# organic compounds

# 7-Bromo-9-(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-en-1-yl)-3,3-dimethyl-2,3,4,9-tetrahydro-1H-xanthen-1-one

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In the xanthene ring system of the title compound, $C_{23}H_{25}BrO_4$, the 4H-pyran ring is almost planar [maximum deviation = 0.040 (3) Å] and the cyclohexene ring adopts a sofa conformation. The cyclohexene ring attached to the xanthene system is puckered [$Q_T$ = 0.427 (3) Å, $\theta$ = 55.0 (4) ° and $\varphi$ = 164.4 (6) °]. In the crystal, molecules are linked to each other by O—H···O and C—H···O hydrogen bonds.

## Related literature

For the biological and pharmaceutical properties of xanthenes, see: Mohamed *et al.* (2012); Hilderbrand & Weissleder (2007); Shchekotikhin & Nikolaeva (2006); Fan *et al.* (2005). For related structures, see: Abdelhamid *et al.* (2011); Mohamed *et al.* (2011); Reddy *et al.* (2009); Çelik *et al.* (2009). For puckering parameters, see: Cremer & Pople (1975).
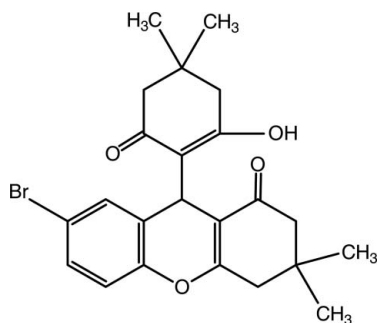


## Experimental

### Crystal data

| | |
|---|---|
| $C_{23}H_{25}BrO_4$ | $a$ = 15.6869 (4) Å |
| $M_r$ = 445.33 | $b$ = 11.0215 (2) Å |
| Orthorhombic, $Pbca$ | $c$ = 23.0217 (16) Å |

| | |
|---|---|
| $V$ = 3980.3 (3) Å$^3$ | $\mu$ = 3.04 mm$^{-1}$ |
| $Z$ = 8 | $T$ = 150 K |
| Cu $K\alpha$ radiation | $0.12 \times 0.08 \times 0.02$ mm |

### Data collection

| | |
|---|---|
| Rigaku RAPID II diffractometer | 17325 measured reflections |
| Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2001) $T_{min}$ = 0.712, $T_{max}$ = 0.942 | 3507 independent reflections |
| | 2728 reflections with $I > 2\sigma(I)$ |
| | $R_{int}$ = 0.044 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.042 | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2)$ = 0.107 | |
| $S$ = 1.07 | |
| 3507 reflections | $\Delta\rho_{max}$ = 0.46 e Å$^{-3}$ |
| 260 parameters | $\Delta\rho_{min}$ = −0.49 e Å$^{-3}$ |
| 1 restraint | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O4—H4$O$···O2$^i$ | 0.80 (3) | 1.86 (3) | 2.650 (3) | 170 (3) |
| C8—H8$A$···O3$^{ii}$ | 0.99 | 2.54 | 3.514 (4) | 167 |

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

Data collection: *CrystalClear* (Rigaku, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5218).

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# supporting information

## 7-Bromo-9-(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-en-1-yl)-3,3-di-methyl-2,3,4,9-tetrahydro-1*H*-xanthen-1-one

**Shaaban K. Mohamed, Mehmet Akkurt, Antar A. Abdelhamid, Phillip E. Fanwick and Herman Potgeiter**

### S1. Comment

Xanthenes are an important class of organic compounds that received considerable attention from many pharmaceuticals and organic chemists. They considered as valuable synthons because of the inherent reactivity of the inbuilt pyran ring. Recently, it has been reported such compounds possess a broad spectrum of biological and pharmaceutical properties such as agricultural bactericide effects, photodynamic therapy, antiviral and anti-inflammatory activities (Hilderbrand & Weissleder 2007; Shchekotikhin, & Nikolaeva 2006; Fan *et al.* 2005).

Following to our recent study (Mohamed *et al.* 2012) on synthesis and structure characterization of tetrahydro xanthenones for biological investigation, herein we report the synthesis and crystal structure of the title compound.

In the title compound (I), (Fig. 1), the 4*H*-pyran ring (O1/C1/C6/C7/C12/C13) of the xanthene ring system (O1/C1–C13) is almost planar with a maximum deviation from the mean plane of 0.040 (3) Å for C13 and the cyclohexene ring (C7–C12) adopts a sofa conformation [puckering parameters (Cremer & Pople, 1975): $Q_T = 0.457$ (3) Å, $\theta = 121.7$ (4) °, $\varphi = 297.3$ (5) °]. The cyclohexene ring (C14–C19) attached to the xanthene system is not planar: the puckering parameters of this ring are $Q_T = 0.427$ (3) Å, $\theta = 55.0$ (4) ° and $\varphi = 164.4$ (6) °. The values of the bond lengths and bond angles are comparable with those of the related structures previously reported (Abdelhamid *et al.*, 2011; Mohamed *et al.*, 2011; Reddy *et al.*, 2009; Çelik *et al.*, 2009).

The crystal structure is stabilized by intermolecular O—H···O and C—H···O hydrogen bonds (Table 1 and Fig. 2).

### S2. Experimental

The title compound (I) has been prepared from reaction of 1 mmol (201 mg) 5-bromo-2-hydroxybenzaldehyde with 1 mmol (140 mg) dimedone in presence of either (4-aminophenyl)methanol or TRIZMA (tris(hydroxymethyl)amino-methane) as a catalyst in 50 ml e thanol at 351 K. The reaction was monitored by TLC till completion after 4 h then left to cool at ambient temperature. The reaction mixture was concentrated under vacuum and the solid formed product was collected and dried using Buckner funnel then recrystallized from ethanol (82% yield; m.p. 521 K). Pure crystals suitable for X-ray structure analysis were obtained by slow evaporation method using ethanol as a solvent.

### S3. Refinement

The hydroxyl H atom was located from a difference Fourier map and refined with a distance restraint of O–H 0.82±0.02. Å. Temperature factor was fixed at 1.5 times the isotropic value of the parent O atom. The hydrogen atoms at C were located geometrically and refined using a riding model with C—H = 0.95 Å (aromatic), 0.98 Å. (methyl), 0.99 Å (methylene) and 1.00 Å (methine), with $U_{iso}(H) = 1.2U_{eq}$ (aromatic, methine, methylene) and $U_{iso}(H) = 1.5U_{eq}$ (methyl).
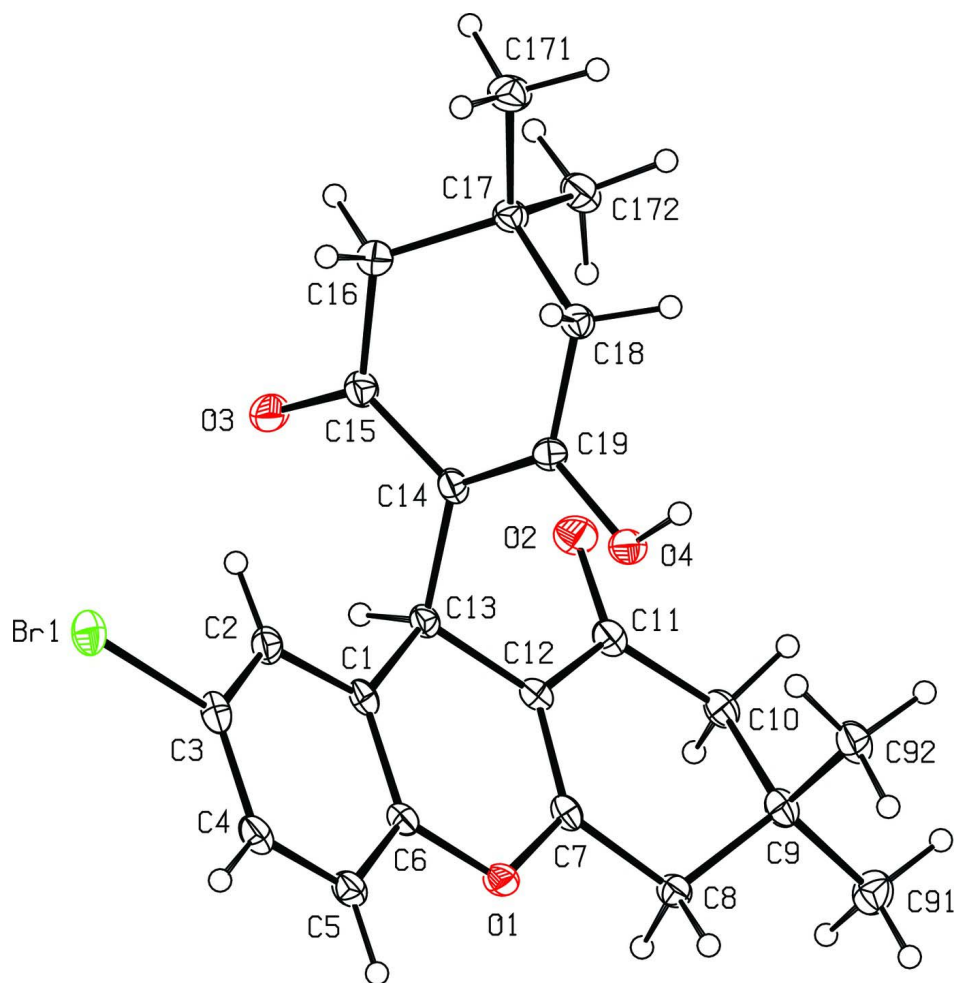
**Figure 1**

View of the title molecule with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.
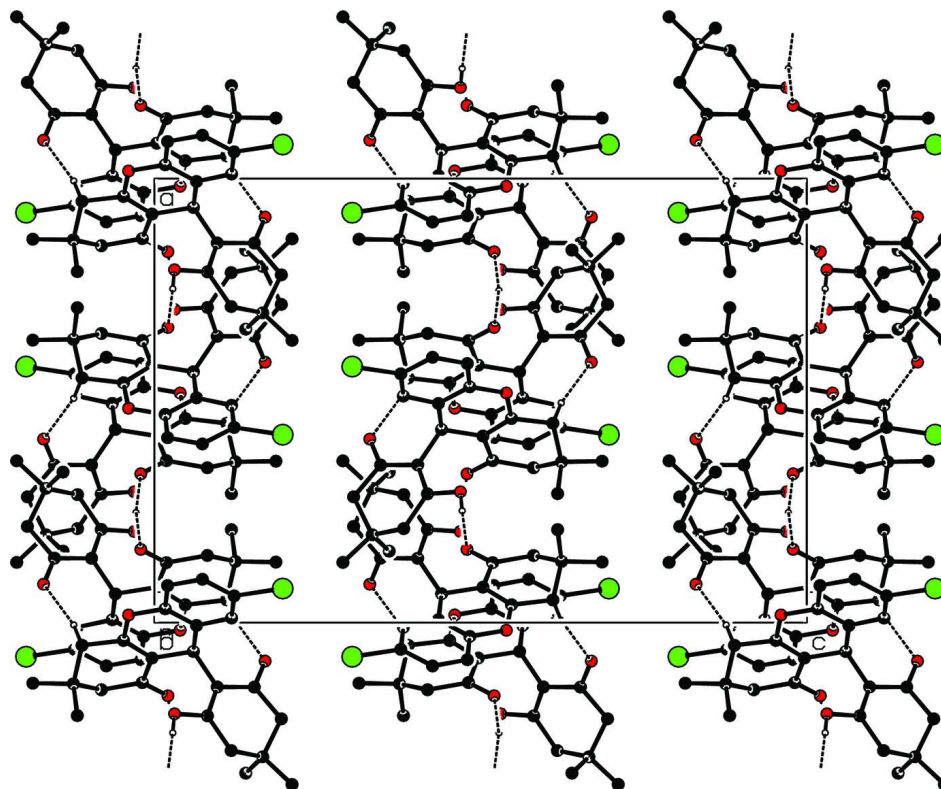
**Figure 2**

The crystal packing of the title compound, viewing along the *b* axis. Dashed lines show the intermolecular hydrogen bonding interactions. H atoms not involved in hydrogen bonds have been omitted for clarity.

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*Crystal data*

| | |
|---|---|
| $C_{23}H_{25}BrO_4$ | $F(000) = 1840$ |
| $M_r = 445.33$ | $D_x = 1.486$ Mg m$^{-3}$ |
| Orthorhombic, *Pbca* | Cu *Kα* radiation, $\lambda = 1.54178$ Å |
| Hall symbol: -P 2ac 2ab | Cell parameters from 18929 reflections |
| $a = 15.6869$ (4) Å | $\theta = 1$–66° |
| $b = 11.0215$ (2) Å | $\mu = 3.04$ mm$^{-1}$ |
| $c = 23.0217$ (16) Å | $T = 150$ K |
| $V = 3980.3$ (3) Å$^3$ | Needle, yellow |
| $Z = 8$ | $0.12 \times 0.08 \times 0.02$ mm |

*Data collection*

| | |
|---|---|
| Rigaku RAPID II diffractometer | 3507 independent reflections |
| Confocal optics monochromator | 2728 reflections with $I > 2\sigma(I)$ |
| *ω* scans | $R_{int} = 0.044$ |
| Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2001) | $\theta_{max} = 66.6°$, $\theta_{min} = 3.8°$ |
| $T_{min} = 0.712$, $T_{max} = 0.942$ | $h = -9 \rightarrow 18$ |
| 17325 measured reflections | $k = -13 \rightarrow 10$ |
| | $l = -27 \rightarrow 24$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.042$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.107$ | |
| $S = 1.07$ | H atoms treated by a mixture of independent and constrained refinement |
| 3507 reflections | |
| 260 parameters | $w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 5.2275P]$ |
| 1 restraint | where $P = (F_o^2 + 2F_c^2)/3$ |
| Primary atom site location: structure-invariant direct methods | $(\Delta/\sigma)_{max} < 0.001$ |
| | $\Delta\rho_{max} = 0.46$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.49$ e Å$^{-3}$ |

*Special details*

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**Refinement**. Refinement on $F^2$ for ALL reflections except those flagged by the user for potential systematic errors. Weighted $R$-factors $wR$ and all 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -$R$-factor-obs *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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Br1 | 0.07678 (2) | 0.50005 (3) | 0.19670 (1) | 0.0308 (1) |
| O1 | 0.01721 (13) | 0.28166 (19) | −0.03953 (8) | 0.0236 (6) |
| O2 | −0.16483 (15) | −0.0333 (2) | 0.02080 (10) | 0.0333 (8) |
| O3 | −0.08661 (14) | 0.0562 (2) | 0.16973 (10) | 0.0344 (8) |
| O4 | −0.20527 (13) | 0.3144 (2) | 0.03091 (9) | 0.0277 (7) |
| C1 | −0.00638 (18) | 0.2718 (3) | 0.06462 (13) | 0.0199 (9) |
| C2 | 0.01035 (19) | 0.3253 (3) | 0.11834 (13) | 0.0242 (10) |
| C3 | 0.06030 (19) | 0.4281 (3) | 0.12230 (13) | 0.0257 (10) |
| C4 | 0.0976 (2) | 0.4792 (3) | 0.07358 (14) | 0.0269 (10) |
| C5 | 0.08353 (19) | 0.4256 (3) | 0.02032 (14) | 0.0251 (10) |
| C6 | 0.03110 (18) | 0.3246 (3) | 0.01653 (12) | 0.0206 (9) |
| C7 | −0.04024 (18) | 0.1897 (3) | −0.04743 (13) | 0.0206 (9) |
| C8 | −0.05102 (19) | 0.1632 (3) | −0.11080 (12) | 0.0229 (9) |
| C9 | −0.13785 (19) | 0.1051 (3) | −0.12419 (13) | 0.0261 (10) |
| C10 | −0.1512 (2) | 0.0005 (3) | −0.08121 (13) | 0.0250 (10) |
| C11 | −0.13510 (19) | 0.0305 (3) | −0.01845 (13) | 0.0242 (9) |
| C12 | −0.07972 (18) | 0.1326 (3) | −0.00369 (13) | 0.0196 (9) |
| C13 | −0.06495 (18) | 0.1619 (3) | 0.05977 (12) | 0.0198 (9) |
| C14 | −0.14483 (18) | 0.1801 (3) | 0.09634 (12) | 0.0215 (9) |
| C15 | −0.14520 (19) | 0.1221 (3) | 0.15319 (13) | 0.0249 (9) |
| C16 | −0.2167 (2) | 0.1536 (3) | 0.19457 (13) | 0.0294 (10) |
| C17 | −0.30235 (19) | 0.1796 (3) | 0.16603 (13) | 0.0228 (9) |
| C18 | −0.28827 (19) | 0.2733 (3) | 0.11776 (13) | 0.0229 (9) |
| C19 | −0.20994 (19) | 0.2527 (3) | 0.08139 (13) | 0.0211 (9) |

| C91 | −0.1364 (2) | 0.0560 (3) | −0.18629 (13) | 0.0339 (11) |
| C92 | −0.2094 (2) | 0.1994 (3) | −0.11809 (15) | 0.0306 (11) |
| C171 | −0.3643 (2) | 0.2305 (3) | 0.21100 (15) | 0.0341 (11) |
| C172 | −0.3390 (2) | 0.0633 (3) | 0.14004 (15) | 0.0315 (11) |
| H2 | −0.01290 | 0.29060 | 0.15260 | 0.0290* |
| H4 | 0.13220 | 0.54960 | 0.07680 | 0.0320* |
| H4O | −0.2481 (16) | 0.353 (3) | 0.0282 (15) | 0.0370* |
| H5 | 0.10960 | 0.45770 | −0.01360 | 0.0300* |
| H8A | −0.00510 | 0.10770 | −0.12360 | 0.0280* |
| H8B | −0.04550 | 0.23960 | −0.13310 | 0.0280* |
| H10A | −0.21060 | −0.02860 | −0.08500 | 0.0300* |
| H10B | −0.11310 | −0.06720 | −0.09240 | 0.0300* |
| H13 | −0.03350 | 0.09150 | 0.07690 | 0.0240* |
| H16A | −0.19950 | 0.22580 | 0.21730 | 0.0350* |
| H16B | −0.22400 | 0.08560 | 0.22220 | 0.0350* |
| H17A | −0.37500 | 0.16920 | 0.24100 | 0.0510* |
| H17B | −0.41820 | 0.25180 | 0.19190 | 0.0510* |
| H17C | −0.33960 | 0.30300 | 0.22890 | 0.0510* |
| H17D | −0.34360 | 0.00140 | 0.17040 | 0.0470* |
| H17E | −0.30120 | 0.03400 | 0.10910 | 0.0470* |
| H17F | −0.39560 | 0.07990 | 0.12390 | 0.0470* |
| H18A | −0.33870 | 0.27310 | 0.09190 | 0.0280* |
| H18B | −0.28440 | 0.35480 | 0.13570 | 0.0280* |
| H91A | −0.12660 | 0.12300 | −0.21350 | 0.0510* |
| H91B | −0.19110 | 0.01720 | −0.19500 | 0.0510* |
| H91C | −0.09040 | −0.00370 | −0.19010 | 0.0510* |
| H92A | −0.19830 | 0.26770 | −0.14430 | 0.0460* |
| H92B | −0.21150 | 0.22870 | −0.07790 | 0.0460* |
| H92C | −0.26410 | 0.16200 | −0.12820 | 0.0460* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br1 | 0.0299 (2) | 0.0320 (2) | 0.0305 (2) | −0.0014 (2) | −0.0083 (1) | −0.0056 (1) |
| O1 | 0.0219 (11) | 0.0251 (12) | 0.0237 (10) | −0.0080 (10) | 0.0012 (9) | −0.0007 (9) |
| O2 | 0.0342 (13) | 0.0301 (13) | 0.0356 (13) | −0.0122 (11) | 0.0017 (11) | 0.0040 (10) |
| O3 | 0.0290 (12) | 0.0449 (16) | 0.0292 (12) | 0.0110 (12) | 0.0030 (10) | 0.0121 (11) |
| O4 | 0.0230 (11) | 0.0304 (14) | 0.0298 (11) | 0.0080 (10) | 0.0034 (10) | 0.0097 (10) |
| C1 | 0.0140 (14) | 0.0170 (16) | 0.0286 (15) | 0.0020 (12) | −0.0039 (12) | 0.0004 (12) |
| C2 | 0.0201 (15) | 0.0278 (19) | 0.0246 (16) | 0.0025 (14) | −0.0041 (13) | 0.0000 (13) |
| C3 | 0.0203 (15) | 0.0274 (19) | 0.0294 (16) | 0.0031 (14) | −0.0072 (13) | −0.0052 (14) |
| C4 | 0.0175 (15) | 0.027 (2) | 0.0361 (18) | −0.0034 (14) | −0.0038 (13) | 0.0018 (14) |
| C5 | 0.0194 (15) | 0.0262 (19) | 0.0298 (16) | −0.0042 (14) | −0.0012 (13) | 0.0005 (14) |
| C6 | 0.0146 (14) | 0.0227 (17) | 0.0244 (15) | −0.0016 (13) | −0.0005 (12) | −0.0015 (13) |
| C7 | 0.0156 (14) | 0.0189 (17) | 0.0274 (15) | 0.0018 (13) | −0.0022 (12) | −0.0021 (13) |
| C8 | 0.0197 (15) | 0.0247 (18) | 0.0244 (15) | −0.0020 (14) | 0.0010 (12) | −0.0014 (13) |
| C9 | 0.0212 (15) | 0.0275 (19) | 0.0295 (16) | −0.0036 (14) | −0.0035 (13) | −0.0022 (14) |
| C10 | 0.0208 (16) | 0.0229 (18) | 0.0312 (17) | −0.0041 (14) | −0.0024 (13) | −0.0030 (13) |

| C11 | 0.0196 (15) | 0.0231 (18) | 0.0299 (16) | −0.0008 (14) | −0.0018 (13) | 0.0015 (13) |
|-----|-------------|-------------|-------------|--------------|--------------|-------------|
| C12 | 0.0153 (14) | 0.0169 (16) | 0.0266 (15) | −0.0001 (12) | 0.0009 (12) | 0.0002 (12) |
| C13 | 0.0157 (14) | 0.0202 (17) | 0.0235 (15) | 0.0012 (13) | −0.0020 (12) | 0.0029 (12) |
| C14 | 0.0170 (15) | 0.0218 (17) | 0.0256 (15) | −0.0004 (13) | −0.0034 (12) | 0.0032 (13) |
| C15 | 0.0212 (15) | 0.0271 (18) | 0.0264 (16) | 0.0006 (15) | −0.0017 (13) | 0.0043 (13) |
| C16 | 0.0242 (17) | 0.039 (2) | 0.0250 (16) | 0.0009 (16) | 0.0011 (13) | 0.0027 (14) |
| C17 | 0.0196 (15) | 0.0239 (18) | 0.0248 (15) | 0.0030 (13) | 0.0020 (13) | 0.0031 (13) |
| C18 | 0.0201 (15) | 0.0237 (18) | 0.0250 (16) | 0.0023 (14) | 0.0002 (13) | 0.0018 (13) |
| C19 | 0.0219 (15) | 0.0185 (16) | 0.0229 (15) | −0.0033 (13) | 0.0002 (13) | 0.0032 (12) |
| C91 | 0.038 (2) | 0.034 (2) | 0.0298 (17) | −0.0031 (18) | −0.0031 (15) | −0.0053 (15) |
| C92 | 0.0225 (16) | 0.031 (2) | 0.0384 (19) | 0.0004 (15) | −0.0053 (14) | 0.0039 (15) |
| C171 | 0.0276 (18) | 0.040 (2) | 0.0347 (18) | 0.0081 (17) | 0.0074 (15) | 0.0039 (16) |
| C172 | 0.0234 (17) | 0.031 (2) | 0.0400 (19) | −0.0016 (16) | −0.0006 (14) | 0.0069 (16) |

*Geometric parameters (Å, º)*

| | | | |
|------|-----------|-----------|-----------|
| Br1—C3 | 1.905 (3) | C17—C18 | 1.533 (4) |
| O1—C6 | 1.392 (3) | C17—C172 | 1.527 (5) |
| O1—C7 | 1.368 (4) | C17—C171 | 1.527 (4) |
| O2—C11 | 1.236 (4) | C18—C19 | 1.504 (4) |
| O3—C15 | 1.232 (4) | C2—H2 | 0.9500 |
| O4—C19 | 1.349 (4) | C4—H4 | 0.9500 |
| O4—H4O | 0.80 (3) | C5—H5 | 0.9500 |
| C1—C6 | 1.382 (4) | C8—H8A | 0.9900 |
| C1—C13 | 1.524 (4) | C8—H8B | 0.9900 |
| C1—C2 | 1.395 (4) | C10—H10A | 0.9900 |
| C2—C3 | 1.381 (5) | C10—H10B | 0.9900 |
| C3—C4 | 1.385 (4) | C13—H13 | 1.0000 |
| C4—C5 | 1.379 (5) | C16—H16A | 0.9900 |
| C5—C6 | 1.387 (4) | C16—H16B | 0.9900 |
| C7—C12 | 1.339 (4) | C18—H18A | 0.9900 |
| C7—C8 | 1.497 (4) | C18—H18B | 0.9900 |
| C8—C9 | 1.536 (4) | C91—H91A | 0.9800 |
| C9—C91 | 1.529 (4) | C91—H91B | 0.9800 |
| C9—C92 | 1.536 (4) | C91—H91C | 0.9800 |
| C9—C10 | 1.534 (4) | C92—H92A | 0.9800 |
| C10—C11 | 1.504 (4) | C92—H92B | 0.9800 |
| C11—C12 | 1.462 (4) | C92—H92C | 0.9800 |
| C12—C13 | 1.514 (4) | C171—H17A | 0.9800 |
| C13—C14 | 1.523 (4) | C171—H17B | 0.9800 |
| C14—C15 | 1.457 (4) | C171—H17C | 0.9800 |
| C14—C19 | 1.342 (4) | C172—H17D | 0.9800 |
| C15—C16 | 1.512 (4) | C172—H17E | 0.9800 |
| C16—C17 | 1.523 (4) | C172—H17F | 0.9800 |
| | | | |
| C6—O1—C7 | 118.6 (2) | C3—C2—H2 | 120.00 |
| C19—O4—H4O | 107 (2) | C3—C4—H4 | 121.00 |
| C2—C1—C13 | 120.9 (3) | C5—C4—H4 | 121.00 |

| | | | |
|---|---|---|---|
| C6—C1—C13 | 122.2 (3) | C4—C5—H5 | 120.00 |
| C2—C1—C6 | 116.9 (3) | C6—C5—H5 | 120.00 |
| C1—C2—C3 | 120.8 (3) | C7—C8—H8A | 109.00 |
| Br1—C3—C4 | 120.1 (2) | C7—C8—H8B | 109.00 |
| C2—C3—C4 | 121.3 (3) | C9—C8—H8A | 109.00 |
| Br1—C3—C2 | 118.6 (2) | C9—C8—H8B | 109.00 |
| C3—C4—C5 | 118.6 (3) | H8A—C8—H8B | 108.00 |
| C4—C5—C6 | 119.7 (3) | C9—C10—H10A | 108.00 |
| O1—C6—C1 | 122.2 (3) | C9—C10—H10B | 108.00 |
| O1—C6—C5 | 115.1 (3) | C11—C10—H10A | 108.00 |
| C1—C6—C5 | 122.7 (3) | C11—C10—H10B | 108.00 |
| O1—C7—C12 | 123.6 (3) | H10A—C10—H10B | 108.00 |
| C8—C7—C12 | 126.1 (3) | C1—C13—H13 | 107.00 |
| O1—C7—C8 | 110.4 (2) | C12—C13—H13 | 107.00 |
| C7—C8—C9 | 112.1 (2) | C14—C13—H13 | 107.00 |
| C8—C9—C10 | 107.8 (2) | C15—C16—H16A | 108.00 |
| C8—C9—C91 | 108.8 (2) | C15—C16—H16B | 108.00 |
| C10—C9—C91 | 109.8 (3) | C17—C16—H16A | 108.00 |
| C10—C9—C92 | 110.5 (3) | C17—C16—H16B | 109.00 |
| C91—C9—C92 | 109.6 (3) | H16A—C16—H16B | 107.00 |
| C8—C9—C92 | 110.3 (3) | C17—C18—H18A | 109.00 |
| C9—C10—C11 | 115.6 (3) | C17—C18—H18B | 109.00 |
| O2—C11—C10 | 120.9 (3) | C19—C18—H18A | 109.00 |
| C10—C11—C12 | 119.5 (3) | C19—C18—H18B | 109.00 |
| O2—C11—C12 | 119.5 (3) | H18A—C18—H18B | 108.00 |
| C7—C12—C11 | 117.5 (3) | C9—C91—H91A | 109.00 |
| C11—C12—C13 | 118.7 (3) | C9—C91—H91B | 109.00 |
| C7—C12—C13 | 123.7 (3) | C9—C91—H91C | 109.00 |
| C1—C13—C12 | 109.4 (2) | H91A—C91—H91B | 110.00 |
| C1—C13—C14 | 110.5 (3) | H91A—C91—H91C | 109.00 |
| C12—C13—C14 | 115.8 (2) | H91B—C91—H91C | 109.00 |
| C13—C14—C15 | 116.3 (3) | C9—C92—H92A | 109.00 |
| C13—C14—C19 | 124.3 (3) | C9—C92—H92B | 109.00 |
| C15—C14—C19 | 119.3 (3) | C9—C92—H92C | 109.00 |
| O3—C15—C16 | 119.6 (3) | H92A—C92—H92B | 110.00 |
| C14—C15—C16 | 117.9 (3) | H92A—C92—H92C | 109.00 |
| O3—C15—C14 | 122.3 (3) | H92B—C92—H92C | 109.00 |
| C15—C16—C17 | 115.2 (2) | C17—C171—H17A | 109.00 |
| C16—C17—C171 | 109.8 (2) | C17—C171—H17B | 109.00 |
| C16—C17—C172 | 110.1 (3) | C17—C171—H17C | 109.00 |
| C16—C17—C18 | 108.2 (2) | H17A—C171—H17B | 109.00 |
| C18—C17—C172 | 109.6 (3) | H17A—C171—H17C | 109.00 |
| C171—C17—C172 | 109.5 (3) | H17B—C171—H17C | 110.00 |
| C18—C17—C171 | 109.6 (3) | C17—C172—H17D | 109.00 |
| C17—C18—C19 | 114.8 (3) | C17—C172—H17E | 109.00 |
| O4—C19—C18 | 116.6 (3) | C17—C172—H17F | 109.00 |
| C14—C19—C18 | 124.7 (3) | H17D—C172—H17E | 109.00 |
| O4—C19—C14 | 118.7 (3) | H17D—C172—H17F | 110.00 |

| C1—C2—H2 | 120.00 | H17E—C172—H17F | 109.00 |
| --- | --- | --- | --- |
| C7—O1—C6—C5 | −173.8 (3) | C9—C10—C11—O2 | 160.4 (3) |
| C6—O1—C7—C8 | 175.6 (2) | C9—C10—C11—C12 | −23.0 (4) |
| C7—O1—C6—C1 | 4.8 (4) | O2—C11—C12—C7 | 171.7 (3) |
| C6—O1—C7—C12 | −4.8 (4) | O2—C11—C12—C13 | −3.8 (4) |
| C6—C1—C2—C3 | 1.7 (4) | C10—C11—C12—C7 | −5.0 (4) |
| C13—C1—C2—C3 | −177.0 (3) | C10—C11—C12—C13 | 179.5 (3) |
| C2—C1—C13—C12 | 173.4 (3) | C7—C12—C13—C1 | 5.4 (4) |
| C2—C1—C13—C14 | 44.7 (4) | C7—C12—C13—C14 | 131.1 (3) |
| C6—C1—C13—C12 | −5.3 (4) | C11—C12—C13—C1 | −179.4 (3) |
| C6—C1—C13—C14 | −134.0 (3) | C11—C12—C13—C14 | −53.7 (4) |
| C2—C1—C6—O1 | −178.1 (3) | C1—C13—C14—C15 | −98.9 (3) |
| C2—C1—C6—C5 | 0.4 (5) | C1—C13—C14—C19 | 75.5 (4) |
| C13—C1—C6—O1 | 0.6 (5) | C12—C13—C14—C15 | 135.9 (3) |
| C13—C1—C6—C5 | 179.1 (3) | C12—C13—C14—C19 | −49.6 (4) |
| C1—C2—C3—Br1 | 177.5 (2) | C13—C14—C15—O3 | −4.1 (5) |
| C1—C2—C3—C4 | −2.1 (5) | C13—C14—C15—C16 | 170.6 (3) |
| Br1—C3—C4—C5 | −179.3 (2) | C19—C14—C15—O3 | −178.8 (3) |
| C2—C3—C4—C5 | 0.4 (5) | C19—C14—C15—C16 | −4.1 (4) |
| C3—C4—C5—C6 | 1.7 (5) | C13—C14—C19—O4 | −0.2 (5) |
| C4—C5—C6—O1 | 176.5 (3) | C13—C14—C19—C18 | −178.7 (3) |
| C4—C5—C6—C1 | −2.1 (5) | C15—C14—C19—O4 | 174.1 (3) |
| O1—C7—C8—C9 | −155.2 (3) | C15—C14—C19—C18 | −4.4 (5) |
| C12—C7—C8—C9 | 25.3 (4) | O3—C15—C16—C17 | −151.6 (3) |
| O1—C7—C12—C11 | −175.9 (3) | C14—C15—C16—C17 | 33.6 (4) |
| O1—C7—C12—C13 | −0.7 (5) | C15—C16—C17—C18 | −50.9 (4) |
| C8—C7—C12—C11 | 3.5 (5) | C15—C16—C17—C171 | −170.5 (3) |
| C8—C7—C12—C13 | 178.8 (3) | C15—C16—C17—C172 | 68.9 (3) |
| C7—C8—C9—C10 | −48.8 (3) | C16—C17—C18—C19 | 42.1 (3) |
| C7—C8—C9—C91 | −167.8 (3) | C171—C17—C18—C19 | 161.8 (3) |
| C7—C8—C9—C92 | 71.9 (3) | C172—C17—C18—C19 | −78.0 (3) |
| C8—C9—C10—C11 | 48.9 (3) | C17—C18—C19—O4 | 165.1 (3) |
| C91—C9—C10—C11 | 167.2 (3) | C17—C18—C19—C14 | −16.3 (4) |
| C92—C9—C10—C11 | −71.7 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| D—H···A | D—H | H···A | D···A | D—H···A |
| --- | --- | --- | --- | --- |
| O4—H4O···O2[i] | 0.80 (3) | 1.86 (3) | 2.650 (3) | 170 (3) |
| C8—H8A···O3[ii] | 0.99 | 2.54 | 3.514 (4) | 167 |
| C13—H13···O3 | 1.00 | 2.33 | 2.807 (4) | 108 |

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