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(1*Z*,2*Z*)-1,2-Bis{2-[3,5-bis(trifluoromethyl)phenyl]-hydrazinylidene}-1,2-bis(4-methoxyphenyl)ethane including an unknown solvate

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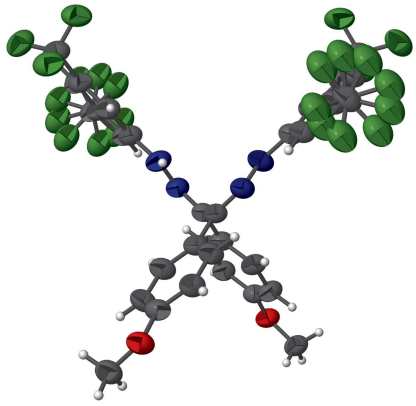
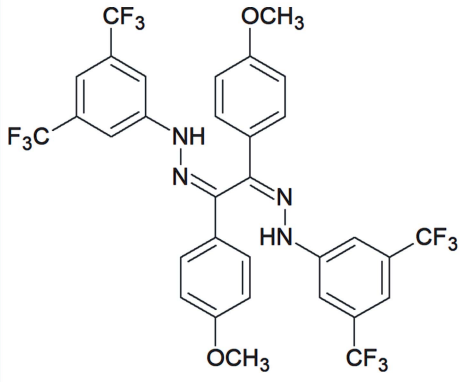
The complete molecule of the title compound, $C_{32}H_{22}F_{12}N_4O_2$, is generated by a crystallographic twofold axis aligned parallel to [010]. The F atoms of one of the CF_3 groups are disordered over three orientations in a 0.6: 0.2: 0.2 ratio. In the crystal, molecules are linked by N—H···O hydrogen bonds, forming zigzag chains propagating along the *a*-axis direction. In addition, weak C—H···O and C—H···F bonds are observed. The contribution of the disordered solvent to the scattering was removed using the SQUEEZE routine [Spek (2015). Acta Cryst. C71, 9–18] of *PLATON*. The solvent contribution is not included in the reported molecular weight and density.

3D view



Chemical scheme



Structure description

In our recent work we have functionalized dye molecules with a C—Cl group, which facilitated halogen bonding (Shixaliyev *et al.*, 2018, 2019; Atioğlu *et al.*, 2019). In a continuation of this work, we have attached –CF_3 groups (potential halogen-bond donor/acceptor sites) to the aromatic moiety of the title hydrazone, $C_{32}H_{22}F_{12}N_4O_2$, which may lead to C—H···F, C—H···O, N—H···O and C—F···F non-covalent interactions in the crystal.

As shown in Fig. 1, the complete molecule is generated by a crystallographic twofold axis passing through the midpoint of the central C—C bond. The bond lengths and the bond angles are comparable to those observed in related structures, for example, 3-methyl-4-[(*Z*)-2-(4-methylphenyl)hydrazin-1-ylidene]-1-(3-nitrophenyl)-1*H*-pyrazol-

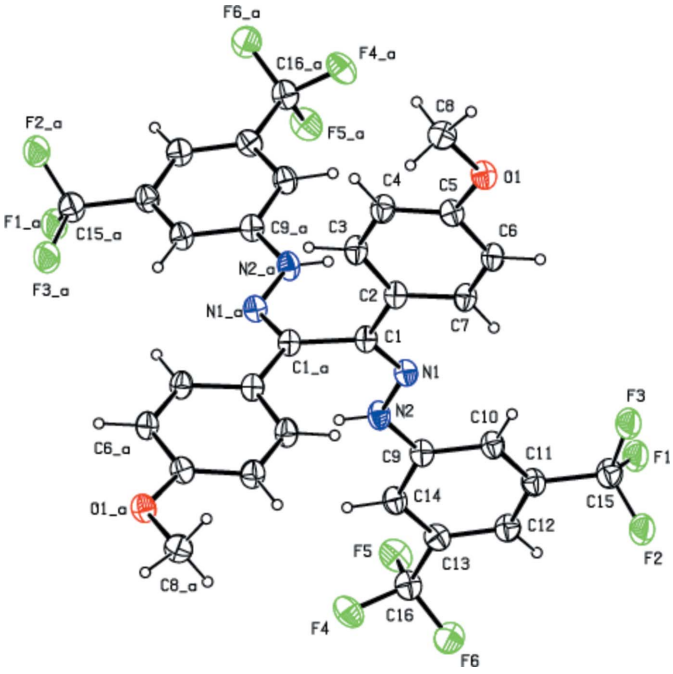


Figure 1
The molecular structure of the title compound with displacement ellipsoids for non-H atoms drawn at the 20% probability level. The minor components of the disordered CF_3 group have been omitted.

5(4*H*)-one and 3-methyl-4-[(*Z*)-2-(4-methylphenyl)hydrazin-1-ylidene]-1-[4-(trifluoromethyl)phenyl]-\1*H*-pyrazol-5(4*H*)-one (Alvarez-Thon *et al.*, 2014).

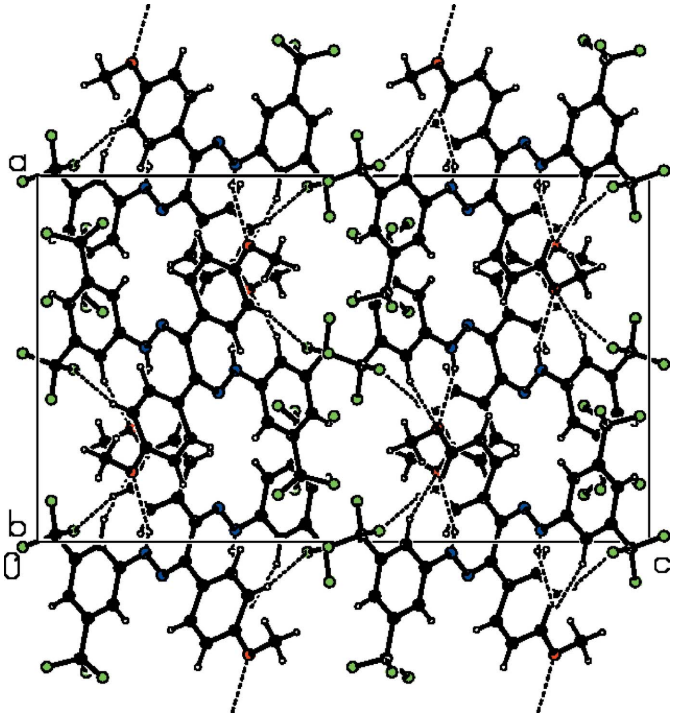


Figure 2
Crystal structure of the title compound viewed along the *b* axis. Dashed lines show hydrogen-bonding interactions. The minor components of the disordered CF_3 group have been omitted.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.79 (4)	2.32 (4)	3.041 (4)	152 (4)
C3—H3···F1^{ii}	0.95	2.42	3.279 (11)	151
C4—H4···F5^{iii}	0.95	2.51	3.439 (4)	165
C14—H14···O1^{i}	0.95	2.57	3.348 (5)	139

Symmetry codes: (i) $x-\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x-\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+1, y-1, -z+\frac{3}{2}$.

In the crystal of the title compound, molecules are linked by N2—H2···O1($x-\frac{1}{2}, y+\frac{1}{2}$) hydrogen bonds (Table 1; Fig. 2), forming zigzag chains propagating along the *a*-axis direction. C—H···O and C—H···F interactions consolidate the packing but π–π stacking and C—H···π interactions are not observed.

Synthesis and crystallization

The title compound was synthesized according to the reported method (Atioğlu *et al.*, 2019; Maharramov *et al.*, 2018; Shikhaliyev *et al.*, 2018, 2019). A 20 ml screw neck vial was charged with DMSO (10 ml), (*E*)-1-[3,5-*bis*(trifluoromethyl)phenyl]-2-(4-methoxybenzylidene)hydrazine (362 mg, 1 mmol), tetramethylethylenediamine (TMEDA) (295 mg, 2.5 mmol), CuCl (2 mg, 0.02 mmol) and CCl_4 (20 mmol, 10 equiv). After 1–3 h (until TLC analysis showed complete consumption of the corresponding Schiff base), the reaction mixture was poured into 0.01 *M* HCl (100 ml, pH = 2–3), and extracted with dichloromethane (3 × 20 ml). The combined organic phase was washed with water (3 × 50 ml), brine (30 ml), dried over anhydrous Na_2SO_4 and concentrated *in vacuo* using a rotary evaporator. The residue was purified by column chromatography on silica gel using appropriate mixtures of hexane and dichloromethane (3/1 − 1/1) giving an orange solid (63%); m.p. 393 K. Analysis calculated for $C_{32}H_{22}F_{12}N_4O_2$ (M = 722.53): C 53.19, H 3.07, N 7.75; found: C 53.07, H 3.01, N 7.74%. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.89 (6*H*, 2OCH_3), 7.00–8.23 (14*H*, Ar). ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.76, 155.49, 148.53, 126.67, 125.49, 119.54, 118.92, 118.66, 118.61, 109.29, 50.67, 25.14. ESI-MS: *m*/*z*: 723.44 [M + H]$^+$. The title compound was dissolved in dichloromethane and then left at room temperature for slow evaporation; colourless prisms started to form after *ca* 2 d.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The residual electron density was difficult to model and therefore, the SQUEEZE routine in *PLATON* (Spek, 2015) was used to remove the contribution of the electron density in the solvent region from the intensity data and the solvent-free model was employed for the final refinement. The solvent formula mass was not taken into account during refinement. The cavity of volume *ca* 384.5 Å^3 (*ca* 11% of the unit-cell volume) contains approximately 105 electrons. Fifteen outliers (0 2 2), (0 2 3), (1 3 3), (3 3 3), (3 5 4),

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{32}H_{22}F_{12}N_4O_2$
M_r	722.54
Crystal system, space group	Orthorhombic, *Pbcn*
Temperature (K)	100
a, *b*, *c* (Å)	13.479 (3), 11.213 (2), 22.552 (5)
V (Å^3)	3408.5 (12)
Z	4
Radiation type	Synchrotron, λ = 0.78790 Å
μ (mm^{-1})	0.17
Crystal size (mm)	0.18 × 0.12 × 0.10
Data collection	
Diffractometer	Rayonix SX165 CCD
Absorption correction	Multi-scan (*SCALA*; Evans, 2006)
T_{min}, T_{max}	0.954, 0.972
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	34331, 3971, 2478
R_{int}	0.062
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.654
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.079, 0.215, 1.02
No. of reflections	3971
No. of parameters	237
No. of restraints	18
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.52, −0.56

Computer programs: *Marccd* (Doyle, 2011), *iMosflm* (Battye *et al.*, 2011), *SHELXT2016/6* (Sheldrick, 2015*a*), *SHELXL2016/6* (Sheldrick, 2015*b*), *ORTEP*3for Windows (Farrugia, 2012) and *PLATON* (Spek, 2009).

(4 1 5), (0 12 1), (6 0 6), (1 7 2), (0 6 6), (3 1 8), (6 0 8), (0 4 2), (5 3 2) and (1 0 6) were omitted in the final cycles of refinement. The fluorine atoms of the $C15F_3$ group are disordered over three orientations in a 0.6: 0.2: 0.2 ratio. Within the disordered CF_3 group, the C—F distances were restrained to 1.330±0.003 Å, and the F—C—F and C—C—F bond angles were limited to near-tetrahedral values by restraining the F···F distances to 2.140±0.003 Å. The disordered F atoms (F1 F2 F3 F1′ F2′ F3′ F1" F2" F3") were restrained using the EADP command in *SHELX*.

Funding information

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References

Alvarez-Thon, L., Bustos, C., Molins, E., Garland, M. T. & Baggio, R. (2014). *Acta Cryst.* C**70**, 837–842.

Atioğlu, Z., Akkurt, M., Shikhaliyev, N. Q., Suleymanova, G. T., Bagirova, K. N. & Toze, F. A. A. (2019). *Acta Cryst.* E**75**, 237–241.

Battye, T. G. G., Kontogiannis, L., Johnson, O., Powell, H. R. & Leslie, A. G. W. (2011). *Acta Cryst*. D**67**, 271–281.

Doyle, R. A. (2011). *Marccd software manual*. Rayonix L. L. C., Evanston, IL 60201, USA.

Evans, P. (2006). *Acta Cryst.* D**62**, 72–82.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Maharramov, A. M., Shikhaliyev, N. Q., Suleymanova, G. T., Gurbanov, A. V., Babayeva, G. V., Mammadova, G. Z., Zubkov, F. I., Nenajdenko, V. G., Mahmudov, K. T. & Pombeiro, A. J. L. (2018). *Dyes Pigments*, **159**, 135–141.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* C**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Shikhaliyev, N. Q., Ahmadova, N. E., Gurbanov, A. V., Maharramov, A. M., Mammadova, G. Z., Nenajdenko, V. G., Zubkov, F. I., Mahmudov, K. T. & Pombeiro, A. J. L. (2018). *Dyes Pigments*, **150**, 377–381.

Spek, A. L. (2009). *Acta Cryst*. D**65**, 148–155.

Spek, A. L. (2015). *Acta Cryst.* C**71**, 9–18.

full crystallographic data

IUCrData (2019). **4**, x191010 [https://doi.org/10.1107/S2414314619010101]

(1*Z*,2*Z*)-1,2-Bis{2-[3,5-bis(trifluoromethyl)phenyl]hydrazinylidene}-1,2-bis(4-methoxyphenyl)ethane including an unknown solvate

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(1*Z*,2*Z*)-1,2-Bis{2-[3,5-bis(trifluoromethyl)phenyl]hydrazinylidene}-1,2-bis(4-methoxyphenyl)ethane

Crystal data

$C_{32}H_{22}F_{12}N_4O_2$
M_r = 722.54
Orthorhombic, *Pbcn*
a = 13.479 (3) Å
b = 11.213 (2) Å
c = 22.552 (5) Å
V = 3408.5 (12) Å^3
Z = 4
F(000) = 1464
D_x = 1.408 Mg m^{-3}
Synchrotron radiation, λ = 0.78790 Å
Cell parameters from 600 reflections
θ = 2.0–30.0°
μ = 0.17 mm^{-1}
T = 100 K
Prism, colourless
0.18 × 0.12 × 0.10 mm

Data collection

Rayonix SX165 CCD diffractometer
/f scan
Absorption correction: multi-scan (*SCALA*; Evans, 2006)
T_{min} = 0.954, T_{max} = 0.972
34331 measured reflections
3971 independent reflections
2478 reflections with $I > 2\sigma(I)$
R_{int} = 0.062
θ_{max} = 31.0°, θ_{min} = 2.0°
h = −17→17
k = −14→14
l = −29→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.079
$wR(F^2)$ = 0.215
S = 1.02
3971 reflections
237 parameters
18 restraints
Primary atom site location: difference Fourier map
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 4P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}
Extinction correction: SHELXL-2018/1 (Sheldrick 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0077 (7)

Special details

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

Refinement. The H atoms of aromatic and methyl groups were placed in calculated positions (C—H = 0.95 and 0.98 Å, respectively) and refined using a riding model withU_{iso}= 1.2U_{eq}(C-aromatic) and 1.5U_{eq}(*C*-methyl).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
F1	0.8573 (3)	0.6496 (2)	0.57941 (18)	0.0832 (7)	0.6
F2	0.8431 (3)	0.5107 (4)	0.51521 (11)	0.0832 (7)	0.6
F3	0.8715 (3)	0.4684 (3)	0.60651 (16)	0.0832 (7)	0.6
F1′	0.8618 (8)	0.6207 (7)	0.6062 (4)	0.0832 (7)	0.2
F2′	0.8426 (8)	0.5731 (9)	0.5152 (2)	0.0832 (7)	0.2
F3′	0.8670 (8)	0.4374 (5)	0.5803 (5)	0.0832 (7)	0.2
F1"	0.8783 (8)	0.5085 (9)	0.6148 (4)	0.0832 (7)	0.2
F2"	0.8552 (7)	0.6378 (6)	0.5458 (5)	0.0832 (7)	0.2
F3"	0.8270 (8)	0.4531 (7)	0.5295 (4)	0.0832 (7)	0.2
F4	0.38927 (16)	0.6493 (2)	0.52250 (11)	0.0811 (7)	
F5	0.47074 (18)	0.79502 (19)	0.55878 (11)	0.0837 (7)	
F6	0.51490 (18)	0.7197 (2)	0.47565 (11)	0.0857 (7)	
O1	0.80893 (17)	−0.1149 (2)	0.84552 (11)	0.0618 (6)	
N1	0.59184 (19)	0.2977 (2)	0.70406 (12)	0.0541 (6)	
N2	0.5315 (2)	0.3763 (2)	0.67618 (14)	0.0586 (7)	
H2	0.474 (3)	0.370 (4)	0.6825 (17)	0.070*	
C1	0.5545 (2)	0.2249 (3)	0.74170 (15)	0.0513 (7)	
C2	0.6199 (2)	0.1371 (3)	0.77029 (15)	0.0543 (8)	
C3	0.5875 (3)	0.0684 (3)	0.81721 (16)	0.0603 (8)	
H3	0.5219	0.0796	0.8317	0.072*	
C4	0.6476 (3)	−0.0165 (3)	0.84393 (17)	0.0627 (9)	
H4	0.6235	−0.0628	0.8761	0.075*	
C5	0.7433 (2)	−0.0327 (3)	0.82289 (15)	0.0558 (8)	
C6	0.7778 (2)	0.0366 (3)	0.77612 (15)	0.0581 (8)	
H6	0.8437	0.0263	0.7621	0.070*	
C7	0.7173 (2)	0.1194 (3)	0.75029 (16)	0.0567 (8)	
H7	0.7417	0.1659	0.7182	0.068*	
C8	0.7725 (3)	−0.1975 (3)	0.88859 (17)	0.0694 (10)	
H8A	0.7180	−0.2439	0.8714	0.104*	
H8B	0.7485	−0.1538	0.9234	0.104*	
H8C	0.8261	−0.2515	0.9005	0.104*	
C9	0.5710 (2)	0.4527 (3)	0.63442 (15)	0.0564 (8)	
C10	0.6731 (2)	0.4553 (3)	0.62320 (15)	0.0580 (8)	
H10	0.7165	0.4028	0.6436	0.070*	
C11	0.7098 (3)	0.5348 (3)	0.58237 (16)	0.0610 (8)	
C12	0.6496 (3)	0.6129 (3)	0.55145 (16)	0.0627 (9)	
H12	0.6768	0.6684	0.5241	0.075*	

C13	0.5475 (3)	0.6076 (3)	0.56162 (16)	0.0595 (8)
C14	0.5086 (3)	0.5286 (3)	0.60240 (16)	0.0600 (8)
H14	0.4389	0.5259	0.6088	0.072*
C15	0.8195 (2)	0.5397 (2)	0.57040 (12)	0.0723 (10)
C16	0.4815 (3)	0.6924 (3)	0.52972 (18)	0.0669 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0625 (9)	0.0989 (17)	0.0883 (16)	−0.0094 (12)	0.0081 (11)	0.0227 (13)
F2	0.0625 (9)	0.0989 (17)	0.0883 (16)	−0.0094 (12)	0.0081 (11)	0.0227 (13)
F3	0.0625 (9)	0.0989 (17)	0.0883 (16)	−0.0094 (12)	0.0081 (11)	0.0227 (13)
F1′	0.0625 (9)	0.0989 (17)	0.0883 (16)	−0.0094 (12)	0.0081 (11)	0.0227 (13)
F2′	0.0625 (9)	0.0989 (17)	0.0883 (16)	−0.0094 (12)	0.0081 (11)	0.0227 (13)
F3′	0.0625 (9)	0.0989 (17)	0.0883 (16)	−0.0094 (12)	0.0081 (11)	0.0227 (13)
F1"	0.0625 (9)	0.0989 (17)	0.0883 (16)	−0.0094 (12)	0.0081 (11)	0.0227 (13)
F2"	0.0625 (9)	0.0989 (17)	0.0883 (16)	−0.0094 (12)	0.0081 (11)	0.0227 (13)
F3"	0.0625 (9)	0.0989 (17)	0.0883 (16)	−0.0094 (12)	0.0081 (11)	0.0227 (13)
F4	0.0686 (13)	0.0683 (13)	0.1064 (17)	0.0058 (11)	−0.0214 (12)	0.0126 (12)
F5	0.0879 (15)	0.0552 (12)	0.1079 (17)	0.0166 (11)	−0.0122 (13)	−0.0034 (12)
F6	0.0849 (15)	0.0845 (16)	0.0877 (16)	0.0244 (13)	0.0063 (13)	0.0265 (12)
O1	0.0574 (13)	0.0502 (12)	0.0779 (15)	0.0070 (10)	−0.0006 (12)	0.0084 (11)
N1	0.0492 (13)	0.0475 (13)	0.0655 (16)	0.0000 (11)	0.0017 (12)	0.0036 (12)
N2	0.0458 (14)	0.0519 (15)	0.0781 (19)	0.0036 (12)	0.0052 (13)	0.0136 (13)
C1	0.0476 (15)	0.0424 (15)	0.0638 (18)	−0.0013 (13)	0.0054 (14)	−0.0024 (13)
C2	0.0501 (16)	0.0450 (15)	0.0677 (19)	−0.0006 (13)	0.0067 (15)	−0.0014 (14)
C3	0.0507 (17)	0.0526 (17)	0.078 (2)	0.0018 (14)	0.0086 (16)	0.0090 (16)
C4	0.0589 (19)	0.0563 (19)	0.073 (2)	0.0023 (16)	0.0088 (17)	0.0146 (16)
C5	0.0529 (17)	0.0455 (16)	0.0691 (19)	0.0036 (13)	−0.0009 (15)	0.0008 (15)
C6	0.0500 (17)	0.0546 (18)	0.070 (2)	0.0044 (14)	0.0083 (15)	0.0011 (16)
C7	0.0509 (17)	0.0499 (16)	0.0692 (19)	0.0015 (14)	0.0098 (15)	0.0064 (15)
C8	0.075 (2)	0.0582 (19)	0.075 (2)	0.0094 (18)	−0.0009 (19)	0.0113 (18)
C9	0.0537 (17)	0.0478 (16)	0.068 (2)	0.0013 (14)	0.0037 (15)	0.0055 (15)
C10	0.0511 (17)	0.0511 (17)	0.072 (2)	−0.0008 (14)	0.0014 (15)	0.0075 (15)
C11	0.0525 (17)	0.0577 (19)	0.073 (2)	−0.0002 (15)	0.0010 (16)	0.0095 (16)
C12	0.063 (2)	0.0541 (18)	0.070 (2)	−0.0006 (16)	0.0032 (17)	0.0106 (16)
C13	0.0610 (19)	0.0474 (16)	0.070 (2)	0.0041 (15)	−0.0047 (17)	0.0054 (15)
C14	0.0524 (17)	0.0525 (18)	0.075 (2)	0.0046 (14)	0.0000 (16)	0.0029 (16)
C15	0.063 (2)	0.071 (2)	0.083 (3)	−0.0008 (18)	0.0034 (19)	0.022 (2)
C16	0.065 (2)	0.0529 (18)	0.083 (3)	0.0053 (16)	−0.0003 (19)	0.0067 (18)

Geometric parameters (Å, º)

F1—C15	1.349 (3)	C3—C4	1.388 (5)
F2—C15	1.325 (3)	C3—H3	0.9500
F3—C15	1.339 (3)	C4—C5	1.386 (5)
F1′—C15	1.342 (3)	C4—H4	0.9500
F2′—C15	1.336 (3)	C5—C6	1.391 (5)

F3′—C15	1.333 (3)	C6—C7	1.366 (4)
F1"—C15	1.324 (3)	C6—H6	0.9500
F2"—C15	1.323 (3)	C7—H7	0.9500
F3"—C15	1.343 (3)	C8—H8A	0.9800
F4—C16	1.344 (4)	C8—H8B	0.9800
F5—C16	1.332 (4)	C8—H8C	0.9800
F6—C16	1.335 (4)	C9—C14	1.398 (5)
O1—C5	1.375 (4)	C9—C10	1.399 (4)
O1—C8	1.429 (4)	C10—C11	1.373 (5)
N1—C1	1.280 (4)	C10—H10	0.9500
N1—N2	1.355 (4)	C11—C12	1.383 (5)
N2—C9	1.380 (4)	C11—C15	1.504 (4)
N2—H2	0.79 (4)	C12—C13	1.396 (5)
C1—C2	1.471 (4)	C12—H12	0.9500
C1—C1^{i}	1.516 (6)	C13—C14	1.380 (5)
C2—C3	1.380 (4)	C13—C16	1.488 (5)
C2—C7	1.403 (4)	C14—H14	0.9500
C5—O1—C8	117.7 (3)	C9—C10—H10	120.3
C1—N1—N2	119.1 (3)	C10—C11—C12	122.5 (3)
N1—N2—C9	119.3 (3)	C10—C11—C15	119.9 (3)
N1—N2—H2	116 (3)	C12—C11—C15	117.6 (3)
C9—N2—H2	124 (3)	C11—C12—C13	118.0 (3)
N1—C1—C2	118.8 (3)	C11—C12—H12	121.0
N1—C1—C1^{i}	123.0 (3)	C13—C12—H12	121.0
C2—C1—C1^{i}	118.2 (3)	C14—C13—C12	120.7 (3)
C3—C2—C7	117.7 (3)	C14—C13—C16	120.4 (3)
C3—C2—C1	121.3 (3)	C12—C13—C16	118.8 (3)
C7—C2—C1	121.0 (3)	C13—C14—C9	120.4 (3)
C2—C3—C4	122.1 (3)	C13—C14—H14	119.8
C2—C3—H3	119.0	C9—C14—H14	119.8
C4—C3—H3	119.0	F2"—C15—F1"	108.6 (3)
C5—C4—C3	119.0 (3)	F3′—C15—F2′	106.6 (3)
C5—C4—H4	120.5	F2—C15—F3	107.4 (3)
C3—C4—H4	120.5	F3′—C15—F1′	106.1 (3)
O1—C5—C4	124.0 (3)	F2′—C15—F1′	105.7 (3)
O1—C5—C6	116.1 (3)	F2"—C15—F3"	106.6 (3)
C4—C5—C6	119.8 (3)	F1"—C15—F3"	106.5 (3)
C7—C6—C5	120.3 (3)	F2—C15—F1	106.0 (2)
C7—C6—H6	119.9	F3—C15—F1	104.8 (2)
C5—C6—H6	119.9	F2"—C15—C11	117.6 (5)
C6—C7—C2	121.2 (3)	F1"—C15—C11	116.3 (6)
C6—C7—H7	119.4	F2—C15—C11	113.3 (3)
C2—C7—H7	119.4	F3′—C15—C11	114.2 (5)
O1—C8—H8A	109.5	F2′—C15—C11	114.0 (5)
O1—C8—H8B	109.5	F3—C15—C11	112.5 (3)
H8A—C8—H8B	109.5	F1′—C15—C11	109.6 (5)
O1—C8—H8C	109.5	F3"—C15—C11	99.8 (5)

H8A—C8—H8C	109.5	F1—C15—C11	112.2 (3)
H8B—C8—H8C	109.5	F6—C16—F5	106.8 (3)
N2—C9—C14	119.9 (3)	F6—C16—F4	106.5 (3)
N2—C9—C10	121.1 (3)	F5—C16—F4	105.6 (3)
C14—C9—C10	119.0 (3)	F6—C16—C13	112.7 (3)
C11—C10—C9	119.3 (3)	F5—C16—C13	112.3 (3)
C11—C10—H10	120.3	F4—C16—C13	112.4 (3)
C1—N1—N2—C9	−178.3 (3)	C12—C13—C14—C9	0.2 (5)
N2—N1—C1—C2	177.9 (3)	C16—C13—C14—C9	−177.0 (3)
N2—N1—C1—C1^{i}	−2.0 (5)	N2—C9—C14—C13	178.3 (3)
N1—C1—C2—C3	170.4 (3)	C10—C9—C14—C13	−1.9 (5)
C1^{i}—C1—C2—C3	−9.6 (5)	C10—C11—C15—F2"	−159.7 (6)
N1—C1—C2—C7	−10.2 (5)	C12—C11—C15—F2"	19.7 (7)
C1^{i}—C1—C2—C7	169.7 (3)	C10—C11—C15—F1"	−28.4 (6)
C7—C2—C3—C4	−0.6 (5)	C12—C11—C15—F1"	151.0 (6)
C1—C2—C3—C4	178.7 (3)	C10—C11—C15—F2	116.9 (4)
C2—C3—C4—C5	0.1 (6)	C12—C11—C15—F2	−63.7 (4)
C8—O1—C5—C4	7.5 (5)	C10—C11—C15—F3′	27.3 (6)
C8—O1—C5—C6	−172.5 (3)	C12—C11—C15—F3′	−153.3 (6)
C3—C4—C5—O1	−179.4 (3)	C10—C11—C15—F2′	150.2 (6)
C3—C4—C5—C6	0.7 (5)	C12—C11—C15—F2′	−30.4 (6)
O1—C5—C6—C7	179.1 (3)	C10—C11—C15—F3	−5.2 (5)
C4—C5—C6—C7	−0.9 (5)	C12—C11—C15—F3	174.2 (3)
C5—C6—C7—C2	0.4 (5)	C10—C11—C15—F1′	−91.5 (6)
C3—C2—C7—C6	0.4 (5)	C12—C11—C15—F1′	87.9 (6)
C1—C2—C7—C6	−179.0 (3)	C10—C11—C15—F3"	85.6 (6)
N1—N2—C9—C14	175.6 (3)	C12—C11—C15—F3"	−95.0 (6)
N1—N2—C9—C10	−4.1 (5)	C10—C11—C15—F1	−123.2 (4)
N2—C9—C10—C11	−178.3 (3)	C12—C11—C15—F1	56.2 (4)
C14—C9—C10—C11	1.9 (5)	C14—C13—C16—F6	−149.8 (3)
C9—C10—C11—C12	−0.1 (6)	C12—C13—C16—F6	32.9 (5)
C9—C10—C11—C15	179.2 (3)	C14—C13—C16—F5	89.5 (4)
C10—C11—C12—C13	−1.6 (6)	C12—C13—C16—F5	−87.8 (4)
C15—C11—C12—C13	179.0 (3)	C14—C13—C16—F4	−29.5 (5)
C11—C12—C13—C14	1.6 (5)	C12—C13—C16—F4	153.3 (3)
C11—C12—C13—C16	178.8 (3)		

Symmetry code: (i) −*x*+1, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{ii}	0.79 (4)	2.32 (4)	3.041 (4)	152 (4)
C3—H3···F1"^{iii}	0.95	2.42	3.279 (11)	151
C4—H4···F5^{iv}	0.95	2.51	3.439 (4)	165

C10—H10···F3	0.95	2.37	2.705 (5)	100
C14—H14···O1^{ii}	0.95	2.57	3.348 (5)	139

Symmetry codes: (ii) $x-1/2, y+1/2, -z+3/2$; (iii) $x-1/2, y-1/2, -z+3/2$; (iv) $-x+1, y-1, -z+3/2$.