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Syntheses, crystal structures and Hirshfeld surface analyses of (3a*R*,4*S*,7*R*,7a*S*)-2-(perfluoropyridin-4-yl)-3a,4,7,7a-tetrahydro-4,7-methanoisoindole-1,3-dione and (3a*R*,4*S*,7*R*,7a*S*)-2-[(perfluoropyridin-4-yl)oxy]-3a,4,7,7a-tetrahydro-4,7-methanoisoindole-1,3-dione

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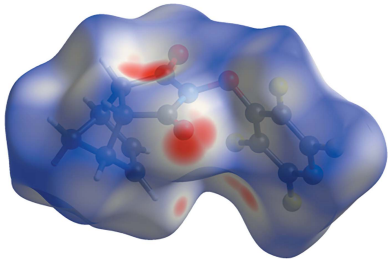
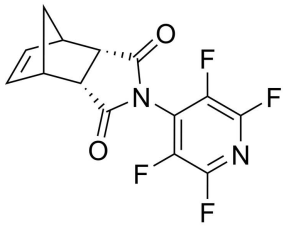
The syntheses and crystal structures of the title compounds, $C_{14}H_8F_4N_2O_2$ and $C_{14}H_8F_4N_2O_3$, are reported. In each crystal, the packing is driven by C—H···F intertactions, along with a variety of C—H···O, C—O···π, and C—F···π contacts. Hirshfeld surface analysis was conducted to aid in the visualization of these various influences on the packing: they showed that the largest contributions to the surface contacts arise from H···F/F···H interactions, followed by H···H and O···H/H···O.

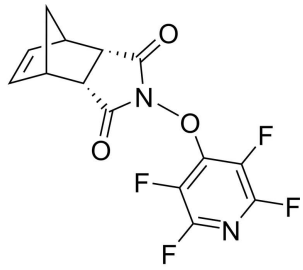


1. Chemical context

Polynorbornenes (PNBs), derived from ring-opening metathesis polymerization reactions, are numerous, owing to their relative ease of synthesis, tolerance of diverse functional groups and high-molecular weights with good processability (Isono *et al.*, 2018). The use of dicarboxyimide-substituted norbornenes allows synthetic control of the substituents on the norbornene ring system, and this feature has been exploited for polymer light-emitting diodes (Zeng *et al.*, 2018) and gas-separation membranes (Yu *et al.*, 2016). With its predictable substitution chemistry (Baker & Muir, 2010; Chambers *et al.*, 1988), perfluoropyridine was added to two dicarboxyimide-norbornene systems, and the resulting crystal structures are herein reported.



I



II

2. Structural commentary

Compound **I** crystallizes in the triclinic space group $P\overline{1}$ with two molecules, *A* and *B*, per asymmetric unit, and compound **II** in the monoclinic space group $P2_1/n$ with one molecule per

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Table 1
Contact geometry (Å, °) for **I**.

*Cg*1 and *Cg*2 are the centroids of the N1/C1–C5 and N3/C15–C19 rings, respectively.

Y—*X*···*A*	*Y*—*X*	*X*···*A*	*Y*···*A*	*Y*—*X*···*A*
C7—H7···O1^{i}	1.00	2.58	3.347 (2)	133
C9—H9···F4^{ii}	0.95	2.49	3.348(20	150
C12—H12···F2^{iii}	1.00	2.30	3.1903 (18)	147
C21—H21···F7^{iii}	1.00	2.55	3.3855 (17)	141
C21—H21.·O3^{iv}	1.00	2.51	3.2764 (18)	133
C26—H26···O4^{v}	1.00	2.51	3.3299 (18)	139
C6—O1···*Cg*1^{i}	1.2075 (17)	3.0462 (14)	4.022 (16)	135.86 (10)
C13—O2···*Cg*2^{vi}	1.2028 (16)	3.1839 (14)	4.0238 (17)	126.98 (9)
C27—O4···*Cg*2^{ii}	1.2102 (16)	3.2978 (14)	4.0644 (16)	121.58 (9)

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

asymmetric unit (Fig. 1). The synthesis of both compounds is conducted using *endo* starting materials, and the same configuration is observed in the resulting crystal structures. In **I**, steric interactions between the *ortho*-fluorine atoms and the carbonyl oxygen atoms prevents free rotation about the

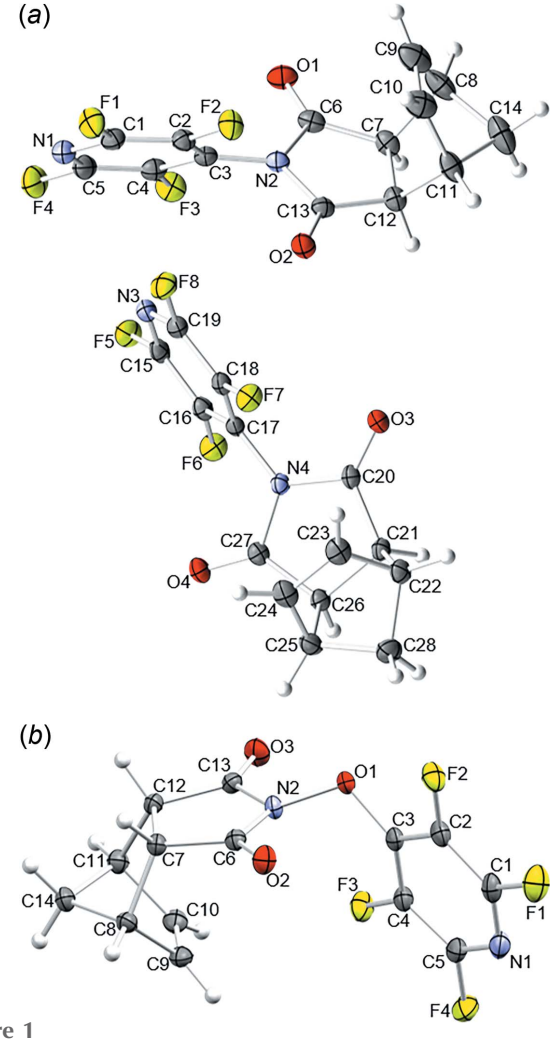

Figure 1
The molecular structures of (*a*) **I** and (*b*) **II**. Displacement ellipsoids are shown at the 50% probability level.

Table 2
Contact geometry (Å, °) for **II**.

*Cg*1 is the centroid of the N1/C1–C5 ring.

Y—*X*···*A*	*Y*—*X*	*X*···*A*	*Y*···*A*	*Y*—*X*···*A*
C7—H7···O2^{i}	1.00	2.58	3.347 (2)	133
C9—H9···O3^{ii}	0.95	2.49	3.348(20	150
C14—H14*B*···F4^{iii}	1.00	2.30	3.1903 (18)	147
C5—F4···*Cg*1^{iv}	1.3234 (13)	3.1543 (18)	3.892 (2)	114.36 (7)
C13—O3···*Cg*1^{v}	1.2060 (14)	3.3224 (19)	4.231 (2)	132.8 (8)

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

nitrogen–*ipso*-carbon bond (C3—N2 and C17—N4 in the crystal): this is evidenced by separate ^{19}F NMR peaks in solution for the *ortho*-F atoms (F2/F7 and F3/F6 in the crystal). In molecule *A*, the N1/C1–C5 plane is rotated by 58.05 (5)° relative to the N2/C6/C7/C12/C13 plane and the corresponding dihedral angle for molecule *B* is 61.65 (7)°. The addition of an oxygen atom between N2 and C3 in **II** alleviates this steric restriction and only one ^{19}F NMR peak in solution is observed for the *ortho*-F atoms; even so, the dihedral angle between the N1/C1–C5 and N2/C6/C7/C12/C13 planes in the crystal of **II** of 84.01 (5)° is larger than those found in **I**.

3. Supramolecular features and Hirshfeld surface analysis

The main directional interactions in the crystal structures of **I** and **II** are of the type C—H···O, C—H···F, C—O···π, and C—F···π (Tables 1 and 2). In both compounds, weak hydrogen-bonding interactions are observed for the hydrogen atom(s) α to the carbonyl groups (C7—H6···O1^{i}, C12—H12···F2^{iii}, C21—H21···F7^{iii}, C21—H21···O3^{iv} and C26—H26···O4^{v} in **I**; C7—H7···O2^{i} in **II**) and the olefinic hydrogen atoms (C9—H9···F4^{ii} in **I**; C9—H9···O3^{ii} in **II**). A weak interaction is also observed for a bridge hydrogen atom in **II**, C14—H14*B*···F4^{iii}. The packing is further aided by π-interactions with the pyridine ring (C6—O1···*Cg*^{i}, C13—O2···*Cg*^{vi} and C27—O4···*Cg*^{ii} in **I**; C5—F4···*Cg*^{iv} and C13—O3···*Cg*^{v} in **II**).

Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) was used to investigate the presence of hydrogen bonds and other intermolecular interactions in the crystal structures. The analyses and associated two-dimensional fingerprint plots (Fig. 3) (Spackman & McKinnon, 2002) were generated with *CrystalExplorer17.5* (Turner *et al.*, 2017) using a standard surface resolution with the three-dimensional d_{norm} surfaces plotted over a fixed color scale of −0.02500 (red) to 1.3800 (blue) a.u. The pale-red spots symbolize short contacts and negative d_{norm} values on the corresponding surface plots shown in Fig. 2, associated with their relative contributions to the Hirshfeld surface.

The largest contribution to the overall crystal packing in both compounds is from F···H/H···F interactions (36.5% in **I**; 39.2% in **II**; Table 3). The F···H/H···F contacts appear as a pair of characteristic tips in the fingerprint plots at 0.95 Å <

Table 3
Percentage contribution of inter-atomic contacts to the Hirshfeld surfaces for **I** and **II**.

Contact	Percentage contribution to **I**	Percentage contribution to **II**
F···H/H···F	36.5	39.2
H···H	20.2	14.1
O···H/H···O	13.0	14.0
O···C/C···O	6.2	6.2
N···H/H···N	5.3	0.1
F···F	5.1	2.1
F···C/C···F	3.6	4.9
F···N/N···F	2.4	4.4
O···N/N···O	0.6	4.1
C···H/H···C	3.8	3.2
F···O/O···F	2.4	2.7
O···O	1.2	2.7
N···C/C···N	0.0	2.4

($d_i + d_e$) < 1.25 Å in **I** and 1.10 Å < ($d_i + d_e$) < 1.35 Å in **II**. H···H contacts make the second largest contribution (20.2% in **I** and 14.1% in **II**), shown in the middle region 1.10 Å < ($d_i + d_e$) < 1.18 Å in **I** and **II**. The third largest contribution is from O···H/H···O contacts. In **I**, the corresponding spike is partially overlapped with the spike representing F···H/H···F contacts, appearing at 1.05 Å < ($d_i + d_e$) < 1.40 Å. The O···H/H···O spike is clearly visible in the fingerprint plot of **II**, shown in the region of 1.10 Å < ($d_i + d_e$) < 1.40 Å.

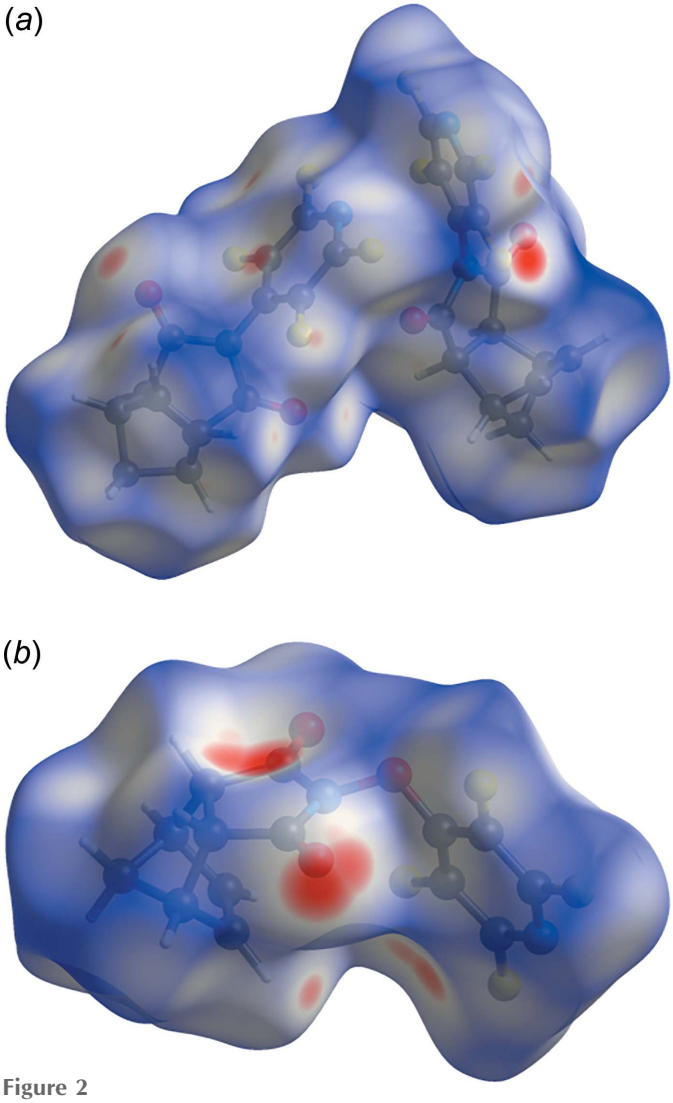

Figure 2
Hirshfeld surfaces of (*a*) **I** and (*b*) **II** mapped with d_{norm}.

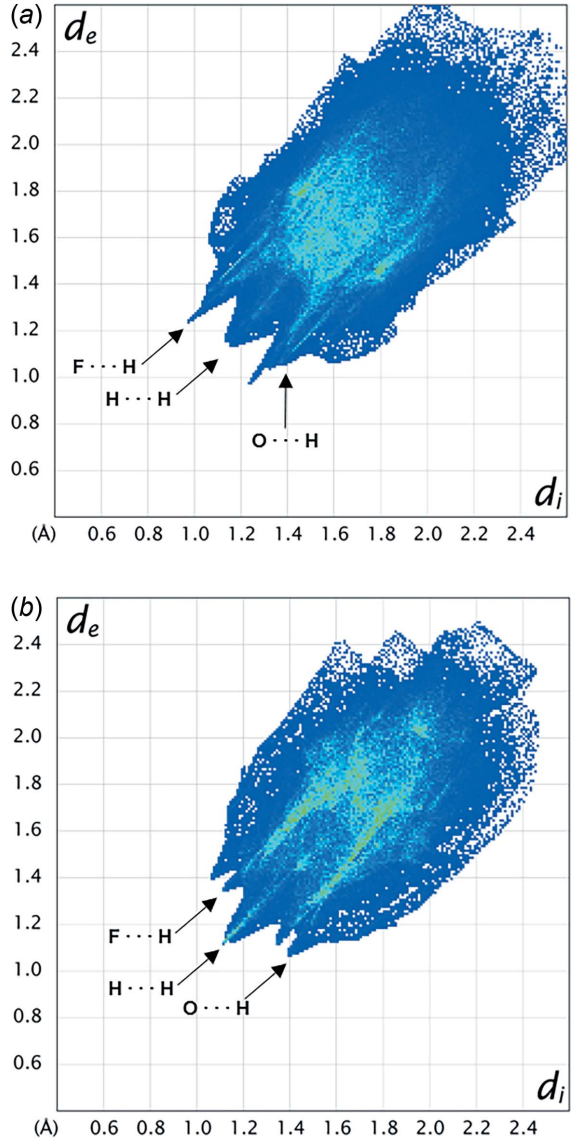

Figure 3
The overall two-dimensional fingerprint plots for (*a*) **I** and (*b*) **II**.

4. Database survey

A search of the November 2018 release of the Cambridge Structure Database (Groom *et al.*, 2016), with updates through May 2019, was performed using the program *ConQuest* (Bruno *et al.*, 2002). The search was limited to organic structures with $R \leq 0.1$. A search for tetrahydro-1*H*-4,7-methanoisoindole-1,3(2*H*)-dione-based compounds with an aromatic substituent on the nitrogen atom yielded 58 results. The dihedral angle between the aromatic ring plane and the succinimide plane is bimodally distributed between 43 and 90°, with peaks near 60 and 75°.

Table 4
Experimental details.

	I	II
Crystal data		
Chemical formula	$C_{14}H_8F_4N_2O_2$	$C_{14}H_8F_4N_2O_3$
M_r	312.22	328.22
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/n$
Temperature (K)	100	100
a, b, c (Å)	7.0347 (15), 10.256 (2), 18.129 (4)	12.285 (6), 5.945 (3), 17.888 (8)
α, β, γ (°)	78.844 (10), 80.165 (10), 87.242 (10)	90, 93.44 (3), 90
V (Å^3)	1264.2 (5)	1304.1 (10)
Z	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.15	0.16
Crystal size (mm)	0.26 × 0.23 × 0.20	0.55 × 0.40 × 0.36
Data collection		
Diffractometer	Bruker SMART APEX CCD	Bruker SMART APEX CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2017)	Multi-scan (*SADABS*; Bruker, 2017)
T_{min}, T_{max}	0.89, 0.97	0.80, 0.95
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	72226, 7674, 6373	18507, 3823, 3443
R_{int}	0.034	0.025
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.714	0.704
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.126, 1.02	0.038, 0.105, 1.04
No. of reflections	7674	3823
No. of parameters	397	208
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.77, −0.41	0.47, −0.25

Computer programs: *APEX3* and *SAINT* (Bruker, 2017), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).

5. Synthesis and crystallization

Synthesis of (I): pentafluoropyridine (2.68 ml, 24.5 mmol), (3a*R*,4*S*,7*R*,7a*S*)-3a,4,7,7a-tetrahydro-1*H*-4,7-methanoisoindole-1,3(2*H*)-dione (4.0 g, 24.5 mmol), and triethylamine (8.56 ml, 61.2 mmol) were combined in DMF (150 ml). The resulting solution was stirred at room temperature for 24 h. Diethyl ether (150 ml) and saturated aqueous ammonium chloride (100 ml) were added and the biphasic solution stirred vigorously for 2 h. The organic layer was separated and the remaining aqueous portion extracted with diethyl ether (2 × 150 ml). The combined organic fractions were washed with water (2 × 1 l) and brine (2 × 300 ml), dried over $MgSO_4$, and the solvent removed *via* rotary evaporation. The resulting off-white solid was dissolved in refluxing EtOH (20 ml) and cooled to 278 K for 12 h. Vacuum filtration, washing with cold EtOH (20 ml), and vacuum drying afforded the target compound as a white, crystalline solid (6.18 g, 81%). ^{1}H NMR (500 MHz, $CDCl_3$): 6.28 (*s*, 2H), 3.58 (*s*, 2H), 3.54 (*s*, 2H), 1.74 (*dd*, 2H, J = 96, 9.0 Hz).^{19}F NMR (471 MHz, $CDCl_3$): −90.5 (2F), −141.7 (1F), −143.1 (1F).

Synthesis of (II): to a stirred solution of potassium carbonate (1 *M*, 140 ml), (3a*R*,4*S*,7*R*,7a*S*)-2-hydroxy-3a,4,7,7a-tetrahydro-1*H*-4,7-methanoisoindole-1,3(2*H*)-dione (10.0 g, 55.9 mmol), pentafluoropyridine (6.1 ml, 56 mmol), and 140 ml of DMF were added. The resulting solution was stirred at room temperature for 24 h. Diethyl ether (150 ml) and saturated aqueous ammonium chloride (100 ml) were added and the biphasic solution stirred vigorously for 2 h. The organic layer was separated and the remaining aqueous portion extracted with diethyl ether (2 × 150 ml). The combined organic fractions were washed with water (2 × 1 l) and brine (2 × 300 ml), dried over $MgSO_4$, and the solvent removed *via* rotary evaporation. The resulting off-white solid was dissolved in refluxing EtOH (50 ml) and cooled to 278 K for 12 h. Vacuum filtration, washing with cold EtOH (20 ml) and vacuum drying afforded the target compound as a white, crystalline solid (12.04 g, 66%). ^{1}H NMR (500 MHz, $CDCl_3$): 6.23 (*s*, 2H), 3.50 (*s*, 2H), 3.34 (*s*, 2H), 1.68 (*dd*, 2H, J = 142, 9.0 Hz).^{19}F NMR (471 MHz, $CDCl_3$): −87.4 (2F), −156.3 (2F).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. H atoms were positioned geometrically and refined using a riding model with C—H = 0.95–1.0Å and U_{iso}(H) = 1.2U_{eq}(C).

Funding information

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supporting information

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Syntheses, crystal structures and Hirshfeld surface analyses of (3a*R*,4*S*,7*R*,7a*S*)-2-(perfluoropyridin-4-yl)-3a,4,7,7a-tetrahydro-4,7-methanoisoindole-1,3-dione and (3a*R*,4*S*,7*R*,7a*S*)-2-[(perfluoropyridin-4-yl)oxy]-3a,4,7,7a-tetrahydro-4,7-methanoisoindole-1,3-dione

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Computing details

For both structures, data collection: *APEX3* (Bruker, 2017); cell refinement: *SAINT* (Bruker, 2017); data reduction: *SAINT* (Bruker, 2017); program(s) used to solve structure: SHELXT (Sheldrick, 2015*a*); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

(I)

Crystal data

$C_{14}H_8F_4N_2O_2$
M_r = 312.22
Triclinic, $P\overline{1}$
a = 7.0347 (15) Å
b = 10.256 (2) Å
c = 18.129 (4) Å
α = 78.844 (10)°
β = 80.165 (10)°
γ = 87.242 (10)°
V = 1264.2 (5) Å^3
Z = 4
$F(000)$ = 632
D_x = 1.640 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9823 reflections
θ = 2.5–30.7°
μ = 0.15 mm^{-1}
T = 100 K
Rectangular prism, clear colourless
0.26 × 0.23 × 0.20 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine focus sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
ω Scans scans
Absorption correction: multi-scan (SADABS; Bruker, 2017)
T_{min} = 0.89, T_{max} = 0.97
72226 measured reflections
7674 independent reflections
6373 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 30.5°, θ_{min} = 2.0°
h = −10→10
k = −14→14
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.126$
$S = 1.02$
7674 reflections
397 parameters
0 restraints

Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0625P)^2 + 0.7892P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.77$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
F1	−0.32163 (13)	0.66535 (9)	0.80280 (6)	0.0299 (2)
F2	−0.04925 (12)	0.46899 (8)	0.81310 (5)	0.02556 (18)
F3	0.29897 (13)	0.74112 (9)	0.92385 (5)	0.02742 (19)
F4	0.01062 (15)	0.92480 (9)	0.90912 (6)	0.0340 (2)
F5	0.40224 (14)	0.92684 (9)	0.76901 (5)	0.02754 (19)
F6	0.72380 (12)	0.90629 (9)	0.66496 (5)	0.02446 (18)
F7	0.34503 (12)	0.67973 (9)	0.53929 (5)	0.02272 (17)
F8	0.04320 (12)	0.70789 (9)	0.64982 (5)	0.02795 (19)
O1	0.27798 (16)	0.45448 (11)	1.00080 (6)	0.0269 (2)
O2	0.34535 (16)	0.50335 (10)	0.74150 (6)	0.0233 (2)
O3	0.71312 (14)	0.54609 (9)	0.56707 (6)	0.02119 (19)
O4	0.75913 (15)	0.99783 (9)	0.49291 (6)	0.02099 (19)
N1	−0.15423 (18)	0.79402 (12)	0.85635 (7)	0.0235 (2)
N2	0.28410 (16)	0.50134 (11)	0.87085 (6)	0.0178 (2)
N3	0.22520 (17)	0.81750 (11)	0.70839 (7)	0.0208 (2)
N4	0.70733 (15)	0.77544 (10)	0.54298 (6)	0.0154 (2)
C1	−0.16515 (19)	0.68212 (14)	0.83262 (8)	0.0214 (3)
C2	−0.02687 (19)	0.58195 (13)	0.83713 (8)	0.0192 (2)
C3	0.13494 (18)	0.59829 (13)	0.86851 (7)	0.0172 (2)
C4	0.1460 (2)	0.71642 (14)	0.89443 (7)	0.0204 (2)
C5	−0.0016 (2)	0.80966 (14)	0.88622 (8)	0.0237 (3)
C6	0.3481 (2)	0.43565 (14)	0.93807 (7)	0.0201 (2)
C7	0.5095 (2)	0.34170 (14)	0.91564 (8)	0.0214 (3)
H7	0.632591	0.364803	0.930317	0.026*
C8	0.4631 (3)	0.19043 (16)	0.94528 (9)	0.0332 (3)
H8	0.479722	0.15207	0.998913	0.04*
C9	0.2739 (3)	0.16766 (17)	0.92242 (10)	0.0355 (4)
H9	0.157787	0.146875	0.9574	0.043*
C10	0.2883 (3)	0.17988 (17)	0.84872 (10)	0.0335 (3)

H10	0.188245	0.170573	0.821019	0.04*
C11	0.4917 (2)	0.21108 (14)	0.81733 (9)	0.0269 (3)
H11	0.535408	0.194148	0.764515	0.032*
C12	0.52885 (19)	0.35524 (13)	0.82814 (8)	0.0191 (2)
H12	0.661556	0.384198	0.802588	0.023*
C13	0.38067 (19)	0.45892 (13)	0.80420 (7)	0.0176 (2)
C14	0.6008 (3)	0.13049 (17)	0.87797 (12)	0.0400 (4)
H14A	0.737652	0.155649	0.871307	0.048*
H14B	0.5897	0.033186	0.88305	0.048*
C15	0.3933 (2)	0.86501 (13)	0.71103 (7)	0.0201 (2)
C16	0.55752 (19)	0.85513 (13)	0.65845 (7)	0.0181 (2)
C17	0.54593 (18)	0.79044 (12)	0.59868 (7)	0.0157 (2)
C18	0.36863 (19)	0.73991 (12)	0.59592 (7)	0.0169 (2)
C19	0.21476 (19)	0.75621 (13)	0.65218 (8)	0.0195 (2)
C20	0.77807 (18)	0.64904 (12)	0.52953 (7)	0.0164 (2)
C21	0.93999 (18)	0.67181 (12)	0.46283 (7)	0.0167 (2)
H21	1.0639	0.631685	0.477318	0.02*
C22	0.8943 (2)	0.62467 (13)	0.39011 (7)	0.0197 (2)
H22	0.919783	0.528529	0.388159	0.024*
C23	0.6930 (2)	0.67662 (14)	0.38012 (8)	0.0225 (3)
H23	0.580506	0.624899	0.387553	0.027*
C24	0.7034 (2)	0.80880 (14)	0.35888 (8)	0.0235 (3)
H24	0.598626	0.867847	0.348918	0.028*
C25	0.9114 (2)	0.84821 (13)	0.35344 (8)	0.0223 (3)
H25	0.950085	0.936047	0.320653	0.027*
C26	0.95440 (19)	0.82471 (12)	0.43732 (7)	0.0176 (2)
H26	1.085803	0.856148	0.438844	0.021*
C27	0.80172 (18)	0.88246 (12)	0.49164 (7)	0.0164 (2)
C28	1.0183 (2)	0.72422 (15)	0.32846 (8)	0.0263 (3)
H28A	1.004935	0.715936	0.276088	0.032*
H28B	1.156118	0.719809	0.334083	0.032*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0193 (4)	0.0288 (5)	0.0432 (5)	−0.0001 (3)	−0.0118 (4)	−0.0050 (4)
F2	0.0233 (4)	0.0211 (4)	0.0368 (5)	−0.0025 (3)	−0.0098 (3)	−0.0115 (3)
F3	0.0301 (5)	0.0295 (4)	0.0273 (4)	−0.0059 (4)	−0.0112 (4)	−0.0099 (4)
F4	0.0406 (5)	0.0235 (4)	0.0406 (5)	−0.0017 (4)	−0.0029 (4)	−0.0158 (4)
F5	0.0396 (5)	0.0255 (4)	0.0190 (4)	−0.0009 (4)	−0.0005 (3)	−0.0116 (3)
F6	0.0252 (4)	0.0264 (4)	0.0253 (4)	−0.0041 (3)	−0.0062 (3)	−0.0109 (3)
F7	0.0219 (4)	0.0269 (4)	0.0230 (4)	−0.0013 (3)	−0.0059 (3)	−0.0114 (3)
F8	0.0181 (4)	0.0307 (5)	0.0352 (5)	−0.0019 (3)	−0.0016 (3)	−0.0086 (4)
O1	0.0308 (5)	0.0333 (6)	0.0160 (4)	−0.0034 (4)	−0.0036 (4)	−0.0031 (4)
O2	0.0312 (5)	0.0217 (5)	0.0169 (4)	0.0030 (4)	−0.0050 (4)	−0.0036 (4)
O3	0.0229 (5)	0.0152 (4)	0.0238 (5)	0.0005 (3)	−0.0014 (4)	−0.0020 (4)
O4	0.0255 (5)	0.0145 (4)	0.0242 (5)	0.0000 (3)	−0.0055 (4)	−0.0054 (4)
N1	0.0226 (6)	0.0203 (5)	0.0254 (6)	−0.0001 (4)	0.0005 (4)	−0.0031 (4)

N2	0.0188 (5)	0.0197 (5)	0.0148 (5)	−0.0010 (4)	−0.0036 (4)	−0.0025 (4)
N3	0.0246 (6)	0.0153 (5)	0.0201 (5)	0.0027 (4)	0.0003 (4)	−0.0017 (4)
N4	0.0174 (5)	0.0132 (4)	0.0161 (5)	0.0007 (4)	−0.0024 (4)	−0.0043 (4)
C1	0.0169 (6)	0.0224 (6)	0.0241 (6)	−0.0026 (5)	−0.0020 (5)	−0.0027 (5)
C2	0.0194 (6)	0.0184 (6)	0.0202 (6)	−0.0034 (4)	−0.0024 (5)	−0.0049 (5)
C3	0.0173 (6)	0.0189 (6)	0.0147 (5)	−0.0018 (4)	−0.0013 (4)	−0.0021 (4)
C4	0.0220 (6)	0.0234 (6)	0.0166 (5)	−0.0042 (5)	−0.0025 (5)	−0.0053 (5)
C5	0.0276 (7)	0.0197 (6)	0.0236 (6)	−0.0025 (5)	0.0008 (5)	−0.0075 (5)
C6	0.0221 (6)	0.0218 (6)	0.0172 (5)	−0.0057 (5)	−0.0063 (5)	−0.0012 (5)
C7	0.0242 (6)	0.0210 (6)	0.0207 (6)	−0.0008 (5)	−0.0099 (5)	−0.0024 (5)
C8	0.0500 (10)	0.0231 (7)	0.0274 (7)	−0.0034 (6)	−0.0171 (7)	0.0037 (6)
C9	0.0504 (10)	0.0253 (7)	0.0336 (8)	−0.0101 (7)	−0.0105 (7)	−0.0069 (6)
C10	0.0407 (9)	0.0289 (8)	0.0327 (8)	−0.0103 (7)	−0.0069 (7)	−0.0072 (6)
C11	0.0374 (8)	0.0175 (6)	0.0303 (7)	0.0006 (5)	−0.0164 (6)	−0.0064 (5)
C12	0.0197 (6)	0.0179 (6)	0.0213 (6)	−0.0004 (4)	−0.0067 (5)	−0.0048 (5)
C13	0.0194 (6)	0.0167 (5)	0.0172 (5)	−0.0037 (4)	−0.0024 (4)	−0.0039 (4)
C14	0.0480 (10)	0.0213 (7)	0.0546 (11)	0.0049 (7)	−0.0255 (9)	−0.0033 (7)
C15	0.0297 (7)	0.0146 (5)	0.0160 (5)	0.0019 (5)	−0.0026 (5)	−0.0047 (4)
C16	0.0220 (6)	0.0155 (5)	0.0181 (5)	0.0002 (4)	−0.0055 (5)	−0.0042 (4)
C17	0.0188 (6)	0.0128 (5)	0.0151 (5)	0.0021 (4)	−0.0029 (4)	−0.0025 (4)
C18	0.0201 (6)	0.0147 (5)	0.0168 (5)	0.0020 (4)	−0.0047 (4)	−0.0041 (4)
C19	0.0191 (6)	0.0163 (5)	0.0220 (6)	0.0010 (4)	−0.0027 (5)	−0.0018 (5)
C20	0.0176 (5)	0.0149 (5)	0.0184 (5)	0.0019 (4)	−0.0059 (4)	−0.0051 (4)
C21	0.0168 (5)	0.0147 (5)	0.0195 (5)	0.0010 (4)	−0.0029 (4)	−0.0059 (4)
C22	0.0254 (6)	0.0161 (5)	0.0187 (6)	0.0002 (5)	−0.0029 (5)	−0.0065 (5)
C23	0.0275 (7)	0.0226 (6)	0.0204 (6)	−0.0013 (5)	−0.0081 (5)	−0.0079 (5)
C24	0.0314 (7)	0.0227 (6)	0.0187 (6)	0.0019 (5)	−0.0098 (5)	−0.0052 (5)
C25	0.0305 (7)	0.0170 (6)	0.0176 (6)	−0.0016 (5)	−0.0002 (5)	−0.0021 (5)
C26	0.0188 (6)	0.0148 (5)	0.0195 (6)	−0.0010 (4)	−0.0018 (4)	−0.0050 (4)
C27	0.0184 (5)	0.0158 (5)	0.0167 (5)	−0.0011 (4)	−0.0056 (4)	−0.0047 (4)
C28	0.0332 (8)	0.0245 (7)	0.0199 (6)	−0.0012 (6)	0.0023 (5)	−0.0069 (5)

Geometric parameters (Å, º)

F1—C1	1.3384 (16)	C9—C10	1.304 (2)
F2—C2	1.3390 (15)	C9—H9	0.95
F3—C4	1.3343 (16)	C10—C11	1.473 (2)
F4—C5	1.3364 (16)	C10—H10	0.95
F5—C15	1.3398 (15)	C11—C14	1.535 (2)
F6—C16	1.3370 (15)	C11—C12	1.5683 (19)
F7—C18	1.3325 (14)	C11—H11	1.0
F8—C19	1.3370 (16)	C12—C13	1.5084 (18)
O1—C6	1.2075 (17)	C12—H12	1.0
O2—C13	1.2028 (16)	C14—H14A	0.99
O3—C20	1.2038 (16)	C14—H14B	0.99
O4—C27	1.2102 (16)	C15—C16	1.3794 (19)
N1—C1	1.3111 (18)	C16—C17	1.3909 (17)
N1—C5	1.312 (2)	C17—C18	1.3858 (18)

N2—C6	1.4077 (17)	C18—C19	1.3825 (18)
N2—C3	1.4107 (17)	C20—C21	1.5033 (18)
N2—C13	1.4161 (17)	C21—C26	1.5499 (18)
N3—C19	1.3103 (18)	C21—C22	1.5753 (18)
N3—C15	1.3125 (19)	C21—H21	1.0
N4—C27	1.4056 (17)	C22—C23	1.516 (2)
N4—C17	1.4086 (16)	C22—C28	1.537 (2)
N4—C20	1.4165 (16)	C22—H22	1.0
C1—C2	1.3809 (19)	C23—C24	1.338 (2)
C2—C3	1.3871 (18)	C23—H23	0.95
C3—C4	1.3917 (18)	C24—C25	1.519 (2)
C4—C5	1.385 (2)	C24—H24	0.95
C6—C7	1.507 (2)	C25—C28	1.550 (2)
C7—C12	1.5482 (19)	C25—C26	1.5717 (19)
C7—C8	1.571 (2)	C25—H25	1.0
C7—H7	1.0	C26—C27	1.5091 (18)
C8—C9	1.501 (3)	C26—H26	1.0
C8—C14	1.623 (3)	C28—H28A	0.99
C8—H8	1.0	C28—H28B	0.99

C1—N1—C5	116.94 (12)	C8—C14—H14A	113.4
C6—N2—C3	124.33 (11)	C11—C14—H14B	113.4
C6—N2—C13	113.71 (11)	C8—C14—H14B	113.4
C3—N2—C13	121.94 (11)	H14A—C14—H14B	110.7
C19—N3—C15	117.26 (12)	N3—C15—F5	116.65 (12)
C27—N4—C17	123.69 (10)	N3—C15—C16	124.23 (12)
C27—N4—C20	113.87 (11)	F5—C15—C16	119.11 (12)
C17—N4—C20	122.21 (11)	F6—C16—C15	120.74 (11)
N1—C1—F1	117.06 (12)	F6—C16—C17	120.86 (12)
N1—C1—C2	124.07 (13)	C15—C16—C17	118.40 (12)
F1—C1—C2	118.87 (12)	C18—C17—C16	117.45 (12)
F2—C2—C1	120.87 (12)	C18—C17—N4	120.70 (11)
F2—C2—C3	119.96 (12)	C16—C17—N4	121.85 (12)
C1—C2—C3	119.15 (12)	F7—C18—C19	120.46 (12)
C2—C3—C4	116.92 (12)	F7—C18—C17	120.92 (11)
C2—C3—N2	121.00 (11)	C19—C18—C17	118.60 (12)
C4—C3—N2	122.02 (12)	N3—C19—F8	117.01 (12)
F3—C4—C5	120.87 (12)	N3—C19—C18	124.05 (13)
F3—C4—C3	120.66 (12)	F8—C19—C18	118.94 (12)
C5—C4—C3	118.42 (12)	O3—C20—N4	123.24 (12)
N1—C5—F4	116.75 (13)	O3—C20—C21	129.39 (12)
N1—C5—C4	124.49 (13)	N4—C20—C21	107.37 (10)
F4—C5—C4	118.75 (13)	C20—C21—C26	105.67 (10)
O1—C6—N2	123.41 (13)	C20—C21—C22	113.60 (11)
O1—C6—C7	129.08 (13)	C26—C21—C22	103.31 (10)
N2—C6—C7	107.50 (11)	C20—C21—H21	111.3
C6—C7—C12	105.79 (11)	C26—C21—H21	111.3
C6—C7—C8	114.60 (13)	C22—C21—H21	111.3

C12—C7—C8	102.66 (11)	C23—C22—C28	100.97 (11)
C6—C7—H7	111.1	C23—C22—C21	106.32 (10)
C12—C7—H7	111.1	C28—C22—C21	98.84 (11)
C8—C7—H7	111.1	C23—C22—H22	116.1
C9—C8—C7	106.69 (13)	C28—C22—H22	116.1
C9—C8—C14	97.02 (13)	C21—C22—H22	116.1
C7—C8—C14	98.55 (13)	C24—C23—C22	107.28 (13)
C9—C8—H8	117.1	C24—C23—H23	126.4
C7—C8—H8	117.1	C22—C23—H23	126.4
C14—C8—H8	117.1	C23—C24—C25	108.23 (13)
C10—C9—C8	111.81 (17)	C23—C24—H24	125.9
C10—C9—H9	124.1	C25—C24—H24	125.9
C8—C9—H9	124.1	C24—C25—C28	100.25 (11)
C9—C10—C11	105.78 (16)	C24—C25—C26	106.61 (11)
C9—C10—H10	127.1	C28—C25—C26	98.57 (11)
C11—C10—H10	127.1	C24—C25—H25	116.3
C10—C11—C14	103.19 (15)	C28—C25—H25	116.3
C10—C11—C12	107.98 (13)	C26—C25—H25	116.3
C14—C11—C12	99.67 (12)	C27—C26—C21	105.73 (10)
C10—C11—H11	114.8	C27—C26—C25	113.65 (11)
C14—C11—H11	114.8	C21—C26—C25	102.89 (10)
C12—C11—H11	114.8	C27—C26—H26	111.4
C13—C12—C7	105.61 (11)	C21—C26—H26	111.4
C13—C12—C11	115.44 (11)	C25—C26—H26	111.4
C7—C12—C11	103.66 (11)	O4—C27—N4	123.64 (12)
C13—C12—H12	110.6	O4—C27—C26	128.99 (12)
C7—C12—H12	110.6	N4—C27—C26	107.35 (10)
C11—C12—H12	110.6	C22—C28—C25	94.29 (11)
O2—C13—N2	123.30 (12)	C22—C28—H28A	112.9
O2—C13—C12	129.34 (12)	C25—C28—H28A	112.9
N2—C13—C12	107.35 (11)	C22—C28—H28B	112.9
C11—C14—C8	91.50 (13)	C25—C28—H28B	112.9
C11—C14—H14A	113.4	H28A—C28—H28B	110.3

C5—N1—C1—F1	179.36 (12)	C19—N3—C15—F5	−179.67 (11)
C5—N1—C1—C2	−0.2 (2)	C19—N3—C15—C16	0.4 (2)
N1—C1—C2—F2	178.68 (13)	N3—C15—C16—F6	179.92 (12)
F1—C1—C2—F2	−0.9 (2)	F5—C15—C16—F6	0.03 (19)
N1—C1—C2—C3	0.0 (2)	N3—C15—C16—C17	−0.2 (2)
F1—C1—C2—C3	−179.51 (12)	F5—C15—C16—C17	179.89 (11)
F2—C2—C3—C4	−178.07 (11)	F6—C16—C17—C18	179.90 (11)
C1—C2—C3—C4	0.57 (19)	C15—C16—C17—C18	0.04 (18)
F2—C2—C3—N2	4.45 (19)	F6—C16—C17—N4	0.29 (19)
C1—C2—C3—N2	−176.90 (12)	C15—C16—C17—N4	−179.57 (11)
C6—N2—C3—C2	−122.28 (14)	C27—N4—C17—C18	115.77 (14)
C13—N2—C3—C2	56.10 (17)	C20—N4—C17—C18	−58.41 (16)
C6—N2—C3—C4	60.38 (18)	C27—N4—C17—C16	−64.63 (17)
C13—N2—C3—C4	−121.24 (14)	C20—N4—C17—C16	121.18 (13)

C2—C3—C4—F3	−178.53 (12)	C16—C17—C18—F7	178.24 (11)
N2—C3—C4—F3	−1.08 (19)	N4—C17—C18—F7	−2.15 (18)
C2—C3—C4—C5	−1.01 (19)	C16—C17—C18—C19	−0.10 (18)
N2—C3—C4—C5	176.44 (12)	N4—C17—C18—C19	179.52 (11)
C1—N1—C5—F4	178.87 (12)	C15—N3—C19—F8	179.93 (11)
C1—N1—C5—C4	−0.3 (2)	C15—N3—C19—C18	−0.5 (2)
F3—C4—C5—N1	178.43 (13)	F7—C18—C19—N3	−178.00 (12)
C3—C4—C5—N1	0.9 (2)	C17—C18—C19—N3	0.3 (2)
F3—C4—C5—F4	−0.7 (2)	F7—C18—C19—F8	1.56 (19)
C3—C4—C5—F4	−178.23 (12)	C17—C18—C19—F8	179.90 (11)
C3—N2—C6—O1	1.5 (2)	C27—N4—C20—O3	−178.97 (12)
C13—N2—C6—O1	−176.98 (13)	C17—N4—C20—O3	−4.26 (19)
C3—N2—C6—C7	−179.43 (11)	C27—N4—C20—C21	1.03 (14)
C13—N2—C6—C7	2.06 (15)	C17—N4—C20—C21	175.74 (11)
O1—C6—C7—C12	176.95 (14)	O3—C20—C21—C26	178.83 (13)
N2—C6—C7—C12	−2.02 (14)	N4—C20—C21—C26	−1.17 (13)
O1—C6—C7—C8	64.62 (19)	O3—C20—C21—C22	66.28 (17)
N2—C6—C7—C8	−114.36 (13)	N4—C20—C21—C22	−113.72 (11)
C6—C7—C8—C9	51.32 (17)	C20—C21—C22—C23	46.18 (14)
C12—C7—C8—C9	−62.86 (16)	C26—C21—C22—C23	−67.79 (13)
C6—C7—C8—C14	151.35 (12)	C20—C21—C22—C28	150.43 (11)
C12—C7—C8—C14	37.17 (14)	C26—C21—C22—C28	36.46 (12)
C7—C8—C9—C10	68.24 (19)	C28—C22—C23—C24	−33.15 (14)
C14—C8—C9—C10	−32.91 (18)	C21—C22—C23—C24	69.55 (14)
C8—C9—C10—C11	0.42 (19)	C22—C23—C24—C25	0.53 (15)
C9—C10—C11—C14	35.12 (18)	C23—C24—C25—C28	31.90 (14)
C9—C10—C11—C12	−69.79 (17)	C23—C24—C25—C26	−70.35 (14)
C6—C7—C12—C13	1.33 (14)	C20—C21—C26—C27	0.93 (13)
C8—C7—C12—C13	121.79 (12)	C22—C21—C26—C27	120.51 (11)
C6—C7—C12—C11	−120.46 (12)	C20—C21—C26—C25	−118.55 (11)
C8—C7—C12—C11	−0.01 (14)	C22—C21—C26—C25	1.04 (12)
C10—C11—C12—C13	−47.45 (16)	C24—C25—C26—C27	−48.17 (14)
C14—C11—C12—C13	−154.82 (14)	C28—C25—C26—C27	−151.64 (11)
C10—C11—C12—C7	67.53 (14)	C24—C25—C26—C21	65.64 (12)
C14—C11—C12—C7	−39.85 (15)	C28—C25—C26—C21	−37.82 (12)
C6—N2—C13—O2	179.80 (12)	C17—N4—C27—O4	3.78 (19)
C3—N2—C13—O2	1.3 (2)	C20—N4—C27—O4	178.40 (12)
C6—N2—C13—C12	−1.18 (15)	C17—N4—C27—C26	−175.04 (11)
C3—N2—C13—C12	−179.73 (11)	C20—N4—C27—C26	−0.41 (14)
C7—C12—C13—O2	178.76 (14)	C21—C26—C27—O4	−179.08 (13)
C11—C12—C13—O2	−67.39 (19)	C25—C26—C27—O4	−66.97 (17)
C7—C12—C13—N2	−0.18 (14)	C21—C26—C27—N4	−0.35 (13)
C11—C12—C13—N2	113.68 (13)	C25—C26—C27—N4	111.75 (12)
C10—C11—C14—C8	−50.81 (14)	C23—C22—C28—C25	49.19 (12)
C12—C11—C14—C8	60.38 (14)	C21—C22—C28—C25	−59.46 (12)
C9—C8—C14—C11	47.84 (14)	C24—C25—C28—C22	−48.49 (12)
C7—C8—C14—C11	−60.29 (13)	C26—C25—C28—C22	60.24 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O1^{i}	1.00	2.58	3.3467 (18)	133
C12—H12···F2^{ii}	1.00	2.30	3.1902 (17)	147
C21—H21···F7^{ii}	1.00	2.55	3.3855 (16)	141
C21—H21···O3^{iii}	1.00	2.51	3.2765 (16)	133
C26—H26···O4^{iv}	1.00	2.51	3.3299 (17)	139
C6—O1···*Cg*1^{v}		3.05 (1)	4.0022 (16)	136 (1)
C13—O2···*Cg*2		3.18 (1)	4.0238 (17)	127 (1)
C27—O4···*Cg*2^{vi}		3.30 (1)	4.0644 (16)	122 (1)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+2; (ii) *x*+1, *y*, *z*; (iii) −*x*+2, −*y*+1, −*z*+1; (iv) −*x*+2, −*y*+2, −*z*+1; (v) −*x*, −*y*+1, −*z*+2; (vi) −*x*+1, −*y*+2, −*z*+1.

(II)

Crystal data

$C_{14}H_8F_4N_2O_3$
M_r = 328.22
Monoclinic, $P2_1/n$
a = 12.285 (6) Å
b = 5.945 (3) Å
c = 17.888 (8) Å
β = 93.44 (3)°
V = 1304.1 (10) Å^3
Z = 4
$F(000)$ = 664
D_x = 1.672 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5752 reflections
θ = 3.3–30.5°
μ = 0.16 mm^{-1}
T = 100 K
Rectangular prism, clear colourless
0.55 × 0.40 × 0.36 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine focus sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
ω Scans scans
Absorption correction: multi-scan (SADABS; Bruker, 2017)
T_{min} = 0.80, T_{max} = 0.95
18507 measured reflections
3823 independent reflections
3443 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 30.0°, θ_{min} = 2.0°
h = −16→17
k = −8→8
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.105
S = 1.04
3823 reflections
208 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2 + 0.5528P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
F1	0.61173 (7)	0.22134 (14)	0.90365 (4)	0.03327 (19)
F2	0.48454 (5)	0.59673 (13)	0.88542 (4)	0.02357 (16)
F3	0.59308 (6)	0.63688 (13)	0.63887 (4)	0.02478 (16)
F4	0.71117 (6)	0.25516 (14)	0.66738 (4)	0.02993 (18)
O1	0.47406 (6)	0.82103 (13)	0.75548 (4)	0.01844 (16)
O2	0.30179 (7)	0.51138 (14)	0.73480 (4)	0.02086 (17)
O3	0.45672 (7)	1.14224 (14)	0.64230 (5)	0.02420 (18)
N1	0.66094 (8)	0.24215 (17)	0.78498 (6)	0.0230 (2)
N2	0.39641 (7)	0.81650 (15)	0.69490 (5)	0.01503 (17)
C1	0.60565 (9)	0.3286 (2)	0.83821 (6)	0.0215 (2)
C2	0.54134 (8)	0.51817 (19)	0.82990 (6)	0.0178 (2)
C3	0.53515 (8)	0.62643 (17)	0.76138 (6)	0.01569 (19)
C4	0.59438 (8)	0.53864 (18)	0.70529 (6)	0.0178 (2)
C5	0.65454 (9)	0.3444 (2)	0.72060 (6)	0.0208 (2)
C6	0.30836 (8)	0.66728 (17)	0.69255 (6)	0.01506 (18)
C7	0.23148 (8)	0.74245 (16)	0.62858 (5)	0.01440 (18)
H7	0.156899	0.76892	0.646338	0.017*
C8	0.22529 (9)	0.59170 (17)	0.55743 (6)	0.01733 (19)
H8	0.178143	0.454959	0.559446	0.021*
C9	0.34224 (9)	0.55197 (19)	0.53633 (6)	0.0201 (2)
H9	0.381115	0.413986	0.540679	0.024*
C10	0.38003 (9)	0.74523 (19)	0.51076 (6)	0.0204 (2)
H10	0.450329	0.769546	0.49285	0.024*
C11	0.29036 (9)	0.92015 (18)	0.51536 (6)	0.01784 (19)
H11	0.296331	1.055951	0.482876	0.021*
C12	0.28051 (8)	0.96515 (17)	0.60041 (6)	0.01497 (18)
H12	0.230305	1.094276	0.608299	0.018*
C13	0.38855 (8)	0.99715 (17)	0.64552 (6)	0.01595 (19)
C14	0.18776 (9)	0.77180 (18)	0.49978 (6)	0.0193 (2)
H14A	0.119519	0.847875	0.512318	0.023*
H14B	0.180981	0.713441	0.447871	0.023*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0372 (4)	0.0362 (4)	0.0258 (4)	0.0035 (3)	−0.0029 (3)	0.0160 (3)
F2	0.0194 (3)	0.0364 (4)	0.0149 (3)	−0.0003 (3)	0.0015 (2)	0.0005 (3)
F3	0.0241 (3)	0.0353 (4)	0.0149 (3)	0.0052 (3)	0.0016 (2)	0.0048 (3)
F4	0.0248 (4)	0.0381 (4)	0.0268 (4)	0.0120 (3)	0.0013 (3)	−0.0068 (3)

O1	0.0189 (3)	0.0198 (4)	0.0157 (3)	0.0032 (3)	−0.0064 (3)	−0.0024 (3)
O2	0.0214 (4)	0.0224 (4)	0.0189 (4)	−0.0009 (3)	0.0014 (3)	0.0074 (3)
O3	0.0243 (4)	0.0187 (4)	0.0289 (4)	−0.0060 (3)	−0.0042 (3)	0.0032 (3)
N1	0.0184 (4)	0.0233 (5)	0.0265 (5)	0.0023 (3)	−0.0049 (4)	0.0021 (4)
N2	0.0135 (4)	0.0177 (4)	0.0134 (4)	−0.0001 (3)	−0.0031 (3)	0.0017 (3)
C1	0.0195 (5)	0.0242 (5)	0.0202 (5)	−0.0011 (4)	−0.0039 (4)	0.0067 (4)
C2	0.0139 (4)	0.0242 (5)	0.0150 (4)	−0.0023 (4)	−0.0013 (3)	0.0013 (4)
C3	0.0122 (4)	0.0182 (4)	0.0162 (4)	−0.0008 (3)	−0.0029 (3)	0.0006 (3)
C4	0.0151 (4)	0.0231 (5)	0.0148 (4)	0.0003 (4)	−0.0018 (3)	0.0016 (4)
C5	0.0151 (4)	0.0248 (5)	0.0220 (5)	0.0024 (4)	−0.0020 (4)	−0.0032 (4)
C6	0.0138 (4)	0.0170 (4)	0.0146 (4)	0.0005 (3)	0.0021 (3)	0.0000 (3)
C7	0.0135 (4)	0.0155 (4)	0.0141 (4)	0.0004 (3)	−0.0003 (3)	0.0010 (3)
C8	0.0197 (5)	0.0151 (4)	0.0168 (4)	−0.0006 (3)	−0.0020 (3)	−0.0007 (3)
C9	0.0235 (5)	0.0196 (5)	0.0172 (5)	0.0056 (4)	0.0004 (4)	−0.0035 (4)
C10	0.0207 (5)	0.0256 (5)	0.0152 (5)	0.0027 (4)	0.0031 (4)	−0.0012 (4)
C11	0.0217 (5)	0.0180 (4)	0.0136 (4)	0.0009 (4)	−0.0006 (3)	0.0027 (3)
C12	0.0153 (4)	0.0140 (4)	0.0153 (4)	0.0014 (3)	−0.0014 (3)	0.0011 (3)
C13	0.0180 (4)	0.0141 (4)	0.0156 (4)	0.0019 (3)	−0.0001 (3)	−0.0002 (3)
C14	0.0209 (5)	0.0209 (5)	0.0155 (4)	0.0015 (4)	−0.0042 (4)	0.0000 (4)

Geometric parameters (Å, º)

F1—C1	1.3312 (14)	C7—C12	1.5511 (15)
F2—C2	1.3323 (13)	C7—C8	1.5547 (15)
F3—C4	1.3231 (13)	C7—H7	1.0
F4—C5	1.3234 (13)	C8—C9	1.5256 (16)
O1—C3	1.3796 (13)	C8—C14	1.5380 (15)
O1—N2	1.4004 (12)	C8—H8	1.0
O2—C6	1.2017 (13)	C9—C10	1.3305 (17)
O3—C13	1.2060 (14)	C9—H9	0.95
N1—C5	1.3005 (16)	C10—C11	1.5207 (16)
N1—C1	1.3076 (16)	C10—H10	0.95
N2—C13	1.3901 (14)	C11—C14	1.5499 (16)
N2—C6	1.3975 (14)	C11—C12	1.5565 (16)
C1—C2	1.3791 (16)	C11—H11	1.0
C2—C3	1.3824 (15)	C12—C13	1.5233 (15)
C3—C4	1.3769 (15)	C12—H12	1.0
C4—C5	1.3891 (16)	C14—H14A	0.99
C6—C7	1.5071 (15)	C14—H14B	0.99
C3—O1—N2	112.66 (8)	C14—C8—C7	98.38 (9)
C5—N1—C1	117.04 (10)	C9—C8—H8	116.0
C13—N2—C6	116.56 (9)	C14—C8—H8	116.0
C13—N2—O1	119.66 (8)	C7—C8—H8	116.0
C6—N2—O1	121.70 (8)	C10—C9—C8	107.73 (9)
N1—C1—F1	116.61 (11)	C10—C9—H9	126.1
N1—C1—C2	124.18 (10)	C8—C9—H9	126.1
F1—C1—C2	119.20 (11)	C9—C10—C11	107.64 (10)

F2—C2—C1	121.89 (10)	C9—C10—H10	126.2
F2—C2—C3	119.63 (10)	C11—C10—H10	126.2
C1—C2—C3	118.47 (10)	C10—C11—C14	100.67 (9)
C4—C3—O1	124.82 (9)	C10—C11—C12	105.64 (8)
C4—C3—C2	117.79 (10)	C14—C11—C12	99.41 (8)
O1—C3—C2	117.32 (9)	C10—C11—H11	116.2
F3—C4—C3	120.56 (10)	C14—C11—H11	116.2
F3—C4—C5	121.43 (10)	C12—C11—H11	116.2
C3—C4—C5	118.00 (10)	C13—C12—C7	106.11 (8)
N1—C5—F4	116.30 (10)	C13—C12—C11	115.01 (9)
N1—C5—C4	124.49 (10)	C7—C12—C11	103.02 (8)
F4—C5—C4	119.20 (10)	C13—C12—H12	110.8
O2—C6—N2	123.48 (10)	C7—C12—H12	110.8
O2—C6—C7	130.06 (10)	C11—C12—H12	110.8
N2—C6—C7	106.45 (9)	O3—C13—N2	124.12 (10)
C6—C7—C12	105.18 (8)	O3—C13—C12	130.51 (10)
C6—C7—C8	116.63 (9)	N2—C13—C12	105.38 (8)
C12—C7—C8	103.12 (8)	C8—C14—C11	93.88 (8)
C6—C7—H7	110.5	C8—C14—H14A	112.9
C12—C7—H7	110.5	C11—C14—H14A	112.9
C8—C7—H7	110.5	C8—C14—H14B	112.9
C9—C8—C14	101.00 (9)	C11—C14—H14B	112.9
C9—C8—C7	106.93 (8)	H14A—C14—H14B	110.4

C3—O1—N2—C13	−130.66 (10)	N2—C6—C7—C8	107.89 (10)
C3—O1—N2—C6	66.28 (12)	C6—C7—C8—C9	−50.39 (11)
C5—N1—C1—F1	179.27 (10)	C12—C7—C8—C9	64.30 (10)
C5—N1—C1—C2	0.48 (17)	C6—C7—C8—C14	−154.65 (9)
N1—C1—C2—F2	178.42 (10)	C12—C7—C8—C14	−39.97 (9)
F1—C1—C2—F2	−0.34 (16)	C14—C8—C9—C10	31.98 (11)
N1—C1—C2—C3	−0.67 (17)	C7—C8—C9—C10	−70.39 (11)
F1—C1—C2—C3	−179.43 (10)	C8—C9—C10—C11	1.02 (12)
N2—O1—C3—C4	55.79 (13)	C9—C10—C11—C14	−33.35 (11)
N2—O1—C3—C2	−127.50 (10)	C9—C10—C11—C12	69.69 (11)
F2—C2—C3—C4	−179.44 (9)	C6—C7—C12—C13	4.58 (10)
C1—C2—C3—C4	−0.33 (15)	C8—C7—C12—C13	−118.12 (9)
F2—C2—C3—O1	3.61 (14)	C6—C7—C12—C11	125.81 (8)
C1—C2—C3—O1	−177.28 (9)	C8—C7—C12—C11	3.12 (9)
O1—C3—C4—F3	−2.24 (16)	C10—C11—C12—C13	45.57 (11)
C2—C3—C4—F3	−178.94 (9)	C14—C11—C12—C13	149.54 (9)
O1—C3—C4—C5	178.10 (9)	C10—C11—C12—C7	−69.40 (10)
C2—C3—C4—C5	1.40 (15)	C14—C11—C12—C7	34.56 (9)
C1—N1—C5—F4	179.84 (10)	C6—N2—C13—O3	178.19 (10)
C1—N1—C5—C4	0.74 (17)	O1—N2—C13—O3	14.27 (15)
F3—C4—C5—N1	178.64 (10)	C6—N2—C13—C12	−1.96 (12)
C3—C4—C5—N1	−1.70 (17)	O1—N2—C13—C12	−165.88 (8)
F3—C4—C5—F4	−0.44 (16)	C7—C12—C13—O3	177.99 (11)
C3—C4—C5—F4	179.22 (10)	C11—C12—C13—O3	64.81 (15)

C13—N2—C6—O2	−175.80 (10)	C7—C12—C13—N2	−1.85 (10)
O1—N2—C6—O2	−12.23 (15)	C11—C12—C13—N2	−115.02 (9)
C13—N2—C6—C7	5.00 (12)	C9—C8—C14—C11	−48.57 (9)
O1—N2—C6—C7	168.57 (8)	C7—C8—C14—C11	60.61 (9)
O2—C6—C7—C12	175.23 (11)	C10—C11—C14—C8	49.16 (9)
N2—C6—C7—C12	−5.64 (10)	C12—C11—C14—C8	−58.86 (9)
O2—C6—C7—C8	−71.24 (14)		