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# (Z)-3-([3-Methoxy-5-(trifluoromethyl)phenyl]- imino)methyl)benzene-1,2-diol

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The title compound, C<sub>15</sub>H<sub>12</sub>F<sub>3</sub>NO<sub>3</sub>, crystallizes with one molecule in the asymmetric unit. The mean planes of the two phenyl rings of the Schiff base moiety, bearing the OH groups and the imine group, respectively, are inclined to each other by 4.91 (1)°. In the crystal, molecules are linked *via* pairs of bifurcated O—H...O hydrogen bonds between the phenol OH groups, forming inversion dimers with an R<sub>1</sub><sup>2</sup>(5) ring motif. The structure exhibits also intramolecular O—H...N and C—H...F hydrogen-bonding interactions. Hirshfeld surfaces analysis and two-dimensional fingerprint plots were applied to quantify the intermolecular interactions. The three F atoms of the trifluoromethyl group are disordered over two sets of sites, with occupancy factors of 0.578 (8) and 0.422 (8). The crystal studied was refined as an inversion twin

## 1. Chemical context

Schiff bases (azomethines, imines) belong to a widely used group of organic compounds or intermediates that are important for production of certain chemical specialties, e.g. pharmaceuticals, or additives to rubber. The synthesis involves an aromatic amine and an aldehyde (Schiff *et al.*, 1881). Schiff bases are also employed as catalyst carriers (Grigoras *et al.*, 2001), thermo-stable materials (Vančo *et al.*, 2004), metal-cation complexing agents or in biological systems (Taggi *et al.*, 2002). Furthermore, they are used as starting materials in the synthesis of significant drugs with properties such as anti-fungal, antibacterial, antimalarial, antiproliferative, anti-inflammatory, antiviral, and antipyretic (Hadjoudis *et al.*, 1987). On an industrial scale, they have a wide range of applications such as dyes and pigments.

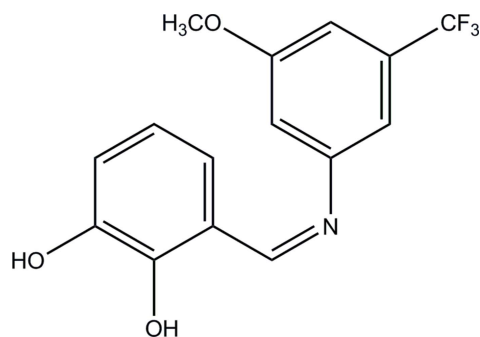
In general, Schiff bases display two possible tautomeric forms, *viz.* phenol-imine and keto-amine. Depending on the tautomers, two types of intramolecular hydrogen bonds are observed in Schiff bases: O—H...N in phenol-imine and N—H...O in keto-amine tautomers. In the present study, a new Schiff base, (Z)-3-([3-methoxy-5-(trifluoromethyl)phenyl]-imino)methyl)benzene-1,2-diol, was obtained in crystalline form from the reaction of 2,3-dihydroxybenzaldehyde with 3-methoxy-5-(trifluoromethyl)aniline.



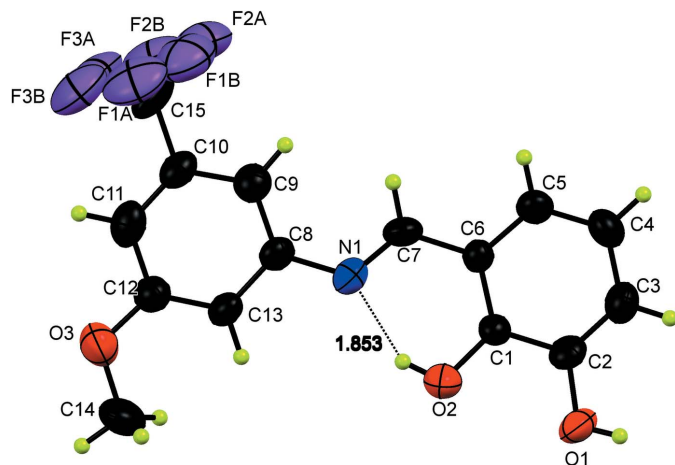
## 2. Structural commentary

The title compound crystallizes as the phenol-imine tautomer with one molecule in the asymmetric unit (Fig. 1). The two

phenyl rings of the Schiff base (C1–C6 and C8–C13) are inclined at an angle of  $4.91(1)^\circ$  with respect to one another. The orientation of the two hydroxy groups with respect to their tautomeric counterparts is defined by the torsion angles  $T1(C1-C6-C7-N1)$  and  $T2(C7-N1-C8-C9)$ . The respective values of  $= 2.0(10)$  and  $-5.5(11)^\circ$  indicate that the molecule is not planar (Ünver *et al.*, 2016).



In the molecule, the C=N group has a strong electron-withdrawing character as revealed by the double-bond character of N1=C7 [1.269(8) Å] and the single bond character of O1–C2 [1.368(6) Å] in the phenol–imine tautomer. These values and other bond lengths and angles (Table 1) are in good agreement with those previously reported for C=N and O–C bonds (Koşar *et al.*, 2010; Demir Kanmazalp *et al.*, 2019). One of the hydroxy groups (O2) makes a strong intramolecular O–H...N bond to the imine N atom (Fig. 1, Table 2) with an  $S(6)$  ring motif, characteristic of *o*-hydroxysalicylidene systems. Other intramolecular hydrogen bonding interactions involve the disordered  $-CF_3$  group and adjacent aromatic H atoms bonded to C9 and C11 (Table 2). As a result of the strongly bent C–H...F angles of about  $100^\circ$ , these contributions are of minor importance.



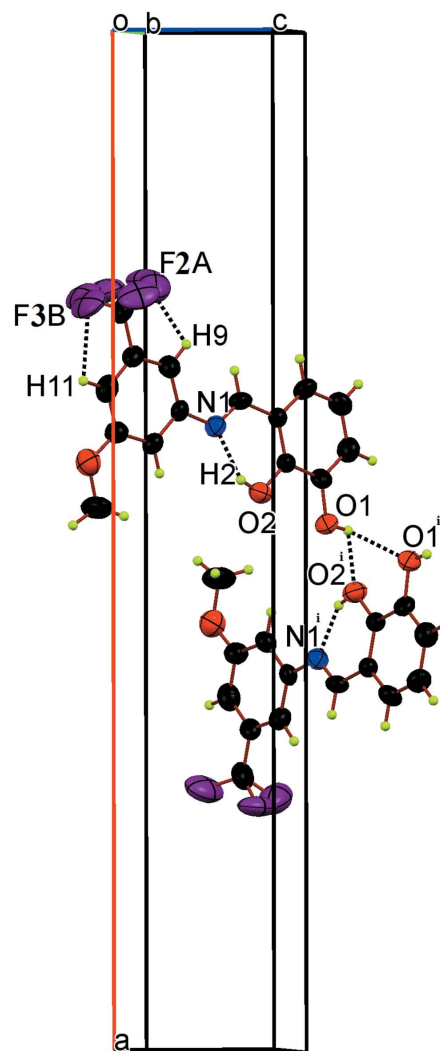
**Figure 1**  
The molecular structure of the title compound with the atomic numbering scheme. The dashed line shows the intramolecular O–H...N hydrogen bond. Displacement ellipsoids are drawn at the 50% probability level.

**Table 1**  
Selected geometric parameters (Å, °).

O1–C2	1.368 (6)	C1–C6	1.394 (8)
O2–C1	1.350 (7)	C6–C5	1.385 (8)
N1–C7	1.269 (8)	C6–C7	1.462 (9)
N1–C8	1.409 (8)		
C12–O3–C14	116.5 (6)	C9–C8–N1	125.6 (7)
C7–N1–C8	123.1 (6)	N1–C7–C6	122.1 (6)
C7–N1–C8–C9	$-5.5(11)$	C1–C6–C7–N1	$2.0(10)$
C5–C6–C7–N1	$-178.7(7)$		

### 3. Supramolecular features

Between adjacent molecules there are bifurcated intermolecular O1–H... $(O1,O2)$  hydrogen bonds with an  $R_1^2(5)$  graph-set motif (Fig. 2, Table 2), leading to the formation of chains parallel to [001]. Despite the presence of aromatic systems, the molecule is arranged in such a way that  $\pi$ – $\pi$  or C–H... $\pi$  interactions are not favoured.



**Figure 2**  
Unit-cell packing diagram for the title compound. Intra- and intermolecular hydrogen bonds are shown as dashed lines. [Symmetry code: (i)  $-x - 1, -y, z - \frac{1}{2}$ .]

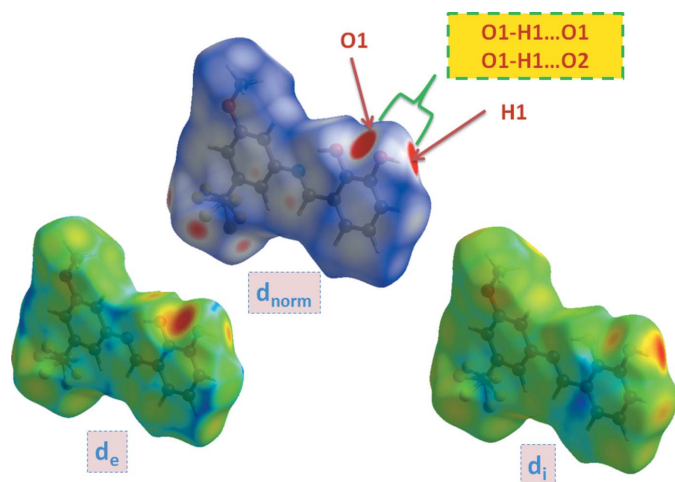
**Table 2**  
 Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O1-H1\cdots O1^i$	0.82	1.99	2.770 (4)	159
$O1-H1\cdots O2^i$	0.82	2.72	3.190 (6)	118
$O2-H2\cdots N1$	0.82	1.85	2.581 (7)	147
$C9-H9\cdots F2A$	0.93	2.44	2.757 (12)	100
$C11-H11\cdots F3B$	0.93	2.39	2.725 (18)	101

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

#### 4. Database survey

A search of the Cambridge Structural Database (CSD, version 5.40, update Nov 2018; Groom *et al.*, 2016) for the (*Z*)-1-phenyl-*N*-[3-(trifluoromethyl)phenyl]methanimine skeleton yielded eight matches. Distinctive bond lengths (here  $N1=C7$ ,  $C1-O2$ ) in the Schiff base structure are the same within standard uncertainties as those of the corresponding bond lengths in the structures of 4*N*-[3,5-bis(trifluoromethyl)phenyl]-3-methoxysalicylalimine (Karadayı *et al.*, 2003), 2-[[3,5-bis(trifluoromethyl)phenyl]carboimidoyl]phenol (Yıldız *et al.*, 2015), 2-[[3,5-bis(trifluoromethyl)phenyl]carboimidoyl]phenol (Ünver *et al.*, 2016), (*E*)-3-[[3-(trifluoromethyl)phenylimino]methyl]benzene-1,2-diol (Koşar *et al.*, 2010), 2-fluoro-*N*-(3-nitrobenzylidene)-5-(trifluoromethyl)aniline (Yang *et al.*, 2007), (*E*)-2-methyl-6-[3-(trifluoromethyl)phenyliminomethyl]phenol (Akkaya *et al.*, 2007), (*E*)-2-[(4-chlorophenyl)iminomethyl]-4-(trifluoromethoxy)phenol (Tüfekçi *et al.*, 2009) and (*E*)-4-methyl-2-[3-(trifluoromethyl)phenyliminomethyl]phenol (Gül *et al.*, 2007). The  $C=N$  bond lengths in these structures vary from 1.270 (3) to 1.295 (5) Å and the  $C-O$  bond lengths from 1.336 (5) to 1.366 (2) Å. The molecular conformations of these structures are also not planar, with dihedral angles between the phenyl rings varying between 5.00 (5) and 47.62 (9)°. It is likely that the intramolecular  $O-H\cdots N$  hydrogen bond, where the imine N atom acts as a hydrogen-bond acceptor, is an important pre-

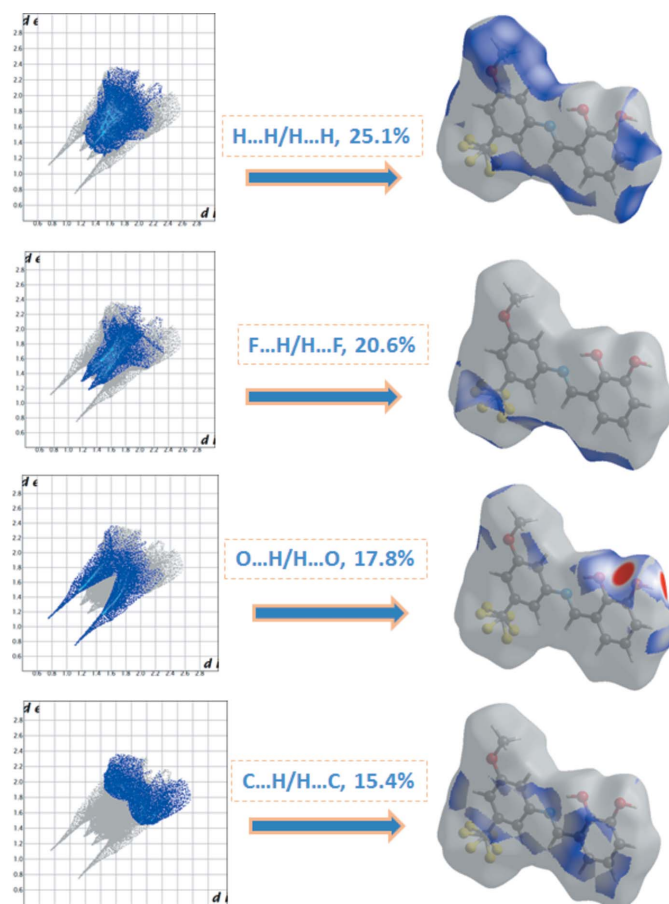


**Figure 3**  
 View of the three-dimensional Hirshfeld surface of the title compound plotted over  $d_{\text{norm}}$  (in the range  $-0.211$  to  $1.077$  a.u.),  $d_e$  and  $d_i$ .

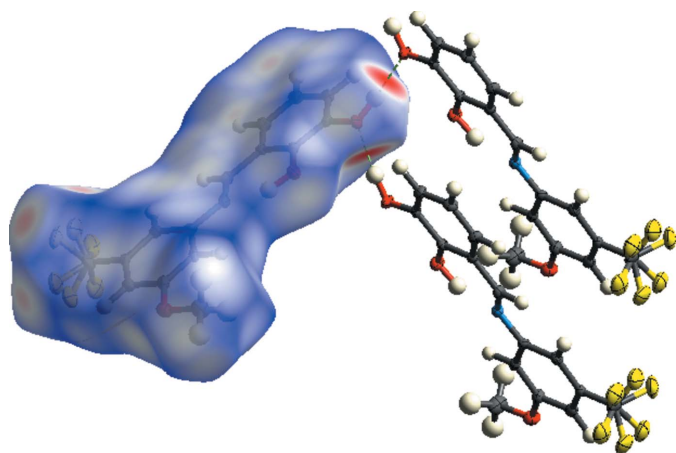
quisite for the tautomeric shift toward the phenol–imine form. In fact, in all eight structures of the phenol–imine tautomers, hydrogen bonds of this type are observed.

#### 5. Hirshfeld surface analysis

Hirshfeld surface analysis of the title compound was performed utilizing the *CrystalExplorer* program (Turner *et al.*, 2017). The three-dimensional  $d_{\text{norm}}$  surface is a useful tool for analysing and visualizing the intermolecular interactions and utilizes the function of the normalized distances  $d_e$  and  $d_i$ , where  $d_e$  and  $d_i$  are the distances from a given point on the surface to the nearest atom outside and inside, respectively. The blue, white and red colour convention used for the  $d_{\text{norm}}$ -mapped Hirshfeld surfaces indicates the interatomic contacts longer, equal to or shorter than the van der Waals separations. The standard-resolution molecular 3D ( $d_{\text{norm}}$ ) plot with  $d_e$  and  $d_i$  for the title compound is shown in Fig. 3. The bright-red spots near the oxygen and hydrogen atoms indicate donors and acceptors of a potential  $O-H\cdots O$  interaction. As can be seen from the two-dimensional fingerprint plots (scattering points spread up to  $d_e = d_i = 1.5$  Å; Fig. 4), the dominant interaction in the title compound originates from  $H\cdots H$  contacts, which are the major contributor (25.1%) to the total



**Figure 4**  
 Two-dimensional fingerprint plots of the crystal with the relative contributions of the atom pairs to the Hirshfeld surface.



**Figure 5**  
Hirshfeld surface mapped over  $d_{\text{norm}}$  to visualize the intermolecular interactions.

Hirshfeld surface. The contribution from the  $\text{O} \cdots \text{H}/\text{H} \cdots \text{O}$  contacts, corresponding to medium  $\text{O1} - \text{H1} \cdots \text{O1}$  and  $\text{O1} - \text{H1} \cdots \text{O2}$  intermolecular interactions (9.6% + 8.2% = 17.8%), is represented by a pair of sharp spikes that are the characteristics of hydrogen-bonding interactions (Fig. 4). Other significant interactions are  $\text{F} \cdots \text{H}/\text{H} \cdots \text{F}$  (20.6%) and  $\text{C} \cdots \text{H}/\text{H} \cdots \text{C}$  (15.4%). While it is likely there are other identifiable points of contact that can be highlighted in the crystal, these may be of limited significance and do not require detailed discussion nor illustration. The interactions are visualized in Fig. 5.

## 6. Synthesis and crystallization

The title compound was prepared by refluxing mixed solutions of 2,3-dihydroxybenzaldehyde (34.5 mg, 0.25 mmol) in ethanol (15 ml) and 3-methoxy-5-(trifluoromethyl)aniline (47.8 mg, 0.25 mmol) in ethanol (15 ml). The reaction mixture was stirred for 5 h under reflux. Single crystals of the title compound for X-ray analysis were obtained by slow evaporation of an ethanol solution (yield 65%, m.p. 396–398 K).

## 7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The carbon-bound H atoms were positioned with idealized geometry and refined isotropically with  $\text{C} - \text{H}$  distances of 0.93–0.96 Å and  $U_{\text{iso}}(\text{H})$  set to 1.2–1.5 $U_{\text{eq}}(\text{C})$ , and with  $\text{O} - \text{H} = 0.82$  Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ . The three F atoms of the trifluoromethyl group are disordered over two sets of sites, with occupancy factors of 0.578 (8) for F atoms with suffix *A* and 0.422 (8) for those with suffix *B* (Fig. 1). A similar behaviour for a disordered  $-\text{CF}_3$  group was observed in a previous study (Demir *et al.*, 2006). Restraints (SIMU, DELU and ISOR; Sheldrick *et al.*, 2015b) were finally applied to the disordered trifluoromethyl group. As a result of

**Table 3**  
Experimental details.

Crystal data	
Chemical formula	$\text{C}_{15}\text{H}_{12}\text{F}_3\text{NO}_3$
$M_r$	311.26
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	296
$a, b, c$ (Å)	30.790 (3), 9.0703 (6), 4.8579 (3)
$V$ (Å <sup>3</sup> )	1356.69 (17)
$Z$	4
Radiation type	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	0.13
Crystal size (mm)	0.79 × 0.32 × 0.05
Data collection	
Diffractometer	Stoe IPDS 2
Absorption correction	Integration ( <i>X-RED32</i> ; Stoe & Cie, 2002)
$T_{\text{min}}, T_{\text{max}}$	0.957, 0.995
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	7150, 2140, 1110
$R_{\text{int}}$	0.099
$(\sin \theta/\lambda)_{\text{max}}$ (Å <sup>-1</sup> )	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.062, 0.088, 0.95
No. of reflections	2140
No. of parameters	231
No. of restraints	73
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	0.14, -0.16
Absolute structure	Refined as an inversion twin
Absolute structure parameter	3 (3)

Computer programs: *X-AREA* and *X-RED32* (Stoe & Cie, 2002), *SHELXT2018* (Sheldrick, 2015a), *SHELXL2018* (Sheldrick, 2015b), *Mercury* (Macrae *et al.*, 2006), *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

missing anomalous dispersion, the absolute structure of the title compound could not be determined reliably (Table 3).

## Funding information

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## supporting information

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**(Z)-3-([3-Methoxy-5-(trifluoromethyl)phenyl]imino)methylbenzene-1,2-diol**

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**Computing details**

Data collection: *X-Area* (Stoe & Cie, 2002); cell refinement: *X-Area* (Stoe & Cie, 2002); data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXT2018* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL2018* (Sheldrick, 2015b), *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

**(Z)-3-([3-Methoxy-5-(trifluoromethyl)phenyl]imino)methylbenzene-1,2-diol***Crystal data*

C<sub>15</sub>H<sub>12</sub>F<sub>3</sub>NO<sub>3</sub>

*M<sub>r</sub>* = 311.26

Orthorhombic, *Pna*2<sub>1</sub>

*a* = 30.790 (3) Å

*b* = 9.0703 (6) Å

*c* = 4.8579 (3) Å

*V* = 1356.69 (17) Å<sup>3</sup>

*Z* = 4

*F*(000) = 640

*D<sub>x</sub>* = 1.524 Mg m<sup>-3</sup>

Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 5897 reflections

θ = 2.2–27.0°

μ = 0.13 mm<sup>-1</sup>

*T* = 296 K

Prism, red

0.79 × 0.32 × 0.05 mm

*Data collection*

Stoe IPDS 2

diffractometer

Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus

Plane graphite monochromator

Detector resolution: 6.67 pixels mm<sup>-1</sup>

rotation method scans

Absorption correction: integration (X-RED32; Stoe & Cie, 2002)

*T<sub>min</sub>* = 0.957, *T<sub>max</sub>* = 0.995

7150 measured reflections

2140 independent reflections

1110 reflections with *I* > 2σ(*I*)

*R<sub>int</sub>* = 0.099

θ<sub>max</sub> = 25.0°, θ<sub>min</sub> = 2.3°

*h* = -30→36

*k* = -10→10

*l* = -4→5

*Refinement*

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.062

*wR*(*F*<sup>2</sup>) = 0.088

*S* = 0.95

2140 reflections

231 parameters

73 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0144*P*)<sup>2</sup>]

where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

(Δσ)<sub>max</sub> < 0.001

Δρ<sub>max</sub> = 0.14 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.16 e Å<sup>-3</sup>

Absolute structure: Refined as an inversion twin.

Absolute structure parameter: 3 (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.

**Refinement.** Refined as a 2-component inversion twin.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
F1A	-0.2587 (3)	-0.7222 (12)	-0.112 (3)	0.109 (3)	0.578 (8)
F2A	-0.2437 (3)	-0.4973 (12)	-0.058 (4)	0.110 (3)	0.578 (8)
F3A	-0.2630 (4)	-0.6345 (18)	0.281 (3)	0.117 (4)	0.578 (8)
F1B	-0.2486 (5)	-0.605 (3)	-0.165 (4)	0.115 (4)	0.422 (8)
F2B	-0.2558 (4)	-0.4953 (17)	0.212 (5)	0.111 (4)	0.422 (8)
F3B	-0.2668 (6)	-0.7256 (19)	0.191 (5)	0.112 (4)	0.422 (8)
O1	-0.48182 (12)	-0.0397 (5)	-1.1221 (12)	0.0559 (13)	
H1	-0.488061	0.002147	-1.266405	0.084*	
O2	-0.45187 (14)	-0.2219 (5)	-0.7485 (11)	0.0566 (13)	
H2	-0.439356	-0.273123	-0.634283	0.085*	
O3	-0.42289 (17)	-0.6770 (5)	0.2365 (12)	0.0675 (15)	
N1	-0.38682 (18)	-0.3355 (6)	-0.4892 (13)	0.0462 (15)	
C13	-0.4043 (2)	-0.5057 (6)	-0.1300 (19)	0.0429 (18)	
H13	-0.433339	-0.485521	-0.165359	0.051*	
C8	-0.3718 (2)	-0.4343 (7)	-0.2864 (14)	0.0422 (17)	
C2	-0.4376 (2)	-0.0449 (7)	-1.0943 (16)	0.0444 (18)	
C12	-0.3936 (2)	-0.6048 (7)	0.0740 (16)	0.050 (2)	
C1	-0.4223 (2)	-0.1434 (7)	-0.8926 (14)	0.0383 (17)	
C6	-0.37787 (19)	-0.1545 (7)	-0.8426 (13)	0.0367 (17)	
C7	-0.3617 (2)	-0.2539 (7)	-0.6293 (16)	0.0453 (18)	
H7	-0.331950	-0.257523	-0.595114	0.054*	
C10	-0.3189 (2)	-0.5665 (8)	-0.0246 (15)	0.0475 (19)	
C5	-0.3498 (2)	-0.0686 (7)	-0.9968 (15)	0.0477 (19)	
H5	-0.320085	-0.075095	-0.962902	0.057*	
C11	-0.3506 (3)	-0.6359 (7)	0.1267 (16)	0.056 (2)	
H11	-0.343162	-0.703200	0.263109	0.067*	
C9	-0.3286 (2)	-0.4645 (7)	-0.2283 (15)	0.051 (2)	
H9	-0.306471	-0.417196	-0.324352	0.061*	
C3	-0.4091 (2)	0.0371 (7)	-1.2431 (17)	0.054 (2)	
H3	-0.419422	0.101663	-1.376554	0.065*	
C4	-0.3642 (2)	0.0258 (7)	-1.1982 (16)	0.053 (2)	
H4	-0.344674	0.080776	-1.302170	0.064*	
C15	-0.2733 (3)	-0.5977 (14)	0.035 (2)	0.088 (3)	
C14	-0.4674 (2)	-0.6454 (10)	0.189 (2)	0.087 (3)	
H14A	-0.475930	-0.685003	0.013742	0.130*	
H14B	-0.471687	-0.540545	0.188861	0.130*	
H14C	-0.484666	-0.689170	0.331567	0.130*	

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
F1A	0.078 (5)	0.107 (7)	0.142 (8)	0.048 (5)	-0.008 (6)	-0.030 (8)
F2A	0.055 (5)	0.125 (7)	0.148 (9)	0.008 (6)	-0.018 (6)	0.015 (8)
F3A	0.080 (5)	0.155 (10)	0.116 (8)	0.043 (8)	-0.034 (6)	0.014 (8)
F1B	0.074 (6)	0.138 (9)	0.133 (9)	0.034 (8)	-0.001 (7)	0.003 (9)
F2B	0.059 (6)	0.136 (8)	0.138 (8)	0.018 (7)	-0.034 (7)	-0.017 (9)
F3B	0.087 (6)	0.118 (9)	0.132 (9)	0.042 (7)	-0.029 (8)	0.010 (9)
O1	0.041 (3)	0.075 (4)	0.051 (3)	0.014 (2)	-0.006 (3)	0.009 (3)
O2	0.043 (3)	0.067 (3)	0.060 (4)	0.002 (2)	0.003 (3)	0.012 (3)
O3	0.072 (4)	0.067 (3)	0.063 (4)	-0.011 (3)	0.002 (4)	0.012 (3)
N1	0.045 (3)	0.049 (3)	0.044 (4)	0.009 (3)	-0.004 (3)	0.000 (3)
C13	0.042 (4)	0.037 (4)	0.050 (5)	0.005 (3)	-0.009 (4)	0.000 (4)
C8	0.042 (4)	0.052 (4)	0.032 (5)	0.001 (3)	-0.006 (4)	-0.003 (4)
C2	0.039 (4)	0.047 (4)	0.047 (5)	0.013 (3)	0.004 (4)	-0.004 (4)
C12	0.046 (5)	0.049 (4)	0.057 (6)	0.002 (3)	0.000 (5)	0.002 (4)
C1	0.035 (4)	0.044 (4)	0.036 (4)	0.002 (3)	0.004 (4)	0.002 (3)
C6	0.040 (4)	0.036 (4)	0.034 (5)	-0.001 (3)	-0.002 (4)	0.000 (4)
C7	0.030 (3)	0.055 (4)	0.051 (5)	0.000 (3)	-0.004 (4)	-0.004 (4)
C10	0.048 (5)	0.053 (4)	0.042 (5)	0.016 (4)	-0.003 (4)	-0.005 (4)
C5	0.045 (5)	0.050 (4)	0.048 (5)	-0.008 (4)	-0.007 (4)	0.000 (4)
C11	0.077 (6)	0.042 (4)	0.049 (5)	0.009 (4)	-0.005 (5)	-0.002 (4)
C9	0.053 (4)	0.051 (4)	0.048 (5)	0.007 (3)	0.005 (5)	-0.001 (4)
C3	0.061 (5)	0.049 (4)	0.051 (5)	0.009 (4)	-0.002 (5)	0.007 (4)
C4	0.056 (5)	0.052 (4)	0.052 (6)	-0.011 (3)	0.008 (5)	0.006 (4)
C15	0.070 (7)	0.136 (10)	0.059 (7)	0.044 (6)	-0.004 (7)	0.029 (7)
C14	0.051 (5)	0.113 (7)	0.096 (9)	-0.017 (5)	0.008 (6)	0.026 (6)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

F1A—C15	1.410 (14)	C2—C1	1.407 (9)
F2A—C15	1.364 (14)	C12—C11	1.377 (9)
F3A—C15	1.282 (15)	C1—C6	1.394 (8)
F1B—C15	1.236 (19)	C6—C5	1.385 (8)
F2B—C15	1.375 (19)	C6—C7	1.462 (9)
F3B—C15	1.40 (2)	C7—H7	0.9300
O1—C2	1.368 (6)	C10—C11	1.376 (9)
O1—H1	0.8200	C10—C9	1.387 (8)
O2—C1	1.350 (7)	C10—C15	1.462 (10)
O2—H2	0.8200	C5—C4	1.374 (9)
O3—C12	1.366 (8)	C5—H5	0.9300
O3—C14	1.419 (8)	C11—H11	0.9300
N1—C7	1.269 (8)	C9—H9	0.9300
N1—C8	1.409 (8)	C3—C4	1.403 (9)
C13—C12	1.377 (9)	C3—H3	0.9300
C13—C8	1.412 (9)	C4—H4	0.9300
C13—H13	0.9300	C14—H14A	0.9600



C8—C9	1.389 (8)	C14—H14B	0.9600
C2—C3	1.360 (9)	C14—H14C	0.9600
C2—O1—H1	109.5	C10—C11—C12	119.3 (7)
C1—O2—H2	109.5	C10—C11—H11	120.3
C12—O3—C14	116.5 (6)	C12—C11—H11	120.3
C7—N1—C8	123.1 (6)	C10—C9—C8	118.9 (7)
C12—C13—C8	121.2 (6)	C10—C9—H9	120.6
C12—C13—H13	119.4	C8—C9—H9	120.6
C8—C13—H13	119.4	C2—C3—C4	120.9 (7)
C9—C8—N1	125.6 (7)	C2—C3—H3	119.5
C9—C8—C13	118.5 (7)	C4—C3—H3	119.5
N1—C8—C13	115.9 (6)	C5—C4—C3	118.4 (7)
C3—C2—O1	124.9 (7)	C5—C4—H4	120.8
C3—C2—C1	120.1 (6)	C3—C4—H4	120.8
O1—C2—C1	115.0 (6)	F3A—C15—F2A	108.6 (13)
O3—C12—C11	115.4 (6)	F1B—C15—F2B	106.8 (16)
O3—C12—C13	124.9 (6)	F1B—C15—F3B	107.0 (14)
C11—C12—C13	119.7 (7)	F2B—C15—F3B	99.5 (14)
O2—C1—C6	122.2 (6)	F3A—C15—F1A	100.6 (11)
O2—C1—C2	118.1 (6)	F2A—C15—F1A	98.8 (11)
C6—C1—C2	119.7 (6)	F1B—C15—C10	116.4 (12)
C5—C6—C1	118.6 (6)	F3A—C15—C10	118.3 (10)
C5—C6—C7	121.1 (6)	F2A—C15—C10	116.5 (9)
C1—C6—C7	120.2 (6)	F2B—C15—C10	111.7 (10)
N1—C7—C6	122.1 (6)	F3B—C15—C10	113.9 (13)
N1—C7—H7	119.0	F1A—C15—C10	111.1 (10)
C6—C7—H7	119.0	O3—C14—H14A	109.5
C11—C10—C9	122.2 (7)	O3—C14—H14B	109.5
C11—C10—C15	119.2 (8)	H14A—C14—H14B	109.5
C9—C10—C15	118.5 (8)	O3—C14—H14C	109.5
C4—C5—C6	122.2 (7)	H14A—C14—H14C	109.5
C4—C5—H5	118.9	H14B—C14—H14C	109.5
C6—C5—H5	118.9		
C7—N1—C8—C9	-5.5 (11)	O3—C12—C11—C10	-179.1 (6)
C7—N1—C8—C13	173.7 (7)	C13—C12—C11—C10	0.5 (11)
C12—C13—C8—C9	-0.8 (11)	C11—C10—C9—C8	-1.7 (11)
C12—C13—C8—N1	179.9 (6)	C15—C10—C9—C8	180.0 (8)
C14—O3—C12—C11	179.2 (7)	N1—C8—C9—C10	-179.0 (6)
C14—O3—C12—C13	-0.3 (11)	C13—C8—C9—C10	1.7 (11)
C8—C13—C12—O3	179.1 (7)	O1—C2—C3—C4	179.2 (7)
C8—C13—C12—C11	-0.3 (11)	C1—C2—C3—C4	-0.2 (12)
C3—C2—C1—O2	179.6 (7)	C6—C5—C4—C3	1.5 (11)
O1—C2—C1—O2	0.0 (9)	C2—C3—C4—C5	-1.0 (12)
C3—C2—C1—C6	1.1 (11)	C11—C10—C15—F1B	141.8 (17)
O1—C2—C1—C6	-178.4 (6)	C9—C10—C15—F1B	-40 (2)
O2—C1—C6—C5	-179.1 (6)	C11—C10—C15—F3A	-28.9 (18)

C2—C1—C6—C5	-0.7 (10)	C9—C10—C15—F3A	149.5 (13)
O2—C1—C6—C7	0.3 (10)	C11—C10—C15—F2A	-161.1 (11)
C2—C1—C6—C7	178.6 (6)	C9—C10—C15—F2A	17.3 (17)
C8—N1—C7—C6	178.7 (6)	C11—C10—C15—F2B	-95.2 (15)
C5—C6—C7—N1	-178.7 (7)	C9—C10—C15—F2B	83.2 (14)
C1—C6—C7—N1	2.0 (10)	C11—C10—C15—F3B	16.6 (18)
C1—C6—C5—C4	-0.6 (11)	C9—C10—C15—F3B	-165.0 (13)
C7—C6—C5—C4	-179.9 (6)	C11—C10—C15—F1A	86.8 (12)
C9—C10—C11—C12	0.6 (11)	C9—C10—C15—F1A	-94.9 (13)
C15—C10—C11—C12	178.9 (8)		

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O1—H1 $\cdots$ O1 <sup>i</sup>	0.82	1.99	2.770 (4)	159
O1—H1 $\cdots$ O2 <sup>i</sup>	0.82	2.72	3.190 (6)	118
O2—H2 $\cdots$ N1	0.82	1.85	2.581 (7)	147
C9—H9 $\cdots$ F2 <i>A</i>	0.93	2.44	2.757 (12)	100
C11—H11 $\cdots$ F3 <i>B</i>	0.93	2.39	2.725 (18)	101

Symmetry code: (i)  $-x-1, -y, z-1/2$ .