

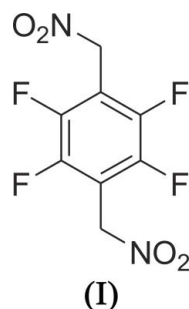
1,2,4,5-Tetrafluoro-3,6-bis(nitromethyl)benzene

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Key indicators

Single-crystal X-ray study
 $T = 299$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.038
 wR factor = 0.106
Data-to-parameter ratio = 11.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The title compound, $\text{C}_8\text{H}_4\text{F}_4\text{N}_2\text{O}_4$, crystallizes on a site of crystallographic inversion symmetry.Received 21 March 2006
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Comment

Interest in the field of crystal engineering, with the prediction of crystal structures and the design of organic compounds with specific properties, has increased significantly in the last few years. In the last decade, interactions of fluorine substituents in a variety of organic compounds have gained interest in life sciences and solid-state materials (Reichenbacher *et al.*, 2005).In the crystal structure of the centrosymmetric title compound, (I), the C1–F1, C2–F3 and C4–N1 bond distances (Table 1) are in quite good agreement with those found in the Cambridge Structural Database (CSD; Version 5.27, 2006 release; Allen, 2002); for the compound with refcode DOBQAM (Martin *et al.*, 1999), C–F bond distances fall in the range 1.329–1.346 Å and C–N = 1.498 Å, and in an analogous compound containing aromatic rings, C–F distances are in the range 1.340–1.349 Å (CSD refcode LABROW; Krebs *et al.*, 2003).

The crystal structure is stabilized by weak intramolecular and intermolecular C–H···F and C–H···O hydrogen bonds.

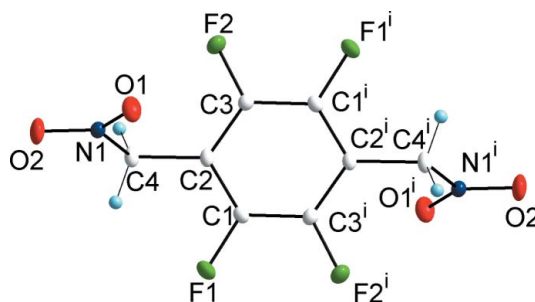


Figure 1

The numbering scheme of (I). Displacement ellipsoids are drawn at the 10% probability level. [Symmetry code: (i) $2 - x, 1 - y, 1 - z$.]

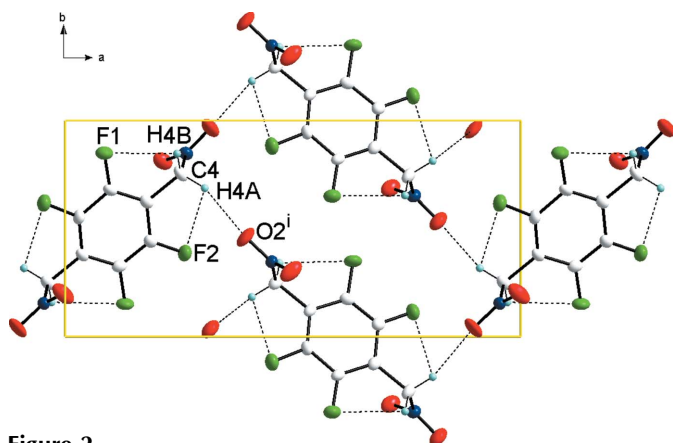


Figure 2
Packing diagram of (I), viewed along the *c* axis. Hydrogen-bonding interactions are indicated by dashed lines. Symmetry code as in Table 2.

Experimental

Nitromethane (5.9 g, 0.097 mol) in DMSO (15 ml) was added dropwise to a suspension of NaH (2.32 g, 0.097 mol) in DMSO (50 ml) with stirring. After the bubbling had subsided (*ca* 1 h), hexafluorobenzene (3 g, 0.016 mol) was added; the mixture was stirred for 20 h at room temperature and then poured into ice-water, acidified with 6 M HCl, then extracted with ethyl acetate. The organic extract was washed with water and brine and dried over anhydrous Na₂SO₄. Evaporation of the solvent in a vacuum gave a residue (mixture of mono- and disubstituted derivatives), which was separated by column chromatography to afford 1.6 g of 1,2,3,4,5-pentafluoro-6-(nitromethyl)benzene (44%) and 1.07 g of (I) (25%). The product was recrystallized from ethanol to afford 1.07 g of (I).

Crystal data

C ₈ H ₄ F ₄ N ₂ O ₄	<i>Z</i> = 4
<i>M_r</i> = 268.13	<i>D_x</i> = 1.848 Mg m ⁻³
Monoclinic, <i>C</i> 2/ <i>c</i>	Mo <i>K</i> α radiation
<i>a</i> = 17.570 (4) Å	<i>μ</i> = 0.20 mm ⁻¹
<i>b</i> = 7.2870 (15) Å	<i>T</i> = 299 (2) K
<i>c</i> = 8.5746 (17) Å	Block, colourless
<i>β</i> = 118.62 (3)°	0.5 × 0.5 × 0.32 mm
<i>V</i> = 963.7 (4) Å ³	

Data collection

Oxford Diffraction Xcalibur CCD diffractometer	2981 measured reflections
<i>ω</i> and <i>φ</i> scans	977 independent reflections
Absorption correction: analytical (Clark & Reid, 1995)	825 reflections with <i>I</i> > 2σ(<i>I</i>)
<i>T</i> _{min} = 0.877, <i>T</i> _{max} = 0.958	<i>R</i> _{int} = 0.016
	<i>θ</i> _{max} = 26.4°

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2 + 0.7257P]$
$R[F^2 > 2\sigma(F^2)] = 0.038$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.106$	(Δ/σ) _{max} = 0.001
<i>S</i> = 1.05	$\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$
977 reflections	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
85 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

F1—C1	1.339 (2)	N1—C4	1.494 (2)
F2—C3	1.337 (2)	C2—C1	1.376 (2)
N1—O1	1.198 (2)	C2—C3	1.377 (3)
N1—O2	1.200 (2)	C2—C4	1.495 (2)
O1—N1—O2	123.6 (2)	F1—C1—C2	119.5 (2)
F2—C3—C2	119.8 (2)		

Table 2

Hydrogen-bond 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>
C4—H4A...F2	0.97	2.54	2.830 (3)	97
C4—H4B...F1	0.97	2.48	2.826 (2)	101
C4—H4A...O2 ⁱ	0.97	2.54	3.435 (5)	153

Symmetry code: (i) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$.

H atoms were positioned geometrically and allowed to ride on their corresponding parent atom at a distance of 0.97 Å, with *U*_{iso} values freely refined.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2001); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2005).

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