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2,4-Dibromo-1,3-dihydroxy-9*H*-xanthen-9-one

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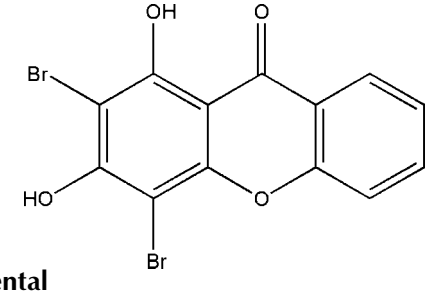
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.034; wR factor = 0.070; data-to-parameter ratio = 12.3.

The title compound, $C_{13}H_6Br_2O_4$, derived from xanthone, a fundamental structural framework of active ingredients in many medicinal plants, and was synthesized by bromination of 1,3-dihydroxyxanthen-9-one with *N*-bromosuccinimide. The molecular conformation is essentially planar, the dihedral angle between the benzene rings being 1.1 (4)°. This conformation is favorable for the formation of an intramolecular O—H···O hydrogen bond between a hydroxy group and the xanthone carbonyl group. In the crystal, molecules are associated into chains along the *b*-axis direction *via* C=O···H—O hydrogen bonds involving the other hydroxy group.

Related literature

For the pharmacological activity of xanthone derivatives, see: Cheng *et al.* (2011); Dao *et al.* (2012); Sousa *et al.* (2009); Szkaradek *et al.* (2013). For the synthesis of the xanthone used as a starting material, see: Liu *et al.* (2006). For related xanthone structures, see: Corrêa *et al.* (2010).



Experimental

Crystal data

$C_{13}H_6Br_2O_4$
M_r = 386.00
Orthorhombic, $Pna2_1$
a = 18.4489 (15) Å
b = 16.9049 (13) Å
c = 3.8564 (3) Å
V = 1202.72 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 6.75 mm^{-1}
T = 298 K
0.28 × 0.09 × 0.06 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.254, T_{max} = 0.688
6188 measured reflections
2120 independent reflections
1830 reflections with $I > 2\sigma(I)$
R_{int} = 0.076

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.070
S = 1.04
2120 reflections
172 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Absolute structure: Flack (1983), 881 Friedel pairs
Absolute structure parameter: −0.008 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O2	0.82	1.81	2.555 (7)	149
O3—H3···O2^{i}	0.82	2.02	2.741 (7)	147

Symmetry code: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXL97* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2480).

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2,4-Dibromo-1,3-dihydroxy-9*H*-xanthen-9-one

Shi-Wen Huang, Zheng-Min Yang, Fu-Ping Huang and Jiang-Ke Qin

S1. Comment

Xanthone, also named as dibenzo-γ-pyrone, is a fundamental structural framework of active ingredients in many medicinal plants, which derivatives have broad pharmacological activities, such as antioxidant (Cheng *et al.*, 2011), antitumor (Sousa *et al.*, 2009), anticonvulsant (Szkaradek *et al.*, 2013) and inhibition of neuraminidase activity (Dao *et al.*, 2012). The title compound in this study is a new xanthone derivative, which was synthesized by bromination of 1,3-dihydroxy-xanthen-9-one (Liu *et al.*, 2006) with NBS.

We report here the synthesis and crystal structure of 2,4-dibromo-1,3-dihydroxy-xanthen-9-one ($C_{13}H_6Br_2O_4$, Fig. 1). The molecule of the title compound has a planar conformation, with characteristic bond lengths C2—Br1 = 1.893 (5) Å and C4—Br2 = 1.902 (5) Å. The molecular conformation is mainly controlled by the O4—H4···O2 intramolecular hydrogen bond between the hydroxy OH group and the carbonyl O atom. Molecules are further connected into a one-dimensional supramolecular architecture *via* O3—H3···O2 intermolecular hydrogen bonds (Fig. 2). The title compound has the same planar molecular conformation as that reported for other 1-hydroxy-9*H*-xanthen-9-one derivatives (Corrêa *et al.*, 2010), while the carbonyl bond length C7═O2, 1.262 (7) Å, is slightly larger than the corresponding carbonyl bond lengths in these derivatives.

S2. Experimental

The title compound was synthesized using the following procedure: in a 50 ml flask, 1,3-dihydroxy-xanthen-9-one (1010 mg, 4.43 mmol; Liu *et al.*, 2006) was dissolved in CCl_4 (15 ml), then NBS (500 mg, 2.83 mmol) was added. The mixture was stirred at room temperature for 24 h. The residue was washed with acetone and then filtered. The yellow solid was collected and dried. Recrystallization from methanol solution afforded 2,4-dibromo-1,3-dihydroxy-xanthen-9-one as yellow crystals. The compound identity was confirmed by NMR spectroscopy. ^{1}H NMR (500 MHz, DMSO-d_6): 13.63 (*s*, 1H), 8.09 (*dd*, *J* = 7.9, 1.6 Hz, 1H), 7.87 (*td*, *J* = 7.8, 1.5 Hz, 1H), 7.59 (*d*, *J* = 8.4 Hz, 1H), 7.48 (*t*, *J* = 7.5 Hz, 1H).

S3. Refinement

The H atoms on C and O atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C), and with O—H = 0.82 Å and U_{iso}(H) = 1.5U_{eq}(O).

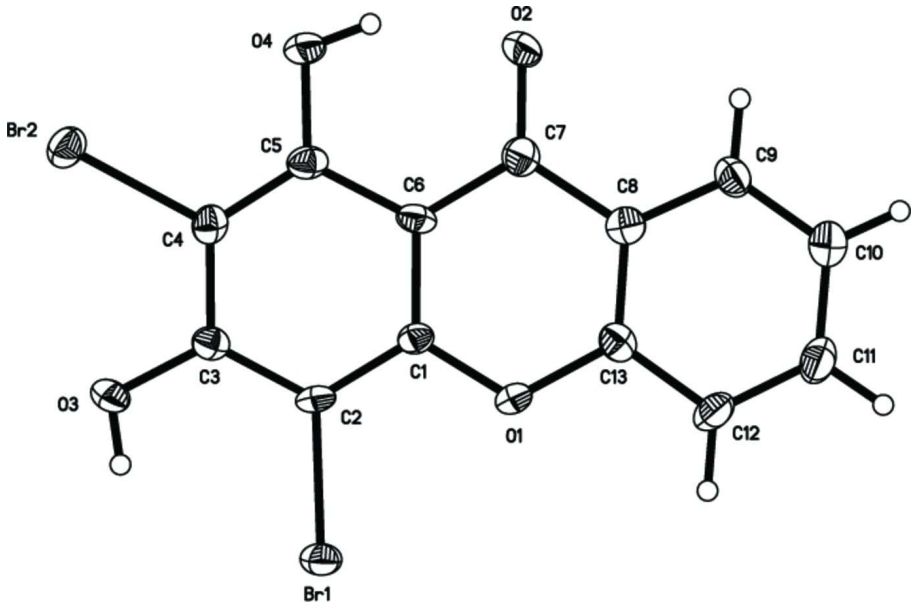

Figure 1

The structure of the title compound and the atom-numbering scheme.

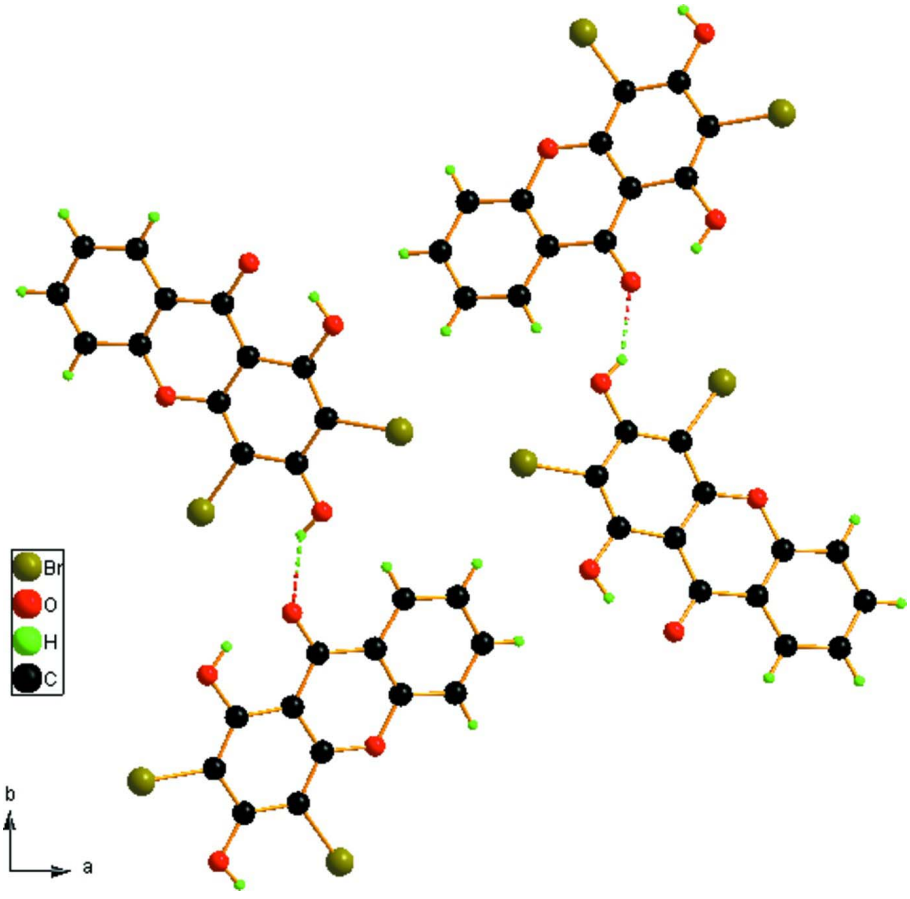

Figure 2

The packing diagram of the title compound.

2,4-Dibromo-1,3-dihydroxy-9*H*-xanthen-9-one

Crystal data

$C_{13}H_6Br_2O_4$
M_r = 386.00
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
a = 18.4489 (15) Å
b = 16.9049 (13) Å
c = 3.8564 (3) Å
V = 1202.72 (16) Å^3
Z = 4
$F(000)$ = 744
D_x = 2.132 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2778 reflections
θ = 3.3–26.2°
μ = 6.75 mm^{-1}
T = 298 K
Block, yellow
0.28 × 0.09 × 0.06 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.254, T_{max} = 0.688
6188 measured reflections
2120 independent reflections
1830 reflections with $I > 2\sigma(I)$
R_{int} = 0.076
θ_{max} = 25.0°, θ_{min} = 1.6°
h = −21→21
k = −20→15
l = −4→4

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.070
S = 1.04
2120 reflections
172 parameters
1 restraint
0 constraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0232P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Absolute structure: Flack (1983), 881 Friedel pairs
Absolute structure parameter: −0.008 (16)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.65912 (3)	1.09241 (3)	−0.07056 (16)	0.03711 (16)
Br2	0.91885 (3)	0.97740 (3)	0.54899 (17)	0.03972 (17)
O1	0.61335 (17)	0.9267 (2)	0.0466 (12)	0.0366 (9)
O2	0.72160 (18)	0.7368 (2)	0.4658 (13)	0.0483 (11)
O3	0.8177 (2)	1.0920 (2)	0.2248 (11)	0.0408 (11)
H3	0.7904	1.1248	0.1377	0.061*
O4	0.83232 (18)	0.8253 (2)	0.5513 (13)	0.0452 (11)
H4	0.8074	0.7851	0.5521	0.068*

C1	0.6824 (3)	0.9348 (3)	0.1582 (13)	0.0262 (13)
C2	0.7137 (3)	1.0086 (3)	0.1241 (13)	0.0277 (13)
C3	0.7844 (3)	1.0223 (3)	0.2401 (14)	0.0293 (13)
C4	0.8233 (3)	0.9589 (3)	0.3828 (15)	0.0310 (14)
C5	0.7941 (3)	0.8853 (3)	0.4141 (16)	0.0323 (13)
C6	0.7210 (3)	0.8704 (3)	0.2998 (14)	0.0305 (14)
C7	0.6879 (3)	0.7943 (4)	0.3340 (15)	0.0351 (15)
C8	0.6131 (3)	0.7875 (3)	0.2015 (14)	0.0337 (14)
C9	0.5753 (3)	0.7164 (4)	0.2048 (17)	0.0400 (15)
H9	0.5977	0.6707	0.2858	0.048*
C10	0.5053 (3)	0.7135 (4)	0.0887 (19)	0.0479 (16)
H10	0.4797	0.6661	0.0978	0.057*
C11	0.4724 (3)	0.7802 (4)	−0.0412 (18)	0.0458 (15)
H11	0.4251	0.7770	−0.1236	0.055*
C12	0.5080 (3)	0.8511 (3)	−0.0515 (17)	0.0410 (14)
H12	0.4853	0.8965	−0.1333	0.049*
C13	0.5793 (3)	0.8533 (3)	0.0642 (17)	0.0314 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0367 (3)	0.0309 (3)	0.0438 (3)	0.0061 (2)	−0.0024 (3)	0.0080 (3)
Br2	0.0277 (3)	0.0461 (4)	0.0454 (3)	−0.0007 (2)	−0.0040 (3)	0.0016 (3)
O1	0.0249 (19)	0.033 (2)	0.052 (2)	0.0044 (15)	−0.007 (2)	0.008 (2)
O2	0.037 (2)	0.027 (2)	0.081 (3)	0.0009 (17)	−0.006 (3)	0.015 (2)
O3	0.040 (2)	0.025 (2)	0.058 (3)	−0.0020 (19)	−0.005 (2)	0.012 (2)
O4	0.032 (2)	0.037 (2)	0.067 (3)	0.0062 (16)	−0.010 (3)	0.015 (2)
C1	0.025 (3)	0.026 (3)	0.027 (3)	0.006 (2)	0.000 (2)	−0.002 (2)
C2	0.028 (3)	0.028 (3)	0.026 (4)	0.012 (2)	−0.002 (2)	0.001 (2)
C3	0.028 (3)	0.028 (3)	0.032 (3)	0.003 (3)	0.005 (2)	0.003 (3)
C4	0.022 (3)	0.036 (3)	0.034 (3)	0.000 (2)	0.006 (3)	−0.002 (3)
C5	0.027 (3)	0.036 (3)	0.034 (3)	0.006 (2)	0.005 (3)	0.008 (3)
C6	0.033 (3)	0.023 (3)	0.036 (3)	0.008 (2)	0.003 (2)	0.005 (2)
C7	0.028 (3)	0.037 (4)	0.041 (4)	0.001 (3)	0.008 (3)	0.003 (3)
C8	0.037 (4)	0.034 (4)	0.031 (3)	0.002 (3)	−0.001 (3)	−0.003 (3)
C9	0.038 (4)	0.028 (3)	0.054 (4)	−0.004 (3)	0.002 (3)	0.001 (3)
C10	0.040 (4)	0.048 (4)	0.056 (4)	−0.008 (3)	−0.003 (3)	0.003 (3)
C11	0.030 (3)	0.056 (4)	0.051 (4)	−0.007 (3)	−0.004 (3)	0.001 (4)
C12	0.028 (3)	0.048 (4)	0.048 (4)	0.006 (3)	−0.008 (3)	−0.001 (3)
C13	0.034 (3)	0.032 (3)	0.029 (3)	−0.001 (2)	0.010 (3)	0.001 (3)

Geometric parameters (Å, º)

Br1—C2	1.893 (5)	C5—C6	1.442 (7)
Br2—C4	1.902 (5)	C6—C7	1.429 (8)
O1—C1	1.351 (6)	C7—C8	1.476 (8)
O1—C13	1.393 (6)	C8—C13	1.379 (7)
O2—C7	1.262 (7)	C8—C9	1.390 (8)

O3—C3	1.331 (7)	C9—C10	1.368 (8)
O3—H3	0.8200	C9—H9	0.9300
O4—C5	1.343 (6)	C10—C11	1.374 (8)
O4—H4	0.8200	C10—H10	0.9300
C1—C2	1.381 (7)	C11—C12	1.368 (8)
C1—C6	1.411 (7)	C11—H11	0.9300
C2—C3	1.398 (7)	C12—C13	1.389 (7)
C3—C4	1.402 (7)	C12—H12	0.9300
C4—C5	1.361 (7)		

C1—O1—C13	119.9 (4)	O2—C7—C6	121.3 (5)
C3—O3—H3	109.5	O2—C7—C8	122.7 (5)
C5—O4—H4	109.5	C6—C7—C8	115.9 (5)
O1—C1—C2	117.1 (4)	C13—C8—C9	118.4 (5)
O1—C1—C6	121.4 (5)	C13—C8—C7	119.5 (5)
C2—C1—C6	121.5 (5)	C9—C8—C7	122.2 (5)
C1—C2—C3	120.6 (5)	C10—C9—C8	120.0 (6)
C1—C2—Br1	119.4 (4)	C10—C9—H9	120.0
C3—C2—Br1	120.0 (4)	C8—C9—H9	120.0
O3—C3—C2	124.2 (5)	C9—C10—C11	120.5 (6)
O3—C3—C4	117.3 (5)	C9—C10—H10	119.8
C2—C3—C4	118.5 (5)	C11—C10—H10	119.8
C5—C4—C3	122.1 (5)	C12—C11—C10	121.2 (5)
C5—C4—Br2	119.1 (4)	C12—C11—H11	119.4
C3—C4—Br2	118.8 (4)	C10—C11—H11	119.4
O4—C5—C4	121.2 (5)	C11—C12—C13	117.9 (5)
O4—C5—C6	118.6 (5)	C11—C12—H12	121.1
C4—C5—C6	120.2 (5)	C13—C12—H12	121.1
C1—C6—C7	121.0 (5)	C8—C13—C12	122.0 (5)
C1—C6—C5	117.1 (5)	C8—C13—O1	122.3 (5)
C7—C6—C5	121.9 (5)	C12—C13—O1	115.7 (5)

C13—O1—C1—C2	178.1 (5)	O4—C5—C6—C7	1.1 (9)
C13—O1—C1—C6	−1.2 (8)	C4—C5—C6—C7	−178.8 (5)
O1—C1—C2—C3	178.6 (5)	C1—C6—C7—O2	−178.7 (5)
C6—C1—C2—C3	−2.1 (8)	C5—C6—C7—O2	−0.1 (9)
O1—C1—C2—Br1	0.3 (6)	C1—C6—C7—C8	2.1 (8)
C6—C1—C2—Br1	179.6 (4)	C5—C6—C7—C8	−179.2 (5)
C1—C2—C3—O3	−178.2 (5)	O2—C7—C8—C13	179.3 (6)
Br1—C2—C3—O3	0.1 (8)	C6—C7—C8—C13	−1.6 (8)
C1—C2—C3—C4	1.7 (8)	O2—C7—C8—C9	−1.8 (9)
Br1—C2—C3—C4	−180.0 (4)	C6—C7—C8—C9	177.3 (5)
O3—C3—C4—C5	179.3 (5)	C13—C8—C9—C10	−3.0 (9)
C2—C3—C4—C5	−0.6 (9)	C7—C8—C9—C10	178.0 (6)
O3—C3—C4—Br2	1.0 (7)	C8—C9—C10—C11	1.9 (11)
C2—C3—C4—Br2	−178.9 (4)	C9—C10—C11—C12	−1.3 (12)
C3—C4—C5—O4	179.9 (6)	C10—C11—C12—C13	1.9 (10)
Br2—C4—C5—O4	−1.8 (8)	C9—C8—C13—C12	3.7 (9)

C3—C4—C5—C6	−0.2 (9)	C7—C8—C13—C12	−177.4 (6)
Br2—C4—C5—C6	178.1 (4)	C9—C8—C13—O1	−179.2 (6)
O1—C1—C6—C7	−0.8 (8)	C7—C8—C13—O1	−0.3 (9)
C2—C1—C6—C7	179.9 (5)	C11—C12—C13—C8	−3.1 (10)
O1—C1—C6—C5	−179.5 (5)	C11—C12—C13—O1	179.6 (6)
C2—C1—C6—C5	1.2 (8)	C1—O1—C13—C8	1.7 (9)
O4—C5—C6—C1	179.8 (5)	C1—O1—C13—C12	179.0 (5)
C4—C5—C6—C1	−0.1 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···Br1	0.82	2.61	3.139 (5)	124
O4—H4···O2	0.82	1.81	2.555 (7)	149
O3—H3···O2^{i}	0.82	2.02	2.741 (7)	147

Symmetry code: (i) −*x*+3/2, *y*+1/2, *z*−1/2.