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Structure of (Trifluoromethylbenzene)(*p*-xylene)chromium(0) at 163 K

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Abstract. $[Cr(C_7H_5F_3)(C_8H_{10})]$, $M_r = 304{\cdot}27$, orthorhombic, $Pca2_1$, $a = 10{\cdot}9592\,(20)$, $b = 7{\cdot}7732\,(11)$, $c = 15{\cdot}1243\,(33)$ Å, $V = 1288{\cdot}4\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}569$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 8{\cdot}831$ cm^{-1}, $F(000) = 624$, $R = 0{\cdot}0345$ for 1755 reflections ($F \geq 4\sigma_F$). The average Cr—C(trifluoromethylbenzene) distance is 2·133 (15) Å [range: 2·113 (5)–2·146 (4) Å]; the average Cr—C(xylene) distance is 2·164 (16) Å [range: 2·149 (3)–2·188 (4) Å]. For the trifluoromethylbenzene, the shortest distances are adjacent to the CF_3 group; in contrast, the longest Cr—C distances in xylene are to the methylated ring C atoms. The dihedral angle between least-squares planes of the rings is 2·9 (1)°. The rings are staggered, rotated approximately 22° with respect to each other. Average ring C—C distances are 1·408 (17) Å for trifluoromethylbenzene [range: 1·389 (7)–1·438 (6) Å] and 1·412 (12) Å for xylene [range: 1·398 (5)–1·430 (5) Å].

Experimental. The title compound, (1), was prepared by the reaction of resistively vaporized Cr and a 1:1 mole ratio mixture of trifluoromethylbenzene and *p*-xylene at 77 K and $2{\cdot}6 \times 10^{-4}$ Pa (Seymour, Ssekaalo & Lagowski, 1987). Brownish crystals were obtained by

Table 1. *Summary of data collection and structure refinement for* (1)

(*a*) Data collection (163 K)$^{\text{i,ii}}$	
Mode	ω scan
Scan range	Symmetrically over 1·0° about $K\alpha_{1,2}$ maximum
Background	Offset 1·0 and −1·0° in ω from $K\alpha_{1,2}$ maximum
Scan rate (° min^{-1})	3·0–6·0
Exposure time (h)	51·5
Stability analysis	
Check reflections	004; $0\bar{4}0$; 400; $\bar{2}\bar{1}\bar{1}$
Computed *s*	−0·00054 (12)
t	0·000007 (2)
Correction range (on *I*)	1·000–1·011
2θ range (°)	4·0–60·0
Range in *hkl*, min.	0, −10, 0
max.	15, 10, 20
Reflections, measured, unique	3762, 1955
R_{int}	0·0316
Crystal dimensions (mm)	0·82 × 0·19 × 0·15
Crystal volume (mm^3)	0·0170
Transmission-factor range	0·807–0·862
(*b*) Structure refinement	
Instability factor *p*	0·04
Reflections used ($F \geq 4\sigma_F$)	1755
No. of variables	231
Goodness of fit, *S*	1·395
R, *wR*	0·0345, 0·0345
R for all data	0·0408
Max. shift/e.s.d.	0·018
Max., min. density in difference map (e Å^{-3})	0·55, −0·48

Notes: (i) Unit-cell parameters were obtained by least-squares refinement of the setting angles of 45 reflections with $26{\cdot}4 < 2\theta < 28{\cdot}5°$. (ii) Syntex $P2_1$ autodiffractometer with a graphite monochromator and a Syntex LT-1 inert-gas (N_2) low-temperature delivery system. Data reduction was carried out as described by Riley & Davis (1976). Crystal and instrument stability were monitored by remeasurement of four check reflections after every 96 reflections. As detailed by Henslee & Davis (1975), these data were analyzed to relate intensity to exposure time by the equation $y = 1{\cdot}0 + sx + tx^2$ where x is exposure time (h), y is fractional intensity relative to $x = 0$ and s and t are coefficients determined by least-squares fit.

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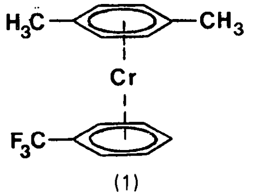
thermal-gradient sublimation as hexagonal needles. The crystal was affixed with grease in a capillary tube. A summary of data collection and structural refinement is given in Table 1.



(1)

Initial Cr and ring C-atom positions were deduced from a Patterson map; remaining C and F atoms were obtained from an electron density map. All atomic positional parameters and thermal parameters (anisotropic non-H atoms) were refined by full-matrix least squares (*SHELX*76, Sheldrick, 1976); function minimized was $\sum w(F_o - F_c)^2$, where $w = \sigma_F^{-2}$. An electron density difference map calculated at $R = 0{\cdot}05$ revealed H atoms as peaks of 0·35–0·67 e Å^{-3}. Scattering factors and anomalous-dispersion corrections for all non-H atoms were taken from *International Tables for X-ray Crystallography* (1974); H scattering factors from Stewart, Davidson & Simpson (1965). Atomic parameters are listed in Table 2; bond lengths and angles in Table 3.[*] Atom labeling is shown in Fig. 1 and

Table 2. *Positional and equivalent isotropic thermal parameters for atoms in* (1)

	x	y	z	U/U_{eq}(Å^2)
Cr	0·51876 (3)	0·73096 (4)	0·5	0·02133 (10)
C(1)	0·3904 (3)	0·8702 (4)	0·5761 (3)	0·0314 (10)
C(2)	0·3259 (2)	0·7725 (3)	0·5124 (3)	0·0288 (10)
C(3)	0·3585 (3)	0·7840 (5)	0·4231 (3)	0·0321 (10)
C(4)	0·4537 (4)	0·8933 (5)	0·3959 (3)	0·0400 (12)
C(5)	0·5160 (4)	0·9931 (7)	0·4573 (4)	0·046 (2)
C(6)	0·4867 (4)	0·9827 (6)	0·5473 (4)	0·0417 (14)
C(7)	0·3549 (4)	0·8643 (5)	0·6702 (3)	0·0500 (14)
C(8)	0·5623 (3)	0·5155 (5)	0·5877 (3)	0·0255 (9)
C(9)	0·5306 (2)	0·4540 (3)	0·5036 (4)	0·0246 (7)
C(10)	0·5903 (3)	0·5156 (5)	0·4267 (3)	0·0255 (9)
C(11)	0·6839 (3)	0·6429 (4)	0·4338 (2)	0·0248 (9)
C(12)	0·7126 (2)	0·7063 (3)	0·5178 (2)	0·0246 (9)
C(13)	0·6533 (3)	0·6444 (5)	0·5938 (2)	0·0259 (9)
C(14)	0·5022 (4)	0·4425 (5)	0·6689 (3)	0·0397 (12)
C(15)	0·7482 (4)	0·7075 (5)	0·3525 (3)	0·0382 (11)
F(1)	0·2925 (2)	0·7214 (4)	0·6924 (2)	0·0598 (9)
F(2)	0·4493 (3)	0·8729 (4)	0·7261 (2)	0·0728 (11)
F(3)	0·2812 (3)	0·9985 (4)	0·6916 (2)	0·0905 (13)
H(2)	0·262 (3)	0·699 (4)	0·531 (2)	0·028 (9)
H(3)	0·327 (5)	0·728 (6)	0·387 (4)	0·06 (2)
H(4)	0·474 (4)	0·896 (6)	0·340 (4)	0·043 (12)
H(5)	0·569 (4)	1·056 (6)	0·437 (3)	0·053 (13)
H(6)	0·517 (5)	1·025 (7)	0·593 (4)	0·06 (2)
H(9)	0·468 (3)	0·373 (4)	0·498 (3)	0·030 (8)
H(10)	0·560 (3)	0·475 (4)	0·373 (2)	0·006 (7)
H(12)	0·769 (3)	0·799 (4)	0·519 (2)	0·031 (9)
H(13)	0·674 (3)	0·689 (4)	0·643 (3)	0·018 (8)
H(14*A*)	0·544 (4)	0·347 (6)	0·685 (3)	0·049 (13)
H(14*B*)	0·507 (4)	0·521 (6)	0·714 (3)	0·036 (11)
H(14*C*)	0·421 (4)	0·401 (6)	0·659 (3)	0·053 (13)
H(15*A*)	0·699 (4)	0·697 (6)	0·305 (3)	0·043 (12)
H(15*B*)	0·773 (5)	0·837 (6)	0·364 (3)	0·061 (14)
H(15*C*)	0·824 (5)	0·645 (6)	0·339 (4)	0·061 (15)

For non-H atoms, the U value is U_{eq}, calculated as $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{A}_{ij}$, where $\mathbf{A}_{ij}$ is the dot product of the ith and jth direct-space unit-cell vectors.

Table 3. *Bond lengths* (Å) *and bond angles* (°) *in* (1)

1	2	3	1–2	1–2–3
C(2)	C(1)	C(6)	1·416 (5)	119·1 (4)
C(6)	C(1)	C(7)	1·438 (6)	120·3 (4)
C(7)	C(1)	C(2)	1·476 (6)	120·5 (3)
C(3)	C(2)	C(1)	1·400 (6)	119·7 (3)
C(4)	C(3)	C(2)	1·407 (5)	120·7 (4)
C(5)	C(4)	C(3)	1·389 (7)	120·4 (5)
C(6)	C(5)	C(4)	1·400 (9)	120·3 (5)
C(1)	C(6)	C(5)		119·8 (5)
F(1)	C(7)	F(2)	1·347 (5)	106·0 (3)
F(1)	C(7)	F(3)		105·8 (4)
F(1)	C(7)	C(1)		113·5 (3)
F(2)	C(7)	F(3)	1·338 (6)	105·7 (4)
F(2)	C(7)	C(1)		113·8 (4)
F(3)	C(7)	C(1)	1·358 (5)	111·2 (4)
C(9)	C(8)	C(13)	1·402 (6)	118·4 (3)
C(13)	C(8)	C(14)	1·416 (5)	121·4 (3)
C(14)	C(8)	C(9)	1·505 (6)	120·1 (3)
C(10)	C(9)	C(8)	1·418 (6)	120·9 (3)
C(11)	C(10)	C(9)	1·430 (5)	120·3 (3)
C(12)	C(11)	C(15)	1·398 (5)	121·4 (3)
C(12)	C(11)	C(10)		118·3 (3)
C(15)	C(11)	C(10)	1·504 (5)	120·4 (3)
C(13)	C(12)	C(11)	1·406 (5)	121·3 (3)
C(8)	C(13)	C(12)		120·9 (3)

1	2	1–2	1	2	1–2
Cr	C(1)	2·116 (4)	Cr	C(8)	2·188 (4)
Cr	C(2)	2·146 (3)	Cr	C(9)	2·158 (2)
Cr	C(3)	2·146 (4)	Cr	C(10)	2·155 (4)
Cr	C(4)	2·140 (4)	Cr	C(11)	2·179 (3)
Cr	C(5)	2·137 (6)	Cr	C(12)	2·149 (3)
Cr	C(6)	2·113 (5)	Cr	C(13)	2·154 (4)

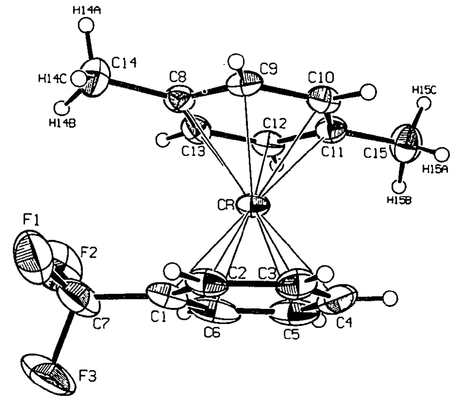


Fig. 1. View of (1) indicating atom labeling. Thermal ellipsoids drawn with 50% probability surfaces.

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[*] Lists of anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles, selected inter-ring distances, least-squares planes and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43844 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

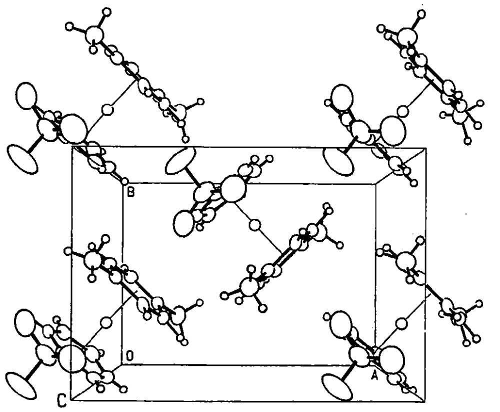

Fig. 2. Packing diagram of (1) as viewed down the *c* axis. The closest intermolecular contact is 2·51 (5) Å for H(10)···H(14*B*)(1−*x*, 1−*y*, *z*−0·5).

packing in Fig. 2. Principal computer programs are given by Gadol & Davis (1982); program for least-squares-planes calculations from Cordes (1983).

Related literature. A (benzene)(napthalene)Cr^0 complex has recently been reported (Kündig, Perret, Spicahiger & Bernardinelli, 1985). Jellinek (1963), Cotton, Dollase & Wood (1963), Ibers (1964) and Keulen & Jellinek (1966) have reported the structures of bisbenzeneCr^0. Elschenbroich, Mockel, Massa, Birkhahn & Zenneck (1982) reported a bisnaphthalene complex. Eyring, Zuerner & Radonovich (1981) and Larson, Seymour & Lagowski (1987) have reported trifluoromethylbenzene complexes of Cr^0.

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Structure of Chloro(isothiocyanato)bis(triphenylphosphine oxide)manganese(II)

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Abstract. $[MnCl(NCS)\{OP(C_6H_5)_3\}_2]$, $M_r = 705{\cdot}06$, orthorhombic, *Pbca*, $a = 10{\cdot}992$ (3), $b = 22{\cdot}331$ (2), $c = 27{\cdot}722$ (4) Å, $V = 6805$ (3) Å^3, $Z = 8$, $D_m = 1{\cdot}37$ (1), $D_x = 1{\cdot}376$ (5) g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 5{\cdot}98$ cm^{-1}, $F(000) = 2904$, $T = ca$ 293 K, $R = 0{\cdot}102$ for 2486 observed reflections. Pairs of molecules of the complex link through Mn—Cl′(1 − *x*, 1 − *y*, 1 − *z*) bonds to form dimeric units. Because of this dimerization, the Mn atom assumes a distorted trigonal-bipyramidal geometry. The Mn atom is 0·08 Å out of the equatorial plane formed by Cl, O and N atoms. Pairs of these bipyramids share Cl—Cl′ edges.

Experimental. Synthesized and characterized by Davolos (1978), colourless plates, 0·2 × 0·2 × 0·4 mm, D_m by flotation ($CH_3OH + CHBr_3$), CAD-4 diffractometer, ω–2θ scan mode, unit-cell dimensions from 25 reflections ($10 < \theta < 17°$), absorption ignored, $\theta < 24°$, $0 \le h \le 12$, $0 \le k \le 25$, $0 \le l \le 31$, three standard reflections showed no appreciable