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1,5-Dimethyl-4-[3-oxo-1,3-bis(trifluoromethyl)prop-1-enylamino]-2-phenyl-1*H*-pyrazol-3(2*H*)-one

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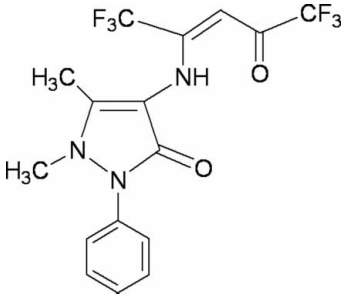
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.043; wR factor = 0.093; data-to-parameter ratio = 10.8.

In the title molecule, $C_{16}H_{13}F_6N_3O_2$, the phenyl and pyrazolone rings make a dihedral angle of 43.1 (3)°. Intramolecular N—H···O hydrogen bonding contributes to the essential coplanarity of the side-chain carbonyl group, the adjacent double bond and the amine N atom; the latter has the largest deviation [0.017 (3) Å] from the mean plane. In the crystal structure, weak intermolecular C—H···O hydrogen bonds link the molecules into chains extending along the c axis. The F atoms of one CF_3 group are disordered approximately equally over two sets of positions.

Related literature

For related crystal structures, see: Wang *et al.* (2002, 2003); Yu *et al.* (2002).



Experimental

Crystal data

$C_{16}H_{13}F_6N_3O_2$
M_r = 393.29
Monoclinic, $P2_1/c$
a = 15.807 (5) Å
b = 8.324 (3) Å
c = 13.715 (5) Å
β = 107.644 (6)°
V = 1719.6 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 293 (2) K
0.30 × 0.30 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.987, T_{max} = 1.000
(expected range = 0.956–0.969)
6854 measured reflections
3008 independent reflections
1508 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.093
S = 0.98
3008 reflections
279 parameters
42 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O2	0.85 (3)	2.04 (3)	2.710 (4)	136 (3)
C9—H9B···O1^{i}	0.96	2.54	3.453 (3)	160 (3)
C10—H10A···O1^{i}	0.96	2.58	3.448 (3)	150 (3)

Symmetry code: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2343).

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supplementary materials

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1,5-Dimethyl-4-[3-oxo-1,3-bis(trifluoromethyl)prop-1-enylamino]-2-phenyl-1*H*-pyrazol-3(2*H*)-one

S.-M. Zhang, W.-J. Zhang and J.-L. Wang

Comment

Schiff base ligands derived from 4-aminoantipyrine and their metal complexes have attracted great attentions of several research groups due to their potential biological activities, such as analgesic, anti-inflammatory, antimicrobial and anticancer activity. As a part of our continuing interest on the syntheses of potential bioactive compounds, we have synthesized several novel schiff base ligands with 4-aminoantipyrine in search for antibacterial agents. And we report here the structure of the title compound (I).

A view of the molecule of (I) is shown in Fig. 1. Atoms C4, C5, C6 and O2 of the HFAA moiety and atom N3 of 4-AATP are essentially coplanar, the largest deviation from the mean plane being 0.017 (3) Å for atom C6. This mean plane is nearly perpendicular to the pyrazolone ring with the dihedral angle of 87.34 (2) °, which close to the value of 84.10 (6) ° reported by Yu *et al.* (2002), reducing their steric hindrance. Bond lengths within this part of the molecule lie between the classical double- and single-bond lengths, clearly indicating that there is electron delocalization over this segment. The dihedral angle between phenyl and pyrazolone rings is 43.1 (3) °. Small torsion angles for O2—C6—C5—C4 [−4.9 (5) °] and N3—C4—C5—C6 [4.0 (4) °] show that atoms O2 and N3 may coordinate with metal ion to form a six-membered chelating ring. Atoms O1, C1, C3 and N3 are also coplanar, the largest deviation from this mean plane being 0.006 (2) Å both for C1 and C3. The dihedral angle between this plane and the adjacent pyrazolone ring of 4-AATP is 6.26 (3) °, closing to the value of 6.64 (3) ° in 4-{[3,4-Dihydro-5-methyl-3-oxo-2-phenyl- 2*H*-pyrazol-4-ylidene](phenyl)methylamino}- 1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one (Wang *et al.*, 2003). The bond lengths in this part of the molecule also indicate delocalization for the antipyrine group. Small torsion angles for O1—C1—C3—N3 [−1.7 (5) °] show that atoms O1 and N3 could chelate with metal and forming a stable five-membered ring. The displacements of atoms C10 and C11 from the pyrazolone ring are −0.588 (7) and 0.397 (5) Å, respectively, showing that the methyl group bonded to N2 and the phenyl group bonded to N1 are on opposite sides of the ring, and the torsion angle of C11—N1—N2—C10 is 54.8 (3) °. These are similar to the related reports (Yu *et al.*, 2002; Wang *et al.*, 2003). A strong intramolecular N—H—O hydrogen bond is observed (Table 1), which shows that the molecule exists as the enamine-keto tautomeric form. This case is completely different from that of 3-(2,3-dihydro-1,5-dimethyl 3-oxo-2-phenylpyrazol-4-ylimino)-4,4,4-trifluoro-1-(2-thienyl)butane −1,2-dione (Wang *et al.*, 2002), but similar to that in 1,5-Dimethyl-4-{[(E)-3-oxo-3-(2-thienyl)- 1-(trifluoromethyl)-1-propenyl] amino}-2-phenyl-1,2-dihydro-3*H*-pyrazol-3-one (Yu *et al.*, 2002). In addition, in the crystal the weak intermolecular C—H···O hydrogen bonds (Table 1) link the molecules into chains extended along *c* axis.

Experimental

The ethanol solutions of 4-aminoantipyrine (0.01 mol) and HFAA (0.01 mol) were refluxed together for 3 h over a steam bath. The excess solvent was removed by evaporation and the concentrated solution was cooled in an ice bath with stirring. Title compound separated out as a cream-colored powder, which was filtered and washed with ethanol, then dried in a vacuum over $CaCl_2$. Melting point: 440–442 K, Elemental analysis for $C_{16}H_{13}F_6N_3O_2$, calculated: C 48.86, H 3.33, N

10.69%; found: C 48.71, H 3.42, N 10.56%. Bright-yellow single crystals suitable for X-ray analysis were obtained by slow cooling of the warmed solution in ethanol.

Refinement

C-bound H atoms were included in calculated positions (C—H 0.93, 0.96 Å) and treated in the subsequent refinement as riding atoms, with U_{iso}(H) = 1.2 or 1.5 U_{eq}(C). Atom H3 was located in Fourier difference map and refined isotropically. One CF_3 group (at C7) was treated as disordered between two orientations with the refined occupancies of 0.54 (3) and 0.46 (3), respectively. The C7—F' bond lengths were restrained to 1.32 (2) Å.

Figures

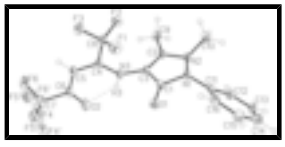


Fig. 1. The molecular structure of (I) showing atomic numbering, disordered CF_3 group and 20% probability displacement ellipsoids. Dashed line denotes hydrogen bond.

1,5-Dimethyl-4-[3-oxo-1,3-bis(trifluoromethyl)prop-1-enylamino]-2-phenyl- 1*H*-pyrazol-3(2*H*)-one

Crystal data

$C_{16}H_{13}F_6N_3O_2$	F_{000} = 800
M_r = 393.29	D_x = 1.519 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 440-442 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 15.807 (5) Å	Cell parameters from 2548 reflections
b = 8.324 (3) Å	θ = 2.7–25.0º
c = 13.715 (5) Å	μ = 0.15 mm^{-1}
β = 107.644 (6)º	T = 293 (2) K
V = 1719.6 (11) Å^3	Block, yellow
Z = 4	0.30 × 0.30 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer	3008 independent reflections
Radiation source: fine-focus sealed tube	1508 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.055
T = 293(2) K	θ_{max} = 25.0º
φ and ω scan	θ_{min} = 2.7º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	h = −10→18
T_{min} = 0.987, T_{max} = 1.000	k = −9→8
6854 measured reflections	l = −15→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.043$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.093$	$w = 1/[\sigma^2(F_o^2) + (0.0307P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.98$	$(\Delta/\sigma)_{max} < 0.001$
3008 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
279 parameters	$\Delta\rho_{min} = -0.14$ e Å^{-3}
42 restraints	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: 0.0223 (11)

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 of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness 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 threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *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}	Occ. (<1)
O1	0.60740 (12)	0.2162 (3)	0.45906 (15)	0.0638 (6)	
O2	0.85903 (15)	0.5621 (3)	0.51390 (17)	0.0811 (8)	
N1	0.56744 (14)	0.1180 (3)	0.29188 (15)	0.0447 (6)	
N2	0.60661 (14)	0.1235 (3)	0.21240 (15)	0.0440 (6)	
N3	0.78198 (15)	0.2900 (4)	0.41904 (18)	0.0497 (7)	
C4	0.85016 (16)	0.2205 (4)	0.4883 (2)	0.0433 (7)	
C8	0.8539 (2)	0.0401 (4)	0.4874 (2)	0.0559 (8)	
F1	0.78609 (13)	−0.0247 (2)	0.51143 (15)	0.0811 (6)	
F2	0.85204 (11)	−0.0198 (2)	0.39737 (13)	0.0770 (6)	
F3	0.92697 (12)	−0.0163 (2)	0.55386 (14)	0.0882 (7)	
C6	0.91335 (19)	0.4700 (5)	0.5685 (2)	0.0550 (9)	
C7	0.9825 (2)	0.5457 (5)	0.6604 (3)	0.0662 (10)	
C1	0.62664 (18)	0.1871 (3)	0.3800 (2)	0.0463 (8)	
C2	0.69184 (17)	0.1733 (3)	0.2536 (2)	0.0431 (7)	
C3	0.70543 (16)	0.2138 (3)	0.35296 (19)	0.0423 (7)	

C5	0.91552 (17)	0.3029 (4)	0.55891 (19)	0.0490 (8)	
H5	0.9629	0.2461	0.6019	0.059*	
C9	0.75420 (18)	0.1735 (4)	0.1918 (2)	0.0632 (9)	
H9A	0.8061	0.2348	0.2265	0.095*	
H9B	0.7258	0.2208	0.1262	0.095*	
H9C	0.7712	0.0651	0.1827	0.095*	
C11	0.47364 (17)	0.1192 (3)	0.2692 (2)	0.0449 (7)	
C12	0.4196 (2)	0.1857 (4)	0.1797 (2)	0.0599 (9)	
H12	0.4439	0.2289	0.1316	0.072*	
C13	0.3289 (2)	0.1871 (4)	0.1628 (3)	0.0823 (12)	
H13	0.2918	0.2286	0.1019	0.099*	
C14	0.2929 (2)	0.1281 (5)	0.2345 (4)	0.0886 (13)	
H14	0.2319	0.1317	0.2228	0.106*	
C15	0.3469 (3)	0.0638 (4)	0.3232 (3)	0.0791 (11)	
H15	0.3222	0.0246	0.3720	0.095*	
C16	0.4372 (2)	0.0562 (4)	0.3413 (2)	0.0566 (9)	
H16	0.4735	0.0095	0.4010	0.068*	
C10	0.58075 (19)	0.0024 (4)	0.1319 (2)	0.0580 (9)	
H10A	0.5903	0.0436	0.0706	0.087*	
H10B	0.5191	−0.0234	0.1186	0.087*	
H10C	0.6159	−0.0926	0.1534	0.087*	
F4	0.9963 (12)	0.6977 (12)	0.6454 (13)	0.114 (5)	0.54 (3)
F5	0.9608 (7)	0.524 (2)	0.7431 (5)	0.092 (3)	0.54 (3)
F6	1.0624 (5)	0.4819 (17)	0.6763 (13)	0.110 (4)	0.54 (3)
F4'	0.9411 (9)	0.609 (3)	0.7246 (13)	0.111 (4)	0.46 (3)
F5'	1.0448 (10)	0.4480 (14)	0.7120 (11)	0.107 (4)	0.46 (3)
F6'	1.0209 (11)	0.6712 (16)	0.6377 (12)	0.085 (4)	0.46 (3)
H3	0.7816 (18)	0.391 (4)	0.423 (2)	0.060 (11)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0513 (13)	0.0989 (18)	0.0381 (13)	−0.0008 (11)	0.0090 (10)	−0.0144 (12)
O2	0.0821 (17)	0.0659 (17)	0.0671 (16)	−0.0058 (12)	−0.0198 (13)	−0.0016 (13)
N1	0.0341 (14)	0.0650 (18)	0.0309 (13)	−0.0040 (12)	0.0036 (11)	−0.0015 (12)
N2	0.0380 (14)	0.0610 (17)	0.0293 (13)	−0.0061 (12)	0.0047 (11)	−0.0065 (12)
N3	0.0417 (16)	0.055 (2)	0.0415 (15)	−0.0024 (14)	−0.0043 (11)	−0.0037 (15)
C4	0.0354 (17)	0.059 (2)	0.0333 (16)	−0.0008 (14)	0.0066 (14)	0.0005 (15)
C8	0.045 (2)	0.073 (3)	0.0419 (19)	0.0023 (17)	0.0021 (15)	−0.0001 (18)
F1	0.0845 (14)	0.0723 (14)	0.0914 (14)	−0.0154 (11)	0.0341 (12)	0.0030 (11)
F2	0.0842 (13)	0.0816 (15)	0.0581 (12)	0.0151 (10)	0.0109 (10)	−0.0187 (11)
F3	0.0791 (13)	0.0793 (15)	0.0773 (13)	0.0174 (10)	−0.0196 (11)	0.0024 (11)
C6	0.044 (2)	0.078 (3)	0.0373 (18)	−0.0119 (18)	0.0040 (15)	−0.0011 (19)
C7	0.062 (3)	0.072 (3)	0.055 (2)	−0.016 (2)	0.002 (2)	0.001 (2)
C1	0.0436 (19)	0.058 (2)	0.0303 (16)	0.0028 (15)	0.0013 (14)	−0.0025 (15)
C2	0.0340 (17)	0.057 (2)	0.0338 (16)	−0.0016 (14)	0.0033 (13)	0.0006 (15)
C3	0.0290 (16)	0.060 (2)	0.0333 (16)	−0.0019 (14)	0.0028 (13)	−0.0048 (15)
C5	0.0397 (18)	0.067 (2)	0.0350 (17)	−0.0062 (16)	0.0037 (13)	0.0002 (17)

C9	0.0502 (19)	0.094 (3)	0.0461 (18)	−0.0017 (17)	0.0160 (15)	−0.0013 (18)
C11	0.0347 (17)	0.046 (2)	0.0461 (17)	−0.0042 (14)	−0.0006 (14)	−0.0053 (15)
C12	0.050 (2)	0.053 (2)	0.062 (2)	−0.0039 (15)	−0.0066 (16)	0.0052 (17)
C13	0.046 (2)	0.074 (3)	0.096 (3)	0.0039 (19)	−0.023 (2)	−0.002 (2)
C14	0.040 (2)	0.085 (3)	0.134 (4)	−0.010 (2)	0.016 (3)	−0.019 (3)
C15	0.067 (3)	0.077 (3)	0.103 (3)	−0.020 (2)	0.039 (2)	−0.018 (2)
C16	0.051 (2)	0.055 (2)	0.064 (2)	−0.0056 (16)	0.0172 (17)	−0.0026 (17)
C10	0.068 (2)	0.064 (2)	0.0358 (16)	−0.0081 (16)	0.0064 (14)	−0.0108 (16)
F4	0.116 (8)	0.084 (5)	0.112 (6)	−0.029 (4)	−0.010 (5)	−0.013 (4)
F5	0.097 (5)	0.133 (8)	0.036 (3)	−0.051 (4)	0.007 (3)	−0.008 (3)
F6	0.055 (4)	0.130 (7)	0.119 (7)	−0.011 (4)	−0.012 (4)	−0.028 (5)
F4'	0.108 (6)	0.155 (9)	0.079 (6)	−0.052 (6)	0.043 (5)	−0.057 (6)
F5'	0.097 (7)	0.101 (5)	0.077 (6)	−0.025 (5)	−0.044 (4)	0.010 (4)
F6'	0.075 (6)	0.109 (7)	0.068 (4)	−0.048 (5)	0.014 (4)	0.005 (5)

Geometric parameters (Å, °)

O1—C1	1.236 (3)	C7—F4'	1.352 (8)
O2—C6	1.223 (3)	C1—C3	1.420 (3)
N1—C1	1.408 (3)	C2—C3	1.356 (3)
N1—N2	1.408 (3)	C2—C9	1.482 (3)
N1—C11	1.420 (3)	C5—H5	0.9300
N2—C2	1.358 (3)	C9—H9A	0.9600
N2—C10	1.459 (3)	C9—H9B	0.9600
N3—C4	1.333 (3)	C9—H9C	0.9600
N3—C3	1.422 (3)	C11—C12	1.381 (4)
N3—H3	0.85 (3)	C11—C16	1.389 (4)
C4—C5	1.367 (3)	C12—C13	1.381 (4)
C4—C8	1.503 (4)	C12—H12	0.9300
C8—F3	1.322 (3)	C13—C14	1.367 (5)
C8—F2	1.323 (3)	C13—H13	0.9300
C8—F1	1.328 (3)	C14—C15	1.365 (5)
C6—C5	1.398 (4)	C14—H14	0.9300
C6—C7	1.532 (4)	C15—C16	1.375 (4)
C7—F6'	1.293 (8)	C15—H15	0.9300
C7—F5	1.294 (7)	C16—H16	0.9300
C7—F5'	1.307 (8)	C10—H10A	0.9600
C7—F4	1.310 (8)	C10—H10B	0.9600
C7—F6	1.325 (7)	C10—H10C	0.9600
C1—N1—N2	108.5 (2)	O1—C1—N1	123.9 (3)
C1—N1—C11	124.3 (2)	O1—C1—C3	131.8 (2)
N2—N1—C11	120.3 (2)	N1—C1—C3	104.3 (2)
C2—N2—N1	107.5 (2)	C3—C2—N2	109.2 (2)
C2—N2—C10	122.7 (2)	C3—C2—C9	130.1 (2)
N1—N2—C10	118.4 (2)	N2—C2—C9	120.7 (2)
C4—N3—C3	127.5 (3)	C2—C3—C1	109.8 (2)
C4—N3—H3	114 (2)	C2—C3—N3	126.6 (3)
C3—N3—H3	117.7 (19)	C1—C3—N3	123.2 (2)
N3—C4—C5	124.1 (3)	C4—C5—C6	122.1 (3)

N3—C4—C8	116.9 (3)	C4—C5—H5	119.0
C5—C4—C8	118.9 (3)	C6—C5—H5	119.0
F3—C8—F2	106.1 (3)	C2—C9—H9A	109.5
F3—C8—F1	106.7 (3)	C2—C9—H9B	109.5
F2—C8—F1	106.7 (3)	H9A—C9—H9B	109.5
F3—C8—C4	112.2 (3)	C2—C9—H9C	109.5
F2—C8—C4	113.2 (3)	H9A—C9—H9C	109.5
F1—C8—C4	111.6 (3)	H9B—C9—H9C	109.5
O2—C6—C5	126.8 (3)	C12—C11—C16	120.4 (3)
O2—C6—C7	116.1 (3)	C12—C11—N1	121.4 (3)
C5—C6—C7	117.0 (3)	C16—C11—N1	118.2 (3)
F6'—C7—F5	126.5 (9)	C13—C12—C11	118.9 (3)
F6'—C7—F5'	107.6 (9)	C13—C12—H12	120.5
F5—C7—F5'	78.2 (6)	C11—C12—H12	120.5
F6'—C7—F4	21.5 (11)	C14—C13—C12	120.9 (3)
F5—C7—F4	112.1 (8)	C14—C13—H13	119.6
F5'—C7—F4	123.5 (8)	C12—C13—H13	119.6
F6'—C7—F6	82.6 (8)	C15—C14—C13	119.9 (4)
F5—C7—F6	107.0 (6)	C15—C14—H14	120.1
F5'—C7—F6	30.4 (6)	C13—C14—H14	120.1
F4—C7—F6	102.6 (8)	C14—C15—C16	120.9 (4)
F6'—C7—F4'	101.5 (9)	C14—C15—H15	119.6
F5—C7—F4'	34.3 (6)	C16—C15—H15	119.6
F5'—C7—F4'	108.9 (8)	C15—C16—C11	119.1 (3)
F4—C7—F4'	82.2 (8)	C15—C16—H16	120.5
F6—C7—F4'	132.3 (7)	C11—C16—H16	120.5
F6'—C7—C6	113.8 (8)	N2—C10—H10A	109.5
F5—C7—C6	110.5 (5)	N2—C10—H10B	109.5
F5'—C7—C6	115.0 (6)	H10A—C10—H10B	109.5
F4—C7—C6	112.3 (7)	N2—C10—H10C	109.5
F6—C7—C6	112.1 (6)	H10A—C10—H10C	109.5
F4'—C7—C6	109.2 (6)	H10B—C10—H10C	109.5
C1—N1—N2—C2	8.2 (3)	C10—N2—C2—C3	−148.5 (3)
C11—N1—N2—C2	160.4 (2)	N1—N2—C2—C9	172.9 (2)
C1—N1—N2—C10	152.8 (2)	C10—N2—C2—C9	30.1 (4)
C11—N1—N2—C10	−55.0 (3)	N2—C2—C3—C1	1.2 (3)
C3—N3—C4—C5	−170.5 (3)	C9—C2—C3—C1	−177.2 (3)
C3—N3—C4—C8	9.3 (4)	N2—C2—C3—N3	−171.6 (3)
N3—C4—C8—F3	175.5 (2)	C9—C2—C3—N3	10.0 (5)
C5—C4—C8—F3	−4.7 (4)	O1—C1—C3—C2	−174.8 (3)
N3—C4—C8—F2	55.5 (4)	N1—C1—C3—C2	3.8 (3)
C5—C4—C8—F2	−124.7 (3)	O1—C1—C3—N3	−1.7 (5)
N3—C4—C8—F1	−64.9 (3)	N1—C1—C3—N3	176.9 (3)
C5—C4—C8—F1	115.0 (3)	C4—N3—C3—C2	−98.4 (4)
O2—C6—C7—F6'	−49.1 (11)	C4—N3—C3—C1	89.7 (4)
C5—C6—C7—F6'	134.5 (10)	N3—C4—C5—C6	4.0 (4)
O2—C6—C7—F5	100.1 (10)	C8—C4—C5—C6	−175.8 (3)
C5—C6—C7—F5	−76.3 (10)	O2—C6—C5—C4	−4.9 (5)
O2—C6—C7—F5'	−173.8 (11)	C7—C6—C5—C4	171.1 (3)

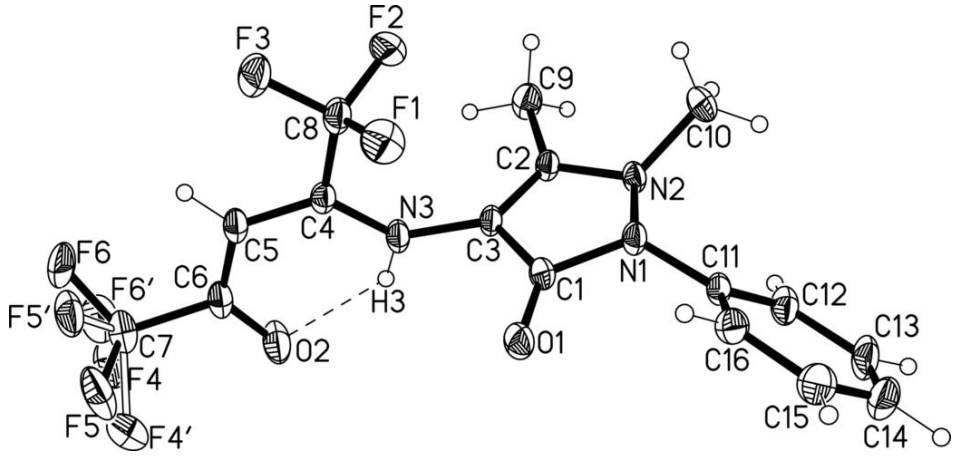
C5—C6—C7—F5'	9.8 (11)	C1—N1—C11—C12	122.3 (3)
O2—C6—C7—F4	−25.8 (11)	N2—N1—C11—C12	−25.3 (4)
C5—C6—C7—F4	157.8 (11)	C1—N1—C11—C16	−55.6 (4)
O2—C6—C7—F6	−140.6 (10)	N2—N1—C11—C16	156.8 (3)
C5—C6—C7—F6	42.9 (10)	C16—C11—C12—C13	−0.6 (4)
O2—C6—C7—F4'	63.5 (14)	N1—C11—C12—C13	−178.4 (3)
C5—C6—C7—F4'	−112.9 (13)	C11—C12—C13—C14	1.9 (5)
N2—N1—C1—O1	171.6 (3)	C12—C13—C14—C15	−1.4 (6)
C11—N1—C1—O1	20.7 (4)	C13—C14—C15—C16	−0.5 (6)
N2—N1—C1—C3	−7.2 (3)	C14—C15—C16—C11	1.8 (5)
C11—N1—C1—C3	−158.1 (2)	C12—C11—C16—C15	−1.2 (4)
N1—N2—C2—C3	−5.7 (3)	N1—C11—C16—C15	176.7 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O2	0.85 (3)	2.04 (3)	2.710 (4)	136 (3)
C9—H9B···O1^{i}	0.96	2.54	3.453 (3)	160 (3)
C10—H10A···O1^{i}	0.96	2.58	3.448 (3)	150 (3)

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2.

Fig. 1


F2
F3
C9
F1
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C4
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C1
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C11
F6′
H3
C12
F5′
C7
O2
O1
C13
C16
F4
F5
F4′
C15
C14