

Matthias Zeller and
Allen D. Hunter*Department of Chemistry, Youngstown State
University, One University Plaza, Youngstown,
OH 44555, USA

Correspondence e-mail: adhunter@ysu.edu

Key indicators

Single-crystal X-ray study
T = 100 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.025
wR factor = 0.069
Data-to-parameter ratio = 16.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Tricarbonyl(η^6 -1,3,5-trimethoxybenzene)-
chromium(0)

The title compound, $(1,3,5\text{-}(\text{MeO})_3\text{C}_6\text{H}_3)\text{Cr}(\text{CO})_3$ or $[\text{Cr}(\text{C}_9\text{H}_{12}\text{O}_3)(\text{CO})_3]$, exhibits the three-legged piano-stool structure expected for a metal tricarbonyl with an η^6 -coordinated arene. The molecule exhibits threefold symmetry. The methoxy groups display an out-of-plane distortion.

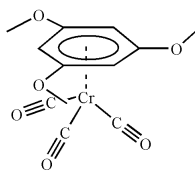
Received 14 October 2003

Accepted 22 October 2003

Online 31 October 2003

Comment

In the course of our work on the preparation and property exploration of substituted arenes coordinated to chromium carbonyls, we isolated and structurally characterized the title compound, (I) (Fig. 1 and Table 1).



The title molecule exhibits a threefold axis, passing through the center of the benzene ring and the Cr atom. The complex crystallizes in the non-centrosymmetric space group $R3c$. The crystal under investigation proved to be a partial racemic twin and the absolute structure parameter has been refined to 0.12 (3). The absolute configuration of this chiral compound is defined by the orientation of the arene methoxy groups, which can easily rotate in solution, and no attempts have been made to assign the major absolute configuration present here.

Both the methoxy functional groups as well as the aromatic H atoms are distorted out of the least-squares plane defined by atoms $\text{C}1/\text{C}2/\text{C}1^1/\text{C}2^1/\text{C}1^j/\text{C}2^j$ of the arene [symmetry codes: (i) $-y, x - y, z$; (j) $-x + y, -x, z$]. The methoxy groups are bent by $0.0587(19) \text{ \AA}$ away from the Cr atom, while the H atoms, as far as their positions can be measured accurately, are bent towards the metal center by $0.12(3) \text{ \AA}$. This distortion is consistent with an earlier structure-property relationship study of (η^6 -arene)tricarbonylchromium complexes (Hunter *et al.*, 1992). That study 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, the degree of bending being correlated with the π -donor/acceptor strength of the substituents.

Experimental

The title compound, (I), was prepared from hexacarbonylchromium(0) and 1,3,5-trimethoxybenzene in a mixture of dibutyl ether and tetrahydrofuran, as previously described (Hunter *et al.*,

1992). Yellow-to-colourless single crystals of (I) were grown by slow diffusion of a layer of hexane into a methylene chloride solution.

Crystal data

[Cr(C₉H₁₂O₃)(CO)₃]
M_r = 304.22
 Trigonal, *R*3*c*
a = 12.6892 (9) Å
c = 13.3798 (19) Å
V = 1865.7 (3) Å³
Z = 6
D_x = 1.625 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 5768 reflections
 θ = 2.4–28.4°
 μ = 0.94 mm⁻¹
T = 100 (2) K
 Block, colourless
 0.56 × 0.20 × 0.16 mm

Data collection

Bruker AXS SMART APEX CCD diffractometer
 ω scans
 Absorption correction: multi-scan (*SADABS* in *SAINTE-Plus*; Bruker, 1997–1999)
T_{min} = 0.568, *T_{max}* = 0.86
 5069 measured reflections

1040 independent reflections
 1038 reflections with *I* > 2σ(*I*)
R_{int} = 0.032
 θ_{max} = 28.4°
h = -16 → 16
k = -16 → 16
l = -17 → 17

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.026
wR (*F*²) = 0.069
S = 1.07
 1040 reflections
 64 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.4118P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.54 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.68 \text{ e \AA}^{-3}$
 Absolute structure: Flack (1983),
 510 Friedel pairs
 Flack parameter = 0.12 (3)

Table 1

Selected geometric parameters (Å, °).

Cr1–C4	1.8459 (17)	C1–H1	0.97 (3)
Cr1–C1	2.2491 (18)	C2–O1	1.343 (2)
Cr1–C2	2.2705 (18)	C3–O1	1.4402 (15)
C1–C2	1.4118 (18)	C4–O2	1.1515 (19)
C4–Cr1–C1	163.27 (7)	C1–C2–C1 ⁱⁱ	120.83 (17)
C2–C1–C2 ⁱ	119.03 (17)	O2–C4–Cr1	178.85 (15)
O1–C2–C1	124.38 (13)	C2–O1–C3	117.19 (11)

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

The single independent aromatic H atom was located from a difference density map and refined isotropically. The C–H length is 0.97 (3) Å. The methoxy H atoms were placed at calculated positions

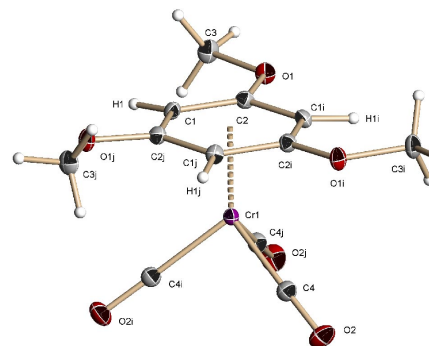


Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids. [Symmetry codes: (i) $-y, x - y, z$; (j) $-x + y, -x, z$].

and refined with *U_{iso}* values defined as 1.5 times *U_{eq}*(parent atom). The s.u. values of the cell parameters are taken from the software and the values are unreasonably small (Herbstein, 2000).

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

MZ was supported by NSF grant No. 0111511. The diffractometer was funded by NSF grant No. 0087210, by the Ohio Board of Regents grant No. CAP-491 and by YSU.

References

- Bruker (1997–2000). *SMART* for WNT/2000. Version 5.625. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (1997–1999). *SAINTE-Plus*. Version 6.02. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
 Herbstein, F. H. (2000). *Acta Cryst.* **B56**, 547–557.
 Hunter, A. D., Shilliday, L., Furey, W. S. & Zaworotko, M. (1992). *J. Organomet.* **11**, 1550–1560.
 Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.