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Acta Cryst. (1987). C**43**, 1624–1626

Structure of Bis(trifluoromethylbenzene)chromium(0) at 163 K

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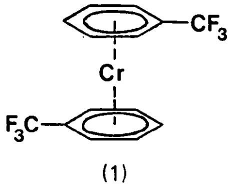
Abstract. $[Cr(C_7H_5F_3)_2]$, $M_r = 344{\cdot}22$, monoclinic, $P2_1/n$, $a = 8{\cdot}5435\ (12)$, $b = 7{\cdot}7583\ (14)$, $c = 10{\cdot}5058\ (20)$ Å, $\beta = 111{\cdot}801\ (12)°$, $V = 646{\cdot}5\ (2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}768$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 9{\cdot}220$ cm^{-1}, $F(000) = 344$, $R = 0{\cdot}0391$ for 1545 reflections ($F \geq 4\sigma_F$). The Cr lies on a crystallographic center of inversion and hence the rings are parallel and eclipsed. The average Cr—C distance is 2·136 (12) Å [range: 2·114 (2)–2·149 (3) Å], the distances increasing for C atoms furthest from the CF_3 group. The F atoms have large thermal parameters suggesting rotational disorder about the C—CF_3 bond. The average C—C distance in the rings is 1·408 (7) Å [range: 1·396 (4)–1·418 (3) Å] and the rings are 3·212 (4) Å apart.

Experimental. The title compound, (1), was prepared by the reaction of resistively vaporized Cr with trifluoromethylbenzene at liquid-nitrogen temperature and a pressure of $2{\cdot}6 \times 10^{-4}$ Pa (Seymour, Ssekaalo & Lagowski, 1987). Crystals were obtained by thermal-gradient sublimation as malformed needles. A summary of data collection and structural refinement is given in Table 1.



(1)

The initial Cr position was obtained from a Patterson map; initial C positions for rings from Cr–C vectors found in the Patterson map; remaining C and F atoms from electron density map. All atomic positional parameters, anisotropic thermal parameters for non-H and isotropic for H atoms refined by full-matrix least squares (*SHELX*76, Sheldrick, 1976); function minimized was $\sum w(F_o - F_c)^2$, where $w = \sigma_F^{-2}$. Electron density difference map calculated at $R = 0{\cdot}09$ revealed H atoms as peaks of 0·75–1·02 e Å^{-3}. Scattering factors and anomalous-dispersion correc-

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

(*a*) Data collection (163 K)[i,ii]	
Mode	ω scan
Scan range	Symmetrically over 1·2° 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)	55·9
Stability analysis	
Check reflections	002; 301; 110; 121
Computed s	0·00148 (16)
t	−0·000022 (3)
Correction range (on I)	0·976–1·000
2θ range (°)	4·0–60·0
Range in hkl, min.	0, −10, −14
max.	12, 10, 13
Reflections, measured, unique	3634, 1884
R_{int}	0·0300
Crystal dimensions (mm)	0·52 × 0·24 × 0·17
Crystal volume (mm^3)	0·0196
Transmission-factor range	0·867–0·892
(*b*) Structure refinement	
Instability factor p	0·04
Reflections used ($F \geq 4\sigma_F$)	1545
No. of variables	117
Goodness of fit, S	1·815
R, wR	0·0391, 0·0440
R for all data	0·0539
Max. shift/e.s.d.	0·0003
Max, min. density in difference map (e Å^{-3})	0·60, −0·44

Notes: (i) Unit-cell parameters were obtained by least-squares refinement of the setting angles of 45 reflections with $25{\cdot}1 < 2\theta < 29{\cdot}8°$. (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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tions for all non-H atoms taken from *International Tables for X-ray Crystallography* (1974); H-atom scattering factors from Stewart, Davidson & Simpson (1965). Atomic parameters in Table 2;* bond lengths and bond angles in Table 3. Atom labeling given on projection in Fig. 1; packing diagram in Fig. 2. Principal computer programs are given by Gadol & Davis (1982); program for least-squares-planes calculations from Cordes (1983).

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

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.

	x	y	z	U/U_{eq}(Å^2)
Cr	0·0	0·0	0·0	0·01613 (13)
C(1)	0·1785 (3)	0·1934 (3)	0·0102 (2)	0·239 (7)
C(2)	0·0244 (3)	0·2731 (3)	−0·0026 (3)	0·0270 (8)
C(3)	−0·0527 (3)	0·2302 (3)	0·0899 (3)	0·0305 (8)
C(4)	0·0225 (3)	0·1080 (3)	0·1944 (2)	0·0309 (8)
C(5)	0·1747 (3)	0·0302 (3)	0·2066 (2)	0·0291 (8)
C(6)	0·2551 (3)	0·0714 (3)	0·1155 (2)	0·0268 (7)
C(7)	0·2644 (3)	0·2415 (4)	−0·0852 (3)	0·0397 (10)
F(1)	0·3581 (2)	0·1154 (3)	−0·1053 (2)	0·0626 (9)
F(2)	0·1580 (3)	0·2876 (3)	−0·2085 (2)	0·0623 (8)
F(3)	0·3667 (3)	0·3774 (3)	−0·0371 (2)	0·0790 (11)
H(2)	−0·017 (3)	0·347 (4)	−0·074 (3)	0·039 (8)
H(3)	−0·153 (4)	0·277 (4)	0·081 (3)	0·046 (8)
H(4)	−0·030 (4)	0·083 (4)	0·250 (3)	0·035 (7)
H(5)	0·222 (3)	−0·058 (4)	0·270 (3)	0·036 (8)
H(6)	0·359 (3)	0·017 (3)	0·113 (3)	0·034 (7)

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

1	2	3	1–2	1–2–3
C(2)	C(1)	C(6)	1·415 (3)	120·6 (2)
C(6)	C(1)	C(7)	1·418 (3)	119·5 (2)
C(7)	C(1)	C(2)	1·493 (4)	119·9 (2)
C(3)	C(2)	C(1)	1·403 (4)	119·4 (2)
C(4)	C(3)	C(2)	1·411 (3)	120·2 (2)
C(5)	C(4)	C(3)	1·396 (4)	120·1 (3)
C(6)	C(5)	C(4)	1·406 (4)	120·8 (2)
C(1)	C(6)	C(5)		118·8 (2)
F(1)	C(7)	F(2)	1·330 (4)	106·2 (2)
F(1)	C(7)	F(3)		106·9 (2)
F(1)	C(7)	C(1)		113·4 (2)
F(2)	C(7)	F(3)	1·325 (3)	105·7 (2)
F(2)	C(7)	C(1)		113·2 (2)
F(3)	C(7)	C(1)	1·343 (4)	110·9 (2)
H(2)	C(2)	C(3)	0·90 (3)	126· (2)
H(2)	C(2)	C(1)		115· (2)
H(3)	C(3)	C(4)	0·90 (3)	120· (2)
H(3)	C(3)	C(2)		120· (2)
H(4)	C(4)	C(5)	0·89 (3)	122· (2)
H(4)	C(4)	C(3)		118· (2)
H(5)	C(5)	C(6)	0·94 (3)	118· (2)
H(5)	C(5)	C(4)		121· (2)
H(6)	C(6)	C(1)	0·99 (3)	115· (2)
H(6)	C(6)	C(5)		126· (2)

1	2	1–2	1	2	1–2
Cr	C(1)	2·114 (2)	Cr	C(4)	2·149 (3)
Cr	C(2)	2·130 (2)	Cr	C(5)	2·142 (2)
Cr	C(3)	2·145 (3)	Cr	C(6)	2·136 (2)

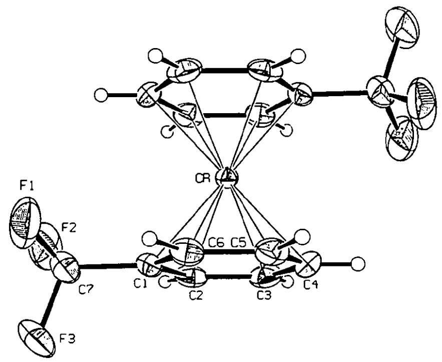

Fig. 1. View of (1) indicating atom labeling. Thermal ellipsoids drawn with 50% probability surfaces.

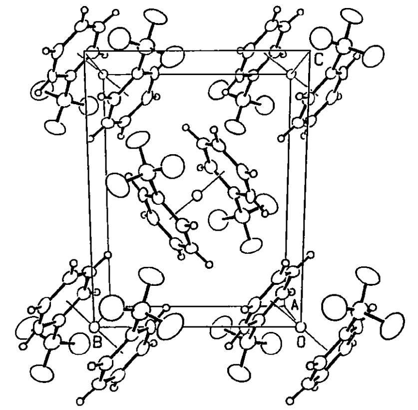

Fig. 2. Packing diagram of (1) as viewed down the a axis. The closest intermolecular contact is 2·66 (3) Å for F(1)···H(6)-($1-x, -y, -z$).

Related literature. Bisbenzene–Cr^0 structures have been reported by Jellinek (1963), Cotton, Dollase & Wood (1963), Ibers (1964) and Keulen & Jellinek (1966). Elschenbroich, Mockel, Massa, Birkhahn & Zenneck (1982) reported a bisnaphthalene complex and Eyring, Zuerner & Radonovich (1981) reported trifluoromethylbenzene complexes. Mixed arene–Cr^0 complexes have also been reported (Kündig, Perret, Spicahiger & Bernardinelli, 1985; Larson, Seymour & Lagowski, 1987).

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* Lists of anisotropic thermal parameters, torsion angles, interplanar distances, least-squares planes and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43843 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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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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