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# 6-Bromo-3-hydroxy-4-oxo-2-phenyl-4*H*-chromene-8-carboxylic acid dimethylformamide disolvate

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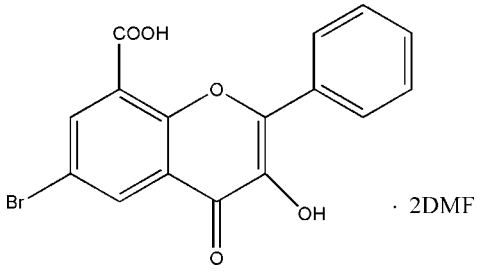
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.036; $wR$ factor = 0.098; data-to-parameter ratio = 14.3.

In the title compound, $C_{16}H_9BrO_5\cdot 2C_3H_7NO$, the chromene ring system is essentially planar. The two dimethylformamide solvent molecules are linked by intermolecular O—H···O hydrogen bonds to the 6-bromo-3-hydroxy-4-oxo-2-phenyl-4H-chromene-8-carboxylic acid molecules.

## Related literature

For related literature, see: Gills *et al.* (1980); Liu *et al.* (2007); Jin & Xiao (2005); Kagechika *et al.* (1989); Valenti *et al.* (1998); Walenta *et al.* (1991); Zwaagstra *et al.* (1996, 1998*a*,*b*).




## Experimental

### Crystal data

$C_{16}H_9BrO_5\cdot 2C_3H_7NO$
$M_r$ = 507.33
Monoclinic, $P2_1/n$
$a$ = 10.489 (2) Å
$b$ = 11.470 (2) Å
$c$ = 18.803 (4) Å
$\beta$ = 92.127 (3)°
$V$ = 2260.6 (8) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 1.86 mm$^{-1}$
$T$ = 294 (2) K
0.49 × 0.38 × 0.17 mm

### Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.462, $T_{max}$ = 0.742
14295 measured reflections
4203 independent reflections
2889 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.028

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.098
$S$ = 0.99
4203 reflections
294 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.34 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2$A$···O6 | 0.82 | 1.78 | 2.598 (2) | 173 |
| O5—H5···O7 | 0.82 | 1.89 | 2.627 (2) | 149 |
| O5—H5···O4 | 0.82 | 2.32 | 2.741 (3) | 113 |

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson,1996); *ORTEP-3 for Windows* (Farrugia, 1997); 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: DN2358).

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# supporting information

*Acta Cryst.* (2008). E**64**, o1415 [doi:10.1107/S1600536808019454]

# 6-Bromo-3-hydroxy-4-oxo-2-phenyl-4*H*-chromene-8-carboxylic acid dimethylformamide disolvate

**Hui-Liang Wen, Dan-Dan Chen and Chong-Bo Liu**

## S1. Comment

Flavonoids are widely present in nature, which have potential biological activity such as antiviral (Zwaagstra *et al.*, 1996; Zwaagstra *et al.*, 1998*a*), anticancer (Valenti *et al.*, 1998), treating leukemia (Kagechika *et al.*, 1989), antihypertensive, antimicrobial (Gills *et al.*, 1980; Walenta *et al.*, 1991) *et al.* Due to the varieties of its biological activity, the structure-activity relationships study of flavonoids carboxylic acids has been the hot spot all along. In a continuation of our recent studies of flavonoids carboxylic acids (Liu *et al.*, 2007), we report here the title compound, $C_{16}H_9BrO_5$—$C_6H_{14}N_2O_2$, (I).

In compound (I), the chromene molecule is roughly planar, with a mean deviation of 0.0521 Å. The dihedral angle between the chromene ring and the phenyl ring is 7.5 (2)°. Two O—H···O hydrogen bonds (Table 1, Fig. 1) involving the H atoms of hydroxyl group and carboxylic acid group connect the dimethylformamide molecules and 6-bromo-3-hydroxy-4-oxo-2-phenyl-4*H*-chromene-8-carboxylic acid.

## S2. Experimental

The title compound was synthesized by the ring closure of 5′-bromo-3′-carboxy-2′-hydroxychalcone under the existence of a certain oxidant, according to the route published by Zwaagstra *et al.* (Zwaagstra *et al.*, 1998*b*). Single crystals of (I) suitable for X-ray diffraction analysis were obtained from a solution in *N*,*N*-dimethylformamide.

## S3. Refinement

All H atoms attached to C atoms and O atom were fixed geometrically and treated as riding with C—H = 0.96 Å (methyl) or 0.93 Å (aromatic) and O—H = 0.82 Å with $U_{iso}(H) = 1.2U_{eq}(C_{aromatic})$ or $U_{iso}(H) = 1.5U_{eq}(C_{methyl}$ and O).

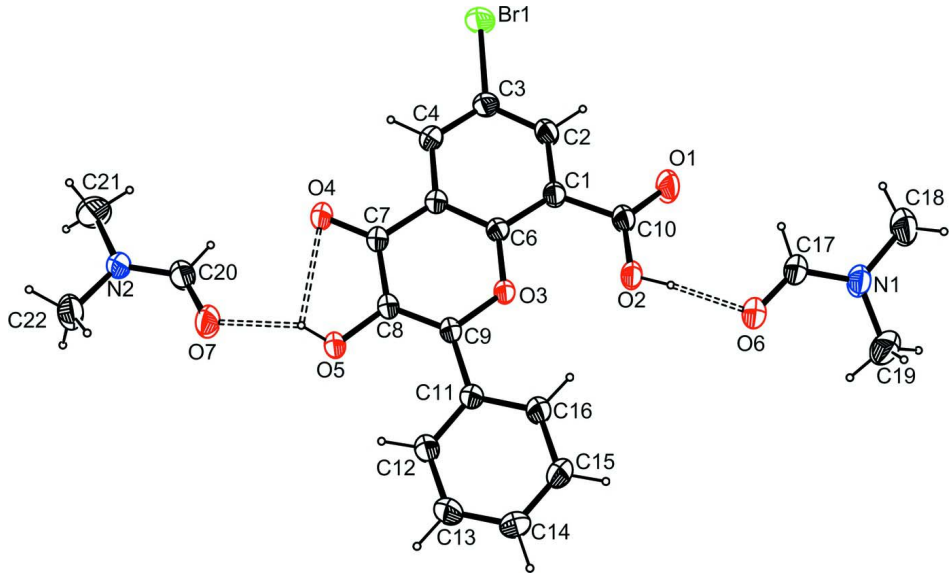


**Figure 1**

The molecular structure of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented as small spheres of arbitrary radii. The hydrogen bonds are shown as dashed lines.

### 6-Bromo-3-hydroxy-4-oxo-2-phenyl-4*H*-chromene-8-carboxylic acid dimethylformamide solvate

*Crystal data*

$C_{16}H_9BrO_5 \cdot 2C_3H_7NO$
$M_r = 507.33$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 10.489$ (2) Å
$b = 11.470$ (2) Å
$c = 18.803$ (4) Å
$\beta = 92.127$ (3)°
$V = 2260.6$ (8) Å$^3$
$Z = 4$
$F(000) = 1040$
$D_x = 1.491$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3645 reflections
$\theta = 2.6$–23.5°
$\mu = 1.86$ mm$^{-1}$
$T = 294$ K
Block, yellow
0.49 × 0.38 × 0.17 mm

*Data collection*

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.462$, $T_{max} = 0.742$
14295 measured reflections
4203 independent reflections
2889 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.6°$
$h = -12 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -22 \rightarrow 22$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.098$
$S = 1.00$
4203 reflections
294 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-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.6824P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.017$
$\Delta\rho_{max} = 0.32$ e Å$^{-3}$
$\Delta\rho_{min} = -0.35$ 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 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Br1 | 0.06013 (3) | 0.88028 (2) | 0.582768 (19) | 0.07113 (15) |
| O1 | −0.0368 (2) | 0.44074 (18) | 0.65009 (12) | 0.0911 (8) |
| O2 | 0.06856 (17) | 0.31912 (16) | 0.58290 (9) | 0.0577 (5) |
| H2A | 0.0279 | 0.2704 | 0.6046 | 0.087* |
| O3 | 0.24560 (16) | 0.40314 (13) | 0.50359 (9) | 0.0460 (4) |
| O4 | 0.41242 (18) | 0.68075 (16) | 0.41187 (10) | 0.0652 (6) |
| O5 | 0.49923 (18) | 0.46087 (15) | 0.38343 (11) | 0.0610 (5) |
| H5 | 0.5303 | 0.5237 | 0.3727 | 0.091* |
| C1 | 0.1082 (2) | 0.5216 (2) | 0.57023 (13) | 0.0439 (6) |
| C2 | 0.0684 (2) | 0.6327 (2) | 0.58763 (14) | 0.0517 (7) |
| H2 | 0.0041 | 0.6416 | 0.6199 | 0.062* |
| C3 | 0.1222 (2) | 0.7312 (2) | 0.55796 (14) | 0.0495 (7) |
| C4 | 0.2183 (2) | 0.7207 (2) | 0.51073 (14) | 0.0485 (6) |
| H4 | 0.2544 | 0.7869 | 0.4912 | 0.058* |
| C5 | 0.2615 (2) | 0.6101 (2) | 0.49221 (13) | 0.0422 (6) |
| C6 | 0.2067 (2) | 0.5112 (2) | 0.52147 (12) | 0.0411 (6) |
| C7 | 0.3634 (2) | 0.5972 (2) | 0.44191 (14) | 0.0456 (6) |
| C8 | 0.4030 (2) | 0.4787 (2) | 0.42805 (13) | 0.0443 (6) |
| C9 | 0.3421 (2) | 0.3864 (2) | 0.45729 (13) | 0.0429 (6) |
| C10 | 0.0406 (3) | 0.4222 (2) | 0.60528 (14) | 0.0518 (7) |
| C11 | 0.3633 (2) | 0.2608 (2) | 0.44578 (13) | 0.0442 (6) |
| C12 | 0.4628 (3) | 0.2186 (3) | 0.40674 (19) | 0.0821 (11) |
| H12 | 0.5188 | 0.2708 | 0.3865 | 0.099* |
| C13 | 0.4800 (4) | 0.1012 (3) | 0.3976 (2) | 0.0944 (13) |
| H13 | 0.5482 | 0.0752 | 0.3716 | 0.113* |
| C14 | 0.3998 (3) | 0.0217 (2) | 0.42550 (17) | 0.0686 (9) |
| H14 | 0.4114 | −0.0578 | 0.4184 | 0.082* |
| C15 | 0.3021 (3) | 0.0619 (2) | 0.46419 (18) | 0.0736 (9) |
| H15 | 0.2464 | 0.0089 | 0.4839 | 0.088* |
| C16 | 0.2840 (3) | 0.1793 (2) | 0.47476 (16) | 0.0629 (8) |

| | | | | |
|---|---|---|---|---|
| H16 | 0.2169 | 0.2041 | 0.5020 | 0.075* |
| N1 | −0.2166 (3) | 0.1136 (2) | 0.71663 (13) | 0.0631 (7) |
| O6 | −0.0563 (2) | 0.15290 (18) | 0.64329 (11) | 0.0722 (6) |
| C17 | −0.1516 (3) | 0.1828 (3) | 0.67549 (16) | 0.0626 (8) |
| H17 | −0.1792 | 0.2595 | 0.6703 | 0.075* |
| C18 | −0.3276 (4) | 0.1567 (3) | 0.7524 (2) | 0.0983 (13) |
| H18A | −0.3072 | 0.1649 | 0.8024 | 0.147* |
| H18B | −0.3968 | 0.1026 | 0.7456 | 0.147* |
| H18C | −0.3521 | 0.2311 | 0.7329 | 0.147* |
| C19 | −0.1781 (3) | −0.0070 (3) | 0.72818 (18) | 0.0840 (10) |
| H19A | −0.2251 | −0.0565 | 0.6955 | 0.126* |
| H19B | −0.1953 | −0.0294 | 0.7761 | 0.126* |
| H19C | −0.0885 | −0.0147 | 0.7206 | 0.126* |
| N2 | 0.7254 (2) | 0.78411 (18) | 0.28823 (12) | 0.0554 (6) |
| O7 | 0.6435 (2) | 0.60874 (17) | 0.31631 (12) | 0.0773 (7) |
| C20 | 0.6539 (3) | 0.7141 (3) | 0.32545 (16) | 0.0642 (8) |
| H20 | 0.6076 | 0.7473 | 0.3615 | 0.077* |
| C21 | 0.7365 (4) | 0.9061 (3) | 0.3036 (3) | 0.1108 (15) |
| H21A | 0.6909 | 0.9239 | 0.3456 | 0.166* |
| H21B | 0.7012 | 0.9503 | 0.2642 | 0.166* |
| H21C | 0.8248 | 0.9260 | 0.3113 | 0.166* |
| C22 | 0.8037 (3) | 0.7385 (3) | 0.23293 (16) | 0.0746 (9) |
| H22A | 0.8853 | 0.7160 | 0.2532 | 0.112* |
| H22B | 0.8151 | 0.7974 | 0.1975 | 0.112* |
| H22C | 0.7626 | 0.6718 | 0.2114 | 0.112* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br1 | 0.0723 (2) | 0.04462 (18) | 0.0983 (3) | 0.00670 (14) | 0.02689 (18) | −0.00929 (15) |
| O1 | 0.1120 (19) | 0.0564 (13) | 0.1106 (18) | −0.0065 (12) | 0.0794 (16) | −0.0021 (12) |
| O2 | 0.0641 (13) | 0.0440 (11) | 0.0672 (12) | −0.0095 (9) | 0.0321 (10) | 0.0013 (9) |
| O3 | 0.0491 (10) | 0.0358 (9) | 0.0546 (10) | −0.0028 (7) | 0.0219 (8) | 0.0004 (7) |
| O4 | 0.0696 (14) | 0.0428 (11) | 0.0860 (14) | −0.0029 (9) | 0.0389 (11) | 0.0106 (10) |
| O5 | 0.0621 (13) | 0.0421 (10) | 0.0814 (13) | −0.0017 (9) | 0.0392 (10) | 0.0058 (9) |
| C1 | 0.0440 (15) | 0.0429 (14) | 0.0454 (14) | −0.0045 (11) | 0.0098 (12) | 0.0000 (11) |
| C2 | 0.0483 (16) | 0.0522 (16) | 0.0557 (16) | −0.0010 (12) | 0.0179 (13) | −0.0043 (12) |
| C3 | 0.0501 (16) | 0.0402 (14) | 0.0587 (17) | 0.0007 (12) | 0.0073 (13) | −0.0050 (12) |
| C4 | 0.0486 (16) | 0.0375 (13) | 0.0600 (17) | −0.0043 (11) | 0.0085 (13) | 0.0032 (12) |
| C5 | 0.0411 (14) | 0.0389 (13) | 0.0472 (14) | −0.0012 (11) | 0.0078 (11) | 0.0025 (11) |
| C6 | 0.0411 (14) | 0.0372 (13) | 0.0455 (14) | −0.0011 (11) | 0.0080 (11) | −0.0015 (11) |
| C7 | 0.0425 (15) | 0.0412 (14) | 0.0540 (15) | −0.0033 (11) | 0.0125 (12) | 0.0060 (11) |
| C8 | 0.0413 (15) | 0.0429 (14) | 0.0494 (14) | −0.0025 (11) | 0.0131 (12) | 0.0040 (11) |
| C9 | 0.0401 (14) | 0.0432 (13) | 0.0460 (14) | −0.0022 (11) | 0.0116 (11) | 0.0022 (11) |
| C10 | 0.0528 (17) | 0.0501 (15) | 0.0537 (16) | −0.0041 (13) | 0.0197 (14) | −0.0009 (12) |
| C11 | 0.0448 (15) | 0.0383 (13) | 0.0500 (15) | −0.0009 (11) | 0.0102 (12) | −0.0001 (11) |
| C12 | 0.083 (2) | 0.0446 (16) | 0.123 (3) | −0.0012 (15) | 0.061 (2) | 0.0002 (17) |
| C13 | 0.099 (3) | 0.0519 (19) | 0.137 (3) | 0.0085 (18) | 0.070 (3) | −0.0067 (19) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C14 | 0.079 (2) | 0.0396 (16) | 0.089 (2) | 0.0024 (15) | 0.0210 (18) | −0.0071 (15) |
| C15 | 0.085 (2) | 0.0397 (15) | 0.099 (2) | −0.0087 (16) | 0.0338 (19) | 0.0043 (16) |
| C16 | 0.069 (2) | 0.0440 (15) | 0.078 (2) | −0.0023 (14) | 0.0344 (16) | −0.0023 (14) |
| N1 | 0.0694 (17) | 0.0582 (15) | 0.0630 (15) | −0.0122 (12) | 0.0213 (13) | 0.0075 (11) |
| O6 | 0.0710 (15) | 0.0588 (12) | 0.0889 (15) | −0.0066 (10) | 0.0320 (12) | 0.0131 (11) |
| C17 | 0.064 (2) | 0.0532 (17) | 0.072 (2) | −0.0080 (15) | 0.0209 (16) | 0.0106 (15) |
| C18 | 0.096 (3) | 0.088 (3) | 0.114 (3) | −0.014 (2) | 0.057 (2) | 0.004 (2) |
| C19 | 0.104 (3) | 0.064 (2) | 0.084 (2) | −0.0043 (19) | 0.016 (2) | 0.0247 (18) |
| N2 | 0.0544 (14) | 0.0404 (12) | 0.0727 (15) | −0.0045 (10) | 0.0200 (12) | 0.0015 (11) |
| O7 | 0.0906 (17) | 0.0485 (13) | 0.0959 (16) | −0.0178 (10) | 0.0453 (13) | −0.0037 (10) |
| C20 | 0.065 (2) | 0.0550 (18) | 0.075 (2) | −0.0054 (15) | 0.0276 (16) | −0.0033 (15) |
| C21 | 0.114 (3) | 0.0420 (18) | 0.180 (4) | −0.0070 (19) | 0.056 (3) | −0.007 (2) |
| C22 | 0.087 (2) | 0.072 (2) | 0.067 (2) | −0.0156 (17) | 0.0320 (18) | −0.0041 (16) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Br1—C3 | 1.894 (2) | C14—C15 | 1.359 (4) |
| O1—C10 | 1.210 (3) | C14—H14 | 0.9300 |
| O2—C10 | 1.293 (3) | C15—C16 | 1.376 (4) |
| O2—H2A | 0.8200 | C15—H15 | 0.9300 |
| O3—C6 | 1.351 (3) | C16—H16 | 0.9300 |
| O3—C9 | 1.372 (3) | N1—C17 | 1.316 (3) |
| O4—C7 | 1.235 (3) | N1—C18 | 1.453 (4) |
| O5—C8 | 1.352 (3) | N1—C19 | 1.455 (4) |
| O5—H5 | 0.8200 | O6—C17 | 1.236 (3) |
| C1—C2 | 1.384 (3) | C17—H17 | 0.9300 |
| C1—C6 | 1.412 (3) | C18—H18A | 0.9600 |
| C1—C10 | 1.507 (3) | C18—H18B | 0.9600 |
| C2—C3 | 1.389 (4) | C18—H18C | 0.9600 |
| C2—H2 | 0.9300 | C19—H19A | 0.9600 |
| C3—C4 | 1.373 (3) | C19—H19B | 0.9600 |
| C4—C5 | 1.396 (3) | C19—H19C | 0.9600 |
| C4—H4 | 0.9300 | N2—C20 | 1.318 (3) |
| C5—C6 | 1.394 (3) | N2—C21 | 1.433 (4) |
| C5—C7 | 1.460 (3) | N2—C22 | 1.447 (3) |
| C7—C8 | 1.447 (3) | O7—C20 | 1.225 (3) |
| C8—C9 | 1.363 (3) | C20—H20 | 0.9300 |
| C9—C11 | 1.475 (3) | C21—H21A | 0.9600 |
| C11—C16 | 1.377 (3) | C21—H21B | 0.9600 |
| C11—C12 | 1.386 (4) | C21—H21C | 0.9600 |
| C12—C13 | 1.371 (4) | C22—H22A | 0.9600 |
| C12—H12 | 0.9300 | C22—H22B | 0.9600 |
| C13—C14 | 1.359 (4) | C22—H22C | 0.9600 |
| C13—H13 | 0.9300 | | |
| | | | |
| C10—O2—H2A | 109.5 | C13—C14—H14 | 121.0 |
| C6—O3—C9 | 121.48 (18) | C14—C15—C16 | 121.4 (3) |
| C8—O5—H5 | 109.5 | C14—C15—H15 | 119.3 |

| | | | |
|---|---|---|---|
| C2—C1—C6 | 117.8 (2) | C16—C15—H15 | 119.3 |
| C2—C1—C10 | 116.2 (2) | C15—C16—C11 | 121.2 (3) |
| C6—C1—C10 | 126.0 (2) | C15—C16—H16 | 119.4 |
| C1—C2—C3 | 121.6 (2) | C11—C16—H16 | 119.4 |
| C1—C2—H2 | 119.2 | C17—N1—C18 | 120.6 (3) |
| C3—C2—H2 | 119.2 | C17—N1—C19 | 120.9 (3) |
| C4—C3—C2 | 120.5 (2) | C18—N1—C19 | 118.5 (3) |
| C4—C3—Br1 | 120.38 (19) | O6—C17—N1 | 124.5 (3) |
| C2—C3—Br1 | 119.14 (19) | O6—C17—H17 | 117.7 |
| C3—C4—C5 | 119.6 (2) | N1—C17—H17 | 117.7 |
| C3—C4—H4 | 120.2 | N1—C18—H18A | 109.5 |
| C5—C4—H4 | 120.2 | N1—C18—H18B | 109.5 |
| C6—C5—C4 | 119.9 (2) | H18A—C18—H18B | 109.5 |
| C6—C5—C7 | 119.7 (2) | N1—C18—H18C | 109.5 |
| C4—C5—C7 | 120.4 (2) | H18A—C18—H18C | 109.5 |
| O3—C6—C5 | 121.0 (2) | H18B—C18—H18C | 109.5 |
| O3—C6—C1 | 118.3 (2) | N1—C19—H19A | 109.5 |
| C5—C6—C1 | 120.6 (2) | N1—C19—H19B | 109.5 |
| O4—C7—C8 | 121.3 (2) | H19A—C19—H19B | 109.5 |
| O4—C7—C5 | 123.0 (2) | N1—C19—H19C | 109.5 |
| C8—C7—C5 | 115.7 (2) | H19A—C19—H19C | 109.5 |
| O5—C8—C9 | 120.3 (2) | H19B—C19—H19C | 109.5 |
| O5—C8—C7 | 118.7 (2) | C20—N2—C21 | 122.1 (3) |
| C9—C8—C7 | 120.9 (2) | C20—N2—C22 | 120.7 (2) |
| C8—C9—O3 | 121.0 (2) | C21—N2—C22 | 117.0 (2) |
| C8—C9—C11 | 128.6 (2) | O7—C20—N2 | 125.2 (3) |
| O3—C9—C11 | 110.38 (19) | O7—C20—H20 | 117.4 |
| O1—C10—O2 | 123.6 (2) | N2—C20—H20 | 117.4 |
| O1—C10—C1 | 120.7 (2) | N2—C21—H21A | 109.5 |
| O2—C10—C1 | 115.7 (2) | N2—C21—H21B | 109.5 |
| C16—C11—C12 | 116.8 (2) | H21A—C21—H21B | 109.5 |
| C16—C11—C9 | 120.6 (2) | N2—C21—H21C | 109.5 |
| C12—C11—C9 | 122.7 (2) | H21A—C21—H21C | 109.5 |
| C13—C12—C11 | 121.0 (3) | H21B—C21—H21C | 109.5 |
| C13—C12—H12 | 119.5 | N2—C22—H22A | 109.5 |
| C11—C12—H12 | 119.5 | N2—C22—H22B | 109.5 |
| C14—C13—C12 | 121.6 (3) | H22A—C22—H22B | 109.5 |
| C14—C13—H13 | 119.2 | N2—C22—H22C | 109.5 |
| C12—C13—H13 | 119.2 | H22A—C22—H22C | 109.5 |
| C15—C14—C13 | 118.0 (3) | H22B—C22—H22C | 109.5 |
| C15—C14—H14 | 121.0 | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2*A*···O6 | 0.82 | 1.78 | 2.598 (2) | 173 |
| O5—H5···O7 | 0.82 | 1.89 | 2.627 (2) | 149 |
| O5—H5···O4 | 0.82 | 2.32 | 2.741 (3) | 113 |