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The (1*S*,9a*R*) and (1*S*,9a*S*) Relative Configurations of 3-Fluoro-1,9a-dihydro-1-phenyl-1,2-bis(trifluoromethyl)-4*H*-quinolizin-4-one

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Abstract

The cycloaddition reaction of (*Z*)-1,1,2,5,5,5-hexafluoro-4-phenyl-3-trifluoromethyl-1,3-pentadiene (1) with pyridine gave a major and minor product. Crystal structures identified the major product to be the racemate of the diastereomer (1*S*,9aR)-3-fluoro-9a-hydro-1,2-bistrifluoromethyl-1- phenyl-4*H*-quinolizin-4-one (2), ($C_{17}H_{10}F_7NO$), and the minor product to be the racemate of the diastereomer (1*S*,9aS)- 3-fluoro-9a-hydro-1,2-bistrifluoromethyl-1-phenyl-4*H*-quinolizin-4-one (3), ($C_{17}H_{10}F_7NO$).

Comment

(*Z*)-1,1,2,5,5,5-Hexafluoro-4-phenyl-3-trifluoromethyl-1,3-pentadiene (1) can be prepared in several steps from perfluorovinyl bromide (Hansen, 1984). The reaction of (1) with pyridine gives a unique entry to 4-quinolizone derivatives (Yamamoto, Burton & Swenson, 1994). Two isomers (2) and (3) were isolated from the reaction in yields of 54% and 21%, respectively. The crystal structures of these isomers were done to confirm the structures and establish the relative configurations of the subject compounds. The major (2) and minor (3) products are the rac-(1*S*,9aR) and rac-(1*S*,9aS) diastereomers of 3-fluoro-9a-hydro-1,2-bistrifluoromethyl-1-phenyl-4*H*-quinolizin-4-one, respectively.

Experimental

Compounds (2) and (3) were prepared and crystallized as described in Yamamoto, Burton & Swenson, (1995).

Refinement

Hydrogen atoms in compound (3) were included using the riding model with default parameters.

Computing details

For both compounds, data collection: CAD4 (Enraf-Nonius, 1977); cell refinement: CAD4 (Enraf-Nonius, 1977); data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: direct methods (*MULTAN*, Main, *et al.*,1980); program(s) used to refine structure: *SHELXTL* v5.0 (Sheldrick, 1995); molecular graphics: *SHELXTL* v5.0 (Sheldrick, 1995); software used to prepare material for publication: *SHELXTL* v5.0 (Sheldrick, 1995)

(2)

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Crystal data

$C_{17}H_{10}F_7NO$	$V = 1557.0\ (7)\ Å^3$
$M_r = 377.26$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$
$a = 7.533\ (2)$ Å	$\mu = 0.16\ mm^{-1}$
$b = 10.306\ (3)$ Å	$T = 293\ (2)$ K
$c = 20.139\ (5)$ Å	0.46 × 0.41 × 0.35 mm
$\beta = 95.24\ (2)^o$	

Data collection

Enraf Nonius CAD4 diffractometer	$R_{int} = 0.034$
Absorption correction: none	4 standard reflections
8055 measured reflections	every 240 min
3808 independent reflections	intensity decay: none
2291 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$	236 parameters
$wR(F^2) = 0.127$	Riding
$S = 1.03$	$\Delta\rho_{max} = 0.27\ e\ Å^{-3}$
3808 reflections	$\Delta\rho_{min} = -0.17\ e\ Å^{-3}$

Table 1
Selected geometric parameters (Å, °)

C1—C2	1.529 (3)	C7—C8	1.317 (3)
C1—C12	1.535 (2)	C8—C9	1.503 (3)
C1—C10	1.558 (2)	C10—F10A	1.332 (2)
C1—C9	1.592 (2)	C10—F10B	1.333 (2)
C2—C3	1.325 (3)	C10—F10C	1.343 (3)
C2—C11	1.529 (3)	C11—F11B	1.326 (3)
C3—F3	1.341 (2)	C11—F11A	1.327 (3)
C3—C4	1.486 (3)	C11—F11C	1.329 (3)
C4—O4	1.220 (2)	C12—C17	1.392 (3)
C4—N	1.364 (2)	C12—C13	1.393 (2)
N—C5	1.409 (2)	C13—C14	1.383 (3)
N—C9	1.484 (2)	C14—C15	1.381 (3)
C5—C6	1.328 (3)	C15—C16	1.369 (3)
C6—C7	1.433 (3)	C16—C17	1.379 (3)
C2—C1—C12	111.40 (13)	N—C9—C1	112.98 (13)
C2—C1—C10	106.14 (15)	C8—C9—C1	112.38 (15)
C12—C1—C10	113.84 (15)	F10A—C10—F10B	106.4 (2)
C2—C1—C9	110.29 (14)	F10A—C10—F10C	106.6 (2)
C12—C1—C9	111.38 (13)	F10B—C10—F10C	106.2 (2)

C10—C1—C9	103.40 (13)	F10A—C10—C1	113.7 (2)
C3—C2—C1	120.0 (2)	F10B—C10—C1	113.2 (2)
C3—C2—C11	118.3 (2)	F10C—C10—C1	110.3 (2)
C1—C2—C11	121.8 (2)	F11B—C11—F11A	106.4 (2)
C2—C3—F3	121.9 (2)	F11B—C11—F11C	107.3 (2)
C2—C3—C4	126.2 (2)	F11A—C11—F11C	107.0 (2)
F3—C3—C4	111.3 (2)	F11B—C11—C2	111.8 (2)
O4—C4—N	124.1 (2)	F11A—C11—C2	112.7 (2)
O4—C4—C3	121.3 (2)	F11C—C11—C2	111.3 (2)
N—C4—C3	114.3 (2)	C17—C12—C13	118.1 (2)
C4—N—C5	118.1 (2)	C17—C12—C1	124.35 (15)
C4—N—C9	119.54 (14)	C13—C12—C1	117.41 (15)
C5—N—C9	120.7 (2)	C14—C13—C12	120.4 (2)
C6—C5—N	122.0 (2)	C15—C14—C13	120.7 (2)
C5—C6—C7	119.5 (2)	C16—C15—C14	119.2 (2)
C8—C7—C6	120.9 (2)	C15—C16—C17	120.8 (2)
C7—C8—C9	123.5 (2)	C16—C17—C12	120.8 (2)
N—C9—C8	110.06 (15)		

(3)

Crystal data

$C_{17}H_{10}F_7NO$	γ = 88.36 (3)°
M_r = 377.26	V = 762.8 (5) Å^3
Triclinic, $P\overline{1}$	Z = 2
a = 8.543 (3) Å	Mo $K\alpha$
b = 12.760 (4) Å	μ = 0.16 mm^{-1}
c = 7.800 (3) Å	T = 293 (2) K
α = 98.39 (3)°	0.36 × 0.13 × 0.05 mm
β = 114.84 (3)°	

Data collection

Enraf-Nonius CAD4 diffractometer	R_{int} = 0.063
Absorption correction: none	4 standard reflections
3375 measured reflections	every 240 min
2506 independent reflections	intensity decay: 1%
1149 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059	236 parameters
$wR(F^2)$ = 0.186	Riding
S = 0.98	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
2648 reflections	$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 2
Selected geometric parameters (Å, °)

C1—C2	1.518 (7)	C7—C8	1.319 (7)
C1—C12	1.532 (6)	C8—C9	1.477 (7)
C1—C10	1.538 (7)	C10—F10B	1.327 (6)
C1—C9	1.577 (6)	C10—F10A	1.337 (5)
C2—C3	1.312 (6)	C10—F10C	1.347 (5)
C2—C11	1.505 (7)	C11—F11A	1.308 (6)
C3—F3	1.342 (5)	C11—F11B	1.313 (7)
C3—C4	1.486 (7)	C11—F11C	1.358 (6)
C4—O4	1.214 (5)	C12—C17	1.371 (7)
C4—N	1.356 (6)	C12—C13	1.381 (8)
N—C5	1.421 (6)	C13—C14	1.396 (7)
N—C9	1.492 (6)	C14—C15	1.348 (9)
C5—C6	1.296 (7)	C15—C16	1.351 (10)
C6—C7	1.432 (7)	C16—C17	1.429 (9)
C2—C1—C12	111.7 (4)	C8—C9—C1	114.0 (4)
C2—C1—C10	105.7 (4)	N—C9—C1	113.1 (3)
C12—C1—C10	114.3 (4)	F10B—C10—F10A	106.0 (4)
C2—C1—C9	109.3 (4)	F10B—C10—F10C	105.6 (4)
C12—C1—C9	105.1 (4)	F10A—C10—F10C	105.4 (4)
C10—C1—C9	110.6 (4)	F10B—C10—C1	115.3 (4)
C3—C2—C11	118.4 (5)	F10A—C10—C1	111.8 (4)
C3—C2—C1	118.8 (4)	F10C—C10—C1	112.0 (4)
C11—C2—C1	122.8 (4)	F11A—C11—F11B	107.7 (5)
C2—C3—F3	120.8 (4)	F11A—C11—F11C	105.5 (5)
C2—C3—C4	128.3 (4)	F11B—C11—F11C	105.6 (5)
F3—C3—C4	110.8 (4)	F11A—C11—C2	114.9 (5)
O4—C4—N	125.4 (5)	F11B—C11—C2	112.4 (5)
O4—C4—C3	120.7 (5)	F11C—C11—C2	110.1 (4)
N—C4—C3	113.9 (4)	C17—C12—C13	118.8 (5)
C4—N—C5	118.3 (4)	C17—C12—C1	124.6 (5)
C4—N—C9	120.6 (4)	C13—C12—C1	116.6 (5)
C5—N—C9	119.2 (4)	C12—C13—C14	121.5 (5)
C6—C5—N	121.1 (5)	C15—C14—C13	119.4 (6)
C5—C6—C7	121.3 (5)	C14—C15—C16	120.7 (6)
C8—C7—C6	119.7 (5)	C15—C16—C17	120.7 (6)
C7—C8—C9	123.4 (5)	C12—C17—C16	118.8 (6)
C8—C9—N	110.8 (4)		

Acknowledgements

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References

Enraf-Nonius, CAD4 Operations Manual, Delft, 1977.
Fair, C. K. (1990). *MolEN*, An Interactive Intelligent System for Crystal Structure Analysis, User Manual, Enraf - Nonius, Delft, The Netherlands.
Hansen, S.W., Ph.D. Thesis, University of Iowa, 1984.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., DeClercq, J. P. & Woolfson, M. M. (1980). *MULTAN80*. University of York, England.
Sheldrick, G.M., *SHELXTL* v5.0, Siemens Analytical X-ray Instruments,Inc., 1995, Madison, WI, USA.
Yamamoto, M., Burton, D. J. & Swenson, D. C. (1995). *J. Fluorine Chem.* **72**, 49–54.

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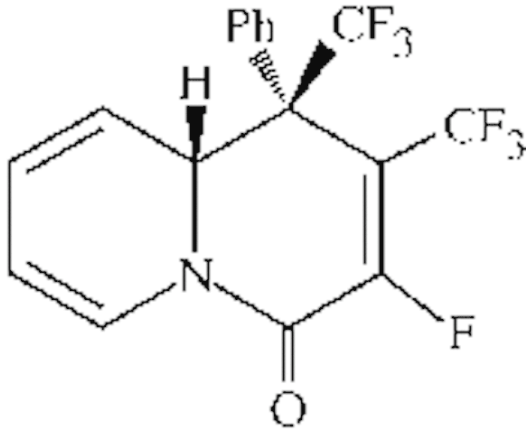
Scheme 1



supplementary materials

(2)

Crystal data

$C_{17}H_{10}F_7NO$	F_{000} = 760
M_r = 377.26	D_x = 1.609 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71070 Å
Hall symbol: -P 2ybc	Cell parameters from 25 reflections
a = 7.533 (2) Å	θ = 8–19º
b = 10.306 (3) Å	μ = 0.16 mm^{-1}
c = 20.139 (5) Å	T = 293 (2) K
β = 95.24 (2)º	Prism, yellow
V = 1557.0 (7) Å^3	0.46 × 0.41 × 0.35 mm
Z = 4	

Data collection

Enraf Nonius CAD4 diffractometer	R_{int} = 0.034
Radiation source: fine-focus sealed tube	θ_{max} = 30.0º
Monochromator: graphite	θ_{min} = 2.0º
T = 293(2) K	h = −10→10
θ/2θ scans	k = −1→9
Absorption correction: none	l = −28→28
8055 measured reflections	4 standard reflections
3808 independent reflections	every 240 min
2291 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.038	Riding
$wR(F^2)$ = 0.127	Calculated $w = 1/[\sigma^2(F_o^2) + (0.0398P)^2 + 0.423P]$ where $P = (F_o^2 + 2F_c^2)/3$?
S = 1.03	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
3808 reflections	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
236 parameters	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0087 (14)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement on F^2 for ALL reflections except for 0 with very negative F^2 or flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factor-obs *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6635 (2)	−0.1996 (2)	0.37095 (7)	0.0411 (4)
C2	0.7840 (2)	−0.0840 (2)	0.35939 (9)	0.0480 (4)
C3	0.8094 (2)	0.0077 (2)	0.40535 (10)	0.0530 (5)
F3	0.8993 (2)	0.11686 (12)	0.39455 (8)	0.0801 (4)
C4	0.7705 (2)	−0.0028 (2)	0.47620 (9)	0.0492 (5)
O4	0.8308 (2)	0.07524 (15)	0.51793 (8)	0.0682 (4)
N	0.6793 (2)	−0.11206 (15)	0.49061 (7)	0.0442 (4)
C5	0.6629 (3)	−0.1410 (2)	0.55815 (9)	0.0576 (5)
H5	0.7181	−0.0872	0.59088	0.069*
C6	0.5710 (3)	−0.2428 (3)	0.57646 (10)	0.0698 (6)
H6	0.5594	−0.2583	0.62135	0.084*
C7	0.4899 (3)	−0.3286 (2)	0.52656 (11)	0.0673 (6)
H7	0.4340	−0.4038	0.53947	0.081*
C8	0.4932 (3)	−0.3025 (2)	0.46266 (10)	0.0578 (5)
H8	0.4485	−0.3643	0.43188	0.069*
C9	0.5655 (2)	−0.1783 (2)	0.43686 (8)	0.0425 (4)
H9	0.4632	−0.1214	0.42496	0.051*
C10	0.5082 (3)	−0.1943 (2)	0.31429 (10)	0.0606 (5)
F10A	0.5567 (2)	−0.2278 (2)	0.25467 (6)	0.0853 (5)
F10B	0.3716 (2)	−0.27076 (14)	0.32569 (6)	0.0777 (4)
F10C	0.4423 (2)	−0.07343 (14)	0.30811 (6)	0.0803 (4)
C11	0.8802 (3)	−0.0715 (3)	0.29602 (11)	0.0687 (6)
F11A	0.9004 (2)	−0.1847 (2)	0.26625 (7)	0.0907 (5)
F11B	1.0422 (2)	−0.0220 (2)	0.30857 (8)	0.1045 (6)
F11C	0.7913 (2)	0.0055 (2)	0.25165 (8)	0.1140 (6)
C12	0.7680 (2)	−0.3276 (2)	0.37366 (7)	0.0405 (4)
C13	0.9290 (2)	−0.3312 (2)	0.41361 (9)	0.0476 (4)
H13	0.9719	−0.2564	0.43540	0.057*
C14	1.0252 (3)	−0.4453 (2)	0.42106 (10)	0.0581 (5)
H14	1.1320	−0.4468	0.44813	0.070*
C15	0.9643 (3)	−0.5571 (2)	0.38870 (11)	0.0615 (5)

H15	1.0300	−0.6335	0.39356	0.074*
C16	0.8062 (3)	−0.5542 (2)	0.34941 (10)	0.0603 (5)
H16	0.7646	−0.6292	0.32747	0.072*
C17	0.7080 (3)	−0.4415 (2)	0.34198 (9)	0.0527 (5)
H17	0.6002	−0.4415	0.31547	0.063*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0423 (8)	0.0459 (11)	0.0345 (7)	0.0006 (7)	0.0008 (6)	−0.0010 (7)
C2	0.0483 (9)	0.0480 (12)	0.0483 (9)	0.0062 (8)	0.0074 (7)	0.0097 (8)
C3	0.0474 (9)	0.0364 (12)	0.0759 (13)	−0.0005 (8)	0.0102 (8)	0.0089 (9)
F3	0.0811 (9)	0.0435 (8)	0.1186 (11)	−0.0116 (6)	0.0244 (8)	0.0107 (7)
C4	0.0425 (8)	0.0409 (12)	0.0642 (11)	0.0047 (8)	0.0037 (8)	−0.0114 (9)
O4	0.0598 (8)	0.0548 (10)	0.0891 (10)	−0.0033 (7)	0.0024 (7)	−0.0307 (8)
N	0.0468 (8)	0.0428 (9)	0.0431 (7)	0.0015 (7)	0.0041 (6)	−0.0074 (6)
C5	0.0629 (11)	0.0684 (15)	0.0409 (9)	0.0107 (10)	0.0018 (8)	−0.0104 (9)
C6	0.0841 (15)	0.079 (2)	0.0481 (11)	0.0059 (13)	0.0178 (10)	0.0104 (11)
C7	0.0807 (14)	0.0563 (14)	0.0694 (13)	−0.0059 (12)	0.0319 (11)	0.0031 (11)
C8	0.0631 (12)	0.0545 (14)	0.0589 (11)	−0.0150 (10)	0.0226 (9)	−0.0110 (9)
C9	0.0398 (8)	0.0454 (11)	0.0424 (8)	0.0000 (7)	0.0047 (6)	−0.0054 (7)
C10	0.0577 (11)	0.073 (2)	0.0486 (10)	0.0090 (11)	−0.0074 (8)	−0.0028 (10)
F10A	0.0958 (10)	0.1181 (12)	0.0388 (6)	0.0182 (9)	−0.0112 (6)	−0.0083 (7)
F10B	0.0539 (7)	0.0959 (10)	0.0791 (8)	−0.0084 (7)	−0.0165 (6)	−0.0130 (8)
F10C	0.0770 (8)	0.0840 (10)	0.0751 (8)	0.0261 (7)	−0.0186 (6)	0.0088 (7)
C11	0.0754 (14)	0.072 (2)	0.0622 (12)	0.0071 (12)	0.0228 (10)	0.0204 (12)
F11A	0.1170 (12)	0.0953 (11)	0.0668 (8)	0.0081 (9)	0.0464 (8)	0.0031 (8)
F11B	0.0820 (10)	0.1320 (14)	0.1062 (11)	−0.0215 (10)	0.0457 (8)	0.0083 (10)
F11C	0.1369 (14)	0.1274 (14)	0.0820 (10)	0.0390 (12)	0.0344 (9)	0.0611 (10)
C12	0.0430 (8)	0.0440 (11)	0.0349 (7)	−0.0008 (7)	0.0062 (6)	−0.0029 (7)
C13	0.0471 (9)	0.0404 (11)	0.0542 (10)	−0.0014 (8)	−0.0008 (7)	−0.0030 (8)
C14	0.0497 (10)	0.0549 (14)	0.0683 (12)	0.0018 (9)	−0.0017 (8)	0.0056 (10)
C15	0.0657 (13)	0.0443 (13)	0.0763 (13)	0.0076 (10)	0.0165 (10)	0.0002 (10)
C16	0.0716 (13)	0.0464 (13)	0.0638 (12)	−0.0035 (10)	0.0105 (10)	−0.0164 (10)
C17	0.0558 (10)	0.0546 (13)	0.0473 (9)	−0.0034 (9)	0.0022 (8)	−0.0126 (9)

Geometric parameters (Å, °)

C1—C2	1.529 (3)	C8—H8	0.93
C1—C12	1.535 (2)	C9—H9	0.98
C1—C10	1.558 (2)	C10—F10A	1.332 (2)
C1—C9	1.592 (2)	C10—F10B	1.333 (2)
C2—C3	1.325 (3)	C10—F10C	1.343 (3)
C2—C11	1.529 (3)	C11—F11B	1.326 (3)
C3—F3	1.341 (2)	C11—F11A	1.327 (3)
C3—C4	1.486 (3)	C11—F11C	1.329 (3)
C4—O4	1.220 (2)	C12—C17	1.392 (3)
C4—N	1.364 (2)	C12—C13	1.393 (2)
N—C5	1.409 (2)	C13—C14	1.383 (3)

N—C9	1.484 (2)	C13—H13	0.93
C5—C6	1.328 (3)	C14—C15	1.381 (3)
C5—H5	0.93	C14—H14	0.93
C6—C7	1.433 (3)	C15—C16	1.369 (3)
C6—H6	0.93	C15—H15	0.93
C7—C8	1.317 (3)	C16—C17	1.379 (3)
C7—H7	0.93	C16—H16	0.93
C8—C9	1.503 (3)	C17—H17	0.93
C2—C1—C12	111.40 (13)	N—C9—H9	107.01 (9)
C2—C1—C10	106.14 (15)	C8—C9—H9	107.01 (10)
C12—C1—C10	113.84 (15)	C1—C9—H9	107.01 (9)
C2—C1—C9	110.29 (14)	F10A—C10—F10B	106.4 (2)
C12—C1—C9	111.38 (13)	F10A—C10—F10C	106.6 (2)
C10—C1—C9	103.40 (13)	F10B—C10—F10C	106.2 (2)
C3—C2—C1	120.0 (2)	F10A—C10—C1	113.7 (2)
C3—C2—C11	118.3 (2)	F10B—C10—C1	113.2 (2)
C1—C2—C11	121.8 (2)	F10C—C10—C1	110.3 (2)
C2—C3—F3	121.9 (2)	F11B—C11—F11A	106.4 (2)
C2—C3—C4	126.2 (2)	F11B—C11—F11C	107.3 (2)
F3—C3—C4	111.3 (2)	F11A—C11—F11C	107.0 (2)
O4—C4—N	124.1 (2)	F11B—C11—C2	111.8 (2)
O4—C4—C3	121.3 (2)	F11A—C11—C2	112.7 (2)
N—C4—C3	114.3 (2)	F11C—C11—C2	111.3 (2)
C4—N—C5	118.1 (2)	C17—C12—C13	118.1 (2)
C4—N—C9	119.54 (14)	C17—C12—C1	124.35 (15)
C5—N—C9	120.7 (2)	C13—C12—C1	117.41 (15)
C6—C5—N	122.0 (2)	C14—C13—C12	120.4 (2)
C6—C5—H5	119.02 (12)	C14—C13—H13	119.82 (11)
N—C5—H5	119.02 (11)	C12—C13—H13	119.82 (11)
C5—C6—C7	119.5 (2)	C15—C14—C13	120.7 (2)
C5—C6—H6	120.24 (12)	C15—C14—H14	119.65 (12)
C7—C6—H6	120.24 (12)	C13—C14—H14	119.65 (11)
C8—C7—C6	120.9 (2)	C16—C15—C14	119.2 (2)
C8—C7—H7	119.53 (14)	C16—C15—H15	120.39 (13)
C6—C7—H7	119.53 (13)	C14—C15—H15	120.39 (12)
C7—C8—C9	123.5 (2)	C15—C16—C17	120.8 (2)
C7—C8—H8	118.26 (14)	C15—C16—H16	119.62 (13)
C9—C8—H8	118.26 (10)	C17—C16—H16	119.62 (12)
N—C9—C8	110.06 (15)	C16—C17—C12	120.8 (2)
N—C9—C1	112.98 (13)	C16—C17—H17	119.59 (11)
C8—C9—C1	112.38 (15)	C12—C17—H17	119.59 (11)
C12—C1—C2—C3	−116.9 (2)	C2—C1—C9—C8	−161.77 (15)
C10—C1—C2—C3	118.6 (2)	C12—C1—C9—C8	−37.5 (2)
C9—C1—C2—C3	7.3 (2)	C10—C1—C9—C8	85.1 (2)
C12—C1—C2—C11	62.1 (2)	C2—C1—C10—F10A	72.6 (2)
C10—C1—C2—C11	−62.3 (2)	C12—C1—C10—F10A	−50.3 (2)
C9—C1—C2—C11	−173.7 (2)	C9—C1—C10—F10A	−171.3 (2)
C1—C2—C3—F3	−173.2 (2)	C2—C1—C10—F10B	−165.9 (2)

C11—C2—C3—F3	7.7 (3)	C12—C1—C10—F10B	71.2 (2)
C1—C2—C3—C4	16.8 (3)	C9—C1—C10—F10B	−49.8 (2)
C11—C2—C3—C4	−162.3 (2)	C2—C1—C10—F10C	−47.1 (2)
C2—C3—C4—O4	164.8 (2)	C12—C1—C10—F10C	−170.00 (15)
F3—C3—C4—O4	−6.1 (2)	C9—C1—C10—F10C	69.0 (2)
C2—C3—C4—N	−9.0 (3)	C3—C2—C11—F11B	36.7 (3)
F3—C3—C4—N	−179.88 (14)	C1—C2—C11—F11B	−142.3 (2)
O4—C4—N—C5	−4.0 (3)	C3—C2—C11—F11A	156.6 (2)
C3—C4—N—C5	169.5 (2)	C1—C2—C11—F11A	−22.5 (3)
O4—C4—N—C9	161.3 (2)	C3—C2—C11—F11C	−83.2 (3)
C3—C4—N—C9	−25.1 (2)	C1—C2—C11—F11C	97.7 (2)
C4—N—C5—C6	177.5 (2)	C2—C1—C12—C17	−136.1 (2)
C9—N—C5—C6	12.4 (3)	C10—C1—C12—C17	−16.2 (2)
N—C5—C6—C7	1.9 (3)	C9—C1—C12—C17	100.3 (2)
C5—C6—C7—C8	−5.4 (3)	C2—C1—C12—C13	47.6 (2)
C6—C7—C8—C9	−5.6 (3)	C10—C1—C12—C13	167.6 (2)
C4—N—C9—C8	174.46 (14)	C9—C1—C12—C13	−76.0 (2)
C5—N—C9—C8	−20.6 (2)	C17—C12—C13—C14	0.1 (3)
C4—N—C9—C1	47.9 (2)	C1—C12—C13—C14	176.6 (2)
C5—N—C9—C1	−147.1 (2)	C12—C13—C14—C15	0.5 (3)
C7—C8—C9—N	17.7 (3)	C13—C14—C15—C16	−0.5 (3)
C7—C8—C9—C1	144.5 (2)	C14—C15—C16—C17	0.0 (3)
C2—C1—C9—N	−36.5 (2)	C15—C16—C17—C12	0.7 (3)
C12—C1—C9—N	87.7 (2)	C13—C12—C17—C16	−0.7 (3)
C10—C1—C9—N	−149.6 (2)	C1—C12—C17—C16	−176.9 (2)

(3)

Crystal data

$C_{17}H_{10}F_7NO$	$Z = 2$
$M_r = 377.26$	$F_{000} = 380$
Triclinic, $P\overline{1}$	$D_x = 1.642$ Mg m^{-3}
$a = 8.543$ (3) Å	Mo $K\alpha$ radiation $\lambda = 0.71070$ Å
$b = 12.760$ (4) Å	Cell parameters from 30 reflections
$c = 7.800$ (3) Å	$\theta = 10.0$–13.0º
$\alpha = 98.39$ (3)º	$\mu = 0.16$ mm^{-1}
$\beta = 114.84$ (3)º	$T = 293$ (2) K
$\gamma = 88.36$ (3)º	Needle, orange
$V = 762.8$ (5) Å^3	0.36 × 0.13 × 0.05 mm

Data collection

Enraf-Nonius CAD4 diffractometer	$R_{int} = 0.063$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.0$º
Monochromator: graphite	$\theta_{min} = 2.5$º
$T = 293$(2) K	$h = -10 \rightarrow 9$

θ/2θ scans
Absorption correction: none
3375 measured reflections
2506 independent reflections
1149 reflections with $I > 2\sigma(I)$
$k = -15 \rightarrow 15$
$l = -1 \rightarrow 9$
4 standard reflections
every 240 min
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.186$
$S = 0.98$
2648 reflections
236 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
Riding
Calculated $w = 1/[\sigma^2(F_o^2) + (0.0878P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$?
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0063 (45)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement on F^2 for ALL reflections except for 0 with very negative F^2 or flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factor-obs *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2065 (6)	0.7412 (4)	0.9650 (7)	0.0362 (13)
C2	0.0446 (6)	0.7425 (4)	1.0002 (6)	0.0336 (12)
C3	−0.0586 (6)	0.6578 (4)	0.9358 (7)	0.0357 (12)
F3	−0.2043 (4)	0.6560 (2)	0.9612 (5)	0.0577 (10)
C4	−0.0438 (6)	0.5576 (4)	0.8208 (7)	0.0379 (12)
O4	−0.1590 (4)	0.4898 (3)	0.7548 (5)	0.0540 (11)
N	0.1070 (5)	0.5507 (3)	0.8017 (6)	0.0376 (10)
C5	0.1332 (7)	0.4583 (4)	0.6922 (7)	0.0464 (14)
H5	0.0471	0.4051	0.6355	0.056*
C6	0.2751 (7)	0.4471 (4)	0.6701 (7)	0.0498 (15)
H6	0.2917	0.3842	0.6038	0.060*

C7	0.4069 (7)	0.5295 (4)	0.7458 (8)	0.0466 (14)
H7	0.4983	0.5255	0.7103	0.056*
C8	0.3981 (6)	0.6111 (4)	0.8662 (8)	0.0426 (13)
H8	0.4847	0.6639	0.9131	0.051*
C9	0.2578 (5)	0.6229 (3)	0.9304 (7)	0.0349 (12)
H9	0.3027	0.5981	1.0549	0.042*
C10	0.1590 (7)	0.7938 (4)	0.7850 (8)	0.0438 (14)
F10A	0.0247 (4)	0.7435 (2)	0.6356 (4)	0.0588 (9)
F10B	0.2840 (4)	0.7990 (3)	0.7274 (5)	0.0645 (10)
F10C	0.1108 (4)	0.8944 (2)	0.8090 (4)	0.0600 (9)
C11	−0.0040 (7)	0.8374 (5)	1.1041 (8)	0.0487 (15)
F11A	0.0999 (4)	0.9207 (2)	1.1547 (5)	0.0655 (10)
F11B	−0.0171 (5)	0.8161 (3)	1.2573 (5)	0.0771 (12)
F11C	−0.1620 (4)	0.8696 (3)	0.9906 (5)	0.0713 (11)
C12	0.3617 (6)	0.7941 (4)	1.1408 (7)	0.0404 (13)
C13	0.3911 (6)	0.7624 (4)	1.3133 (8)	0.0486 (15)
H13	0.3174	0.7112	1.3167	0.058*
C14	0.5288 (7)	0.8052 (5)	1.4826 (8)	0.062 (2)
H14	0.5475	0.7821	1.5971	0.075*
C15	0.6346 (8)	0.8803 (6)	1.4786 (10)	0.071 (2)
H15	0.7251	0.9101	1.5916	0.085*
C16	0.6105 (8)	0.9130 (5)	1.3124 (12)	0.080 (2)
H16	0.6865	0.9634	1.3116	0.096*
C17	0.4695 (7)	0.8707 (5)	1.1378 (9)	0.059 (2)
H17	0.4512	0.8947	1.0241	0.071*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.031 (3)	0.032 (3)	0.035 (3)	−0.003 (2)	0.006 (2)	−0.001 (2)
C2	0.027 (3)	0.036 (3)	0.031 (3)	0.004 (2)	0.006 (2)	0.004 (2)
C3	0.029 (3)	0.043 (3)	0.039 (3)	0.002 (2)	0.017 (2)	0.008 (2)
F3	0.037 (2)	0.071 (2)	0.066 (2)	−0.0032 (15)	0.023 (2)	0.004 (2)
C4	0.035 (3)	0.035 (3)	0.039 (3)	0.001 (2)	0.009 (2)	0.013 (2)
O4	0.045 (2)	0.051 (2)	0.052 (2)	−0.016 (2)	0.006 (2)	0.003 (2)
N	0.034 (2)	0.036 (2)	0.033 (2)	0.003 (2)	0.006 (2)	0.001 (2)
C5	0.050 (3)	0.043 (3)	0.028 (3)	0.005 (3)	0.001 (3)	−0.002 (2)
C6	0.054 (3)	0.044 (3)	0.040 (3)	0.013 (3)	0.013 (3)	−0.002 (3)
C7	0.040 (3)	0.050 (3)	0.047 (3)	0.017 (3)	0.015 (3)	0.011 (3)
C8	0.030 (3)	0.047 (3)	0.045 (3)	0.008 (2)	0.010 (3)	0.009 (3)
C9	0.025 (2)	0.034 (3)	0.032 (3)	0.002 (2)	0.000 (2)	0.004 (2)
C10	0.043 (3)	0.039 (3)	0.049 (4)	0.000 (3)	0.018 (3)	0.010 (3)
F10A	0.061 (2)	0.058 (2)	0.036 (2)	−0.005 (2)	−0.001 (2)	0.0085 (15)
F10B	0.066 (2)	0.071 (2)	0.071 (2)	0.010 (2)	0.038 (2)	0.026 (2)
F10C	0.069 (2)	0.041 (2)	0.063 (2)	0.012 (2)	0.019 (2)	0.017 (2)
C11	0.035 (3)	0.057 (4)	0.048 (4)	0.009 (3)	0.013 (3)	0.004 (3)
F11A	0.054 (2)	0.044 (2)	0.080 (2)	0.002 (2)	0.018 (2)	−0.014 (2)
F11B	0.095 (3)	0.086 (3)	0.060 (2)	0.016 (2)	0.046 (2)	0.001 (2)

F11C	0.043 (2)	0.066 (2)	0.085 (3)	0.022 (2)	0.012 (2)	0.002 (2)
C12	0.030 (3)	0.040 (3)	0.040 (3)	−0.001 (2)	0.008 (2)	−0.005 (2)
C13	0.035 (3)	0.051 (3)	0.048 (4)	−0.001 (2)	0.010 (3)	−0.003 (3)
C14	0.043 (3)	0.080 (4)	0.041 (4)	0.003 (3)	0.002 (3)	−0.009 (3)
C15	0.039 (4)	0.081 (5)	0.064 (5)	−0.004 (3)	0.004 (3)	−0.017 (4)
C16	0.047 (4)	0.063 (4)	0.105 (6)	−0.030 (3)	0.015 (4)	−0.019 (4)
C17	0.048 (3)	0.057 (4)	0.063 (4)	−0.014 (3)	0.017 (3)	−0.001 (3)

Geometric parameters (Å, °)

C1—C2	1.518 (7)	C8—H8	0.93
C1—C12	1.532 (6)	C9—H9	0.98
C1—C10	1.538 (7)	C10—F10B	1.327 (6)
C1—C9	1.577 (6)	C10—F10A	1.337 (5)
C2—C3	1.312 (6)	C10—F10C	1.347 (5)
C2—C11	1.505 (7)	C11—F11A	1.308 (6)
C3—F3	1.342 (5)	C11—F11B	1.313 (7)
C3—C4	1.486 (7)	C11—F11C	1.358 (6)
C4—O4	1.214 (5)	C12—C17	1.371 (7)
C4—N	1.356 (6)	C12—C13	1.381 (8)
N—C5	1.421 (6)	C13—C14	1.396 (7)
N—C9	1.492 (6)	C13—H13	0.93
C5—C6	1.296 (7)	C14—C15	1.348 (9)
C5—H5	0.93	C14—H14	0.93
C6—C7	1.432 (7)	C15—C16	1.351 (10)
C6—H6	0.93	C15—H15	0.93
C7—C8	1.319 (7)	C16—C17	1.429 (9)
C7—H7	0.93	C16—H16	0.93
C8—C9	1.477 (7)	C17—H17	0.93
C2—C1—C12	111.7 (4)	C8—C9—H9	106.1 (3)
C2—C1—C10	105.7 (4)	N—C9—H9	106.1 (3)
C12—C1—C10	114.3 (4)	C1—C9—H9	106.1 (3)
C2—C1—C9	109.3 (4)	F10B—C10—F10A	106.0 (4)
C12—C1—C9	105.1 (4)	F10B—C10—F10C	105.6 (4)
C10—C1—C9	110.6 (4)	F10A—C10—F10C	105.4 (4)
C3—C2—C11	118.4 (5)	F10B—C10—C1	115.3 (4)
C3—C2—C1	118.8 (4)	F10A—C10—C1	111.8 (4)
C11—C2—C1	122.8 (4)	F10C—C10—C1	112.0 (4)
C2—C3—F3	120.8 (4)	F11A—C11—F11B	107.7 (5)
C2—C3—C4	128.3 (4)	F11A—C11—F11C	105.5 (5)
F3—C3—C4	110.8 (4)	F11B—C11—F11C	105.6 (5)
O4—C4—N	125.4 (5)	F11A—C11—C2	114.9 (5)
O4—C4—C3	120.7 (5)	F11B—C11—C2	112.4 (5)
N—C4—C3	113.9 (4)	F11C—C11—C2	110.1 (4)
C4—N—C5	118.3 (4)	C17—C12—C13	118.8 (5)
C4—N—C9	120.6 (4)	C17—C12—C1	124.6 (5)
C5—N—C9	119.2 (4)	C13—C12—C1	116.6 (5)
C6—C5—N	121.1 (5)	C12—C13—C14	121.5 (5)
C6—C5—H5	119.5 (3)	C12—C13—H13	119.3 (3)

N—C5—H5	119.5 (3)	C14—C13—H13	119.3 (4)
C5—C6—C7	121.3 (5)	C15—C14—C13	119.4 (6)
C5—C6—H6	119.4 (3)	C15—C14—H14	120.3 (4)
C7—C6—H6	119.4 (3)	C13—C14—H14	120.3 (4)
C8—C7—C6	119.7 (5)	C14—C15—C16	120.7 (6)
C8—C7—H7	120.1 (3)	C14—C15—H15	119.6 (4)
C6—C7—H7	120.1 (3)	C16—C15—H15	119.6 (4)
C7—C8—C9	123.4 (5)	C15—C16—C17	120.7 (6)
C7—C8—H8	118.3 (3)	C15—C16—H16	119.7 (4)
C9—C8—H8	118.3 (3)	C17—C16—H16	119.7 (4)
C8—C9—N	110.8 (4)	C12—C17—C16	118.8 (6)
C8—C9—C1	114.0 (4)	C12—C17—H17	120.6 (3)
N—C9—C1	113.1 (3)	C16—C17—H17	120.6 (4)
C12—C1—C2—C3	−138.9 (5)	C2—C1—C9—N	43.5 (5)
C10—C1—C2—C3	96.1 (5)	C12—C1—C9—N	163.6 (4)
C9—C1—C2—C3	−23.0 (6)	C10—C1—C9—N	−72.5 (5)
C12—C1—C2—C11	41.8 (6)	C2—C1—C10—F10B	−178.9 (4)
C10—C1—C2—C11	−83.2 (5)	C12—C1—C10—F10B	57.8 (6)
C9—C1—C2—C11	157.7 (4)	C9—C1—C10—F10B	−60.7 (5)
C11—C2—C3—F3	0.3 (7)	C2—C1—C10—F10A	−57.7 (5)
C1—C2—C3—F3	−179.0 (4)	C12—C1—C10—F10A	179.0 (4)
C11—C2—C3—C4	176.3 (5)	C9—C1—C10—F10A	60.5 (5)
C1—C2—C3—C4	−3.1 (8)	C2—C1—C10—F10C	60.4 (5)
C2—C3—C4—O4	−172.4 (5)	C12—C1—C10—F10C	−62.9 (5)
F3—C3—C4—O4	3.8 (6)	C9—C1—C10—F10C	178.6 (4)
C2—C3—C4—N	8.4 (7)	C3—C2—C11—F11A	−176.4 (4)
F3—C3—C4—N	−175.3 (4)	C1—C2—C11—F11A	2.9 (7)
O4—C4—N—C5	1.6 (7)	C3—C2—C11—F11B	59.9 (6)
C3—C4—N—C5	−179.3 (4)	C1—C2—C11—F11B	−120.8 (5)
O4—C4—N—C9	−162.9 (5)	C3—C2—C11—F11C	−57.5 (7)
C3—C4—N—C9	16.2 (6)	C1—C2—C11—F11C	121.8 (5)
C4—N—C5—C6	−179.5 (5)	C2—C1—C12—C17	−130.1 (5)
C9—N—C5—C6	−14.9 (7)	C10—C1—C12—C17	−10.1 (7)
N—C5—C6—C7	−3.5 (8)	C9—C1—C12—C17	111.5 (5)
C5—C6—C7—C8	11.2 (8)	C2—C1—C12—C13	48.9 (6)
C6—C7—C8—C9	0.3 (8)	C10—C1—C12—C13	168.9 (4)
C7—C8—C9—N	−16.7 (7)	C9—C1—C12—C13	−69.5 (5)
C7—C8—C9—C1	−145.7 (5)	C17—C12—C13—C14	−1.0 (8)
C4—N—C9—C8	−172.1 (4)	C1—C12—C13—C14	180.0 (5)
C5—N—C9—C8	23.6 (6)	C12—C13—C14—C15	0.9 (8)
C4—N—C9—C1	−42.7 (6)	C13—C14—C15—C16	−1.3 (10)
C5—N—C9—C1	153.0 (4)	C14—C15—C16—C17	1.8 (11)
C2—C1—C9—C8	171.3 (4)	C13—C12—C17—C16	1.4 (8)
C12—C1—C9—C8	−68.6 (5)	C1—C12—C17—C16	−179.6 (5)
C10—C1—C9—C8	55.3 (5)	C15—C16—C17—C12	−1.9 (10)