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Structure of 5,6,7,8-Tetrafluoro-9-methyl-1,4-dihydro-1,4-iminonaphthalene, $C_{11}H_7F_4N$

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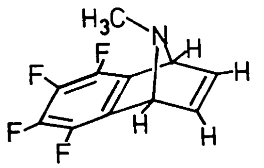
Abstract. $M_r = 229{\cdot}18$, monoclinic, $P2_1/n$, $a = 6{\cdot}258\,(4)$, $b = 31{\cdot}85\,(1)$, $c = 9{\cdot}481\,(6)$ Å, $\beta = 99{\cdot}71\,(5)°$, $V = 1862{\cdot}60$ Å^3, $Z = 8$, $D_x = 1{\cdot}63$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 1{\cdot}46$ cm^{-1}, $F(000) = 928$, $T = 115$ K, $R = 0{\cdot}057$ for 1687 unique observed reflections. The distances from the *N*-methyl carbon atom to the center of the benzene ring in the two independent molecules are 3·608 (8) and 3·616 (11) Å; the corresponding imine bridge bond angles are 95·0 (7) and 96·0 (4)°, which are small for an sp^3-hybridized N atom.

Introduction. The crystal structure determination of the title compound was undertaken in continuation of our studies of the chemical and physical properties of arenimines (Gribble, Sibi, Kumar & Kelly, 1983; LeHoullier & Gribble, 1983). It was of particular interest to establish the configuration of the methyl group in the solid state and to determine the imine bridge bond angle in this presumably strained molecule.



Experimental. Preparation described by Gribble, Allen, LeHoullier, Eaton, Easton, Slayton & Sibi (1981). Recrystallization from hexane; colorless, fragment cut from large irregular single crystal, 0·25 × 0·50 × 0·50 mm; Syntex $P\bar{1}$ diffractometer equipped with variable-temperature device (Strouse, 1976); graphite monochromator; unit-cell parameters by least-squares refinement of 15 reflections ($17{\cdot}50 \le 2\theta \le 27{\cdot}57°$), θ–2θ scan, 24·0° min^{-1}, $2\theta_{\max} = 45°$ for range $0 \le h \le 6$, $0 \le k \le 33$, $-10 \le l \le 10$; 3 reflections monitored every 97 reflections with no significant variation; 2863 measured intensities, 2453 unique, 1687 with $I > 3\sigma(I)$, 766 unobserved; Lorentz and polarization but no absorption correction. Structure solved by direct methods; N(2), C(8), C(11), C(27), C(28), C(30) and all hydrogen atoms located in difference Fourier maps. Refinement included positional and anisotropic thermal parameters for all non-hydrogen atoms in high-occupancy fragments. Hydrogen atoms included in structure factor calculation. Difference maps revealed disorder in imine bridge and ethylene fragments of both molecules in asymmetric unit. Low-occupancy fragments labeled *L*1 [consisting of N(1*D*), C(7*D*)–C(11*D*) and H(7*D*)–H(11*D*)] and *L*2 [consisting of N(2*D*), C(26*D*)–C(30*D*) and H(26*D*)–H(30*D*)] located and fragment *L*2 included in structure factor calculation. Fragment *L*1 non-hydrogen atoms with isotropic temperature factors included in refinement with high-occupancy fragments *H*1 [consisting of N(1), C(7)–C(11) and H(7)–H(11)] and *H*2 [consisting of N(2), C(26)–C(30) and H(26)–H(30)]. Sums of occupancies of two fragments in each molecule constrained to unity. Refined occupancies of *H*1 and *H*2 fragments 0·70 (1) and 0·94 (1), respectively. Refinement in $P2_1/m$ did not account for disorder nor were

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any alternate symmetry elements found. Structure refined by full-matrix least-squares procedure based on F with max. $\sin\theta/\lambda = 0{\cdot}54$ Å^{-1}; refinement of 315 parameters converged to $R_w = 0{\cdot}073$, $w = 1/\sigma^2(F_o)$, error of fit $S = 2{\cdot}18$; $\Delta_{\max}/\sigma = 0{\cdot}042$, av. $\Delta/\sigma = 0{\cdot}003$; max. height of $0{\cdot}32$ e Å^{-3} in final difference Fourier synthesis; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974); all calculations performed on UCLA Departmental DEC VAX 11/780 using the *UCLA Crystallographic Package* (1983) (locally edited versions of *CARESS, PROFILE, MULTAN, ORFLS, ORFFE, ABSORB* and *ORTEP*).

Table 1. *Positional parameters and equivalent isotropic temperature factors for H*1 *and H*2

	x	y	z	U_{eq} (Å$^2\times10^4$)*
C(1)	0·1362 (9)	−0·0270 (2)	0·2751 (6)	283 (38)
C(2)	0·0187 (8)	0·0061 (2)	0·3097 (5)	259 (37)
C(3)	0·0821 (9)	0·0468 (2)	0·2809 (6)	279 (38)
C(4)	0·2603 (9)	0·0529 (2)	0·2191 (6)	257 (38)
C(5)	0·3820 (9)	0·0187 (2)	0·1832 (6)	271 (38)
C(6)	0·3193 (9)	−0·0210 (2)	0·2125 (6)	305 (39)
C(7)	0·3952 (16)	−0·0666 (3)	0·1754 (12)	191 (61)
C(8)	0·2194 (16)	−0·0857 (2)	0·0603 (8)	190 (58)
C(9)	0·0439 (15)	−0·0913 (3)	0·1224 (9)	191 (58)
C(10)	0·1055 (16)	−0·0758 (3)	0·2776 (13)	190 (65)
C(11)	0·4586 (18)	−0·0761 (3)	0·4429 (9)	213 (67)
C(20)	0·6501 (8)	0·2667 (2)	0·2192 (5)	232 (36)
C(21)	0·5920 (8)	0·2268 (2)	0·2519 (5)	232 (36)
C(22)	0·7178 (9)	0·1931 (2)	0·2220 (6)	262 (38)
C(23)	0·8968 (9)	0·1990 (2)	0·1600 (5)	236 (37)
C(24)	0·9579 (8)	0·2392 (2)	0·1263 (5)	209 (35)
C(25)	0·8364 (8)	0·2729 (2)	0·1552 (5)	213 (34)
C(26)	0·8503 (9)	0·3209 (2)	0·1354 (6)	188 (37)
C(27)	0·6517 (9)	0·3324 (2)	0·0194 (6)	234 (38)
C(28)	0·4802 (9)	0·3264 (2)	0·0786 (6)	258 (39)
C(29)	0·5616 (9)	0·3111 (2)	0·2361 (6)	252 (40)
C(30)	0·9048 (10)	0·3270 (2)	0·3979 (6)	291 (41)
F(1)	−0·1602 (5)	0·0012 (1)	0·3730 (3)	323 (21)
F(2)	−0·0331 (5)	0·0801 (1)	0·3153 (3)	418 (24)
F(3)	0·3228 (5)	0·0924 (1)	0·1910 (3)	420 (24)
F(4)	0·5588 (5)	0·0267 (1)	0·1220 (3)	339 (22)
F(5)	0·4168 (5)	0·2192 (1)	0·3153 (3)	302 (21)
F(6)	0·6609 (5)	0·1535 (1)	0·2563 (3)	378 (23)
F(7)	1·0190 (5)	0·1656 (1)	0·1344 (3)	369 (23)
F(8)	1·1373 (4)	0·2442 (1)	0·0671 (3)	268 (20)
N(1)	0·3367 (10)	−0·0893 (2)	0·3040 (8)	172 (47)
N(2)	0·7624 (7)	0·3364 (1)	0·2621 (5)	244 (33)

* $U_{eq} = [1/(6\pi^2)] \times \sum\beta_{ij}a_ia_j$.

Table 2. *Positional parameters and isotropic temperature factors for L*1 *and L*2 *fragments*

	x	y	z	U(Å$^2\times10^4$)
C(7*D*)	0·4345 (38)	−0·0642 (8)	0·2357 (25)	146 (69)
C(8*D*)	0·5187 (34)	−0·0754 (6)	0·3970 (22)	111 (64)
C(9*D*)	0·3555 (36)	−0·0814 (7)	0·4601 (23)	211 (67)
C(10*D*)	0·1514 (47)	−0·0732 (9)	0·3352 (29)	309 (93)
C(11*D*)	0·1067 (46)	−0·0851 (9)	0·0763 (29)	219 (82)
N(1*D*)	0·2486 (30)	−0·0914 (5)	0·2174 (22)	325 (58)
C(26*D*)	0·9189	0·3146	0·2138	253
C(27*D*)	0·9889	0·3325	0·3591	253
C(28*D*)	0·8003	0·3254	0·4202	253
C(29*D*)	0·6101	0·3117	0·3253	253
C(30*D*)	0·5280	0·3315	0·0297	253
N(2*D*)	0·6648	0·3414	0·1691	253

Discussion. Atomic coordinates and equivalent isotropic temperature factors are listed in Table 1 for the high-occupancy fragments.* For the low-occupancy fragments the positional parameters and isotropic temperature factors are given in Table 2. An *ORTEP* drawing is shown in Fig. 1 with the atom numbering scheme for both molecules in the asymmetric unit. All C—F bonds are normal [average = 1·358 (6) Å] and C—C bonds all fall in the normal range; the C—N distances range from 1·468 (11) Å for C(11)—N(1) to 1·515 (11) Å for C(7)—N(1).

The disorder is created by a twofold rotation of the molecule, which superimposes the fluorinated rings. The resulting arrangement of the dihydro ring and imine bridge fragments for the N(1) molecule is depicted in Fig. 2. The ethylene hydrogens are from 2·18 to 2·26 Å below the plane of the benzene ring and the imine methyl hydrogens are from 2·42 to 2·66 Å above this plane.

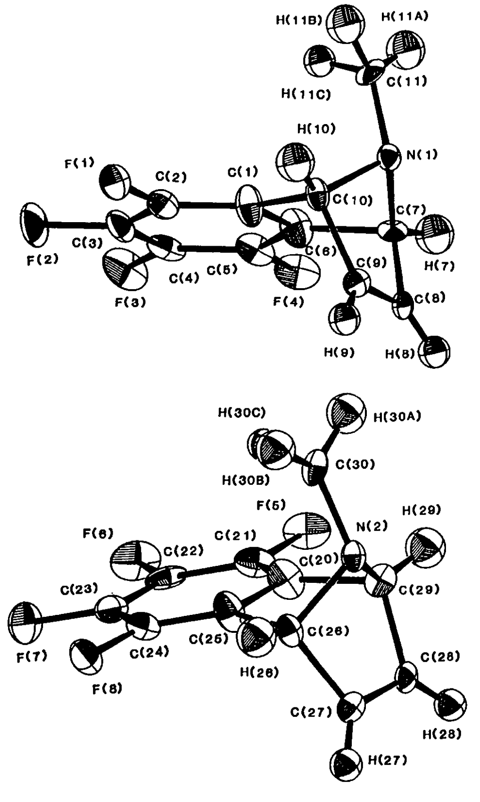

Fig. 1. *ORTEP* drawings of the high-occupancy fragments for both molecules in the asymmetric unit and the atom numbering scheme.

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* Lists of structure factors, anisotropic thermal parameters, hydrogen-atom positional parameters, all bond lengths and angles and a packing diagram have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39709 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

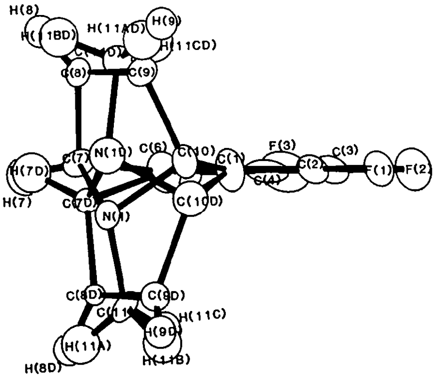

Fig. 2. *ORTEP* drawing of the disordered N(1) molecule. Atoms F(4), H(10) and H(10*D*) are omitted for clarity.

The structure analysis confirms the position of the *N*-methyl group over the benzene ring with a distance from the center of the benzene ring to the methyl carbon atom of 3·608 (8) for C(30) and 3·616 (11) Å for C(11).

As expected, the N(1) and N(2) imine bridge bond angles of 95·0 (7) and 96·0 (4)° are very small for an sp^3-hybridized nitrogen, accounting for the relatively high barrier to nitrogen inversion observed for this compound (Gribble, Easton & Eaton, 1970). Similarly small bond angles of 92·3 (7) and 96° are found for the methano bridge of 5,6,7,8-tetrafluoro-1,2,3,4-tetrahydro-9-isopropyl-1,4-methanonaphthalene and the epoxy bridge of the photodimer of 1,4-epoxy-1,4-dihydronaphthalene, respectively (Brown & Mason, 1978; Bordner, Stanford & Dickerson, 1970).

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Structures of 2-(5-Amino-1,3,4-thiadiazol-2-yl)benzenesulphonamide (I), $C_8H_8N_4O_2S_2$, and 2-(5-Amino-1,3,4-thiadiazol-2-yl)-*N*-methylbenzenesulphonamide (II), $C_9H_{10}N_4O_2S_2$

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Abstract. (I): $M_r = 256{\cdot}3$, monoclinic, $P2_1/n$, $a = 14{\cdot}604\ (5)$, $b = 14{\cdot}605\ (6)$, $c = 5{\cdot}206\ (3)$ Å, $\beta = 94{\cdot}78\ (1)°$, $V = 1106{\cdot}5\ (9)$ Å^3, $Z = 4$, $D_x = 1{\cdot}539$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}413$ mm^{-1}, $F(000) = 528$, $R = 0{\cdot}038$ for 1781 observed reflections, $T = 295$ K. (II): $M_r = 270{\cdot}3$, monoclinic, $P2_1/a$, $a = 21{\cdot}308\ (8)$, $b = 13{\cdot}420\ (6)$, $c = 8{\cdot}155\ (2)$ Å, $\beta = 94{\cdot}49\ (1)°$, $V = 2325\ (1)$ Å^3, $Z = 8$,