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Structure of 1,2,4,5-Tetrakis(trifluoromethyl)benzene

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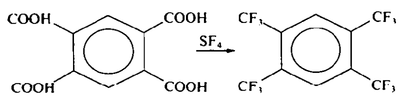
Abstract. $C_{10}H_2F_{12}$, $M_r = 350.11$, orthorhombic, *Pbca*, $a = 9.203$ (3), $b = 9.811$ (3), $c = 12.705$ (6) Å, $V = 1147.0$ (7) Å^3, $Z = 4$, $D_x = 2.03$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 2.470$ cm^{-1}, $F(000) = 680$, $T = 173$ K, $R = 0.0513$ for 828 reflections [$F_o \geq 6\sigma(F_o)$]. The molecule lies around an inversion center at 0, $\frac{1}{2}$, 0. Steric crowding caused by adjacent CF_3 groups is reflected in the exocyclic bond angles at C1 and C2. The angles for C4—C1—C2 and C5—C2—C1 are 123.5 (3) and 123.4 (3)°, respectively, while those for C5—C2—C3 and C4—C1—C3′ are 117.8 (3) and 117.2 (3)°, respectively. The C—F bond lengths are normal and average 1.330 (2) Å.



(I)

Experimental. The title compound (I) was synthesized by the action of SF_4 on benzene tetracarboxylic acid (Hasek, Smith & Engelhardt, 1960). In a typical reaction, 5 g of the tetraacid was placed into an 80 cm^3, stainless-steel high-pressure cylinder which was then fitted with a high-pressure valve and evacuated. A 50% stoichiometric excess of SF_4 was condensed into the cylinder at 77 K and subsequently heated to 393 K for 12 h. After heating, the cylinder was allowed to cool to room temperature and the volatile components were removed *in vacuo*. The solid residue was placed in a sublimator. Large well formed colorless crystals of (I) were obtained by sublimation onto a 'cold finger' at 273 K at approximately 10 mm Hg in a matter of a few hours. The data crystal was a colourless needle of approximate dimensions 0.27 × 0.35 × 0.83 mm. The data were collected on a Nicolet *R*3 diffractometer using a graphite monochromator and a Nicolet LT2 low-temperature delivery system. Lattice parameters were obtained from the least-squares refinement of 32 reflections with $18.4 < 2\theta < 22.0°$. The data were collected using the ω-scan technique, with a 1.2° ω scan at 8–12° min^{-1} and a 2θ range of 4.0–50.0° (h −10→10, k 0→11, l 0→15). A total of 2248 reflections was collected, of which 1009 were unique, with $R_{\text{int}} = 0.013$. Three reflections ($23\bar{3}$, $4\bar{2}2$, $\bar{4}\bar{2}\bar{2}$) were remeasured every 97 reflections to monitor instrument and crystal stability. A smoothed curve of the intensities of these check reflections was used to scale the data. The scaling factor range was 0.948–1.00. The data were also corrected for Lp effects but not for absorption. Data reduction and decay correction were performed using *SHELXTL-Plus* (Sheldrick, 1989). Reflections having $F_o < 6\sigma(F_o)$ were considered unobserved (411 reflections). The structure was solved by direct methods and refined by full-matrix least squares (Sheldrick, 1989) on *F*. In all, 105 parameters were refined. The non-H atoms were refined with anisotropic thermal parameters. The H-atom position was obtained from a ΔF map and refined with an isotropic thermal parameter. The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = 0.5kI^{-\frac{1}{2}}\{[\sigma(I)]^2 + (0.02I)^2\}^{1/2}$; the intensity, *I*, is given by $(I_{\text{peak}} - I_{\text{background}})$(scan rate), 0.02 is a factor to downweight intense reflections and to account for instrument instability, and *k* is the correction for Lp effects and decay. $\sigma(I)$ was

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Table 1. *Fractional coordinates and isotropic or equivalent isotropic thermal parameters* (Å^2) *for* 1,2,4,5-*tetrakis*(*trifluoromethyl*)*benzene*

The H atom was refined isotropically. For non-H atoms $U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{A}_{ij}$ where $\mathbf{A}_{ij}$ is the dot product of the ith and jth direct-space unit-cell vectors.

	x	y	z	U_{iso}/U_{eq}
C1	0.0508 (3)	0.5285 (3)	0.1010 (2)	0.0247 (8)
C2	0.1113 (3)	0.5950 (3)	0.0141 (2)	0.0251 (8)
C3	0.0583 (3)	0.5654 (3)	−0.0854 (3)	0.0262 (9)
C4	0.0984 (4)	0.5536 (3)	0.2126 (3)	0.0363 (10)
C5	0.2326 (4)	0.6974 (4)	0.0226 (3)	0.0418 (11)
F1	0.0416 (3)	0.6681 (2)	0.2518 (2)	0.0508 (8)
F2	0.0550 (3)	0.4539 (2)	0.2779 (2)	0.0575 (9)
F3	0.2415 (2)	0.5598 (3)	0.2241 (2)	0.0557 (8)
F4	0.3610 (2)	0.6399 (3)	0.0356 (2)	0.0629 (8)
F5	0.2146 (3)	0.7856 (2)	0.1003 (2)	0.0688 (10)
F6	0.2461 (3)	0.7704 (3)	−0.0653 (2)	0.0745 (10)
H3	0.089 (4)	0.598 (3)	−0.133 (3)	0.029 (9)

estimated from counting statistics: $\sigma(I) = (I_{peak} + I_{background})^{1/2}$(scan rate). Final $R = 0.0513$ for 828 reflections, with $wR = 0.0728$ ($R_{all} = 0.0605$, $wR_{all} = 0.0758$) and a goodness of fit = 2.748. Maximum $|\Delta/\sigma| < 0.1$ in the final refinement cycle and the minimum and maximum peaks in the final ΔF map were −0.24 and 0.71 e Å^{-3} (near *F*), respectively. The scattering factors for the non-H atoms were taken from Cromer & Mann (1968), with the anomalous-dispersion corrections taken from the work of Cromer & Liberman (1970). The scattering factors for the H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient were from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 55).* Fig. 1 was generated using *SHELXTL-Plus* (Sheldrick, 1989). The positional and thermal parameters for non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H atoms are listed in Table 2. The atom-labeling scheme is shown in Fig. 1. Other computer programs used in this work are listed in reference 11 of Gadol & Davis (1982).

Table 2. *Bond lengths* (Å) *and angles* (°) *for atoms of* 1,2,4,5-*tetrakis*(*trifluoromethyl*)*benzene*

1	2	3	1—2	1—2—3
C2	C1	C4	1.398 (4)	123.5 (3)
C4	C1		1.505 (4)	
C3	C2	C5	1.385 (4)	117.8 (3)
C3	C2	C1		118.8 (3)
C5	C2	C1	1.506 (4)	123.4 (3)
F1	C4	F2	1.336 (4)	105.4 (3)
F1	C4	F3		108.0 (3)
F1	C4	C1		112.0 (3)
F2	C4	F3	1.343 (4)	105.1 (3)
F2	C4	C1		112.1 (3)
F3	C4	C1	1.326 (4)	113.6 (3)
F4	C5	F5	1.320 (4)	107.3 (3)
F4	C5	F6		104.6 (3)
F4	C5	C2		112.9 (3)
F5	C5	F6	1.324 (4)	106.5 (3)
F5	C5	C2		113.3 (3)
F6	C5	C2	1.332 (4)	111.6 (3)
C3′	C1	C2	1.377 (4)	119.2 (3)
C3′	C1	C4		117.2 (3)
C2	C3	C1′		121.9 (3)
H3	C3	C2	0.75 (3)	121 (3)
H3	C3	C1′		117 (3)

Symmetry code: (′) $-x$, $1-y$, $-z$.

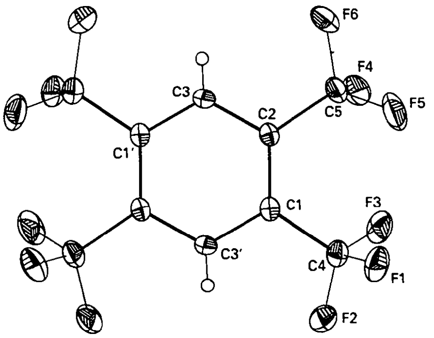

Fig. 1. View of $C_{10}H_2F_{12}$ (I) showing the atom-labeling scheme. Thermal ellipsoids are scaled to the 30% probability level. H atoms are scaled to an arbitrary size. The molecule lies around an inversion center at 0,½,0. Symmetry code: (′) $-x$, $1-y$, $-z$.

Related literature. The synthesis of (I) was undertaken as part of a study on the metalation and derivatization of trifluoromethyl substituted benzenes. Previous work (Carr, Chambers, Holmes & Parker, 1987) using 1,3,5-tris(trifluoromethyl)benzene has shown 2,4,6-tris(trifluoromethyl)phenyllithium to be thermally stable. Additionally, numerous derivatives of this reagent have been prepared (Carr *et al.*, 1987; Filler, Gnandt, Chen & Lin, 1991; Scholz, Roesky, Stalke, Keller & Edelmann, 1989) along with derivatives of 2,4- and 2,6-bis(trifluoromethyl)phenyllithium (Heuer, Jones & Schmutzler, 1990).

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* Lists of structure factors, anisotropic thermal parameters and torsion angles, and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54887 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0561]

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Structure of *cis*-2-Methyl-4-tosyloxymethyl-1,3-dioxolane

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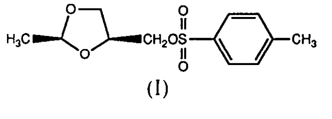
Abstract. $C_{12}H_{16}O_5S$, $M_r = 272.3$, orthorhombic, *Pbca*, $a = 20.301$ (5), $b = 8.952$ (2), $c = 14.707$ (6) Å, $V = 2673$ (2) Å^3, $Z = 8$, $D_x(105\ K) = 1.353$ Mg m^{-3}, m.p. (hot-stage microscope) 339.5–340.5 K (uncorrected), λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.240$ mm^{-1}, $F(000) = 1152$, $T = 105$ K, $R = 0.047$ for 764 observed reflections [$I \geq 3\sigma(I)$]. The conformation of the 1,3-dioxolane ring is a distorted envelope, E_2, with the pseudo-rotational parameters $P = 131.3$ (7)° and $\theta_m = 38.7$ (4)°. The five-membered ring is more puckered than expected from *ab initio* calculations [Cremer & Pople (1975). *J. Am. Chem. Soc.* **97**, 1354–1358], but the overall conformation is a favoured low-energy form. The tosyloxymethyl group adopts a conformation with τ(O8—S9—C21—C26) = ±89.3 (6)°. This is the usual orientation of the tosyloxymethyl group, illustrated by a search in the *Cambridge Structural Database* (1991, version 4.4). Crystal packing is governed by electrostatic interactions and van der Waals contacts.

Experimental. The title compound (I) was prepared by the method described by Triggle & Belleau (1962). Single crystals were obtained by slow cooling of a saturated hot solution in toluene. Intensity data was sampled from a crystal of size 0.05 × 0.20 × 0.30 mm on an Enraf–Nonius CAD-4 diffractometer with low-temperature device. Graphite-monochromated Mo $K\alpha$ radiation was used. Temperature was recorded with a thermocouple, variation within 1 K. Cell parameters and orientation matrix were determined from 17 reflections ($11.47 \leq \theta \leq 18.48°$). No corrections for absorption were performed. Three intensity control reflections measured every 10^4 s showed no systematic variation. ω scan, $\theta_{\max} = 23°$, range of *hkl* $-22 \leq h \leq 22$, $0 \leq k \leq 9$, $-16 \leq l \leq 16$. 4993 reflections were measured of which 1851 were unique and 1087 unobserved [$I < 3\sigma(I)$], $R_{\rm int} = 0.031$ (on I).



(I)

All non-H atoms were localized by direct methods (*MULTAN*80; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The positions of all H atoms were observed in the $\Delta\rho$ map in expected positions. Structure refinement on F (positional parameters for all non-H atoms, anisotropic displacement parameters for eight non-H atoms, isotropic displacement parameters for the remaining ten non-H atoms, H atoms fixed) by least squares, $w^{-1} = \sigma^2(F_o) + \frac{1}{4}(0.08F_o)^2$, $R = 0.047$, $wR = 0.063$ for 764 observed reflections [$I \geq 3\sigma(I)$] and 113 variables. $S = 1.19$, $(\Delta/\sigma)_{\max} = 0.02$, $\Delta\rho_{\max}/\Delta\rho_{\min} = 0.53/-0.24$ e Å^{-3}. Atomic scattering factors for uncharged atoms were used as implemented in the *SDP Structure Determination Package* (B. A. Frenz & Associates, Inc., 1982), which was used for all calculations (including *MULTAN*80). Final atomic coordinates and displacement parameters are listed in Table 1.* The bond lengths, bond angles, selected torsion angles and pseudo-rotational parameters involving the non-H atoms are listed in Table 2. Fig.

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* Lists of structure factors, anisotropic displacement parameters for eight non-H atoms, atomic coordinates for H atoms and information on selected intermolecular contacts have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54952 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0261]

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