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2,4-Dibromo-6-[(2-hydroxy-5-methylanilino)-methylidene]cyclohexa-2,4-dienone

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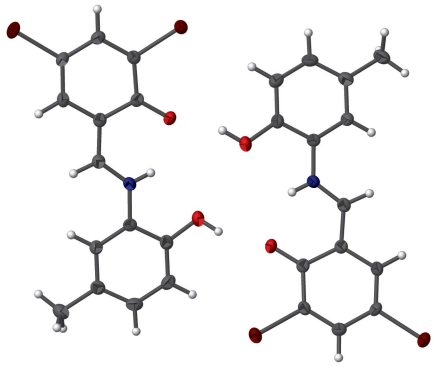
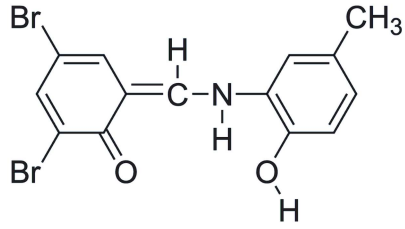
The new bromo-substituted title compound, $C_{14}H_{11}Br_2NO_2$, was synthesized by the condensation of 3,5-dibromosalicylaldehyde and 2-amino-4-methyl phenol. The asymmetric unit consists of two crystallographically independent molecules (*A* and *B*), which are related to each other by a pseudo-inversion centre. Both molecules are almost planar; dihedral angles between the two benzene rings are 11.40 (11)° for *A* and 3.05 (12)° for *B*. In each molecule, there is an intramolecular N—H···O hydrogen bond with an *S*(6) ring motif. In the crystal, two independent molecules are linked by O—H···O hydrogen bonds, forming a pseudo-inversion *A*–*B* dimer.

3D view



Chemical scheme



Structure description

Organic compounds containing N and O donor atoms are widely used as ligands (Dong *et al.*, 2015; Khalaji *et al.*, 2015; de Blas *et al.*, 1991) and their metal complexes have received considerable attention for their possible bioactivities (Zhang *et al.*, 2012; Khandar *et al.*, 2010). In addition, such compounds also containing halogen atoms have been shown to have attractive biological properties (Dong *et al.*, 2015).

The asymmetric unit of the title compound consists of two independent molecules as shown in Fig. 1. The bond lengths of both molecules are almost same and within normal ranges, but the conformations are slightly different. The lengths of C2—O1 and C16—O3 correspond to a C=O double bond, while C9—O2 and C23—O4 are single C—O bonds (Xu *et al.*, 2007). The NH group in each molecule forms an intramolecular N—H···O hydrogen bond (Fig. 1 and Table 1).

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3	0.90 (4)	1.86 (4)	2.728 (3)	163 (4)
O4—H4···O1	0.86 (4)	1.92 (4)	2.753 (3)	164 (3)
N1—H1···O1	0.80 (4)	1.97 (3)	2.616 (3)	139 (3)
N2—H2*A*···O3	0.81 (3)	1.99 (3)	2.632 (3)	136 (3)

In the crystal, the two independent molecules are linked *via* two O—H···O hydrogen bonds, forming a pseudo-inversion dimer with an $R_2^2(18)$ ring motif (Fig. 1 and Table 1).

Synthesis and crystallization

A saturated ethanolic solution of 2-amino-4-methyl phenol (123 mg, 1 mmol) was added dropwise to a saturated ethanolic solution of 3,5-dibromosalicylaldehyde (280 mg, 1 mmol) with continuous stirring at 60°C. The mixture was heated with continuous stirring whereupon a precipitate was formed. The mixture was stirred for a further hour at room temperature. The resulting orange solid product was collected by filtration, washed several times with hot ethanol and dried in a vacuum (300 mg, 78%). Orange single crystals were obtained by slow evaporation from an ethanol/acetonitrile (5:1) solution at room temperature over 20 days (m.p. 190°C).

Refinement

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

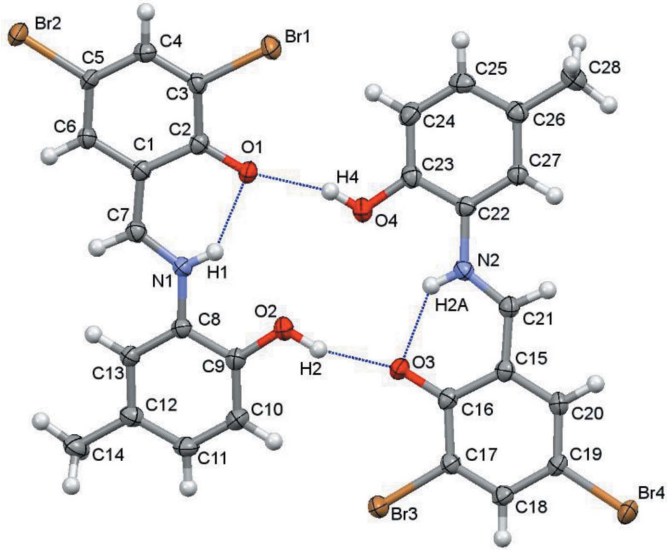


Figure 1
The asymmetric unit of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii. Intramolecular N—H···O and intermolecular O—H···O hydrogen bonds are indicated by dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{11}Br_2NO_2$
M_r	385.05
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	173
a, b, c (Å)	16.7219 (3), 6.90389 (13), 23.0811 (4)
β (°)	99.502 (7)
V (Å^3)	2628.06 (10)
Z	8
Radiation type	Cu $K\alpha$
μ (mm^{-1})	7.81
Crystal size (mm)	0.36 × 0.29 × 0.08
Data collection	
Diffractometer	Rigaku R-AXIS RAPID
Absorption correction	Multi-scan (*ABSCOR*; Higashi, 1995)
T_{min}, T_{max}	0.239, 0.535
No. of measured, independent and observed [$F^2 > 2.0\sigma(F^2)$] reflections	28830, 4799, 4558
R_{int}	0.048
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.602
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.074, 1.08
No. of reflections	4799
No. of parameters	361
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.99, −0.61

Computer programs: *RAPID-AUTO* (Rigaku, 2001), *SIR92* (Altomare *et al.*, 1993), *SHELXL97* (Sheldrick, 2008) and *CrystalStructure* (Rigaku, 2017).

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References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
Blas, A. de, Bastida, R. & Fucntcs, M. J. (1991). *Synth. React. Inorg. Met.-Org. Chem.* **21**, 1273–1298.
Dong, Y. L., Li, C., Meng, X. F., Zhou, X. & Ma, J. J. (2015). *J. Struct. Chem.* **56**, 1426–1430.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Khalaji, A. D., Hafez Ghoran, S., Pojarová, M. & Dušek, M. (2015). *J. Struct. Chem.* **56**, 1410–1414.
Khandar, A. A., Hosseini-Yazdi, S. A., Khatamian, M. & Zarei, S. A. (2010). *Polyhedron*, **29**, 995–1000.
Rigaku (2001). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku (2017). *CrystalStructure*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Xu, T.-T., Xu, X.-Y., Gao, J., Ni, S.-S., Wang, D.-Q., Lu, L.-D., Yang, X.-J. & Wang, X. (2007). *J. Struct. Chem.* **48**, 193–197.
Zhang, M., Xian, D. M., Li, H. H., Zhang, J. C. & You, Z. L. (2012). *Aust. J. Chem.* **65**, 343–350.

full crystallographic data

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Crystal data

$C_{14}H_{11}Br_2NO_2$
M_r = 385.05
Monoclinic, $P2_1/n$
a = 16.7219 (3) Å
b = 6.90389 (13) Å
c = 23.0811 (4) Å
β = 99.502 (7)°
V = 2628.06 (10) Å^3
Z = 8
$F(000)$ = 1504.00
D_x = 1.946 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54187 Å
Cell parameters from 27726 reflections
θ = 3.0–68.2°
μ = 7.81 mm^{-1}
T = 173 K
Platelet, orange
0.36 × 0.29 × 0.08 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Detector resolution: 10.000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
T_{min} = 0.239, T_{max} = 0.535
28830 measured reflections
4799 independent reflections
4558 reflections with $F^2 > 2.0\sigma(F^2)$
R_{int} = 0.048
θ_{max} = 68.2°, θ_{min} = 3.0°
h = −19→20
k = −8→8
l = −27→27

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.074
S = 1.08
4799 reflections
361 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0323P)^2 + 3.250P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.99 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-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. Refinement was performed using all reflections. The weighted R-factor (wR) and goodness of fit (S) are based on F^2. R-factor (gt) are based on F. The threshold expression of $F^2 > 2.0$ sigma(F^2) is used only for calculating R-factor (gt).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	−0.01727 (2)	0.20364 (5)	0.64304 (2)	0.03261 (10)
Br2	0.24115 (2)	0.12206 (6)	0.51554 (2)	0.03943 (11)
Br3	0.25614 (2)	0.09612 (5)	1.09293 (2)	0.03620 (10)
Br4	−0.01488 (2)	0.11048 (5)	1.20799 (2)	0.03135 (9)
O1	0.12485 (11)	0.2391 (3)	0.74614 (8)	0.0298 (4)
O2	0.21228 (12)	0.1592 (3)	0.89639 (9)	0.0347 (5)
O3	0.12662 (11)	0.2105 (3)	0.98606 (8)	0.0295 (4)
O4	0.04812 (12)	0.3579 (3)	0.83675 (9)	0.0325 (5)
N1	0.27421 (14)	0.1839 (3)	0.79845 (9)	0.0231 (5)
N2	−0.01980 (14)	0.2985 (3)	0.93165 (10)	0.0231 (5)
C1	0.23343 (16)	0.1772 (4)	0.69402 (11)	0.0229 (5)
C2	0.14904 (16)	0.2072 (4)	0.69752 (11)	0.0230 (5)
C3	0.09544 (15)	0.1935 (4)	0.64211 (11)	0.0231 (5)
C4	0.12240 (16)	0.1692 (4)	0.58993 (12)	0.0258 (6)
H4A	0.084848	0.163499	0.554274	0.031*
C5	0.20591 (16)	0.1527 (4)	0.58916 (11)	0.0250 (6)
C6	0.26057 (16)	0.1522 (4)	0.63966 (12)	0.0248 (6)
H6	0.316657	0.135172	0.638484	0.030*
C7	0.29174 (16)	0.1680 (4)	0.74565 (11)	0.0242 (6)
H7	0.346874	0.149222	0.741733	0.029*
C8	0.32862 (16)	0.1779 (4)	0.85205 (11)	0.0231 (5)
C9	0.29418 (16)	0.1666 (4)	0.90288 (12)	0.0255 (6)
C10	0.34561 (18)	0.1645 (5)	0.95692 (12)	0.0321 (7)
H10	0.323451	0.156331	0.992180	0.039*
C11	0.42851 (17)	0.1742 (5)	0.95951 (12)	0.0315 (6)
H11	0.462526	0.173001	0.996797	0.038*
C12	0.46372 (17)	0.1858 (4)	0.90902 (12)	0.0278 (6)
C13	0.41242 (16)	0.1874 (4)	0.85515 (12)	0.0242 (6)
H13	0.434787	0.195000	0.819967	0.029*
C14	0.55430 (17)	0.1992 (5)	0.91222 (14)	0.0367 (7)
H14A	0.580797	0.101170	0.939574	0.044*
H14B	0.572862	0.328325	0.926028	0.044*
H14C	0.568130	0.176676	0.873133	0.044*
C15	0.01183 (16)	0.2188 (4)	1.03431 (11)	0.0231 (6)
C16	0.09673 (16)	0.1904 (4)	1.03296 (11)	0.0226 (5)
C17	0.14371 (16)	0.1393 (4)	1.08883 (12)	0.0240 (6)

C18	0.11088 (17)	0.1191 (4)	1.13890 (12)	0.0252 (6)
H18	0.144597	0.085854	1.174796	0.030*
C19	0.02747 (16)	0.1473 (4)	1.13744 (11)	0.0235 (6)
C20	−0.02172 (16)	0.1947 (4)	1.08629 (12)	0.0245 (6)
H20	−0.078274	0.211589	1.085518	0.029*
C21	−0.04189 (16)	0.2698 (4)	0.98261 (11)	0.0244 (6)
H21	−0.097709	0.284149	0.985166	0.029*
C22	−0.07113 (16)	0.3483 (4)	0.87846 (11)	0.0222 (5)
C23	−0.03394 (16)	0.3733 (4)	0.82909 (12)	0.0254 (6)
C24	−0.08220 (18)	0.4147 (4)	0.77557 (12)	0.0300 (6)
H24	−0.058047	0.430591	0.741398	0.036*
C25	−0.16540 (17)	0.4330 (4)	0.77181 (12)	0.0301 (6)
H25	−0.197437	0.461047	0.734813	0.036*
C26	−0.20333 (17)	0.4115 (4)	0.82072 (12)	0.0266 (6)
C27	−0.15469 (16)	0.3695 (4)	0.87419 (12)	0.0240 (6)
H27	−0.178942	0.354950	0.908375	0.029*
C28	−0.29388 (17)	0.4308 (5)	0.81661 (14)	0.0361 (7)
H28A	−0.307472	0.441898	0.856187	0.043*
H28B	−0.320532	0.316250	0.797111	0.043*
H28C	−0.312521	0.546832	0.793864	0.043*
H1	0.227 (2)	0.199 (5)	0.8001 (14)	0.036 (10)*
H2	0.193 (2)	0.164 (6)	0.9303 (18)	0.061 (12)*
H2A	0.028 (2)	0.284 (5)	0.9301 (14)	0.031 (9)*
H4	0.063 (2)	0.328 (5)	0.8042 (15)	0.041 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.01857 (15)	0.0492 (2)	0.03065 (17)	−0.00132 (12)	0.00568 (11)	−0.00265 (13)
Br2	0.02797 (17)	0.0714 (3)	0.02070 (16)	0.00151 (15)	0.00913 (12)	0.00201 (14)
Br3	0.02106 (16)	0.0624 (2)	0.02539 (16)	0.00287 (13)	0.00467 (12)	0.00489 (14)
Br4	0.03446 (18)	0.03847 (19)	0.02417 (16)	0.00327 (12)	0.01380 (12)	0.00366 (12)
O1	0.0220 (10)	0.0471 (12)	0.0215 (9)	0.0004 (9)	0.0077 (7)	0.0000 (9)
O2	0.0206 (10)	0.0570 (14)	0.0280 (11)	0.0031 (9)	0.0080 (8)	0.0017 (10)
O3	0.0234 (10)	0.0457 (12)	0.0202 (9)	−0.0010 (9)	0.0065 (7)	0.0025 (9)
O4	0.0231 (10)	0.0467 (13)	0.0291 (11)	0.0026 (9)	0.0088 (8)	−0.0018 (10)
N1	0.0182 (12)	0.0292 (13)	0.0215 (12)	0.0002 (10)	0.0025 (9)	0.0019 (9)
N2	0.0183 (12)	0.0266 (12)	0.0242 (12)	0.0004 (9)	0.0028 (9)	−0.0009 (9)
C1	0.0227 (13)	0.0235 (14)	0.0229 (13)	−0.0005 (11)	0.0050 (10)	0.0025 (11)
C2	0.0230 (13)	0.0230 (14)	0.0237 (13)	−0.0021 (11)	0.0061 (10)	0.0025 (11)
C3	0.0180 (13)	0.0256 (14)	0.0261 (14)	−0.0005 (10)	0.0045 (10)	0.0018 (11)
C4	0.0253 (14)	0.0293 (14)	0.0219 (13)	0.0004 (11)	0.0014 (10)	0.0016 (11)
C5	0.0259 (14)	0.0332 (15)	0.0171 (12)	−0.0004 (12)	0.0074 (10)	0.0026 (11)
C6	0.0199 (13)	0.0302 (14)	0.0254 (14)	0.0002 (11)	0.0065 (10)	0.0036 (12)
C7	0.0216 (13)	0.0254 (14)	0.0258 (14)	−0.0004 (11)	0.0046 (10)	0.0010 (11)
C8	0.0220 (13)	0.0255 (14)	0.0214 (13)	−0.0001 (11)	0.0028 (10)	0.0024 (11)
C9	0.0234 (14)	0.0295 (14)	0.0246 (14)	0.0020 (11)	0.0069 (11)	0.0023 (11)
C10	0.0314 (16)	0.0433 (18)	0.0221 (14)	0.0035 (13)	0.0058 (11)	0.0041 (13)

C11	0.0287 (15)	0.0405 (17)	0.0231 (14)	−0.0002 (13)	−0.0027 (11)	0.0032 (12)
C12	0.0240 (14)	0.0293 (15)	0.0287 (14)	−0.0006 (11)	0.0004 (11)	0.0017 (12)
C13	0.0223 (14)	0.0288 (14)	0.0218 (13)	−0.0005 (11)	0.0046 (10)	0.0032 (11)
C14	0.0236 (15)	0.0484 (19)	0.0360 (16)	−0.0030 (13)	−0.0013 (12)	0.0063 (14)
C15	0.0242 (13)	0.0219 (13)	0.0236 (13)	0.0002 (11)	0.0055 (10)	−0.0019 (11)
C16	0.0244 (13)	0.0223 (13)	0.0217 (13)	−0.0034 (11)	0.0058 (10)	−0.0016 (11)
C17	0.0206 (13)	0.0276 (14)	0.0237 (13)	−0.0021 (11)	0.0034 (10)	0.0002 (11)
C18	0.0274 (14)	0.0286 (15)	0.0192 (13)	−0.0005 (11)	0.0033 (10)	−0.0012 (11)
C19	0.0275 (14)	0.0250 (14)	0.0192 (13)	0.0000 (11)	0.0075 (10)	−0.0015 (11)
C20	0.0218 (13)	0.0267 (14)	0.0265 (14)	0.0018 (11)	0.0082 (11)	−0.0015 (11)
C21	0.0233 (13)	0.0261 (14)	0.0242 (13)	0.0015 (11)	0.0051 (10)	−0.0013 (11)
C22	0.0237 (13)	0.0214 (13)	0.0211 (13)	0.0011 (11)	0.0025 (10)	−0.0015 (10)
C23	0.0238 (14)	0.0263 (14)	0.0267 (14)	0.0000 (11)	0.0056 (11)	−0.0045 (11)
C24	0.0344 (16)	0.0351 (16)	0.0217 (14)	−0.0008 (13)	0.0078 (11)	−0.0031 (12)
C25	0.0313 (15)	0.0322 (16)	0.0244 (14)	0.0010 (12)	−0.0027 (11)	−0.0012 (12)
C26	0.0251 (14)	0.0272 (15)	0.0268 (14)	−0.0001 (11)	0.0024 (11)	−0.0020 (11)
C27	0.0239 (14)	0.0246 (14)	0.0241 (13)	0.0010 (11)	0.0056 (10)	0.0003 (11)
C28	0.0255 (15)	0.0438 (18)	0.0375 (17)	0.0030 (13)	0.0010 (12)	0.0009 (14)

Geometric parameters (Å, º)

Br1—C3	1.890 (3)	C11—C12	1.392 (4)
Br2—C5	1.900 (3)	C11—H11	0.9500
Br3—C17	1.890 (3)	C12—C13	1.389 (4)
Br4—C19	1.897 (3)	C12—C14	1.507 (4)
O1—C2	1.273 (3)	C13—H13	0.9500
O2—C9	1.354 (3)	C14—H14A	0.9800
O2—H2	0.90 (4)	C14—H14B	0.9800
O3—C16	1.273 (3)	C14—H14C	0.9800
O4—C23	1.358 (3)	C15—C21	1.415 (4)
O4—H4	0.86 (3)	C15—C20	1.416 (4)
N1—C7	1.304 (3)	C15—C16	1.438 (4)
N1—C8	1.410 (3)	C16—C17	1.439 (4)
N1—H1	0.80 (3)	C17—C18	1.366 (4)
N2—C21	1.305 (3)	C18—C19	1.403 (4)
N2—C22	1.419 (3)	C18—H18	0.9500
N2—H2A	0.81 (3)	C19—C20	1.363 (4)
C1—C7	1.411 (4)	C20—H20	0.9500
C1—C6	1.414 (4)	C21—H21	0.9500
C1—C2	1.442 (4)	C22—C27	1.392 (4)
C2—C3	1.439 (4)	C22—C23	1.396 (4)
C3—C4	1.364 (4)	C23—C24	1.389 (4)
C4—C5	1.404 (4)	C24—C25	1.385 (4)
C4—H4A	0.9500	C24—H24	0.9500
C5—C6	1.357 (4)	C25—C26	1.391 (4)
C6—H6	0.9500	C25—H25	0.9500
C7—H7	0.9500	C26—C27	1.392 (4)
C8—C9	1.392 (4)	C26—C28	1.507 (4)

C8—C13	1.393 (4)	C27—H27	0.9500
C9—C10	1.393 (4)	C28—H28A	0.9800
C10—C11	1.379 (4)	C28—H28B	0.9800
C10—H10	0.9500	C28—H28C	0.9800

C9—O2—H2	114 (3)	H14A—C14—H14B	109.5
C23—O4—H4	110 (2)	C12—C14—H14C	109.5
C7—N1—C8	127.3 (2)	H14A—C14—H14C	109.5
C7—N1—H1	115 (2)	H14B—C14—H14C	109.5
C8—N1—H1	117 (2)	C21—C15—C20	117.3 (2)
C21—N2—C22	126.6 (2)	C21—C15—C16	120.5 (2)
C21—N2—H2A	117 (2)	C20—C15—C16	122.2 (2)
C22—N2—H2A	117 (2)	O3—C16—C15	122.1 (2)
C7—C1—C6	117.8 (2)	O3—C16—C17	123.8 (2)
C7—C1—C2	120.4 (2)	C15—C16—C17	114.1 (2)
C6—C1—C2	121.9 (2)	C18—C17—C16	123.2 (2)
O1—C2—C3	123.6 (2)	C18—C17—Br3	118.5 (2)
O1—C2—C1	122.0 (2)	C16—C17—Br3	118.35 (19)
C3—C2—C1	114.4 (2)	C17—C18—C19	120.1 (2)
C4—C3—C2	123.0 (2)	C17—C18—H18	119.9
C4—C3—Br1	119.4 (2)	C19—C18—H18	119.9
C2—C3—Br1	117.62 (19)	C20—C19—C18	120.6 (2)
C3—C4—C5	119.7 (2)	C20—C19—Br4	121.2 (2)
C3—C4—H4A	120.2	C18—C19—Br4	118.12 (19)
C5—C4—H4A	120.2	C19—C20—C15	119.7 (3)
C6—C5—C4	121.3 (2)	C19—C20—H20	120.2
C6—C5—Br2	120.2 (2)	C15—C20—H20	120.2
C4—C5—Br2	118.44 (19)	N2—C21—C15	124.3 (3)
C5—C6—C1	119.5 (2)	N2—C21—H21	117.9
C5—C6—H6	120.2	C15—C21—H21	117.9
C1—C6—H6	120.2	C27—C22—C23	120.4 (2)
N1—C7—C1	123.7 (3)	C27—C22—N2	122.9 (2)
N1—C7—H7	118.2	C23—C22—N2	116.7 (2)
C1—C7—H7	118.2	O4—C23—C24	123.9 (3)
C9—C8—C13	120.8 (2)	O4—C23—C22	117.4 (2)
C9—C8—N1	116.4 (2)	C24—C23—C22	118.6 (3)
C13—C8—N1	122.8 (2)	C25—C24—C23	120.3 (3)
O2—C9—C8	117.4 (2)	C25—C24—H24	119.8
O2—C9—C10	124.2 (2)	C23—C24—H24	119.8
C8—C9—C10	118.4 (3)	C24—C25—C26	121.8 (3)
C11—C10—C9	120.4 (3)	C24—C25—H25	119.1
C11—C10—H10	119.8	C26—C25—H25	119.1
C9—C10—H10	119.8	C25—C26—C27	117.6 (3)
C10—C11—C12	121.8 (3)	C25—C26—C28	121.8 (2)
C10—C11—H11	119.1	C27—C26—C28	120.5 (3)
C12—C11—H11	119.1	C26—C27—C22	121.1 (3)
C13—C12—C11	117.8 (3)	C26—C27—H27	119.4
C13—C12—C14	120.7 (3)	C22—C27—H27	119.4

C11—C12—C14	121.5 (3)	C26—C28—H28A	109.5
C12—C13—C8	120.9 (2)	C26—C28—H28B	109.5
C12—C13—H13	119.6	H28A—C28—H28B	109.5
C8—C13—H13	119.6	C26—C28—H28C	109.5
C12—C14—H14A	109.5	H28A—C28—H28C	109.5
C12—C14—H14B	109.5	H28B—C28—H28C	109.5
C7—C1—C2—O1	4.8 (4)	C21—C15—C16—O3	0.3 (4)
C6—C1—C2—O1	−176.5 (3)	C20—C15—C16—O3	179.4 (3)
C7—C1—C2—C3	−173.9 (3)	C21—C15—C16—C17	179.9 (2)
C6—C1—C2—C3	4.8 (4)	C20—C15—C16—C17	−1.0 (4)
O1—C2—C3—C4	176.5 (3)	O3—C16—C17—C18	179.7 (3)
C1—C2—C3—C4	−4.8 (4)	C15—C16—C17—C18	0.1 (4)
O1—C2—C3—Br1	−5.1 (4)	O3—C16—C17—Br3	−0.8 (4)
C1—C2—C3—Br1	173.57 (19)	C15—C16—C17—Br3	179.56 (19)
C2—C3—C4—C5	1.3 (4)	C16—C17—C18—C19	0.4 (4)
Br1—C3—C4—C5	−177.1 (2)	Br3—C17—C18—C19	−179.1 (2)
C3—C4—C5—C6	2.8 (4)	C17—C18—C19—C20	0.1 (4)
C3—C4—C5—Br2	−179.3 (2)	C17—C18—C19—Br4	178.4 (2)
C4—C5—C6—C1	−2.8 (4)	C18—C19—C20—C15	−1.0 (4)
Br2—C5—C6—C1	179.3 (2)	Br4—C19—C20—C15	−179.2 (2)
C7—C1—C6—C5	177.5 (3)	C21—C15—C20—C19	−179.4 (3)
C2—C1—C6—C5	−1.2 (4)	C16—C15—C20—C19	1.5 (4)
C8—N1—C7—C1	−179.5 (3)	C22—N2—C21—C15	179.5 (3)
C6—C1—C7—N1	−178.2 (3)	C20—C15—C21—N2	179.4 (3)
C2—C1—C7—N1	0.5 (4)	C16—C15—C21—N2	−1.5 (4)
C7—N1—C8—C9	−169.9 (3)	C21—N2—C22—C27	−1.3 (4)
C7—N1—C8—C13	11.6 (4)	C21—N2—C22—C23	179.6 (3)
C13—C8—C9—O2	179.5 (3)	C27—C22—C23—O4	177.6 (2)
N1—C8—C9—O2	1.0 (4)	N2—C22—C23—O4	−3.3 (4)
C13—C8—C9—C10	−0.1 (4)	C27—C22—C23—C24	−1.6 (4)
N1—C8—C9—C10	−178.6 (3)	N2—C22—C23—C24	177.5 (2)
O2—C9—C10—C11	−179.4 (3)	O4—C23—C24—C25	−178.3 (3)
C8—C9—C10—C11	0.2 (5)	C22—C23—C24—C25	0.8 (4)
C9—C10—C11—C12	−0.2 (5)	C23—C24—C25—C26	0.2 (5)
C10—C11—C12—C13	0.0 (5)	C24—C25—C26—C27	−0.3 (4)
C10—C11—C12—C14	179.2 (3)	C24—C25—C26—C28	−179.8 (3)
C11—C12—C13—C8	0.1 (4)	C25—C26—C27—C22	−0.5 (4)
C14—C12—C13—C8	−179.1 (3)	C28—C26—C27—C22	179.0 (3)
C9—C8—C13—C12	0.0 (4)	C23—C22—C27—C26	1.4 (4)
N1—C8—C13—C12	178.4 (3)	N2—C22—C27—C26	−177.6 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O3	0.90 (4)	1.86 (4)	2.728 (3)	163 (4)
O4—H4···O1	0.86 (4)	1.92 (4)	2.753 (3)	164 (3)
N1—H1···O1	0.80 (4)	1.97 (3)	2.616 (3)	139 (3)

N2—H2*A*···O3	0.81 (3)	1.99 (3)	2.632 (3)	136 (3)