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(1*R*,2*R*)-8-Bromo-1-[(*E*)-2-(4-bromophenyl)ethenyl]-2-nitro-1,2-dihydronaphtho[2,1-*b*]furan

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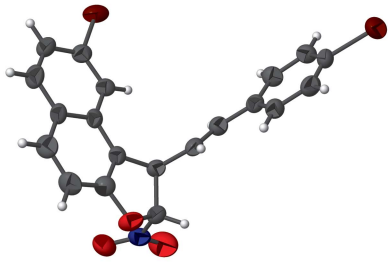
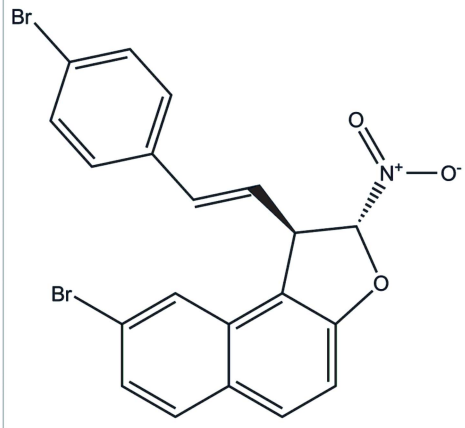
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{20}H_{13}Br_2NO_3$, contains the dihydrobenzofuran moiety, which is present in the physiologically active components of many medicinal plants. The naphthyl ring system is nearly perpendicular to the phenyl ring, while the mean plane of the double bond is almost coplanar with the phenyl ring [dihedral angles of 79.14 (3) and 13.56 (1)°, respectively]. The nitro group and bromobenzene alkenyl group are *trans* to one another on opposite sides of the furan ring. There are two stereogenic centres, and each has the *R* configuration. In the crystal, there are very weak intermolecular C—H···Br interactions.

3D view



Chemical scheme



Structure description

The title compound has a dihydrobenzofuran skeleton, which is the effective physiologically active component in many medicinal plants(Ohkawa *et al.* 1997; Snyder *et al.*, 2011). The molecular structure of the title compound is shown in Fig. 1. The five-membered ring involving C9/C10/C11/C12/O1adopts an envelope conformation and is characterized by torsion angle values of −140.9 (6) and −18.7 (7)° for C13—C12—C11—N1 and C10—C12—C11—O1, respectively. The naphthyl group is nearly perpendicular to the phenyl ring, while the mean plane through the double bond and its attached substituents is almost coplanar with the phenyl ring [dihedral angles of 79.14 (3) and 13.56 (1)°, respectively]. Furthermore, the C12—C10—C9 and C10—C9—O1 bond angles are 109.7 (5)° and 112.0 (5)°, respectively. The nitro group and bromobenzene alkenyl group are mutually *trans* to one another, on opposite sides of the furan ring. The molecule possesses two stereogenic centres, C11 and C12, and both have the *R* configuration. In the crystal, there are very weak intermolecular C—H···Br interactions present (Fig. 2 and Table 1).

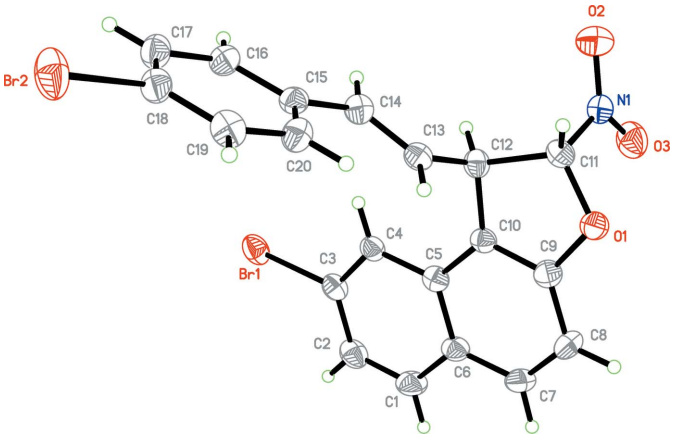


Figure 1
The structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

Synthesis and crystallization

The title compound was synthesized from 1-bromo-4-[(1*E*,3*Z*)-4-bromo-4-nitrobuta-1,3-dienyl]benzene and 7-bromonaphthalen-2-ol (Jarava-Barrera *et al.*, 2013; Pan *et al.*, 2013). To a solution of the chiral amine catalyst 3-(benzylamino)-4-({*R*-(6-methoxyquinolin-4-yl)[(1*S*,2*R*,4*S*,5*R*)-5-vinylquinuclidin-2-yl]methyl}amino)cyclobut-3-ene-1,2-dione (5.08 mg, 5 mol%) and 1-bromo-4-[(1*E*,3*Z*)-4-bromo-4-nitrobuta-1,3-dienyl]benzene (66.2 mg, 0.2 mmol) in chloroform (6 ml) was added sequentially a solution of 7-bromonaphthalen-2-ol (88.8 mg, 0. 4 mmol) and potassium carbonate (27.6 mg, 0.2 mmol) in water (3.0 ml) with vigorous stirring. The reaction was monitored by TLC. After completion of the reaction, the mixture was extracted with DCM (3 × 5 mL), washed with water, dried and concentrated. The residue was purified by flash chromatography to give a white solid. Single crystals were obtained by slow evaporation of a solution in ethyl acetate.

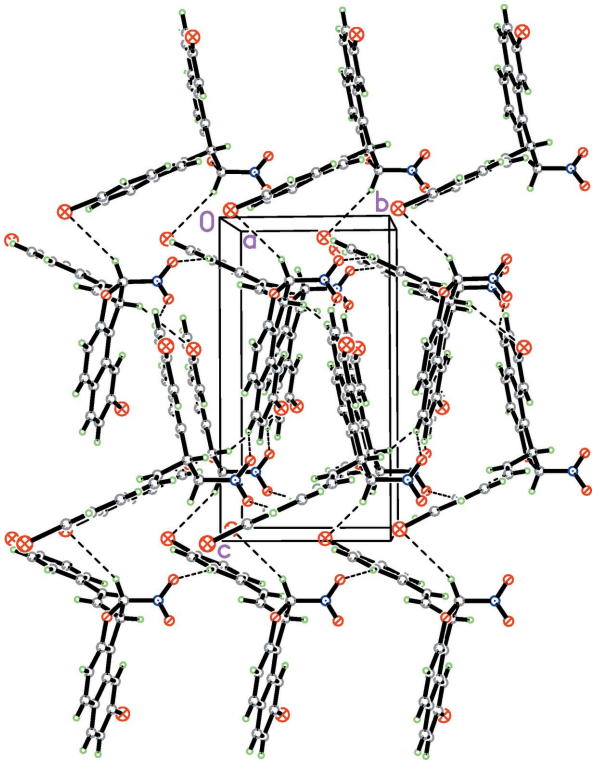


Figure 2
The crystal packing of the title compound viewed down the crystallographic *a* axis. C—H···Br interactions are shown with dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···Br1^{i}	0.98	3.04	3.786 (7)	134
C11—H11···Br2^{ii}	0.98	3.04	3.942 (7)	154

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{13}Br_2NO_3$
M_r	475.13
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	293
a, *b*, *c* (Å)	9.4796 (15), 7.1550 (12), 13.651 (2)
β (°)	90.968 (4)
V (Å^3)	925.8 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	4.40
Crystal size (mm)	0.20 × 0.14 × 0.10
Data collection	
Diffractometer	CCD area detector
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.308, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5364, 3269, 2731
R_{int}	0.032
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.606
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.041, 0.093, 0.97
No. of reflections	3269
No. of parameters	235
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.46, −0.33
Absolute structure	Flack *x* determined using 998 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.020 (14)

Computer programs: *SMART* and *SAINT* (Bruker, 2013), *SHELXTL* (Sheldrick, 2008) and *SHELXL2013* (Sheldrick, 2015).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

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Crystal data

$C_{20}H_{13}Br_2NO_3$
M_r = 475.13
Monoclinic, $P2_1$
a = 9.4796 (15) Å
b = 7.1550 (12) Å
c = 13.651 (2) Å
β = 90.968 (4)°
V = 925.8 (3) Å^3
Z = 2
$F(000)$ = 468
D_x = 1.704 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1587 reflections
θ = 4.3–42.0°
μ = 4.40 mm^{-1}
T = 293 K
Prismatic, colorless
0.20 × 0.14 × 0.10 mm

Data collection

CCD area detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min} = 0.308, T_{max} = 0.746
5364 measured reflections
3269 independent reflections
2731 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.5°, θ_{min} = 2.2°
h = −11→10
k = −8→8
l = −16→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.093
S = 0.97
3269 reflections
235 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0287P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Absolute structure: Flack x determined using 998 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.020 (14)

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}
Br1	0.34554 (7)	0.79114 (10)	0.40480 (6)	0.0566 (2)
Br2	0.15172 (9)	0.04620 (14)	0.97004 (6)	0.0734 (3)
N1	0.8641 (6)	1.1062 (9)	0.8084 (5)	0.0505 (17)
O1	0.9655 (4)	0.8187 (7)	0.7633 (4)	0.0494 (13)
O2	0.7964 (6)	1.1871 (10)	0.8702 (5)	0.087 (2)
O3	0.9374 (6)	1.1853 (8)	0.7504 (5)	0.0684 (16)
C1	0.7710 (8)	0.6837 (11)	0.3851 (6)	0.055 (2)
H1	0.8322	0.6469	0.3362	0.066*
C2	0.6321 (8)	0.7072 (11)	0.3625 (6)	0.0526 (19)
H2	0.5983	0.6878	0.2990	0.063*
C3	0.5398 (7)	0.7617 (10)	0.4377 (5)	0.0443 (16)
C4	0.5847 (6)	0.7928 (11)	0.5314 (5)	0.0393 (14)
H4	0.5215	0.8284	0.5793	0.047*
C5	0.7298 (6)	0.7699 (10)	0.5543 (5)	0.0377 (14)
C6	0.8249 (7)	0.7137 (10)	0.4806 (5)	0.0428 (16)
C7	0.9695 (8)	0.6893 (11)	0.5051 (6)	0.053 (2)
H7	1.0307	0.6499	0.4567	0.064*
C8	1.0220 (7)	0.7214 (11)	0.5972 (6)	0.0530 (19)
H8	1.1172	0.7060	0.6126	0.064*
C9	0.9269 (6)	0.7780 (11)	0.6662 (5)	0.0431 (15)
C10	0.7863 (6)	0.8018 (10)	0.6493 (4)	0.0364 (13)
C11	0.8482 (7)	0.8968 (10)	0.8071 (5)	0.0429 (17)
H11	0.8380	0.8485	0.8738	0.052*
C12	0.7149 (6)	0.8505 (9)	0.7436 (5)	0.0406 (16)
H12	0.6551	0.9613	0.7352	0.049*
C13	0.6332 (7)	0.6920 (10)	0.7872 (5)	0.0393 (15)
H13	0.6821	0.5818	0.8000	0.047*
C14	0.4995 (7)	0.6957 (10)	0.8085 (5)	0.0435 (16)
H14	0.4525	0.8081	0.7975	0.052*
C15	0.4160 (6)	0.5434 (11)	0.8476 (4)	0.0401 (14)
C16	0.2702 (6)	0.5486 (12)	0.8480 (5)	0.0486 (16)
H16	0.2250	0.6550	0.8241	0.058*
C17	0.1888 (7)	0.4044 (11)	0.8820 (6)	0.0510 (19)
H17	0.0909	0.4115	0.8804	0.061*
C18	0.2568 (7)	0.2504 (11)	0.9182 (5)	0.049 (2)
C19	0.4018 (7)	0.2356 (10)	0.9217 (5)	0.0497 (18)
H19	0.4455	0.1295	0.9472	0.060*
C20	0.4800 (7)	0.3820 (11)	0.8865 (5)	0.0457 (17)
H20	0.5779	0.3740	0.8886	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0575 (4)	0.0457 (4)	0.0659 (5)	0.0035 (4)	−0.0212 (3)	−0.0074 (4)
Br2	0.0799 (6)	0.0793 (6)	0.0607 (5)	−0.0367 (5)	−0.0063 (4)	0.0148 (5)

N1	0.038 (3)	0.058 (4)	0.055 (4)	0.000 (3)	−0.003 (3)	−0.012 (3)
O1	0.041 (2)	0.058 (4)	0.049 (3)	0.013 (2)	−0.011 (2)	−0.012 (3)
O2	0.074 (4)	0.085 (5)	0.102 (6)	0.006 (4)	0.015 (4)	−0.040 (4)
O3	0.069 (4)	0.061 (4)	0.075 (4)	−0.019 (3)	0.001 (3)	0.000 (3)
C1	0.064 (5)	0.062 (5)	0.041 (5)	−0.001 (4)	0.018 (4)	−0.009 (4)
C2	0.066 (5)	0.056 (5)	0.036 (4)	−0.010 (4)	−0.001 (4)	−0.002 (4)
C3	0.052 (4)	0.036 (4)	0.045 (4)	−0.003 (3)	−0.004 (3)	−0.001 (3)
C4	0.042 (3)	0.033 (3)	0.043 (4)	−0.001 (4)	−0.001 (3)	−0.003 (4)
C5	0.047 (3)	0.029 (3)	0.037 (4)	−0.003 (3)	0.004 (3)	0.006 (3)
C6	0.050 (4)	0.043 (4)	0.035 (4)	−0.003 (3)	0.011 (3)	−0.002 (3)
C7	0.052 (4)	0.054 (5)	0.054 (5)	0.004 (4)	0.014 (4)	−0.013 (4)
C8	0.034 (4)	0.060 (5)	0.065 (5)	0.007 (3)	0.007 (3)	−0.004 (4)
C9	0.044 (3)	0.037 (3)	0.048 (4)	0.005 (3)	−0.002 (3)	0.001 (4)
C10	0.042 (3)	0.030 (3)	0.038 (4)	−0.001 (3)	0.004 (3)	0.001 (3)
C11	0.047 (4)	0.046 (4)	0.036 (4)	−0.001 (3)	−0.002 (3)	−0.002 (3)
C12	0.039 (3)	0.045 (4)	0.037 (4)	−0.001 (3)	−0.006 (3)	0.002 (3)
C13	0.047 (4)	0.039 (4)	0.032 (4)	0.001 (3)	−0.003 (3)	0.001 (3)
C14	0.045 (4)	0.045 (4)	0.041 (4)	0.000 (3)	−0.002 (3)	0.003 (3)
C15	0.042 (3)	0.045 (4)	0.034 (3)	0.001 (3)	0.002 (3)	−0.003 (3)
C16	0.041 (4)	0.050 (4)	0.055 (4)	0.004 (4)	0.002 (3)	−0.003 (4)
C17	0.042 (4)	0.062 (5)	0.049 (5)	−0.006 (4)	−0.002 (3)	−0.001 (4)
C18	0.053 (4)	0.060 (6)	0.034 (4)	−0.016 (4)	0.005 (3)	0.002 (3)
C19	0.060 (4)	0.049 (5)	0.040 (4)	−0.003 (3)	−0.006 (3)	0.008 (3)
C20	0.041 (4)	0.056 (4)	0.041 (4)	−0.002 (3)	−0.002 (3)	0.006 (3)

Geometric parameters (Å, º)

Br1—C3	1.900 (6)	C8—H8	0.9300
Br2—C18	1.911 (7)	C9—C10	1.359 (8)
N1—O3	1.204 (8)	C10—C12	1.505 (9)
N1—O2	1.215 (8)	C11—C12	1.556 (9)
N1—C11	1.506 (10)	C11—H11	0.9800
O1—C11	1.389 (8)	C12—C13	1.501 (9)
O1—C9	1.400 (8)	C12—H12	0.9800
C1—C2	1.358 (10)	C13—C14	1.306 (9)
C1—C6	1.408 (11)	C13—H13	0.9300
C1—H1	0.9300	C14—C15	1.454 (10)
C2—C3	1.415 (10)	C14—H14	0.9300
C2—H2	0.9300	C15—C16	1.382 (8)
C3—C4	1.359 (9)	C15—C20	1.404 (10)
C4—C5	1.415 (8)	C16—C17	1.374 (10)
C4—H4	0.9300	C16—H16	0.9300
C5—C10	1.413 (9)	C17—C18	1.365 (11)
C5—C6	1.421 (9)	C17—H17	0.9300
C6—C7	1.416 (10)	C18—C19	1.378 (10)
C7—C8	1.364 (11)	C19—C20	1.374 (10)
C7—H7	0.9300	C19—H19	0.9300
C8—C9	1.376 (9)	C20—H20	0.9300

O3—N1—O2	123.4 (7)	O1—C11—C12	108.8 (5)
O3—N1—C11	121.2 (7)	N1—C11—C12	107.3 (6)
O2—N1—C11	115.4 (7)	O1—C11—H11	110.5
C11—O1—C9	107.0 (5)	N1—C11—H11	110.5
C2—C1—C6	121.7 (7)	C12—C11—H11	110.5
C2—C1—H1	119.1	C13—C12—C10	114.0 (6)
C6—C1—H1	119.1	C13—C12—C11	111.0 (5)
C1—C2—C3	118.6 (7)	C10—C12—C11	98.8 (5)
C1—C2—H2	120.7	C13—C12—H12	110.8
C3—C2—H2	120.7	C10—C12—H12	110.8
C4—C3—C2	122.7 (6)	C11—C12—H12	110.8
C4—C3—Br1	119.4 (5)	C14—C13—C12	125.6 (7)
C2—C3—Br1	117.9 (5)	C14—C13—H13	117.2
C3—C4—C5	118.5 (6)	C12—C13—H13	117.2
C3—C4—H4	120.8	C13—C14—C15	127.1 (7)
C5—C4—H4	120.8	C13—C14—H14	116.5
C10—C5—C4	122.4 (6)	C15—C14—H14	116.5
C10—C5—C6	117.4 (6)	C16—C15—C20	116.5 (7)
C4—C5—C6	120.1 (6)	C16—C15—C14	122.1 (7)
C1—C6—C7	122.3 (6)	C20—C15—C14	121.4 (6)
C1—C6—C5	118.3 (6)	C17—C16—C15	123.2 (8)
C7—C6—C5	119.4 (6)	C17—C16—H16	118.4
C8—C7—C6	122.3 (6)	C15—C16—H16	118.4
C8—C7—H7	118.9	C18—C17—C16	117.7 (6)
C6—C7—H7	118.9	C18—C17—H17	121.2
C7—C8—C9	116.6 (6)	C16—C17—H17	121.2
C7—C8—H8	121.7	C17—C18—C19	122.6 (7)
C9—C8—H8	121.7	C17—C18—Br2	120.4 (5)
C10—C9—C8	124.9 (7)	C19—C18—Br2	117.0 (6)
C10—C9—O1	112.0 (5)	C20—C19—C18	118.2 (7)
C8—C9—O1	123.1 (6)	C20—C19—H19	120.9
C9—C10—C5	119.4 (6)	C18—C19—H19	120.9
C9—C10—C12	109.7 (5)	C19—C20—C15	121.8 (6)
C5—C10—C12	130.8 (5)	C19—C20—H20	119.1
O1—C11—N1	109.0 (6)	C15—C20—H20	119.1

C6—C1—C2—C3	−0.5 (11)	C9—O1—C11—C12	17.4 (7)
C1—C2—C3—C4	0.5 (11)	O3—N1—C11—O1	24.1 (9)
C1—C2—C3—Br1	−179.4 (6)	O2—N1—C11—O1	−157.1 (6)
C2—C3—C4—C5	0.1 (11)	O3—N1—C11—C12	−93.6 (7)
Br1—C3—C4—C5	179.9 (5)	O2—N1—C11—C12	85.2 (8)
C3—C4—C5—C10	179.1 (6)	C9—C10—C12—C13	−104.2 (7)
C3—C4—C5—C6	−0.6 (10)	C5—C10—C12—C13	72.0 (9)
C2—C1—C6—C7	179.8 (7)	C9—C10—C12—C11	13.6 (8)
C2—C1—C6—C5	0.0 (11)	C5—C10—C12—C11	−170.2 (7)
C10—C5—C6—C1	−179.1 (7)	O1—C11—C12—C13	101.3 (6)
C4—C5—C6—C1	0.6 (10)	N1—C11—C12—C13	−140.9 (6)

C10—C5—C6—C7	1.1 (10)	O1—C11—C12—C10	−18.7 (7)
C4—C5—C6—C7	−179.2 (7)	N1—C11—C12—C10	99.1 (6)
C1—C6—C7—C8	178.8 (7)	C10—C12—C13—C14	−123.6 (7)
C5—C6—C7—C8	−1.4 (11)	C11—C12—C13—C14	125.9 (7)
C6—C7—C8—C9	0.5 (11)	C12—C13—C14—C15	177.8 (6)
C7—C8—C9—C10	0.7 (12)	C13—C14—C15—C16	−165.8 (7)
C7—C8—C9—O1	−179.1 (7)	C13—C14—C15—C20	13.8 (11)
C11—O1—C9—C10	−8.4 (8)	C20—C15—C16—C17	−1.5 (10)
C11—O1—C9—C8	171.3 (7)	C14—C15—C16—C17	178.1 (7)
C8—C9—C10—C5	−0.9 (11)	C15—C16—C17—C18	1.0 (11)
O1—C9—C10—C5	178.8 (6)	C16—C17—C18—C19	0.1 (11)
C8—C9—C10—C12	175.8 (7)	C16—C17—C18—Br2	178.4 (5)
O1—C9—C10—C12	−4.4 (9)	C17—C18—C19—C20	−0.5 (11)
C4—C5—C10—C9	−179.7 (7)	Br2—C18—C19—C20	−178.8 (6)
C6—C5—C10—C9	0.0 (10)	C18—C19—C20—C15	−0.2 (11)
C4—C5—C10—C12	4.4 (12)	C16—C15—C20—C19	1.1 (10)
C6—C5—C10—C12	−175.9 (7)	C14—C15—C20—C19	−178.6 (7)
C9—O1—C11—N1	−99.4 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···Br1^{i}	0.98	3.04	3.786 (7)	134
C11—H11···Br2^{ii}	0.98	3.04	3.942 (7)	154

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