of chlorotris(2,2,6,6-tetramethyl-3,5-heptanedionato)-zirconium(IV), $[\text{ZrCl}\{(\text{CH}_3)_3\text{CCOCHCOC}(\text{CH}_3)_3\}_3]$

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$\text{C}_{33}\text{H}_{57}\text{ClO}_6\text{Zr}$, monoclinic, $P2_1$ (No. 4), $a = 10.679(5)$ Å, $b = 18.263(7)$ Å, $c = 10.973(5)$ Å, $\beta = 117.68(5)^\circ$, $V = 1895.2$ Å³, $Z = 2$, $R(F) = 0.049$, $R_w(F) = 0.049$.

Table 1. Parameters used for the X-ray data collection

Crystal:	white prism, max. dim. 0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	261.00 cm ⁻¹
Diffractometer:	Philips
Scan mode:	2 θ
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	54°
$N(hkl)_{\text{unique}}$:	2524
Criterion for F_o :	$F_o > 3 \sigma(F_o)$
$N(\text{param})_{\text{refined}}$:	379
Program:	SHELX-76

Source of material: The title compound was obtained by reacting 2,2,6,6-tetramethyl-3,5-heptanedione with ZrCl_4 (molar ratio 1:4) in ethanol. White crystals separated from the mother solution on standing.

Methyl H atoms were not introduced in the calculations. One chlorine atom and three bidentate ligands are directly bonded to the Zr atom forming an irregular coordination polyhedron. Zr–O bonds range from 2.09 Å to 2.15 Å; the Zr–Cl distance is 2.496(3) Å.

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{iso}
H(6)	2a	1.169(1)	0.483(1)	0.828(1)	0.07
H(17)	2a	0.699(1)	0.709(1)	0.253(1)	0.07
H(28)	2a	0.711(1)	0.256(1)	0.237(1)	0.07

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zr(1)	2a	0.8482(1)	0.4593	0.3747(1)	0.0333(3)	0.0565(4)	0.0348(3)	−0.0017(8)	0.0149(2)	0.0001(8)
Cl(1)	2a	0.7498(2)	0.4611(5)	0.1192(2)	0.0719(8)	0.0904(9)	0.0346(7)	0.0147(9)	0.0152(6)	−0.0035(9)
O(1)	2a	1.0691(4)	0.4570(7)	0.5131(4)	0.0365(9)	0.0781(9)	0.0362(9)	−0.007(1)	0.0136(8)	−0.008(1)
O(2)	2a	0.8539(4)	0.4600(8)	0.5706(4)	0.0435(9)	0.0984(9)	0.0366(9)	0.012(1)	0.0229(8)	0.010(1)
O(3)	2a	0.6782(7)	0.5325(5)	0.3425(6)	0.0551(9)	0.061(1)	0.0604(9)	0.0004(9)	0.0470(9)	0.0055(9)
O(4)	2a	0.9253(6)	0.5637(5)	0.3496(6)	0.0375(9)	0.0371(9)	0.0543(9)	−0.0096(9)	0.0295(9)	0.0017(9)
O(5)	2a	0.9210(7)	0.3563(6)	0.3542(7)	0.0505(9)	0.086(1)	0.0508(9)	−0.0139(9)	0.0064(9)	0.001(1)
O(6)	2a	0.6797(7)	0.3855(6)	0.3434(7)	0.0353(9)	0.067(1)	0.0609(9)	−0.0147(9)	0.0075(9)	−0.0060(9)
C(1)	2a	1.3069(6)	0.4616(8)	0.6871(6)	0.0312(9)	0.068(1)	0.0475(9)	−0.008(1)	0.0120(9)	−0.001(1)
C(2)	2a	1.3556(8)	0.3836(4)	0.7210(8)	0.048(1)	0.099(1)	0.188(1)	0.011(1)	0.036(1)	0.002(1)
C(3)	2a	1.3349(8)	0.4888(5)	0.5742(6)	0.061(1)	0.209(1)	0.102(1)	−0.021(1)	0.037(1)	0.048(1)
C(4)	2a	1.3893(8)	0.5079(5)	0.8148(6)	0.048(1)	0.175(1)	0.109(1)	−0.038(1)	0.010(1)	−0.075(1)
C(5)	2a	1.1497(6)	0.4627(8)	0.6406(6)	0.0321(9)	0.0480(9)	0.0443(9)	−0.012(1)	0.0130(9)	−0.012(1)
C(6)	2a	1.0969(6)	0.4591(9)	0.7373(6)	0.0411(9)	0.085(1)	0.0386(9)	−0.012(1)	0.0129(9)	−0.003(1)

Table 3. (Continued)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(7)	2a	0.9524(6)	0.4564(8)	0.6988(6)	0.0367(9)	0.065(1)	0.0409(9)	0.015(1)	0.0128(9)	-0.017(1)
C(8)	2a	0.8934(6)	0.4540(7)	0.8012(6)	0.0510(9)	0.069(1)	0.0399(9)	0.006(1)	0.0253(9)	-0.005(1)
C(9)	2a	0.8029(9)	0.3854(7)	0.7803(8)	0.106(1)	0.096(1)	0.078(1)	-0.036(1)	0.025(1)	0.007(1)
C(10)	2a	1.0141(7)	0.4695(8)	0.9509(6)	0.073(1)	0.132(1)	0.0385(9)	0.029(1)	0.0239(9)	-0.009(1)
C(11)	2a	0.7965(7)	0.5228(6)	0.7684(7)	0.0853(9)	0.076(1)	0.0790(9)	0.013(1)	0.0745(9)	0.004(1)
C(12)	2a	0.5089(8)	0.6190(7)	0.2759(8)	0.061(1)	0.065(1)	0.071(1)	0.012(1)	0.0290(9)	0.002(1)
C(13)	2a	0.4511(9)	0.7017(7)	0.2044(9)	0.080(1)	0.108(1)	0.187(1)	0.035(1)	0.017(1)	0.043(1)
C(14)	2a	0.5000(8)	0.6170(7)	0.4122(8)	0.062(1)	0.151(1)	0.099(1)	0.026(1)	0.0520(9)	-0.007(1)
C(15)	2a	0.3828(8)	0.5677(8)	0.1694(9)	0.068(1)	0.145(1)	0.149(1)	0.000(1)	0.032(1)	-0.044(1)
C(16)	2a	0.6423(8)	0.5950(7)	0.2938(8)	0.038(1)	0.067(1)	0.051(1)	-0.009(1)	0.0090(9)	-0.018(1)
C(17)	2a	0.7310(8)	0.6410(6)	0.2634(8)	0.055(1)	0.044(1)	0.061(1)	0.007(1)	0.012(1)	0.012(1)
C(18)	2a	0.8617(7)	0.6211(5)	0.2874(7)	0.0382(9)	0.036(1)	0.0414(9)	-0.0107(9)	0.0299(9)	-0.0093(9)
C(19)	2a	0.9497(8)	0.6765(7)	0.2521(8)	0.073(1)	0.079(1)	0.079(1)	0.024(1)	0.037(1)	0.019(1)
C(20)	2a	0.9338(8)	0.6435(8)	0.1168(8)	0.149(1)	0.165(1)	0.105(1)	-0.029(1)	0.093(1)	0.013(1)
C(21)	2a	0.9203(8)	0.7564(7)	0.2679(9)	0.117(1)	0.071(1)	0.177(1)	-0.011(1)	0.079(1)	0.024(1)
C(22)	2a	1.1166(8)	0.6659(7)	0.3887(9)	0.068(1)	0.137(1)	0.164(1)	-0.034(1)	0.036(1)	0.018(1)
C(23)	2a	0.9633(7)	0.2491(6)	0.2554(8)	0.0450(9)	0.053(1)	0.078(1)	0.012(1)	0.0342(9)	-0.011(1)
C(24)	2a	0.8831(8)	0.1793(7)	0.1673(8)	0.078(1)	0.126(1)	0.142(1)	-0.018(1)	0.037(1)	-0.093(1)
C(25)	2a	1.0913(8)	0.2275(7)	0.3715(8)	0.091(1)	0.151(1)	0.088(1)	0.056(1)	0.005(1)	-0.034(1)
C(26)	2a	1.0143(8)	0.2959(7)	0.1688(8)	0.116(1)	0.155(1)	0.114(1)	0.033(1)	0.081(1)	0.010(1)
C(27)	2a	0.8739(8)	0.2912(7)	0.3002(8)	0.046(1)	0.065(1)	0.056(1)	-0.008(1)	-0.010(1)	0.002(1)
C(28)	2a	0.7274(8)	0.2773(7)	0.2652(8)	0.055(1)	0.089(1)	0.077(1)	-0.009(1)	0.0412(9)	-0.004(1)
C(29)	2a	0.6457(7)	0.3205(6)	0.2966(7)	0.0441(9)	0.052(1)	0.0347(9)	-0.0176(9)	0.0222(9)	0.0071(9)
C(30)	2a	0.4903(7)	0.2969(6)	0.2667(8)	0.0199(9)	0.064(1)	0.059(1)	-0.0104(9)	0.0128(9)	-0.002(1)
C(31)	2a	0.4643(8)	0.3301(8)	0.3829(8)	0.086(1)	0.152(1)	0.120(1)	-0.002(1)	0.064(1)	-0.021(1)
C(32)	2a	0.3974(8)	0.3379(7)	0.1326(9)	0.041(1)	0.109(1)	0.121(1)	0.007(1)	-0.014(1)	0.029(1)
C(33)	2a	0.4784(8)	0.2171(6)	0.2577(8)	0.073(1)	0.078(1)	0.103(1)	-0.037(1)	0.0528(9)	-0.024(1)

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