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Tricarbonyl(η^6-*N*,*N*-dimethylaniline)chromium(0)

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Key indicators

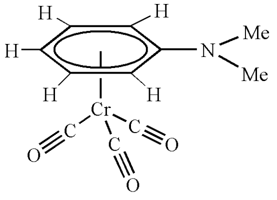
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.042
wR factor = 0.100
Data-to-parameter ratio = 14.4

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

The title compound, [Cr{($C_6H_5N(CH_3)_2$}(CO)$_3$], exhibits the three-legged piano-stool structure expected for a tricarbonylmetal with an η^6-coordinated arene. The arene moiety is distorted with the amino functional group bent away from the chromium atom.

Comment

In the course of our work on the preparation and property exploration of substituted arenes coordinated to chromium carbonyls, we have isolated and structurally characterized the title compound, (I).



(I)

The complex crystallizes in the space group $P\overline{1}$ with two crystallographically independent molecules in the asymmetric unit. The compound exhibits the three-legged piano-stool structure expected for a tricarbonylmetal complex. Both molecules have very similar geometries with a weighted r.m.s. deviation of 0.2161 Å. Their main difference lies within the three carbonyl ligands, showing slightly different rotational angles relative to the arene group for the two molecules. When omitting the carbonyl groups, the weighted r.m.s. deviation decreases to 0.0272 Å.

The amine substituents are only slightly pyramidalized, with C—N—C angles between 115.5 (2)° for C30—N2—C31 and 119.9 (2)° for C4—N1—C11. The lone electron pairs are pointing away from the tricarbonylchromium units.

Both the amine N atoms as well as the directly adjacent arene C atoms are significantly bent out of the plane of the other five aromatic C atoms. The values are 0.108 (3) and 0.099 (3) Å for C4 and C24, and 0.234 (4) and 0.219 (4) Å for N1 and N2, respectively. This kind of distortion is consistent with an earlier structural study of several (η^6-arene)tricarbonylchromium complexes (Hunter *et al.*, 1992). That investigation revealed that π-donor substituents on the arene bend away from the tricarbonylchromium fragment, while π-accepting groups remain in the plane of the arene or are bent slightly towards the chromium fragment.

Experimental

The title compound, (I), was prepared from hexacarbonylchromium(0) and dimethylaniline in a mixture of dibutyl ether and

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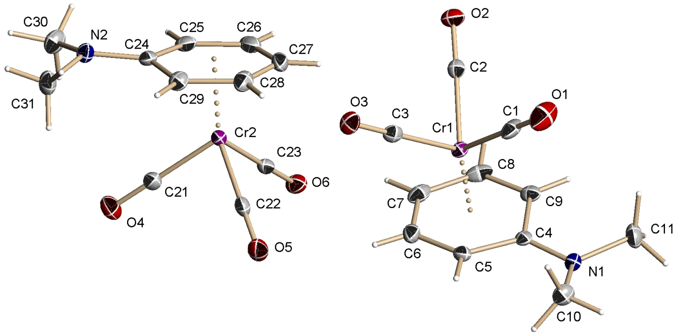

Figure 1
The structure of the asymmetric unit of (I), showing 50% probability displacement ellipsoids.

tetrahydrofuran, as described previously (Hunter *et al.*, 1992). Yellow needle-shaped single crystals of (I) were grown by the slow diffusion of a layer of hexane into a dichloromethane solution.

Crystal data

$[Cr(C_8H_{11}N)(CO)_3]$
$M_r = 257.21$
Triclinic, $P\bar{1}$
$a = 7.0974$ (5) Å
$b = 12.6169$ (8) Å
$c = 14.0477$ (9) Å
$\alpha = 115.397$ (1)°
$\beta = 90.959$ (1)°
$\gamma = 102.666$ (1)°
$V = 1099.92$ (13) Å^3

$Z = 4$
$D_x = 1.553$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9302 reflections
$\theta = 2.9$–30.5°
$\mu = 1.03$ mm^{-1}
$T = 100$ (2) K
Needle, yellow
0.60 × 0.15 × 0.05 mm

Data collection

Bruker AXS SMART APEX CCD diffractometer
φ and ω scans
Absorption correction: multi scan (*SADABS* in *SAINT-Plus*; Bruker, 1997–1999)
$T_{min} = 0.743$, $T_{max} = 0.95$
11969 measured reflections

5444 independent reflections
5013 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 28.3°$
$h = -9 \rightarrow 9$
$k = -16 \rightarrow 16$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.100$
$S = 1.18$
5444 reflections
377 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0417P)^2 + 0.8635P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.68$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

All H atoms were located in difference density maps and refined isotropically. The refined C—H bond lengths are between 0.84 (3) and 1.00 (3) Å. The s.u. values of the cell parameters are taken from the software, recognizing that the values are unreasonably small (Herbstein, 2000).

Data collection: *SMART* (Bruker, 1997–2002); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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