

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55725 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1020]

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Structure of *trans*-Decafluoroazobenzene

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Abstract

The planar *trans*-decafluoroazobenzene molecules are stacked along the [102] direction with an interlayer distance of 4.22 Å.

Comment

The title compound was prepared (Birchall, Haszeldine & Kemp, 1970) by the coupling of penta-

fluoroaniline in benzene using lead tetraacetate as a catalyst and crystallized from methanol. Individual molecules are situated in the unit cell so that a crystallographic inversion centre bisects the N—N bond. The crystal structure is composed of layers of decafluoroazobenzene molecules which are stacked along the [102] direction with an interlayer spacing of 4.22 Å. The bond lengths and distances are comparable with those reported for the *trans*-decafluoroazobenzene–*trans*-stilbene (1/1) complex (Bruce, Snow & Tiekink, 1987).

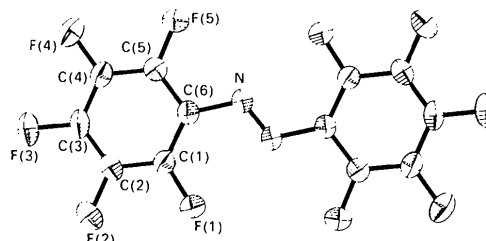


Fig. 1. View of the molecule showing the labelling of the non-H atoms. Thermal ellipsoids are shown at 50% probability levels.

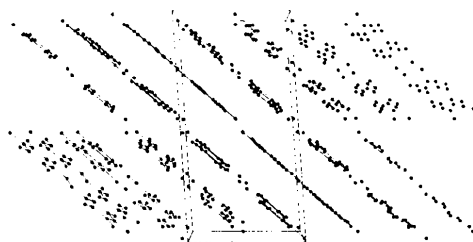


Fig. 2. Packing of the molecules in the unit cell viewed down the *b* axis.

Experimental

Crystal data

$C_{12}F_{10}N_2$
 $M_r = 362.1$
 Monoclinic
 $C2/c$
 $a = 17.371$ (6) Å
 $b = 7.621$ (2) Å
 $c = 8.862$ (3) Å
 $\beta = 94.03$ (2)°
 $V = 1170.3$ (6) Å³
 $Z = 4$
 $D_x = 2.055$ Mg m⁻³
 $D_m = 2.038$ Mg m⁻³

Mo $K\alpha$ radiation
 $\lambda = 0.71069$ Å
 Cell parameters from 50 reflections
 $\theta = 7.5$ – 17.5 °
 $\mu = 0.237$ mm⁻¹
 $T = 298$ K
 Thin plate
 $1.0 \times 0.66 \times 0.06$ mm
 Orange

Data collection

Siemens P4 diffractometer
 $2\theta/\theta$ scans

$R_{int} = 0.044$
 $\theta_{max} = 55.0$ °

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Absorption correction: $h = -22 \rightarrow 22$
 empirical (Ugozzoli, 1987) $k = 0 \rightarrow 9$
 $T_{\min} = 0.930$, $T_{\max} = 1.052$ $l = 0 \rightarrow 11$
 2420 measured reflections 2 standard reflections
 1347 independent reflections monitored every 200 reflections
 981 observed reflections intensity variation: insignificant
 $[F > 4\sigma(F)]$ (Sheldrick, 1990)

Refinement

Refinement on F $(\Delta/\sigma)_{\max} = 0.000$
 Final $R = 0.0317$ $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$
 $wR = 0.0438$ $\Delta\rho_{\min} = -0.15 \text{ e } \text{\AA}^{-3}$
 $S = 1.44$
 981 reflections Atomic scattering factors from *SHELXTL/PC*
 109 parameters (Sheldrick, 1990)
 $w = 1.0/[\sigma^2(F) + 0.0005F^2]$

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)

The coordinates for the second half of the molecule are generated by the symmetry operation $\frac{1}{2} - x, -\frac{1}{2} - y, 2 - z$; U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
N	0.2666 (1)	-0.3122 (2)	0.9721 (1)	0.044 (1)
C(1)	0.3388 (1)	-0.1013 (2)	0.8145 (2)	0.038 (1)
C(2)	0.3898 (1)	-0.0788 (2)	0.7032 (2)	0.042 (1)
C(3)	0.4205 (1)	-0.2228 (2)	0.6358 (2)	0.045 (1)
C(4)	0.4020 (1)	-0.3894 (2)	0.6803 (2)	0.042 (1)
C(5)	0.3512 (1)	-0.4109 (2)	0.7920 (2)	0.040 (1)
C(6)	0.3180 (1)	-0.2683 (2)	0.8611 (2)	0.037 (1)
F(1)	0.3112 (1)	0.0439 (1)	0.8749 (1)	0.051 (1)
F(2)	0.4089 (1)	0.0818 (1)	0.6607 (1)	0.061 (1)
F(3)	0.4690 (1)	-0.2009 (2)	0.5276 (1)	0.065 (1)
F(4)	0.4327 (1)	-0.5287 (1)	0.6166 (1)	0.061 (1)
F(5)	0.3337 (1)	-0.5734 (1)	0.8324 (1)	0.054 (1)

Table 2. Bond lengths (\AA) and angles ($^\circ$)

C(1)—F(1)	1.333 (2)	C(3)—C(4)	1.375 (2)
C(2)—F(2)	1.330 (2)	C(4)—C(5)	1.381 (2)
C(3)—F(3)	1.330 (2)	C(5)—C(6)	1.393 (2)
C(4)—F(4)	1.332 (2)	C(1)—C(6)	1.393 (2)
C(5)—F(5)	1.330 (2)	N—C(6)	1.414 (2)
C(1)—C(2)	1.382 (2)	N—N ⁱ	1.231 (3)
C(2)—C(3)	1.375 (2)		
C(6)—N—N ⁱ	115.6 (1)	C(3)—C(4)—C(5)	119.3 (1)
C(2)—C(1)—C(6)	121.2 (1)	C(3)—C(4)—F(4)	120.4 (1)
C(2)—C(1)—F(1)	116.8 (1)	C(5)—C(4)—F(4)	120.3 (1)
C(6)—C(1)—F(1)	122.1 (1)	C(4)—C(5)—C(6)	121.9 (1)
C(1)—C(2)—C(3)	119.9 (1)	C(4)—C(5)—F(5)	118.3 (1)
C(1)—C(2)—F(2)	120.1 (1)	C(6)—C(5)—F(5)	119.9 (1)
C(3)—C(2)—F(2)	120.0 (1)	N—C(6)—C(1)	127.7 (1)
C(2)—C(3)—C(4)	120.5 (1)	N—C(6)—C(5)	115.0 (1)
C(2)—C(3)—F(3)	119.8 (1)	C(1)—C(6)—C(5)	117.3 (1)
C(4)—C(3)—F(3)	119.7 (1)		

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

The density was measured by flotation in a mixture of CCl_4 and bromoform. The data were collected using a variable scan speed of $5.33\text{--}29.3^\circ \text{ min}^{-1}$ in ω . The structure was solved by direct methods and refined by full-matrix least squares. *SHELXTL/PC* (Sheldrick, 1990) was used for all calculations.

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Structure of Zizyberannalic Acid

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Abstract

In the pentacyclic triterpenoid zizyberannalic acid several C—C bonds and C—C—C angles deviate by more than 3σ from their expected values. The five-membered rings *A* and *E* are in a distorted envelope conformation. The six-membered rings *B*, *C* and *D* are in a slightly distorted chair conformation with mean torsion angles of $55.7(6)$, $58.1(6)$ and $55.7(6)^\circ$, respectively. The structure is stabilized by O—H \cdots O hydrogen bonds in addition to van der Waals forces.

Comment

The pentacyclic triterpenoid zizyberannalic acid was isolated by Kundu and co-workers from both the bark and the roots of *Zizyphus jujuba* (Kundu,

* Contribution No. 803.