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Tricarbonyl[η^6-(*R*)-*N*,*N*-dimethyl-1-phenylethylamine]chromium(0)

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The title complex, $[Cr(C_{10}H_{15}N)(CO)_3]$, has a typical three-legged piano-stool structure, as is expected for an η^6-arene–tricarbonylchromium compound. The conformation is staggered with respect to the side chain and one of the carbonyl ligands. The side chain bearing the stereogenic centre adopts a conformation with the H atom pointing downwards, leaving the dimethylamine group above the ring plane.

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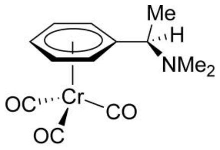
Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.002 Å
R factor = 0.022
wR factor = 0.061
Data-to-parameter ratio = 17.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Tricarbonyl[η^6-(*R*)-*N*,*N*-dimethyl-1-phenylethylamine]chromium(0), (I), a common starting material for various *ortho*-functionalizations to afford planar chiral [*i.e.* a kind of chirality in which a plane is the decisive element (*cf* center, axis and helix in the other three kinds of chirality)] compounds (Berger *et al.*, 2003; Uemura *et al.*, 1993; Christian *et al.*, 1994; Fukuzawa *et al.*, 1997; Weber & Jones, 2001; Braun *et al.*, 2003), has been structurally characterized.



(I)

The conformation of the tricarbonylchromium moiety is staggered with respect to the side chain and one of the carbonyl ligands. The α-chain itself adopts a conformation in which the smallest atom (H) on the stereogenic C atom points downwards to the sterically demanding tricarbonylchromium group. This causes the dimethylamine group to be placed

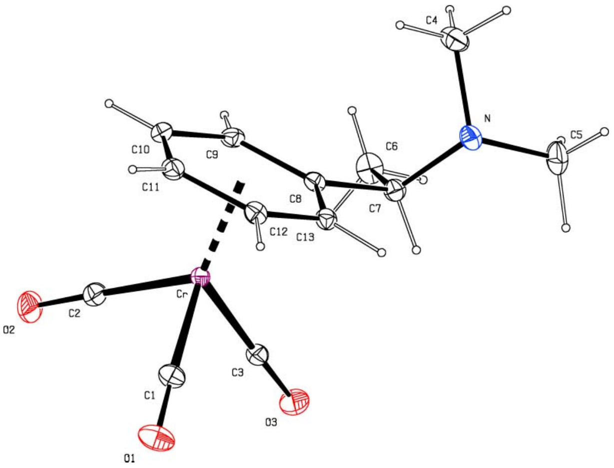


Figure 1
The crystal structure of the molecule. Displacement ellipsoids are drawn at the 30% probability level and H atoms are drawn with arbitrary radii.

above the ring plane of the arene in close proximity to the *ortho*-H atom. The torsion angle N—C7—C8—C13 is −63.57 (18)°. This confirms that the results obtained by Heppert *et al.* (1990) for solutions by NOE (nuclear Overhauser effect) measurements apply also in the solid state. This preferred conformation enables the diastereospecific deprotonation of the pro-(*R*) H atom in the reaction with ^{t}BuLi due to the pre-coordination of the Li atom by the N atom.

Experimental

The title chromium tricarbonyl complex was synthesized by double methylation of (*R*)-1-phenylethylamine with formaldehyde and formic acid in an Eschweiler–Clarke reaction and subsequent thermal complexation of dimethylamine with hexacarbonylchromium in a mixture of n-Bu$_2$O and tetrahydrofuran. After work-up, single crystals were obtained by slow evaporation of a solution in diethyl ether under nitrogen at room temperature.

Crystal data

$[Cr(C_{10}H_{15}N)(CO)_3]$
$M_r = 285.26$
Orthorhombic, $P2_12_12_1$
$a = 10.6453\ (18)$ Å
$b = 10.7928\ (19)$ Å
$c = 11.458\ (2)$ Å
$V = 1316.5\ (4)$ Å^3
$Z = 4$
$D_x = 1.439$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8096 reflections
$\theta = 2.6$–27.0°
$\mu = 0.87$ mm^{-1}
$T = 110\ (2)$ K
Irregular fragment, yellow
0.63 × 0.37 × 0.13 mm

Data collection

Bruker SMART APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.611$, $T_{\max} = 0.892$
16 290 measured reflections
2869 independent reflections
2807 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\max} = 27.0°$
$h = -13 \rightarrow 13$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.061$
$S = 1.07$
2869 reflections
165 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.075P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = −0.003 (14)

Table 1
Selected geometric parameters (Å, °).

Cr—C3	1.8246 (17)	N—C4	1.462 (2)
Cr—C2	1.8479 (17)	N—C7	1.472 (2)
Cr—C1	1.8537 (18)	C7—C8	1.529 (2)
N—C5	1.460 (2)	C7—C6	1.529 (2)
C3—Cr—C2	88.00 (7)	N—C7—C8	107.03 (12)
C3—Cr—C1	88.82 (7)	N—C7—C6	116.25 (14)
C2—Cr—C1	90.44 (8)	C8—C7—C6	113.84 (13)
C5—N—C4	110.56 (14)	C9—C8—C13	118.76 (14)
C5—N—C7	112.06 (13)	C9—C8—C7	123.79 (14)
C4—N—C7	113.57 (13)		

H atoms were placed in calculated positions and refined using a riding model, with C—H distances ranging from 0.95 to 1.00 Å and $U_{\rm iso}$(H) = 1.2 or 1.5 times $U_{\rm eq}$(C). The methyl groups bonded to N atoms were allowed to rotate as rigid groups.

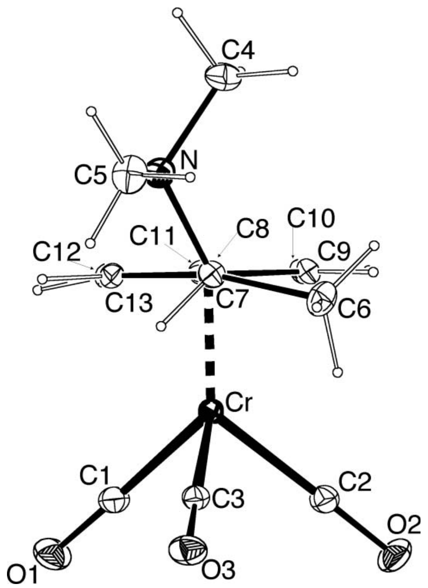


Figure 2
View of the molecule along the α-C7–*ipso*-C8-axis, displaying the conformation of the side chain. Displacement ellipsoids are drawn at the 30% probability level and H atoms are drawn with arbitrary radii.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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