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5-Bromo-1-octylindoline-2,3-dione

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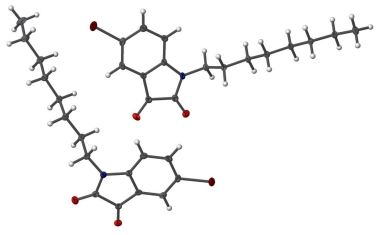
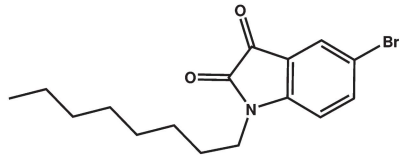
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{16}H_{20}BrNO_2$, crystallizes with two molecules in the asymmetric unit. The indoline ring system and the two ketone O atoms are nearly coplanar, with the largest deviations from the mean plane being 0.077 (2) and 0.055 (2) Å in the two molecules. In each molecule, the mean plane through the octyl chain is nearly perpendicular to the mean plane of the indoline ring system, as indicated by the dihedral angles between them of 86.6 (1) and 76.1 (1)°. In the crystal, molecules are linked by week C—H···O hydrogen bonds, forming a three-dimensional network.

3D view



Chemical scheme



Structure description

Isatin and 5-bromoisatin have shown anxiolytic, sedative and anticonvulsant activities. They have proven to be good antagonists of natriuretic peptide receptors. In fact, some derivatives of isatins are already in use for the treatment of gastrointestinal stromal tumors and advanced renal cell carcinoma, while many other halogenated derivatives are in use for the treatment of cancer and leukemia. Probably the most important aspect of research surrounding isatin derivatives has evolved in the context of their antifungal and antiviral activities (Sridhar *et al.*, 2001*a*; Sarangapani & Reddy, 1994; Verma *et al.*, 2004; Pandeya *et al.*, 1999; Sridhar & Sreenivasulu, 2001*b*; Aboul-Fadl *et al.*, 2010; Varma & Nobles, 1975). We are concerned in developing new heteroatom-containing 5-bromoisatin derivatives and continue research work to explore other applications (Qachchachi *et al.*, 2013, 2014*a*,*b*)

The asymmetric unit of the title compound is built up from two independent molecules with different orientations, as shown in Fig. 1. The two fused five- and six-membered ring

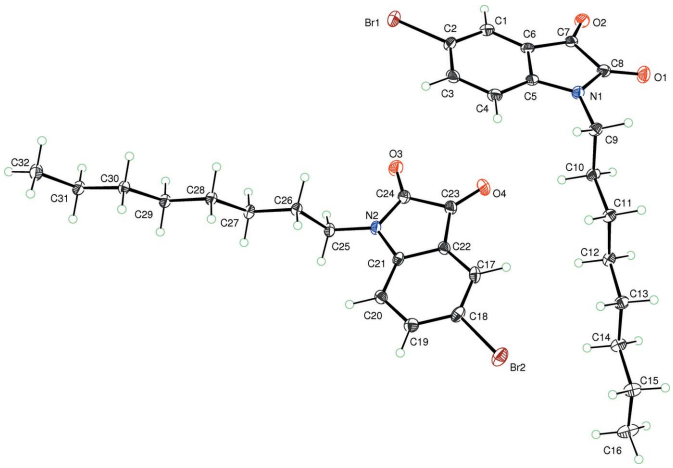


Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

systems in each molecule are almost planar, with maximum deviation of 0.038 (2) Å for C7 in the first molecule (N1/C1–C8) and -.029 (2) Å for C24 in the second molecule (N2/C17–C24). The dihedral angle between the two indoline ring systems is 18.54 (6)°. Moreover, the mean plans through the octyl chains are almost perpendicular to the fused rings systems, as indicated by the torsion angles of C10—C9—N1—C5 = 86.0 (2) and C26—C25—N2—C21 = 85.3 (2)°. In the crystal, molecules are linked by weak C—H···O hydrogen bonds (Table 1), forming a three-dimensional network as shown in Fig. 2.

Synthesis and crystallization

A mixture of 5-bromoisatin (0.4 g, 1.76 mmol) and 1-bromo-octane (0.33 ml, 1.70 mmol) in DMF (25 ml) in the presence of a catalytic amount of tetra-*n*-butylammonium bromide (0.1 g, 0.4 mmol) and potassium carbonate (0.6 g, 4.4 mmol) was stirred for 48 h. After filtering, the reaction was monitored by thin layer chromatography. Orange crystals of the title compound (m.p. = 347 K) were obtained in 69% yield.

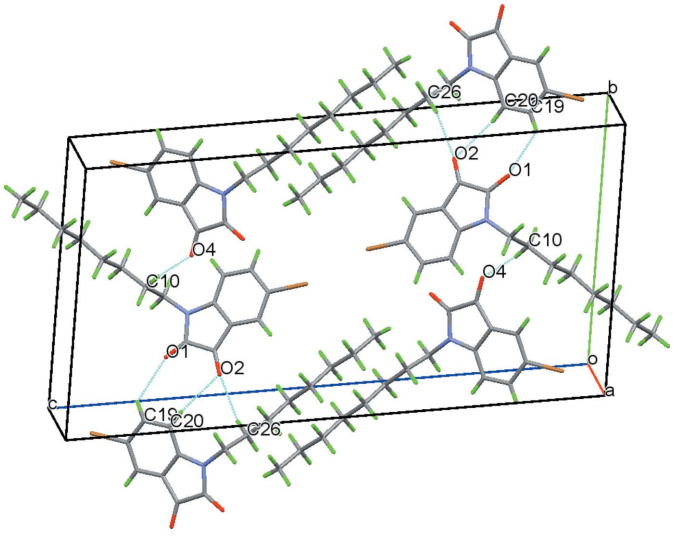


Figure 2
Molecules linked by C—H···O hydrogen bonds, forming a three-dimensional network.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*A*···O4	0.99	2.59	3.572 (2)	174
C19—H19···O1^{i}	0.95	2.51	3.181 (2)	128
C20—H20···O2^{i}	0.95	2.56	3.457 (2)	159
C26—H26*B*···O2^{i}	0.99	2.50	3.159 (2)	124

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{20}BrNO_2$
M_r	338.24
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	100
a, *b*, *c* (Å)	4.5284 (2), 13.2044 (6), 25.9133 (11)
α, β, γ (°)	96.663 (3), 90.728 (3), 95.393 (3)
V (Å^3)	1531.77 (12)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	2.69
Crystal size (mm)	0.13 × 0.12 × 0.10
Data collection	
Diffractometer	Bruker X8 *APEX*
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.649, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	32458, 7501, 6054
R_{int}	0.035
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.667
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.032, 0.070, 1.22
No. of reflections	7501
No. of parameters	363
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.41, −0.40

Computer programs: *APEX2* (Bruker, 2009), *SAINT* (Bruker, 2009), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2009), *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The reflection (1 0 0) affected by the beamstop was removed during the final refinement.

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full crystallographic data

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Crystal data

$C_{16}H_{20}BrNO_2$
M_r = 338.24
Triclinic, $P\overline{1}$
a = 4.5284 (2) Å
b = 13.2044 (6) Å
c = 25.9133 (11) Å
α = 96.663 (3)°
β = 90.728 (3)°
γ = 95.393 (3)°
V = 1531.77 (12) Å^3
Z = 4
$F(000)$ = 696
D_x = 1.467 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7501 reflections
θ = 1.6–28.3°
μ = 2.69 mm^{-1}
T = 100 K
Irregular shape, red
0.13 × 0.12 × 0.10 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.649, T_{max} = 0.746
32458 measured reflections
7501 independent reflections
6054 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 28.3°, θ_{min} = 1.6°
h = −6→6
k = −17→17
l = −34→34

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.070
S = 1.22
7501 reflections
363 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0287P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.006
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.33986 (5)	0.53518 (2)	0.43877 (2)	0.02445 (6)
Br2	0.95415 (5)	0.09935 (2)	0.08242 (2)	0.02568 (7)
C1	0.4271 (4)	0.66359 (14)	0.35847 (7)	0.0167 (4)
H1	0.2996	0.7066	0.3775	0.020*
C2	0.4956 (4)	0.57285 (15)	0.37545 (7)	0.0175 (4)
C3	0.6762 (4)	0.50825 (15)	0.34690 (7)	0.0176 (4)
H3	0.7146	0.4455	0.3590	0.021*
C4	0.8007 (4)	0.53444 (15)	0.30092 (7)	0.0168 (4)
H4	0.9240	0.4906	0.2814	0.020*
C5	0.7395 (4)	0.62592 (14)	0.28468 (7)	0.0138 (4)
C6	0.5515 (4)	0.68921 (14)	0.31275 (7)	0.0142 (4)
C7	0.5225 (4)	0.77703 (14)	0.28406 (7)	0.0152 (4)
C8	0.7297 (4)	0.76111 (15)	0.23685 (7)	0.0164 (4)
C9	1.0208 (4)	0.62114 (14)	0.19977 (7)	0.0154 (4)
H9A	1.1547	0.5771	0.2152	0.018*
H9B	1.1451	0.6742	0.1837	0.018*
C10	0.8240 (4)	0.55667 (14)	0.15815 (7)	0.0158 (4)
H10B	0.6973	0.6016	0.1417	0.019*
H10A	0.6921	0.5063	0.1747	0.019*
C11	0.9994 (4)	0.49967 (14)	0.11622 (7)	0.0160 (4)
H11B	1.1389	0.5496	0.1009	0.019*
H11A	1.1179	0.4518	0.1323	0.019*
C12	0.7999 (4)	0.43955 (14)	0.07312 (7)	0.0156 (4)
H12B	0.6498	0.3939	0.0889	0.019*
H12A	0.6933	0.4882	0.0553	0.019*
C13	0.9682 (4)	0.37547 (15)	0.03312 (7)	0.0166 (4)
H13B	1.1101	0.4216	0.0159	0.020*
H13A	1.0843	0.3297	0.0512	0.020*
C14	0.7673 (4)	0.31093 (15)	−0.00806 (7)	0.0181 (4)
H14B	0.6475	0.3566	−0.0255	0.022*
H14A	0.6289	0.2636	0.0091	0.022*
C15	0.9363 (4)	0.24857 (15)	−0.04907 (7)	0.0211 (4)
H15B	1.0709	0.2959	−0.0670	0.025*
H15A	1.0600	0.2041	−0.0316	0.025*
C16	0.7334 (5)	0.18244 (17)	−0.08919 (8)	0.0283 (5)
H16A	0.6089	0.1321	−0.0721	0.042*
H16C	0.8533	0.1467	−0.1154	0.042*
H16B	0.6069	0.2258	−0.1061	0.042*
C17	0.6291 (4)	0.21003 (15)	0.15971 (7)	0.0168 (4)
H17	0.6944	0.2691	0.1438	0.020*
C18	0.7167 (4)	0.11479 (15)	0.14196 (7)	0.0170 (4)
C19	0.6272 (4)	0.02868 (15)	0.16610 (7)	0.0169 (4)
H19	0.6949	−0.0354	0.1536	0.020*
C20	0.4396 (4)	0.03486 (14)	0.20842 (7)	0.0153 (4)
H20	0.3771	−0.0239	0.2249	0.018*

C21	0.3485 (4)	0.12914 (14)	0.22545 (7)	0.0136 (4)
C22	0.4430 (4)	0.21578 (14)	0.20151 (7)	0.0149 (4)
C23	0.3020 (4)	0.30251 (15)	0.22828 (7)	0.0170 (4)
C24	0.1096 (4)	0.25545 (15)	0.27056 (7)	0.0176 (4)
C25	0.0036 (4)	0.08215 (14)	0.29796 (7)	0.0148 (4)
H25A	−0.0544	0.0170	0.2757	0.018*
H25B	−0.1806	0.1100	0.3110	0.018*
C26	0.1880 (4)	0.05958 (14)	0.34417 (7)	0.0126 (4)
H26A	0.2513	0.1243	0.3664	0.015*
H26B	0.3682	0.0284	0.3316	0.015*
C27	0.0073 (4)	−0.01333 (14)	0.37592 (7)	0.0142 (4)
H27B	−0.1545	0.0229	0.3927	0.017*
H27A	−0.0855	−0.0719	0.3520	0.017*
C28	0.1859 (4)	−0.05445 (14)	0.41761 (7)	0.0133 (4)
H28B	0.2807	0.0039	0.4415	0.016*
H28A	0.3456	−0.0919	0.4009	0.016*
C29	−0.0021 (4)	−0.12577 (14)	0.44903 (7)	0.0140 (4)
H29B	−0.1616	−0.0881	0.4656	0.017*
H29A	−0.0976	−0.1837	0.4250	0.017*
C30	0.1708 (4)	−0.16839 (14)	0.49094 (7)	0.0133 (4)
H30A	0.3237	−0.2091	0.4742	0.016*
H30B	0.2745	−0.1105	0.5140	0.016*
C31	−0.0209 (4)	−0.23528 (15)	0.52385 (7)	0.0157 (4)
H31A	−0.1172	−0.2950	0.5011	0.019*
H31B	−0.1796	−0.1956	0.5393	0.019*
C32	0.1519 (4)	−0.27373 (15)	0.56740 (7)	0.0202 (4)
H32A	0.3006	−0.3171	0.5524	0.030*
H32C	0.0150	−0.3136	0.5880	0.030*
H32B	0.2513	−0.2151	0.5898	0.030*
N1	0.8458 (3)	0.67046 (11)	0.24066 (6)	0.0152 (3)
N2	0.1553 (3)	0.15416 (12)	0.26640 (6)	0.0143 (3)
O1	0.7723 (3)	0.81734 (10)	0.20352 (5)	0.0234 (3)
O2	0.3696 (3)	0.84731 (10)	0.29132 (5)	0.0209 (3)
O3	−0.0522 (3)	0.30056 (11)	0.30020 (5)	0.0248 (3)
O4	0.3105 (3)	0.39090 (10)	0.22037 (5)	0.0234 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.03487 (14)	0.02370 (12)	0.01569 (10)	0.00250 (9)	0.00435 (8)	0.00609 (8)
Br2	0.02694 (13)	0.03326 (14)	0.01880 (11)	0.00805 (9)	0.00766 (8)	0.00649 (9)
C1	0.0176 (10)	0.0166 (10)	0.0153 (9)	0.0029 (8)	−0.0007 (7)	−0.0021 (8)
C2	0.0215 (11)	0.0202 (11)	0.0105 (8)	−0.0005 (8)	−0.0008 (7)	0.0028 (7)
C3	0.0226 (11)	0.0127 (10)	0.0174 (9)	0.0014 (8)	−0.0050 (8)	0.0023 (8)
C4	0.0185 (10)	0.0151 (10)	0.0165 (9)	0.0033 (8)	−0.0004 (7)	−0.0011 (8)
C5	0.0150 (10)	0.0139 (10)	0.0116 (8)	−0.0011 (8)	−0.0034 (7)	−0.0004 (7)
C6	0.0168 (10)	0.0127 (10)	0.0123 (8)	0.0016 (8)	−0.0028 (7)	−0.0017 (7)
C7	0.0187 (10)	0.0136 (10)	0.0123 (9)	−0.0001 (8)	−0.0029 (7)	−0.0006 (7)

C8	0.0202 (11)	0.0144 (10)	0.0145 (9)	0.0026 (8)	−0.0010 (7)	0.0004 (8)
C9	0.0151 (10)	0.0175 (10)	0.0141 (9)	0.0046 (8)	0.0040 (7)	0.0009 (7)
C10	0.0147 (10)	0.0184 (10)	0.0140 (9)	0.0019 (8)	0.0023 (7)	0.0006 (8)
C11	0.0156 (10)	0.0184 (10)	0.0141 (9)	0.0028 (8)	0.0016 (7)	0.0013 (8)
C12	0.0158 (10)	0.0182 (10)	0.0135 (9)	0.0027 (8)	0.0020 (7)	0.0034 (8)
C13	0.0155 (10)	0.0192 (11)	0.0151 (9)	0.0035 (8)	0.0023 (7)	0.0000 (8)
C14	0.0182 (10)	0.0209 (11)	0.0152 (9)	0.0042 (8)	0.0007 (8)	−0.0004 (8)
C15	0.0207 (11)	0.0238 (12)	0.0177 (10)	0.0043 (9)	−0.0002 (8)	−0.0036 (8)
C16	0.0269 (12)	0.0343 (13)	0.0211 (11)	0.0053 (10)	0.0007 (9)	−0.0096 (9)
C17	0.0159 (10)	0.0180 (10)	0.0178 (9)	−0.0002 (8)	−0.0013 (7)	0.0089 (8)
C18	0.0140 (10)	0.0253 (11)	0.0125 (9)	0.0030 (8)	−0.0006 (7)	0.0046 (8)
C19	0.0177 (10)	0.0185 (11)	0.0148 (9)	0.0039 (8)	−0.0037 (7)	0.0018 (8)
C20	0.0174 (10)	0.0139 (10)	0.0149 (9)	0.0014 (8)	−0.0040 (7)	0.0035 (7)
C21	0.0126 (10)	0.0180 (10)	0.0109 (8)	0.0016 (8)	−0.0017 (7)	0.0046 (7)
C22	0.0169 (10)	0.0131 (10)	0.0150 (9)	0.0019 (8)	−0.0022 (7)	0.0035 (7)
C23	0.0199 (11)	0.0151 (10)	0.0165 (9)	0.0019 (8)	−0.0013 (8)	0.0041 (8)
C24	0.0223 (11)	0.0162 (10)	0.0148 (9)	0.0028 (8)	−0.0021 (8)	0.0037 (8)
C25	0.0147 (10)	0.0152 (10)	0.0148 (9)	−0.0008 (8)	−0.0009 (7)	0.0050 (7)
C26	0.0122 (9)	0.0146 (10)	0.0115 (8)	0.0026 (7)	0.0009 (7)	0.0028 (7)
C27	0.0132 (10)	0.0174 (10)	0.0129 (8)	0.0019 (8)	0.0019 (7)	0.0049 (7)
C28	0.0126 (10)	0.0153 (10)	0.0123 (8)	0.0008 (7)	−0.0006 (7)	0.0033 (7)
C29	0.0138 (10)	0.0152 (10)	0.0131 (8)	0.0004 (8)	−0.0009 (7)	0.0033 (7)
C30	0.0123 (9)	0.0154 (10)	0.0126 (8)	0.0008 (7)	0.0012 (7)	0.0036 (7)
C31	0.0167 (10)	0.0174 (10)	0.0133 (9)	0.0010 (8)	−0.0007 (7)	0.0039 (7)
C32	0.0222 (11)	0.0204 (11)	0.0188 (10)	0.0001 (8)	0.0001 (8)	0.0073 (8)
N1	0.0189 (9)	0.0138 (9)	0.0136 (7)	0.0037 (7)	0.0028 (6)	0.0019 (6)
N2	0.0171 (8)	0.0146 (8)	0.0124 (7)	0.0025 (7)	0.0017 (6)	0.0054 (6)
O1	0.0322 (9)	0.0197 (8)	0.0205 (7)	0.0067 (6)	0.0043 (6)	0.0081 (6)
O2	0.0287 (8)	0.0161 (7)	0.0191 (7)	0.0089 (6)	0.0019 (6)	0.0016 (6)
O3	0.0343 (9)	0.0221 (8)	0.0203 (7)	0.0114 (7)	0.0086 (6)	0.0043 (6)
O4	0.0317 (9)	0.0141 (8)	0.0259 (8)	0.0045 (6)	0.0014 (6)	0.0071 (6)

Geometric parameters (Å, º)

Br1—C2	1.8948 (19)	C16—H16B	0.9800
Br2—C18	1.8943 (18)	C17—C22	1.380 (3)
C1—C2	1.384 (3)	C17—C18	1.384 (3)
C1—C6	1.382 (3)	C17—H17	0.9500
C1—H1	0.9500	C18—C19	1.390 (3)
C2—C3	1.394 (3)	C19—C20	1.396 (3)
C3—C4	1.391 (3)	C19—H19	0.9500
C3—H3	0.9500	C20—C21	1.374 (2)
C4—C5	1.375 (3)	C20—H20	0.9500
C4—H4	0.9500	C21—C22	1.399 (3)
C5—C6	1.400 (2)	C21—N2	1.413 (2)
C5—N1	1.413 (2)	C22—C23	1.471 (3)
C6—C7	1.463 (3)	C23—O4	1.205 (2)
C7—O2	1.208 (2)	C23—C24	1.558 (3)

C7—C8	1.558 (3)	C24—O3	1.213 (2)
C8—O1	1.208 (2)	C24—N2	1.365 (2)
C8—N1	1.365 (2)	C25—N2	1.455 (2)
C9—N1	1.461 (2)	C25—C26	1.525 (2)
C9—C10	1.517 (2)	C25—H25A	0.9900
C9—H9A	0.9900	C25—H25B	0.9900
C9—H9B	0.9900	C26—C27	1.525 (2)
C10—C11	1.524 (2)	C26—H26A	0.9900
C10—H10B	0.9900	C26—H26B	0.9900
C10—H10A	0.9900	C27—C28	1.521 (2)
C11—C12	1.526 (2)	C27—H27B	0.9900
C11—H11B	0.9900	C27—H27A	0.9900
C11—H11A	0.9900	C28—C29	1.523 (2)
C12—C13	1.520 (2)	C28—H28B	0.9900
C12—H12B	0.9900	C28—H28A	0.9900
C12—H12A	0.9900	C29—C30	1.521 (2)
C13—C14	1.520 (3)	C29—H29B	0.9900
C13—H13B	0.9900	C29—H29A	0.9900
C13—H13A	0.9900	C30—C31	1.520 (2)
C14—C15	1.526 (2)	C30—H30A	0.9900
C14—H14B	0.9900	C30—H30B	0.9900
C14—H14A	0.9900	C31—C32	1.526 (2)
C15—C16	1.517 (3)	C31—H31A	0.9900
C15—H15B	0.9900	C31—H31B	0.9900
C15—H15A	0.9900	C32—H32A	0.9800
C16—H16A	0.9800	C32—H32C	0.9800
C16—H16C	0.9800	C32—H32B	0.9800

C2—C1—C6	117.35 (17)	C17—C18—C19	121.35 (18)
C2—C1—H1	121.3	C17—C18—Br2	119.87 (14)
C6—C1—H1	121.3	C19—C18—Br2	118.76 (14)
C1—C2—C3	121.45 (18)	C18—C19—C20	121.06 (18)
C1—C2—Br1	119.35 (14)	C18—C19—H19	119.5
C3—C2—Br1	119.20 (14)	C20—C19—H19	119.5
C2—C3—C4	120.91 (18)	C21—C20—C19	117.54 (17)
C2—C3—H3	119.5	C21—C20—H20	121.2
C4—C3—H3	119.5	C19—C20—H20	121.2
C5—C4—C3	117.75 (18)	C20—C21—C22	121.12 (17)
C5—C4—H4	121.1	C20—C21—N2	128.01 (17)
C3—C4—H4	121.1	C22—C21—N2	110.86 (16)
C4—C5—C6	121.11 (17)	C17—C22—C21	121.52 (17)
C4—C5—N1	128.32 (17)	C17—C22—C23	131.21 (17)
C6—C5—N1	110.56 (16)	C21—C22—C23	107.25 (16)
C1—C6—C5	121.38 (17)	O4—C23—C22	131.15 (18)
C1—C6—C7	131.27 (17)	O4—C23—C24	124.06 (17)
C5—C6—C7	107.35 (16)	C22—C23—C24	104.72 (15)
O2—C7—C6	131.13 (18)	O3—C24—N2	127.65 (18)
O2—C7—C8	123.80 (16)	O3—C24—C23	126.12 (17)

C6—C7—C8	105.02 (15)	N2—C24—C23	106.21 (15)
O1—C8—N1	127.77 (18)	N2—C25—C26	114.12 (15)
O1—C8—C7	126.40 (17)	N2—C25—H25A	108.7
N1—C8—C7	105.82 (15)	C26—C25—H25A	108.7
N1—C9—C10	111.50 (15)	N2—C25—H25B	108.7
N1—C9—H9A	109.3	C26—C25—H25B	108.7
C10—C9—H9A	109.3	H25A—C25—H25B	107.6
N1—C9—H9B	109.3	C25—C26—C27	110.31 (15)
C10—C9—H9B	109.3	C25—C26—H26A	109.6
H9A—C9—H9B	108.0	C27—C26—H26A	109.6
C9—C10—C11	112.97 (15)	C25—C26—H26B	109.6
C9—C10—H10B	109.0	C27—C26—H26B	109.6
C11—C10—H10B	109.0	H26A—C26—H26B	108.1
C9—C10—H10A	109.0	C28—C27—C26	114.35 (15)
C11—C10—H10A	109.0	C28—C27—H27B	108.7
H10B—C10—H10A	107.8	C26—C27—H27B	108.7
C12—C11—C10	112.51 (15)	C28—C27—H27A	108.7
C12—C11—H11B	109.1	C26—C27—H27A	108.7
C10—C11—H11B	109.1	H27B—C27—H27A	107.6
C12—C11—H11A	109.1	C27—C28—C29	112.95 (15)
C10—C11—H11A	109.1	C27—C28—H28B	109.0
H11B—C11—H11A	107.8	C29—C28—H28B	109.0
C13—C12—C11	113.37 (15)	C27—C28—H28A	109.0
C13—C12—H12B	108.9	C29—C28—H28A	109.0
C11—C12—H12B	108.9	H28B—C28—H28A	107.8
C13—C12—H12A	108.9	C30—C29—C28	114.15 (15)
C11—C12—H12A	108.9	C30—C29—H29B	108.7
H12B—C12—H12A	107.7	C28—C29—H29B	108.7
C14—C13—C12	113.30 (15)	C30—C29—H29A	108.7
C14—C13—H13B	108.9	C28—C29—H29A	108.7
C12—C13—H13B	108.9	H29B—C29—H29A	107.6
C14—C13—H13A	108.9	C31—C30—C29	113.80 (15)
C12—C13—H13A	108.9	C31—C30—H30A	108.8
H13B—C13—H13A	107.7	C29—C30—H30A	108.8
C13—C14—C15	113.41 (15)	C31—C30—H30B	108.8
C13—C14—H14B	108.9	C29—C30—H30B	108.8
C15—C14—H14B	108.9	H30A—C30—H30B	107.7
C13—C14—H14A	108.9	C30—C31—C32	113.48 (15)
C15—C14—H14A	108.9	C30—C31—H31A	108.9
H14B—C14—H14A	107.7	C32—C31—H31A	108.9
C16—C15—C14	112.96 (16)	C30—C31—H31B	108.9
C16—C15—H15B	109.0	C32—C31—H31B	108.9
C14—C15—H15B	109.0	H31A—C31—H31B	107.7
C16—C15—H15A	109.0	C31—C32—H32A	109.5
C14—C15—H15A	109.0	C31—C32—H32C	109.5
H15B—C15—H15A	107.8	H32A—C32—H32C	109.5
C15—C16—H16A	109.5	C31—C32—H32B	109.5
C15—C16—H16C	109.5	H32A—C32—H32B	109.5

H16A—C16—H16C	109.5	H32C—C32—H32B	109.5
C15—C16—H16B	109.5	C8—N1—C5	111.17 (15)
H16A—C16—H16B	109.5	C8—N1—C9	122.78 (15)
H16C—C16—H16B	109.5	C5—N1—C9	125.47 (15)
C22—C17—C18	117.40 (17)	C24—N2—C21	110.93 (15)
C22—C17—H17	121.3	C24—N2—C25	123.13 (16)
C18—C17—H17	121.3	C21—N2—C25	125.78 (15)
C6—C1—C2—C3	−1.8 (3)	N2—C21—C22—C17	−178.74 (16)
C6—C1—C2—Br1	178.44 (13)	C20—C21—C22—C23	178.98 (16)
C1—C2—C3—C4	1.8 (3)	N2—C21—C22—C23	−0.3 (2)
Br1—C2—C3—C4	−178.37 (14)	C17—C22—C23—O4	0.7 (4)
C2—C3—C4—C5	0.0 (3)	C21—C22—C23—O4	−177.6 (2)
C3—C4—C5—C6	−1.7 (3)	C17—C22—C23—C24	177.60 (19)
C3—C4—C5—N1	177.88 (17)	C21—C22—C23—C24	−0.65 (19)
C2—C1—C6—C5	0.0 (3)	O4—C23—C24—O3	0.0 (3)
C2—C1—C6—C7	179.01 (18)	C22—C23—C24—O3	−177.20 (18)
C4—C5—C6—C1	1.8 (3)	O4—C23—C24—N2	178.57 (18)
N1—C5—C6—C1	−177.87 (16)	C22—C23—C24—N2	1.39 (19)
C4—C5—C6—C7	−177.43 (16)	N2—C25—C26—C27	177.96 (15)
N1—C5—C6—C7	2.9 (2)	C25—C26—C27—C28	170.44 (15)
C1—C6—C7—O2	−4.6 (3)	C26—C27—C28—C29	179.20 (15)
C5—C6—C7—O2	174.48 (19)	C27—C28—C29—C30	179.83 (15)
C1—C6—C7—C8	178.04 (19)	C28—C29—C30—C31	177.15 (16)
C5—C6—C7—C8	−2.85 (19)	C29—C30—C31—C32	−177.35 (15)
O2—C7—C8—O1	3.1 (3)	O1—C8—N1—C5	−178.94 (19)
C6—C7—C8—O1	−179.34 (19)	C7—C8—N1—C5	−0.2 (2)
O2—C7—C8—N1	−175.73 (17)	O1—C8—N1—C9	−7.3 (3)
C6—C7—C8—N1	1.85 (19)	C7—C8—N1—C9	171.50 (15)
N1—C9—C10—C11	−177.08 (15)	C4—C5—N1—C8	178.64 (18)
C9—C10—C11—C12	−177.01 (16)	C6—C5—N1—C8	−1.7 (2)
C10—C11—C12—C13	−175.47 (16)	C4—C5—N1—C9	7.3 (3)
C11—C12—C13—C14	176.69 (16)	C6—C5—N1—C9	−173.12 (16)
C12—C13—C14—C15	178.53 (16)	C10—C9—N1—C8	−84.4 (2)
C13—C14—C15—C16	178.57 (17)	C10—C9—N1—C5	86.0 (2)
C22—C17—C18—C19	−1.6 (3)	O3—C24—N2—C21	176.94 (19)
C22—C17—C18—Br2	176.54 (13)	C23—C24—N2—C21	−1.61 (19)
C17—C18—C19—C20	1.5 (3)	O3—C24—N2—C25	1.3 (3)
Br2—C18—C19—C20	−176.63 (13)	C23—C24—N2—C25	−177.24 (15)
C18—C19—C20—C21	−0.4 (3)	C20—C21—N2—C24	−177.93 (17)
C19—C20—C21—C22	−0.6 (3)	C22—C21—N2—C24	1.3 (2)
C19—C20—C21—N2	178.50 (17)	C20—C21—N2—C25	−2.4 (3)
C18—C17—C22—C21	0.6 (3)	C22—C21—N2—C25	176.75 (16)
C18—C17—C22—C23	−177.44 (18)	C26—C25—N2—C24	−99.72 (19)
C20—C21—C22—C17	0.5 (3)	C26—C25—N2—C21	85.3 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*A*···O4	0.99	2.59	3.572 (2)	174
C19—H19···O1^{i}	0.95	2.51	3.181 (2)	128
C20—H20···O2^{i}	0.95	2.56	3.457 (2)	159
C26—H26*B*···O2^{i}	0.99	2.50	3.159 (2)	124

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