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2-(1,4-Dioxo-1,4-dihydro-2-naphthyl)-2-methylpropanoic acid

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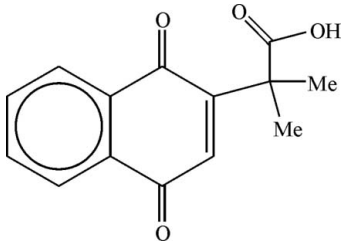
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.092; data-to-parameter ratio = 12.7.

The sterically crowded title compound, $C_{14}H_{12}O_4$, crystallizes as centrosymmetric hydrogen-bonded dimers involving the carboxyl groups. The naphthoquinone ring system is folded by 11.5 (1)° about a vector joining the 1,4-C atoms, and the quinone O atoms are displaced from the ring plane, presumably because of steric interactions with the bulky substituent.

Related literature

For synthesis details, see: Petersen & Heitzer (1972). For related studies of *o*-hydroxycinnamic acids and benzoquinones, see: Karle & Karle (1972); Wang *et al.* (1996). For related naphthoquinone structures, see: Gaultier & Hauw (1965); Gaultier *et al.* (1971).



Experimental

Crystal data

$C_{14}H_{12}O_4$
M_r = 244.24
Monoclinic, $P2_1/c$
a = 8.3070 (2) Å
b = 6.5027 (2) Å
c = 22.0392 (6) Å
β = 97.219 (1)°
V = 1181.07 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 200 (2) K
0.38 × 0.18 × 0.10 mm

Data collection

Siemens SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.790, T_{max} = 0.990
6372 measured reflections
2145 independent reflections
1722 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.092
S = 1.03
2145 reflections
169 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H1···O4^{i}	0.935 (19)	1.711 (19)	2.6455 (15)	176.5 (16)

Symmetry code: (i) $-x, -y+1, -z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We thank Dr Jan Wikaira, University of Canterbury, for collection of X-ray intensity data.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2238).

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supplementary materials

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2-(1,4-Dioxo-1,4-dihydro-2-naphthyl)-2-methylpropanoic acid

K. J. Dempster-Rivett, L. Main, B. K. Nicholson and W. A. Denny

Comment

The title compound was prepared as part of a study to examine constrained conformational effects on lactonization of naphthohydroquinone carboxylic acid derivatives for comparison with related work on sterically crowded *o*-hydroxycinnamic acids and benzoquinone analogues with a "trimethyl-lock" effect (Karle & Karle, 1972; Wang *et al.*, 1996).

The compound crystallizes as a dimer formed about an inversion centre by O—H···O hydrogen bonding between the carboxyl groups. The naphthoquinone ring is folded by 11.5 (1)° about a vector joining the 1,4-carbon atoms and the quinone O atoms are displaced from the ring plane, presumably because of steric interactions with the adjacent geminal dimethyl groups, and with the carboxyl group. Even with the bending, there is still a close intramolecular contact between O1 and O3 (2.922 (2) Å) which suggests that intramolecular O3—H1···O1 hydrogen bonding might be possible if the intermolecular dimer form was not preferred. Naphthoquinone itself, and other 2-substituted naphthoquinones (*e.g.* the 2-iodo derivative) are planar (Gaultier and Hauw, 1965; Gaultier *et al.*, 1971). However, benzoquinones with tri-substituted carbon atoms in the 2-position also show displacement of the quinone O atoms from the plane of the ring (Wang *et al.*, 1996).

Experimental

The title compound was prepared using the method of Petersen & Heitzer (1972). Crystals suitable for X-ray analysis were grown from a solution in toluene.

Refinement

The carboxyl H atom was located in a penultimate difference Fourier map and its position was refined freely with U_{iso} = 0.05 Å^2. All other H atoms were positioned geometrically and refined using a riding model with C—H = 0.95 Å, U_{iso}(H) = 1.2U_{eq}(C) for aromatic H, and with C—H = 0.96 Å, U_{iso}(H) = 1.5U_{eq}(C) for the methyl groups.

Figures

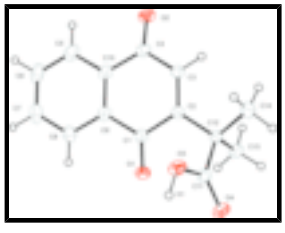

Fig. 1. The molecular structure of the title compound with displacement parameters drawn at the 30% probability level for non-H atoms.

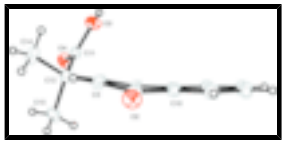

Fig. 2. Side-on view showing the folding of the aromatic ring.

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Crystal data

$C_{14}H_{12}O_4$	F_{000} = 512
M_r = 244.24	D_x = 1.374 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3955 reflections
a = 8.3070 (2) Å	θ = 2–25º
b = 6.5027 (2) Å	μ = 0.10 mm^{-1}
c = 22.0392 (6) Å	T = 200 (2) K
β = 97.219 (1)º	Needle, yellow
V = 1181.07 (6) Å^3	0.38 × 0.18 × 0.10 mm
Z = 4	

Data collection

Siemens SMART CCD diffractometer	2145 independent reflections
Radiation source: fine-focus sealed tube	1722 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
T = 200(2) K	θ_{max} = 25.4º
ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	h = −9→9
T_{min} = 0.790, T_{max} = 0.990	k = 0→7
6372 measured reflections	l = 0→26

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.033	$w = 1/[\sigma^2(F_o^2) + (0.0458P)^2 + 0.2618P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.092	$(\Delta/\sigma)_{max} < 0.001$
S = 1.03	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
2145 reflections	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
169 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.014 (2)
Secondary atom site location: difference Fourier map	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.22816 (14)	0.56642 (15)	0.14194 (5)	0.0427 (3)
O2	0.40002 (14)	−0.08507 (17)	0.27913 (5)	0.0483 (3)
O3	0.03089 (13)	0.29681 (18)	0.05640 (5)	0.0412 (3)
O4	0.19621 (13)	0.46089 (17)	0.00002 (5)	0.0435 (3)
H1	−0.046 (2)	0.386 (3)	0.0360 (8)	0.050*
C1	0.24736 (16)	0.4101 (2)	0.17254 (6)	0.0307 (3)
C2	0.29793 (16)	0.2139 (2)	0.14460 (6)	0.0289 (3)
C3	0.33983 (17)	0.0523 (2)	0.18060 (6)	0.0330 (3)
H3	0.3707	−0.0713	0.1622	0.040*
C4	0.34047 (17)	0.0569 (2)	0.24761 (6)	0.0350 (3)
C5	0.23413 (19)	0.2327 (3)	0.33547 (7)	0.0469 (4)
H5	0.2649	0.1169	0.3605	0.056*
C6	0.1598 (2)	0.3998 (3)	0.35918 (7)	0.0540 (5)
H6	0.1387	0.3978	0.4006	0.065*
C7	0.11625 (19)	0.5692 (3)	0.32321 (8)	0.0507 (5)
H7	0.0662	0.6835	0.3401	0.061*
C8	0.14484 (18)	0.5740 (3)	0.26257 (7)	0.0400 (4)
H8	0.1146	0.6912	0.2380	0.048*
C9	0.21794 (16)	0.4066 (2)	0.23789 (6)	0.0328 (3)
C10	0.26363 (16)	0.2354 (2)	0.27450 (6)	0.0351 (3)
C11	0.17484 (18)	0.3414 (2)	0.04156 (6)	0.0322 (3)
C12	0.31285 (17)	0.2190 (2)	0.07637 (6)	0.0327 (3)
C13	0.47669 (18)	0.3179 (3)	0.06739 (7)	0.0423 (4)
H13A	0.4896	0.3180	0.0238	0.063*
H13B	0.5651	0.2390	0.0901	0.063*
H13C	0.4797	0.4597	0.0826	0.063*
C14	0.3015 (2)	0.0004 (3)	0.04859 (7)	0.0491 (4)
H14A	0.3959	−0.0803	0.0658	0.074*
H14B	0.2993	0.0095	0.0041	0.074*
H14C	0.2021	−0.0665	0.0582	0.074*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0608 (7)	0.0313 (6)	0.0360 (6)	0.0057 (5)	0.0062 (5)	0.0046 (4)
O2	0.0507 (7)	0.0516 (7)	0.0408 (6)	0.0089 (5)	−0.0010 (5)	0.0170 (5)
O3	0.0335 (6)	0.0501 (7)	0.0389 (6)	−0.0016 (5)	0.0012 (4)	0.0127 (5)
O4	0.0414 (6)	0.0559 (7)	0.0333 (6)	0.0028 (5)	0.0054 (4)	0.0165 (5)
C1	0.0292 (7)	0.0327 (8)	0.0294 (7)	−0.0012 (6)	0.0004 (5)	−0.0002 (6)
C2	0.0273 (7)	0.0312 (7)	0.0277 (7)	−0.0016 (5)	0.0013 (5)	−0.0001 (6)
C3	0.0347 (7)	0.0313 (7)	0.0322 (8)	0.0009 (6)	0.0009 (6)	0.0019 (6)
C4	0.0300 (7)	0.0400 (8)	0.0334 (8)	−0.0016 (6)	−0.0016 (6)	0.0085 (6)
C5	0.0378 (9)	0.0710 (12)	0.0310 (8)	0.0018 (8)	0.0008 (6)	0.0071 (8)
C6	0.0410 (9)	0.0934 (14)	0.0277 (8)	0.0036 (9)	0.0052 (7)	−0.0085 (9)

C7	0.0377 (9)	0.0736 (12)	0.0407 (9)	0.0059 (8)	0.0044 (7)	−0.0174 (9)
C8	0.0339 (8)	0.0476 (9)	0.0382 (8)	0.0022 (7)	0.0030 (6)	−0.0070 (7)
C9	0.0273 (7)	0.0399 (8)	0.0305 (7)	−0.0021 (6)	0.0015 (5)	−0.0024 (6)
C10	0.0277 (7)	0.0485 (9)	0.0280 (7)	−0.0017 (6)	−0.0003 (6)	0.0018 (6)
C11	0.0369 (8)	0.0352 (8)	0.0242 (7)	−0.0020 (6)	0.0025 (5)	−0.0009 (6)
C12	0.0381 (8)	0.0339 (8)	0.0261 (7)	0.0033 (6)	0.0034 (6)	0.0006 (6)
C13	0.0370 (8)	0.0554 (10)	0.0351 (8)	0.0052 (7)	0.0071 (6)	0.0072 (7)
C14	0.0724 (12)	0.0417 (9)	0.0321 (8)	0.0088 (8)	0.0024 (7)	−0.0062 (7)

Geometric parameters (Å, °)

O1—C1	1.2193 (17)	C6—H6	0.950
O2—C4	1.2219 (17)	C7—C8	1.387 (2)
O3—C11	1.3113 (17)	C7—H7	0.950
O3—H1	0.935 (19)	C8—C9	1.391 (2)
O4—C11	1.2303 (17)	C8—H8	0.950
C1—C9	1.4910 (19)	C9—C10	1.399 (2)
C1—C2	1.4991 (19)	C11—C12	1.522 (2)
C2—C3	1.3364 (19)	C12—C13	1.540 (2)
C2—C12	1.5246 (18)	C12—C14	1.546 (2)
C3—C4	1.476 (2)	C13—H13A	0.980
C3—H3	0.950	C13—H13B	0.980
C4—C10	1.484 (2)	C13—H13C	0.980
C5—C6	1.384 (3)	C14—H14A	0.980
C5—C10	1.396 (2)	C14—H14B	0.980
C5—H5	0.950	C14—H14C	0.980
C6—C7	1.378 (3)		
C11—O3—H1	109.5 (10)	C8—C9—C1	119.91 (13)
O1—C1—C9	121.39 (13)	C10—C9—C1	120.24 (13)
O1—C1—C2	120.41 (12)	C5—C10—C9	119.82 (14)
C9—C1—C2	118.18 (12)	C5—C10—C4	120.75 (14)
C3—C2—C1	119.38 (12)	C9—C10—C4	119.42 (12)
C3—C2—C12	123.74 (12)	O4—C11—O3	123.14 (13)
C1—C2—C12	116.64 (11)	O4—C11—C12	122.37 (13)
C2—C3—C4	123.10 (13)	O3—C11—C12	114.29 (12)
C2—C3—H3	118.4	C11—C12—C2	111.05 (11)
C4—C3—H3	118.4	C11—C12—C13	109.80 (12)
O2—C4—C3	120.25 (14)	C2—C12—C13	108.57 (11)
O2—C4—C10	121.85 (13)	C11—C12—C14	106.06 (12)
C3—C4—C10	117.88 (12)	C2—C12—C14	111.23 (12)
C6—C5—C10	119.63 (16)	C13—C12—C14	110.11 (13)
C6—C5—H5	120.2	C12—C13—H13A	109.5
C10—C5—H5	120.2	C12—C13—H13B	109.5
C7—C6—C5	120.48 (14)	H13A—C13—H13B	109.5
C7—C6—H6	119.8	C12—C13—H13C	109.5
C5—C6—H6	119.8	H13A—C13—H13C	109.5
C6—C7—C8	120.53 (16)	H13B—C13—H13C	109.5
C6—C7—H7	119.7	C12—C14—H14A	109.5
C8—C7—H7	119.7	C12—C14—H14B	109.5

C7—C8—C9	119.69 (16)	H14A—C14—H14B	109.5
C7—C8—H8	120.2	C12—C14—H14C	109.5
C9—C8—H8	120.2	H14A—C14—H14C	109.5
C8—C9—C10	119.84 (13)	H14B—C14—H14C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1···O4^{i}	0.935 (19)	1.711 (19)	2.6455 (15)	176.5 (16)

Symmetry codes: (i) −*x*, −*y*+1, −*z*.

Fig. 1

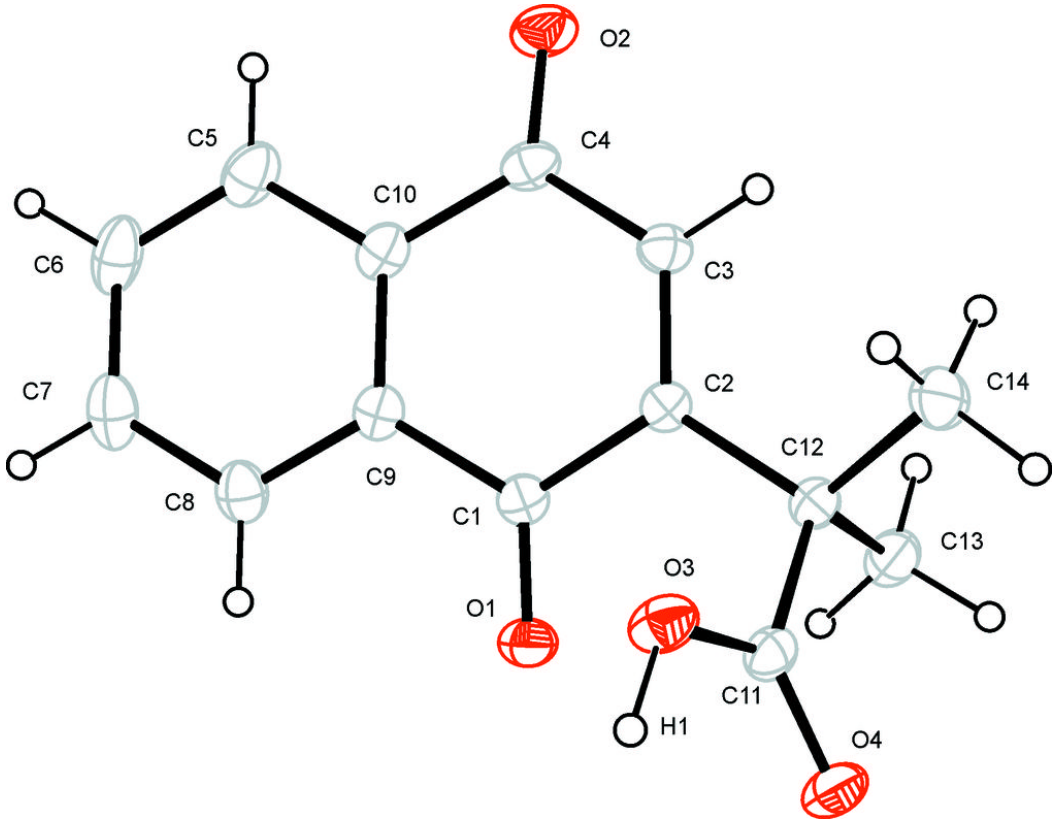

Fig. 2