Table 2. S	elected geom	ietric parameters	(A, °)
N1-C5	1.343 (3)	C9-C10	1.384 (4)
N1C6	1.455 (3)	C10-C11	1.388 (4)
D1C5	1.354 (3)	C11-C12	1.376 (5)
01—C1	1.477 (3)	C12—C13	1.371 (4)
02—C5	1.214 (3)	C13-C14	1.391 (4)
03—C7	1.202 (3)	C15-C16	1.506 (3)
O4—C7	1.339 (3)	C16C17	1.476 (3)
O4—C8	1.444 (3)	C17-C18	1.333 (3)
O5—C16	1.218 (3)	C18—C19	1.468 (3)
C1—C3	1.512 (3)	C19-C20	1.399 (3)
C1-C2	1.517 (3)	C19—C24	1.402 (3)
C1-C4	1.524 (3)	C20-C21	1.386 (3)
C6—C7	1.518 (3)	C21-C22	1.386 (4)
C6—C15	1.526 (3)	C22-C23	1.382 (4)
C8—C9	1.505 (3)	C23C24	1.385 (3)
C9—C14	1.382 (3)		
C5—N1—C6	120.7 (2)	C10-C9-C8	117.9 (2)
C5-01-C1	119.8 (2)	C9-C10-C11	121.1 (3)
C7-04-C8	116.8 (2)	C12-C11-C10	119.8 (3)
01-C1-C3	102.7 (2)	C13-C12-C11	119.7 (3)
01-C1-C2	110.5 (2)	C12-C13-C14	120.5 (3)
C3-C1-C2	110.3 (2)	C9-C14-C13	120.4 (3)
O1-C1-C4	110.1 (2)	C16-C15-C6	113.0 (2)
C3-C1-C4	110.5 (2)	O5-C16-C17	119.6 (2)
C2-C1-C4	112.4 (2)	O5-C16-C15	120.8 (2)
O2-C5-N1	124.7 (2)	C17-C16-C15	119.6 (2)
02-C5-01	125.4 (2)	C18-C17-C16	125.5 (2)
N1-C5-01	109.9 (2)	C17—C18—C19	125.6 (2)
N1-C6-C7	108.2 (2)	C20-C19-C24	118.0 (2
N1-C6-C15	113.5 (2)	C20-C19-C18	122.5 (2)
C7-C6-C15	114.3 (2)	C24—C19—C18	119.5 (2)
O3—C7—O4	125.2 (2)	C21-C20-C19	120.9 (2
O3-C7-C6	124.5 (2)	C22-C21-C20	120.5 (2
O4-C7-C6	110.2 (2)	C23-C22-C21	119.2 (2
O4—C8—C9	112.0 (2)	C22-C23-C24	120.9 (2
C14-C9-C10	118.5 (2)	C23-C24-C19	120.5 (2
C14-C9-C8	123.6 (2)		

1 01

Isotropic H atoms were constrained to give N-H 0.90 and C-H 0.95-1.00 Å depending on position, H-C-H 109.5°, aromatic and olefinic H atoms on angle external bisectors and U(H)= $1.2U_{eq}(C)$. Data collection and cell refinement: DIF4 (Stoe & Cie, 1992). Data reduction: local programs. Program(s) used to solve structure: SHELXTL/PC (Sheldrick, 1990). Program(s) used to refine structure: SHELXL (Sheldrick, 1994). Molecular graphics: SHELXTL/PC. Software used to prepare material for publication: SHELXL and local programs.

We thank SERC and Pfizer Central Research for financial support.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: HA1079). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Clegg, W. (1981). Acta Cryst. A37, 22-28.

- Jackson, R. F. W., Wishart, N., Wood, A., James, K. & Wythes, M. J. (1992), J. Org. Chem. 57, 3397-3404.
- Sheldrick, G. M. (1990). SHELXTL/PC User's Manual. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1994). J. Appl. Cryst. In preparation.
- Stoe & Cie (1992). DIF4. Diffractometer Control Program. Version 7.04. Stoe & Cie, Darmstadt, Germany.
- Williams, R. M. (1989). In Synthesis of Optically Active α -Amino Acids. Oxford: Pergamon.

©1994 International Union of Crystallography Printed in Great Britain - all rights reserved

Acta Cryst. (1994). C50, 967-971

p-Methyl-N-(pentafluorobenzylidene)aniline (1), 1,2,3,4-Tetrafluoro-7-methoxyacridine (2), 1,2,3,4,7-Pentafluoroacridine (3) and 3-(p-Methylanilino)-1,2,4-trifluoro-7-methylacridine (4): Four Molecules **Representing Key Stages in the One-Pot** Synthesis of 1,2,3,4-Tetrafluoroacridines by Treating Pentafluorobenzaldehyde with para-Substituted Anilines

A. J. ADAMSON, Y. ARCHAMBEAU, R. E. BANKS AND B. BEAGLEY

Department of Chemistry, University of Manchester Institute of Science and Technology, PO Box 88, Manchester M60 1QD, England

M. HELLIWELL

Department of Chemistry, University of Manchester, Manchester M13 9PL, England

R. G. PRITCHARD AND A. E. TIPPING

Department of Chemistry, University of Manchester Institute of Science and Technology, PO Box 88, Manchester M60 1QD, England

(Received 3 August 1993; accepted 4 January 1994)

Abstract

All of the title compounds, $C_{14}H_8F_5N$, $C_{14}H_7F_4NO$, $C_{13}H_4F_5N$ and $C_{21}H_{15}F_3N_2$, display a characteristic stacking of their π systems in which adjacent members of the stack are related by crystallographic inversion centres. This leads to a particularly efficient mode of packing in which fluorinated rings overlap unfluorinated sections of the molecules at interplanar spacings ranging from 3.40 to 3.51 Å.

Comment

During the purification of a crude sample of the Schiff base $C_6F_5CH = NC_6H_4(OCH_3-4)$ [(1) with $R = OCH_3$] obtained by heating pentafluorobenzaldehyde with pmethoxyaniline in boiling *n*-butyl acetate, Flowers & DeFigueredo (1990) isolated a by-product which they concluded was 7,8,9,10-tetrafluoro-2-methoxyphenanthridine on the basis of the results of elemental, NMR and mass spectometric analyses. Their interest centred only on the preparation of Schiff bases of the type C₆F₅CH=NAr for mass spectral studies. For our part we wished to compare the mechanism of this intramolecular ring closure with that involved in the production of fluorinated diareno-1,2-diazepines from phenylazo derivatives of fluoroaromatic compounds (Alty, Banks, Fishwick, Pritchard & Thompson, 1984; Alty, Banks, Fishwick & Thompson, 1985).

We prepared the compound thought to be the phenanthridine, but found that the ¹H and ¹⁹F NMR signals could not be assigned unambiguously. The crystal structure determination, described here, showed that the by-product from the reaction of C_6F_5 CHO with $H_2NC_6H_4(OCH_3-4)$ was in fact 1,2,3,4-tetrafluoro-7-methoxyacridine (2).

Subsequent detailed work on the mechanism of formation of various tetrafluoroacridines [*e.g.* (3)] from pentafluorobenzaldehyde and a range of substituted anilines showed that, rather than the simple ring closure expected, another aniline molecule attacks the *ortho*position in the fluorinated ring, initiating a process which ultimately yields substituted acridines. Full details of the reaction mechanism have been presented by Adamson, Banks & Tipping (1993) and are summarized in the scheme below. Further evidence for the role of the aniline component in the ring closure is given by the formation of compound (4), in which the *para*-F substituent has also been replaced by *p*-methylaniline.





Fig. 1. Molecule (1) including atomic labelling scheme.



Fig. 2. Molecule (2) including atomic labelling scheme [an identical labelling scheme has been used for molecule (3)].



Fig. 3. Crystal packing in (2) showing the type of overlapping π systems seen in all four crystal structures.



Fig. 4. Molecule (4) including atomic labelling scheme.

In connection with the mechanism of acridine formation, the determination of the structure of (1) establishes that Schiff bases of the type $C_6F_5CH=NC_6H_4R$ -4 prepared in this manner adopt an *E* configuration at the C=N bond.

Experimental

The Schiff base [(1) m.p. 412 K] was obtained in 93% yield (after recrystallization from boiling acetone) by stirring penta-fluorobenzaldehyde and p-methylaniline together (1:1 molar

ratio) in dichloromethane at room temperature for approximately 1 h. The fluoroacridines were isolated from reaction mixtures produced by heating pentafluorobenzaldehyde with p-methoxyaniline [(2), m.p. 484 K], p-fluoroaniline [(3), m.p. 438 K] or p-methylaniline [(4), m.p. 512 K] in an inert solvent (n-butyl acetate or, preferentially, toluene or 1,2dichlorobenzene) (Adamson, Banks & Tipping, 1993). All three acridines were sublimed in vacuo then recrystallized from acetone; each one provided a satisfactory elemental analysis (C, H, F, N).

Mo $K\alpha$ radiation

 $\theta = 23.93 - 37.10^{\circ}$

 $\mu = 0.14 \text{ mm}^{-1}$

T = 296 K

Colourless

 $R_{\rm int} = 0.034$

 $\theta_{\rm max} = 25.0^{\circ}$

 $k = -15 \rightarrow 15$

3 standard reflections

reflections

monitored every 150

intensity variation: 0.03%

 $l = -7 \rightarrow 7$

 $h = 0 \rightarrow 8$

Needle

Cell parameters from 25 reflections

 $0.40 \times 0.20 \times 0.10$ mm

 $\lambda = 0.71069 \text{ Å}$

Compound (1)

Crystal data

C14H8F5N $M_r = 285.22$ Triclinic $P\overline{1}$ a = 7.437 (3) Å *b* = 13.176 (4) Å c = 6.210 (2) Å $\alpha = 92.00 (3)^{\circ}$ $\beta = 96.90 (3)^{\circ}$ $\gamma = 91.76 (3)^{\circ}$ V = 603.4 (7) Å³ Z = 2 $D_{\rm x} = 1.570 {\rm Mg} {\rm m}^{-3}$

Data collection AFC-6S diffractometer $\omega/2\theta$ scans Absorption correction: empirical $T_{\rm min} = 0.88, T_{\rm max} = 1.00$ 2308 measured reflections 2126 independent reflections 793 observed reflections $[I > 3\sigma(I)]$

Refinement

Refinement on F	Weighting scheme b
R = 0.042	measured e.s.d.'s
wR = 0.046	$(\Delta/\sigma)_{\rm max} = 0.10$
<i>S</i> = 1.75	$\Delta \rho_{\rm max} = 0.15 \ {\rm e} \ {\rm \AA}^{-3}$
793 reflections	$\Delta \rho_{\rm min}$ = -0.21 e Å
201 parameters	Atomic scattering fa
All H-atom parameters	from Internationa
refined except for methyl	for X-ray Crystall
H atoms, fixed	(1974, Vol. IV)

Weighting scheme based on
measured e.s.d.'s
$(\Delta/\sigma)_{\rm max} = 0.10$
$\Delta \rho_{\rm max} = 0.15 \ {\rm e} \ {\rm \AA}^{-3}$
$\Delta \rho_{\rm min} = -0.21 \ {\rm e} \ {\rm \AA}^{-3}$
Atomic scattering factors
from International Tables
for X-ray Crystallography
(1974, Vol. IV)

ag scheme based on red e.s.d.'s $_x = 0.10$ $0.15 \text{ e} \text{ Å}^{-3}$ $-0.21 \text{ e} \text{ Å}^{-3}$ scattering factors International Tables ray Crystallography	Data collection AFC-5S diffrac $\omega/2\theta$ scans Absorption cor- empirical $T_{min} = 0.70$, 1827 measured
ray Crystallography	1676 independ

AFC-5S diffractometer
$\omega/2\theta$ scans
Absorption correction:
empirical
$T_{\rm min} = 0.70, \ T_{\rm max} = 1.00$
1827 measured reflections
1676 independent reflections
1167 observed reflections

 $[I > 3\sigma(I)]$

Refinement

refined

Refinement on FR = 0.078wR = 0.088S = 3.241167 reflections 181 parameters H-atom parameters not

	•			
	x	v	z	U_{eq}
F10	0.6136 (4)	0.6027 (2)	0.0214 (4)	0.065 (2)
F11	0.6590 (5)	0.8003 (2)	-0.0592 (5)	0.077 (2)
F12	0.8344 (5)	0.9295 (2)	0.2493 (6)	0.085 (2)
F13	0.9644 (4)	0.8603 (2)	0.6410 (5)	0.071 (2)
F14	0.9229 (4)	0.6655 (2)	0.7259 (4)	0.060 (2)
N7	0.7687 (6)	0.4803 (3)	0.5915 (7)	0.060 (3)
CI	0.7385 (7)	0.3744 (4)	0.6225 (9)	0.048 (3)
C2	0.7954 (8)	0.3409 (4)	0.8286 (9)	0.049 (3)
C3	0.7748 (8)	0.2401 (5)	0.8742 (9)	0.055 (3)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters $(Å^2)$ for (1)

 $U_{\text{eq}} = (1/3) \sum_{i} \sum_{j} U_{ij} a_i^* a_i^* \mathbf{a}_i . \mathbf{a}_j.$

C4	0.6950 (7)	0.1705 (4)	0.7182 (9)	0.051 (3)
C5	0.6371 (8)	0.2039 (5)	0.5171 (10)	0.058 (3)
C6	0.6570 (8)	0.3042 (4)	0.4644 (10)	0.056 (3)
C8	0.7336 (7)	0.5175 (4)	0.4128 (10)	0.048 (3)
C9	0.7659 (7)	0.6261 (4)	0.3783 (9)	0.044 (3)
C10	0.7019 (7)	0.6651 (4)	0.1797 (9)	0.048 (3)
C11	0.7236 (8)	0.7662 (5)	0.1346 (9)	0.054 (3)
C12	0.8120 (8)	0.8308 (4)	0.2907 (9)	0.055 (3)
C13	0.8761 (8)	0.7958 (4)	0.4891 (9)	0.052 (3)
C14	0.8552 (7)	0.6957 (4)	0.5313 (8)	0.044 (3)
C15	0.6717 (9)	0.0599 (4)	0.7710 (10)	0.079 (3)

Table 2. Selected geometric parameters (Å, °) for (1)

1.431 (6)	C4-C15	1.514 (7)
1.232 (6)	C8-C9	1.471 (6)
1.390 (7)	C9-C10	1.389 (6)
1.395 (7)	C9-C14	1.391 (7)
1.378 (7)	C12—C13	1.368 (7)
1.364 (7)	C13—C14	1.360 (7)
121.8 (5)	C3-C4-C5	118.4 (6)
116.0 (5)	N7-C8-C9	122.3 (6)
125.3 (5)	C11-C12-C13	120.3 (6)
118.7 (6)		
	1.431 (6) 1.232 (6) 1.390 (7) 1.395 (7) 1.378 (7) 1.364 (7) 121.8 (5) 116.0 (5) 125.3 (5) 118.7 (6)	

Compound (2)

Crystal data C₁₄H₇F₄NO $M_r = 281.21$ Triclinic PΪ a = 7.366 (7) Åb = 12.411 (2) Å c = 6.312 (2) Å $\alpha = 100.13 (2)^{\circ}$ $\beta = 94.28 (5)^{\circ}$ $\gamma = 86.69 (4)^{\circ}$ $V = 565.9 (5) \text{ Å}^3$ Z = 2 $D_{\rm r} = 1.650 {\rm Mg m}^{-3}$

Cu $K\alpha$ radiation $\lambda = 1.5418 \text{ Å}$ Cell parameters from 18 reflections $\theta = 76.90 - 79.37^{\circ}$ $\mu = 1.29 \text{ mm}^{-1}$ T = 295 KPlate $0.60 \times 0.40 \times 0.03~\text{mm}$ Yellow

 $R_{\rm int} = 0.048$ $\theta_{\rm max} = 60^{\circ}$ $h = -4 \rightarrow 8$ $k = -13 \rightarrow 13$ $l = -7 \rightarrow 7$ 3 standard reflections monitored every 150 reflections intensity variation: 4.56%

Weighting scheme based on
measured e.s.d.'s
$(\Delta/\sigma)_{\rm max} = 0.0001$
$\Delta \rho_{\rm max} = 0.34 \ {\rm e} \ {\rm \AA}^{-3}$
$\Delta \rho_{\rm min} = -0.33 \ {\rm e} \ {\rm \AA}^{-3}$
Atomic scattering factors
from International Tables
for X-ray Crystallography
(1974, Vol. IV)

 Table 3. Fractional atomic coordinates and equivalent
 Refinement isotropic displacement parameters $(Å^2)$ for (2) Refinement on F

	- -	$(1/2)\sum \sum$	777.****			R = 0.045		
	U_{eq}	$= (1/3) \Sigma_i Z_i$	$a_j U_{ij} a_i^* a_j^* \mathbf{a}_i$. a _j .		wR = 0.052	3	
	x	У		z	U_{eq}	S = 2.15		
F1	0.5355 (4)	0.1683	(3) 0.	0975 (4)	0.067 (2)	951 reflect	ions	
F2	0.5470 (5)	0.3790	(3) 0.	2984 (5)	0.080 (2)	188 param	eters	
F3 E4	0.0969 (5)	0.4292	(3) U. (3) D.	/03/(5)	0.082(2)	All H-aton	n narameter	2
F4 07	0.8312 (4)	-0.3828	(3) 0.	3828 (6)	0.076(2)	All II-atoli	ii parameter	5
N10	0.8233 (6)	0.0552	(3) 0.7	7362 (6)	0.049 (2)	renned		
CI	0.6116 (7)	0.1935	(4) 0.	3017 (8)	0.053 (3)			
C2	0.6140 (8)	0.2971	(5) 0.	3975 (9)	0.057 (3)			
C3	0.6929 (8)	0.3235	(5) 0.	6122 (9)	0.061 (3)	Table 5.	Fractional	4
C4	0.7574 (7)	0.2430	(5) 0.	7171 (8)	0.055 (3)	isotr	onic displa	ı
C4a	0.7561(7)	0.1319	(4) U. (5) O	0229 (8) 7584 (8)	0.047(2)	10011	opic andpia	
C6	0.8931 (8)	-0.2386	(5) 0.	6672 (9)	0.058(3)		U _{eq} =	-
C7	0.8241 (7)	-0.2727	(5) 0.	4498 (9)	0.054 (3)			
C8	0.7572 (7)	-0.1982	(4) 0.	3282 (8)	0.052 (2)	E1	X 0.0479 (2)	
C8a	0.7512 (7)	-0.0846	(4) 0.	4199 (8)	0.046 (2)	F1 F2	0.0478(3) 0.0448(3)	
C9	0.6814 (7)	-0.0033	(4) 0.	3060 (7)	0.048 (2)	F3	0.1842(3)	
C9a	0.6803 (6)	0.1055	(4) 0.	4047 (8)	0.045 (2)	F4	0.3292 (3)	
C10a	0.8210(7) 0.7795(10)	-0.0490	(4) 0. (5) 0	0407 (8)	0.047(2) 0.082(4)	F5	0.3638 (3)	
Ch	0.7795 (10)	- 0.4245	(5) 0.	105 (1)	0.002 (4)	N10	0.3368 (4)	
T-1-1-4	C - I				for (2)	C1	0.1190 (5)	
Table 4	. Selectea	geometri	c parame	ters (A, ⁻)	Jor (2)	C2	0.1165 (5)	
O7—C7		1.359 (6)	C4a—C9a		1.437 (6)	C3	0.1892 (5)	
O7-C71		1.439 (7)	C5-C6		1.343 (7)	C4 C4a	0.2656 (4)	
N10-C4a		1.340 (6)	C5-C10a		1.431 (7)	C5	0.4125 (5)	
N10-C10a		1.328 (6)	C6C7		1.427 (7)	C6	0.4187 (5)	
C1 - C2		1.322(7) 1.417(7)	$C_{1} = C_{2}$		1.331(7) 1.427(7)	C7	0.3515 (5)	
$C_{2}-C_{3}$		1.425 (8)	C8a-C9		1.393 (7)	C8	0.2808 (5)	
C3—C4		1.339 (7)	C8a-C10a	1	1.452 (7)	C8a	0.2715 (4)	
C4—C4a		1.404 (7)	C9—C9a		1.384 (7)	C9	0.1989(5) 0.1031(4)	
C40 N10	C10a	118 3 (4)	C8a	-C9a	110 0 (4)	C3a	0.3385 (4)	
C4a—1410—	CIUU					0.10/2		
N10-C4a-	C9a	122.6 (5)	C4a-C9a-	-C9	118.5 (5)	CTUa	0.5505 (4)	
N10-C4a- C9-C8a-C	C9a 210a	122.6 (5) 117.0 (5)	C4a—C9a- N10—C10a		119.5 (4) 118.5 (5) 123.6 (5)	Table 6	Selected g	30
C4a=N10= N10-C4a= C9-C8a=C	C9a 210a	122.6 (5) 117.0 (5)	C4a—C9a- N10—C10a	C9 aC8a	119.5 (4) 118.5 (5) 123.6 (5)	Table 6	Selected g	30 .3
C9-C8a-C	C9a 210a .d (3)	122.6 (5) 117.0 (5)	C4a—C9a- N10—C10	-C9 a-C8a	119.5 (5) 118.5 (5) 123.6 (5)	Table 6 N10C4a N10C10a	. Selected §	30 .3
Compoun Crystal da	C9a 210a ad (3) <i>ta</i>	122.6 (5) 117.0 (5)	C4a—C9a- N10—C10	-C9 a-C8a	118.5 (5) 123.6 (5)	Table 6 N10	5. Selected §	30 .3 .3
Compoun Crystal da	C9a 210a dd (3)	122.6 (5) 117.0 (5)	C4a-C9a- N10-C10a	-C9 a-C8a	118.5 (5) 123.6 (5)	Table 6 N10C4a N10C10a C1C2 C1C9a C2	. Selected §	.3 .3 .3
$\begin{array}{c} \text{C}_{4a} = \text{M}_{10} \\ \text{N}_{10} = \text{C}_{4a} = 0 \\ \text{C}_{9} = \text{C}_{8a} = \text{C}_{12} \\ \text{Compoun} \\ \text{C}_{13}\text{H}_{4}\text{F}_{5}\text{N} \\ \text{M} = 260 \end{array}$	C9a C9a c10a d (3) ta	122.6 (5) 117.0 (5)	C4a - C9a - N10 - C10a Mo K α 1	-C9 a-C8a	118.5 (5) 123.6 (5)	Table 6 N10C4a N10C10a C1C2 C1C9a C2C3 C3C4	. Selected g	30.3
$C_{4a} = M_{10} = M_{10} = C_{4a} = C$	C9a 10a d (3) ta 17	122.6 (5) 117.0 (5)	$Mo K\alpha n$ $\lambda = 0.71$	-C9 a-C8a radiation 069 Å	118.5 (5) 123.6 (5)	Table 6 N10C4a N10C10a C1C2 C1C9a C2C3 C3C4 C4C4a	. Selected g	3.3 .3 .4 .4
$C_{13} = 10^{-10}$ $N_{10} = C_{4a} = -C$ $C_{7} = C_{8a} = C$ $C_{13} = C_{13} = C_{13}$ $M_r = 269.$ $Triclinic$	C9a 210a d (3) ta 17	122.6 (5) 117.0 (5)	$Mo K\alpha n$ $\lambda = 0.71$ Cell para	-C9 a-C8a radiation 069 Å umeters from	118.5 (5) 1123.6 (5) m 25	Table 6 N10C4a N10C10a C1C2 C1C9a C2C3 C3C4 C4C4a C4aC9a	. Selected §	3.3.3.4.4.3.4.4.3.4.4.
$C_{13} = 100^{-1}$ $C_{23} = C_{23} = C_{23}$ $Compoun$ $Crystal da$ $C_{13}H_4F_5N$ $M_r = 269.$ $Triclinic$ $P\overline{1}$	croa croa d (3) <i>ta</i> 17	122.6 (5) 117.0 (5)	$Mo \ K\alpha \ n$ $Mo \ K\alpha \ n$ $\lambda = 0.71$ Cell para reflect	-C9 a-C8a radiation 069 Å umeters from ions	m 25	Table 6 N10C4a N10C10a C1C2 C1C9a C2C3 C3C4 C4C4a C4aC9a C4a-N10	. Selected s	3.3.3.4.4.3.4.4.1
$C_{13} = 100^{-1}$ $C_{13} = 10^{-1}$ $C_{13} = 10^{-1}$ $M_r = 269^{-1}$ $M_r = 269^{-1}$ Triclinic $P\bar{1}$ $a = 8.270^{-1}$	c:9a c:10a d (3) <i>ta</i> 17 (5) Å	122.6 (5) 117.0 (5)	$Mo \ K\alpha \ n$ $Mo \ K\alpha \ n$ $\lambda = 0.71$ Cell para reflect $\theta = 35.5$	-C9 a-C8a radiation 069 Å meters from ions 5-40.85°	m 25	Table 6 N10C4a N10C10a C1C2 C1C2a C2C3 C3C4 C4C4a C4aC9a C4aC9a C4a-N10 N10C4a	. Selected s	3334434
$C_{13} = 100^{-1}$ $C_{13} = 100^{-1}$ $C_{13} = 100^{-1}$ $C_{13} = 100^{-1}$ $M_r = 100^{-1}$	(5) Å (5) Å (5) Å (5) Å	122.6 (5) 117.0 (5)	$Mo \ K\alpha \ n$ $Mo \ K\alpha \ n$ $\lambda = 0.71$ Cell para reflect $\theta = 35.5.$ $\mu = 0.15$	-C9 a-C8a radiation 069 Å meters from ions 5-40.85° 9 mm ⁻¹	m 25	Table 6 N10C4a N10C10a C1C2 C1C2a C2C3 C3C4 C4aC4a C4aN10 N10C4a- C9C8aC0	. Selected s	3334434
$C_{4a} = (C_{4a} = (C_{4a} = (C_{4a} = C_{4a} $	(5) Å (5) Å (5) Å (5) Å (2) Å	122.6 (5) 117.0 (5)	$Mo \ K\alpha \ n$ $Mo \ K\alpha \ n$ $\lambda = 0.71$ Cell para reflect $\theta = 35.5$ $\mu = 0.15$ $T = 296$	-C9 a-C8a radiation 069 Å meters from ions 5-40.85° 9 mm ⁻¹ K	m 25	Table 6 N10C4a N10C10a C1C2 C1C2a C2C3 C3C4 C4C4a C4aC9a C4aN10 N10C4a C9C8aC0	. Selected s	3.3.3.4.4.3.4.1.2.1
Compoun Crystal da Crystal da Crystal da C13H4F5N $M_r = 269$. Triclinic $P\overline{1}$ a = 8.270 b = 10.721 c = 5.995 $\alpha = 102.50$	(5) Å (5) Å (5) Å (5) Å (1 (5) Å (2) Å (2) Å (3)°	122.6 (5) 117.0 (5)	C4a-C9a- N10-C10k Mo K α f λ = 0.71 Cell para reflect θ = 35.5 μ = 0.15 T = 296 Needle	-C9 a-C8a radiation 069 Å meters from ions 5-40.85° 9 mm ⁻¹ K	m 25	Table 6 N10C4a N10C10a C1C2 C1C2 C1C3a C2C3 C3C4 C4C4a C4aC9a C-Campout	. Selected g 1 1 1 1 1 1 1 1 1 1 1 1 1	3334434
Compoun Crystal da Crystal da Crystal da C13H4F5N $M_r = 269$. Triclinic $P\overline{1}$ a = 8.270 b = 10.721 c = 5.995 $\alpha = 102.55$ $\beta = 92.60$	d (3) ta (5) Å (5) Å (2) Å (2) Å (3)° (4)°	122.6 (5) 117.0 (5)	$Mo \ K\alpha \ p$ $N10-C10k$ $Mo \ K\alpha \ p$ $\lambda = 0.71$ $Cell \ para reflect$ $\theta = 35.5, \mu = 0.15$ $T = 296$ $Needle$ $0.30 \times C$	-C9 a-C8 a-C8a radiation 069 Å mmeters from ions 5-40.85° 9 mm ⁻¹ K	m 25	Table 6 N10C4a N10C10a C1C2 C1C2 C1C3a C2C3 C3C4 C4C4a C4aC9a C4aC9a C4aC9a C4aC9a C4aC9a C4aC9a C4aC9a C4aC9a C9C8aC0 Compoun Compoun	. Selected g . Selected g 1 1 1 1 1 1 1 1 1 1 1 1 1	33344344
Compount Cy=C4a C9-C4a Crystal da C ₁₃ H ₄ F ₅ N $M_r = 269$. Triclinic $P\overline{1}$ a = 8.270 b = 10.721 c = 5.995 $\alpha = 102.57$ $\beta = 92.60$ $\gamma = 83.30$	$\begin{array}{c} \text{(S)} a \\ \text{(C)} a \\ \text{(C)} a \\ \text{(C)} a \\ \text{(C)} b \\ (C$	122.6 (5) 117.0 (5)	C4a-C9a- N10-C10k Mo K α f λ = 0.71 Cell para reflect θ = 35.5 μ = 0.15 T = 296 Needle 0.30 × C Yellow	-C9 a-C8 a-C8a radiation 069 Å immeters from ions 5-40.85° 9 mm ⁻¹ K 0.20 × 0.20	m 25	Table 6 N10C4a N10C10a C1C2 C1C2 C1C2 C1C2 C1C3 C3C4 C4C4a C4aC9a C4aN10 N10C4a C9C8aC9 Compound Crystal data	. Selected s . Selected s . 1 . 1 . 1 . 1 . 1 . 1 . 1 . 1	30 .3 .3 .3 .3 .3 .3 .4 .4 .4 .4 .4 .4 .1
Compount $C_{13}H_4F_5N$ $M_r = 269$. $C_{13}H_4F_5N$ $M_r = 269$. $Triclinic P\overline{1}a = 8.270b = 10.721c = 5.995\alpha = 102.55\beta = 92.60\gamma = 83.30V = 515.2$	$\begin{array}{c} \text{(S)} a \\ \text{(C)} a \\ (C$	122.6 (5) 117.0 (5)	$\begin{aligned} &\text{Mo } K\alpha \text{ is } \\ &\text{Mo } K\alpha \text{ is } \\ &\lambda = 0.71\\ &\text{Cell parareflect} \\ &\theta = 35.5, \\ &\mu = 0.15\\ &T = 296\\ &\text{Needle}\\ &0.30 \times C\\ &\text{Yellow} \end{aligned}$	-C9 a-C8 a-C8a radiation 069 Å immeters from ions 5-40.85° 9 mm ⁻¹ K 0.20 × 0.20	m 25	$\begin{array}{c} \text{Table 6} \\ \text{N10C4a} \\ \text{N10C10a} \\ \text{C1C2} \\ \text{C1C2} \\ \text{C2C3} \\ \text{C3C4} \\ \text{C4C4a} \\ \text{C4aC4a} \\ \text{C4aC9a} \\ \text{C4aN10N10C4a} \\ \text{C9C8aC9} \\ \hline \\ $	Selected s . Selected s . 1 . 1 . 1 . 1 . 1 . 1 . 1 . 1	20 .3 .3 .3 .4 .4 .4 .4 .1 .2 .2
Compount $C_{13}H_4F_5N$ $M_r = 269$. $C_{13}H_4F_5N$ $M_r = 269$. Triclinic $P\overline{1}$ a = 8.270 b = 10.721 c = 5.995 $\alpha = 102.57$ $\beta = 92.60$ $\gamma = 83.30$ V = 515.2 Z = 2	$\begin{array}{c} \text{(S)} a \\ \text{(C)} a \\ (C$	122.6 (5) 117.0 (5)	$Mo \ K\alpha \ i \lambda = 0.71$ $Mo \ K\alpha \ i \lambda = 0.71$ $Cell \ para reflect$ $\theta = 35.5$ $\mu = 0.15$ $T = 296$ Needle $0.30 \times C$ Yellow	-C9 a-C8 a-C8 meters from ions 5-40.85° 9 mm ⁻¹ K 0.20 × 0.20	m 25	Table 6 N10C4a N10C10a C1C2 C1C9a C2C3 C3C4 C4C4a C4aC9a C4a-N10 N10C4a C9C8aC9 Compound Crystal dat C21H15F3N $M_r = 352$	Selected s . Selected s 1. 1. 1. 1. 1. 1. 1. 1. 1. 1.	3.3 3.3 4.4 4.1 22 1
Compount $C_{13}H_4F_5N$ $M_r = 269$. $C_{13}H_4F_5N$ $M_r = 269$. Triclinic $P\overline{1}$ a = 8.270 b = 10.721 c = 5.995 $\alpha = 102.57$ $\beta = 92.60$ $\gamma = 83.30$ V = 515.2 Z = 2 $D_r = 1.737$	$d (3)$ ta $(5) Å$ $(5) Å$ $(5) Å$ $(5) Å$ $(2) Å$ $(3)^{\circ}$ $(4)^{\circ}$ $(3)^{\circ}$ $(9) Å^{3}$ $5 Mg m^{-3}$	122.6 (5) 117.0 (5)	Mo K α 10 N10-C100 Mo K α 11 $\lambda = 0.711$ Cell para reflect $\theta = 35.5$ $\mu = 0.15$ T = 296 Needle 0.30×0 Yellow	-C9 a-C8 a-C8 a meters from ions 5-40.85° 9 mm ⁻¹ K 0.20 × 0.20	m 25	Table 6 N10C4a N10C10a C1C2 C1C9a C2C3 C3C4 C4aC4a C4aC9a C4a-N10 N10C4a C9C8aC9 Compound Crystal da C21H15F3N $M_r = 352$. Monoclini	Selected g . Selected g 1. 1. 1. 1. 1. 1. 1. 1. 1. 1.	3.3 3.3 4.4 4.3 4.4 1 2 2
Compount $C_{13}H_4F_5N$ $M_r = 269$. Triclinic $P\overline{1}$ a = 8.270 b = 10.721 c = 5.995 $\alpha = 102.57$ $\beta = 92.60$ $\gamma = 83.30$ V = 515.2 Z = 2 $D_x = 1.733$	$d (3)$ ta $(5) Å$ $(5) Å$ $(5) Å$ $(2) Å$ $(3)^{\circ}$ $(9) Å^{3}$ $5 Mg m^{-3}$	122.6 (5) 117.0 (5)	Mo K α 1 N10-C10 N10-C10 N10-C10 N10-C10 Needle 0.15 T = 296 Needle 0.30 × 0 Yellow	-C9 a-C8 a-C8 a meters from ions 5-40.85° 9 mm ⁻¹ K 0.20 × 0.20	m 25	Table 6 N10C4a N10C10a C1C2 C1C9a C2C3 C3C4 C4aC4a C4aC9a C4aN10 N10C4a C9C8aC0 Compound C21H15F3N Mr = 3522. Monoclinii P2, /c	. Selected s . Selected s . 1 . 1 . 1 . 1 . 1 . 1 . 1 . 1	30 33 33 44 43 44 11 22 11
Compoun $C_{13}H_4F_5N$ $M_r = 269$. Triclinic $P\overline{1}$ a = 8.270 b = 10.721 c = 5.995 $\alpha = 102.57$ $\beta = 92.60$ $\gamma = 83.30$ V = 515.2 Z = 2 $D_x = 1.733$ Data colle	$d (3)$ ta $(5) Å$ $(5) Å$ $(5) Å$ $(2) Å$ $(3)^{\circ}$ $(9) Å^{3}$ $5 Mg m^{-3}$	122.6 (5) 117.0 (5)	Mo K α 1 $\lambda = 0.71$ Cell para reflect $\theta = 35.5$ $\mu = 0.15$ T = 296 Needle 0.30×0 Yellow	-C9 a-C8 a-C8 a meters from ions 5-40.85° 9 mm ⁻¹ K 0.20 × 0.20	m 25	Table 6 N10C4a N10C10a C1C2 C1C9a C2C3 C3C4 C4aC4a C4aC4a C4aC4a C4aC4a C4aC4a C2C3 C4aC4a C4aC4a C4aC4a C4aC4a C9C8aC0 Compound C21H15F3N Mr G21-C1 C3C5 C3C5	Selected g . Selected g 1 1 1 1 1 1 1 1 1 1 1 1 1	3.3.3.4 4.4 12:11
Compoun $C_{13}H_4F_5N$ $M_r = 269$. Triclinic $P\overline{1}$ a = 8.270 b = 10.721 c = 5.995 $\alpha = 102.55$ $\beta = 92.60$ $\gamma = 83.30$ V = 515.2 Z = 2 $D_x = 1.735$ Data colle	$d (3)$ ta $(5) Å$ $(5) Å$ $(5) Å$ $(2) Å$ $(2) Å$ $(3)^{\circ}$ $(3)^{\circ}$ $(9) Å^{3}$ $5 Mg m^{-3}$ $ection$	122.6 (5) 117.0 (5)	Mo K α 1 N10-C10 N10-C10 Mo K α 1 λ = 0.71 Cell para reflect θ = 35.5 μ = 0.15 T = 296 Needle 0.30 × 0 Yellow	-C9 a-C8 a-C8 a meters from ions 5-40.85° 9 mm ⁻¹ K 0.20 × 0.20	m 25	Cloa Table 6 N10C4a N10C10a C1C2 C1C9a C2C3 C3C4 C4aC4a C4aC9a C9C8aC9 C0mpoun C71H15F3N Mr = 352. Monoclinii P21/c a = 5.8711 b = 7.9820	Selected ξ . Selected ξ 1. 1. 1. 1. 1. 1. 1. 1. 1. 1.	30 .3 .3 .4 .4 .4 .4 .1 .2 .1
Compound $C_{13}H_4F_5N$ $M_r = 269$. Triclinic $P\overline{1}$ a = 8.270 b = 10.721 c = 5.995 $\alpha = 102.5$; $\beta = 92.60$ $\gamma = 83.30$ V = 515.2 Z = 2 $D_x = 1.735$ Data colle	$d (3)$ ta $(5) Å$ $(5) Å$ $(5) Å$ $(2) Å$ $(2) Å$ $(3)^{\circ}$ $(3)^{\circ}$ $(9) Å^{3}$ $5 Mg m^{-3}$ $ection$ liffractome	122.6 (5) 117.0 (5)	$Mo \ K\alpha \ i$ $Ni0-C10k$ $Mo \ K\alpha \ i$ $\lambda = 0.71$ $Cell \ para reflect$ $\theta = 35.5.$ $\mu = 0.15$ $T = 296$ $Needle$ $0.30 \times C$ $Yellow$ $\theta_{max} = 2$	-C9 a-C8 a-C8 a meters from ions 5-40.85° 9 mm ⁻¹ K 0.20 × 0.20 4.99°	m 25	Cloa Table 6 N10C4a N10C10a C1C2 C1C9a C2C3 C3C4 C4aC4a C4aC4a C4aC9a C4aC4a C9C8aC0 Compound Crystal da C2-1H15F3N M_r = 352. Monoclinii P21/c a = 5.871 b = 7.889	Selected g . Selected g 1. 1. 1. 1. 1. 1. 1. 1. 1. 1.	30 33 33 44 34 44 11 22 11
Compound $C_{13}H_4F_5N$ $C_{7}=C8a-CC$ Compound $C_{7}ystal da$ $C_{13}H_4F_5N$ $M_r = 269.$ Triclinic $P\overline{1}$ a = 8.270 b = 10.721 c = 5.995 $\alpha = 102.5$ $\beta = 92.60$ $\gamma = 83.30$ V = 515.2 Z = 2 $D_x = 1.733$ Data colle AFC-6S d $\omega/2\theta$ scan	$cost = 10^{10} cost = 10^{10} cost$	122.6 (5) 117.0 (5)	$\begin{aligned} &\text{Mo } K\alpha \text{ if } \\ &\text{Mo } K\alpha \text{ if } \\ &\lambda = 0.71\\ &\text{Cell parareflect} \\ &\theta = 35.5, \\ &\mu = 0.15\\ &T = 296\\ &\text{Needle}\\ &0.30 \times &\text{C}\\ &\text{Yellow} \end{aligned}$	-C9 a-C8a a-C8a a-C8a a-C8a a-C8a a-C8a a-C8a a-C8a a-C8a b-C8a a-C8a b-C8a a-C8a b-C8a a-C8a b-C8a a-C8a b-C9a b-C8a b-C9a b-C	m 25	Cloa Table 6 N10C4a N10C10a C1C2 C1C9a C2C3 C3C4 C4aC4a C4aC4a C4aC4a C4aC4a C4aC4a C4aC4a C4aC4a C4aC4a C4aC4a C2C8aC0 Compoun Crystal da C21H15F3N M_r = 352. Monoclini P21/c a = 5.8711 b = 7.889 c = 34.830 c = 34.830	Selected g . Selected g 1 1 1 1 1 1 1 1 1 1 1 1 1	3.3 3.4 4.4 11 22 11
Compound Crystal da Crystal da Crystal da Crystal da Crystal da $C_{13}H_4F_5N$ $M_r = 269$. Triclinic $P\overline{1}$ a = 8.270 b = 10.721 c = 5.995 $\alpha = 102.5$ $\beta = 92.60$ $\gamma = 83.30$ V = 515.2 Z = 2 $D_x = 1.733$ Data colle AFC-6S d $\omega/2\theta$ scan Absorption	c) Source 20° c) So	122.6 (5) 117.0 (5)	$\theta_{\text{max}} = 2$ $h = 0$	-C9 a-C8 a-C8a radiation 069 Å mmeters from 5-40.85° 9 mm ⁻¹ K 0.20 × 0.20 4.99° 9 9 2 → 12	m 25	Cloa Table 6 N10C4a N10C10a C1C2 C1C9a C2C3 C3C4 C4aC4a C4aC9a C4aN10 N10C4a C9C8aC0 Compound Crystal da C21H15F3N $M_r = 352$. Monoclinii P21/c $a = 5.8711$ $b = 7.889$ $c = 34.830$ $\beta = 91.23$	Selected g . Selected g 1 1 1 1 1 1 1 1 1 1 1 1 1	3.3 .3 .4 .4 .4 .3 .4 .4 .1 .2 .1
Compound $C_{13}H_4F_5N$ $C_{7}=C8a-CC$ Compound Crystal da $C_{13}H_4F_5N$ $M_r = 269.$ Triclinic $P\overline{1}$ a = 8.270 b = 10.721 c = 5.995 $\alpha = 102.5$ $\beta = 92.60$ $\gamma = 83.30$ V = 515.2 Z = 2 $D_x = 1.733$ Data colle AFC-6S d $\omega/2\theta$ scan Absorption empiric	c) Sqa c) Sqa c) Sqa c) Sqa ta ta 17 (5) Å (1 (5) Å (2) Å 6 (3)° (4)° (3)° (9) Å ³ 5 Mg m ⁻³ ection liffractome s n correctic al	122.6 (5) 117.0 (5)	$\theta_{\text{max}} = 2$ $h = 0$ $h = -12$ $h = -6$	-C9 a-C8 a-C8 a meters from $5-40.85^{\circ}$ 9 mm ⁻¹ K 0.20 × 0.20 4.99° 9 2 → 12 → 6	m 25	Cloa Table 6 N10C4a N10C10a C1C2 C1C9a C2C3 C3C4 C4aC4a C4aC9a C4aC4a C4aC4a C4aC9a C21H15F3N Mr = 352. Monoclini P21/c a = 5.871 b = 7.889 c = 34.830 $\beta = 91.23$ V = 1612	Selected g . Selected g 1 1 1 1 1 1 1 1 1 1 1 1 1	30 .3 .3 .4 .4 .4 .4 .1 .2 .1
Compound $C_{13}H_4F_5N$ $C_{7}=C8a-CC$ Compound Crystal da $C_{13}H_4F_5N$ $M_r = 269.$ Triclinic $P\overline{1}$ a = 8.270 b = 10.721 c = 5.995 $\alpha = 102.5$ $\beta = 92.60$ $\gamma = 83.30$ V = 515.2 Z = 2 $D_x = 1.733$ Data colle AFC-6S d $\omega/2\theta$ scan Absorption empiric $T_{min} = 0$	c) Solution c) S	122.6 (5) 117.0 (5) eter on: a = 1.00	$\theta_{max} = 2$ $h = 0$ $h = -12$ $h = -6$ $h = -2$ $h = -6$ $h = -2$ $h = -6$ $h = -2$ $h = -6$	-C9 a-C8 a-C8 a meters from ions 5-40.85° 9 mm ⁻¹ K 0.20 × 0.20 4.99° 9 9 2 → 12 → 6 rd reflectio	m 25) mm	Table 6 N10-C4a N10-C10a C1-C2 C1-C9a C2-C3 C3-C4 C4a-C9a C4a-N10- N10-C4a- C9-C8a-C9 Compoun Crystal da C ₂₁ H ₁₅ F ₃ N $M_r = 352$. Monoclini $P2_1/c$ a = 5.871 b = 7.889 c = 34.830 $\beta = 91.23$ V = 1612 Z = 4	Selected g . Selected g 1 1 1 1 1 1 1 1 1 1 1 1 1	30 .3 .3 .4 .4 .4 .4 .1 .2 .1
Compount Cy=C4a C9-C4a C9-C8a-C Compount Crystal da C ₁₃ H ₄ F ₅ N $M_r = 269$. Triclinic $P\overline{1}$ a = 8.270 b = 10.721 c = 5.995 $\alpha = 102.5^{\circ}$ $\beta = 92.60$ $\gamma = 83.30$ V = 515.2 Z = 2 Data colle AFC-6S d $\omega/2\theta$ scan Absorption empiric $T_{min} = (1945 mean)$	c) Solution c) S	122.6 (5) 117.0 (5) 117.0 (5)	$\begin{aligned} & \theta_{max} = 2\\ & h = 0 \\ & h = 0 \\ & h = 0 \end{aligned}$	-C9 a-C8 a-C8 a a-C8 a a-C8 a a-C8 a a-C8 a a a-C8 a a a-C8 a b a b a b a b a b a b a b a b a b a	m 25) mm	Table 6 N10-C4a N10-C10a C1-C2 C1-C9a C2-C3 C3-C4 C4a-C4a C4a-C9a C4a-N10- N10-C4a- C9-C8a-C9 Compoun Crystal da C21H15F3N $M_r = 352$. Monoclini $P2_1/c$ a = 5.871 b = 7.889 c = 34.830 $\beta = 91.23$ V = 1612 Z = 4 $D_x = 1.45$	Selected g . Selected g 1 1 1 1 1 1 1 1 1 1 1 1 1	30 .3.3.3.4 .4 .4 .4 .4 .1 .2 .1
Compount $C_{3} - C_{4a} - C_{5} - C_{5a} - C_$	c) Ga C	22.6 (5) 117.0 (5) 117.0 (5) etter e	$\begin{array}{l} \text{Mo } K\alpha \text{ fr} \\ \lambda = 0.71\\ \text{Cell para} \\ reflect\\ \theta = 35.5\\ \mu = 0.15\\ T = 296\\ \text{Needle} \\ 0.30 \times \text{C}\\ \text{Yellow} \\ \end{array}$ $\begin{array}{l} \theta_{\text{max}} = 2\\ h = 0 \\ -k = -12\\ l = -6\\ 3 \text{ standa} \\ \text{monit} \\ refl \end{array}$	-C9 a-C8 a-C8a radiation 069 Å meters from ions 5-40.85° 9 mm ⁻¹ K 0.20 × 0.20 4.99° → 9 2 → 12 → 6 rd reflectio ored every ections	ns 150	Table 6 N10-C4a N10-C10a C1-C2 C1-C9a C2-C3 C3-C4 C4-C4a C4a-C9a C4a-N10- N10-C4a- C9-C8a-C9 Compoun <i>Crystal da</i> C ₂₁ H ₁₅ F ₃ N <i>M_r</i> = 352. Monoclini <i>P</i> 2 ₁ / <i>c</i> <i>a</i> = 5.871 <i>b</i> = 7.889 <i>c</i> = 34.830 β = 91.23 <i>V</i> = 1612 <i>Z</i> = 4 <i>D_x</i> = 1.45	Selected g . Selected g 1 1 1 1 1 1 1 1 1 1 1 1 1	3.3 3.3 4.4 4.1 22 1
Compount $C_{13}H_4F_5N$ $M_r = 269$. $C_{13}H_4F_5N$ $M_r = 269$. $Triclinic P\overline{1}a = 8.270b = 10.721c = 5.995\alpha = 102.5^{\circ}\beta\beta = 92.60\gamma = 83.30V = 515.2Z = 2D_x = 1.733Data colleAFC-6S d\omega/2\theta scanAbsorptionempiricT_{min} = (1945 mear1808 inde951 obser$	c) Solution c) S	22.6 (5) 117.0 (5) 117.0 (5) 117.0 (5) 117.0 (5)	$\begin{array}{l} \text{Mo } K\alpha = 0\\ \lambda = 0.71\\ \text{Cell para}\\ reflect\\ \theta = 35.5\\ \mu = 0.15\\ T = 296\\ \text{Needle}\\ 0.30 \times C\\ \text{Yellow} \end{array}$ $\begin{array}{l} \theta_{\text{max}} = 2\\ h = 0\\ -k = -12\\ l = -6\\ 3 \text{ standa}\\ \text{monit}\\ refl\\ \text{intense} \end{array}$	-C9 a-C8 a-C8 a meters from ions 5-40.85° 9 mm ⁻¹ K 0.20 × 0.20 4.99° → 9 2 → 12 → 6 rd reflections ity variation	ns 150 n:	Table 6 N10-C4a N10-C10a C1-C2 C1-C9a C2-C3 C3-C4 C4-C4a C4a-C9a C4a-N10- N10-C4a- C9-C8a-C9 Compoun <i>Crystal da</i> C ₂₁ H ₁₅ F ₃ N <i>M_r</i> = 352. Monoclini <i>P</i> 2 ₁ / <i>c</i> <i>a</i> = 5.871 <i>b</i> = 7.889 <i>c</i> = 34.830 β = 91.23 <i>V</i> = 1612 <i>Z</i> = 4 <i>D_x</i> = 1.45 <i>Data colle</i>	Selected g . Selected g 1 1 1 1 1 1 1 1 1 1 1 1 1	20 33 33 44 34 4 12 21
Compount $C_{13}H_4F_5N$ $M_r = 269$. Triclinic $P\overline{1}$ a = 8.270 b = 10.721 c = 5.995 $\alpha = 102.55$ $\beta = 92.60$ $\gamma = 83.30$ V = 515.2 Z = 2 $D_x = 1.733$ Data colle AFC-6S d $\omega/2\theta$ scan Absorption empiric $T_{min} = ($ 1945 mear 1808 inde 951 obser [I > 30	c) Solution c) S	ter = 1.00 ctions effections fions	$\begin{array}{c} \text{Mo } K\alpha \text{ fr} \\ \lambda = 0.71\\ \text{Cell para} \\ reflect \\ \theta = 35.5, \\ \mu = 0.15\\ T = 296\\ \text{Needle} \\ 0.30 \times \text{C} \\ \text{Yellow} \\ \end{array}$ $\begin{array}{c} \theta_{\text{max}} = 2\\ h = 0 \\ -k = -12\\ l = -6\\ 3 \text{ standa} \\ \text{monit} \\ refl\\ \text{intens} \\ -0 \end{array}$	-C9 a-C8 a-C8 a a-C8 a a-C8 a a-C8 a a-C8 a a a-C8 a a a-C8 a b a b a b a b a b a b a b a b a b a	ns 150 m:	Table 6 N10-C4a N10-C10a C1-C2 C1-C9a C2-C3 C3-C4 C4-C4a C4a-C9a C4a-N10- N10-C4a- C9-C8a-C9 Compoun <i>Crystal da</i> C ₂₁ H ₁₅ F ₃ N <i>M_r</i> = 352. Monoclini <i>P</i> 2 ₁ / <i>c</i> <i>a</i> = 5.871 <i>b</i> = 7.889 <i>c</i> = 34.830 β = 91.23 <i>V</i> = 1612 <i>Z</i> = 4 <i>D_x</i> = 1.45 <i>Data colle</i> AFC-65 d	Selected g . Selected g 1 1 1 1 1 1 1 1 1 1 1 1 1	20 3.3.3.4.4.3.4.4 11211

Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{\rm max} = 0.08$
$\Delta \rho_{\rm max} = 0.19 \ {\rm e} \ {\rm \AA}^{-3}$
$\Delta \rho_{\rm min} = -0.27 \ {\rm e} \ {\rm \AA}^{-3}$
Atomic scattering factors
from International Tables
for X-ray Crystallography
(1974, Vol. IV)

actional atomic coordinates and equivalent ic displacement parameters $(Å^2)$ for (3)

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

4 (3)		r	ν		7	Um
2 (2)	F1	0.0478 (3)	0.8677	(2)	0.8655 (4)	0.060(1)
6 (2)	F7	0.0448(3)	0.6227	(2)	0.6302 (5)	0.070(1)
8 (2)	F3	0.1842(3)	0 5441 ((2)	0.2195 (5)	0.075(1)
5 (2)	F4	0.3292(3)	0 7085	(2)	0.0316(4)	0.061(1)
7 (2)	F5	0.3638 (3)	1 4606 ((2)	0 6779 (5)	0.077(1)
2 (4)	N10	0.3368 (4)	0 9583	(3)	0.2503 (5)	0.039(1)
	Cl	0.1100 (5)	0.9385	(4)	0.6508 (7)	0.033(1)
(2)	C1	0.1150(5)	0.0200	(*) (4)	0.5463 (8)	0.040 (2)
(2)	C2 C3	0.1103(3)	0.7070	(4)	0.3405 (8)	0.030(2)
7 (6)	C3	0.1692(3)	0.0000	(4)	0.3290(7)	0.049(2)
3 (7)	C4	0.2393(3)	0.7469	(4)	0.2505(7)	0.040(2)
1 (7)	C4a	0.2030 (4)	1 1661	(4)	0.3510 (0)	0.039(2)
7 (7)	C5	0.4123(3)	1.1001	(4) (4)	0.2348 (7)	0.043(2)
1 (7)	C0	0.4167(3)	1.2099	(4)	0.3330 (8)	0.049(2)
7 (7)		0.3313(3)	1.3330	(4)	0.5780 (8)	0.031(2)
3 (7)		0.2606(3)	1.2021	(4) (4)	0.0903(0)	0.047 (2)
2 (7)	Coa	0.2713 (4)	1.1510	(4)	0.3847 (0)	0.039(2)
4 (7)	09	0.1989 (5)	1.0464	(4)	0.0838 (8)	0.042 (2)
0 (4)	C9a	0.1931 (4)	0.9204	(4)	0.5722 (6)	0.038 (2)
9 (4) 5 (5)	C10a	0.3385 (4)	1.0812	(4)	0.3625 (6)	0.038 (2)
5 (5) 6 (5)	Table 6.	Selected	l geometric	: param	eters (Å, °) for (3)
	N10 C4a		1 226 (4)	C5 C6		1 344 (6)
	N10C4a		1.330 (4)	C5 C10	0	1.344 (0)
	NIU-CIUa		1.343 (3)	C = C I 0	a	1.429 (3)
	C1 - C2		1.334 (3)	\mathcal{C}		1.412(0)
	$C_1 - C_{9a}$		1.421 (5)			1.338(0)
	$C_2 - C_3$		1.413(0)	$-c_0$		1.420(0)
	$C_3 - C_4$		1.336 (3)	Coa-Cy	0	1.397 (3)
	C4		1.432 (5)	$c_{0a} - c_{1}$	Ua	1.434 (3)
	C4aC9a		1.435 (5)	C9-C9a		1.379(3)
	C4a-N10-0	C10a	117.5 (3)	C8aC9	C9a	120.1 (4)
	N10-C4a-0	C9a	123.5 (3)	C4aC9	a—C9	117.9 (3)
	C9—C8a—C	10a	117.4 (4)	N10-C1	0a—C8a	123.5 (4)
	Compour	d (4)			•	
1	Compoun	u (+)				
	Crystal da	ta				
	C21H15F3N	12		Μο Κα	radiation	
	$M = 352^{\circ}$	36		$\lambda = 0.7$	1069 Å	
	$W_{r} = 552.$	50		C-11		20
	Monoclini	c		Cell pa	rameters fro	om 20
	$P2_{1}/c$			reflec	ctions	
	a = 5 871	(A) Å		$\theta = 14.$	99-23.39°	
	u = 5.871			u = 0.1	04 mm^{-1}	
	b = 7.889	(3) A		$\mu = 0.1$		
	c = 34.830) (9) Å		T = 290	5 K	
	$\beta = 91.23$	(À)°		Plate		
	V = 1612	à 13		$0.40 \times$	0.35×0.1	5 mm
	v = 1012	$(2) \mathbf{A}^{-}$		Dolo rio	1100	
	Z = 4			rale ye	now	
	$D_x = 1.45$	1 Mg m ⁻³	3			
	Data colle	ction				
	AEC 65 A	iffractome	ter	$R_{1} = 0$	070	
	Arc-03 0			$\Lambda_{int} = 0$	1070 15 ⁰	
	ω scans w	iin profile	anaiysis	$\sigma_{\rm max} = 2$	23-	

Absorption correction: refined from ΔF (*DI*-*FABS*; Walker & Stuart, 1983) $T_{min} = 0.83$, $T_{max} = 1.18$ 3372 measured reflections 3049 independent reflections 1317 observed reflections $[I > 3\sigma(I)]$

Refinement

Refinement on F R = 0.068 wR = 0.066 S = 4.251317 reflections 283 parameters All H-atom parameters refined except H on methyl C18, fixed $h = 0 \rightarrow 6$ $k = 0 \rightarrow 20$ $l = -40 \rightarrow 40$ 3 standard reflections monitored every 150 reflections intensity variation: 1.09%

Weighting scheme based on measured e.s.d.'s $(\Delta/\sigma)_{max} = 0.081$ $\Delta\rho_{max} = 0.30 \text{ e} \text{ Å}^{-3}$ $\Delta\rho_{min} = -0.33 \text{ e} \text{ Å}^{-3}$ Atomic scattering factors from International Tables for X-ray Crystallography (1974, Vol. IV)

...

Table 7. Fractional atomic coordinates and equivalentisotropic displacement parameters (Å²) for (4)

$$U_{\text{eq}} = (1/3) \sum_{i} \sum_{j} U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	у		Z	U_{ea}
Fl	0.9691 (6)	0.0290 (5) 0.5	589 (1)	0.050 (2)
F2	0.8437 (6)	0.0300 (6) 0.6	321 (1)	0.052 (2)
F4	0.1739 (6)	0.3322 (6) 0.5	990 (1)	0.046 (2)
N10	0.2821 (9)	0.3259 (8) 0.5	232 (2)	0.034 (3)
N11	0.439 (1)	0.1846 (10) 0.6	548 (2)	0.049 (4)
C1	0.772 (1)	0.1084 (10) 0.5	679 (2)	0.037 (4)
C2	0.712 (1)	0.1089 (10) 0.6	054 (2)	0.039 (4)
C3	0.507 (1)	0.191 (1) 0.6	173 (2)	0.037 (4)
C4	0.372 (1)	0.2570 (9) 0.5	884 (2)	0.029 (3)
C4a	0.426 (1)	0.2579 (9) 0.5	487 (2)	0.033 (4)
C5	0.194 (1)	0.393 (1) 0.4	576 (2)	0.037 (4)
C6	0.247 (1)	0.388 (1) 0.4	193 (2)	0.044 (4)
C7	0.453 (1)	0.315 (1) 0.4	060 (2)	0.041 (4)
C8	0.603 (1)	0.2495 (10) 0.4	334 (2)	0.037 (4)
C8a	0.553 (1)	0.2499 (9) 0.4	724 (2)	0.032 (4)
C9	0.696 (1)	0.1795 (10) 0.5	006 (2)	0.034 (4)
C9a	0.637 (1)	0.1806 (9) 0.5	387 (2)	0.032 (4)
C10a	0.341 (1)	0.3228 (10) 0.4	855 (2)	0.033 (4)
C12	0.580 (1)	0.2243 (10) 0.6	867 (2)	0.039 (4)
C13	0.785 (1)	0.309 (1) 0.6	846 (3)	0.046 (5)
C14	0.918 (1)	0.345 (1)) 0.7	160 (3)	0.046 (4)
C15	0.853 (1)	0.295 (1) 0.7	528 (2)	0.042 (4)
C16	0.646 (1)	0.211 (1)) 0.7	553 (2)	0.050 (5)
C17	0.512(1)	0.175 (1) 0.7	235 (2)	0.043 (4)
C18	1.002 (1)	0.330 (1)) 0.7	882 (3)	0.069 (5)
C19	0.507 (2)	0.317 (2) 0.3	649 (3)	0.066 (6)
Table 8	. Selected	d geometric	paramete	ers (Å, °)) for (4)
N10—C4a		1.326 (7)	C5-C6		1.37 (1)
N10-C10a		1.366 (8)	C5-C10a		1.403 (9)
N11-C3		1.375 (9)	C6—C7		1.43 (1)
N11-C12		1.408 (9)	C7-C8		1.39 (1)
C1-C2		1.36 (1)	C7-C19		1.48 (1)
C1C9a		1.401 (9)	C8—C8a		1.40(1)
C2—C3		1.43 (1)	C8aC9		1.394 (9)
C3C4		1.372 (9)	C8a-C10a		1.449 (9)
C4—C4a		1.421 (8)	C9—C9a		1.379 (9)
C4a—C9a		1.432 (9)			

The crystals of (2) were extremely thin and a rotating-anode source had to be used. Data collection: MSC/AFC Diffractome-

C8a-C9-C9a

C4a-C9a-C9

N10-C10a-C8a

© 1994 International Union of Crystallography Printed in Great Britain – all rights reserved

117.8 (6)

123.3 (7)

116.4 (7)

C4a-N10-C10a

N10-C4a-C9a

C9-C8a-C10a

ter Control Software (Molecular Structure Corporation, 1988). Cell refinement: MSC/AFC Diffractometer Control Software. Data reduction: TEXSAN PROCESS (Molecular Structure Corporation, 1985). Program(s) used to solve structure: TEXSAN, MITHRIL (Gilmore, 1984). Program(s) used to refine structure: TEXSAN LS. Molecular graphics: TEXSAN, PLUTO (Motherwell & Clegg, 1978). Software used to prepare material for publication: TEXSAN FINISH. Literature survey: CSSR (1984).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1078). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Adamson, A. J., Banks, R. E. & Tipping A. E. (1993). J. Fluorine Chem. 64, 5-10.
- Alty, A. C., Banks, R. E., Fishwick, B. R., Pritchard, R. G. & Thompson, A. R. (1984). J. Chem. Soc. Chem. Commun. pp. 832-833.
- Alty, A. C., Banks, R. E., Fishwick, B. R. & Thompson, A. R. (1985). Tetrahedron Lett. pp. 1345–1348.
- CSSR (1984). Crystal Structure Search and Retrieval Instruction Manual. SERC Daresbury Laboratory, Warrington, England.

Flowers, W. T. & DeFigueredo, P. (1990). Personal communication. Gilmore, C. J. (1984). J. Appl. Cryst. 17, 42-46.

- Molecular Structure Corporation (1985). TEXSAN. TEXRAY Structure Analysis Package. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Molecular Structure Corporation (1988). MSC/AFC Diffractometer Control Software. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Motherwell, W. D. S. & Clegg, W. (1978). PLUTO. Program for Plotting Molecular and Crystal Structures. Univ. of Cambridge, England. Walker, N. & Stuart, D. (1983). Acta Cryst. A39, 158-166.

Acta Cryst. (1994). C50, 971-974

3-Selenocyanatopyridine

S. J. DUNNE, L. A. SUMMERS AND

E. I. VON NAGY-FELSOBUKI

Department of Chemistry, The University of Newcastle, Callaghan, NSW 2308, Australia

M. F. MACKAY

Department of Chemistry, La Trobe University, Bundoora, Victoria 3083, Australia

(Received 11 October 1993; accepted 13 December 1993)

Abstract

120.9 (7)

118.4 (6)

123.1 (6)

The crystal structure of the title compound (3-pyridyl selenocyanate, $C_6H_4N_2Se$) has been determined by X-ray analysis. As expected, the Se- C_{sp} bond of length