

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

6,8-Dibromo-5-hydroxy-4-oxo-2-phenyl-4H-chromen-7-yl acetate

Angannan Nallasivam,^a Munirathinam Nethaji,^b
Nagarajan Vembu,^{c*} Venkatraman Ragunathan^d and
Nagarajan Sulochana^a

^aDepartment of Chemistry, National Institute of Technology, Tiruchirappalli 620 015, India, ^bDepartment of Inorganic and Physical Chemistry, Indian Institute of Science, Bangalore 560 012, India, ^cDepartment of Chemistry, Urumu Dhanalakshmi College, Tiruchirappalli 620 019, India, and ^dDepartment of Chemistry, Kandasamy Kandar College, Velur 638 182, India

Correspondence e-mail: vembu57@yahoo.com

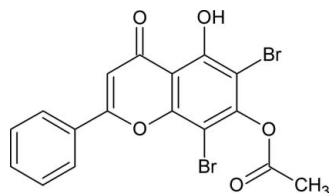
Received 12 January 2009; accepted 16 February 2009

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.044; wR factor = 0.105; data-to-parameter ratio = 17.1.

In the title compound, $\text{C}_{17}\text{H}_{10}\text{Br}_2\text{O}_5$, the chromene ring is almost planar with minimal puckering [total puckering amplitude = $0.067(4)$ Å]. The dihedral angle between chromene ring system and phenyl ring is $3.7(2)^\circ$. The crystal structure is stabilized by intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions and an intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond also occurs.

Related literature

For the biological and pharmacological properties of benzopyrans and their derivatives, see: Brooks (1998); Hatakeyama *et al.* (1988); Hyana & Saimoto (1987); Tang *et al.* (2007). For the importance of 4H-chromenes, see: Liu *et al.* (2007); Wang, Fang *et al.* (2003); Wang, Zhang *et al.* (2003). For hydrogen bonding, see: Bernstein *et al.* (1995); Desiraju (1989); Desiraju & Steiner (1999); Etter (1990).



Experimental

Crystal data

$\text{C}_{17}\text{H}_{10}\text{Br}_2\text{O}_5$
 $M_r = 454.07$
Monoclinic, $P2_1/n$
 $a = 14.072(3)$ Å
 $b = 5.5586(13)$ Å

$c = 21.333(5)$ Å
 $\beta = 104.501(4)^\circ$
 $V = 1615.6(7)$ Å³
 $Z = 4$
Mo $K\alpha$ radiation

$\mu = 5.04$ mm⁻¹
 $T = 293$ K

$0.55 \times 0.23 \times 0.12$ mm

Data collection

Bruker SMART APEX CCD
diffractometer
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1998)
 $T_{\min} = 0.157$, $T_{\max} = 0.547$

13326 measured reflections
3773 independent reflections
2245 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.105$
 $S = 0.99$
3773 reflections

221 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.57$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.37$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O}12-\text{H}12\cdots\text{O}11$	0.82	1.86	2.584 (4)	147
$\text{C}3-\text{H}3\cdots\text{O}11^i$	0.93	2.57	3.497 (4)	171
$\text{C}24-\text{H}24\cdots\text{O}11^i$	0.93	2.48	3.387 (5)	166
$\text{C}17-\text{H}17A\cdots\text{O}16^{ii}$	0.96	2.58	3.309 (6)	133

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

AN thanks Dr S. Kannan and Dr B. S. Krishnamurthy, School of Chemistry, Bharathidasan University, Tiruchirappalli, and Organica Aromatics Pvt Ltd Bangalore, India, for providing laboratory facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2060).

References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Brooks, G. T. (1998). *Pestic. Sci.* **22**, 41–50.
- Bruker (2007). *SMART* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Desiraju, G. R. (1989). *Crystal Engineering: The Design of Organic Solids*, pp. 125–167. Amsterdam: Elsevier.
- Desiraju, G. R. & Steiner, T. (1999). *The Weak Hydrogen Bond in Structural Chemistry and Biology*, pp. 11–40. New York: Oxford University Press.
- Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
- Hatakeyama, S., Ochi, N., Numata, H. & Takano, S. (1988). *J. Chem. Soc. Chem. Commun.* pp. 1022–1024.
- Hyana, T. & Saimoto, H. (1987). Jpn Patent JP 621 812 768.
- Liu, C.-B., Chen, Y.-H., Zhou, X.-Y., Ding, L. & Wen, H.-L. (2007). *Acta Cryst.* **E63**, o90–o91.
- Sheldrick, G. M. (1998). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Tang, Q.-G., Wu, W.-Y., He, W., Sun, H.-S. & Guo, C. (2007). *Acta Cryst.* **E63**, o1437–o1438.
- Wang, J.-F., Fang, M.-J., Huang, H.-Q., Li, G.-L., Su, W.-J. & Zhao, Y.-F. (2003). *Acta Cryst.* **E59**, o1517–o1518.
- Wang, J.-F., Zhang, Y.-J., Fang, M.-J., Huang, Y.-J., Wei, Z.-B., Zheng, Z.-H., Su, W.-J. & Zhao, Y.-F. (2003). *Acta Cryst.* **E59**, o1244–o1245.

supporting information

Acta Cryst. (2009). E65, o568 [doi:10.1107/S1600536809005431]

6,8-Dibromo-5-hydroxy-4-oxo-2-phenyl-4H-chromen-7-yl acetate

Angannan Nallasivam, Munirathinam Nethaji, Nagarajan Vembu, Venkatraman Ragunathan and Nagarajan Sulochana

S1. Comment

Chromenes (benzopyrans) and their derivatives have numerous biological and pharmacological properties (Tang *et al.*, 2007) such as antisterility (Brooks, 1998) and anticancer activity (Hyana & Saimoto, 1987). In addition, polyfunctionalized chromene units are present in numerous natural products (Hatakeyama *et al.*, 1988). 4H-chromenes are important synthons for some natural products (Liu *et al.*, 2007). As a part of our structural investigations on 4H-chromene derivatives and compounds containing the benzopyran fragment, the single-crystal X-ray diffraction study on the title compound was carried out.

The chromene ring is almost planar similarly as those found in the related chromene derivatives (Wang, Fang *et al.*, 2003; Wang, Zhang *et al.*, 2003). The total puckering amplitude of the chromene ring is 0.067 (4) Å in the title structure. The interplanar angle between the chromene ring and the 2-phenyl ring is 3.7 (2)° thereby indicating the almost coplanar arrangement (Fig. 1). The OCOCH₃ substituent at C7 is non-coplanar with the chromene ring as discerned from the interplanar angle of 87.4 (1)°.

The crystal structure is stabilized by the interplay of C–H⋯O and O–H⋯O interactions (Fig. 2 & Table 1). The H-bond distances agree with those reported in literature (Desiraju & Steiner, 1999; Desiraju, 1989). The C20–H20⋯O1 interaction generates a motif of graph set (Bernstein *et al.*, 1995; Etter, 1990) S(5). An S(6) motif is formed by O12–H12⋯O11 interaction. This interaction is also responsible for the formation of a cooperative H-bonded network (Fig. 3). The C3–H3⋯O11ⁱ and C24–H24⋯O11ⁱ interactions constitute a pair of bifurcated acceptor bonds generating a ring of graph set R¹₂(7). There are no significant C–H⋯π and π⋯π interactions.

S2. Experimental

In to the RBF, a suspension of chrysin (1 g, 3.93 mmol) and potassium carbonate (1.64 g, 11.81 mmol) in dimethyl formamide (10 ml) were added. The reaction mixture was heated to 383 K for 2–3 hrs. The reaction mixture was cooled to 313 K and acetyl chloride (1.23 g, 15.74 mmol) was slowly added with the help of dropping funnel. The reaction mixture was maintained for 8–9 hr at 313 K and monitored by HPLC. After completion of the reaction, the contents were quenched with water and stirred for 30–45 min at 303 K. The crude solid obtained was filtered and washed with plenty of water followed by methanol and dried under vacuum at 343 K. The acetylated compound was then taken in RBF and dissolved in dichloromethane (10 ml) and cooled to 273 K. Bromine (0.6 ml, 11.81 mmol) was added dropwise over a period of 15–20 min. The reaction mixture was maintained at 273 K for 5–6 hr. After completion of the reaction, the reaction mixture was quenched in ice water and extracted with dichloromethane (10 ml) and purified by column chromatography using ethyl acetate: n-hexane (20:80). The crude brominated product was then dissolved in dichloromethane (10 ml) and equal amount of n-Hexane (10 ml). The clear solution was kept for a week without stirring. Diffraction quality needle shaped crystals of average size 0.23 mm were obtained which were filtered and washed with n-

hexane and dried under vacuum at 343 K. Yield: 80%

S3. Refinement

All the H-atoms were observed in the difference electron density map. However, they were situated into idealized positions with C—H = 0.93 and 0.96 Å for aryl and methylene H, respectively and O—H = 0.82 Å for hydroxyl H-atoms and and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}(\text{C})$ for C—H and $U_{\text{iso}}(\text{H})=1.5U_{\text{eq}}(\text{O})$ for O—H.

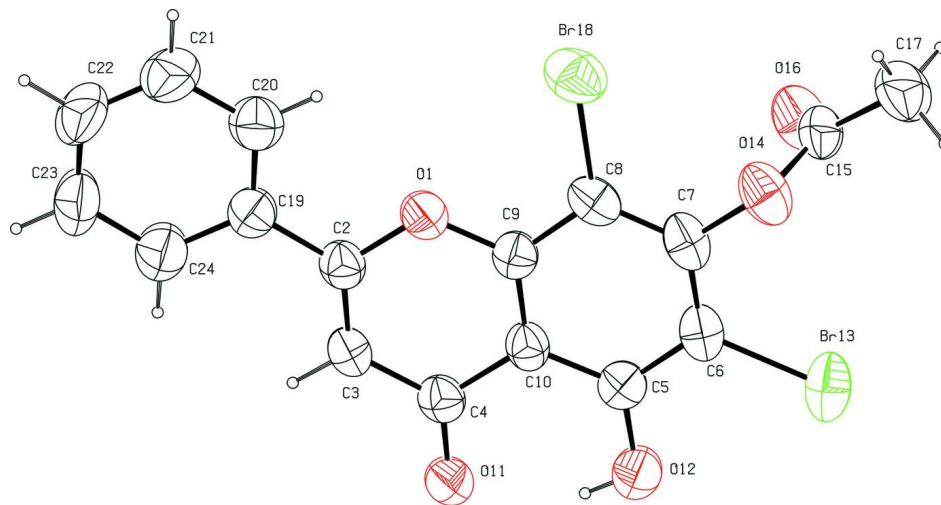
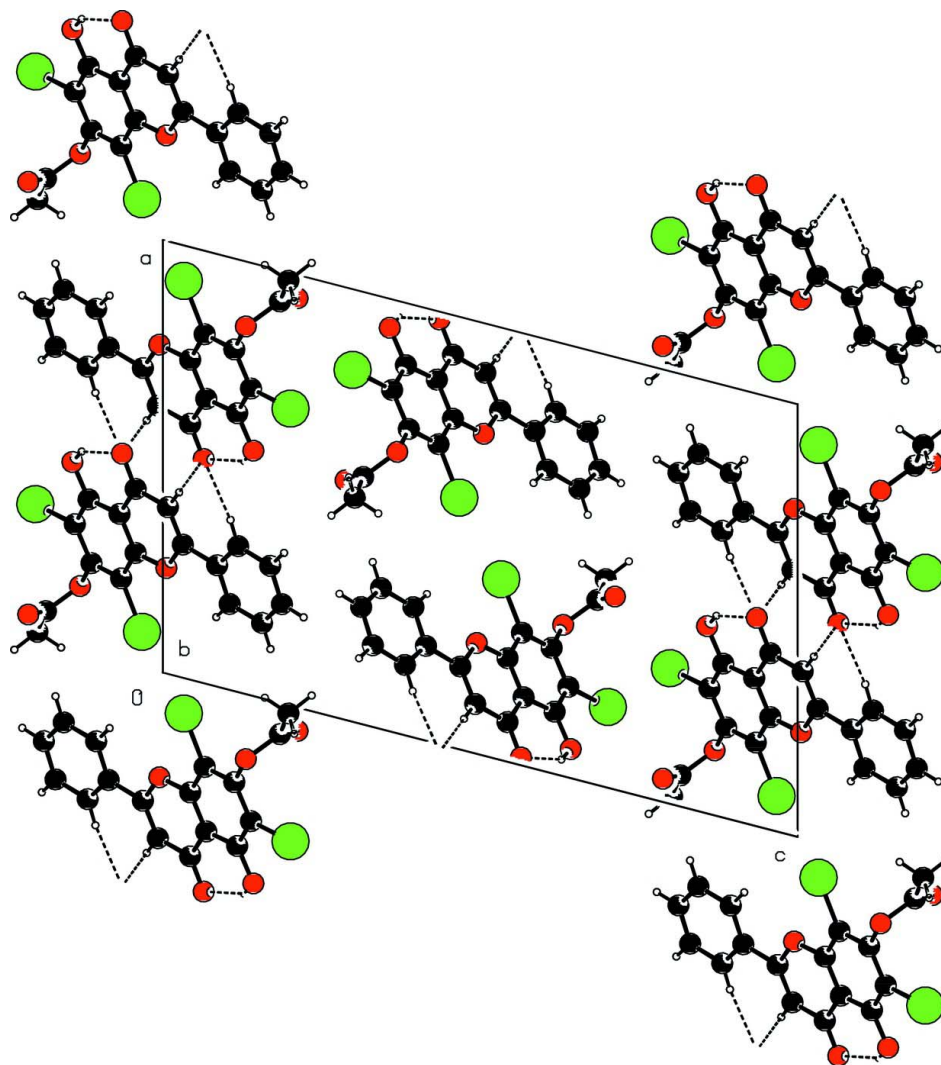
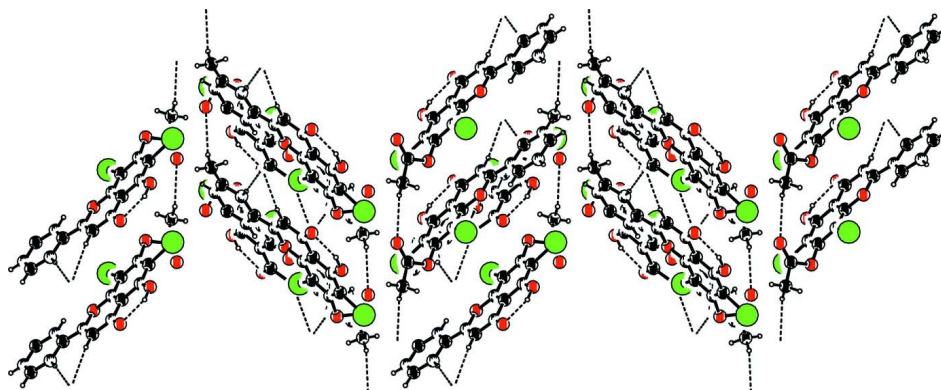


Figure 1

The asymmetric unit with the atoms labelled and displacement ellipsoids depicted at the 50% probability level for all non-H atoms. H-atoms are drawn as spheres of arbitrary radius.

**Figure 2**

The molecular packing viewed down the *b*-axis. Dashed lines represent the weak C–H···O interactions within the lattice.

**Figure 3**

Cooperative H-bonded network of O–H···O interactions viewed down the *a*-axis

6,8-Dibromo-5-hydroxy-4-oxo-2-phenyl-4*H*-chromen-7-yl acetate

Crystal data

C₁₇H₁₀Br₂O₅ $M_r = 454.07$ Monoclinic, $P2_1/n$ Hall symbol: $-P\ 2_1n$ $a = 14.072\ (3)\ \text{\AA}$ $b = 5.5586\ (13)\ \text{\AA}$ $c = 21.333\ (5)\ \text{\AA}$ $\beta = 104.501\ (4)^\circ$ $V = 1615.6\ (7)\ \text{\AA}^3$ $Z = 4$ $F(000) = 888$ $D_x = 1.867\ \text{Mg m}^{-3}$

Melting point = 433–435 K

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 475 reflections

 $\theta = 2.0\text{--}27.0^\circ$ $\mu = 5.04\ \text{mm}^{-1}$ $T = 293\ \text{K}$

Rectangular, brown

 $0.55 \times 0.23 \times 0.12\ \text{mm}$

Data collection

Bruker SMART APEX CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.3 pixels mm^{-1} ω scansAbsorption correction: multi-scan
(*SADABS*; Sheldrick, 1998) $T_{\min} = 0.157$, $T_{\max} = 0.547$

13326 measured reflections

3773 independent reflections

2245 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.046$ $\theta_{\max} = 28.0^\circ$, $\theta_{\min} = 2.0^\circ$ $h = -18 \rightarrow 18$ $k = -7 \rightarrow 7$ $l = -25 \rightarrow 28$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.044$ $wR(F^2) = 0.105$ $S = 0.99$

3773 reflections

221 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0505P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.57\ \text{e \AA}^{-3}$ $\Delta\rho_{\min} = -0.37\ \text{e \AA}^{-3}$

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.

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.75605 (17)	-0.0593 (5)	-0.00552 (13)	0.0490 (7)
C2	0.6921 (3)	-0.2392 (7)	-0.03148 (18)	0.0441 (9)
C3	0.6143 (3)	-0.2872 (7)	-0.00800 (19)	0.0444 (9)

H3	0.5734	-0.4146	-0.0256	0.069 (6)*
C4	0.5916 (3)	-0.1506 (7)	0.04288 (19)	0.0429 (9)
C5	0.6440 (3)	0.1983 (7)	0.11713 (18)	0.0424 (9)
C6	0.7094 (3)	0.3853 (7)	0.13882 (18)	0.0455 (9)
C7	0.7897 (3)	0.4160 (7)	0.11257 (19)	0.0484 (10)
C8	0.8070 (3)	0.2636 (7)	0.0658 (2)	0.0474 (10)
C9	0.7396 (3)	0.0821 (7)	0.04300 (18)	0.0416 (9)
C10	0.6588 (3)	0.0450 (6)	0.06832 (18)	0.0419 (9)
O11	0.51819 (18)	-0.1904 (5)	0.06422 (13)	0.0506 (7)
O12	0.5671 (2)	0.1687 (5)	0.14257 (14)	0.0550 (7)
H12	0.5375	0.0458	0.1279	0.101 (9)*
Br13	0.68667 (3)	0.60136 (8)	0.20112 (2)	0.06240 (17)
O14	0.8497 (2)	0.6151 (5)	0.13072 (15)	0.0600 (8)
C15	0.9279 (3)	0.5925 (8)	0.1845 (2)	0.0523 (10)
O16	0.9461 (2)	0.4080 (6)	0.21218 (15)	0.0660 (8)
C17	0.9808 (3)	0.8239 (8)	0.1978 (3)	0.0747 (15)
H17A	0.9374	0.9448	0.2069	0.101 (9)*
H17B	1.0033	0.8711	0.1607	0.101 (9)*
H17C	1.0361	0.8062	0.2345	0.101 (9)*
Br18	0.91815 (3)	0.30002 (10)	0.03298 (2)	0.06851 (18)
C19	0.7192 (3)	-0.3583 (7)	-0.08556 (19)	0.0471 (10)
C20	0.8013 (3)	-0.2850 (9)	-0.1052 (2)	0.0607 (12)
H20	0.8397	-0.1587	-0.0841	0.069 (6)*
C21	0.8258 (3)	-0.4012 (10)	-0.1563 (2)	0.0741 (14)
H21	0.8810	-0.3525	-0.1694	0.069 (6)*
C22	0.7703 (4)	-0.5856 (10)	-0.1876 (2)	0.0751 (14)
H22	0.7879	-0.6629	-0.2217	0.069 (6)*
C23	0.6888 (4)	-0.6571 (9)	-0.1692 (2)	0.0781 (15)
H23	0.6507	-0.7828	-0.1908	0.069 (6)*
C24	0.6626 (3)	-0.5442 (8)	-0.1186 (2)	0.0650 (12)
H24	0.6065	-0.5930	-0.1065	0.069 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0447 (14)	0.0548 (18)	0.0464 (16)	-0.0054 (12)	0.0096 (12)	-0.0050 (14)
C2	0.044 (2)	0.046 (2)	0.039 (2)	0.0008 (17)	0.0044 (18)	-0.0001 (19)
C3	0.044 (2)	0.042 (2)	0.043 (2)	-0.0052 (18)	0.0043 (18)	-0.0035 (19)
C4	0.042 (2)	0.046 (3)	0.038 (2)	0.0027 (17)	0.0031 (17)	0.0030 (18)
C5	0.045 (2)	0.043 (2)	0.037 (2)	0.0052 (18)	0.0067 (18)	0.0068 (19)
C6	0.058 (2)	0.034 (2)	0.038 (2)	0.0010 (18)	0.0007 (18)	0.0031 (18)
C7	0.057 (2)	0.035 (2)	0.045 (2)	-0.0042 (19)	-0.003 (2)	0.005 (2)
C8	0.043 (2)	0.046 (3)	0.049 (3)	-0.0046 (18)	0.0034 (18)	0.009 (2)
C9	0.044 (2)	0.041 (2)	0.036 (2)	0.0044 (17)	0.0052 (17)	0.0021 (19)
C10	0.044 (2)	0.039 (2)	0.039 (2)	0.0016 (17)	0.0027 (17)	-0.0013 (17)
O11	0.0463 (14)	0.0556 (17)	0.0504 (17)	-0.0071 (13)	0.0128 (13)	-0.0059 (14)
O12	0.0583 (17)	0.054 (2)	0.0540 (19)	-0.0002 (14)	0.0171 (15)	-0.0058 (14)
Br13	0.0923 (4)	0.0435 (3)	0.0465 (3)	0.0055 (2)	0.0083 (2)	-0.0046 (2)

O14	0.0682 (18)	0.0392 (16)	0.064 (2)	-0.0116 (14)	0.0006 (16)	0.0060 (15)
C15	0.054 (2)	0.040 (2)	0.062 (3)	-0.005 (2)	0.011 (2)	-0.013 (2)
O16	0.071 (2)	0.0481 (19)	0.067 (2)	-0.0012 (16)	-0.0046 (16)	0.0045 (17)
C17	0.067 (3)	0.052 (3)	0.101 (4)	-0.014 (2)	0.015 (3)	-0.014 (3)
Br18	0.0579 (3)	0.0773 (4)	0.0723 (4)	-0.0162 (2)	0.0200 (2)	0.0048 (3)
C19	0.048 (2)	0.052 (3)	0.038 (2)	0.0083 (19)	0.0036 (18)	0.0017 (19)
C20	0.056 (2)	0.075 (3)	0.050 (3)	-0.003 (2)	0.012 (2)	-0.003 (2)
C21	0.064 (3)	0.105 (4)	0.058 (3)	0.003 (3)	0.025 (2)	-0.011 (3)
C22	0.083 (3)	0.094 (4)	0.051 (3)	0.011 (3)	0.024 (3)	-0.020 (3)
C23	0.091 (4)	0.081 (4)	0.065 (3)	-0.015 (3)	0.026 (3)	-0.033 (3)
C24	0.070 (3)	0.066 (3)	0.063 (3)	-0.006 (2)	0.024 (2)	-0.012 (3)

Geometric parameters (Å, °)

O1—C9	1.365 (4)	O12—H12	0.8200
O1—C2	1.366 (4)	O14—C15	1.383 (5)
C2—C3	1.340 (5)	C15—O16	1.179 (5)
C2—C19	1.461 (5)	C15—C17	1.478 (6)
C3—C4	1.425 (5)	C17—H17A	0.9600
C3—H3	0.9300	C17—H17B	0.9600
C4—O11	1.249 (4)	C17—H17C	0.9600
C4—C10	1.454 (5)	C19—C20	1.385 (5)
C5—O12	1.337 (4)	C19—C24	1.385 (6)
C5—C6	1.388 (5)	C20—C21	1.383 (6)
C5—C10	1.401 (5)	C20—H20	0.9300
C6—C7	1.392 (6)	C21—C22	1.359 (7)
C6—Br13	1.877 (4)	C21—H21	0.9300
C7—C8	1.376 (6)	C22—C23	1.362 (7)
C7—O14	1.387 (4)	C22—H22	0.9300
C8—C9	1.387 (5)	C23—C24	1.376 (6)
C8—Br18	1.878 (4)	C23—H23	0.9300
C9—C10	1.391 (5)	C24—H24	0.9300
C9—O1—C2	120.6 (3)	C15—O14—C7	117.5 (3)
C3—C2—O1	120.7 (3)	O16—C15—O14	121.5 (4)
C3—C2—C19	127.2 (4)	O16—C15—C17	128.7 (4)
O1—C2—C19	112.1 (3)	O14—C15—C17	109.8 (4)
C2—C3—C4	122.6 (4)	C15—C17—H17A	109.5
C2—C3—H3	118.7	C15—C17—H17B	109.5
C4—C3—H3	118.7	H17A—C17—H17B	109.5
O11—C4—C3	123.1 (3)	C15—C17—H17C	109.5
O11—C4—C10	121.1 (3)	H17A—C17—H17C	109.5
C3—C4—C10	115.8 (3)	H17B—C17—H17C	109.5
O12—C5—C6	119.5 (4)	C20—C19—C24	118.9 (4)
O12—C5—C10	120.8 (3)	C20—C19—C2	120.5 (4)
C6—C5—C10	119.6 (3)	C24—C19—C2	120.6 (4)
C5—C6—C7	119.6 (4)	C21—C20—C19	119.5 (4)
C5—C6—Br13	120.0 (3)	C21—C20—H20	120.3

C7—C6—Br13	120.4 (3)	C19—C20—H20	120.3
C8—C7—O14	119.2 (4)	C22—C21—C20	121.0 (5)
C8—C7—C6	121.7 (4)	C22—C21—H21	119.5
O14—C7—C6	119.0 (4)	C20—C21—H21	119.5
C7—C8—C9	118.2 (4)	C21—C22—C23	120.0 (5)
C7—C8—Br18	121.3 (3)	C21—C22—H22	120.0
C9—C8—Br18	120.5 (3)	C23—C22—H22	120.0
O1—C9—C8	117.0 (3)	C22—C23—C24	120.3 (5)
O1—C9—C10	121.3 (3)	C22—C23—H23	119.9
C8—C9—C10	121.7 (4)	C24—C23—H23	119.9
C9—C10—C5	119.1 (3)	C23—C24—C19	120.4 (4)
C9—C10—C4	119.0 (3)	C23—C24—H24	119.8
C5—C10—C4	121.9 (3)	C19—C24—H24	119.8
C5—O12—H12	109.5		
C9—O1—C2—C3	-2.9 (5)	O1—C9—C10—C4	0.8 (5)
C9—O1—C2—C19	176.1 (3)	C8—C9—C10—C4	-179.1 (3)
O1—C2—C3—C4	2.4 (6)	O12—C5—C10—C9	179.8 (3)
C19—C2—C3—C4	-176.5 (4)	C6—C5—C10—C9	0.7 (5)
C2—C3—C4—O11	178.4 (4)	O12—C5—C10—C4	0.4 (5)
C2—C3—C4—C10	-0.3 (5)	C6—C5—C10—C4	-178.7 (3)
O12—C5—C6—C7	179.8 (3)	O11—C4—C10—C9	180.0 (3)
C10—C5—C6—C7	-1.1 (5)	C3—C4—C10—C9	-1.3 (5)
O12—C5—C6—Br13	-2.3 (5)	O11—C4—C10—C5	-0.6 (6)
C10—C5—C6—Br13	176.8 (3)	C3—C4—C10—C5	178.2 (3)
C5—C6—C7—C8	-0.7 (6)	C8—C7—O14—C15	-95.3 (4)
Br13—C6—C7—C8	-178.6 (3)	C6—C7—O14—C15	89.3 (4)
C5—C6—C7—O14	174.6 (3)	C7—O14—C15—O16	3.5 (6)
Br13—C6—C7—O14	-3.2 (5)	C7—O14—C15—C17	-177.8 (4)
O14—C7—C8—C9	-172.6 (3)	C3—C2—C19—C20	178.7 (4)
C6—C7—C8—C9	2.7 (6)	O1—C2—C19—C20	-0.3 (5)
O14—C7—C8—Br18	7.0 (5)	C3—C2—C19—C24	-0.4 (6)
C6—C7—C8—Br18	-177.7 (3)	O1—C2—C19—C24	-179.4 (4)
C2—O1—C9—C8	-178.8 (3)	C24—C19—C20—C21	-1.1 (6)
C2—O1—C9—C10	1.3 (5)	C2—C19—C20—C21	179.7 (4)
C7—C8—C9—O1	177.0 (3)	C19—C20—C21—C22	0.2 (7)
Br18—C8—C9—O1	-2.6 (5)	C20—C21—C22—C23	0.5 (8)
C7—C8—C9—C10	-3.1 (6)	C21—C22—C23—C24	-0.3 (8)
Br18—C8—C9—C10	177.4 (3)	C22—C23—C24—C19	-0.6 (8)
O1—C9—C10—C5	-178.7 (3)	C20—C19—C24—C23	1.4 (7)
C8—C9—C10—C5	1.4 (5)	C2—C19—C24—C23	-179.5 (4)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O12—H12 \cdots O11	0.82	1.86	2.584 (4)	147
C20—H20 \cdots O1	0.93	2.34	2.679 (5)	101
C3—H3 \cdots O11 ⁱ	0.93	2.57	3.497 (4)	171

C24—H24···O11 ⁱ	0.93	2.48	3.387 (5)	166
C17—H17A···O16 ⁱⁱ	0.96	2.58	3.309 (6)	133

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