Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including bond distances and angles involving H atoms, have been deposited with the IUCr (Reference: AS1140). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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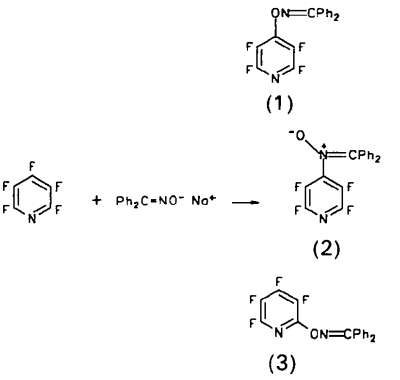
Comment

The structure determination reported herein forms part of an investigation into the substitution of salts of hydroxylamines, oximes and hydrazones with pentafluoropyridine, in which novel competing 2-substitution was observed in addition to the expected 4-substitution (Banks, Jondi & Tipping, 1989; Jondi, 1989). The structural information was required to confirm that the 4-substituted product was the title oxime (1), and not the alternative immonium oxide (2), which would also aid identification of the 2-substituted product (3), for which crystals suitable for X-ray crystallographic analysis could not be obtained.



Acta Cryst. (1995). C**51**, 291–293

Benzophenone *O*-(2,3,5,6-Tetrafluoro-4-pyridyl)oxime, Formed by 4-Substitution of Pentafluoropyridine by Benzophenone Oximate

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Abstract

The asymmetric unit of the title compound, $C_{18}H_{10}F_4N_2O$, is composed of two identical molecules having different configurations. Although both molecules adopt a *trans* configuration about the N—O bond [C—N—O—C 164.2 (3) and −162.0 (2)°], substantial twists of all the aromatic rings relative to the C=N—O plane show no correlation between the conformers. Despite the apparently random molecular configurations, the packing arrangement involves several π interactions between adjacent molecules.

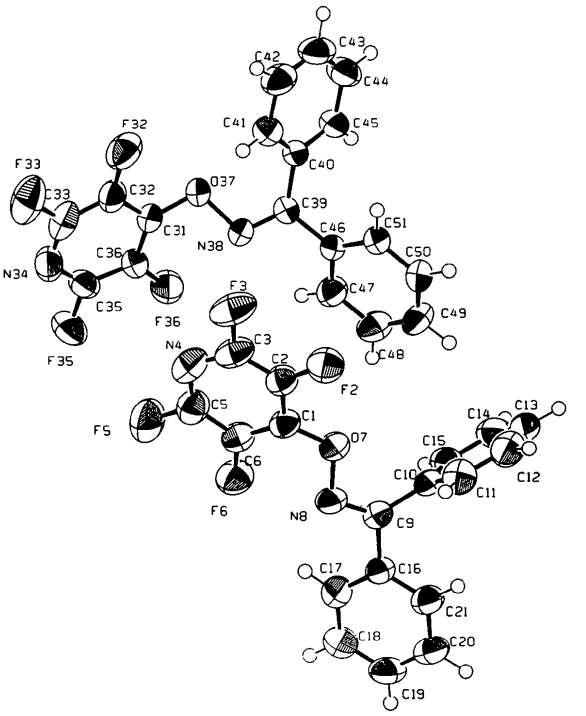

Fig. 1. An *ORTEP*II (Johnson, 1976) view of the asymmetric unit of the title compound, comprising two molecules, showing the atomic numbering scheme with ellipsoids set at the 50% probability level.

Experimental

Pentafluoropyridine (4.30 g, 25.4 mmol) was added slowly to a stirred slurry of sodium benzophenone oximate [prepared *in situ* from sodium hydride (0.60 g, 25.0 mmol) and benzophenone oxime (4.60 g, 23.4 mmol) in diethyl ether (*ca* 50 ml)] and the mixture was stirred for 4 h. The mixture was then filtered and the solvent removed from the filtrate *in vacuo* to give a residue (8.70 g) which was shown by TLC (eluant 1:4 *v/v* $CHCl_3$:*n*-C_6H_{14}) to contain two components (R_F = 0.40 and 0.30). Separation by dry-column chromatography (Merck Kieselgel 60 GF_{254}; eluant 1:4 *v/v* $CHCl_3$:*n*-C_6H_{14}) afforded (*a*) the title compound (1) (4.90 g, 14.2 mmol, 56%; analysis found C 62.7, H 2.9, F 22.4, N 8.1%, M^+ = 346; $C_{18}H_{10}F_4N_2O$ requires C 62.4, H 2.9, F 22.0, N 8.1%, M = 346), m.p. 325–327 K; and (*b*) 2-(diphenylmethyliminooxy)tetrafluoropyridine (3) (1.90 g, 5.5 mmol, 22%; analysis found C 62.3, 22.2, N 8.0%, M^+ = 346; $C_{18}H_{10}F_4N_2O$ requires C 62.4, H 2.9, F 22.0, N 8.1%, M = 346), m.p. 370–372 K. The crystals of compound (1), obtained by slow evaporation of the eluant, were suitable for the structure determination.

Crystal data

$C_{18}H_{10}F_4N_2O$
M_r = 346.28
Triclinic
$P\bar{1}$
a = 5.603 (2) Å
b = 23.641 (4) Å
c = 12.643 (2) Å
α = 73.42 (1)°
β = 76.34 (3)°
γ = 84.92 (2)°
V = 1559 (1) Å^3
Z = 4
D_x = 1.475 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 9.53–12.78°
μ = 0.1194 mm^{-1}
T = 286 K
Plate
0.40 × 0.40 × 0.15 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4950 measured reflections
4848 independent reflections
2663 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.0167

θ_{max} = 24.0°
$h = 0 \rightarrow 6$
$k = -25 \rightarrow 25$
$l = -13 \rightarrow 13$
3 standard reflections monitored every 200 reflections
intensity decay: not significant

Refinement

Refinement on F
R = 0.037
wR = 0.025
S = 2.320
2663 reflections
531 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F) + 0.00023F^2]$
$(\Delta/\sigma)_{max}$ = 0.008
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.167 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)

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

	x	y	z	U_{eq}
F2	0.2899 (3)	0.19669 (8)	1.0084 (2)	0.087 (2)
F3	0.2833 (4)	0.25008 (8)	0.7912 (2)	0.104 (2)
F5	1.0279 (4)	0.17466 (9)	0.6702 (2)	0.110 (2)
F6	1.0826 (3)	0.12219 (8)	0.8769 (2)	0.088 (2)
O7	0.6769 (4)	0.13532 (9)	1.0662 (2)	0.069 (2)
N4	0.6561 (7)	0.2123 (1)	0.7301 (3)	0.080 (3)
N8	0.8423 (4)	0.0850 (1)	1.0892 (2)	0.065 (2)
C1	0.6846 (6)	0.1569 (1)	0.9535 (3)	0.058 (3)
C2	0.4821 (6)	0.1906 (1)	0.9259 (3)	0.063 (3)
C3	0.4812 (7)	0.2171 (2)	0.8152 (4)	0.074 (3)
C5	0.8440 (7)	0.1800 (2)	0.7567 (3)	0.076 (3)
C6	0.8729 (6)	0.1520 (1)	0.8639 (3)	0.064 (3)
C9	0.8735 (5)	0.0759 (1)	1.1898 (3)	0.052 (2)
C10	0.7611 (6)	0.1102 (1)	1.2716 (2)	0.050 (3)
C11	0.5241 (7)	0.1002 (2)	1.3363 (3)	0.073 (3)
C12	0.4335 (8)	0.1307 (2)	1.4161 (3)	0.082 (4)
C13	0.5729 (10)	0.1703 (2)	1.4322 (3)	0.078 (4)
C14	0.8056 (9)	0.1802 (2)	1.3688 (3)	0.075 (4)
C15	0.9004 (7)	0.1498 (2)	1.2893 (3)	0.060 (3)
C16	1.0497 (6)	0.0269 (1)	1.2235 (3)	0.056 (3)
C17	1.2468 (7)	0.0140 (2)	1.1452 (3)	0.082 (3)
C18	1.4168 (8)	−0.0295 (2)	1.1788 (4)	0.090 (4)
C19	1.3894 (8)	−0.0601 (2)	1.2897 (4)	0.079 (4)
C20	1.1950 (7)	−0.0480 (2)	1.3687 (4)	0.079 (3)
C21	1.0264 (7)	−0.0039 (2)	1.3351 (3)	0.070 (3)
F32	0.3508 (3)	0.42862 (9)	0.4752 (2)	0.094 (2)
F33	0.4843 (4)	0.37205 (9)	0.3132 (2)	0.112 (2)
F35	1.1203 (4)	0.28096 (8)	0.4574 (2)	0.097 (2)
F36	1.0195 (3)	0.33188 (7)	0.6262 (2)	0.078 (2)
O37	0.5954 (4)	0.40995 (9)	0.6401 (2)	0.068 (2)
N34	0.8009 (7)	0.3267 (1)	0.3856 (2)	0.076 (3)
N38	0.6031 (4)	0.36876 (10)	0.7495 (2)	0.060 (2)
C31	0.6736 (5)	0.3811 (1)	0.5591 (3)	0.053 (2)
C32	0.5460 (6)	0.3913 (2)	0.4750 (3)	0.063 (3)
C33	0.6156 (8)	0.3632 (2)	0.3932 (3)	0.075 (3)
C35	0.9252 (7)	0.3176 (1)	0.4643 (3)	0.067 (3)
C36	0.8748 (6)	0.3433 (1)	0.5523 (3)	0.057 (3)
C39	0.4699 (5)	0.3885 (1)	0.8291 (2)	0.049 (2)
C40	0.3078 (5)	0.4420 (1)	0.8157 (2)	0.049 (2)
C41	0.1326 (6)	0.4503 (2)	0.7518 (3)	0.065 (3)
C42	−0.0251 (7)	0.4993 (2)	0.7449 (3)	0.079 (3)
C43	−0.0032 (8)	0.5393 (2)	0.8013 (4)	0.085 (4)
C44	0.1699 (8)	0.5323 (2)	0.8634 (4)	0.079 (3)
C45	0.3253 (6)	0.4831 (2)	0.8720 (3)	0.060 (3)
C46	0.4798 (5)	0.3510 (1)	0.9435 (2)	0.049 (2)
C47	0.6893 (7)	0.3173 (2)	0.9641 (3)	0.065 (3)
C48	0.6936 (8)	0.2813 (2)	1.0700 (3)	0.076 (3)
C49	0.4898 (9)	0.2776 (2)	1.1563 (3)	0.079 (4)
C50	0.2832 (8)	0.3103 (2)	1.1379 (3)	0.072 (3)
C51	0.2778 (6)	0.3472 (1)	1.0323 (3)	0.060 (3)

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

O7—N8	1.446 (3)	O37—N38	1.454 (3)
O7—C1	1.361 (3)	O37—C31	1.356 (3)
N8—C9	1.278 (3)	N38—C39	1.281 (3)
C9—C10	1.486 (4)	C39—C40	1.483 (4)
C9—C16	1.487 (4)	C39—C46	1.476 (4)
N8—O7—C1	111.7 (2)	N38—O37—C31	108.5 (2)
O7—N8—C9	108.8 (2)	O37—N38—C39	110.2 (2)
O7—C1—C6	128.3 (3)	O37—C31—C36	125.5 (3)
N8—C9—C10	126.8 (3)	N38—C39—C40	126.6 (3)
N8—C9—C16	114.8 (3)	N38—C39—C46	113.6 (3)

Data collection: CAD-4 diffractometer control software (Enraf–Nonius, 1989). Cell refinement: CAD-4 diffractometer control software. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to

refine structure: *TEXSAN LS*. 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, including bond distances and angles involving H atoms, have been deposited with the IUCr (Reference: LI1121). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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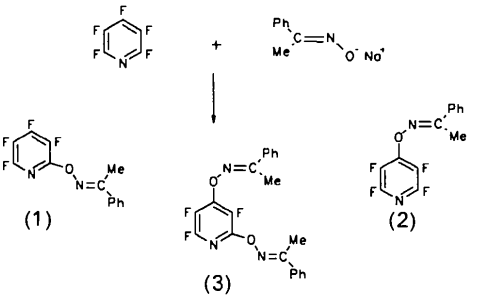
Comment

The structure determination reported herein is part of an investigation into the substitution reactions of hydroxylamine, oxime and hydrozone salts with pentafluoropyridine and related compounds, in which novel 2-substitution was observed to compete with the expected 4-substitution (Banks, Jondi & Tipping, 1989; Jondi, 1989). The structural information was required to confirm that the title compound was a 2-substituted tetrafluoropyridine and that the substituent was the —O—N=C(Me)Ph group having the same configuration, *i.e.* (*E*), as the oximate reactant. Confirmation that the compound was the 2-substituted oxime (1) enabled the other products of the reaction, compounds (2) and (3), to be positively identified.



Acta Cryst. (1995). C51, 293–295

(*E*)-Acetophenone *O*-(3,4,5,6-Tetrafluoro-2-pyridyl)oxime, Formed by 2-Substitution of Pentafluoropyridine by (*E*)-Acetophenone Oximate

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(*Received* 28 *April* 1994; *accepted* 4 *August* 1994)

Abstract

Despite the oxyimino chain being unconjugated [N—O 1.434 (2) and C—N 1.278 (2) Å] in the title compound, $C_{13}H_8F_4N_2O$, the planar α-phenylethylimino and tetrafluoro-2-oxopyridine moieties are only slightly twisted relative to each other [C—O—N—C 167.6 (2)°]. This facilitates stacking along the *ac* diagonal so that fluorinated pyridine substituents alternate with non-fluorinated phenyl rings.

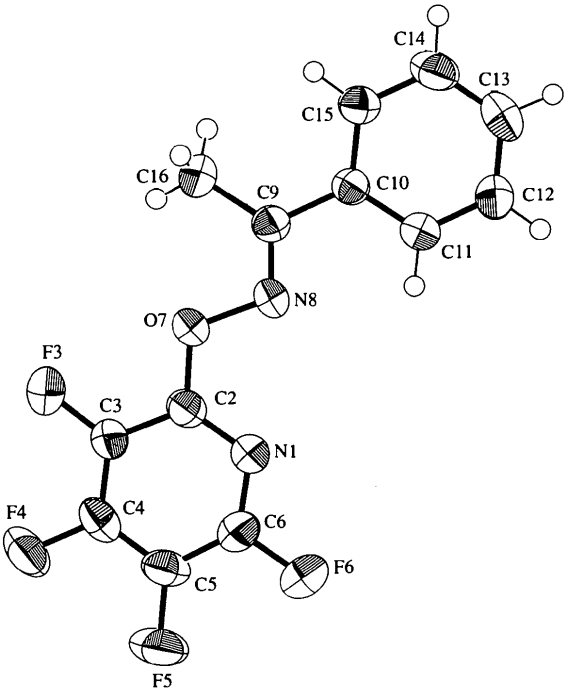

Fig. 1. *ORTEP*II (Johnson, 1976) view of the title molecule showing the atom-numbering scheme and ellipsoids set at the 50% probability level.