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4-(8-Hydroxy-3-methyl-1,4-dioxo-1,4-dihydro-2-naphthyl)butanoic acid

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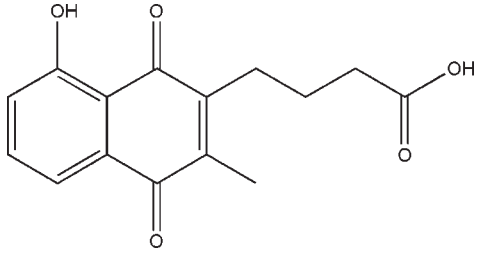
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.073; wR factor = 0.198; data-to-parameter ratio = 13.1.

In the title compound, $C_{15}H_{14}O_5$, an intramolecular O—H···O hydrogen bond occurs. In the crystal, the molecules form inversion dimers linked by pairs of O—H···O bonds, which are further linked by C—H···O interactions.

Related literature

For the synthesis and biological properties of the title compound, see: Salmon-Chemin *et al.* (2001). For crystal structures of similar compounds, see: Vijayalakshmi *et al.* (1987); Ghouse & Rao (1974).



Experimental

Crystal data

$C_{15}H_{14}O_5$
M_r = 274.26
Monoclinic, $P2_1/n$
a = 10.881 (3) Å
b = 9.973 (2) Å
c = 12.705 (3) Å
β = 106.936 (5)°
V = 1319.0 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.45 × 0.30 × 0.24 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (REQAB: Jacobson, 1998)
T_{min} = 0.734, T_{max} = 0.975
11416 measured reflections
2405 independent reflections
1779 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.198
S = 1.09
2405 reflections
184 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2^{i}	0.93	2.43	3.315 (4)	160
O5—H5···O4^{ii}	0.82	1.77	2.589 (3)	174
O1—H1···O3	0.82	1.87	2.582 (3)	145

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

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC & Rigaku, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2192).

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Salmon-Chemin, L., Buisine, E., Yardley, V., Kohler, S., Debreu, M.-A., Landry, V., Sergheraert, C., Croft, S. L., Krauth-Siegel, L. & Davioud-Charvet, E. (2001). *J. Med. Chem.* **44**, 548–565.
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supplementary materials

Acta Cryst. (2009). E**65**, o2737 [**doi:10.1107/S1600536809040021**]

4-(8-Hydroxy-3-methyl-1,4-dioxo-1,4-dihydro-2-naphthyl)butanoic acid

Y.-F. Wang, H. Tang, Y.-C. Liu, Z.-F. Chen and H. Liang

Comment

Plumbagin is a potent toxic natural product extracted from Plumbago Zeylanica *L*. (Plumbaginaceae), which has been used in China as well as other Asian countries for the treatment of rheumatoid arthritis, dysmenorrhea, injury by bumping, and even cancer. The title compound is a 2-substituted 1,4-naphthoquinone derivative. Its synthesis has been reported by Salmon-Chemin *et al.*(2001), we now report its structure. The molecular structure of the title compound is shown in Fig.1. The bond lengths and angles of the napthoquinone molecule are normal and comparable to those of plumbagin (Ghouse & Rao, 1974; Vijayalakshmi, *et al.*, 1987). Geometric parameters for the butanoic acid group are also normal. As shown in Fig.2, a two-dimensional network is generated *via* intermolecular hydrogen bond interactions involving C—H···O, O—H···O.

Experimental

0.2 mmol compound were dissolved in 10 ml methanol and 10 ml CH_2Cl_2. The resulting red solution was filtered. The filtrate was allowed to sit under ambient conditions for two weeks, dark-red block crystals were obtained.

Refinement

The H bound to C atoms of naphthoquinone, and to C(11) as well as to C(12)—C(14) were treated as riding, with C—H distances of 0.93, 0.96 and 0.97Å with $U_{iso}(H) = 1.2U_{eq}(C)$, respectively. Hydroxyl O—H distances were set to 0.82Å and were refined as riding with $U_{iso}(H) = 1.5U_{eq}(O)$.

Figures

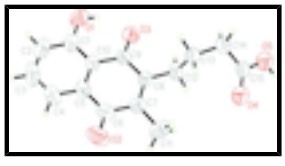

Fig. 1. The molecular structure showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

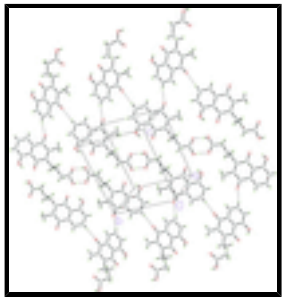

Fig. 2. Packing of molecules roughly down the [100] direction showing the two-dimensional network of molecules. Hydrogen bonds are shown as dashed lines.

4-(8-Hydroxy-3-methyl-1,4-dioxo-1,4-dihydro-2-naphthyl)butanoic acid

Crystal data

$C_{15}H_{14}O_5$	$F_{000} = 576$
$M_r = 274.26$	$D_x = 1.381$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å
Hall symbol: -P 2yn	Cell parameters from 3725 reflections
$a = 10.881$ (3) Å	$\theta = 3.4$–25.3º
$b = 9.973$ (2) Å	$\mu = 0.10$ mm^{-1}
$c = 12.705$ (3) Å	$T = 293$ K
$\beta = 106.936$ (5)º	Block, dark-red
$V = 1319.0$ (6) Å^3	0.45 × 0.30 × 0.24 mm
$Z = 4$	

Data collection

Rigaku Mercury CCD diffractometer	2405 independent reflections
Radiation source: fine-focus sealed tube	1779 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.038$
Detector resolution: 7.31 pixels mm^{-1}	$\theta_{max} = 25.4$º
$T = 293$ K	$\theta_{min} = 3.4$º
ω scans	$h = -13 \rightarrow 13$
Absorption correction: multi-scan (REQAB: Jacobson, 1998)	$k = -11 \rightarrow 12$
$T_{min} = 0.734$, $T_{max} = 0.975$	$l = -13 \rightarrow 15$
11416 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.073$	H-atom parameters constrained
$wR(F^2) = 0.198$	$w = 1/[\sigma^2(F_o^2) + (0.0888P)^2 + 0.5298P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} < 0.001$
2405 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
184 parameters	$\Delta\rho_{min} = -0.25$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.9902 (2)	0.8451 (2)	0.6287 (2)	0.0868 (8)
H1	1.0115	0.8001	0.6851	0.130*
O2	0.7285 (3)	0.3526 (3)	0.4035 (2)	0.0943 (9)
O3	1.0036 (2)	0.6313 (2)	0.74605 (17)	0.0752 (7)
O4	0.9090 (2)	0.1025 (2)	0.90860 (19)	0.0749 (7)
O5	1.0253 (3)	0.1478 (2)	1.08016 (19)	0.0808 (8)
H5	1.0411	0.0674	1.0806	0.121*
C1	0.9241 (3)	0.7683 (3)	0.5446 (3)	0.0619 (8)
C2	0.8795 (3)	0.8269 (4)	0.4409 (3)	0.0741 (10)
H2	0.8964	0.9169	0.4317	0.089*
C3	0.8115 (3)	0.7538 (4)	0.3534 (3)	0.0782 (11)
H3	0.7836	0.7940	0.2846	0.094*
C4	0.7827 (3)	0.6200 (4)	0.3646 (2)	0.0665 (9)
H4	0.7346	0.5716	0.3040	0.080*
C5	0.8260 (2)	0.5593 (3)	0.4661 (2)	0.0516 (7)
C6	0.7964 (3)	0.4165 (3)	0.4808 (2)	0.0587 (8)
C7	0.8472 (3)	0.3520 (3)	0.5893 (2)	0.0523 (7)
C8	0.9164 (2)	0.4235 (3)	0.6766 (2)	0.0475 (7)
C9	0.9428 (3)	0.5675 (3)	0.6644 (2)	0.0490 (7)
C10	0.8974 (2)	0.6322 (3)	0.5572 (2)	0.0480 (7)
C11	0.8153 (3)	0.2063 (3)	0.5962 (3)	0.0781 (10)
H11A	0.8895	0.1597	0.6407	0.117*
H11B	0.7897	0.1682	0.5237	0.117*
H11C	0.7462	0.1978	0.6287	0.117*
C12	0.9674 (3)	0.3671 (3)	0.7905 (2)	0.0565 (8)
H12A	1.0433	0.4168	0.8303	0.068*
H12B	0.9923	0.2744	0.7859	0.068*
C13	0.8682 (3)	0.3741 (3)	0.8535 (2)	0.0593 (8)
H13A	0.8385	0.4658	0.8531	0.071*
H13B	0.7949	0.3189	0.8165	0.071*
C14	0.9219 (3)	0.3269 (3)	0.9717 (3)	0.0640 (8)
H14A	0.9995	0.3771	1.0063	0.077*
H14B	0.8600	0.3470	1.0111	0.077*

C15	0.9521 (3)	0.1824 (3)	0.9828 (3)	0.0582 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1058 (19)	0.0542 (14)	0.0918 (18)	−0.0175 (13)	0.0155 (15)	0.0019 (12)
O2	0.1062 (19)	0.106 (2)	0.0626 (15)	−0.0342 (16)	0.0118 (14)	−0.0271 (14)
O3	0.0995 (17)	0.0634 (13)	0.0507 (12)	−0.0164 (12)	0.0031 (12)	−0.0043 (10)
O4	0.0928 (17)	0.0626 (14)	0.0654 (15)	0.0010 (12)	0.0167 (13)	0.0090 (11)
O5	0.1021 (18)	0.0667 (15)	0.0655 (15)	0.0022 (14)	0.0119 (13)	0.0084 (11)
C1	0.0604 (17)	0.0583 (19)	0.067 (2)	0.0009 (15)	0.0189 (16)	0.0156 (16)
C2	0.074 (2)	0.065 (2)	0.087 (3)	0.0133 (17)	0.030 (2)	0.0290 (19)
C3	0.069 (2)	0.106 (3)	0.064 (2)	0.024 (2)	0.0276 (18)	0.040 (2)
C4	0.0576 (18)	0.097 (3)	0.0443 (16)	0.0080 (17)	0.0132 (14)	0.0075 (16)
C5	0.0471 (15)	0.0657 (19)	0.0428 (15)	0.0011 (13)	0.0143 (13)	0.0042 (13)
C6	0.0525 (16)	0.073 (2)	0.0518 (17)	−0.0070 (15)	0.0166 (14)	−0.0131 (15)
C7	0.0511 (15)	0.0492 (16)	0.0593 (17)	−0.0005 (13)	0.0204 (14)	−0.0020 (13)
C8	0.0454 (14)	0.0504 (16)	0.0496 (15)	0.0017 (12)	0.0183 (12)	0.0047 (12)
C9	0.0519 (15)	0.0507 (16)	0.0427 (15)	−0.0022 (13)	0.0110 (13)	0.0008 (12)
C10	0.0479 (15)	0.0533 (16)	0.0429 (15)	0.0017 (12)	0.0134 (12)	0.0071 (12)
C11	0.079 (2)	0.0543 (19)	0.104 (3)	−0.0133 (17)	0.031 (2)	−0.0053 (18)
C12	0.0590 (17)	0.0578 (18)	0.0545 (17)	0.0092 (14)	0.0195 (14)	0.0184 (13)
C13	0.0690 (19)	0.0570 (18)	0.0561 (17)	0.0093 (14)	0.0247 (15)	0.0090 (14)
C14	0.080 (2)	0.060 (2)	0.0567 (18)	0.0069 (16)	0.0276 (16)	0.0067 (14)
C15	0.0665 (18)	0.062 (2)	0.0472 (16)	−0.0049 (15)	0.0177 (14)	0.0079 (14)

Geometric parameters (Å, °)

O1—C1	1.341 (4)	C7—C8	1.349 (4)
O1—H1	0.8200	C7—C11	1.502 (4)
O2—C6	1.222 (3)	C8—C9	1.482 (4)
O3—C9	1.232 (3)	C8—C12	1.500 (4)
O4—C15	1.220 (4)	C9—C10	1.457 (4)
O5—C15	1.307 (4)	C11—H11A	0.9600
O5—H5	0.8200	C11—H11B	0.9600
C1—C2	1.393 (4)	C11—H11C	0.9600
C1—C10	1.407 (4)	C12—C13	1.521 (4)
C2—C3	1.355 (5)	C12—H12A	0.9700
C2—H2	0.9300	C12—H12B	0.9700
C3—C4	1.387 (5)	C13—C14	1.520 (4)
C3—H3	0.9300	C13—H13A	0.9700
C4—C5	1.377 (4)	C13—H13B	0.9700
C4—H4	0.9300	C14—C15	1.476 (4)
C5—C10	1.395 (4)	C14—H14A	0.9700
C5—C6	1.484 (4)	C14—H14B	0.9700
C6—C7	1.475 (4)		
C1—O1—H1	109.5	C5—C10—C9	120.0 (3)
C15—O5—H5	109.5	C1—C10—C9	120.5 (3)

O1—C1—C2	118.1 (3)	C7—C11—H11A	109.5
O1—C1—C10	122.8 (3)	C7—C11—H11B	109.5
C2—C1—C10	119.1 (3)	H11A—C11—H11B	109.5
C3—C2—C1	120.4 (3)	C7—C11—H11C	109.5
C3—C2—H2	119.8	H11A—C11—H11C	109.5
C1—C2—H2	119.8	H11B—C11—H11C	109.5
C2—C3—C4	121.3 (3)	C8—C12—C13	111.8 (2)
C2—C3—H3	119.3	C8—C12—H12A	109.3
C4—C3—H3	119.3	C13—C12—H12A	109.3
C5—C4—C3	119.5 (3)	C8—C12—H12B	109.3
C5—C4—H4	120.3	C13—C12—H12B	109.3
C3—C4—H4	120.3	H12A—C12—H12B	107.9
C4—C5—C10	120.2 (3)	C14—C13—C12	112.2 (2)
C4—C5—C6	120.8 (3)	C14—C13—H13A	109.2
C10—C5—C6	119.0 (2)	C12—C13—H13A	109.2
O2—C6—C7	119.9 (3)	C14—C13—H13B	109.2
O2—C6—C5	120.1 (3)	C12—C13—H13B	109.2
C7—C6—C5	120.0 (2)	H13A—C13—H13B	107.9
C8—C7—C6	120.4 (3)	C15—C14—C13	114.1 (3)
C8—C7—C11	123.1 (3)	C15—C14—H14A	108.7
C6—C7—C11	116.5 (3)	C13—C14—H14A	108.7
C7—C8—C9	120.3 (2)	C15—C14—H14B	108.7
C7—C8—C12	123.8 (3)	C13—C14—H14B	108.7
C9—C8—C12	115.9 (2)	H14A—C14—H14B	107.6
O3—C9—C10	120.8 (3)	O4—C15—O5	123.4 (3)
O3—C9—C8	119.0 (2)	O4—C15—C14	122.6 (3)
C10—C9—C8	120.2 (2)	O5—C15—C14	113.9 (3)
C5—C10—C1	119.4 (3)		
O1—C1—C2—C3	−179.5 (3)	C7—C8—C9—C10	−1.9 (4)
C10—C1—C2—C3	−0.1 (5)	C12—C8—C9—C10	−180.0 (2)
C1—C2—C3—C4	1.0 (5)	C4—C5—C10—C1	0.3 (4)
C2—C3—C4—C5	−1.2 (5)	C6—C5—C10—C1	−178.9 (3)
C3—C4—C5—C10	0.5 (4)	C4—C5—C10—C9	179.9 (3)
C3—C4—C5—C6	179.7 (3)	C6—C5—C10—C9	0.7 (4)
C4—C5—C6—O2	−3.0 (4)	O1—C1—C10—C5	178.8 (3)
C10—C5—C6—O2	176.2 (3)	C2—C1—C10—C5	−0.5 (4)
C4—C5—C6—C7	178.0 (3)	O1—C1—C10—C9	−0.7 (4)
C10—C5—C6—C7	−2.8 (4)	C2—C1—C10—C9	179.9 (3)
O2—C6—C7—C8	−176.4 (3)	O3—C9—C10—C5	−178.5 (3)
C5—C6—C7—C8	2.6 (4)	C8—C9—C10—C5	1.7 (4)
O2—C6—C7—C11	2.5 (4)	O3—C9—C10—C1	1.0 (4)
C5—C6—C7—C11	−178.5 (3)	C8—C9—C10—C1	−178.8 (3)
C6—C7—C8—C9	−0.2 (4)	C7—C8—C12—C13	−85.5 (3)
C11—C7—C8—C9	−179.1 (3)	C9—C8—C12—C13	92.5 (3)
C6—C7—C8—C12	177.7 (2)	C8—C12—C13—C14	−175.7 (2)
C11—C7—C8—C12	−1.1 (4)	C12—C13—C14—C15	−67.6 (4)
C7—C8—C9—O3	178.3 (3)	C13—C14—C15—O4	−17.6 (5)
C12—C8—C9—O3	0.2 (4)	C13—C14—C15—O5	164.6 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2^{i}	0.93	2.43	3.315 (4)	160
O5—H5···O4^{ii}	0.82	1.77	2.589 (3)	174
O1—H1···O3	0.82	1.87	2.582 (3)	145

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

Fig. 1

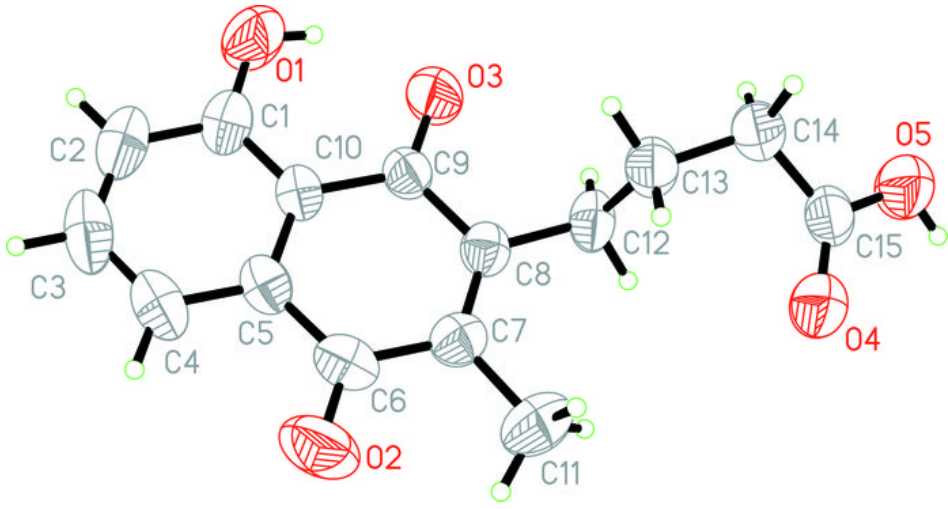

Fig. 2