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N,*N*′-Bis(2,2,2-trifluoroacetyl)hexane-1,6-diamine

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The title molecule, $C_{10}H_{14}F_6N_2O_2$, possesses a crystallographically imposed centre of symmetry. In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules into two-dimensional crimped layers parallel to the *bc* plane.

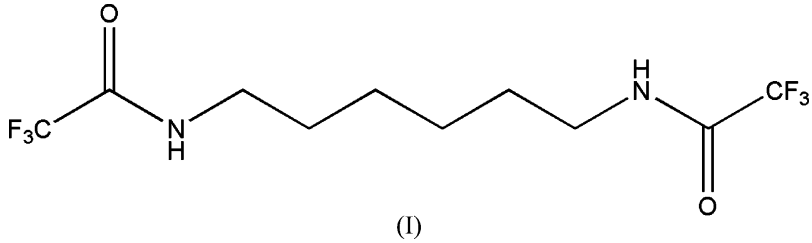
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.060
wR factor = 0.157
Data-to-parameter ratio = 15.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

As a monomer of fluorinated polymers, the title compound, (I), has been widely investigated (Chapman *et al.*, 1995). We present here its crystal structure.



(I)

The title molecule posseses a crystallographically imposed centre of symmetry (Fig. 1). The bond lengths and angles in (I) are normal (Allen *et al.*, 1987). The distance between the two O atoms in the molecule is 11.797 (3) Å. In the crystal structure, intermolecular N—H···O hydrogen bonds (Table 1) link the molecules into two-dimensional crimped layers parallel to the *bc* plane (Fig. 2).

Experimental

1,6-Diaminohexane (2.32 g, 20.0 mmol) was dissolved in 20 ml of dry THF at room temperature. Trifluoroacetic anhydride (4.62 g, 22.0 mmol) was added under ultrasonic conditions for 5 min. THF and the excess trifluoroacetic anhydride were removed under reduced pressure. The resulting white solid was washed with water to give the pure product (yield 90%, 5.54 g; m.p. 403.2 K). Colourless block single crystals suitable for X-ray diffraction were obtained by slow evaporation of an acetone solution at room temperature.

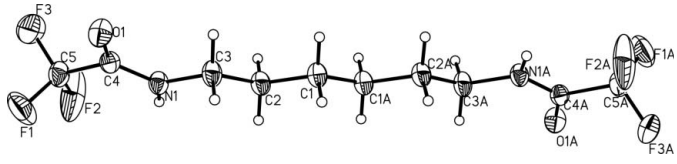


Figure 1
The molecular structure of (I), with atomic labels and displacement ellipsoids drawn at the 20% probability level [symmetry code: (A) $1-x$, $1-y$, $1-z$]. Only major components of the disordered trifluoromethyl groups are shown.

Crystal data

$C_{10}H_{14}F_6N_2O_2$
M_r = 308.23
Monoclinic, $P2_1/c$
a = 7.2009 (14) Å
b = 10.498 (2) Å
c = 8.9607 (18) Å
β = 90.51 (3)°
V = 677.4 (2) Å^3
Z = 2
D_x = 1.511 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 296 (2) K
Block, colourless
0.63 × 0.60 × 0.21 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.907, T_{max} = 0.968
6508 measured reflections
1544 independent reflections
1298 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.157
S = 1.03
1544 reflections
103 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 0.4876P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.13 (2)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.86	2.03	2.883 (2)	169

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

The H-atoms were included in calculated positions and treated in the subsequent refinement as riding atoms, with C—H = 0.97 Å, N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(carrier atom). The trifluoromethyl group was treated as disordered between two orientations with refined occupancies of 0.906 (4) and 0.094 (4), respectively. All C—F bond lengths were restrained to be equal within 0.003 Å. The F atoms from the minor component were refined isotropically with a common U_{iso}(F). In the major component, the large values of atomic displacement parameters for atoms F1, F2 and F3, and strong anisotropy of their displacement ellipsoids, indicate further unresolved disorder of the trifluoromethyl group.

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

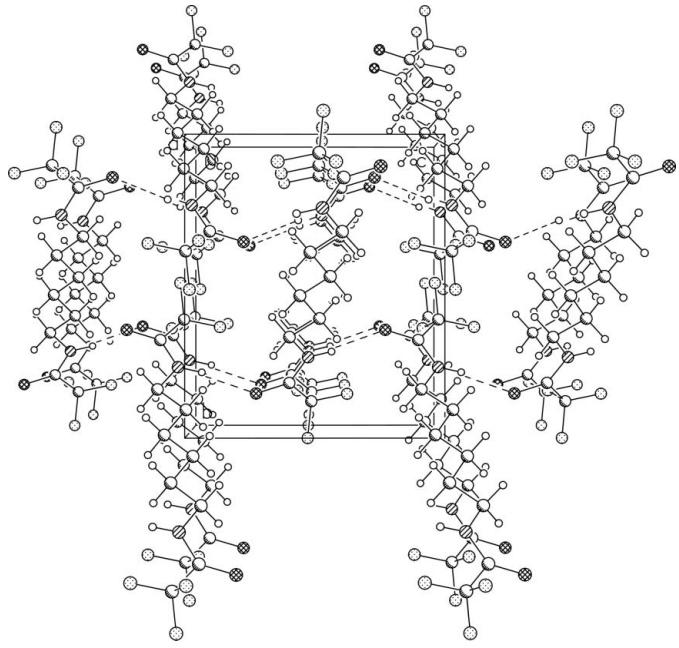


Figure 2
The crystal packing viewed down the *a* axis. The dashed lines denote intermolecular hydrogen bonds. Only major components of the disordered trifluoromethyl groups are shown.

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