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The Crystal Structure of 4-Amino-2,2,5,5-tetrakis(trifluoromethyl)-3-imidazoline*

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Crystals of 4-amino-2,2,5,5-tetrakis(trifluoromethyl)-3-imidazoline, $C_7N_3F_{12}H_3$, are monoclinic, space group $C2/c$, with $a=24{\cdot}812$ (24), $b=6{\cdot}598$ (8), $c=16{\cdot}076$ (18) Å, and $\beta=118{\cdot}51$ (4)°. The data were collected on a diffractometer at room temperature; structure parameters were refined by least-squares methods to R of 0·067 for 1716 reflections. The molecule has no space-group imposed symmetry, but nearly has $C_s(m)$ point symmetry, the mirror plane being the plane of the imidazoline ring. Molecules are associated to form dimers through N–H···N hydrogen bonds involving amino hydrogens and the unprotonated imidazoline nitrogens of adjacent molecules. A short external C–NH$_2$ distance of 1·315 (4) Å indicates a substantial resonance contribution from a $>C=N^+H_2$ form.

Introduction

The chemistry of (polyfluoroalkyl)imidazolines has been pursued because of their pronounced stability and physiological activity (Middleton, Gale, Wiley & Krespan, 1970; Middleton & Krespan, 1970; Middleton, Metzger, Cunningham & Krespan, 1970). The compound 4-amino-2,2,5,5-tetrakis(trifluoromethyl)-3-imidazoline (midaflur, EXP 338) is active as a central nervous system depressant and muscle relaxant (Levine, Jossmann, Friend & De Anglis, 1968; Claghorn & Schoolar, 1968; Clark, Lynes, Price, Smith, Woodward, Marvel & Vernier, 1971). The crystal structure was determined to establish the tautomeric form of the imidazoline system and, the related feature of interest, the nature of the hydrogen bonding.

Experimental

Crystals suitable for X-ray work were obtained from Middleton & Krespan (1968). The systematic absences as observed on Weissenberg and precession photographs indicated that the space group is $C2/c$ or Cc. The correct space group, as confirmed by the structure refinement, is $C2/c$. The cell parameters were averaged from back-reflection-Weissenberg and diffractometer-determined values. The cell data are summarized in Table 1.

Table 1. *Crystal data*

$C_7N_3F_{12}H_3$
Space group: Monoclinic $C2/c$
$a=24{\cdot}812$ (24) Å
$b=6{\cdot}598$ (8)
$c=16{\cdot}076$ (18)
$\beta=118{\cdot}51$ (4)°
$Z=8$
$\varrho(\text{calc})=2{\cdot}05$ g cm^{-3}
$\varrho(\text{obs})=2{\cdot}05$ g cm^{-3}
$\mu(\text{Cu }K\alpha)=25{\cdot}5$ cm^{-1}

Data collection

A prismatic crystal of dimensions 0·22 × 0·45 × 0·18 mm was used in the data collection. Intensity data were measured on a Picker four-circle automatic diffractometer using the θ–2θ scan technique (1°/min scan speed). The crystal was mounted with the b axis along the diffractometer φ axis. Copper radiation (Cu $K\alpha=1{\cdot}5418$ Å) was used with a Ni filter. The scan range for each reflection was 2·0° plus the $K\alpha_1$–$K\alpha_2$ separation; backgrounds of 40 sec were measured before and after each scan. The data were measured in two sets, from 0 to 90° 2θ and then from 90 to 125° 2θ. A total of 1907 reflections were measured including $hk0$ and $\bar{h}k0$ data which were averaged.

The data were corrected for Lorentz and polarization effects in the usual way. The standard reflections measured showed a steady decrease with time; a crystal decomposition correction was made accordingly. The maximum decomposition observed was 5% in F_{obs}. The data were corrected for absorption (Prewitt, 1968) with the crystal defined by seven plane faces. The minimum and maximum calculated transmission factors were 0·52 and 0·66 respectively. The structure-factor errors were estimated according to a scheme reported earlier (Guggenberger, 1968). Those data with $F<\sigma(F)$ were given zero weight in the refinement.

Structure solution and refinement

The structure was solved using the symbolic addition procedure and the Bednowitz (1968) computer program. The Hauptman & Karle (1959) procedure for generally treating centered cells was followed. The cell was transformed to a primitive cell, the phase problem was solved, and then the coordinates were transformed back to the conventional centered cell. The transformation matrix used to obtain the primitive cell was

$$T=\begin{pmatrix} \frac{1}{2} & \frac{1}{2} & 0 \\ -\frac{1}{2} & \frac{1}{2} & 0 \\ 0 & 0 & 1 \end{pmatrix}.$$

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The coordinates transform as the transpose of the inverse of the transformation matrix.

The distribution of E's followed the expected centric distribution; the observed values of 0·80 for $\langle|E|\rangle$, 1·00 for $\langle|E|^2\rangle$, and 0·99 for $\langle|E^2-1|\rangle$, are essentially identical to the theoretical values of 0·798, 1·00, and 0·968 respectively (Karle, Hauptman & Christ, 1958). The 228 largest E's (E's > 1·50) were used in the phase determining process. Seven reflections were assigned symbolic phases and two were used for origin assignment ($\overline{10},12,7$ and $\overline{7},10,8$). The correct phases for the symbols were found by inspection. All the non-hydrogen atoms were found on the resultant E map.

The model was refined stepwise giving R $(=\sum||F_o|-|F_c||/\sum|F_o|)$ values of 0·215 for all atoms with isotropic temperature factors, 0·101 for the fluorine atoms only with anisotropic temperature factors, and 0·083 for all atoms with anisotropic temperature factors. At this point an electron-density difference map was calculated through the plane of the imidazoline ring to look for the hydrogen atoms. All three hydrogen-atom positions were clearly evident; the electron densities were 0·5 for H(1), 0·4 for H(2), and 0·3 e Å^{-3} for H(3). The hydrogen atoms were included in the refinement with isotropic temperature factors. A secondary extinction correction was also applied since the largest observed F's were systematically smaller than the calculated F's. The first-order correction was applied after the method of Zachariasen (1963) according to $F_{\rm calc}$(corrected) $= F_{\rm calc}$(uncorrected)$/(1+\beta C I_{\rm obs})$; β was set equal to 1·0 for all reflections and C was varied in the refinement.

Table 3. *Observed and calculated structure factors*

The columns give the running index h, $10F_o$, and $10F_c$. An asterisk marks an 'unobserved' reflection (see text).

Table 2. *Final coordinates and thermal parameters*

Standard deviations are given in parentheses. Anisotropic temperature factor has the form $\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)]$. Data are $\times 10^5$. The secondary extinction parameter C = 5·1 (4) × 10^{-6}.

	x	y	z	β_{11} or B(Å^2)	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
N(1)	6276 (14)	48878 (55)	−3894 (20)	210 (7)	3988 (110)	441 (16)	340 (23)	182 (9)	559 (33)
N(2)	6226 (11)	34894 (43)	9200 (17)	154 (5)	2627 (77)	373 (13)	174 (17)	111 (7)	243 (26)
N(3)	15703 (11)	19048 (45)	16231 (17)	147 (5)	2500 (76)	338 (13)	137 (16)	92 (7)	156 (26)
C(1)	8770 (13)	38324 (50)	4003 (20)	158 (6)	2309 (85)	331 (15)	136 (19)	113 (8)	108 (30)
C(2)	10189 (13)	22340 (51)	17004 (20)	160 (6)	2440 (86)	321 (15)	123 (19)	109 (8)	142 (29)
C(3)	15116 (13)	28687 (52)	7863 (20)	137 (6)	2485 (88)	336 (15)	67 (19)	96 (8)	38 (30)
C(4)	15393 (17)	13051 (62)	973 (25)	233 (9)	3276 (115)	543 (20)	148 (26)	197 (11)	−100 (40)
C(5)	20182 (15)	44965 (63)	10408 (26)	178 (8)	3368 (122)	582 (22)	−22 (24)	141 (10)	60 (41)
C(6)	7004 (17)	2335 (59)	16761 (25)	228 (8)	2985 (110)	508 (20)	−27 (25)	136 (10)	315 (39)
C(7)	11893 (18)	33607 (61)	26419 (24)	300 (10)	3304 (116)	416 (18)	163 (28)	211 (11)	35 (39)
F(1)	7132 (14)	37768 (58)	27479 (20)	415 (8)	7904 (146)	846 (18)	281 (29)	415 (11)	−497 (43)
F(2)	14721 (14)	50645 (39)	26978 (17)	538 (9)	3266 (72)	679 (15)	−180 (21)	338 (10)	−495 (27)
F(3)	15744 (14)	22804 (48)	33891 (15)	528 (9)	4756 (95)	317 (11)	309 (24)	82 (8)	166 (25)
F(4)	1896 (13)	5171 (51)	17485 (25)	380 (8)	5635 (118)	1474 (26)	−210 (24)	560 (13)	181 (44)
F(5)	5291 (13)	−7148 (42)	8729 (18)	404 (7)	4226 (85)	736 (16)	−570 (22)	243 (9)	−401 (31)
F(6)	10529 (14)	−9973 (44)	23577 (20)	442 (9)	3251 (77)	880 (18)	−42 (21)	84 (10)	800 (31)
F(7)	19503 (13)	59638 (41)	15542 (20)	415 (8)	3345 (74)	970 (19)	−412 (20)	341 (10)	−639 (31)
F(8)	25651 (9)	37107 (46)	15522 (20)	138 (5)	4936 (96)	1137 (20)	−64 (17)	86 (8)	278 (36)
F(9)	20027 (12)	53661 (44)	3032 (18)	346 (7)	4951 (97)	830 (17)	−207 (21)	345 (9)	370 (32)
F(10)	15010 (16)	21644 (48)	−6725 (18)	641 (11)	4922 (97)	626 (15)	198 (27)	479 (11)	47 (31)
F(11)	11037 (12)	−213 (43)	−1603 (19)	380 (7)	4395 (90)	891 (18)	−340 (21)	322 (10)	−1027 (34)
F(12)	20705 (12)	2891 (43)	5008 (20)	335 (7)	4663 (93)	998 (19)	547 (21)	273 (9)	−307 (35)
H(1)	797 (21)	4988 (66)	−779 (31)	3·9 (11)					
H(2)	227 (19)	5500 (57)	−598 (26)	2·1 (8)					
H(3)	1709 (27)	645 (88)	1930 (40)	6·6 (15)					

The final refinements were done in two sections: (*a*) the positional and thermal parameters for the carbon, nitrogen, and hydrogen atoms, and (*b*) the parameters for the fluorine atoms. The final R values for the 1716 reflections with $F < \sigma(F)$ are 0·067 for R and 0·087 for $wR\{=[\sum w(|F_o|-|F_c|)^2/\sum w|F_o|^2]^{1/2}\}$. The corresponding values for all the data (1827 reflections) are 0·071 for R and 0·087 for wR.

The scattering factors used were those for the neutral atoms (Hanson, Herman, Lea & Skillman, 1964). The function minimized in the refinement was $\sum w(|F_o|-|F_c|)^2$. The final parameters are given in Table 2. The observed and calculated structure factors are listed in Table 3.

Discussion

An *ORTEP* plot (Johnson, 1965) of the molecular structure is shown in Fig. 1 illustrating the numbering system used here. The molecule exists as the amino tautomer rather than the imino tautomer. The molecule has no crystallographically imposed symmetry but nearly has $C_s(m)$ point symmetry, with the possible exception of the H(3) atom. The molecular mirror plane is the plane of the imidazoline ring. The thermal parameters of the non-fluorine atoms are normal. The thermal parameters of the fluorine atoms of the trifluoromethyl groups are understandably large, but the individual atom refinement appears to be adequate.

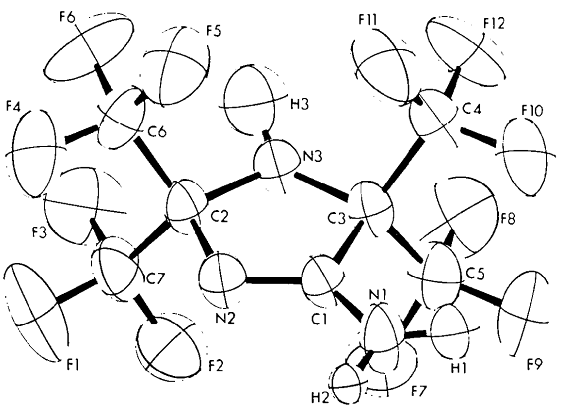


Fig. 1. Molecular structure illustrating the thermal ellipsoids and numbering system.

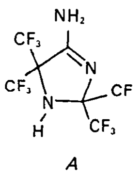
Table 4. *Interatomic distances*

Bonding distances (Å)			
N(1)–C(1)	1·315 (4)	C(7)–F(1)	1·301 (6)
C(1)–N(2)	1·285 (4)	C(7)–F(2)	1·305 (5)
N(2)–C(2)	1·432 (4)	C(7)–F(3)	1·330 (4)
C(2)–N(3)	1·448 (4)	C(6)–F(4)	1·340 (6)
N(3)–C(3)	1·431 (4)	C(6)–F(5)	1·310 (5)
C(3)–C(1)	1·528 (4)	C(6)–F(6)	1·308 (4)
		C(5)–F(7)	1·334 (5)
C(3)–C(4)	1·539 (5)	C(5)–F(8)	1·312 (4)
C(3)–C(5)	1·552 (5)	C(5)–F(9)	1·301 (5)
C(2)–C(6)	1·529 (5)	C(4)–F(10)	1·323 (5)
C(2)–C(7)	1·553 (5)	C(4)–F(11)	1·295 (5)
Av.	1·543 (6)	C(4)–F(12)	1·338 (5)
		Av.	1·316 (5)
N(1)–H(1)	0·91 (6)		
N(1)–H(2)	0·97 (4)		
N(3)–H(3)	0·94 (6)		
Av.	0·94 (2)		
Nonbonding distances (Å)			
C(4)–C(5)	2·535 (6)	N(3)–F(2)	2·791 (4)
C(6)–C(7)	2·518 (6)	N(3)–F(3)	2·845 (4)
H(1)–H(2)	1·61 (8)	N(3)–F(5)	2·853 (4)
N(1)–F(9)	3·058 (4)	N(3)–F(6)	2·856 (5)
N(1)–F(10)	3·012 (5)	N(3)–F(7)	2·859 (4)
C(1)–F(7)	2·792 (4)	N(3)–F(8)	2·792 (4)
C(1)–F(11)	2·842 (5)	N(3)–F(11)	2·830 (4)
N(2)–F(1)	2·844 (5)	N(3)–F(12)	2·841 (5)
N(2)–F(2)	2·818 (3)	C(4)–F(8)	2·964 (4)
N(2)–F(4)	2·853 (5)	C(4)–F(9)	2·872 (5)
N(2)–F(5)	2·782 (4)	C(5)–F(10)	2·868 (5)
F(1)–F(4)	2·627 (5)	C(5)–F(12)	2·930 (5)
F(2)–F(7)	2·687 (5)	C(6)–F(1)	2·895 (6)
F(3)–F(6)	2·656 (4)	C(6)–F(3)	2·900 (4)
F(5)–F(11)	2·695 (5)	C(7)–F(4)	2·887 (5)
F(8)–F(12)	2·735 (4)	C(7)–F(6)	2·906 (5)
F(9)–F(10)	2·571 (4)		

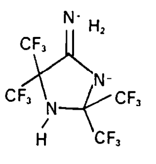
Table 5. *Interatomic angles* (°)

N(1)–C(1)–N(2)	124·8 (3)	C(2)—C(7)–F(1)	112·8 (3)
N(1)–C(1)–C(3)	122·1 (3)	C(2)—C(7)–F(2)	111·5 (3)
N(2)–C(1)–C(3)	113·2 (3)	C(2)—C(7)–F(3)	111·6 (3)
C(1)–N(2)–C(2)	109·1 (2)	C(2)—C(6)–F(4)	112·1 (3)
N(2)–C(2)–N(3)	107·1 (2)	C(2)—C(6)–F(5)	111·0 (3)
C(2)–N(3)–C(3)	109·7 (2)	C(2)—C(6)–F(6)	112·6 (3)
N(3)–C(3)–C(1)	100·9 (2)	C(3)—C(5)–F(7)	109·9 (2)
		C(3)—C(5)–F(8)	110·9 (3)
N(2)–C(2)–C(6)	110·3 (3)	C(3)—C(5)–F(9)	113·4 (3)
N(2)–C(2)–C(7)	109·3 (3)	C(3)—C(4)–F(10)	112·2 (3)
N(3)–C(2)–C(6)	111·5 (3)	C(3)—C(4)–F(11)	111·4 (3)
N(3)–C(2)–C(7)	109·1 (3)	C(3)—C(4)–F(12)	111·0 (3)
C(6)–C(2)–C(7)	109·6 (3)	Av.	111·7 (3)
		F(1)—C(7)–F(2)	107·5 (3)
N(3)–C(3)–C(4)	111·0 (3)	F(1)—C(7)–F(3)	107·5 (3)
N(3)–C(3)–C(5)	110·7 (3)	F(2)—C(7)–F(3)	105·6 (3)
C(1)–C(3)–C(4)	112·2 (3)	F(4)—C(6)–F(5)	106·1 (3)
C(1)–C(3)–C(5)	111·5 (3)	F(4)—C(6)–F(6)	107·2 (3)
C(4)–C(3)–C(5)	110·2 (3)	F(5)—C(6)–F(6)	107·6 (3)
		F(7)—C(5)–F(8)	107·3 (3)
H(1)–N(1)–C(1)	123·0 (26)	F(7)—C(5)–F(9)	106·6 (3)
H(2)–N(1)–C(1)	119·1 (21)	F(8)—C(5)–F(9)	108·5 (3)
H(1)–N(1)–H(2)	117·8 (31)	F(10)–C(4)–F(11)	108·3 (3)
		F(10)–C(4)–F(12)	106·4 (3)
H(3)–N(3)–C(2)	102·2 (23)	F(11)–C(4)–F(12)	107·2 (3)
H(3)–N(3)–C(3)	136·9 (33)	Av.	107·1 (2)

The interatomic distances are given in Table 4 and the interatomic angles in Table 5. The two pertinent resonance hybrids for this structure are *A* and *B*.

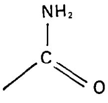


A

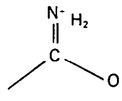


B

The external C(1)–N(1) distance of 1·315 (4) Å and internal C(1)–N(2) distance of 1·285 (4) Å suggest that both hybrids are nearly of equal importance in describing the structure. The external C–N distance is much more like the values found in amides rather than amines. This is not so surprising in view of the similarities between the geometry here and that found in amides (*C* and *D*).



C



D

The C–N distances in amines are frequently close to the single bond value of about 1·47 Å where the π delocalization is minimal, but distances intermediate between the value observed here and the single bond value are found, for example, 1·40 (2) in α-sulphanilamide (O'Connor & Maslen, 1965), 1·367 (17) Å in 2-amino-3-methylbenzoic acid (Brown & Marsh, 1963), and 1·35 Å in melamine (Hughes, 1941). The C–N distances in amides, however, are frequently much shorter because of large contributions from the *D* resonance form, for example, 1·28 Å in L-glutamine (Cochran & Penfold, 1952), and 1·300 (13) Å in formamide (Ladell & Post, 1954). The C(1)–N(2) distance of 1·285 (4) Å is only slightly less than the values frequently found for the C=N distance in imidazole rings; examples are 1·298 (7) Å in 1′-(4-imidazolylsulfonyl)-4′-phenylimidazole (Guggenberger, 1972), 1·312 (8) Å in purine (Watson, Sweet & Marsh, 1965), and 1·326 (5) Å in imidazole (Martinez-Carrera, 1966). The remaining distances and angles are acceptably self consistent according to type within the molecule and are in agreement with literature values. The internal angle at the protonated N(3) site is slightly larger than at N(2) [109·7 *vs.* 109·1 (2)°] consistent with other nitrogen containing ring systems (Singh, 1965; Sletten, Sletten & Jensen, 1968).

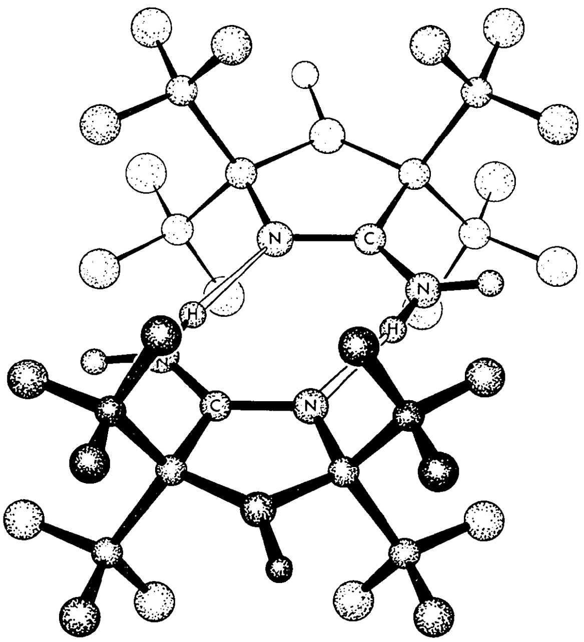
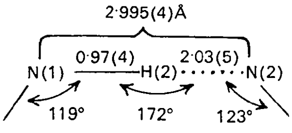


Fig. 2. One dimer unit illustrating the hydrogen bonding (hydrogen bonds are open).

The molecules are associated through N–H···N hydrogen bonds to form dimers. The bridging geometry is a six-membered ring with two opposite sides composed of N–H···N bridges. The hydrogen bonds are characterized by



and the dimer formed is illustrated in Fig. 2. The planarity of the dimeric unit and the presence of the hydrogen bonds tend to increase the contribution of the *B* resonance form.

The equation and the atomic deviations from the molecular plane are given in Table 6. The molecule is acceptably planar and H(1) and H(2) are nearly in the plane as required by resonance form *B*. The C(3)–C(4)–C(5) and C(2)–C(6)–C(7) planes are 89·8 and 90·8°, respectively, from the molecular plane (Table 6). The deviation of H(3) from the plane may mean that H(3) is slightly misplaced in the refinement since the deviation is not apparent from consideration of nearest neighbor distances. The H(3) atom would be expected to be more nearly in the center of the symmetrically placed F(3), F(6), F(8), and F(12) atoms. There is a short intermolecular contact of 2·58 Å involving H(3) and F(8) (at $\frac{1}{2}-x, -\frac{1}{2}+y, \frac{1}{2}-z$) but H(3) does not appear to be optimized with respect to F(8) for either a bonding or non-bonding interaction. Also, the refined position of H(3) leads to short intramolecular contacts [H(3)–F(6) is 2·32 (7) Å].

Table 6. *Deviations* (Å) *from molecular least-squares plane*

Atoms with asterisks were used in the plane calculation. Primed atoms are in the related ring in the dimer.

Plane: $0\cdot1834X+0\cdot8291Y+0\cdot5282Z-2\cdot733=0$

Atomic deviations from plane (Å)

N(1)*	−0·009	H(1)	−0·113
N(2)*	0·016	H(2)	0·017
N(3)*	0·006	H(3)	−0·434
C(1)*	0·005	N(1′)	0·014
C(2)*	−0·018	N(2′)	−0·012
C(3)*	0·000	N(3′)	−0·002
C(4)	−1·260	C(1′)	−0·001
C(5)	1·275	C(2′)	0·022
C(6)	−1·272	C(3′)	0·004
C(7)	1·246	H(2′)	−0·013

Dihedral angles (°)

Molecular plane C(3), C(4), C(5)	89·8	Molecular plane C(2), C(6), C(7)	90·8

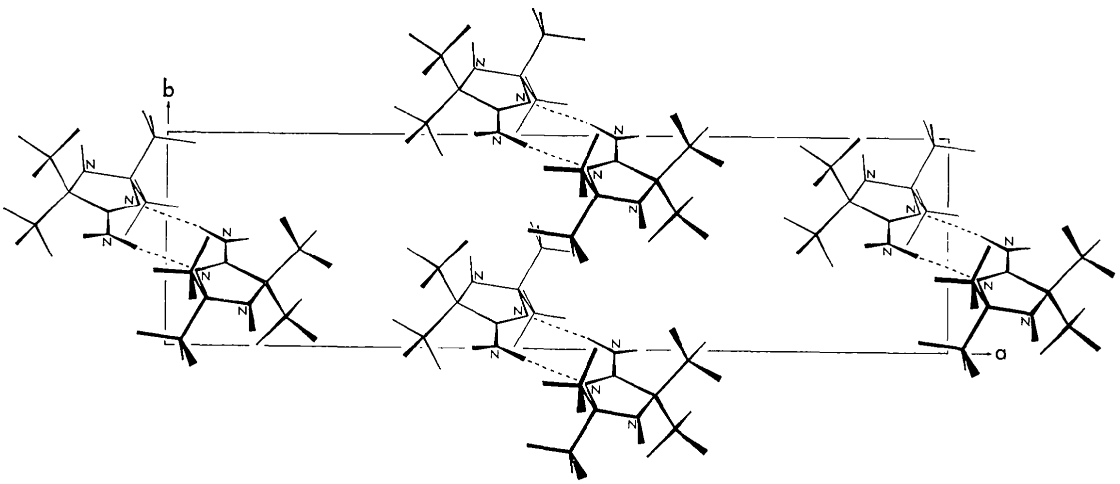


Fig. 3. One layer of the crystal packing (*xy* projection).

The packing of the structure in one layer is shown in Fig. 3. This layer shows one unique dimeric unit and the dimer translated by the cell-centering operation, and these repeated by the unit-cell translations. There is another layer in the cell with similar packing with $\frac{1}{2}c$ translation. All the intermolecular contacts were examined; they are of the normal van der Waals type. The shortest contacts are 2·755 Å between non-hydrogen atoms (F···F), 2·73 Å between hydrogen atoms, and 2·42 Å for the hydrogen–non-hydrogen contacts, the latter between H(1) and F(1) on a neighboring molecule.

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