Table 2. *Selected geometric parameters* (Å, °)

N1—C5	1.343 (3)	C9—C10	1.384 (4)
N1—C6	1.455 (3)	C10—C11	1.388 (4)
O1—C5	1.354 (3)	C11—C12	1.376 (5)
O1—C1	1.477 (3)	C12—C13	1.371 (4)
O2—C5	1.214 (3)	C13—C14	1.391 (4)
O3—C7	1.202 (3)	C15—C16	1.506 (3)
O4—C7	1.339 (3)	C16—C17	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)	C23—C24	1.385 (3)
C9—C14	1.382 (3)		
C5—N1—C6	120.7 (2)	C10—C9—C8	117.9 (2)
C5—O1—C1	119.8 (2)	C9—C10—C11	121.1 (3)
C7—O4—C8	116.8 (2)	C12—C11—C10	119.8 (3)
O1—C1—C3	102.7 (2)	C13—C12—C11	119.7 (3)
O1—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)
O2—C5—O1	125.4 (2)	C18—C17—C16	125.5 (2)
N1—C5—O1	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)		

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 $U(\mathrm{H}) = 1.2U_{\mathrm{eq}}(\mathrm{C})$. Data collection and cell refinement: *DIF*4 (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.

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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

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(*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

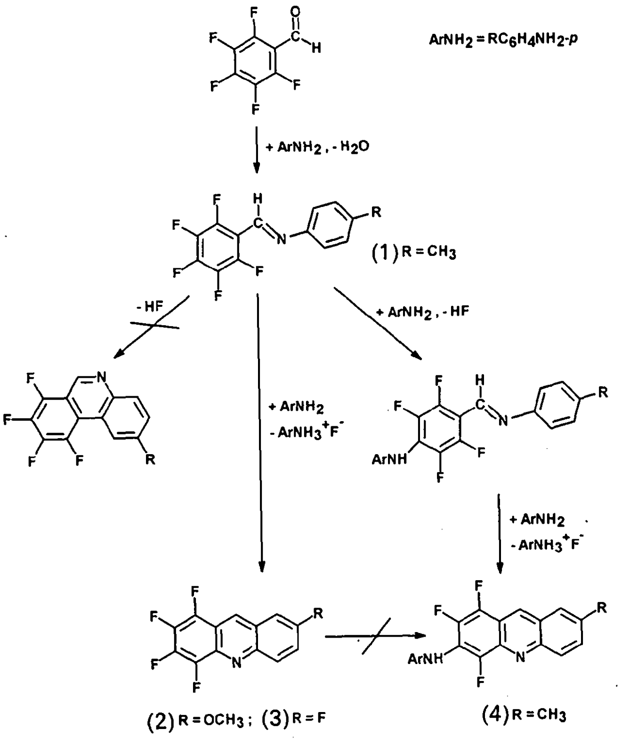
During the purification of a crude sample of the Schiff base $C_6F_5CH{=}NC_6H_4(OCH_3\text{-}4)$ [(1) with $R = OCH_3$] obtained by heating pentafluorobenzaldehyde with *p*-methoxyaniline in boiling *n*-butyl acetate, Flowers & DeFiguеredo (1990) isolated a by-product which they concluded was 7,8,9,10-tetrafluoro-2-methoxyphenanthridine 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 $C_6F_5CH{=}NAr$ for mass spectral studies. For our part we wished to compare the mechanism of this intramolecu-

Acta Crystallographica Section C
ISSN 0108-2701 ©1994

lar 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 ^{19}F NMR signals could not be assigned unambiguously. The crystal structure determination, described here, showed that the by-product from the reaction of C_6F_5CHO with $H_2NC_6H_4(OCH_3\text{-}4)$ 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.



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

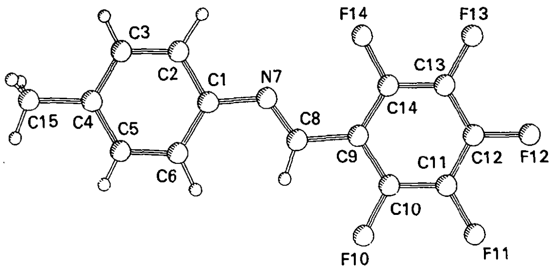


Fig. 1. Molecule (1) including atomic labelling scheme.

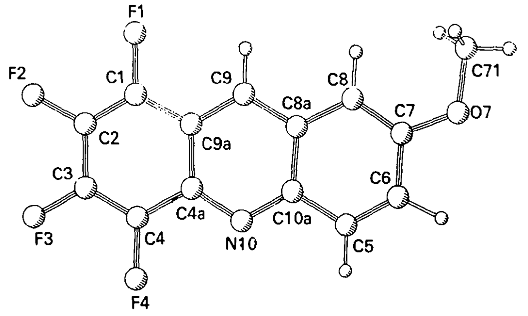


Fig. 2. Molecule (2) including atomic labelling scheme [an identical labelling scheme has been used for molecule (3)].

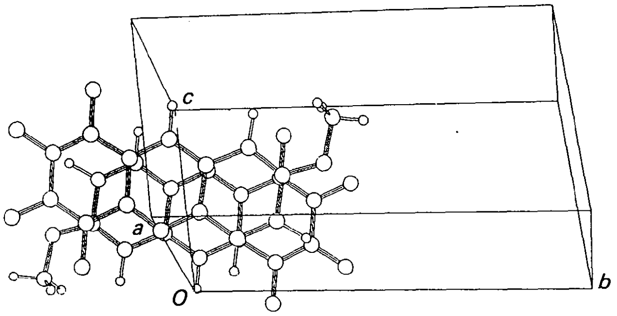


Fig. 3. Crystal packing in (2) showing the type of overlapping π systems seen in all four crystal structures.

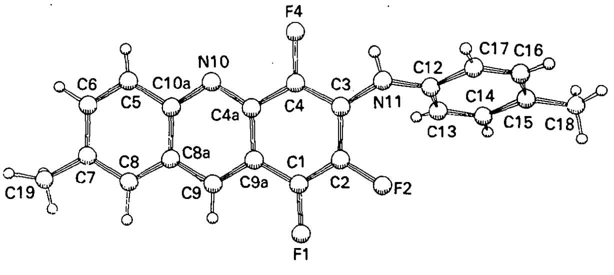


Fig. 4. Molecule (4) including atomic labelling scheme.

Experimental

The Schiff base [(1) m.p. 412 K] was obtained in 93% yield (after recrystallization from boiling acetone) by stirring pentafluorobenzaldehyde 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 1,2-dichlorobenzene) (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).

Compound (1)

Crystal data

$C_{14}H_8F_5N$
M_r = 285.22
Triclinic
$P\bar{1}$
a = 7.437 (3) Å
b = 13.176 (4) Å
c = 6.210 (2) Å
α = 92.00 (3)°
β = 96.90 (3)°
γ = 91.76 (3)°
V = 603.4 (7) Å^3
Z = 2
D_x = 1.570 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 23.93–37.10°
μ = 0.14 mm^{-1}
T = 296 K
Needle
0.40 × 0.20 × 0.10 mm
Colourless

Data collection

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

$R_{\rm int}$ = 0.034
$\theta_{\max}$ = 25.0°
$h = 0 \rightarrow 8$
$k = -15 \rightarrow 15$
$l = -7 \rightarrow 7$
3 standard reflections monitored every 150 reflections intensity variation: 0.03%

Refinement

Refinement on F
R = 0.042
wR = 0.046
S = 1.75
793 reflections
201 parameters
All H-atom parameters refined except for methyl H atoms, fixed

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

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

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

	x	y	z	$U_{\rm eq}$
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)
C1	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)
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)

N7—C1	1.431 (6)	C4—C15	1.514 (7)
N7—C8	1.232 (6)	C8—C9	1.471 (6)
C1—C2	1.390 (7)	C9—C10	1.389 (6)
C1—C6	1.395 (7)	C9—C14	1.391 (7)
C3—C4	1.378 (7)	C12—C13	1.368 (7)
C4—C5	1.364 (7)	C13—C14	1.360 (7)
C1—N7—C8	121.8 (5)	C3—C4—C5	118.4 (6)
N7—C1—C2	116.0 (5)	N7—C8—C9	122.3 (6)
N7—C1—C6	125.3 (5)	C11—C12—C13	120.3 (6)
C2—C1—C6	118.7 (6)		

Compound (2)

Crystal data

$C_{14}H_7F_4NO$
M_r = 281.21
Triclinic
$P\bar{1}$
a = 7.366 (7) Å
b = 12.411 (2) Å
c = 6.312 (2) Å
α = 100.13 (2)°
β = 94.28 (5)°
γ = 86.69 (4)°
V = 565.9 (5) Å^3
Z = 2
D_x = 1.650 Mg m^{-3}

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 18 reflections
θ = 76.90–79.37°
μ = 1.29 mm^{-1}
T = 295 K
Plate
0.60 × 0.40 × 0.03 mm
Yellow

Data collection

AFC-5*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
$T_{\min}$ = 0.70, $T_{\max}$ = 1.00
1827 measured reflections
1676 independent reflections
1167 observed reflections [$I > 3\sigma(I)$]

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

Refinement

Refinement on F
R = 0.078
wR = 0.088
S = 3.24
1167 reflections
181 parameters
H-atom parameters not refined

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

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (2)

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

	x	y	z	U_{eq}
F1	0.5355 (4)	0.1683 (3)	0.0975 (4)	0.067 (2)
F2	0.5470 (5)	0.3790 (3)	0.2984 (5)	0.080 (2)
F3	0.6969 (5)	0.4292 (3)	0.7037 (5)	0.082 (2)
F4	0.8312 (4)	0.2716 (3)	0.9212 (5)	0.070 (2)
O7	0.8400 (6)	−0.3828 (3)	0.3828 (6)	0.076 (2)
N10	0.8233 (6)	0.0552 (3)	0.7362 (6)	0.049 (2)
C1	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)
C4	0.7574 (7)	0.2430 (5)	0.7171 (8)	0.055 (3)
C4a	0.7561 (7)	0.1319 (4)	0.6229 (8)	0.047 (2)
C5	0.8929 (7)	−0.1323 (5)	0.7584 (8)	0.053 (3)
C6	0.8931 (8)	−0.2386 (5)	0.6672 (9)	0.058 (3)
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)
C8a	0.7512 (7)	−0.0846 (4)	0.4199 (8)	0.046 (2)
C9	0.6814 (7)	−0.0033 (4)	0.3060 (7)	0.048 (2)
C9a	0.6803 (6)	0.1055 (4)	0.4047 (8)	0.045 (2)
C10a	0.8216 (7)	−0.0490 (4)	0.6407 (8)	0.047 (2)
C71	0.7795 (10)	−0.4243 (5)	0.163 (1)	0.082 (4)

Table 4. *Selected geometric parameters* (Å, °) *for* (2)

O7—C7	1.359 (6)	C4a—C9a	1.437 (6)
O7—C71	1.439 (7)	C5—C6	1.343 (7)
N10—C4a	1.340 (6)	C5—C10a	1.431 (7)
N10—C10a	1.328 (6)	C6—C7	1.427 (7)
C1—C2	1.322 (7)	C7—C8	1.351 (7)
C1—C9a	1.417 (7)	C8—C8a	1.427 (7)
C2—C3	1.425 (8)	C8a—C9	1.393 (7)
C3—C4	1.339 (7)	C8a—C10a	1.452 (7)
C4—C4a	1.404 (7)	C9—C9a	1.384 (7)
C4a—N10—C10a	118.3 (4)	C8a—C9—C9a	119.9 (4)
N10—C4a—C9a	122.6 (5)	C4a—C9a—C9	118.5 (5)
C9—C8a—C10a	117.0 (5)	N10—C10a—C8a	123.6 (5)

Compound (3)

Crystal data

$C_{13}H_4F_5N$
$M_r = 269.17$
Triclinic
$P\bar{1}$
$a = 8.270\ (5)$ Å
$b = 10.721\ (5)$ Å
$c = 5.995\ (2)$ Å
$\alpha = 102.56\ (3)°$
$\beta = 92.60\ (4)°$
$\gamma = 83.30\ (3)°$
$V = 515.2\ (9)$ Å^3
$Z = 2$
$D_x = 1.735$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 35.55$-$40.85°$
$\mu = 0.159$ mm^{-1}
$T = 296$ K
Needle
0.30 × 0.20 × 0.20 mm
Yellow

Data collection

AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
$T_{\min} = 0.93$, $T_{\max} = 1.00$
1945 measured reflections
1808 independent reflections
951 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int} = 0.041$

$\theta_{\max} = 24.99°$
$h = 0 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -6 \rightarrow 6$
3 standard reflections monitored every 150 reflections intensity variation: −0.80%

Refinement

Refinement on F
$R = 0.045$
$wR = 0.053$
$S = 2.15$
951 reflections
188 parameters
All H-atom parameters refined

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

Table 5. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (3)

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

	x	y	z	U_{eq}
F1	0.0478 (3)	0.8677 (2)	0.8655 (4)	0.060 (1)
F2	0.0448 (3)	0.6227 (2)	0.6302 (5)	0.070 (1)
F3	0.1842 (3)	0.5441 (2)	0.2195 (5)	0.075 (1)
F4	0.3292 (3)	0.7085 (2)	0.0316 (4)	0.061 (1)
F5	0.3638 (3)	1.4606 (2)	0.6779 (5)	0.077 (1)
N10	0.3368 (4)	0.9583 (3)	0.2503 (5)	0.039 (1)
C1	0.1190 (5)	0.8285 (4)	0.6598 (7)	0.043 (2)
C2	0.1165 (5)	0.7070 (4)	0.5463 (8)	0.050 (2)
C3	0.1892 (5)	0.6666 (4)	0.3296 (7)	0.049 (2)
C4	0.2595 (5)	0.7489 (4)	0.2365 (7)	0.046 (2)
C4a	0.2656 (4)	0.8801 (4)	0.3516 (6)	0.039 (2)
C5	0.4125 (5)	1.1661 (4)	0.2548 (7)	0.045 (2)
C6	0.4187 (5)	1.2899 (4)	0.3590 (8)	0.049 (2)
C7	0.3515 (5)	1.3356 (4)	0.5780 (8)	0.051 (2)
C8	0.2808 (5)	1.2621 (4)	0.6903 (8)	0.047 (2)
C8a	0.2715 (4)	1.1310 (4)	0.5847 (6)	0.039 (2)
C9	0.1989 (5)	1.0464 (4)	0.6858 (8)	0.042 (2)
C9a	0.1931 (4)	0.9204 (4)	0.5722 (6)	0.038 (2)
C10a	0.3385 (4)	1.0812 (4)	0.3625 (6)	0.038 (2)

Table 6. *Selected geometric parameters* (Å, °) *for* (3)

N10—C4a	1.336 (4)	C5—C6	1.344 (6)
N10—C10a	1.345 (5)	C5—C10a	1.429 (5)
C1—C2	1.334 (5)	C6—C7	1.412 (6)
C1—C9a	1.421 (5)	C7—C8	1.338 (6)
C2—C3	1.415 (6)	C8—C8a	1.420 (6)
C3—C4	1.338 (5)	C8a—C9	1.397 (5)
C4—C4a	1.432 (5)	C8a—C10a	1.434 (5)
C4a—C9a	1.435 (5)	C9—C9a	1.379 (5)
C4a—N10—C10a	117.5 (3)	C8a—C9—C9a	120.1 (4)
N10—C4a—C9a	123.5 (3)	C4a—C9a—C9	117.9 (3)
C9—C8a—C10a	117.4 (4)	N10—C10a—C8a	123.5 (4)

Compound (4)

Crystal data

$C_{21}H_{15}F_3N_2$
$M_r = 352.36$
Monoclinic
$P2_1/c$
$a = 5.871\ (4)$ Å
$b = 7.889\ (3)$ Å
$c = 34.830\ (9)$ Å
$\beta = 91.23\ (4)°$
$V = 1612\ (2)$ Å^3
$Z = 4$
$D_x = 1.451$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 20 reflections
$\theta = 14.99$-$23.39°$
$\mu = 0.104$ mm^{-1}
$T = 296$ K
Plate
0.40 × 0.35 × 0.15 mm
Pale yellow

Data collection

AFC-6*S* diffractometer
ω scans with profile analysis

$R_{\rm int} = 0.070$
$\theta_{\max} = 25°$

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

$h = 0 \rightarrow 6$
$k = 0 \rightarrow 20$
$l = -40 \rightarrow 40$
3 standard reflections monitored every 150 reflections intensity variation: 1.09%

Refinement

Refinement on F
$R = 0.068$
$wR = 0.066$
$S = 4.25$
1317 reflections
283 parameters
All H-atom parameters refined except H on methyl C18, fixed

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

Table 7. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (4)

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

	x	y	z	$U_{\rm eq}$
F1	0.9691 (6)	0.0290 (5)	0.5589 (1)	0.050 (2)
F2	0.8437 (6)	0.0300 (6)	0.6321 (1)	0.052 (2)
F4	0.1739 (6)	0.3322 (6)	0.5990 (1)	0.046 (2)
N10	0.2821 (9)	0.3259 (8)	0.5232 (2)	0.034 (3)
N11	0.439 (1)	0.1846 (10)	0.6548 (2)	0.049 (4)
C1	0.772 (1)	0.1084 (10)	0.5679 (2)	0.037 (4)
C2	0.712 (1)	0.1089 (10)	0.6054 (2)	0.039 (4)
C3	0.507 (1)	0.191 (1)	0.6173 (2)	0.037 (4)
C4	0.372 (1)	0.2570 (9)	0.5884 (2)	0.029 (3)
C4a	0.426 (1)	0.2579 (9)	0.5487 (2)	0.033 (4)
C5	0.194 (1)	0.393 (1)	0.4576 (2)	0.037 (4)
C6	0.247 (1)	0.388 (1)	0.4193 (2)	0.044 (4)
C7	0.453 (1)	0.315 (1)	0.4060 (2)	0.041 (4)
C8	0.603 (1)	0.2495 (10)	0.4334 (2)	0.037 (4)
C8a	0.553 (1)	0.2499 (9)	0.4724 (2)	0.032 (4)
C9	0.696 (1)	0.1795 (10)	0.5006 (2)	0.034 (4)
C9a	0.637 (1)	0.1806 (9)	0.5387 (2)	0.032 (4)
C10a	0.341 (1)	0.3228 (10)	0.4855 (2)	0.033 (4)
C12	0.580 (1)	0.2243 (10)	0.6867 (2)	0.039 (4)
C13	0.785 (1)	0.309 (1)	0.6846 (3)	0.046 (5)
C14	0.918 (1)	0.345 (1)	0.7160 (3)	0.046 (4)
C15	0.853 (1)	0.295 (1)	0.7528 (2)	0.042 (4)
C16	0.646 (1)	0.211 (1)	0.7553 (2)	0.050 (5)
C17	0.512 (1)	0.175 (1)	0.7235 (2)	0.043 (4)
C18	1.002 (1)	0.330 (1)	0.7882 (3)	0.069 (5)
C19	0.507 (2)	0.317 (2)	0.3649 (3)	0.066 (6)

Table 8. *Selected geometric parameters* (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)
C1—C9a	1.401 (9)	C8—C8a	1.40 (1)
C2—C3	1.43 (1)	C8a—C9	1.394 (9)
C3—C4	1.372 (9)	C8a—C10a	1.449 (9)
C4—C4a	1.421 (8)	C9—C9a	1.379 (9)
C4a—C9a	1.432 (9)		
C4a—N10—C10a	117.8 (6)	C8a—C9—C9a	120.9 (7)
N10—C4a—C9a	123.3 (7)	C4a—C9a—C9	118.4 (6)
C9—C8a—C10a	116.4 (7)	N10—C10a—C8a	123.1 (6)

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

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3-Selenocyanatopyridine

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(*Received* 11 *October* 1993; *accepted* 13 *December* 1993)

Abstract

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

Acta Crystallographica Section C
ISSN 0108-2701 ©1994