worthwhile to investigate the exact nature of this disorder. Fig. 1 shows the molecule with adopted numbering. The packing and hydrogen bonding are illustrated in Fig. 2. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Data on the geometry are assembled in Table 2. Scattering factors of Cromer & Mann (1968) were used. Geometrical calculations were performed and illustrations drawn with the programs *PLATON* and *PLUTON* of the *EUCLID* package (Spek, 1982).

**Related literature.** The title compound was isolated as an intermediate in the total synthesis of $C_{19}$-diterpene alkaloids (van Beek, van der Baan, Klumpp & Bickelhaupt, 1986).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43963 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# 3,8,11,16-Tetrakis(trifluoromethyl)-1,2,4,7,9,10,12,15-octaazacyclohexadeca-2,8,10,16(1)-tetraene

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**Abstract.** $C_{12}H_{12}F_{12}N_8$, $M_r = 496{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}572$ (2), $b = 9{\cdot}509$ (3), $c = 23{\cdot}502$ (5) Å, $V = 1915{\cdot}7$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}72$ Mg m$^{-3}$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}141$ mm$^{-1}$, $F(000) = 992$, $T = 293$ K, $R = 0{\cdot}051$ for 818 unique reflexions $[F > 3\sigma(F)]$. The ring is composed of two distorted *anti* 1,3,4,6-tetraaza-2,4-hexadiene moieties linked by two ethylene bridges, one *anti* and the other *gauche*, so that an approximate twofold axis passes through the C—C bonds.

**Experimental.** The title compound was obtained on mixing 2,5-dichloro-1,1,1,6,6,6-hexafluoro-3,4-diaza-2,4-hexadiene with 1,2-ethanediamine in molar ratio 1:2 in diethyl ether at 273 K for 30 min followed by recrystallization from 50:50 petroleum ether (b.p. 313–333 K) and chloroform. Crystals suitable for X-ray work were obtained from aqueous ethanol (10:90 $H_2O$: EtOH).

Crystal size 0·4 × 0·4 × 0·2 mm; Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; unit-cell dimensions from setting angles of 25 accurately centred reflexions ($5{\cdot}0 < \theta < 15{\cdot}8°$); $\omega$–$2\theta$ scan mode used to measure 1194 reflexions with $I > 0$, $\omega$-scan width of $(1{\cdot}00 + 0{\cdot}35\tan\theta)°$ and scan speed ranging from 0·3 to 5° min$^{-1}$ according to the intensity gathered in a pre-scan; $0 \leq h \leq 9$, $0 \leq k \leq 10$, $0 \leq l \leq 26$; $0 < \theta < 25°$; 818 unique structure amplitudes with $F > 3\sigma(F)$; negligible change in intensity standards ($\bar{1}\bar{2}\bar{3}$; $\bar{1}\bar{2}0$; $\bar{1}\bar{2}4$) measured every 2 h; Lorentz and polarization corrections but absorption ignored; structure solution *via* *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), all non-H atoms found in a Fourier map, H atoms constrained to chemically reasonable positions except for those attached to N atoms, which were located from a difference Fourier map; full-matrix least squares based on $F$ using

*SHELX*76 (Sheldrick, 1976); final $R = 0{\cdot}051$, $wR = 0{\cdot}047$, $w = 1{\cdot}62635/[\sigma^2(F_o) + 0{\cdot}000242F_o^2]$.

In order to maintain a tolerable reflexion-to-parameter ratio only the F and some N atoms were subjected to anisotropic refinement; the remaining N and C atoms were treated isotropically as were the H atoms, which shared a common isotropic parameter [0·037 (7) Å$^2$]. Max. fluctuation in final difference map in range −0·26 to 0·24 e Å$^{-3}$; max. LS shift-to-e.s.d. ratio 0·022 [*z*, H(16)]. Scattering factors from *International Tables for X-ray Crystallography* (1974); computations carried out on the joint CDC 7600/Amdahl 470 system of the University of Manchester Regional Computing Centre.*

Table 1. *Fractional atomic coordinates* (×10$^4$) *and vibrational parameters* (Å$^2$ ×10$^4$) *for non*-H *atoms*

| | $x$ | $y$ | $z$ | $U/U_{eq}$* |
|---|---|---|---|---|
| C(1) | 9488 (11) | 2865 (11) | 7327 (4) | 37 (3) |
| N(2) | 8518 (9) | 3665 (8) | 7070 (3) | 39 (2) |
| N(3) | 7572 (9) | 4453 (8) | 7445 (3) | 42 (2) |
| C(4) | 6410 (11) | 4954 (10) | 7175 (4) | 35 (3) |
| N(5) | 5960 (11) | 4696 (9) | 6633 (4) | 52 |
| C(6) | 5177 (13) | 5667 (12) | 6237 (4) | 51 (3) |
| C(7) | 6191 (12) | 6795 (11) | 6008 (5) | 49 (3) |
| N(8) | 7608 (12) | 6248 (10) | 5725 (4) | 60 |
| C(9) | 7780 (12) | 5793 (10) | 5194 (4) | 38 (3) |
| N(10) | 8852 (9) | 4983 (9) | 4996 (3) | 42 (2) |
| N(11) | 9806 (10) | 4490 (8) | 5444 (3) | 41 (2) |
| C(12) | 10964 (13) | 3808 (11) | 5264 (4) | 42 (3) |
| N(13) | 12034 (11) | 3204 (11) | 5640 (4) | 62 |
| C(14) | 11760 (12) | 3256 (11) | 6246 (4) | 45 (3) |
| C(15) | 10678 (12) | 2111 (11) | 6437 (4) | 40 (3) |
| N(16) | 10521 (12) | 2074 (9) | 7051 (4) | 49 |
| C(17) | 9548 (14) | 2700 (13) | 7977 (5) | 44 (3) |
| F(171) | 8205 (7) | 2371 (7) | 8210 (2) | 71 |
| F(172) | 10053 (8) | 3876 (6) | 8218 (2) | 65 |
| F(173) | 10575 (9) | 1707 (7) | 8129 (2) | 82 |
| C(18) | 5319 (15) | 5846 (13) | 7523 (5) | 53 (3) |
| F(181) | 3848 (8) | 5425 (8) | 7491 (3) | 86 |
| F(182) | 5686 (8) | 5846 (8) | 8068 (3) | 86 |
| F(183) | 5336 (8) | 7187 (7) | 7362 (3) | 96 |
| C(19) | 6739 (17) | 6315 (14) | 4738 (6) | 65 (4) |
| F(191) | 7086 (10) | 5906 (8) | 4229 (3) | 107 |
| F(192) | 6659 (10) | 7705 (7) | 4724 (3) | 106 |
| F(193) | 5255 (10) | 5921 (10) | 4832 (3) | 123 |
| C(20) | 11493 (16) | 3656 (14) | 4649 (5) | 55 (3) |
| F(201) | 12818 (9) | 3021 (9) | 4595 (3) | 105 |
| F(202) | 10455 (9) | 2865 (8) | 4371 (3) | 91 |
| F(203) | 11574 (9) | 4839 (7) | 4373 (3) | 84 |

* $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}\mathbf{a}_i.\mathbf{a}_j a_i^* a_j^*$.

Table 2. *Selected bond lengths* (Å) *and angles* (°)

| | | | |
|---|---|---|---|
| C(1)—N(2) | 1·28 (1) | C(1)—N(16) | 1·33 (1) |
| C(1)—C(17) | 1·54 (2) | N(2)—N(3) | 1·41 (1) |
| N(3)—C(4) | 1·27 (1) | C(4)—N(5) | 1·35 (1) |
| C(4)—C(18) | 1·50 (2) | N(5)—C(6) | 1·47 (1) |
| C(6)—C(7) | 1·48 (1) | C(7)—N(8) | 1·48 (1) |
| N(8)—C(9) | 1·33 (1) | C(9)—N(10) | 1·29 (1) |
| C(9)—C(19) | 1·48 (2) | N(10)—N(11) | 1·41 (1) |
| N(11)—C(12) | 1·26 (1) | C(12)—N(13) | 1·40 (1) |
| C(12)—C(20) | 1·52 (2) | N(13)—C(14) | 1·44 (1) |
| C(14)—C(15) | 1·50 (1) | C(15)—N(16) | 1·45 (1) |
| C(17)—F(171) | 1·31 (1) | C(17)—F(172) | 1·33 (1) |
| C(17)—F(173) | 1·34 (1) | C(18)—F(181) | 1·33 (1) |
| C(18)—F(182) | 1·32 (1) | C(18)—F(183) | 1·33 (1) |
| C(19)—F(191) | 1·29 (1) | C(19)—F(192) | 1·32 (1) |
| C(19)—F(193) | 1·35 (1) | C(20)—F(201) | 1·29 (1) |
| C(20)—F(202) | 1·34 (1) | C(20)—F(203) | 1·30 (1) |
| N(16)—C(1)—N(2) | 122·6 (9) | N(3)—N(2)—C(1) | 113·2 (8) |
| C(4)—N(3)—N(2) | 109·7 (8) | N(5)—C(4)—N(3) | 128·6 (10) |
| C(6)—N(5)—C(4) | 127·7 (9) | C(7)—C(6)—N(5) | 114·6 (9) |
| N(8)—C(7)—C(6) | 113·0 (9) | C(9)—N(8)—C(7) | 128·9 (9) |
| N(10)—C(9)—N(8) | 127·9 (10) | N(11)—N(10)—C(9) | 110·1 (8) |
| C(12)—N(11)—N(10) | 112·2 (8) | N(13)—C(12)—N(11) | 121·2 (10) |
| C(14)—N(13)—C(12) | 120·2 (9) | C(15)—C(14)—N(13) | 111·8 (9) |
| N(16)—C(15)—C(14) | 111·9 (9) | C(15)—N(16)—C(1) | 122·2 (9) |

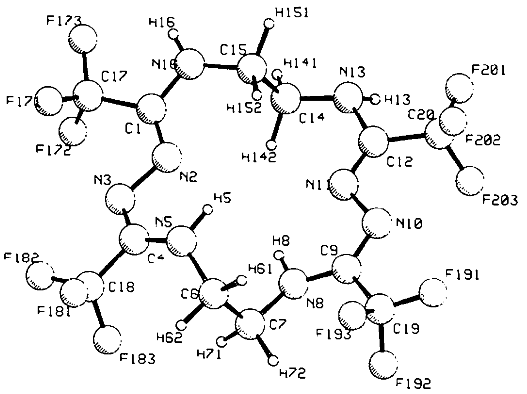

Fig. 1. The title molecule including atomic labelling scheme produced using *PLUTO* (Motherwell & Clegg, 1978).

Table 1 lists coordinates of the non-H atoms and Table 2 selected bond lengths and angles. Fig. 1 shows a view of the molecule with the atomic labelling scheme.

**Related literature.** For further preparative and chemical information see Barlow, Bell, O-Reilly & Tipping (1983).

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* Lists of structure factors, H-atom coordinates, anisotropic vibrational parameters and a full list of bond lengths, bond angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44045 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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