Crystal structure of dichloro-(3-cyclopentadienyl-3-methyl-1-phenyl-butyliden-2,6-dimethylphenylamino)chromium(III), $CrCl_2(C_{24}H_{26}N)$

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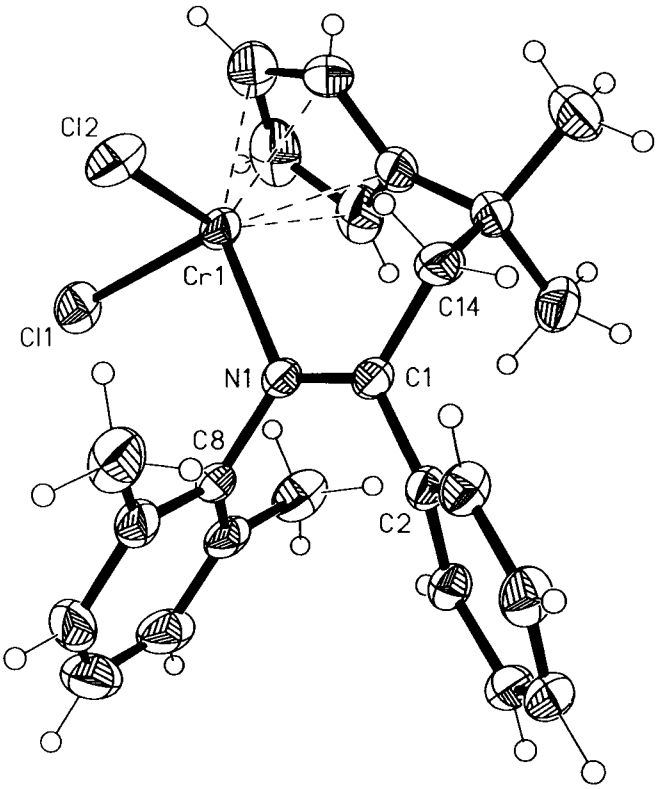

Abstract

$C_{24}H_{26}Cl_2CrN$, monoclinic, $P12_1/c1$ (no. 14), a = 11.5168(5) Å, b = 13.9721(6) Å, c = 14.1146(4) Å, β = 104.628(2)°, V = 2197.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.039, $wR_{ref}(F^2)$ = 0.095, T = 233 K.

Source of material

The chromium complex was obtained by methods analogous to Huang's [1] procedure: First, the ketimine of acetophenone and 2,6-dimethylaniline were formed. Second, deprotonation with lithium diisopropylamide afforded the corresponding α-lithiated ketimine. Third, nucleophilic addition of this anion to the exocyclic bond of 6,6-dimethylfulvene gave the corresponding iminocyclopentadienide. Fourth and last, this ligand was reacted with chromium(III)chloride. X-ray quality crystals of the chromium complex were obtained from toluene.

Discussion

The Cr(III) cation adopts a distorted tetrahedral coordination with Cr—Cl distances of 2.278(1) Å and 2.290(1) Å, Cr—N of 2.141(2) Å and averaged distances to the cyclopentadienyl carbon atoms of 2.233(3) Å. The bond angles Cl–Cr–Cl and Cl–Cr–N are in the range between 93° and 98°. The double bond of the imino function C1—N1 with 1.295(3) Å is increased by the coordination of nitrogen to the chromium cation. A short discussion about the influence to the length of the double bond by different substituents at C1 or N1 in a similar ligand system is given in [2]. The dihedral angle of the main plane (C8, N1, C1, C2, C14) to the xylenyl- and phenyl ring is 78.4(1)° and 51.7(1)°, respectively.

Table 1. Data collection and handling.

Crystal:	blue plate, size 0.04 × 0.15 × 0.15 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	7.73 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ/ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12748, 3870
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2860
$N(param)_{refined}$:	256
Programs:	SHELXS-97 [3], SHELXL-97 [4], SHELXTL [5]

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

Atom	Site	x	y	z	U_{iso}
H(3)	4e	0.9007	0.0701	0.1161	0.041
H(4)	4e	1.0726	0.0603	0.0593	0.046
H(5)	4e	1.2558	0.0260	0.1665	0.051
H(6)	4e	1.2686	0.0024	0.3308	0.051
H(7)	4e	1.0996	0.0194	0.3900	0.046
H(10)	4e	0.8162	−0.2712	0.1471	0.081
H(11)	4e	0.7172	−0.2290	−0.0102	0.095
H(12)	4e	0.6426	−0.0786	−0.0435	0.074
H(14A)	4e	0.9799	0.1435	0.4070	0.054
H(14B)	4e	0.8759	0.0915	0.4417	0.054
H(16A)	4e	0.9503	0.3016	0.4589	0.126
H(16B)	4e	0.8617	0.2549	0.5151	0.126
H(16C)	4e	0.8198	0.3443	0.4460	0.126
H(17A)	4e	0.9160	0.2812	0.2799	0.100
H(17B)	4e	0.7861	0.3258	0.2643	0.100
H(17C)	4e	0.8025	0.2245	0.2189	0.100
H(19)	4e	0.5927	0.2174	0.2156	0.071
H(20)	4e	0.4101	0.1608	0.2653	0.089
H(21)	4e	0.4705	0.1180	0.4420	0.090
H(22)	4e	0.6917	0.1517	0.5052	0.076
H(91A)	4e	0.9479	−0.1391	0.3307	0.095
H(91B)	4e	0.8824	−0.2396	0.3154	0.095
H(91C)	4e	0.8240	−0.1526	0.3585	0.095
H(13A)	4e	0.7042	0.1198	0.0908	0.080
H(13B)	4e	0.5784	0.0814	0.1007	0.080
H(13C)	4e	0.6110	0.0769	−0.0017	0.080

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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}
Cr(1)	4e	0.62336(4)	0.04571(3)	0.32881(3)	0.0327(3)	0.0339(3)	0.0476(3)	−0.0011(2)	0.0215(2)	−0.0008(2)
Cl(1)	4e	0.48861(6)	−0.05268(5)	0.22863(5)	0.0301(4)	0.0538(5)	0.0609(4)	−0.0051(3)	0.0122(3)	−0.0025(4)
Cl(2)	4e	0.69055(7)	−0.06252(6)	0.45238(5)	0.0571(5)	0.0703(5)	0.0435(4)	−0.0009(4)	0.0211(3)	0.0130(4)
N(1)	4e	0.7717(2)	0.0204(1)	0.2663(1)	0.029(1)	0.029(1)	0.032(1)	0.0002(9)	0.0120(9)	0.0027(9)
C(1)	4e	0.8759(2)	0.0593(2)	0.3008(2)	0.031(1)	0.032(1)	0.032(1)	−0.002(1)	0.012(1)	0.002(1)
C(2)	4e	0.9818(2)	0.0448(2)	0.2591(2)	0.027(1)	0.029(1)	0.037(1)	−0.003(1)	0.011(1)	0.000(1)
C(3)	4e	0.9751(2)	0.0573(2)	0.1601(2)	0.027(1)	0.036(2)	0.039(1)	−0.001(1)	0.009(1)	0.002(1)
C(4)	4e	1.0774(2)	0.0509(2)	0.1262(2)	0.036(2)	0.044(2)	0.038(1)	−0.003(1)	0.015(1)	−0.001(1)
C(5)	4e	1.1865(2)	0.0307(2)	0.1900(2)	0.032(2)	0.044(2)	0.056(2)	−0.002(1)	0.021(1)	−0.006(1)
C(6)	4e	1.1943(2)	0.0174(2)	0.2877(2)	0.024(1)	0.049(2)	0.053(2)	−0.001(1)	0.004(1)	−0.001(1)
C(7)	4e	1.0932(2)	0.0261(2)	0.3226(2)	0.031(2)	0.046(2)	0.036(1)	−0.003(1)	0.005(1)	0.004(1)
C(8)	4e	0.7583(2)	−0.0504(2)	0.1884(2)	0.025(1)	0.036(2)	0.042(1)	−0.005(1)	0.017(1)	−0.005(1)
C(9)	4e	0.8047(2)	−0.1420(2)	0.2111(2)	0.037(2)	0.035(2)	0.068(2)	−0.000(1)	0.029(1)	−0.001(1)
C(10)	4e	0.7868(3)	−0.2085(2)	0.1342(3)	0.066(2)	0.038(2)	0.118(3)	−0.009(2)	0.058(2)	−0.023(2)
C(11)	4e	0.7273(4)	−0.1837(3)	0.0405(3)	0.074(3)	0.092(3)	0.085(3)	−0.033(2)	0.045(2)	−0.051(3)
C(12)	4e	0.6831(3)	−0.0944(3)	0.0211(2)	0.044(2)	0.093(3)	0.052(2)	−0.024(2)	0.021(1)	−0.027(2)
C(13)	4e	0.6955(2)	−0.0254(2)	0.0933(2)	0.026(1)	0.061(2)	0.041(2)	−0.013(1)	0.014(1)	−0.006(1)
C(14)	4e	0.8949(2)	0.1258(2)	0.3870(2)	0.039(2)	0.059(2)	0.042(2)	−0.014(1)	0.018(1)	−0.014(1)
C(15)	4e	0.8191(3)	0.2183(2)	0.3678(2)	0.055(2)	0.042(2)	0.066(2)	−0.016(2)	0.034(2)	−0.020(2)
C(16)	4e	0.8672(4)	0.2860(3)	0.4550(3)	0.087(3)	0.077(3)	0.103(3)	−0.036(2)	0.051(2)	−0.053(2)
C(17)	4e	0.8321(3)	0.2669(2)	0.2742(3)	0.082(3)	0.041(2)	0.094(2)	−0.015(2)	0.054(2)	−0.004(2)
C(18)	4e	0.6894(3)	0.1944(2)	0.3625(2)	0.059(2)	0.031(2)	0.072(2)	−0.006(1)	0.040(2)	−0.014(1)
C(19)	4e	0.5903(3)	0.1975(2)	0.2786(3)	0.057(2)	0.032(2)	0.094(3)	0.008(2)	0.030(2)	0.002(2)
C(20)	4e	0.4880(3)	0.1655(2)	0.3065(4)	0.049(2)	0.042(2)	0.139(4)	0.011(2)	0.039(2)	−0.008(2)
C(21)	4e	0.5215(3)	0.1420(3)	0.4051(3)	0.071(3)	0.052(2)	0.128(3)	−0.009(2)	0.073(2)	−0.025(2)
C(22)	4e	0.6461(3)	0.1606(2)	0.4405(2)	0.080(3)	0.055(2)	0.073(2)	−0.017(2)	0.053(2)	−0.026(2)
C(91)	4e	0.8706(3)	−0.1709(2)	0.3129(2)	0.068(2)	0.041(2)	0.094(3)	0.017(2)	0.041(2)	0.020(2)
C(131)	4e	0.6426(3)	0.0716(2)	0.0686(2)	0.035(2)	0.080(2)	0.043(2)	−0.002(2)	0.006(1)	0.010(2)

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