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Acta Cryst. (1994). C**50**, 1002–1003

Bis(η^5-methylcyclopentadienyl)chromium, $[Cr(C_6H_7)_2]$

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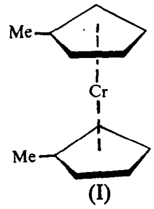
(*Received* 11 *May* 1993; *accepted* 19 *November* 1993)

Abstract

Dimethylchromocene contains planar C_5H_4Me rings in an eclipsed sandwich structure. The Cr atom is 1.788 (2) Å from each MeCp plane. The two substituted MeCp ligands are almost parallel [dihedral angle 5.9 (1)° and $MeCp_{centroid}$—Cr—$MeCp_{centroid}$ 178.1 (1)°].

Comment

The structural investigation of the title compound, (I), was undertaken as part of our work on chromium-deposition thin films, MOCVD (metal-organic chemical vapor deposition). The whole structure consists of well separated $(MeCp)_2Cr$ units where the two Cp ligands are bound to a metal ion in an eclipsed sandwich orientation. The two Cp ligands are almost parallel. The metal–centroid distances of 1.788 (2) Å are short with respect to the value of 1.832 (5) Å found in bis(tetraphenylcyclopentadienyl)chromium, which has a staggered conformation (Castellani, Geib, Rheingold & Trogler, 1987). The structure of Cp_2Cr^{II} has been reported by Weiss & Fischer (1956) to have an average Cr—Cp bond distance of 2.2 Å, comparable with the value of 2.158 (3) Å found in the title compound.



(I)

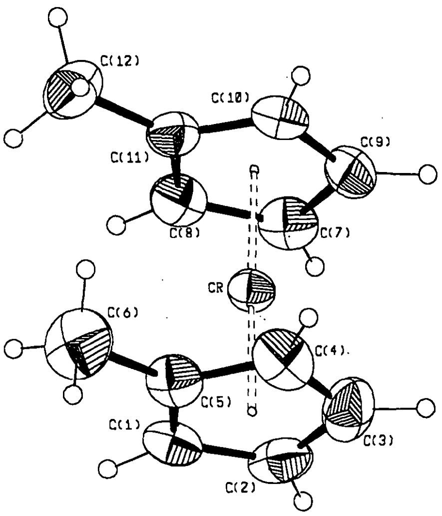


Fig. 1. A perspective view of the molecule, with the atomic numbering scheme. Ellipsoids are drawn at the 50% probability level.

Experimental

Bis(η^5-methylcyclopentadienyl)chromium was obtained following the previously reported procedure (Kohler, 1976) by reacting chromium(II) chloride with potassium methylcyclopentadienide in tetrahydrofuran. Crystals of $[Cr(C_5H_4CH_3)_2]$ suitable for X-ray determination were obtained by slow sublimation at 310 K and 8×10^{-3} mmHg (1.067 Pa).

Crystal data

$[Cr(C_6H_7)_2]$
$M_r = 210.24$
Monoclinic
$P2_1/c$
$a = 12.281\ (2)$ Å
$b = 7.728\ (2)$ Å
$c = 11.159\ (2)$ Å
$\beta = 102.71\ (3)°$
$V = 1033.1\ (4)$ Å^3
$Z = 4$
$D_x = 1.35$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 13–18°$
$\mu = 1.03$ mm^{-1}
$T = 293$ K
Prism
0.62 × 0.52 × 0.16 mm
Transparent red

Data collection

Philips PW1100 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (North, Phillips & Mathews, 1968)
$T_{min} = 0.78$, $T_{max} = 1.00$
2891 measured reflections
2496 independent reflections
2010 observed reflections $[I \geq 3\sigma(I)]$

$R_{int} = 0.013$
$\theta_{max} = 28°$
$h = -15 \rightarrow 15$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 14$
2 standard reflections
frequency: 180 min
intensity variation: none

Acta Crystallographica Section C
ISSN 0108-2701 ©1994

Refinement

Refinement on F
$R = 0.037$
$wR = 0.044$
$S = 1.009$
2010 reflections
125 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F_o) + 0.002777(F_o^2)]$
$(\Delta/\sigma)_{\text{max}} = 0.01$
$\Delta\rho_{\text{max}} = 0.32$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.52$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Cr	0.29760 (3)	−0.01745 (4)	0.09236 (3)	0.0330 (1)
C(1)	0.2443 (2)	−0.2647 (3)	0.1510 (2)	0.0465 (7)
C(2)	0.3554 (2)	−0.2746 (3)	0.1325 (3)	0.0553 (9)
C(3)	0.4219 (2)	−0.1592 (4)	0.2163 (3)	0.0620 (9)
C(4)	0.3518 (2)	−0.0781 (4)	0.2859 (2)	0.0551 (8)
C(5)	0.2422 (2)	−0.1434 (3)	0.2461 (2)	0.0447 (7)
C(6)	0.1433 (3)	−0.0932 (5)	0.2960 (3)	0.072 (1)
C(7)	0.3150 (2)	0.0589 (3)	−0.0864 (2)	0.0475 (8)
C(8)	0.2001 (2)	0.0749 (3)	−0.0819 (2)	0.0471 (7)
C(9)	0.3763 (2)	0.1732 (3)	0.0034 (2)	0.0470 (7)
C(10)	0.2994 (2)	0.2584 (3)	0.0616 (2)	0.0458 (7)
C(11)	0.1904 (2)	0.1979 (3)	0.0085 (2)	0.0452 (7)
C(12)	0.0850 (2)	0.2563 (4)	0.0444 (3)	0.070 (1)

Table 2. *Selected geometric parameters* (Å, °)

Cp(1) and *Cp*(2) are the centroids of the cyclopentadienyl rings.

Cr—C(1)	2.167 (2)	Cr—C(2)	2.124 (3)
Cr—C(3)	2.125 (3)	Cr—C(4)	2.167 (2)
Cr—C(5)	2.206 (3)	Cr—C(7)	2.135 (3)
Cr—C(8)	2.168 (2)	Cr—C(9)	2.125 (3)
Cr—C(10)	2.160 (2)	Cr—C(11)	2.199 (2)
Cr—*Cp*(1)	1.788 (2)	Cr—*Cp*(2)	1.788 (2)
C(1)—C(2)	1.426 (4)	C(1)—C(5)	1.420 (3)
C(2)—C(3)	1.414 (4)	C(3)—C(4)	1.426 (4)
C(4)—C(5)	1.414 (4)	C(5)—C(6)	1.494 (5)
C(7)—C(8)	1.428 (4)	C(7)—C(9)	1.421 (3)
C(8)—C(11)	1.410 (3)	C(9)—C(10)	1.421 (4)
C(10)—C(11)	1.419 (3)	C(11)—C(12)	1.507 (4)
C(2)—C(1)—C(5)	108.6 (2)	C(1)—C(2)—C(3)	107.6 (2)
C(2)—C(3)—C(4)	107.9 (3)	C(3)—C(4)—C(5)	108.6 (2)
C(1)—C(5)—C(4)	107.3 (3)	C(8)—C(7)—C(9)	107.2 (2)
C(7)—C(8)—C(11)	108.8 (2)	C(7)—C(9)—C(10)	107.9 (2)
C(9)—C(10)—C(11)	108.5 (2)	C(8)—C(11)—C(10)	107.5 (2)
C(4)—C(5)—C(6)	125.8 (2)	C(1)—C(5)—C(6)	126.8 (2)
C(10)—C(11)—C(12)	125.3 (2)	C(8)—C(11)—C(12)	127.2 (2)
Cp(1)—Cr—*Cp*(2)	178.1 (1)		

An air-sensitive crystal was lodged in a Lindemann glass capillary and centred on the Philips PW1100 diffractometer. The orientation matrix and preliminary unit-cell dimensions were determined from reflections found by mounting the crystal at random, and varying each of the orientation angles χ and φ over a range of 120°, with $7 < \theta < 10°$. There were no significant fluctuations of intensities other than those expected from Poisson statistics. Intensity data were corrected for Lp effects. The structure was solved by Patterson and Fourier techniques and refined by full-matrix least squares; anisotropic displacement parameters were assigned to all non-H atoms. H atoms were introduced at calculated positions and allowed to ride on the parent C atoms during the least-squares refinement (C—H = 0.98 Å and $U = 0.07$ Å^2). Data processing and computations were carried out using *SHELX76* (Sheldrick, 1976). Molecular graphics were obtained using *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71837 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1054]

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Acta Cryst. (1994). **C50**, 1003–1005

(4,7-Dimethyl-4,7-diazadecanediamide)-diisothiocyanatocopper(II)

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(*Received* 14 *May* 1993; *accepted* 23 *November* 1993)

Abstract

The Cu$^{\text{II}}$ ion of the title complex, [Cu(NCS)$_2$(C$_{10}$H$_{22}$O$_2$N$_4$)], is six-coordinated in a tetragonally elongated octahedral geometry with the two amine N atoms of the quadridentate ligand and the two N atoms of the isothiocyanato groups equatorial and the two amide O atoms axial. The two *N*-methyl groups of the tetradentate ligand are *trans* to each other with respect to the equatorial plane. The complex has a *cis*-α conformation. The central five-membered ring is in a stable skew form and both of the terminal

Acta Crystallographica Section C
ISSN 0108-2701 ©1994