Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N^2,N^2,N^6,N^6-Tetrakis(2,3,4,5,6-pentafluorobenzoyl)pyridine-2,6-diamine

Arto Valkonen,[a]* Erkki Kolehmainen[a] and Borys Ośmiałowski[b]

[a]University of Jyväskylä, Department of Chemistry, PO Box 35, FIN-40014 Jyväskylä, Finland, and [b]University of Technology and Life Sciences, Department of Chemistry, Seminaryjna 3, PL-85-326 Bydgoszcz, Poland
Correspondence e-mail: arto.m.valkonen@jyu.fi

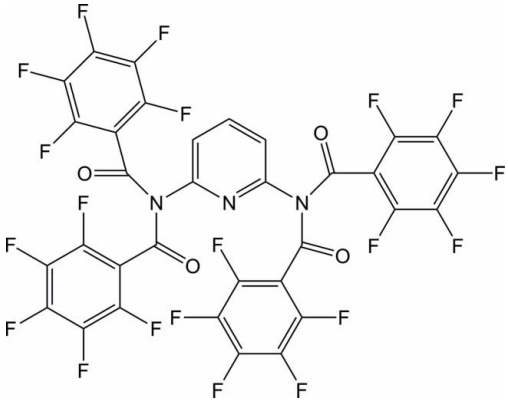
Received 14 November 2011; accepted 16 November 2011

Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.057; wR factor = 0.133; data-to-parameter ratio = 10.3.

The title compound, $C_{33}H_3F_{20}N_3O_4$, is a highly fluorinated organic imide that was isolated as an unexpected product from the reaction of 2,6-diaminopyridine with 2,3,4,5,6-pentafluorobenzoyl chloride in a 1:2 molar ratio. The molecule is located on a twofold axis and one of its symmetry-independent 2,3,4,5,6-pentafluorobenzoyl groups is disordered over two sets of sites, the occupancy of the major component being 0.773 (3). In the major component, the dihedral angle between the perfluorophenyl groups is 63.64 (10)°, and these groups form dihedral angles of 67.14 (7) and 21.1 (2)° with the pyridine core. Short intermolecular C—H···O and C—H···N contacts are found in the crystal structure.

Related literature

For preparation of 2-acylaminopyridines and their structures, see: Ośmiałowski *et al.* (2010*a*,*b*). For related structures, see: Kovalevsky *et al.* (1999).



Experimental

Crystal data

$C_{33}H_3F_{20}N_3O_4$
M_r = 885.38
Monoclinic, $C2/c$
a = 21.2370 (5) Å
b = 6.3940 (1) Å
c = 23.1045 (5) Å
β = 100.585 (1)°
V = 3083.96 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 123 K
0.30 × 0.18 × 0.16 mm

Data collection

Bruker–Nonius KappaCCD with an APEXII detector diffractometer
7138 measured reflections
3785 independent reflections
2526 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.133
S = 1.05
3785 reflections
366 parameters
97 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···N1^{i}	0.95	2.67	3.621 (5)	180
C3—H3···O1^{i}	0.95	2.52	3.318 (3)	141

Symmetry code: (i) $x, y-1, z$.

Data collection: *COLLECT* (Bruker, 2008); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*.

Academy Professor Kari Rissanen and the Academy of Finland (project No. 212588) are thanked for financial support to AV. The Polish Ministry of Science and Higher Education (grant No. N N204 174138 to BO) is also gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2434).

References

Bruker (2008). *COLLECT*. Bruker AXS, Delft, The Netherlands.
Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Kovalevsky, A. Y., Shishkin, O. V. & Ponomarev, I. I. (1999). *Acta Cryst.* C**55**, 1914–1915.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Ośmiałowski, B., Kolehmainen, E., Dobosz, R., Gawinecki, R., Kauppinen, R., Valkonen, A., Koivukorpi, J. & Rissanen, K. (2010*a*). *J. Phys. Chem. A*, **114**, 10421–10426.
Ośmiałowski, B., Kolehmainen, E., Gawinecki, R., Kauppinen, R., Koivukorpi, J. & Valkonen, A. (2010*b*). *Struct. Chem.* **21**, 1061–1067.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2011). E**67**, o3429-o3430 [**doi:10.1107/S1600536811048768**]

N^2,N^2,N^6,N^6-Tetrakis(2,3,4,5,6-pentafluorobenzoyl)pyridine-2,6-diamine

A. Valkonen, E. Kolehmainen and B. Osmialowski

Comment

The title compound was accidentally obtained by reaction of 2,6-diaminopyridine with two equivalents of 2,3,4,5,6-pentafluorobenzoyl chloride, while the preparation of *N*,*N*′-bis(pentafluorobenzoyl)-2,6-diaminopyridine was attempted. The reaction was carried out analogously to our previously reported preparations of 2-acylaminopyridines (Ośmiałowski *et al.*, 2010*a*). Previously we have structurally characterized two related secondary amides (Ośmiałowski *et al.*, 2010*b*). This title imide has not been previously reported in the literature. The crystal structure of closely related imide, *N*,*N*′-(pyridine-2,6-diyl)-bis(naphthalenedicarboximide), has been reported (Kovalevsky *et al.*, 1999).

In the crystal molecules are located on a twofold rotation axis and one of the symmetry independent perfluorobenzoyl group is disordered over two sets of sites with different occupancies (Fig.1). The perfluorophenyl groups are twisted out of the pyridine core by 67.14 (7) and 21.1 (2) ° in the major component whereas in the minor component these angles are 67.14 (7) and 63.4 (4)°. Furthermore, the dihedral angle between perfluorophenyl - group planes is 63.64 (10) ° [the minor component 67.9 (4) °]. Mercury (Macrae *et al.*, 2008) helped us to find a motif along [0-10] direction (Fig. 2), where the molecule is connected to the translation related one by two C—H···O contacts (Table 1). Between these molecules there is also one rather long but linear C—H···N contact. A few F···C_{Ar}, F···F and F···O type contacts were found, which are only slightly shorter than the sum of van der Waals radii.

Experimental

2,3,4,5,6-Pentafluorobenzoyl chloride (2.28 g, 10 mmol) was added dropwise to a magnetically stirred solution of 2,6-diaminopyridine (0.54 g, 5 mmol) and triethylamine (1 ml) in dry methylene chloride (6 ml) at 0 °C. Subsequently the reaction mixture was refluxed for 4 h and the solution was treated with water and extracted with CH_2Cl_2. The organic solvent of the extract was evaporated under reduced pressure and the product recrystallized from hexane/ethyl acetate (10:1) mixture. ^{1}H NMR ($CDCl_3$): δ (p.p.m.) = 8.00 (t, 1H, H4), 7.45 (d, 2H, H3). Single crystals suitable for X-ray diffraction were obtained by very slow evaporation of analytical sample from NMR-tube, where $CDCl_3$ was used as a solvent.

Refinement

All H atoms were visible in electron density maps, but were calculated at their idealized positions and allowed to ride on their parent atoms at C—H distances of 0.95 Å with U_{iso}(H) of 1.2 times U_{eq}(C). A large number of restraints was needed to rationalize the disorder in perfluorobenzoyl group. FLAT (2 restraints, s=0.1) was applied to amide groups of both components to make them more planar. SADI (3 restraints, s=0.02) was applied for amide groups of both components to equalize the bond distances. DELU (2 restraints, s1=s2=0.01) was applied for one C—C and one C—F bond of the major component to equalize the anisotropic displacement parameters. SIMU (84 restraints, s=0.01, st=0.02, dmax=1.7) was applied to carbons of the disordered perfluorophenyl ring and carbonyl group of both components to equalize the anisotropic

displacement parameters. ISOR (6 restraints, s=0.01, st=0.02) was applied to carbonyl oxygen atom of the minor component to prevent the atom to appear as non-positive definite.

Figures

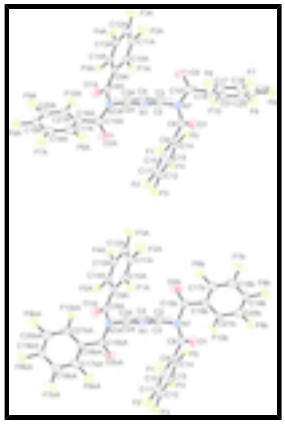


Fig. 1. The structures of the major (top) and the minor (bottom) components of the disordered title molecule showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

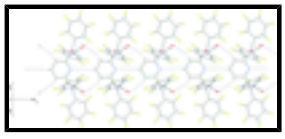


Fig. 2. The short C—H···O/N contacts (blue dashed lines) organizing the molecules in the [0 1 0] direction. The minor component of disordered part of the molecule was removed for clarity.

N^2,N^2,N^6,N^6- Tetrakis(2,3,4,5,6-pentafluorobenzoyl)pyridine-2,6-diamine

Crystal data

$C_{33}H_3F_{20}N_3O_4$	$F(000)$ = 1736
M_r = 885.38	D_x = 1.907 Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 4030 reflections
a = 21.2370 (5) Å	θ = 0.4–28.3°
b = 6.3940 (1) Å	μ = 0.21 mm^{-1}
c = 23.1045 (5) Å	T = 123 K
β = 100.585 (1)°	Block, colourless
V = 3083.96 (11) Å^3	0.30 × 0.18 × 0.16 mm
Z = 4	

Data collection

Bruker–Nonius KappaCCD with an APEXII detector diffractometer	2526 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.039
graphite	θ_{max} = 28.2°, θ_{min} = 1.8°
Detector resolution: 9 pixels mm^{-1}	h = −27→28
φ and ω scans	k = −8→8
7138 measured reflections	l = −30→30
3785 independent reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.057$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.133$	H-atom parameters constrained
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.0381P)^2 + 7.2159P]$ where $P = (F_o^2 + 2F_c^2)/3$
3785 reflections	$(\Delta/\sigma)_{max} < 0.001$
366 parameters	$\Delta\rho_{max} = 0.35$ e Å^{-3}
97 restraints	$\Delta\rho_{min} = -0.42$ e Å^{-3}

Special details

Experimental. ^{13}C NMR ($CDCl_3$): δ (ppm) = 159.4, 149.1, 144.5, 142.4, 141.7, 138.7, 136.6, 123.8, 110.5.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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)
F1	0.86727 (7)	0.9378 (2)	0.69841 (7)	0.0376 (4)	
F2	0.78577 (10)	0.8370 (3)	0.59909 (7)	0.0616 (5)	
F3	0.72661 (9)	0.4583 (3)	0.58930 (7)	0.0579 (5)	
F4	0.75118 (6)	0.1782 (2)	0.67885 (7)	0.0362 (4)	
F5	0.82680 (7)	0.2875 (2)	0.78079 (6)	0.0345 (4)	
O1	0.87489 (9)	0.8475 (3)	0.82339 (8)	0.0380 (4)	
N7	0.93905 (9)	0.5589 (3)	0.82488 (9)	0.0330 (5)	
N1	1.0000	0.5463 (4)	0.7500	0.0278 (6)	
C2	0.97130 (10)	0.4368 (4)	0.78664 (11)	0.0303 (5)	
C3	0.97017 (12)	0.2207 (4)	0.78901 (14)	0.0392 (7)	
H3	0.9496	0.1495	0.8165	0.047*	
C4	1.0000	0.1126 (6)	0.7500	0.0450 (11)	
H4	1.0000	−0.0360	0.7500	0.054*	
C8	0.88798 (11)	0.6875 (4)	0.80002 (11)	0.0295 (5)	
C9	0.84960 (10)	0.6158 (4)	0.74248 (10)	0.0253 (5)	
C10	0.83779 (11)	0.7515 (4)	0.69504 (11)	0.0293 (5)	
C11	0.79688 (13)	0.6999 (4)	0.64361 (11)	0.0373 (6)	

C12	0.76727 (13)	0.5075 (4)	0.63878 (11)	0.0368 (6)	
C13	0.77919 (11)	0.3668 (4)	0.68450 (11)	0.0294 (5)	
C14	0.81941 (10)	0.4222 (4)	0.73576 (10)	0.0257 (5)	
O2	1.02137 (19)	0.5421 (8)	0.9007 (2)	0.0851 (16)	0.773 (2)
C15	0.9663 (3)	0.5901 (8)	0.8850 (2)	0.0484 (16)	0.773 (2)
F6	0.96821 (11)	0.9929 (4)	0.93716 (10)	0.0545 (6)	0.773 (2)
F7	0.90327 (13)	1.0815 (4)	1.02541 (11)	0.0641 (8)	0.773 (2)
F8	0.82644 (14)	0.7899 (4)	1.06022 (10)	0.0548 (7)	0.773 (2)
F9	0.81043 (14)	0.4137 (4)	1.00624 (12)	0.0565 (7)	0.773 (2)
F10	0.87435 (13)	0.3237 (4)	0.91753 (12)	0.0533 (7)	0.773 (2)
C16	0.92394 (13)	0.6546 (4)	0.92708 (10)	0.0413 (10)	0.773 (2)
C17	0.93101 (11)	0.8490 (4)	0.95442 (11)	0.0429 (9)	0.773 (2)
C18	0.89756 (12)	0.8949 (3)	0.99922 (10)	0.0451 (9)	0.773 (2)
C19	0.85705 (12)	0.7464 (4)	1.01668 (10)	0.0432 (9)	0.773 (2)
C20	0.84999 (14)	0.5520 (4)	0.98934 (13)	0.0407 (11)	0.773 (2)
C21	0.88343 (15)	0.5061 (3)	0.94454 (13)	0.0381 (12)	0.773 (2)
O2B	1.0322 (5)	0.4514 (16)	0.8935 (5)	0.041 (3)	0.227 (2)
C15B	0.9785 (6)	0.524 (2)	0.8821 (5)	0.027 (4)	0.227 (2)
F6B	1.0454 (3)	0.7631 (12)	0.9763 (3)	0.048 (2)	0.227 (2)
F7B	0.9953 (4)	0.8597 (13)	1.0711 (3)	0.060 (2)	0.227 (2)
F8B	0.8734 (5)	0.7549 (14)	1.0761 (3)	0.059 (2)	0.227 (2)
F9B	0.8023 (5)	0.5329 (19)	0.9872 (4)	0.058 (3)	0.227 (2)
F10B	0.8522 (4)	0.4215 (13)	0.8935 (3)	0.0397 (18)	0.227 (2)
C16B	0.9465 (4)	0.6015 (14)	0.9305 (3)	0.029 (3)	0.227 (2)
C17B	0.9844 (3)	0.7046 (14)	0.9773 (3)	0.040 (2)	0.227 (2)
C18B	0.9591 (4)	0.7582 (14)	1.0267 (3)	0.043 (3)	0.227 (2)
C19B	0.8959 (4)	0.7086 (15)	1.0293 (3)	0.042 (3)	0.227 (2)
C20B	0.8581 (3)	0.6055 (17)	0.9825 (4)	0.034 (3)	0.227 (2)
C21B	0.8833 (4)	0.5519 (16)	0.9331 (4)	0.025 (3)	0.227 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0398 (8)	0.0247 (7)	0.0498 (9)	−0.0086 (6)	0.0121 (7)	0.0033 (7)
F2	0.0912 (14)	0.0489 (11)	0.0402 (9)	−0.0188 (10)	0.0006 (9)	0.0136 (8)
F3	0.0692 (12)	0.0568 (12)	0.0403 (9)	−0.0210 (10)	−0.0093 (8)	−0.0015 (9)
F4	0.0296 (7)	0.0285 (8)	0.0512 (9)	−0.0101 (6)	0.0095 (6)	−0.0078 (7)
F5	0.0346 (7)	0.0277 (8)	0.0409 (8)	−0.0072 (6)	0.0062 (6)	0.0061 (6)
O1	0.0464 (11)	0.0271 (9)	0.0434 (10)	−0.0083 (8)	0.0159 (8)	−0.0102 (8)
N7	0.0269 (10)	0.0343 (12)	0.0373 (11)	−0.0054 (9)	0.0049 (9)	−0.0024 (10)
N1	0.0174 (12)	0.0214 (14)	0.0433 (17)	0.000	0.0021 (12)	0.000
C2	0.0179 (10)	0.0263 (12)	0.0447 (14)	−0.0012 (10)	0.0000 (10)	0.0012 (11)
C3	0.0236 (12)	0.0271 (13)	0.0656 (19)	−0.0033 (10)	0.0042 (12)	0.0084 (13)
C4	0.0240 (17)	0.0192 (17)	0.089 (3)	0.000	0.0020 (19)	0.000
C8	0.0276 (11)	0.0245 (12)	0.0386 (13)	−0.0081 (10)	0.0118 (10)	−0.0014 (11)
C9	0.0219 (10)	0.0237 (11)	0.0331 (12)	−0.0003 (9)	0.0118 (9)	−0.0031 (10)
C10	0.0304 (12)	0.0201 (11)	0.0400 (14)	−0.0056 (10)	0.0140 (11)	−0.0031 (11)
C11	0.0457 (15)	0.0356 (15)	0.0319 (13)	−0.0049 (12)	0.0105 (11)	0.0059 (12)

C12	0.0383 (14)	0.0381 (15)	0.0329 (13)	−0.0081 (12)	0.0033 (11)	−0.0051 (12)
C13	0.0256 (11)	0.0241 (12)	0.0408 (14)	−0.0059 (10)	0.0121 (10)	−0.0057 (11)
C14	0.0228 (10)	0.0230 (12)	0.0330 (12)	0.0016 (9)	0.0095 (9)	−0.0003 (10)
O2	0.040 (2)	0.106 (4)	0.094 (3)	0.032 (2)	−0.0279 (18)	−0.061 (3)
C15	0.035 (2)	0.041 (3)	0.062 (3)	0.009 (2)	−0.011 (2)	−0.026 (2)
F6	0.0586 (14)	0.0444 (14)	0.0607 (15)	−0.0048 (12)	0.0116 (12)	−0.0191 (12)
F7	0.0776 (17)	0.0436 (14)	0.0734 (17)	−0.0001 (13)	0.0204 (13)	−0.0319 (13)
F8	0.0691 (17)	0.0515 (15)	0.0451 (13)	0.0136 (13)	0.0142 (12)	−0.0149 (11)
F9	0.0761 (18)	0.0438 (16)	0.0508 (16)	0.0007 (15)	0.0151 (14)	−0.0041 (13)
F10	0.0680 (17)	0.0360 (14)	0.0561 (16)	0.0013 (12)	0.0117 (13)	−0.0186 (12)
C16	0.038 (2)	0.036 (2)	0.043 (2)	0.0126 (18)	−0.0102 (16)	−0.0140 (17)
C17	0.0409 (18)	0.0379 (19)	0.045 (2)	0.0075 (16)	−0.0048 (16)	−0.0116 (17)
C18	0.050 (2)	0.0365 (19)	0.044 (2)	0.0138 (17)	−0.0044 (16)	−0.0166 (17)
C19	0.047 (2)	0.044 (2)	0.0341 (18)	0.0151 (18)	−0.0019 (16)	−0.0082 (17)
C20	0.055 (3)	0.029 (2)	0.033 (2)	0.0114 (19)	−0.006 (2)	−0.0009 (18)
C21	0.048 (2)	0.023 (2)	0.037 (2)	0.0125 (17)	−0.0080 (18)	−0.0023 (18)
O2B	0.040 (5)	0.030 (5)	0.044 (5)	0.004 (4)	−0.016 (4)	0.017 (4)
C15B	0.030 (8)	0.027 (8)	0.024 (6)	−0.005 (6)	0.005 (5)	0.009 (6)
F6B	0.048 (4)	0.050 (5)	0.040 (4)	−0.003 (4)	−0.007 (3)	0.011 (4)
F7B	0.083 (6)	0.049 (5)	0.038 (4)	0.001 (5)	−0.012 (4)	−0.003 (4)
F8B	0.077 (6)	0.065 (6)	0.038 (4)	0.022 (5)	0.020 (4)	−0.007 (4)
F9B	0.067 (6)	0.076 (7)	0.036 (5)	−0.012 (6)	0.018 (4)	−0.005 (5)
F10B	0.055 (5)	0.038 (4)	0.025 (3)	−0.013 (4)	0.004 (3)	−0.006 (3)
C16B	0.042 (6)	0.023 (5)	0.020 (4)	0.001 (5)	0.002 (4)	0.000 (4)
C17B	0.053 (6)	0.031 (5)	0.032 (5)	−0.001 (5)	0.000 (4)	0.003 (4)
C18B	0.060 (6)	0.039 (5)	0.027 (5)	0.007 (5)	−0.001 (5)	−0.002 (4)
C19B	0.059 (6)	0.045 (6)	0.024 (5)	0.006 (5)	0.015 (5)	−0.001 (4)
C20B	0.046 (6)	0.021 (5)	0.037 (5)	0.005 (5)	0.009 (5)	0.014 (5)
C21B	0.044 (6)	0.009 (4)	0.020 (5)	0.003 (4)	0.001 (4)	0.002 (4)

Geometric parameters (Å, °)

F1—C10	1.341 (3)	C15—C16	1.498 (6)
F2—C11	1.339 (3)	F6—C17	1.321 (3)
F3—C12	1.338 (3)	F7—C18	1.333 (3)
F4—C13	1.340 (3)	F8—C19	1.324 (3)
F5—C14	1.338 (3)	F9—C20	1.327 (4)
O1—C8	1.213 (3)	F10—C21	1.320 (3)
N7—C8	1.398 (3)	C16—C17	1.3900
N7—C15	1.417 (6)	C16—C21	1.3900
N7—C2	1.443 (3)	C17—C18	1.3900
N7—C15B	1.446 (12)	C18—C19	1.3900
N1—C2^{i}	1.330 (3)	C19—C20	1.3900
N1—C2	1.330 (3)	C20—C21	1.3900
C2—C3	1.383 (4)	O2B—C15B	1.215 (14)
C3—C4	1.379 (3)	C15B—C16B	1.497 (13)
C3—H3	0.9500	F6B—C17B	1.353 (9)
C4—C3^{i}	1.379 (3)	F7B—C18B	1.332 (10)
C4—H4	0.9500	F8B—C19B	1.294 (9)

C8—C9	1.498 (3)	F9B—C20B	1.294 (12)
C9—C10	1.384 (3)	F10B—C21B	1.321 (9)
C9—C14	1.389 (3)	C16B—C17B	1.3900
C10—C11	1.377 (4)	C16B—C21B	1.3900
C11—C12	1.377 (4)	C17B—C18B	1.3900
C12—C13	1.375 (4)	C18B—C19B	1.3900
C13—C14	1.374 (3)	C19B—C20B	1.3900
O2—C15	1.200 (6)	C20B—C21B	1.3900
C8—N7—C15	117.8 (3)	C17—C16—C21	120.0
C8—N7—C2	119.1 (2)	C17—C16—C15	120.9 (3)
C15—N7—C2	121.5 (3)	C21—C16—C15	118.5 (3)
C8—N7—C15B	138.0 (6)	F6—C17—C16	120.4 (2)
C2—N7—C15B	102.4 (6)	F6—C17—C18	119.6 (2)
C2^{i}—N1—C2	116.5 (3)	C16—C17—C18	120.0
N1—C2—C3	124.4 (3)	F7—C18—C19	119.3 (2)
N1—C2—N7	115.5 (2)	F7—C18—C17	120.7 (2)
C3—C2—N7	120.2 (2)	C19—C18—C17	120.0
C4—C3—C2	117.5 (3)	F8—C19—C18	119.6 (2)
C4—C3—H3	121.3	F8—C19—C20	120.4 (2)
C2—C3—H3	121.3	C18—C19—C20	120.0
C3—C4—C3^{i}	119.8 (4)	F9—C20—C21	121.1 (2)
C3—C4—H4	120.1	F9—C20—C19	118.9 (2)
C3^{i}—C4—H4	120.1	C21—C20—C19	120.0
O1—C8—N7	122.5 (2)	F10—C21—C20	119.2 (2)
O1—C8—C9	121.4 (2)	F10—C21—C16	120.7 (2)
N7—C8—C9	116.1 (2)	C20—C21—C16	120.0
C10—C9—C14	117.2 (2)	O2B—C15B—N7	128.4 (11)
C10—C9—C8	120.1 (2)	O2B—C15B—C16B	120.1 (11)
C14—C9—C8	122.4 (2)	N7—C15B—C16B	111.4 (9)
F1—C10—C11	118.5 (2)	C17B—C16B—C21B	120.0
F1—C10—C9	119.7 (2)	C17B—C16B—C15B	117.5 (7)
C11—C10—C9	121.8 (2)	C21B—C16B—C15B	122.0 (7)
F2—C11—C12	120.6 (2)	F6B—C17B—C18B	117.5 (6)
F2—C11—C10	120.0 (2)	F6B—C17B—C16B	122.5 (6)
C12—C11—C10	119.4 (2)	C18B—C17B—C16B	120.0
F3—C12—C13	120.1 (2)	F7B—C18B—C17B	119.8 (7)
F3—C12—C11	119.5 (2)	F7B—C18B—C19B	120.2 (7)
C13—C12—C11	120.4 (2)	C17B—C18B—C19B	120.0
F4—C13—C14	120.7 (2)	F8B—C19B—C20B	120.6 (8)
F4—C13—C12	119.9 (2)	F8B—C19B—C18B	119.4 (8)
C14—C13—C12	119.4 (2)	C20B—C19B—C18B	120.0
F5—C14—C13	118.0 (2)	F9B—C20B—C19B	120.8 (8)
F5—C14—C9	120.1 (2)	F9B—C20B—C21B	118.5 (8)
C13—C14—C9	121.8 (2)	C19B—C20B—C21B	120.0
O2—C15—N7	117.4 (5)	F10B—C21B—C20B	120.3 (7)
O2—C15—C16	122.8 (5)	F10B—C21B—C16B	118.6 (7)
N7—C15—C16	119.3 (4)	C20B—C21B—C16B	120.0
C2^{i}—N1—C2—C3	0.57 (19)	C21—C16—C17—C18	0.0

C2^{i}—N1—C2—N7	−178.4 (2)	C15—C16—C17—C18	−171.1 (3)
C8—N7—C2—N1	62.2 (3)	F6—C17—C18—F7	−0.9 (3)
C15—N7—C2—N1	−103.0 (4)	C16—C17—C18—F7	−179.2 (3)
C15B—N7—C2—N1	−111.0 (6)	F6—C17—C18—C19	178.4 (3)
C8—N7—C2—C3	−116.9 (3)	C16—C17—C18—C19	0.0
C15—N7—C2—C3	78.0 (4)	F7—C18—C19—F8	−2.2 (3)
C15B—N7—C2—C3	70.0 (6)	C17—C18—C19—F8	178.6 (3)
N1—C2—C3—C4	−1.1 (4)	F7—C18—C19—C20	179.3 (3)
N7—C2—C3—C4	177.85 (18)	C17—C18—C19—C20	0.0
C2—C3—C4—C3^{i}	0.50 (17)	F8—C19—C20—F9	2.3 (3)
C15—N7—C8—O1	15.2 (4)	C18—C19—C20—F9	−179.1 (3)
C2—N7—C8—O1	−150.5 (2)	F8—C19—C20—C21	−178.6 (3)
C15B—N7—C8—O1	19.5 (9)	C18—C19—C20—C21	0.0
C15—N7—C8—C9	−164.5 (3)	F9—C20—C21—F10	1.7 (3)
C2—N7—C8—C9	29.8 (3)	C19—C20—C21—F10	−177.5 (3)
C15B—N7—C8—C9	−160.2 (8)	F9—C20—C21—C16	179.1 (3)
O1—C8—C9—C10	51.4 (3)	C19—C20—C21—C16	0.0
N7—C8—C9—C10	−128.9 (2)	C17—C16—C21—F10	177.4 (3)
O1—C8—C9—C14	−122.1 (3)	C15—C16—C21—F10	−11.2 (4)
N7—C8—C9—C14	57.6 (3)	C17—C16—C21—C20	0.0
C14—C9—C10—F1	−177.86 (19)	C15—C16—C21—C20	171.3 (3)
C8—C9—C10—F1	8.3 (3)	C8—N7—C15B—O2B	−155.9 (9)
C14—C9—C10—C11	1.3 (4)	C2—N7—C15B—O2B	15.1 (14)
C8—C9—C10—C11	−172.6 (2)	C8—N7—C15B—C16B	20.5 (14)
F1—C10—C11—F2	−2.1 (4)	C2—N7—C15B—C16B	−168.5 (7)
C9—C10—C11—F2	178.8 (2)	O2B—C15B—C16B—C17B	37.7 (14)
F1—C10—C11—C12	178.5 (2)	N7—C15B—C16B—C17B	−139.0 (8)
C9—C10—C11—C12	−0.6 (4)	O2B—C15B—C16B—C21B	−134.5 (10)
F2—C11—C12—F3	−0.6 (4)	N7—C15B—C16B—C21B	48.8 (11)
C10—C11—C12—F3	178.8 (3)	C21B—C16B—C17B—F6B	−177.6 (9)
F2—C11—C12—C13	179.6 (2)	C15B—C16B—C17B—F6B	10.0 (10)
C10—C11—C12—C13	−1.1 (4)	C21B—C16B—C17B—C18B	0.0
F3—C12—C13—F4	1.6 (4)	C15B—C16B—C17B—C18B	−172.3 (9)
C11—C12—C13—F4	−178.6 (2)	F6B—C17B—C18B—F7B	−1.2 (9)
F3—C12—C13—C14	−177.9 (2)	C16B—C17B—C18B—F7B	−179.0 (9)
C11—C12—C13—C14	2.0 (4)	F6B—C17B—C18B—C19B	177.8 (9)
F4—C13—C14—F5	−3.7 (3)	C16B—C17B—C18B—C19B	0.0
C12—C13—C14—F5	175.7 (2)	F7B—C18B—C19B—F8B	−2.6 (11)
F4—C13—C14—C9	179.26 (19)	C17B—C18B—C19B—F8B	178.4 (10)
C12—C13—C14—C9	−1.3 (4)	F7B—C18B—C19B—C20B	179.0 (9)
C10—C9—C14—F5	−177.3 (2)	C17B—C18B—C19B—C20B	0.0
C8—C9—C14—F5	−3.6 (3)	F8B—C19B—C20B—F9B	−7.9 (12)
C10—C9—C14—C13	−0.3 (3)	C18B—C19B—C20B—F9B	170.5 (12)
C8—C9—C14—C13	173.4 (2)	F8B—C19B—C20B—C21B	−178.4 (10)
C8—N7—C15—O2	−153.1 (5)	C18B—C19B—C20B—C21B	0.0
C2—N7—C15—O2	12.2 (7)	F9B—C20B—C21B—F10B	−3.3 (10)
C8—N7—C15—C16	34.5 (6)	C19B—C20B—C21B—F10B	167.5 (10)
C2—N7—C15—C16	−160.2 (3)	F9B—C20B—C21B—C16B	−170.7 (11)

O2—C15—C16—C17	73.5 (6)	C19B—C20B—C21B—C16B	0.0
N7—C15—C16—C17	−114.5 (4)	C17B—C16B—C21B—F10B	−167.7 (10)
O2—C15—C16—C21	−97.7 (6)	C15B—C16B—C21B—F10B	4.3 (11)
N7—C15—C16—C21	74.2 (5)	C17B—C16B—C21B—C20B	0.0
C21—C16—C17—F6	−178.4 (3)	C15B—C16B—C21B—C20B	172.0 (9)
C15—C16—C17—F6	10.5 (4)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···N1^{ii}	0.95	2.67	3.621 (5)	180
C3—H3···O1^{ii}	0.95	2.52	3.318 (3)	141

Symmetry codes: (ii) *x*, *y*−1, *z*.

Fig. 1

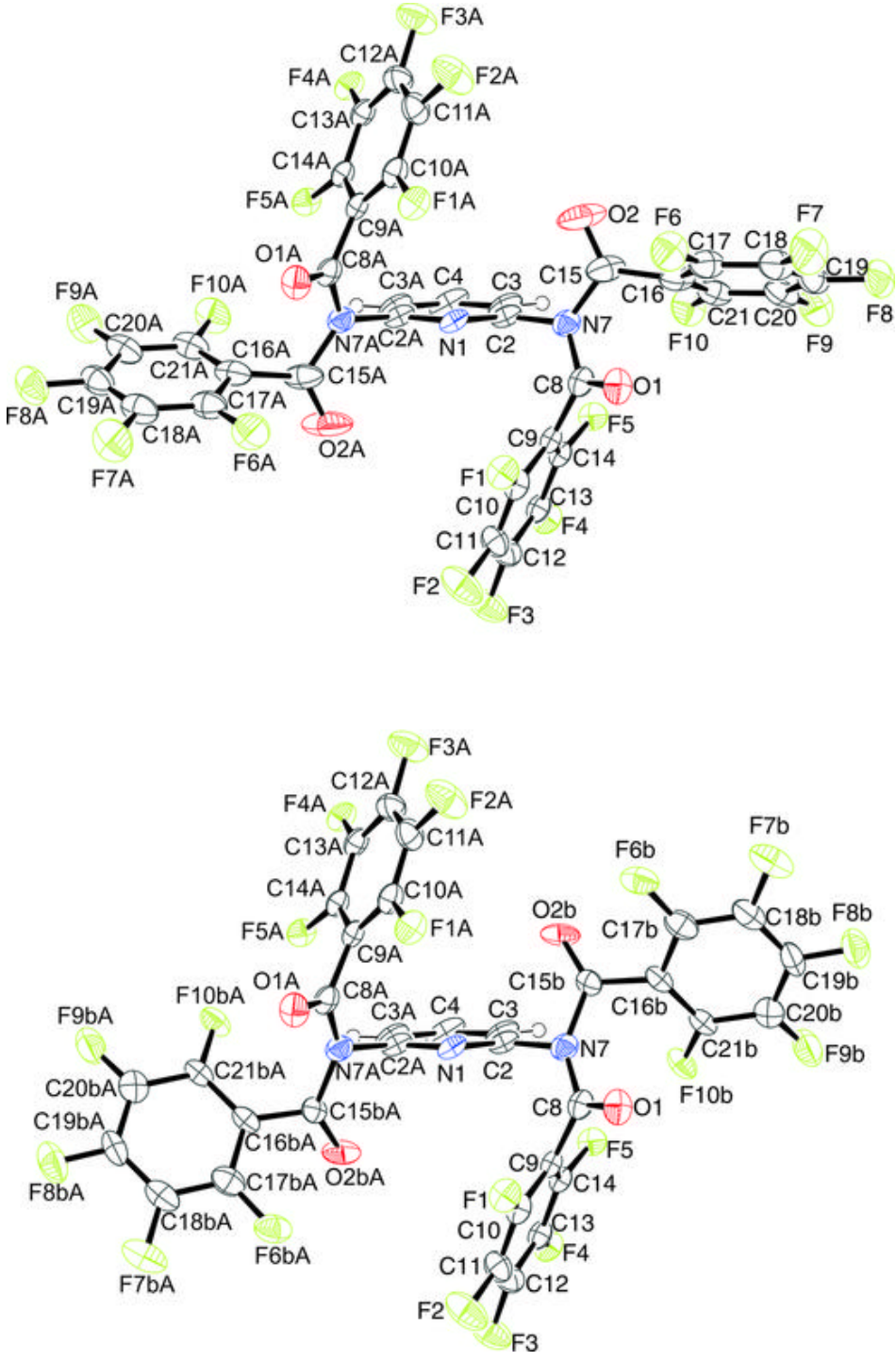

F3A
C12A
F4A
F2A
C13A
C11A
C14A
C10A
F5A
F1A
C9A
O1A
C8A
C3A
C4
C3
C15
O2
F6
C17
C18
F7
C19
F8
C16
C21
C20
F10
F9
N7
C2A
N1
C2
F9A
F10A
C20A
C16A
C21A
C15A
C8
O1
F8A
C19A
C17A
C18A
O2A
F5
C9
C14
F7A
F6A
F1
C10
C13
F4
C11
C12
F2
F3
F3A
C12A
F4A
F2A
C13A
C11A
F7b
F6b
C14A
C10A
O2b
C17b
C18b
F8b
F5A
F1A
C9A
C15b
C19b
O1A
C8A
F10bA
C16b
C20b
C3A
C4
C3
F9bA
C21b
F9b
N7A
C2A
N1
C2
C21bA
C20bA
C15bA
C8
O1
F10b
C19bA
C16bA
C9
F5
C17bA
O2bA
C14
F8bA
F1
C18bA
C10
C13
F6bA
F4
C11
C12
F7bA
F2
F3

Fig. 2