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# Crystal structure of 7-bromo-4-oxo-4*H*-chromene-3-carbaldehyde

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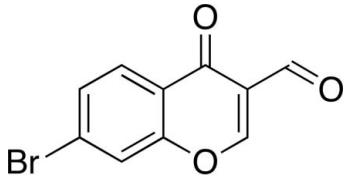
In the title compound, $C_{10}H_5BrO_3$, a brominated 3-formylchromone derivative, all atoms are essentially coplanar (r.m.s. = 0.0631 Å for the non-H atoms), with the largest deviation from the least-squares plane [0.215 (3) Å] being for the formyl O atom. In the crystal, molecules are linked into tapes through C—H···O hydrogen bonds and these tapes are assembled by stacking interactions [centroid–centroid distance between the pyran rings = 3.858 (3) Å] to form supramolecular layers that stack along the *c* axis.

**Keywords:** crystal structure; chromone; C—H···O hydrogen bonding; stacking interaction.

**CCDC reference**: 1018275

## 1. Related literature

For related structures, see: Ishikawa (2014*a*,*b*). For halogen bonding, see: Auffinger *et al.* (2004); Metrangolo *et al.* (2005); Wilcken *et al.* (2013); Sirimulla *et al.* (2013). For halogen–halogen interactions, see: Metrangolo & Resnati (2014); Mukherjee & Desiraju (2014).




## 2. Experimental

### 2.1. Crystal data

$C_{10}H_5BrO_3$
$M_r$ = 253.05
Monoclinic, $P2_1/c$
$a$ = 3.8580 (18) Å
$b$ = 6.054 (4) Å
$c$ = 37.268 (13) Å
$\beta$ = 90.39 (4)°
$V$ = 870.4 (8) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 4.71 mm$^{-1}$
$T$ = 100 K
0.45 × 0.20 × 0.10 mm

### 2.2. Data collection

Rigaku AFC-7R diffractometer
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.339, $T_{max}$ = 0.624
4817 measured reflections
1980 independent reflections
1710 reflections with $F^2 > 2\sigma(F^2)$
$R_{int}$ = 0.024
3 standard reflections
every 150 reflections
intensity decay: 4.8%

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.109
$S$ = 1.07
1980 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.26 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.73 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C7$^{i}$—H4$^{i}$···O2 | 0.95 | 2.30 | 3.149 (4) | 149 (1) |
| C1$^{ii}$—H1$^{ii}$···O3 | 0.95 | 2.37 | 3.228 (5) | 149 (1) |

Symmetry codes: (i) $x+1, y+1, z$; (ii) $-x, -y+1, -z+1$.

Data collection: *WinAFC Diffractometer Control Software* (Rigaku, 1999); cell refinement: *WinAFC Diffractometer Control Software*; data reduction: *WinAFC Diffractometer Control Software*; program(s) used to solve structure: *SIR2008* (Burla *et al.*, 2007); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku, 2010); software used to prepare material for publication: *CrystalStructure*.

## Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5337).

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# supporting information

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## Crystal structure of 7-bromo-4-oxo-4*H*-chromene-3-carbaldehyde

**Yoshinobu Ishikawa**

### S1. Structural commentary

Halogen bonding and halogen···halogen interaction have recently attracted much attention in medicinal chemistry, chemical biology, supramolecular chemistry and crystal engineering (Auffinger *et al.*, 2004, Metrangolo *et al*., 2005, Wilcken *et al.*, 2013, Sirimulla *et al.*, 2013, Mukherjee & Desiraju, 2014, Metrangolo & Resnati, 2014). We have recently reported the crystal structures of a dibrominated 3-formylchromone derivative 6,8-dibromo-4-oxo-4*H*-chromene-3-carbaldehyde (Ishikawa, 2014*a*) and a monobrominated 3-formylchromone derivative 6-bromo-4-oxo-4*H*-chromene-3-carbaldehyde (Ishikawa, 2014*b*). Halogen bonding between the formyl oxygen atom and the bromine atom at 8-position and type II halogen···halogen interaction between the bromine atoms at 6-position are observed in 6,8-dibromo-4-oxo-4*H*-chromene-3-carbaldehyde (Fig.3, top). On the other hand, halogen bonding between the formyl oxygen atom and the bromine atom at 6-position is found in 6-bromo-4-oxo-4*H*-chromene-3-carbaldehyde (Fig.3, middle). As part of our interest in these types of chemical bonding, we herein report the crystal structure of a monobrominated 3-formyl-chromone derivative 7-bromo-4-oxo-4*H*-chromene-3-carbaldehyde.

The objective of this study is to reveal whether a short contact is found for the bromine atom at 7-position. The mean deviation of the least-square planes for the non-hydrogen atoms is 0.0631 Å, and the largest deviation is 0.215 (3) Å for the formyl O3 atom (Fig. 1). In the crystal, the molecules are linked through C–H···O hydrogen bonds between the translation-symmetry[i] and inversion-symmetry equivalents[ii,iii] to form tapes [i: $x + 1$, $y + 1$, $z$, ii: $-x$, $-y + 1$, $-z + 1$, iii: $-x + 1$, $-y + 2$, $-z + 1$], which are further assembled by stacking interactions [centroid–centroid distance between the pyran rings of the 4*H*-chromene units = 3.858 (3) Å], as shown in Fig. 2. A short contact for the bromine atom at 7-position is not observed (Fig. 3, bottom).

### S2. Synthesis and crystallization

To a solution of 4-bromo-2-hydroxyacetophenone (4.7 mmol) in *N*,*N*-dimethylformamide (15 ml) was added drop-wise $POCl_3$ (11.6 mmol) at 0 °C. After the mixture was stirred for 14 h at room temperature, water (50 ml) was added. The precipitates were collected, washed with water, and dried *in vacuo* (yield: 84%). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ = 7.48 (d, 1H, $J$ = 8.8 Hz), 7.57 (s, 1H), 8.24 (d, 1H, $J$ = 8.8 Hz), 8.52 (s, 1H), 10.37 (s, 1H). DART-MS calcd for [$C_{10}H_5BrO_3$ + $H^+$]: 252.950, found 252.981. Single crystals suitable for X-ray diffraction were obtained from a 1,2-dichloro-ethane/cyclohexane solution of the title compound at room temperature.

### S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The C($sp^2$)-bound hydrogen atoms were placed in geometrical positions [C–H 0.95 Å, $U_{iso}$(H) = 1.2$U_{eq}$(C)], and refined using a riding model.

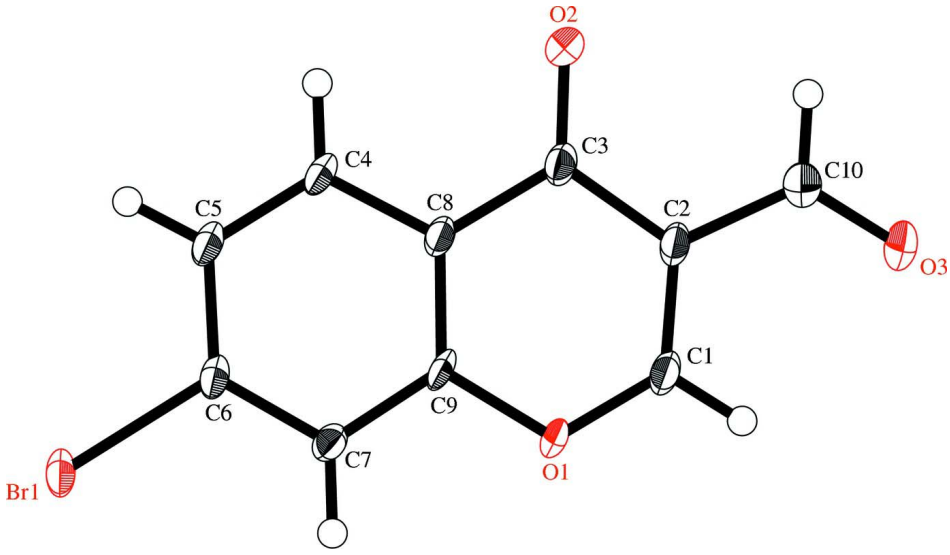


**Figure 1**

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level. Hydrogen atoms are shown as small spheres of arbitrary radius.

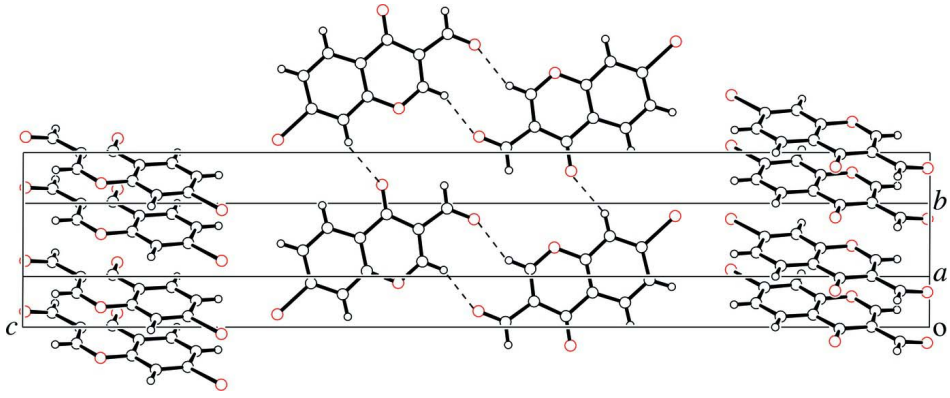


**Figure 2**

A packing view of the title compound. C—H···O hydrogen bonds are represented by dashed lines.

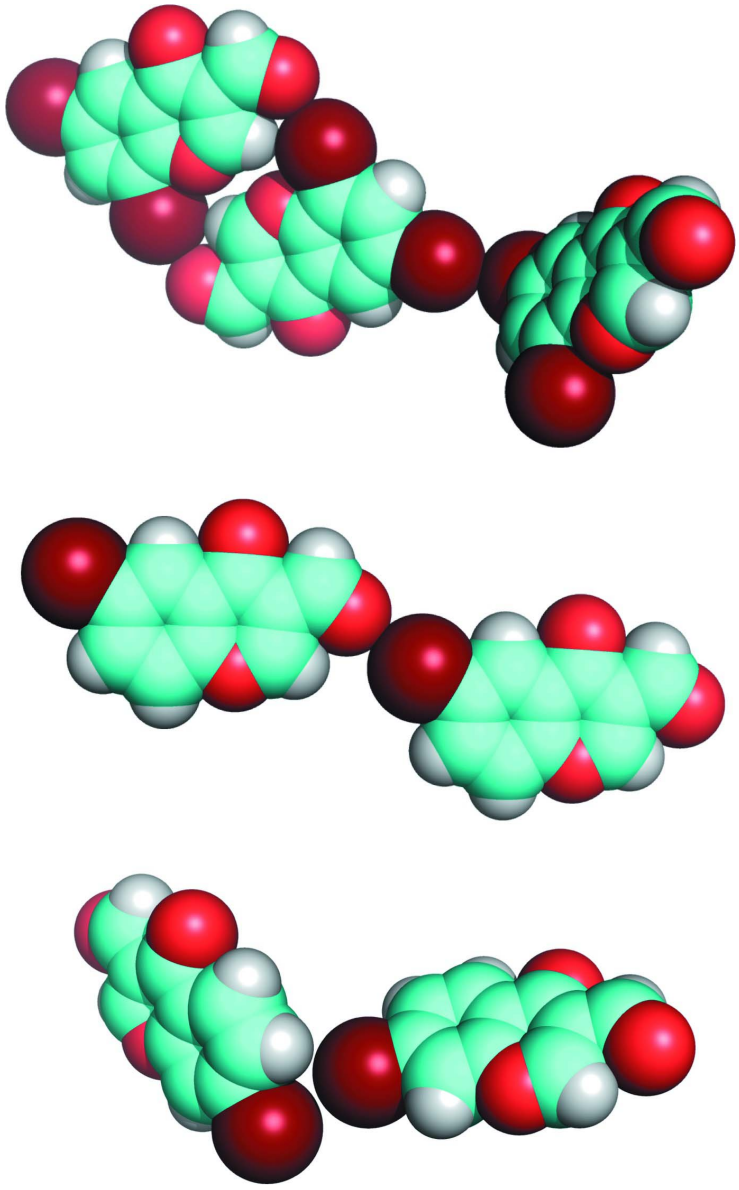

**Figure 3**

Sphere models of the crystal structures of 6,8-dibromo-4-oxo-4*H*-chromene-3-carbaldehyde (top, Ishikawa, 2014*a*), 6-bromo-4-oxo-4*H*-chromene-3-carbaldehyde (middle, Ishikawa, 2014*b*), and the title compound (bottom, this work).

### 7-Bromo-4-oxo-4*H*-chromene-3-carbaldehyde

*Crystal data*

$C_{10}H_5BrO_3$
$M_r = 253.05$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 3.8580\ (18)$ Å
$b = 6.054\ (4)$ Å
$c = 37.268\ (13)$ Å
$\beta = 90.39\ (4)°$
$V = 870.4\ (8)$ Å$^3$
$Z = 4$
$F(000) = 496.00$
$D_x = 1.931$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 15.3–17.5°$
$\mu = 4.71$ mm$^{-1}$

$T$ = 100 K
Plate, colourless

0.45 × 0.20 × 0.10 mm

*Data collection*

Rigaku AFC-7R diffractometer
$\omega$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.339, $T_{max}$ = 0.624
4817 measured reflections
1980 independent reflections

1710 reflections with $F^2 > 2\sigma(F^2)$
$R_{int}$ = 0.024
$\theta_{max}$ = 27.5°
$h$ = −5→2
$k$ = −7→7
$l$ = −48→48
3 standard reflections every 150 reflections
intensity decay: 4.8%

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.109
$S$ = 1.07
1980 reflections
127 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
$w = 1/[\sigma^2(F_o^2) + (0.0756P)^2 + 0.989P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 1.26 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.73 e Å$^{-3}$

*Special details*

**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 | 0.08234 (8) | 0.06978 (6) | 0.719199 (7) | 0.01987 (15) |
| O1 | 0.1286 (6) | 0.3047 (4) | 0.58604 (6) | 0.0198 (5) |
| O2 | 0.6540 (7) | 0.8813 (4) | 0.60418 (6) | 0.0253 (6) |
| O3 | 0.2989 (8) | 0.7487 (5) | 0.50270 (6) | 0.0321 (6) |
| C1 | 0.1969 (9) | 0.4490 (6) | 0.55957 (8) | 0.0199 (7) |
| C2 | 0.3614 (9) | 0.6437 (6) | 0.56382 (8) | 0.0190 (7) |
| C3 | 0.4862 (8) | 0.7122 (6) | 0.59908 (8) | 0.0181 (6) |
| C4 | 0.4730 (9) | 0.6080 (6) | 0.66421 (8) | 0.0173 (6) |
| C5 | 0.3830 (9) | 0.4628 (6) | 0.69123 (8) | 0.0187 (7) |
| C6 | 0.2140 (8) | 0.2670 (6) | 0.68222 (8) | 0.0167 (6) |
| C7 | 0.1322 (8) | 0.2111 (6) | 0.64716 (8) | 0.0170 (6) |
| C8 | 0.3930 (9) | 0.5598 (5) | 0.62828 (8) | 0.0169 (7) |
| C9 | 0.2221 (8) | 0.3622 (6) | 0.62059 (8) | 0.0164 (6) |
| C10 | 0.4165 (9) | 0.7874 (7) | 0.53244 (9) | 0.0256 (8) |
| H1 | 0.1233 | 0.4106 | 0.5360 | 0.0238* |
| H2 | 0.5900 | 0.7415 | 0.6700 | 0.0207* |
| H3 | 0.4357 | 0.4961 | 0.7156 | 0.0224* |
| H4 | 0.0197 | 0.0759 | 0.6415 | 0.0203* |
| H5 | 0.5512 | 0.9174 | 0.5356 | 0.0307* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br1 | 0.0190 (3) | 0.0305 (3) | 0.01010 (19) | −0.00138 (11) | 0.00019 (12) | 0.00367 (11) |
| O1 | 0.0260 (12) | 0.0252 (12) | 0.0083 (10) | −0.0074 (10) | −0.0036 (9) | −0.0010 (9) |
| O2 | 0.0336 (14) | 0.0259 (12) | 0.0162 (11) | −0.0107 (11) | −0.0033 (10) | −0.0010 (10) |
| O3 | 0.0437 (16) | 0.0392 (15) | 0.0133 (12) | −0.0131 (12) | −0.0067 (11) | 0.0046 (11) |
| C1 | 0.0209 (16) | 0.0291 (18) | 0.0095 (14) | −0.0034 (13) | −0.0018 (12) | −0.0010 (12) |
| C2 | 0.0214 (16) | 0.0261 (16) | 0.0096 (13) | −0.0028 (13) | −0.0008 (12) | 0.0006 (12) |
| C3 | 0.0197 (15) | 0.0240 (15) | 0.0107 (14) | −0.0011 (12) | 0.0011 (11) | −0.0015 (12) |
| C4 | 0.0175 (15) | 0.0245 (16) | 0.0098 (14) | −0.0003 (12) | −0.0021 (11) | −0.0049 (12) |
| C5 | 0.0192 (16) | 0.0268 (17) | 0.0099 (14) | −0.0008 (12) | −0.0015 (12) | −0.0030 (12) |
| C6 | 0.0149 (14) | 0.0260 (16) | 0.0091 (13) | 0.0001 (12) | −0.0003 (10) | 0.0001 (12) |
| C7 | 0.0154 (14) | 0.0226 (15) | 0.0128 (14) | −0.0035 (12) | −0.0017 (11) | −0.0022 (12) |
| C8 | 0.0179 (15) | 0.0231 (16) | 0.0095 (14) | −0.0023 (12) | −0.0013 (12) | −0.0014 (11) |
| C9 | 0.0175 (15) | 0.0247 (15) | 0.0070 (13) | −0.0010 (12) | −0.0022 (11) | −0.0043 (12) |
| C10 | 0.0299 (19) | 0.0299 (17) | 0.0171 (16) | −0.0078 (15) | −0.0006 (14) | 0.0021 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Br1—C6 | 1.895 (4) | C4—C8 | 1.403 (5) |
| O1—C1 | 1.345 (4) | C5—C6 | 1.393 (5) |
| O1—C9 | 1.379 (4) | C6—C7 | 1.384 (5) |
| O2—C3 | 1.226 (4) | C7—C9 | 1.394 (5) |
| O3—C10 | 1.217 (5) | C8—C9 | 1.395 (5) |
| C1—C2 | 1.348 (5) | C1—H1 | 0.950 |
| C2—C3 | 1.457 (5) | C4—H2 | 0.950 |
| C2—C10 | 1.474 (5) | C5—H3 | 0.950 |
| C3—C8 | 1.473 (5) | C7—H4 | 0.950 |
| C4—C5 | 1.382 (5) | C10—H5 | 0.950 |
| | | | |
| O1···C3 | 2.866 (5) | C10···H1 | 2.5501 |
| O2···C1 | 3.561 (5) | H1···H5 | 3.4843 |
| O2···C4 | 2.873 (4) | H2···H3 | 2.3357 |
| O2···C10 | 2.877 (5) | Br1···H2$^{i}$ | 3.2968 |
| O3···C1 | 2.820 (4) | Br1···H2$^{ii}$ | 3.3454 |
| C1···C7 | 3.578 (5) | Br1···H3$^{iii}$ | 3.5917 |
| C1···C8 | 2.748 (5) | Br1···H3$^{x}$ | 3.1886 |
| C2···C9 | 2.772 (5) | Br1···H3$^{xi}$ | 3.0827 |
| C4···C7 | 2.810 (5) | O1···H5$^{ii}$ | 3.4250 |
| C5···C9 | 2.769 (5) | O2···H4$^{iv}$ | 3.0581 |
| C6···C8 | 2.771 (5) | O2···H4$^{v}$ | 2.2984 |
| O1···O2$^{i}$ | 3.224 (4) | O3···H1$^{ix}$ | 2.3733 |
| O1···O2$^{ii}$ | 3.334 (4) | O3···H1$^{vii}$ | 2.8324 |
| O1···C3$^{iii}$ | 3.533 (5) | O3···H5$^{iii}$ | 3.3041 |
| O2···O1$^{iv}$ | 3.334 (4) | O3···H5$^{viii}$ | 2.5428 |
| O2···O1$^{v}$ | 3.224 (4) | C2···H1$^{vi}$ | 3.4277 |
| O2···C2$^{vi}$ | 3.440 (5) | C3···H4$^{iv}$ | 3.2588 |

| | | | |
|---|---|---|---|
| O2···C3$^{vi}$ | 3.376 (5) | C3···H4$^{v}$ | 3.3965 |
| O2···C7$^{iv}$ | 3.263 (4) | C4···H2$^{iii}$ | 3.5093 |
| O2···C7$^{v}$ | 3.149 (4) | C4···H4$^{iv}$ | 3.4320 |
| O2···C8$^{vi}$ | 3.562 (5) | C5···H2$^{iii}$ | 3.5776 |
| O2···C9$^{iv}$ | 3.412 (5) | C6···H2$^{ii}$ | 3.5270 |
| O3···O3$^{vii}$ | 3.394 (5) | C6···H3$^{iii}$ | 3.5409 |
| O3···O3$^{viii}$ | 3.422 (5) | C7···H2$^{ii}$ | 3.4509 |
| O3···C1$^{ix}$ | 3.228 (5) | C7···H4$^{vi}$ | 3.5285 |
| O3···C1$^{vii}$ | 3.265 (5) | C8···H4$^{iv}$ | 3.4764 |
| O3···C10$^{iii}$ | 3.595 (5) | C10···H1$^{vi}$ | 3.5572 |
| O3···C10$^{viii}$ | 3.290 (5) | C10···H1$^{ix}$ | 3.4940 |
| C1···O3$^{ix}$ | 3.228 (5) | C10···H1$^{vii}$ | 3.3402 |
| C1···O3$^{vii}$ | 3.265 (5) | C10···H5$^{iii}$ | 3.4327 |
| C1···C2$^{iii}$ | 3.437 (5) | C10···H5$^{viii}$ | 3.1048 |
| C1···C3$^{iii}$ | 3.504 (5) | H1···O3$^{ix}$ | 2.3733 |
| C2···O2$^{iii}$ | 3.440 (5) | H1···O3$^{vii}$ | 2.8324 |
| C2···C1$^{vi}$ | 3.437 (5) | H1···C2$^{iii}$ | 3.4277 |
| C3···O1$^{vi}$ | 3.533 (5) | H1···C10$^{iii}$ | 3.5572 |
| C3···O2$^{iii}$ | 3.376 (5) | H1···C10$^{ix}$ | 3.4940 |
| C3···C1$^{vi}$ | 3.504 (5) | H1···C10$^{vii}$ | 3.3402 |
| C4···C6$^{vi}$ | 3.586 (5) | H1···H1$^{ix}$ | 3.0401 |
| C4···C7$^{vi}$ | 3.559 (5) | H1···H5$^{ii}$ | 3.4115 |
| C5···C6$^{vi}$ | 3.437 (5) | H1···H5$^{vii}$ | 3.5617 |
| C6···C4$^{iii}$ | 3.586 (5) | H2···Br1$^{iv}$ | 3.3454 |
| C6···C5$^{iii}$ | 3.437 (5) | H2···Br1$^{v}$ | 3.2968 |
| C7···O2$^{i}$ | 3.149 (4) | H2···C4$^{vi}$ | 3.5093 |
| C7···O2$^{ii}$ | 3.263 (4) | H2···C5$^{vi}$ | 3.5776 |
| C7···C4$^{iii}$ | 3.559 (5) | H2···C6$^{iv}$ | 3.5270 |
| C8···O2$^{iii}$ | 3.562 (5) | H2···C7$^{iv}$ | 3.4509 |
| C8···C9$^{vi}$ | 3.429 (5) | H2···H4$^{iv}$ | 3.1684 |
| C9···O2$^{ii}$ | 3.412 (5) | H2···H4$^{v}$ | 2.8291 |
| C9···C8$^{iii}$ | 3.429 (5) | H3···Br1$^{vi}$ | 3.5917 |
| C10···O3$^{vi}$ | 3.595 (5) | H3···Br1$^{xii}$ | 3.1886 |
| C10···O3$^{viii}$ | 3.290 (5) | H3···Br1$^{xiii}$ | 3.0827 |
| C10···C10$^{viii}$ | 3.593 (6) | H3···C6$^{vi}$ | 3.5409 |
| Br1···H3 | 2.9226 | H4···O2$^{i}$ | 2.2984 |
| Br1···H4 | 2.9055 | H4···O2$^{ii}$ | 3.0581 |
| O1···H4 | 2.5249 | H4···C3$^{i}$ | 3.3965 |
| O2···H2 | 2.6093 | H4···C3$^{ii}$ | 3.2588 |
| O2···H5 | 2.5934 | H4···C4$^{ii}$ | 3.4320 |
| O3···H1 | 2.4901 | H4···C7$^{iii}$ | 3.5285 |
| C1···H5 | 3.2749 | H4···C8$^{ii}$ | 3.4764 |
| C3···H1 | 3.2825 | H4···H2$^{i}$ | 2.8291 |
| C3···H2 | 2.6775 | H4···H2$^{ii}$ | 3.1684 |
| C3···H5 | 2.6858 | H5···O1$^{iv}$ | 3.4250 |
| C5···H4 | 3.2949 | H5···O3$^{vi}$ | 3.3041 |
| C6···H2 | 3.2513 | H5···O3$^{viii}$ | 2.5428 |
| C7···H3 | 3.2879 | H5···C10$^{vi}$ | 3.4327 |

| | | | |
|---|---|---|---|
| C8···H3 | 3.2793 | H5···C10$^{viii}$ | 3.1048 |
| C8···H4 | 3.3027 | H5···H1$^{iv}$ | 3.4115 |
| C9···H1 | 3.1864 | H5···H1$^{vii}$ | 3.5617 |
| C9···H2 | 3.2628 | H5···H5$^{viii}$ | 2.8597 |

| | | | |
|---|---|---|---|
| C1—O1—C9 | 118.0 (3) | C4—C8—C9 | 118.4 (3) |
| O1—C1—C2 | 125.2 (3) | O1—C9—C7 | 115.7 (3) |
| C1—C2—C3 | 120.5 (3) | O1—C9—C8 | 121.9 (3) |
| C1—C2—C10 | 119.6 (3) | C7—C9—C8 | 122.4 (3) |
| C3—C2—C10 | 120.0 (3) | O3—C10—C2 | 123.6 (4) |
| O2—C3—C2 | 123.3 (3) | O1—C1—H1 | 117.410 |
| O2—C3—C8 | 122.7 (3) | C2—C1—H1 | 117.401 |
| C2—C3—C8 | 114.0 (3) | C5—C4—H2 | 119.709 |
| C5—C4—C8 | 120.6 (3) | C8—C4—H2 | 119.732 |
| C4—C5—C6 | 119.0 (3) | C4—C5—H3 | 120.507 |
| Br1—C6—C5 | 119.2 (3) | C6—C5—H3 | 120.495 |
| Br1—C6—C7 | 118.2 (3) | C6—C7—H4 | 121.481 |
| C5—C6—C7 | 122.6 (3) | C9—C7—H4 | 121.508 |
| C6—C7—C9 | 117.0 (3) | O3—C10—H5 | 118.165 |
| C3—C8—C4 | 121.4 (3) | C2—C10—H5 | 118.205 |
| C3—C8—C9 | 120.2 (3) | | |

| | | | |
|---|---|---|---|
| C1—O1—C9—C7 | 177.4 (3) | C8—C4—C5—C6 | 0.5 (5) |
| C1—O1—C9—C8 | −2.2 (4) | C8—C4—C5—H3 | −179.5 |
| C9—O1—C1—C2 | 2.5 (5) | H2—C4—C5—C6 | −179.5 |
| C9—O1—C1—H1 | −177.5 | H2—C4—C5—H3 | 0.5 |
| O1—C1—C2—C3 | 0.9 (5) | H2—C4—C8—C3 | −0.0 |
| O1—C1—C2—C10 | −179.4 (3) | H2—C4—C8—C9 | 179.8 |
| H1—C1—C2—C3 | −179.1 | C4—C5—C6—Br1 | −178.8 (3) |
| H1—C1—C2—C10 | 0.6 | C4—C5—C6—C7 | 0.1 (5) |
| C1—C2—C3—O2 | 175.8 (3) | H3—C5—C6—Br1 | 1.1 |
| C1—C2—C3—C8 | −4.2 (5) | H3—C5—C6—C7 | −179.9 |
| C1—C2—C10—O3 | 5.6 (5) | Br1—C6—C7—C9 | 178.02 (18) |
| C1—C2—C10—H5 | −174.4 | Br1—C6—C7—H4 | −2.0 |
| C3—C2—C10—O3 | −174.7 (3) | C5—C6—C7—C9 | −0.9 (5) |
| C3—C2—C10—H5 | 5.3 | C5—C6—C7—H4 | 179.1 |
| C10—C2—C3—O2 | −3.9 (5) | C6—C7—C9—O1 | −178.3 (3) |
| C10—C2—C3—C8 | 176.1 (3) | C6—C7—C9—C8 | 1.2 (5) |
| O2—C3—C8—C4 | 4.2 (5) | H4—C7—C9—O1 | 1.7 |
| O2—C3—C8—C9 | −175.6 (3) | H4—C7—C9—C8 | −178.8 |
| C2—C3—C8—C4 | −175.8 (3) | C3—C8—C9—O1 | −1.4 (5) |
| C2—C3—C8—C9 | 4.4 (4) | C3—C8—C9—C7 | 179.2 (3) |
| C5—C4—C8—C3 | 180.0 (3) | C4—C8—C9—O1 | 178.8 (3) |
| C5—C4—C8—C9 | −0.2 (5) | C4—C8—C9—C7 | −0.7 (5) |

Symmetry codes: (i) $x-1, y-1, z$; (ii) $x, y-1, z$; (iii) $x-1, y, z$; (iv) $x, y+1, z$; (v) $x+1, y+1, z$; (vi) $x+1, y, z$; (vii) $-x+1, -y+1, -z+1$; (viii) $-x+1, -y+2, -z+1$; (ix) $-x, -y+1, -z+1$; (x) $-x, y-1/2, -z+3/2$; (xi) $-x+1, y-1/2, -z+3/2$; (xii) $-x, y+1/2, -z+3/2$; (xiii) $-x+1, y+1/2, -z+3/2$.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C7$^{v}$—H4$^{v}$···O2 | 0.95 | 2.30 | 3.149 (4) | 149 (1) |
| C1$^{ix}$—H1$^{ix}$···O3 | 0.95 | 2.37 | 3.228 (5) | 149 (1) |

Symmetry codes: (v) *x*+1, *y*+1, *z*; (ix) −*x*, −*y*+1, −*z*+1.