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(*Z*)-5-(4-Bromophenyl)-3-{[(3,5-dichlorophenyl)-amino]methylidene}furan-2(3*H*)-one

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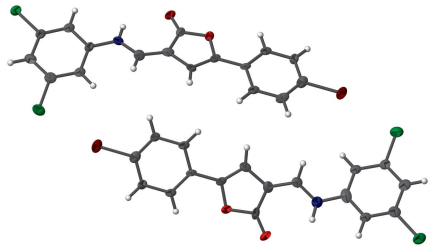
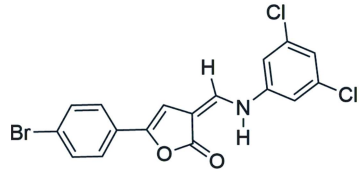
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{17}H_{10}BrCl_2NO_2$, crystallizes in the monoclinic space group *C*2/*c* with a large cell volume of 6207 (3) Å^3. The asymmetric unit of the title compound investigated at 120 K contains two crystallographically independent molecules (Z' = 2). Each molecule demonstrates slight non-planarity in the solid state and a *Z*-configuration for the exocyclic C=C bond. The crystal packing reveals the presence of π-π stacking interactions between the substituted benzene rings [centroid–centroid distances of 3.836 (5) Å, shift distances in the range 1.272–1.843 Å].

3D view



Chemical scheme



Structure description

Push–pull enamines based on furan-2(3*H*)-ones may be of interest for the creation of molecular switches (Osipov *et al.*, 2017). Both crystallographically independent molecules of the title compound are close to planarity and may be aligned together with an r.m.s.d. of 0.297 Å without and 0.561 Å with inversion. Usually, pronounced non-planar molecules differ much more in their alignment with and without inversion. Actually, both molecules are slightly non-planar (Fig. 1) with the 4-bromophenyl substituent rotated about the mean plane of the furanone ring by approximately 2–5° [C18—C17—C6—O1 = 2.1 (12)° while the corresponding C18*A*—C17*A*—C6*A*—O1*A* torsion angle = 5.2 (13)°] . The C4=C7 as well as corresponding C4*A*=C7*A* bonds adopt a *Z* configuration. The benzene ring of the 3,5-dichlorophenyl substituent is also out of the plane of the molecule [the dihedral angles between the mean planes of the furanone and 3,5-dichlorophenyl rings are 33.6 (4) and 14.8 (4)°, respectively, for the two molecules], which is a consequence of the repulsion of hydrogen atoms H16/H16*A* of the aromatic substituent and H7/H7*A* of the enamine fragment with distances H7···H16 = 2.178 Å and H7*A*···H16*A*

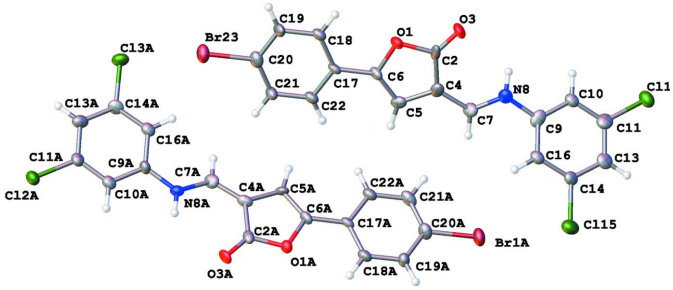


Figure 1
The asymmetric unit of the title compound with the atom labelling and displacement ellipsoids drawn at the 50% probability level (two crystallographically independent molecules are shown).

= 2.063 Å, which is less than the sum of the van der Waals radii (2.38 Å). This is in agreement with the observation that the interatomic distance is slightly larger in the more twisted molecule than in the more planar one.

In contrast to (*Z*)-3-[(3,5-dichloroanilino)methylidene]-5-(*p*-tolyl)furan-2(3*H*)-one (Grinev *et al.*, 2018), which demonstrated only intramolecular hydrogen bond, in crystal of the title molecule there are not only intramolecular, but also intermolecular hydrogen bonds (Table 1, Fig. 2). They are relatively weak but result in dimer formation in the crystal packing. The H8···O3 distance in both molecules is significantly longer than the corresponding distance in the *p*-tolyl-substituted analogue [2.18 (2) Å]. This may be explained by the presence of the bulky electronegative bromine atom as a substituent on the benzene ring instead of a methyl group. The two molecules in the asymmetric unit are oriented in a head-to-tail fashion, the bromine atom of one molecule becoming relatively close to the H atoms of CH fragments of both the aromatic and enamine moieties in the neighbouring second crystallographically independent molecule [H···Br interatomic distances are in the range 3.26—3.82 Å].

The interplanar distances between identically oriented molecules in the *p*-tolyl substituted analogue are larger than 7 Å, excluding non-covalent interactions such as π–π stacking. In contrast to this, in the crystal of the title molecule parallel-displaced π–π stacking interactions are present for both crystallographically independent molecules (Fig. 3). The intercentroid distances between the 3,5-dichlorophenyl as well as the 4-bromophenyl rings are 3.836 (5) Å for both molecules, with shift distances of 1.272 and 1.665 Å for the first molecule and 1.539 and 1.843 Å for the second molecule.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N8—H8···O3	0.87 (13)	2.49 (11)	3.013 (11)	119 (10)
N8—H8···O3^{i}	0.87 (13)	2.26 (13)	3.059 (15)	153 (11)
N8*A*—H8*A*···O3*A*	0.88	2.44	3.050 (11)	126
N8*A*—H8*A*···O3*A*^{ii}	0.88	2.32	3.142 (12)	156

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{10}BrCl_2NO_2$
M_r	411.07
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	120
a, *b*, *c* (Å)	55.051 (17), 3.8355 (11), 35.979 (12)
β (°)	125.214 (6)
V (Å^3)	6207 (3)
Z	16
Radiation type	Mo *K*α
μ (mm^{-1})	3.00
Crystal size (mm)	0.6 × 0.1 × 0.1
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.170, 0.337
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	17633, 6123, 3647
R_{int}	0.136
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.092, 0.220, 1.06
No. of reflections	6123
No. of parameters	359
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.17, −1.30

Computer programs: *APEX2* (Bruker, 2013), *SAINT* (Bruker, 2013), *SHELXT* (Sheldrick, 2015*b*), *SHELXL* (Sheldrick, 2015*a*), *OLEX2* (Dolomanov *et al.*, 2009).

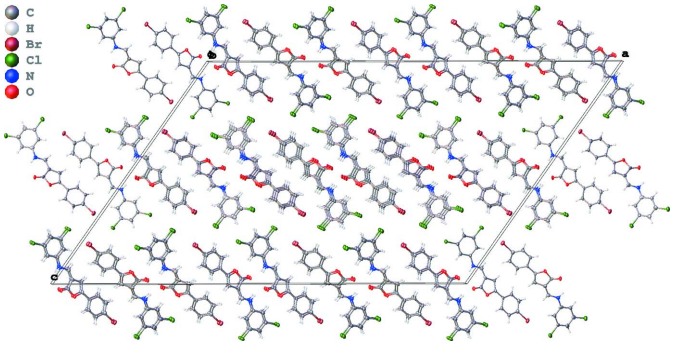


Figure 2
The crystal packing of the title compound, viewed along the *b* axis.

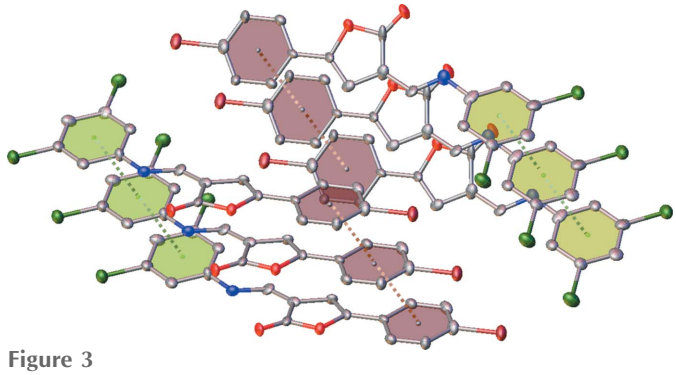


Figure 3
π–π stacking interactions between the dichloro (green) and bromo (brown) substituted aromatic rings of the title compound.

Synthesis and crystallization

The synthesis of the title compound was performed according to the method described by Osipov *et al.* (2017) and Grinev *et al.* (2018). Briefly, about 7 ml of benzene, 1.78 g of (12.02 mmol) triethyl orthoformate, 0.40 g (1.67 mmol) of 5-(4-bromophenyl)furan-2(3*H*)-one and 0.27 g (1.67 mmol) of 3,5-dichloroaniline were placed into a round-bottom flask equipped with a Liebig reflux condenser, and the reaction mixture was refluxed for 2 h. The precipitate of 3-[(3,5-dichloroanilino)methylidene]-5-(4-bromophenyl)furan-2(3*H*)-one was filtered off, washed with benzene and then with chloroform, dried, and recrystallized from DMF. Yield 0.51 g (75%), yellow crystals. A single crystal suitable for X-ray analysis was obtained by slow cooling of a saturated solution of the title compound in benzene.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The relatively high R_{int} and R values as well as the low C—C bond precision are due to poor crystal quality because of probable twinning or clustering.

Funding information

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full crystallographic data

IUCrData (2020). **5**, x200937 [https://doi.org/10.1107/S2414314620009372]

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(*Z*)-5-(4-Bromophenyl)-3-{[(3,5-dichlorophenyl)amino]methylidene}furan-2(3*H*)-one

Crystal data

$C_{17}H_{10}BrCl_2NO_2$
M_r = 411.07
Monoclinic, *C*2/*c*
a = 55.051 (17) Å
b = 3.8355 (11) Å
c = 35.979 (12) Å
β = 125.214 (6)°
V = 6207 (3) Å^3
Z = 16
F(000) = 3264
D_x = 1.760 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 1746 reflections
θ = 2.8–30.7°
μ = 3.00 mm^{-1}
T = 120 K
Needle, metallic orangish yellow
0.6 × 0.1 × 0.1 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.170, T_{max} = 0.337
17633 measured reflections
6123 independent reflections
3647 reflections with $I > 2\sigma(I)$
R_{int} = 0.136
θ_{max} = 26.0°, θ_{min} = 0.9°
h = −66→65
k = −4→4
l = −41→44

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.092
$wR(F^2)$ = 0.220
S = 1.06
6123 reflections
359 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0747P)^2 + 74.2699P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.17 e Å^{-3}
$\Delta\rho_{min}$ = −1.30 e Å^{-3}
Extinction correction: SHELXL2016/6 (Sheldrick 2016), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00133 (13)

Special details

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

Refinement. The structure was solved by the internal phasing method and refined by the least-squares method in the anisotropic full-matrix approximation in accordance with F^2_{hkl}. Aromatic and amine H atoms refined with riding coordinates: C5(H5), C7(H7), C10(H10), C13(H13), C16(H16), C18(H18), C19(H19), C21(H21), C22(H22), N8A(H8A), C5A(H5A), C7A(H7A), C10A(H10A), C13A(H13A), C16A(H16A), C18A(H18A), C19A(H19A), C21A(H21A), C22A(H22A). Other H atoms were placed in calculated positions and refined geometrically using a riding model, with fixed thermal parameters U_{iso}(H) = 1.2U_{iso}(C). In addition, the following restraints and constraints were applied: U_{anis}(C6A) = U_{anis}(C6); U_{anis}(C16A) = U_{anis}(C10A) = U_{anis}(C16) = U_{anis}(C10); U_{anis}(C7A) = U_{anis}(C7); U_{anis}(C20) = U_{anis}(C20A); U_{anis}(C18) = U_{anis}(C22) = U_{anis}(C22A) = U_{anis}(C18A).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br23	0.48087 (2)	1.1512 (3)	0.68139 (4)	0.0309 (3)
Cl1	0.14526 (6)	−0.0626 (7)	0.33686 (10)	0.0340 (7)
Cl15	0.20333 (6)	0.2610 (8)	0.26102 (10)	0.0399 (7)
O1	0.32912 (15)	0.9028 (18)	0.5536 (2)	0.0275 (17)
O3	0.27944 (15)	0.865 (2)	0.5197 (2)	0.0338 (18)
N8	0.2499 (2)	0.467 (2)	0.4316 (3)	0.029 (2)
H8	0.247 (2)	0.48 (3)	0.453 (4)	0.035*
C2	0.3001 (2)	0.796 (3)	0.5180 (3)	0.029 (3)
C4	0.3023 (2)	0.614 (3)	0.4849 (3)	0.026 (2)
C5	0.3334 (2)	0.613 (2)	0.5017 (3)	0.022 (2)
H5	0.341698	0.512110	0.487258	0.027*
C6	0.3483 (2)	0.784 (2)	0.5422 (3)	0.0199 (15)
C7	0.2781 (2)	0.477 (2)	0.4441 (3)	0.0236 (16)
H7	0.281853	0.381941	0.423527	0.028*
C9	0.2261 (2)	0.345 (2)	0.3887 (3)	0.027 (2)
C10	0.2008 (2)	0.216 (2)	0.3845 (3)	0.0223 (11)
H10	0.200137	0.208946	0.410275	0.027*
C11	0.1767 (2)	0.097 (3)	0.3425 (4)	0.030 (3)
C13	0.1772 (2)	0.103 (2)	0.3038 (4)	0.027 (2)
H13	0.160948	0.017098	0.275366	0.033*
C14	0.2023 (2)	0.237 (3)	0.3086 (3)	0.023 (2)
C16	0.2270 (2)	0.363 (2)	0.3509 (3)	0.0223 (11)
H16	0.243784	0.458461	0.353446	0.027*
C17	0.3802 (2)	0.867 (2)	0.5759 (3)	0.0177 (14)
C18	0.3893 (2)	1.041 (2)	0.6163 (3)	0.0216 (11)
H18	0.375138	1.102737	0.622215	0.026*
C19	0.4198 (2)	1.123 (3)	0.6482 (3)	0.026 (2)
H19	0.426177	1.240856	0.675631	0.031*
C20	0.4401 (2)	1.031 (3)	0.6389 (4)	0.0270 (17)
C21	0.4313 (2)	0.856 (2)	0.5991 (3)	0.022 (2)
H21	0.445521	0.793918	0.593232	0.026*
C22	0.4007 (2)	0.771 (2)	0.5672 (3)	0.0216 (11)
H22	0.394359	0.647951	0.540107	0.026*
Br1A	0.28136 (2)	−0.0323 (3)	0.32665 (4)	0.0321 (3)
Cl2A	0.62033 (6)	1.0959 (7)	0.68860 (9)	0.0291 (6)
Cl3A	0.55514 (6)	0.6764 (7)	0.74909 (9)	0.0316 (6)
O1A	0.42611 (14)	0.5740 (16)	0.4455 (2)	0.0212 (15)

O3A	0.47142 (15)	0.8240 (17)	0.4752 (2)	0.0252 (16)
N8A	0.51085 (17)	0.717 (2)	0.5780 (3)	0.0213 (19)
H8A	0.511802	0.804501	0.556191	0.026*
C2A	0.4552 (2)	0.657 (2)	0.4813 (3)	0.020 (2)
C4A	0.4599 (2)	0.516 (2)	0.5227 (3)	0.020 (2)
C5A	0.4325 (2)	0.348 (2)	0.5088 (3)	0.019 (2)
H5A	0.428873	0.227865	0.528260	0.023*
C6A	0.4130 (2)	0.391 (2)	0.4639 (3)	0.0199 (15)
C7A	0.4858 (2)	0.543 (2)	0.5660 (3)	0.0236 (16)
H7A	0.486133	0.428201	0.589717	0.028*
C9A	0.5354 (2)	0.766 (3)	0.6235 (3)	0.021 (2)
C10A	0.5626 (2)	0.887 (2)	0.6326 (3)	0.0223 (11)
H10A	0.564462	0.929666	0.608312	0.027*
C11A	0.5865 (2)	0.944 (3)	0.6769 (3)	0.023 (2)
C13A	0.5849 (2)	0.882 (3)	0.7136 (3)	0.026 (2)
H13A	0.601535	0.923528	0.743953	0.031*
C14A	0.5582 (2)	0.758 (2)	0.7044 (3)	0.023 (2)
C16A	0.5334 (2)	0.706 (2)	0.6597 (3)	0.0223 (11)
H16A	0.515162	0.630053	0.654176	0.027*
C17A	0.3814 (2)	0.289 (2)	0.4306 (3)	0.0177 (14)
C18A	0.3672 (2)	0.348 (2)	0.3842 (3)	0.0216 (11)
H18A	0.377945	0.448435	0.373662	0.026*
C19A	0.3374 (2)	0.260 (2)	0.3532 (3)	0.022 (2)
H19A	0.327384	0.310506	0.321743	0.026*
C20A	0.3224 (2)	0.097 (3)	0.3692 (4)	0.0270 (17)
C21A	0.3361 (2)	0.038 (2)	0.4148 (4)	0.027 (2)
H21A	0.325416	−0.068515	0.425210	0.032*
C22A	0.3657 (2)	0.134 (2)	0.4456 (3)	0.0216 (11)
H22A	0.375192	0.094617	0.477202	0.026*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br23	0.0239 (6)	0.0206 (6)	0.0413 (7)	−0.0040 (4)	0.0149 (5)	−0.0004 (5)
Cl1	0.0339 (15)	0.0288 (15)	0.0475 (17)	−0.0050 (12)	0.0282 (14)	−0.0047 (12)
Cl15	0.0389 (16)	0.0507 (19)	0.0385 (17)	0.0045 (14)	0.0273 (14)	0.0024 (14)
O1	0.025 (4)	0.036 (4)	0.030 (4)	−0.010 (3)	0.021 (3)	−0.007 (3)
O3	0.020 (4)	0.047 (5)	0.040 (5)	−0.004 (3)	0.021 (4)	−0.005 (4)
N8	0.030 (5)	0.031 (5)	0.027 (5)	−0.004 (4)	0.017 (4)	0.005 (4)
C2	0.018 (5)	0.041 (7)	0.020 (5)	−0.014 (5)	0.007 (4)	0.007 (5)
C4	0.029 (6)	0.026 (6)	0.022 (5)	−0.007 (5)	0.014 (5)	0.006 (4)
C5	0.037 (6)	0.013 (5)	0.026 (6)	−0.004 (4)	0.022 (5)	0.001 (4)
C6	0.023 (4)	0.011 (3)	0.033 (4)	−0.004 (3)	0.021 (3)	−0.003 (3)
C7	0.032 (4)	0.009 (4)	0.032 (4)	−0.004 (3)	0.020 (3)	−0.004 (3)
C9	0.033 (6)	0.008 (5)	0.028 (6)	0.005 (4)	0.010 (5)	0.003 (4)
C10	0.026 (3)	0.013 (3)	0.032 (3)	0.002 (2)	0.020 (2)	0.001 (2)
C11	0.035 (6)	0.022 (6)	0.037 (6)	0.007 (5)	0.022 (5)	−0.004 (5)
C13	0.029 (6)	0.017 (5)	0.038 (6)	0.010 (4)	0.020 (5)	0.005 (5)

C14	0.022 (5)	0.019 (5)	0.027 (6)	0.007 (4)	0.014 (5)	0.005 (4)
C16	0.026 (3)	0.013 (3)	0.032 (3)	0.002 (2)	0.020 (2)	0.001 (2)
C17	0.023 (3)	0.012 (3)	0.028 (4)	−0.001 (3)	0.021 (3)	−0.001 (3)
C18	0.027 (3)	0.017 (3)	0.030 (3)	−0.001 (2)	0.022 (2)	0.000 (2)
C19	0.023 (5)	0.028 (6)	0.030 (6)	−0.012 (4)	0.018 (5)	−0.008 (5)
C20	0.027 (4)	0.018 (4)	0.037 (4)	−0.003 (3)	0.019 (3)	0.004 (3)
C21	0.021 (5)	0.017 (5)	0.030 (6)	−0.004 (4)	0.017 (5)	−0.006 (4)
C22	0.027 (3)	0.017 (3)	0.030 (3)	−0.001 (2)	0.022 (2)	0.000 (2)
Br1A	0.0217 (5)	0.0293 (7)	0.0428 (7)	−0.0055 (5)	0.0171 (5)	−0.0041 (5)
Cl2A	0.0262 (13)	0.0328 (15)	0.0328 (14)	−0.0045 (11)	0.0196 (12)	−0.0021 (12)
Cl3A	0.0424 (16)	0.0316 (15)	0.0262 (14)	−0.0033 (12)	0.0228 (13)	0.0014 (11)
O1A	0.020 (3)	0.019 (4)	0.024 (4)	−0.004 (3)	0.013 (3)	−0.001 (3)
O3A	0.025 (4)	0.024 (4)	0.036 (4)	−0.011 (3)	0.023 (3)	−0.004 (3)
N8A	0.028 (5)	0.020 (5)	0.021 (4)	−0.001 (4)	0.017 (4)	0.000 (3)
C2A	0.019 (5)	0.015 (5)	0.030 (6)	−0.003 (4)	0.015 (4)	−0.009 (4)
C4A	0.034 (6)	0.008 (5)	0.031 (6)	0.001 (4)	0.025 (5)	0.002 (4)
C5A	0.027 (5)	0.020 (5)	0.016 (5)	−0.003 (4)	0.016 (4)	0.000 (4)
C6A	0.023 (4)	0.011 (3)	0.033 (4)	−0.004 (3)	0.021 (3)	−0.003 (3)
C7A	0.032 (4)	0.009 (4)	0.032 (4)	−0.004 (3)	0.020 (3)	−0.004 (3)
C9A	0.018 (5)	0.022 (6)	0.025 (5)	−0.003 (4)	0.013 (4)	0.002 (4)
C10A	0.026 (3)	0.013 (3)	0.032 (3)	0.002 (2)	0.020 (2)	0.001 (2)
C11A	0.022 (5)	0.021 (6)	0.029 (6)	0.002 (4)	0.016 (5)	−0.002 (4)
C13A	0.033 (6)	0.023 (6)	0.026 (6)	0.009 (5)	0.019 (5)	0.006 (4)
C14A	0.034 (6)	0.008 (5)	0.031 (6)	0.002 (4)	0.021 (5)	0.006 (4)
C16A	0.026 (3)	0.013 (3)	0.032 (3)	0.002 (2)	0.020 (2)	0.001 (2)
C17A	0.023 (3)	0.012 (3)	0.028 (4)	−0.001 (3)	0.021 (3)	−0.001 (3)
C18A	0.027 (3)	0.017 (3)	0.030 (3)	−0.001 (2)	0.022 (2)	0.000 (2)
C19A	0.019 (5)	0.020 (5)	0.024 (5)	−0.002 (4)	0.011 (4)	0.002 (4)
C20A	0.027 (4)	0.018 (4)	0.037 (4)	−0.003 (3)	0.019 (3)	0.004 (3)
C21A	0.032 (6)	0.014 (5)	0.046 (7)	0.002 (4)	0.029 (6)	0.006 (5)
C22A	0.027 (3)	0.017 (3)	0.030 (3)	−0.001 (2)	0.022 (2)	0.000 (2)

Geometric parameters (Å, º)

Br23—C20	1.907 (10)	Br1A—C20A	1.926 (10)
Cl1—C11	1.732 (11)	Cl2A—C11A	1.756 (10)
Cl15—C14	1.746 (10)	Cl3A—C14A	1.740 (10)
O1—C2	1.412 (11)	O1A—C2A	1.395 (11)
O1—C6	1.409 (11)	O1A—C6A	1.416 (11)
O3—C2	1.204 (12)	O3A—C2A	1.220 (11)
N8—H8	0.86 (10)	N8A—H8A	0.8800
N8—C7	1.342 (13)	N8A—C7A	1.361 (12)
N8—C9	1.411 (13)	N8A—C9A	1.412 (12)
C2—C4	1.445 (15)	C2A—C4A	1.457 (13)
C4—C5	1.448 (14)	C4A—C5A	1.440 (13)
C4—C7	1.397 (14)	C4A—C7A	1.382 (14)
C5—H5	0.9500	C5A—H5A	0.9500
C5—C6	1.360 (13)	C5A—C6A	1.339 (13)

C6—C17	1.486 (13)	C6A—C17A	1.487 (13)
C7—H7	0.9500	C7A—H7A	0.9500
C9—C10	1.400 (14)	C9A—C10A	1.417 (13)
C9—C16	1.389 (14)	C9A—C16A	1.388 (13)
C10—H10	0.9500	C10A—H10A	0.9500
C10—C11	1.393 (14)	C10A—C11A	1.378 (14)
C11—C13	1.410 (15)	C11A—C13A	1.392 (14)
C13—H13	0.9500	C13A—H13A	0.9500
C13—C14	1.390 (14)	C13A—C14A	1.390 (14)
C14—C16	1.418 (14)	C14A—C16A	1.400 (14)
C16—H16	0.9500	C16A—H16A	0.9500
C17—C18	1.403 (13)	C17A—C18A	1.392 (13)
C17—C22	1.380 (12)	C17A—C22A	1.391 (13)
C18—H18	0.9500	C18A—H18A	0.9500
C18—C19	1.417 (13)	C18A—C19A	1.394 (13)
C19—H19	0.9500	C19A—H19A	0.9500
C19—C20	1.385 (14)	C19A—C20A	1.393 (14)
C20—C21	1.391 (14)	C20A—C21A	1.375 (14)
C21—H21	0.9500	C21A—H21A	0.9500
C21—C22	1.427 (13)	C21A—C22A	1.392 (14)
C22—H22	0.9500	C22A—H22A	0.9500

C6—O1—C2	106.7 (8)	C2A—O1A—C6A	107.7 (7)
C7—N8—H8	118 (8)	C7A—N8A—H8A	118.4
C7—N8—C9	122.8 (9)	C7A—N8A—C9A	123.3 (8)
C9—N8—H8	117 (8)	C9A—N8A—H8A	118.4
O1—C2—C4	107.4 (9)	O1A—C2A—C4A	107.1 (8)
O3—C2—O1	119.9 (10)	O3A—C2A—O1A	121.4 (9)
O3—C2—C4	132.6 (9)	O3A—C2A—C4A	131.5 (9)
C2—C4—C5	107.3 (9)	C5A—C4A—C2A	106.1 (8)
C7—C4—C2	124.4 (10)	C7A—C4A—C2A	125.8 (9)
C7—C4—C5	128.3 (10)	C7A—C4A—C5A	128.1 (9)
C4—C5—H5	126.8	C4A—C5A—H5A	125.8
C6—C5—C4	106.4 (9)	C6A—C5A—C4A	108.3 (8)
C6—C5—H5	126.8	C6A—C5A—H5A	125.8
O1—C6—C17	115.2 (8)	O1A—C6A—C17A	115.4 (8)
C5—C6—O1	112.2 (8)	C5A—C6A—O1A	110.8 (8)
C5—C6—C17	132.5 (9)	C5A—C6A—C17A	133.8 (9)
N8—C7—C4	125.5 (10)	N8A—C7A—C4A	126.2 (9)
N8—C7—H7	117.2	N8A—C7A—H7A	116.9
C4—C7—H7	117.2	C4A—C7A—H7A	116.9
C10—C9—N8	118.4 (9)	N8A—C9A—C10A	119.3 (8)
C16—C9—N8	121.2 (10)	C16A—C9A—N8A	121.8 (9)
C16—C9—C10	120.3 (9)	C16A—C9A—C10A	118.9 (9)
C9—C10—H10	120.0	C9A—C10A—H10A	120.2
C11—C10—C9	120.0 (10)	C11A—C10A—C9A	119.7 (9)
C11—C10—H10	120.0	C11A—C10A—H10A	120.2
C10—C11—Cl1	120.5 (8)	C10A—C11A—Cl2A	120.0 (8)

C10—C11—C13	121.0 (10)	C10A—C11A—C13A	122.1 (10)
C13—C11—Cl1	118.6 (8)	C13A—C11A—Cl2A	117.9 (8)
C11—C13—H13	121.0	C11A—C13A—H13A	121.0
C14—C13—C11	118.1 (10)	C14A—C13A—C11A	117.9 (10)
C14—C13—H13	121.0	C14A—C13A—H13A	121.0
C13—C14—Cl15	119.3 (8)	C13A—C14A—Cl3A	119.7 (8)
C13—C14—C16	121.7 (10)	C13A—C14A—C16A	121.3 (9)
C16—C14—Cl15	119.0 (8)	C16A—C14A—Cl3A	119.0 (8)
C9—C16—C14	118.9 (9)	C9A—C16A—C14A	120.1 (9)
C9—C16—H16	120.6	C9A—C16A—H16A	119.9
C14—C16—H16	120.6	C14A—C16A—H16A	119.9
C18—C17—C6	119.6 (8)	C18A—C17A—C6A	120.7 (8)
C22—C17—C6	119.6 (8)	C22A—C17A—C6A	119.9 (9)
C22—C17—C18	120.8 (9)	C22A—C17A—C18A	119.3 (9)
C17—C18—H18	120.2	C17A—C18A—H18A	119.7
C17—C18—C19	119.5 (9)	C17A—C18A—C19A	120.5 (9)
C19—C18—H18	120.2	C19A—C18A—H18A	119.7
C18—C19—H19	120.3	C18A—C19A—H19A	120.6
C20—C19—C18	119.3 (9)	C20A—C19A—C18A	118.7 (9)
C20—C19—H19	120.3	C20A—C19A—H19A	120.6
C19—C20—Br23	119.0 (8)	C19A—C20A—Br1A	119.3 (8)
C19—C20—C21	121.5 (9)	C21A—C20A—Br1A	119.2 (8)
C21—C20—Br23	119.4 (8)	C21A—C20A—C19A	121.5 (10)
C20—C21—H21	120.5	C20A—C21A—H21A	120.4
C20—C21—C22	119.0 (9)	C20A—C21A—C22A	119.3 (9)
C22—C21—H21	120.5	C22A—C21A—H21A	120.4
C17—C22—C21	119.8 (9)	C17A—C22A—C21A	120.6 (9)
C17—C22—H22	120.1	C17A—C22A—H22A	119.7
C21—C22—H22	120.1	C21A—C22A—H22A	119.7

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N8—H8···O3	0.87 (13)	2.49 (11)	3.013 (11)	119 (10)
N8—H8···O3^{i}	0.87 (13)	2.26 (13)	3.059 (15)	153 (11)
N8*A*—H8*A*···O3*A*	0.88	2.44	3.050 (11)	126
N8*A*—H8*A*···O3*A*^{ii}	0.88	2.32	3.142 (12)	156

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