

Tricarbonyl(η^6 -4-fluoroaniline)chromiumMatthias Zeller,^a Allen D.
Hunter,^{a*} Jody L. Regula^a and
Paul S. Szalay^b^aDepartment of Chemistry, Youngstown State University, One University Plaza, Youngstown, OH 44555, USA, and ^bDepartment of Chemistry, Muskingum College, 163 Stormont St., New Concord, OH 43762, USA

Correspondence e-mail: adhunter@ysu.edu

Key indicators

Single-crystal X-ray study

T = 298 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.026

wR factor = 0.076

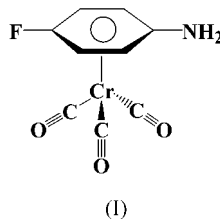
Data-to-parameter ratio = 13.0

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

The title compound, $[\text{Cr}(\text{C}_6\text{H}_6\text{FN})(\text{CO})_3]$, exhibits the three-legged piano-stool structure expected for a metal tricarbonyl with an η^6 -coordinated arene. A crystallographic mirror plane bisects the molecule. The amine and fluoroarene functional groups display an out-of-plane distortion.

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).



The title molecule has a mirror symmetry (Fig. 1 and Table 1). The amine and fluoro functional groups are distorted out of the least-squares plane defined by atoms C2/C2ⁱ/C3/C3ⁱ of the arene [symmetry code: (i) $x, \frac{1}{2} - y, z$]. The torsion angles between the plane of C2/C2ⁱ/C3/C3ⁱ and those of C3/C3ⁱ/C4/N1 and C2/C2ⁱ/C1/F1 are 6.3 (3) and 2.8 (3)°, respectively. This distortion is consistent with an earlier structural study of several tricarbonyl(η^6 -arene)chromium complexes (Hunter *et al.*, 1992). This study revealed that π -donor substituents on the arene bend away from the tricarbonylchromium fragments, while π -accepting groups remain in the plane of the arene or are bent slightly towards the chromium fragment. Previous structural reports of amine substituted arenes coordinated to

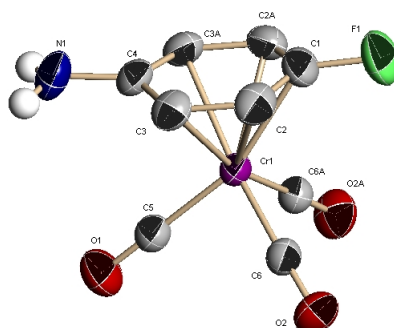


Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids. Arene H atoms have been omitted for clarity. Atoms labeled with the suffix A are at the symmetry position $(x, \frac{1}{2} - y, z)$.

metal carbonyls also revealed extensive hydrogen-bonding networks in the extended structures of these compounds (Camiolo *et al.*, 2000). In these examples, amine groups form hydrogen bonds with the nearest neighbour carbonyl O atoms. In (I), a bifurcated N—H···O hydrogen bond has been observed (Table 2).

Experimental

The title compound, (I), was prepared from hexacarbonylchromium(0) and *p*-fluoroaniline in a mixture of dibutyl ether and tetrahydrofuran, as described previously (Hunter *et al.*, 1992). Yellow needle-shaped single crystals of (I) were grown by slow diffusion of a layer of hexane into a methylene chloride solution.

Crystal data

[Cr(C ₆ H ₆ FN)(CO) ₃]	Mo K α radiation
$M_r = 247.15$	Cell parameters from 5835 reflections
Orthorhombic, <i>Pnma</i>	$\theta = 2.2\text{--}28.1^\circ$
$a = 12.6648(8) \text{ \AA}$	$\mu = 1.20 \text{ mm}^{-1}$
$b = 9.1249(5) \text{ \AA}$	$T = 298(2) \text{ K}$
$c = 8.2136(5) \text{ \AA}$	Needle, yellow
$V = 949.20(10) \text{ \AA}^3$	$0.66 \times 0.27 \times 0.12 \text{ mm}$
$Z = 4$	
$D_x = 1.729 \text{ Mg m}^{-3}$	

Data collection

Bruker SMART APEX CCD diffractometer	1185 independent reflections
ω scans	1122 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (<i>SADABS</i> in <i>SAINT-Plus</i> ; Bruker, 1997–1999)	$R_{\text{int}} = 0.019$
$T_{\text{min}} = 0.728$, $T_{\text{max}} = 0.870$	$\theta_{\text{max}} = 28.2^\circ$
7436 measured reflections	$h = -16 \rightarrow 16$
	$k = -11 \rightarrow 12$
	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.046P)^2 + 0.2515P]$
$R[F^2 > 2\sigma(F^2)] = 0.026$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.077$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.08$	$\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
1185 reflections	$\Delta\rho_{\text{min}} = -0.32 \text{ e \AA}^{-3}$
91 parameters	
All H-atom parameters refined	

Table 1

Selected geometric parameters (Å).

Cr1—C6	1.8250 (15)	O2—C6	1.1577 (19)
Cr1—C5	1.854 (2)	O1—C5	1.146 (3)
Cr1—C2	2.2040 (17)	C4—N1	1.363 (3)
Cr1—C1	2.214 (2)	C4—C3	1.410 (2)
Cr1—C3	2.2462 (15)	C2—C1	1.383 (2)
Cr1—C4	2.350 (2)	C2—C3	1.414 (2)
F1—C1	1.358 (3)		

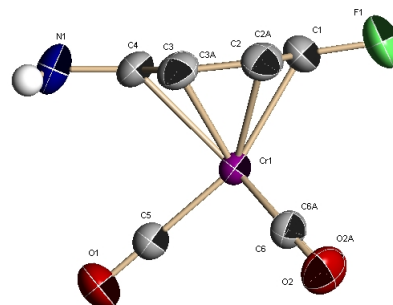


Figure 2

Side-on view of (I), illustrating the out-of-plane displacement of the amine and fluoro groups. Arene H atoms have been omitted for clarity. Atoms labeled with the suffix *A* are at the symmetry position $(x, \frac{1}{2} - y, z)$.

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1···O2 ⁱⁱ	0.86 (2)	2.38 (2)	3.132 (2)	147 (2)
N1—H1···O2 ⁱⁱⁱ	0.86 (2)	2.66 (2)	3.245 (2)	127 (2)

Symmetry codes: (ii) $-x, -y, 1 - z$; (iii) $x, y, 1 + z$.

All H atoms were located from difference density maps and refined isotropically. The C—H and N—H bond lengths are 0.87 (2)–0.90 (2) and 0.86 (2) Å, respectively. The s.u. values of the cell parameters are derived by the software, giving values that are unreasonably small (Herbstein, 2000).

Data collection: *SMART* (Bruker, 1997–2000); cell refinement: *SAINT-Plus* (Bruker, 1997–1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXL97*.

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