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2-Bromo-*N*′-[(*E*)-(4-fluorophenyl)-methylene]-5-methoxybenzohydrazide monohydrate

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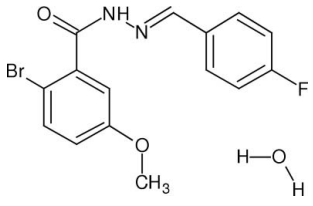
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.013 Å; R factor = 0.080; wR factor = 0.202; data-to-parameter ratio = 13.7.

The crystal packing of the title compound, $C_{15}H_{12}BrFN_2O_2\cdot H_2O$, is stabilized by N—H···O and O—H···O hydrogen bonds. There are two molecules in the asymmetric unit, differing in the dihedral angle between the two aromatic rings, which are 62.3 (2) and 49.9 (2)°.

Related literature

For related structures, see: Bruno *et al.* (1998); Harrison *et al.* (2005); Yathirajan, Sarojini *et al.* (2007); Yathirajan, Narayana, *et al.* (2007). For related literature, see: Varma *et al.* (1986); Misra *et al.* (1981); Agarwal *et al.* (1983); Singh *et al.* (1988); Hodnett *et al.* (1970).



Experimental

Crystal data

$C_{15}H_{12}BrFN_2O_2\cdot H_2O$
M_r = 369.19
Monoclinic, *Cc*
a = 30.1171 (18) Å
b = 8.0187 (7) Å
c = 13.4661 (8) Å
β = 111.902 (4)°
V = 3017.3 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 2.75 mm^{-1}
T = 173 (2) K
0.37 × 0.35 × 0.33 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.379, T_{max} = 0.394
16531 measured reflections
5477 independent reflections
4843 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.080
$wR(F^2)$ = 0.202
S = 1.02
5477 reflections
400 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.76 e Å^{-3}
Absolute structure: Flack (1983), 2642 Friedel pairs
Flack parameter: 0.018 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1*W*	0.88	2.03	2.901 (9)	172
N1*A*—H1*A*···O1*WA*	0.88	2.01	2.860 (9)	163
O1*W*—H1*W*1···O1^{i}	0.84	1.99	2.832 (8)	180
O1*W*—H1*W*2···O1*A*^{ii}	0.84	2.01	2.856 (8)	180
O1*WA*—H1*W*3···O1^{iii}	0.84	2.06	2.897 (8)	180
O1*WA*—H1*W*4···O1*A*^{iv}	0.84	1.96	2.802 (7)	180

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

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2297).

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supplementary materials

Acta Cryst. (2007). E**63**, o2948 [**doi:10.1107/S1600536807024348**]

2-Bromo-*N*'-[(*E*)-(4-fluorophenyl)methylene]-5-methoxybenzohydrazide monohydrate

B. Narayana, K. Sunil, H. S. Yathirajan, B. K. Sarojini and M. Bolte

Comment

Schiff bases are used as substrates in the preparation of number of industrial and biologically active compounds *via* ring closure, cycloaddition and replacement reactions. Some Schiff base derivatives were reported to possess antimicrobial, anti-inflammatory and central nervous system activities. Moreover, Schiff bases are also known to have biological activities such as antimicrobial, antifungal, antitumor, and as herbicides. A new Schiff base, (I), $C_{15}H_{12}BrFN_2O_2.H_2O$ is synthesized and its crystal structure is reported.

The two molecules in the asymmetric unit differ in the dihedral angle between the two aromatic rings, which are 62.3 (2)° and 49.9 (2)°. The crystal packing is stabilized by N—H···O and O—H···O hydrogen bonds.

Experimental

A mixture of 2-bromo-5-methoxybenzohydrazide (0.735 g, 0.003 mol) and 4-fluorobenzaldehyde (0.372 g, 0.003 mol) in 15 ml of absolute alcohol containing 2 drops of 4 *M* sulfuric acid was refluxed for about 3 h (see scheme). On cooling, the solid separated was filtered off and recrystallized from ethyl acetate (m.p.: 440–442 K). Analysis for $C_{15}H_{12}BrFN_2O_2.H_2O$: Found (calculated): C 48.71 (48.80), H 3.76 (3.82), N 7.51 (7.59) %.

Refinement

H atoms were found in a difference map, but they were refined using a riding model with C—H = 0.95 Å, N—H = 0.88 Å, O—H = 0.84Å and U_{iso}(H) = 1.2U_{eq}(C,*N*,*O*) or C—H = 0.98Å and U_{iso}(H) = 1.5U_{eq}(C_{methyl}).

Figures

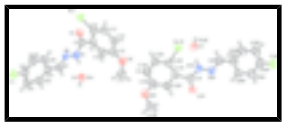


Fig. 1. The structure of the two independent molecules in the asymmetric unit of the title compound, with the atom numbering. Displacement ellipsoids are at the 50% probability level.
Fig. 2. Reaction scheme.

2-Bromo-*N*'-[(1E)-(4-fluorophenyl)methylene]-5-methoxybenzohydrazide monohydrate

Crystal data

$C_{15}H_{12}BrFN_2O_2.H_2O$	$F(000)$ = 1488
M_r = 369.19	D_x = 1.625 Mg m^{-3}
Monoclinic, *Cc*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: C -2yc	Cell parameters from 11319 reflections
a = 30.1171 (18) Å	θ = 3.6–25.1°

b = 8.0187 (7) Å	μ = 2.75 mm^{-1}
c = 13.4661 (8) Å	T = 173 K
β = 111.902 (4)°	Block, colourless
V = 3017.3 (4) Å^3	0.37 × 0.35 × 0.33 mm
Z = 8	

Data collection

STOE IPDS II two-circle-diffractometer	5477 independent reflections
Radiation source: fine-focus sealed tube	4843 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.059
ω scans	θ_{max} = 25.6°, θ_{min} = 3.5°
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)	h = −36→35
T_{min} = 0.379, T_{max} = 0.394	k = −9→9
16531 measured reflections	l = −16→16

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.080	$w = 1/[\sigma^2(F_o^2) + (0.163P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.202	$(\Delta/\sigma)_{max} < 0.001$
S = 1.02	$\Delta\rho_{max}$ = 0.56 e Å^{-3}
5477 reflections	$\Delta\rho_{min}$ = −0.76 e Å^{-3}
400 parameters	Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
2 restraints	Extinction coefficient: 0.0062 (8)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 2642 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.018 (17)

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ 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}
Br1	0.52393 (3)	0.24296 (11)	0.77882 (6)	0.0677 (3)
F1	0.82483 (19)	0.6614 (8)	0.6074 (5)	0.0838 (15)
O1	0.5622 (2)	0.5636 (7)	0.6968 (4)	0.0603 (12)
O2	0.3807 (2)	0.4246 (8)	0.3428 (5)	0.0697 (14)
N1	0.5647 (3)	0.4797 (9)	0.5375 (5)	0.0578 (14)
H1	0.5499	0.4312	0.4752	0.069*
N2	0.6106 (2)	0.5460 (8)	0.5654 (5)	0.0548 (13)
C1	0.5439 (3)	0.4937 (9)	0.6110 (6)	0.0557 (15)
C2	0.6317 (3)	0.5090 (10)	0.5039 (7)	0.0594 (17)
H2	0.6153	0.4475	0.4404	0.071*
C11	0.4947 (3)	0.4167 (8)	0.5755 (6)	0.0540 (15)
C12	0.4806 (3)	0.3189 (9)	0.6429 (5)	0.0555 (15)
C13	0.4333 (3)	0.2595 (9)	0.6109 (7)	0.0584 (17)
H13	0.4237	0.1914	0.6571	0.070*
C14	0.4013 (3)	0.3030 (12)	0.5107 (7)	0.0623 (19)
H14	0.3689	0.2680	0.4893	0.075*
C15	0.4150 (3)	0.3964 (10)	0.4402 (7)	0.0605 (17)
C16	0.4613 (3)	0.4553 (9)	0.4731 (6)	0.0568 (16)
H16	0.4707	0.5222	0.4261	0.068*
C17	0.3953 (3)	0.5087 (12)	0.2651 (7)	0.070 (2)
H17A	0.4090	0.6176	0.2933	0.105*
H17B	0.3674	0.5243	0.1986	0.105*
H17C	0.4193	0.4412	0.2506	0.105*
C21	0.6819 (3)	0.5605 (10)	0.5292 (6)	0.0554 (15)
C22	0.7068 (3)	0.4959 (10)	0.4660 (7)	0.0625 (17)
H22	0.6901	0.4297	0.4049	0.075*
C23	0.7554 (3)	0.5292 (11)	0.4933 (7)	0.0629 (18)
H23	0.7725	0.4847	0.4528	0.076*
C24	0.7774 (3)	0.6282 (11)	0.5806 (7)	0.0648 (19)
C25	0.7545 (3)	0.6977 (11)	0.6432 (7)	0.0661 (18)
H25	0.7712	0.7684	0.7019	0.079*
C26	0.7071 (3)	0.6607 (9)	0.6175 (6)	0.0583 (16)
H26	0.6910	0.7039	0.6605	0.070*
Br1A	0.15181 (3)	0.26881 (10)	0.25235 (4)	0.0646 (3)
C1A	0.1256 (3)	0.4374 (10)	0.0114 (6)	0.0569 (16)
F1A	−0.1718 (2)	0.6188 (9)	−0.1250 (5)	0.0903 (17)
O1A	0.1093 (2)	0.3990 (7)	−0.0840 (4)	0.0620 (12)
O2A	0.2918 (2)	0.3916 (8)	0.0344 (5)	0.0682 (14)
N1A	0.0998 (2)	0.5084 (9)	0.0612 (5)	0.0583 (14)
H1A	0.1135	0.5470	0.1268	0.070*
N2A	0.0509 (3)	0.5199 (8)	0.0075 (5)	0.0576 (15)
C2A	0.0269 (3)	0.5737 (10)	0.0615 (6)	0.0597 (16)
H2A	0.0430	0.6068	0.1339	0.072*
C11A	0.1769 (3)	0.3985 (8)	0.0781 (6)	0.0543 (15)
C12A	0.1936 (3)	0.3191 (9)	0.1777 (6)	0.0551 (15)

C13A	0.2403 (3)	0.2667 (10)	0.2268 (7)	0.0604 (17)
H13A	0.2504	0.2118	0.2941	0.072*
C14A	0.2724 (3)	0.2947 (10)	0.1770 (8)	0.0645 (18)
H14A	0.3048	0.2601	0.2108	0.077*
C15A	0.2577 (3)	0.3727 (10)	0.0784 (6)	0.0606 (16)
C16A	0.2104 (3)	0.4230 (10)	0.0277 (6)	0.0565 (16)
H16A	0.2004	0.4739	−0.0409	0.068*
C17A	0.2772 (3)	0.4652 (12)	−0.0697 (7)	0.071 (2)
H17D	0.2507	0.4004	−0.1200	0.107*
H17E	0.3042	0.4654	−0.0936	0.107*
H17F	0.2666	0.5800	−0.0667	0.107*
C21A	−0.0256 (3)	0.5847 (10)	0.0117 (7)	0.0625 (18)
C22A	−0.0509 (3)	0.6564 (10)	0.0689 (8)	0.0665 (18)
H22A	−0.0343	0.6958	0.1397	0.080*
C23A	−0.1008 (3)	0.6706 (12)	0.0220 (9)	0.073 (2)
H23A	−0.1185	0.7223	0.0590	0.088*
C24A	−0.1232 (3)	0.6070 (13)	−0.0792 (8)	0.077 (2)
C25A	−0.0994 (3)	0.5372 (13)	−0.1390 (8)	0.073 (2)
H25A	−0.1163	0.5000	−0.2101	0.088*
C26A	−0.0501 (3)	0.5231 (9)	−0.0922 (7)	0.0611 (17)
H26A	−0.0328	0.4717	−0.1302	0.073*
O1W	0.5235 (2)	0.2938 (8)	0.3394 (5)	0.0634 (12)
H1W1	0.5349	0.3360	0.2969	0.076*
H1W2	0.5488	0.2372	0.3619	0.076*
O1WA	0.1404 (2)	0.7000 (7)	0.2531 (4)	0.0585 (12)
H1W3	0.1178	0.7686	0.2367	0.070*
H1W4	0.1311	0.6703	0.3020	0.070*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0677 (5)	0.0716 (5)	0.0613 (5)	−0.0041 (4)	0.0213 (4)	0.0093 (3)
F1	0.055 (3)	0.098 (4)	0.102 (4)	−0.007 (2)	0.033 (3)	0.007 (3)
O1	0.059 (3)	0.067 (3)	0.060 (3)	−0.003 (2)	0.027 (2)	−0.004 (2)
O2	0.056 (3)	0.081 (4)	0.068 (3)	−0.003 (3)	0.019 (3)	0.005 (3)
N1	0.057 (4)	0.059 (3)	0.055 (3)	−0.004 (3)	0.018 (3)	−0.004 (3)
N2	0.051 (3)	0.057 (3)	0.055 (3)	0.001 (2)	0.019 (3)	0.001 (2)
C1	0.057 (4)	0.053 (3)	0.057 (4)	0.000 (3)	0.020 (3)	−0.003 (3)
C2	0.058 (4)	0.055 (4)	0.060 (4)	0.001 (3)	0.017 (3)	0.001 (3)
C11	0.049 (4)	0.048 (3)	0.068 (4)	0.002 (3)	0.025 (3)	−0.003 (3)
C12	0.056 (4)	0.060 (4)	0.045 (3)	0.007 (3)	0.013 (3)	0.002 (3)
C13	0.063 (4)	0.055 (4)	0.065 (4)	−0.003 (3)	0.032 (4)	−0.003 (3)
C14	0.050 (4)	0.066 (5)	0.076 (5)	−0.002 (3)	0.029 (4)	−0.006 (3)
C15	0.062 (4)	0.054 (4)	0.066 (4)	0.000 (3)	0.025 (4)	−0.004 (3)
C16	0.065 (4)	0.051 (3)	0.061 (4)	−0.004 (3)	0.032 (3)	0.001 (3)
C17	0.062 (5)	0.079 (5)	0.061 (4)	−0.004 (4)	0.015 (4)	0.003 (4)
C21	0.054 (4)	0.057 (4)	0.058 (4)	0.003 (3)	0.024 (3)	0.010 (3)
C22	0.071 (5)	0.055 (4)	0.062 (4)	−0.002 (3)	0.025 (4)	0.004 (3)

C23	0.062 (4)	0.067 (4)	0.071 (4)	0.007 (3)	0.038 (4)	0.007 (3)
C24	0.058 (4)	0.070 (5)	0.070 (4)	0.003 (3)	0.029 (4)	0.017 (4)
C25	0.066 (5)	0.064 (4)	0.063 (4)	−0.006 (4)	0.018 (4)	0.006 (3)
C26	0.058 (4)	0.056 (4)	0.067 (4)	0.005 (3)	0.030 (3)	0.007 (3)
Br1A	0.0716 (5)	0.0638 (4)	0.0619 (5)	0.0006 (4)	0.0291 (4)	0.0058 (4)
C1A	0.058 (4)	0.052 (4)	0.060 (4)	−0.001 (3)	0.021 (3)	0.001 (3)
F1A	0.059 (3)	0.117 (5)	0.097 (4)	0.014 (3)	0.031 (3)	0.027 (3)
O1A	0.055 (3)	0.076 (3)	0.053 (2)	0.003 (2)	0.019 (2)	−0.004 (2)
O2A	0.055 (3)	0.073 (3)	0.077 (4)	0.001 (3)	0.026 (3)	−0.001 (3)
N1A	0.055 (3)	0.061 (3)	0.056 (3)	0.001 (3)	0.018 (3)	−0.005 (2)
N2A	0.056 (4)	0.057 (3)	0.058 (3)	0.000 (3)	0.018 (3)	−0.001 (3)
C2A	0.059 (4)	0.053 (4)	0.063 (4)	0.003 (3)	0.018 (3)	0.002 (3)
C11A	0.054 (4)	0.048 (3)	0.061 (4)	−0.001 (3)	0.022 (3)	−0.002 (3)
C12A	0.058 (4)	0.056 (3)	0.058 (4)	−0.004 (3)	0.029 (3)	0.000 (3)
C13A	0.062 (5)	0.060 (4)	0.054 (4)	0.007 (3)	0.017 (3)	0.000 (3)
C14A	0.059 (4)	0.056 (4)	0.074 (5)	0.004 (3)	0.019 (4)	−0.006 (3)
C15A	0.057 (4)	0.058 (4)	0.068 (4)	−0.002 (3)	0.025 (3)	−0.010 (3)
C16A	0.058 (4)	0.054 (4)	0.054 (3)	−0.001 (3)	0.017 (3)	−0.004 (3)
C17A	0.069 (5)	0.075 (5)	0.074 (5)	0.000 (4)	0.031 (4)	0.000 (4)
C21A	0.059 (4)	0.055 (4)	0.074 (5)	0.002 (3)	0.026 (4)	0.003 (3)
C22A	0.065 (5)	0.061 (4)	0.079 (5)	−0.001 (3)	0.033 (4)	−0.001 (3)
C23A	0.071 (5)	0.055 (4)	0.106 (7)	0.007 (3)	0.046 (5)	0.009 (4)
C24A	0.059 (5)	0.087 (6)	0.085 (6)	0.003 (4)	0.026 (4)	0.021 (5)
C25A	0.062 (5)	0.087 (6)	0.072 (5)	−0.009 (4)	0.027 (4)	0.004 (4)
C26A	0.061 (4)	0.057 (4)	0.069 (4)	0.005 (3)	0.029 (4)	0.012 (3)
O1W	0.062 (3)	0.067 (3)	0.062 (3)	0.002 (2)	0.023 (3)	−0.003 (2)
O1WA	0.060 (3)	0.060 (3)	0.057 (3)	0.006 (2)	0.023 (2)	0.004 (2)

Geometric parameters (Å, °)

Br1—C12	1.908 (7)	C1A—N1A	1.331 (11)
F1—C24	1.363 (10)	C1A—C11A	1.501 (11)
O1—C1	1.216 (9)	F1A—C24A	1.363 (11)
O2—C15	1.352 (11)	O2A—C15A	1.371 (10)
O2—C17	1.444 (11)	O2A—C17A	1.431 (11)
N1—C1	1.358 (10)	N1A—N2A	1.378 (10)
N1—N2	1.396 (10)	N1A—H1A	0.8800
N1—H1	0.8800	N2A—C2A	1.276 (11)
N2—C2	1.254 (11)	C2A—C21A	1.474 (12)
C1—C11	1.510 (10)	C2A—H2A	0.9500
C2—C21	1.480 (12)	C11A—C12A	1.398 (10)
C2—H2	0.9500	C11A—C16A	1.425 (11)
C11—C12	1.381 (11)	C12A—C13A	1.380 (12)
C11—C16	1.405 (11)	C13A—C14A	1.385 (14)
C12—C13	1.408 (12)	C13A—H13A	0.9500
C13—C14	1.379 (13)	C14A—C15A	1.382 (13)
C13—H13	0.9500	C14A—H14A	0.9500
C14—C15	1.386 (12)	C15A—C16A	1.390 (12)
C14—H14	0.9500	C16A—H16A	0.9500

C15—C16	1.380 (11)	C17A—H17D	0.9800
C16—H16	0.9500	C17A—H17E	0.9800
C17—H17A	0.9800	C17A—H17F	0.9800
C17—H17B	0.9800	C21A—C22A	1.393 (12)
C17—H17C	0.9800	C21A—C26A	1.405 (12)
C21—C26	1.401 (12)	C22A—C23A	1.400 (13)
C21—C22	1.423 (12)	C22A—H22A	0.9500
C22—C23	1.396 (13)	C23A—C24A	1.373 (16)
C22—H22	0.9500	C23A—H23A	0.9500
C23—C24	1.368 (13)	C24A—C25A	1.382 (15)
C23—H23	0.9500	C25A—C26A	1.384 (13)
C24—C25	1.389 (13)	C25A—H25A	0.9500
C25—C26	1.370 (12)	C26A—H26A	0.9500
C25—H25	0.9500	O1W—H1W1	0.8401
C26—H26	0.9500	O1W—H1W2	0.8424
Br1A—C12A	1.923 (7)	O1WA—H1W3	0.8399
C1A—O1A	1.231 (10)	O1WA—H1W4	0.8400
C15—O2—C17	116.9 (6)	O1A—C1A—C11A	119.5 (7)
C1—N1—N2	116.9 (6)	N1A—C1A—C11A	116.8 (7)
C1—N1—H1	121.6	C15A—O2A—C17A	117.5 (7)
N2—N1—H1	121.6	C1A—N1A—N2A	118.3 (7)
C2—N2—N1	115.4 (7)	C1A—N1A—H1A	120.8
O1—C1—N1	124.3 (7)	N2A—N1A—H1A	120.8
O1—C1—C11	122.1 (7)	C2A—N2A—N1A	116.4 (7)
N1—C1—C11	113.6 (6)	N2A—C2A—C21A	120.1 (7)
N2—C2—C21	120.6 (8)	N2A—C2A—H2A	120.0
N2—C2—H2	119.7	C21A—C2A—H2A	120.0
C21—C2—H2	119.7	C12A—C11A—C16A	117.0 (7)
C12—C11—C16	119.2 (7)	C12A—C11A—C1A	126.2 (7)
C12—C11—C1	122.1 (7)	C16A—C11A—C1A	116.1 (6)
C16—C11—C1	118.6 (6)	C13A—C12A—C11A	122.4 (7)
C11—C12—C13	121.0 (7)	C13A—C12A—Br1A	115.8 (6)
C11—C12—Br1	122.9 (6)	C11A—C12A—Br1A	121.7 (6)
C13—C12—Br1	116.0 (6)	C12A—C13A—C14A	119.4 (8)
C14—C13—C12	118.2 (7)	C12A—C13A—H13A	120.3
C14—C13—H13	120.9	C14A—C13A—H13A	120.3
C12—C13—H13	120.9	C15A—C14A—C13A	120.4 (8)
C13—C14—C15	121.9 (7)	C15A—C14A—H14A	119.8
C13—C14—H14	119.0	C13A—C14A—H14A	119.8
C15—C14—H14	119.0	O2A—C15A—C14A	116.1 (8)
O2—C15—C16	124.6 (7)	O2A—C15A—C16A	123.4 (7)
O2—C15—C14	116.2 (7)	C14A—C15A—C16A	120.4 (8)
C16—C15—C14	119.2 (7)	C15A—C16A—C11A	120.3 (7)
C15—C16—C11	120.4 (7)	C15A—C16A—H16A	119.9
C15—C16—H16	119.8	C11A—C16A—H16A	119.9
C11—C16—H16	119.8	O2A—C17A—H17D	109.5
O2—C17—H17A	109.5	O2A—C17A—H17E	109.5
O2—C17—H17B	109.5	H17D—C17A—H17E	109.5
H17A—C17—H17B	109.5	O2A—C17A—H17F	109.5

O2—C17—H17C	109.5	H17D—C17A—H17F	109.5
H17A—C17—H17C	109.5	H17E—C17A—H17F	109.5
H17B—C17—H17C	109.5	C22A—C21A—C26A	120.3 (8)
C26—C21—C22	118.6 (7)	C22A—C21A—C2A	119.2 (8)
C26—C21—C2	122.6 (7)	C26A—C21A—C2A	120.5 (7)
C22—C21—C2	118.7 (8)	C21A—C22A—C23A	120.0 (9)
C23—C22—C21	120.4 (8)	C21A—C22A—H22A	120.0
C23—C22—H22	119.8	C23A—C22A—H22A	120.0
C21—C22—H22	119.8	C24A—C23A—C22A	117.8 (9)
C24—C23—C22	117.5 (8)	C24A—C23A—H23A	121.1
C24—C23—H23	121.3	C22A—C23A—H23A	121.1
C22—C23—H23	121.3	F1A—C24A—C23A	118.1 (9)
F1—C24—C23	117.7 (8)	F1A—C24A—C25A	117.9 (9)
F1—C24—C25	117.9 (8)	C23A—C24A—C25A	123.9 (9)
C23—C24—C25	124.3 (8)	C24A—C25A—C26A	118.1 (9)
C26—C25—C24	117.7 (8)	C24A—C25A—H25A	121.0
C26—C25—H25	121.1	C26A—C25A—H25A	121.0
C24—C25—H25	121.1	C25A—C26A—C21A	119.9 (8)
C25—C26—C21	121.5 (7)	C25A—C26A—H26A	120.0
C25—C26—H26	119.3	C21A—C26A—H26A	120.0
C21—C26—H26	119.3	H1W1—O1W—H1W2	85.1
O1A—C1A—N1A	123.6 (8)	H1W3—O1WA—H1W4	85.2
C1—N1—N2—C2	169.4 (7)	O1A—C1A—N1A—N2A	8.9 (12)
N2—N1—C1—O1	2.2 (11)	C11A—C1A—N1A—N2A	−169.1 (6)
N2—N1—C1—C11	−179.1 (6)	C1A—N1A—N2A—C2A	173.4 (7)
N1—N2—C2—C21	−175.2 (6)	N1A—N2A—C2A—C21A	−177.9 (7)
O1—C1—C11—C12	−45.4 (10)	O1A—C1A—C11A—C12A	−130.6 (8)
N1—C1—C11—C12	135.9 (7)	N1A—C1A—C11A—C12A	47.4 (11)
O1—C1—C11—C16	130.4 (8)	O1A—C1A—C11A—C16A	39.6 (10)
N1—C1—C11—C16	−48.4 (9)	N1A—C1A—C11A—C16A	−142.4 (7)
C16—C11—C12—C13	−0.8 (10)	C16A—C11A—C12A—C13A	0.3 (11)
C1—C11—C12—C13	174.9 (7)	C1A—C11A—C12A—C13A	170.5 (7)
C16—C11—C12—Br1	174.6 (5)	C16A—C11A—C12A—Br1A	−177.3 (5)
C1—C11—C12—Br1	−9.7 (10)	C1A—C11A—C12A—Br1A	−7.1 (10)
C11—C12—C13—C14	−0.6 (11)	C11A—C12A—C13A—C14A	0.8 (12)
Br1—C12—C13—C14	−176.3 (6)	Br1A—C12A—C13A—C14A	178.5 (6)
C12—C13—C14—C15	2.8 (12)	C12A—C13A—C14A—C15A	−0.8 (12)
C17—O2—C15—C16	6.1 (12)	C17A—O2A—C15A—C14A	177.3 (7)
C17—O2—C15—C14	−174.6 (8)	C17A—O2A—C15A—C16A	−0.8 (11)
C13—C14—C15—O2	177.2 (7)	C13A—C14A—C15A—O2A	−178.6 (7)
C13—C14—C15—C16	−3.5 (13)	C13A—C14A—C15A—C16A	−0.4 (12)
O2—C15—C16—C11	−178.8 (7)	O2A—C15A—C16A—C11A	179.6 (7)
C14—C15—C16—C11	1.9 (12)	C14A—C15A—C16A—C11A	1.6 (11)
C12—C11—C16—C15	0.2 (11)	C12A—C11A—C16A—C15A	−1.6 (10)
C1—C11—C16—C15	−175.7 (7)	C1A—C11A—C16A—C15A	−172.7 (7)
N2—C2—C21—C26	−4.3 (12)	N2A—C2A—C21A—C22A	−174.8 (8)
N2—C2—C21—C22	171.0 (7)	N2A—C2A—C21A—C26A	5.4 (12)
C26—C21—C22—C23	1.4 (11)	C26A—C21A—C22A—C23A	−1.4 (12)
C2—C21—C22—C23	−174.1 (7)	C2A—C21A—C22A—C23A	178.8 (8)

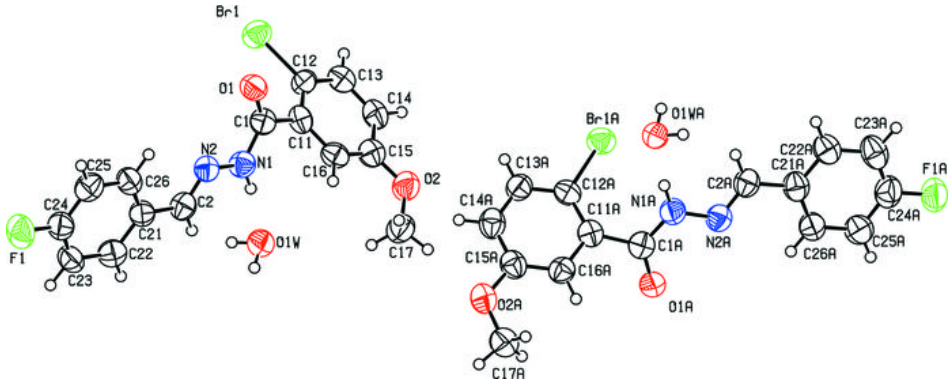
C21—C22—C23—C24	−1.5 (12)	C21A—C22A—C23A—C24A	2.0 (13)
C22—C23—C24—F1	−179.8 (7)	C22A—C23A—C24A—F1A	179.4 (8)
C22—C23—C24—C25	−0.2 (13)	C22A—C23A—C24A—C25A	−3.1 (14)
F1—C24—C25—C26	−178.5 (7)	F1A—C24A—C25A—C26A	−179.1 (8)
C23—C24—C25—C26	1.9 (13)	C23A—C24A—C25A—C26A	3.3 (15)
C24—C25—C26—C21	−1.9 (12)	C24A—C25A—C26A—C21A	−2.4 (13)
C22—C21—C26—C25	0.4 (11)	C22A—C21A—C26A—C25A	1.6 (12)
C2—C21—C26—C25	175.7 (7)	C2A—C21A—C26A—C25A	−178.6 (8)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1W	0.88	2.03	2.901 (9)	172
N1A—H1A···O1WA	0.88	2.01	2.860 (9)	163
O1W—H1W1···O1^{i}	0.84	1.99	2.832 (8)	180
O1W—H1W2···O1A^{ii}	0.84	2.01	2.856 (8)	180
O1WA—H1W3···O1^{iii}	0.84	2.06	2.897 (8)	180
O1WA—H1W4···O1A^{iv}	0.84	1.96	2.802 (7)	180

Symmetry codes: (i) *x*, −*y*+1, *z*−1/2; (ii) *x*+1/2, −*y*+1/2, *z*+1/2; (iii) *x*−1/2, −*y*+3/2, *z*−1/2; (iv) *x*, −*y*+1, *z*+1/2.

Fig. 1


Br1
O1
C1
C12
C13
C14
C11
C15
C16
O2
C17
N2
N1
O1W
C25
C26
C2
C24
C21
F1
C22
C23
Br1A
O1WA
C13A
C12A
C14A
C11A
N1A
C2A
C23A
C22A
C21A
F1A
C15A
C1A
N2A
C24A
C16A
C26A
C25A
O2A
O1A
C17A