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(1,1′-Binaphthyl-2,2′-dioxy)diacetic acid

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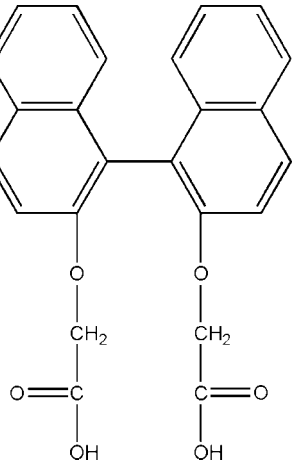
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.062; wR factor = 0.185; data-to-parameter ratio = 14.2.

The asymmetric unit of the title compound, $C_{24}H_{18}O_6$, contains two crystallographically independent molecules. In the crystal structure, there are intramolecular C—H···O and intermolecular O—H···O hydrogen bonds.,

Related literature

For related literature, see: Lehn *et al.* (1978). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{24}H_{18}O_6$
M_r = 402.38
Triclinic, $P\overline{1}$
a = 7.3160 (15) Å
b = 14.067 (3) Å
c = 19.386 (4) Å
α = 85.55 (3)°
β = 83.11 (3)°
γ = 85.50 (3)°
V = 1969.9 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (2) K
0.40 × 0.20 × 0.10 mm

Data collection

Nonius CAD4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.969, T_{max} = 0.988
8349 measured reflections
7704 independent reflections
4677 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.185
S = 0.97
7704 reflections
541 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O7^{i}	0.82	1.78	2.598 (4)	174
O6—H6A···O2^{ii}	0.82	1.96	2.760 (3)	167
O8—H8B···O2^{i}	0.82	1.95	2.770 (4)	179
O12—H12B···O11^{i}	0.82	1.87	2.671 (4)	166
C2—H2A···O5	0.97	2.42	3.360 (5)	162
C47—H47A···O5	0.97	2.39	3.139 (4)	134
C47—H47B···O7	0.97	2.44	3.236 (4)	139

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); 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: HK2284).

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supplementary materials

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(1,1'-Binaphthyl-2,2'-dioxy)diacetic acid

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Comment

The chemistry associated with chiral compounds, such as chiral recognition and asymmetric synthesis, is of particular importance. 1,1'-Bi-2-naphthol (BINOL) and its derivatives have been widely used in asymmetric synthesis, *e.g.* as effective chiral ligands for various metal complex catalysts. We report herein the crystal structure of the title compound, (I).

The asymmetric unit of the title compound, (I), contains two crystallographicaly independent molecules, in which the naphthyl ring systems are planar (Fig. 1). The bond lengths and angles are generally within normal ranges (Allen *et al.*, 1987).

In the crystal structure, the intramolecular C—H···O and intermolecular O—H···O hydrogen bonds (Table 1, Fig. 2) link the molecules, in which they seem to be effective in the stabilization of the structure.

Experimental

The title compound, (I), was prepared by the literature method with a minor change (Lehn *et al.*, 1978). Crystals of (I) suitable for X-ray analysis were obtained by dissolving (I) (1.5 g) in acetic acid (20 ml) and evaporating the solvent slowly at room temperature for about 60 d.

Refinement

H atoms were positioned geometrically, with O—H = 0.82 Å (for OH) and C—H = 0.93 and 0.97 Å for aromatic and methylene H, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,*O*), where x = 1.5 for OH H, and x = 1.2 for all other H atoms.

Figures

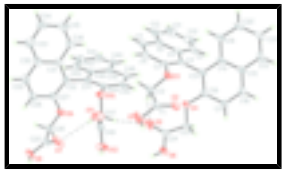


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

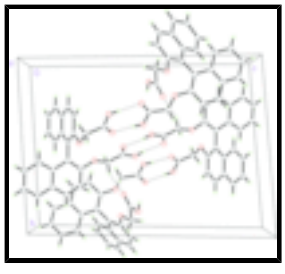


Fig. 2. A partial packing diagram of (I). Hydrogen bonds are shown as dashed lines.

(1,1'-Binaphthyl-2,2'-dioxy)diacetic acid

Crystal data

$C_{24}H_{18}O_6$	$Z = 4$
$M_r = 402.38$	$F_{000} = 840$
Triclinic, $P\overline{1}$	$D_x = 1.357$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 211 K
$a = 7.3160$ (15) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 14.067$ (3) Å	Cell parameters from 25 reflections
$c = 19.386$ (4) Å	$\theta = 9$–12º
$\alpha = 85.55$ (3)º	$\mu = 0.10$ mm^{-1}
$\beta = 83.11$ (3)º	$T = 298$ (2) K
$\gamma = 85.50$ (3)º	Block, white
$V = 1969.9$ (7) Å^3	0.40 × 0.20 × 0.10 mm

Data collection

Nonius CAD4 diffractometer	$R_{int} = 0.045$
Radiation source: fine-focus sealed tube	$\theta_{max} = 26.0$º
Monochromator: graphite	$\theta_{min} = 1.1$º
$T = 298(2)$ K	$h = -8 \rightarrow 9$
$\omega/2\theta$ scans	$k = -17 \rightarrow 17$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = 0 \rightarrow 23$
$T_{min} = 0.969$, $T_{max} = 0.988$	3 standard reflections
8349 measured reflections	every 120 min
7704 independent reflections	intensity decay: none
4677 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.062$	H-atom parameters constrained
$wR(F^2) = 0.185$	$w = 1/[\sigma^2(F_o^2) + (0.060P)^2 + 3.P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.97$	$(\Delta/\sigma)_{max} < 0.001$
7704 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
541 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 $F^2 > 2\text{sigma}(F^2)$ 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	0.1282 (5)	0.6169 (3)	0.49477 (15)	0.1021 (14)
H1A	0.0835	0.6059	0.5352	0.153*
O2	−0.0989 (3)	0.73119 (17)	0.49653 (12)	0.0496 (6)
O3	−0.0114 (3)	0.77416 (16)	0.35876 (11)	0.0439 (6)
O4	0.2512 (3)	0.92721 (18)	0.37989 (13)	0.0565 (7)
O5	0.5061 (4)	0.7891 (2)	0.4021 (2)	0.0967 (12)
O6	0.6600 (3)	0.88120 (19)	0.45775 (14)	0.0645 (8)
H6A	0.7205	0.8311	0.4666	0.097*
C1	0.0299 (5)	0.6857 (3)	0.46559 (19)	0.0489 (9)
C2	0.0985 (5)	0.7004 (3)	0.39056 (18)	0.0516 (9)
H2A	0.2258	0.7172	0.3856	0.062*
H2B	0.0942	0.6417	0.3679	0.062*
C3	0.0591 (4)	0.8019 (2)	0.29089 (17)	0.0383 (7)
C4	0.0597 (4)	0.8975 (2)	0.27175 (17)	0.0385 (7)
C5	0.1318 (4)	0.9274 (3)	0.20207 (17)	0.0446 (8)
C6	0.1987 (4)	0.8567 (3)	0.15491 (18)	0.0490 (9)
C7	0.1890 (5)	0.7598 (3)	0.1771 (2)	0.0567 (10)
H7A	0.2306	0.7135	0.1461	0.068*
C8	0.1191 (5)	0.7324 (3)	0.24370 (19)	0.0480 (9)
H8A	0.1115	0.6679	0.2576	0.058*
C9	0.1391 (5)	1.0245 (3)	0.1785 (2)	0.0558 (10)
H9A	0.0919	1.0719	0.2079	0.067*
C10	0.2173 (6)	1.0496 (4)	0.1109 (2)	0.0780 (14)
H10A	0.2263	1.1137	0.0963	0.094*
C11	0.2825 (6)	0.9791 (5)	0.0648 (2)	0.0844 (16)
H11A	0.3305	0.9968	0.0194	0.101*
C12	0.2758 (5)	0.8867 (4)	0.0860 (2)	0.0742 (14)
H12A	0.3223	0.8407	0.0553	0.089*
C13	−0.0051 (4)	0.9716 (2)	0.32173 (16)	0.0372 (7)
C14	−0.1729 (4)	1.0295 (2)	0.31468 (17)	0.0412 (8)
C15	−0.2220 (5)	1.1072 (2)	0.35663 (19)	0.0457 (8)
C16	−0.1044 (5)	1.1245 (3)	0.4057 (2)	0.0518 (9)
H16A	−0.1338	1.1762	0.4330	0.062*

C17	0.0530 (5)	1.0671 (3)	0.41447 (19)	0.0512 (9)
H17A	0.1288	1.0797	0.4474	0.061*
C18	0.0981 (5)	0.9890 (2)	0.37306 (18)	0.0442 (8)
C19	−0.2954 (4)	1.0097 (3)	0.26709 (19)	0.0482 (9)
H19A	−0.2672	0.9582	0.2395	0.058*
C20	−0.4542 (5)	1.0658 (3)	0.2616 (2)	0.0610 (11)
H20A	−0.5334	1.0514	0.2303	0.073*
C21	−0.5009 (5)	1.1439 (3)	0.3016 (2)	0.0613 (11)
H21A	−0.6089	1.1818	0.2964	0.074*
C22	−0.3881 (5)	1.1643 (3)	0.3482 (2)	0.0542 (9)
H22A	−0.4198	1.2164	0.3750	0.065*
C23	0.3781 (4)	0.9458 (2)	0.42561 (18)	0.0438 (8)
H23A	0.3154	0.9531	0.4721	0.053*
H23B	0.4373	1.0041	0.4097	0.053*
C24	0.5175 (5)	0.8630 (3)	0.42612 (19)	0.0497 (9)
O7	0.9951 (4)	0.4283 (2)	0.37652 (15)	0.0893 (11)
O8	1.2485 (4)	0.33147 (19)	0.37122 (14)	0.0630 (7)
H8B	1.2045	0.3133	0.4105	0.095*
O9	1.0627 (3)	0.48722 (16)	0.23955 (12)	0.0485 (6)
O10	0.5807 (3)	0.59017 (17)	0.28705 (11)	0.0474 (6)
O11	0.4509 (4)	0.5109 (2)	0.41261 (13)	0.0653 (8)
O12	0.6800 (4)	0.56388 (19)	0.46200 (13)	0.0593 (7)
H12B	0.6259	0.5375	0.4970	0.089*
C25	1.1379 (5)	0.3985 (3)	0.34557 (18)	0.0482 (9)
C26	1.2070 (5)	0.4393 (3)	0.27429 (18)	0.0513 (9)
H26A	1.2994	0.4838	0.2783	0.062*
H26B	1.2653	0.3881	0.2468	0.062*
C27	0.9372 (4)	0.4332 (2)	0.21556 (16)	0.0401 (7)
C28	0.9505 (5)	0.3324 (2)	0.22164 (18)	0.0497 (9)
H28A	1.0445	0.2999	0.2442	0.060*
C29	0.8276 (5)	0.2822 (3)	0.19487 (19)	0.0535 (9)
H29A	0.8391	0.2157	0.1996	0.064*
C30	0.6842 (5)	0.3280 (3)	0.16040 (18)	0.0486 (9)
C31	0.6660 (4)	0.4297 (2)	0.15515 (17)	0.0427 (8)
C32	0.7939 (4)	0.4826 (2)	0.18415 (16)	0.0379 (7)
C33	0.5600 (6)	0.2773 (3)	0.1296 (2)	0.0675 (11)
H33A	0.5688	0.2109	0.1345	0.081*
C34	0.4273 (6)	0.3232 (3)	0.0929 (2)	0.0729 (12)
H34A	0.3484	0.2885	0.0720	0.088*
C35	0.4119 (6)	0.4234 (3)	0.0871 (2)	0.0683 (12)
H35A	0.3226	0.4551	0.0617	0.082*
C36	0.5238 (5)	0.4749 (3)	0.11768 (19)	0.0551 (9)
H36A	0.5076	0.5413	0.1141	0.066*
C37	0.7715 (4)	0.5891 (2)	0.17765 (16)	0.0389 (7)
C38	0.8468 (4)	0.6408 (2)	0.11561 (16)	0.0417 (8)
C39	0.8064 (5)	0.7410 (2)	0.10647 (17)	0.0432 (8)
C40	0.8834 (5)	0.7916 (3)	0.04569 (19)	0.0552 (10)
H40A	0.8547	0.8570	0.0390	0.066*
C41	0.9990 (6)	0.7457 (3)	−0.0032 (2)	0.0660 (12)

H41A	1.0511	0.7801	−0.0426	0.079*
C42	1.0399 (6)	0.6477 (3)	0.0054 (2)	0.0644 (11)
H42A	1.1179	0.6166	−0.0285	0.077*
C43	0.9660 (5)	0.5965 (3)	0.06357 (18)	0.0534 (9)
H43A	0.9954	0.5310	0.0687	0.064*
C44	0.6597 (4)	0.6392 (2)	0.22741 (16)	0.0413 (8)
C45	0.6222 (5)	0.7385 (3)	0.21914 (18)	0.0469 (8)
H45A	0.5512	0.7706	0.2543	0.056*
C46	0.6894 (5)	0.7875 (2)	0.15982 (18)	0.0483 (9)
H46A	0.6588	0.8527	0.1537	0.058*
C47	0.6854 (4)	0.5974 (2)	0.34339 (16)	0.0418 (8)
H47A	0.7004	0.6643	0.3487	0.050*
H47B	0.8071	0.5658	0.3330	0.050*
C48	0.5953 (5)	0.5537 (2)	0.40964 (17)	0.0444 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.100 (2)	0.125 (3)	0.0570 (19)	0.072 (2)	0.0118 (17)	0.0372 (18)
O2	0.0481 (14)	0.0529 (15)	0.0468 (14)	0.0151 (11)	−0.0153 (11)	−0.0012 (11)
O3	0.0453 (13)	0.0425 (13)	0.0418 (13)	0.0081 (10)	−0.0111 (10)	0.0080 (10)
O4	0.0537 (15)	0.0574 (16)	0.0638 (17)	0.0224 (12)	−0.0366 (13)	−0.0196 (13)
O5	0.075 (2)	0.067 (2)	0.163 (4)	0.0281 (16)	−0.068 (2)	−0.048 (2)
O6	0.0557 (16)	0.0639 (17)	0.081 (2)	0.0169 (13)	−0.0441 (15)	−0.0138 (14)
C1	0.047 (2)	0.052 (2)	0.046 (2)	0.0129 (17)	−0.0149 (16)	0.0039 (16)
C2	0.049 (2)	0.055 (2)	0.048 (2)	0.0143 (17)	−0.0112 (17)	0.0089 (17)
C3	0.0336 (16)	0.0438 (19)	0.0392 (18)	0.0014 (14)	−0.0150 (14)	0.0000 (14)
C4	0.0326 (16)	0.0440 (19)	0.0410 (18)	−0.0020 (13)	−0.0188 (14)	0.0051 (14)
C5	0.0401 (18)	0.053 (2)	0.0420 (19)	−0.0010 (15)	−0.0206 (15)	0.0079 (16)
C6	0.0354 (18)	0.073 (3)	0.0380 (19)	0.0085 (17)	−0.0094 (15)	−0.0023 (17)
C7	0.052 (2)	0.074 (3)	0.046 (2)	0.0091 (19)	−0.0164 (18)	−0.0129 (19)
C8	0.048 (2)	0.0422 (19)	0.054 (2)	0.0096 (15)	−0.0149 (17)	−0.0074 (16)
C9	0.049 (2)	0.064 (2)	0.054 (2)	−0.0004 (18)	−0.0195 (18)	0.0143 (19)
C10	0.072 (3)	0.096 (4)	0.064 (3)	−0.012 (3)	−0.021 (2)	0.037 (3)
C11	0.068 (3)	0.128 (5)	0.050 (3)	−0.002 (3)	−0.007 (2)	0.030 (3)
C12	0.049 (2)	0.125 (4)	0.044 (2)	0.007 (2)	−0.0062 (18)	0.009 (3)
C13	0.0368 (17)	0.0347 (17)	0.0412 (18)	−0.0003 (13)	−0.0148 (14)	0.0047 (14)
C14	0.0429 (18)	0.0371 (18)	0.0454 (19)	−0.0006 (14)	−0.0208 (15)	0.0072 (14)
C15	0.0454 (19)	0.0401 (19)	0.052 (2)	−0.0010 (15)	−0.0152 (16)	0.0065 (16)
C16	0.048 (2)	0.045 (2)	0.063 (2)	0.0119 (16)	−0.0148 (18)	−0.0103 (17)
C17	0.051 (2)	0.051 (2)	0.057 (2)	0.0043 (17)	−0.0279 (18)	−0.0131 (18)
C18	0.0428 (19)	0.0413 (19)	0.051 (2)	0.0065 (15)	−0.0232 (16)	0.0002 (15)
C19	0.0359 (18)	0.055 (2)	0.055 (2)	−0.0006 (15)	−0.0178 (16)	0.0002 (17)
C20	0.051 (2)	0.069 (3)	0.066 (3)	0.0055 (19)	−0.030 (2)	0.005 (2)
C21	0.042 (2)	0.066 (3)	0.076 (3)	0.0097 (18)	−0.025 (2)	0.012 (2)
C22	0.046 (2)	0.046 (2)	0.070 (3)	0.0088 (16)	−0.0147 (19)	0.0042 (18)
C23	0.0411 (18)	0.051 (2)	0.0417 (19)	0.0079 (15)	−0.0215 (15)	−0.0062 (15)
C24	0.051 (2)	0.048 (2)	0.053 (2)	0.0108 (16)	−0.0249 (17)	−0.0068 (17)

O7	0.083 (2)	0.119 (3)	0.0491 (17)	0.056 (2)	0.0016 (15)	0.0225 (17)
O8	0.0590 (16)	0.0675 (18)	0.0552 (16)	0.0246 (13)	−0.0069 (13)	0.0137 (13)
O9	0.0542 (14)	0.0470 (14)	0.0458 (14)	−0.0003 (11)	−0.0205 (11)	0.0062 (11)
O10	0.0498 (14)	0.0608 (15)	0.0323 (12)	−0.0097 (11)	−0.0069 (10)	0.0017 (11)
O11	0.0611 (17)	0.093 (2)	0.0433 (15)	−0.0236 (15)	−0.0045 (13)	0.0043 (14)
O12	0.0638 (17)	0.0755 (19)	0.0393 (14)	−0.0134 (14)	−0.0126 (12)	0.0104 (12)
C25	0.049 (2)	0.049 (2)	0.046 (2)	0.0165 (16)	−0.0166 (17)	−0.0023 (16)
C26	0.048 (2)	0.058 (2)	0.048 (2)	0.0098 (17)	−0.0168 (17)	0.0008 (17)
C27	0.0417 (18)	0.0448 (19)	0.0324 (17)	−0.0032 (14)	−0.0053 (14)	0.0083 (14)
C28	0.054 (2)	0.045 (2)	0.049 (2)	0.0071 (17)	−0.0145 (17)	0.0050 (16)
C29	0.066 (2)	0.046 (2)	0.047 (2)	−0.0014 (18)	−0.0104 (18)	0.0088 (17)
C30	0.058 (2)	0.047 (2)	0.0397 (19)	−0.0085 (17)	−0.0036 (16)	0.0073 (16)
C31	0.0380 (18)	0.052 (2)	0.0372 (18)	−0.0017 (15)	−0.0071 (14)	0.0045 (15)
C32	0.0383 (17)	0.0440 (18)	0.0294 (16)	−0.0010 (14)	−0.0024 (13)	0.0054 (13)
C33	0.083 (3)	0.057 (3)	0.067 (3)	−0.018 (2)	−0.020 (2)	0.006 (2)
C34	0.072 (3)	0.074 (3)	0.080 (3)	−0.023 (2)	−0.032 (2)	0.000 (2)
C35	0.053 (2)	0.087 (3)	0.068 (3)	−0.010 (2)	−0.024 (2)	0.009 (2)
C36	0.056 (2)	0.059 (2)	0.050 (2)	0.0007 (18)	−0.0141 (18)	0.0067 (18)
C37	0.0402 (17)	0.0447 (19)	0.0316 (17)	0.0036 (14)	−0.0119 (14)	0.0048 (14)
C38	0.0422 (18)	0.051 (2)	0.0315 (17)	0.0014 (15)	−0.0113 (14)	0.0055 (14)
C39	0.050 (2)	0.0458 (19)	0.0344 (18)	0.0070 (15)	−0.0159 (15)	0.0025 (14)
C40	0.071 (3)	0.052 (2)	0.043 (2)	−0.0036 (19)	−0.0196 (19)	0.0168 (17)
C41	0.091 (3)	0.069 (3)	0.037 (2)	−0.022 (2)	−0.002 (2)	0.0161 (19)
C42	0.078 (3)	0.069 (3)	0.041 (2)	−0.007 (2)	0.009 (2)	0.0014 (19)
C43	0.066 (2)	0.053 (2)	0.0374 (19)	0.0066 (18)	0.0020 (17)	0.0022 (16)
C44	0.0367 (17)	0.056 (2)	0.0300 (17)	0.0035 (15)	−0.0064 (13)	0.0026 (15)
C45	0.047 (2)	0.051 (2)	0.042 (2)	0.0071 (16)	−0.0059 (16)	−0.0073 (16)
C46	0.055 (2)	0.0407 (19)	0.049 (2)	0.0085 (16)	−0.0173 (17)	0.0016 (16)
C47	0.0404 (18)	0.049 (2)	0.0351 (18)	0.0022 (15)	−0.0059 (14)	0.0029 (15)
C48	0.047 (2)	0.048 (2)	0.0375 (19)	0.0061 (16)	−0.0083 (15)	−0.0023 (15)

Geometric parameters (Å, °)

O1—C1	1.295 (4)	O7—C25	1.203 (4)
O1—H1A	0.8200	O8—C25	1.302 (4)
O2—C1	1.219 (4)	O8—H8B	0.8200
O3—C3	1.392 (4)	O9—C27	1.380 (4)
O3—C2	1.411 (4)	O9—C26	1.418 (4)
O4—C18	1.376 (4)	O10—C44	1.387 (4)
O4—C23	1.410 (4)	O10—C47	1.421 (4)
O5—C24	1.184 (4)	O11—C48	1.250 (4)
O6—C24	1.322 (4)	O12—C48	1.273 (4)
O6—H6A	0.8200	O12—H12B	0.8200
C1—C2	1.485 (5)	C25—C26	1.500 (5)
C2—H2A	0.9700	C26—H26A	0.9700
C2—H2B	0.9700	C26—H26B	0.9700
C3—C4	1.367 (4)	C27—C32	1.388 (4)
C3—C8	1.400 (5)	C27—C28	1.411 (5)
C4—C5	1.434 (5)	C28—C29	1.358 (5)

C4—C13	1.490 (4)	C28—H28A	0.9300
C5—C9	1.410 (5)	C29—C30	1.399 (5)
C5—C6	1.422 (5)	C29—H29A	0.9300
C6—C7	1.402 (5)	C30—C33	1.410 (5)
C6—C12	1.431 (5)	C30—C31	1.423 (5)
C7—C8	1.369 (5)	C31—C36	1.422 (5)
C7—H7A	0.9300	C31—C32	1.430 (5)
C8—H8A	0.9300	C32—C37	1.492 (4)
C9—C10	1.395 (6)	C33—C34	1.365 (6)
C9—H9A	0.9300	C33—H33A	0.9300
C10—C11	1.403 (7)	C34—C35	1.403 (6)
C10—H10A	0.9300	C34—H34A	0.9300
C11—C12	1.336 (7)	C35—C36	1.350 (5)
C11—H11A	0.9300	C35—H35A	0.9300
C12—H12A	0.9300	C36—H36A	0.9300
C13—C18	1.367 (4)	C37—C44	1.386 (4)
C13—C14	1.433 (4)	C37—C38	1.431 (4)
C14—C15	1.412 (5)	C38—C43	1.403 (5)
C14—C19	1.417 (4)	C38—C39	1.418 (5)
C15—C16	1.403 (5)	C39—C40	1.409 (5)
C15—C22	1.422 (5)	C39—C46	1.426 (5)
C16—C17	1.373 (5)	C40—C41	1.359 (6)
C16—H16A	0.9300	C40—H40A	0.9300
C17—C18	1.406 (5)	C41—C42	1.390 (6)
C17—H17A	0.9300	C41—H41A	0.9300
C19—C20	1.363 (5)	C42—C43	1.367 (5)
C19—H19A	0.9300	C42—H42A	0.9300
C20—C21	1.391 (6)	C43—H43A	0.9300
C20—H20A	0.9300	C44—C45	1.403 (5)
C21—C22	1.354 (5)	C45—C46	1.354 (5)
C21—H21A	0.9300	C45—H45A	0.9300
C22—H22A	0.9300	C46—H46A	0.9300
C23—C24	1.487 (4)	C47—C48	1.483 (5)
C23—H23A	0.9700	C47—H47A	0.9700
C23—H23B	0.9700	C47—H47B	0.9700
C1—O1—H1A	109.5	C25—O8—H8B	109.5
C3—O3—C2	113.5 (2)	C27—O9—C26	118.5 (3)
C18—O4—C23	119.9 (3)	C44—O10—C47	110.3 (2)
C24—O6—H6A	109.5	C48—O12—H12B	109.5
O2—C1—O1	123.8 (3)	O7—C25—O8	123.8 (3)
O2—C1—C2	126.1 (3)	O7—C25—C26	122.0 (3)
O1—C1—C2	110.1 (3)	O8—C25—C26	114.2 (3)
O3—C2—C1	109.8 (3)	O9—C26—C25	112.0 (3)
O3—C2—H2A	109.7	O9—C26—H26A	109.2
C1—C2—H2A	109.7	C25—C26—H26A	109.2
O3—C2—H2B	109.7	O9—C26—H26B	109.2
C1—C2—H2B	109.7	C25—C26—H26B	109.2
H2A—C2—H2B	108.2	H26A—C26—H26B	107.9
C4—C3—O3	118.1 (3)	O9—C27—C32	116.9 (3)

C4—C3—C8	122.0 (3)	O9—C27—C28	122.8 (3)
O3—C3—C8	119.8 (3)	C32—C27—C28	120.2 (3)
C3—C4—C5	118.9 (3)	C29—C28—C27	120.7 (3)
C3—C4—C13	122.1 (3)	C29—C28—H28A	119.6
C5—C4—C13	118.9 (3)	C27—C28—H28A	119.6
C9—C5—C6	118.6 (3)	C28—C29—C30	121.6 (3)
C9—C5—C4	122.4 (3)	C28—C29—H29A	119.2
C6—C5—C4	119.0 (3)	C30—C29—H29A	119.2
C7—C6—C5	119.3 (3)	C29—C30—C33	122.5 (4)
C7—C6—C12	121.8 (4)	C29—C30—C31	118.5 (3)
C5—C6—C12	118.9 (4)	C33—C30—C31	119.0 (3)
C8—C7—C6	121.0 (4)	C36—C31—C30	117.7 (3)
C8—C7—H7A	119.5	C36—C31—C32	122.3 (3)
C6—C7—H7A	119.5	C30—C31—C32	120.0 (3)
C7—C8—C3	119.7 (3)	C27—C32—C31	118.9 (3)
C7—C8—H8A	120.2	C27—C32—C37	122.3 (3)
C3—C8—H8A	120.2	C31—C32—C37	118.7 (3)
C10—C9—C5	119.9 (4)	C34—C33—C30	121.7 (4)
C10—C9—H9A	120.0	C34—C33—H33A	119.1
C5—C9—H9A	120.0	C30—C33—H33A	119.1
C9—C10—C11	120.8 (5)	C33—C34—C35	118.9 (4)
C9—C10—H10A	119.6	C33—C34—H34A	120.6
C11—C10—H10A	119.6	C35—C34—H34A	120.6
C12—C11—C10	120.1 (4)	C36—C35—C34	121.4 (4)
C12—C11—H11A	120.0	C36—C35—H35A	119.3
C10—C11—H11A	120.0	C34—C35—H35A	119.3
C11—C12—C6	121.5 (5)	C35—C36—C31	121.2 (4)
C11—C12—H12A	119.2	C35—C36—H36A	119.4
C6—C12—H12A	119.2	C31—C36—H36A	119.4
C18—C13—C14	118.6 (3)	C44—C37—C38	118.5 (3)
C18—C13—C4	120.8 (3)	C44—C37—C32	120.9 (3)
C14—C13—C4	120.5 (3)	C38—C37—C32	120.2 (3)
C15—C14—C19	118.2 (3)	C43—C38—C39	117.9 (3)
C15—C14—C13	120.2 (3)	C43—C38—C37	122.3 (3)
C19—C14—C13	121.6 (3)	C39—C38—C37	119.8 (3)
C16—C15—C14	118.2 (3)	C40—C39—C38	119.4 (3)
C16—C15—C22	122.6 (3)	C40—C39—C46	122.0 (3)
C14—C15—C22	119.2 (3)	C38—C39—C46	118.6 (3)
C17—C16—C15	121.9 (3)	C41—C40—C39	120.6 (4)
C17—C16—H16A	119.1	C41—C40—H40A	119.7
C15—C16—H16A	119.1	C39—C40—H40A	119.7
C16—C17—C18	119.2 (3)	C40—C41—C42	120.3 (3)
C16—C17—H17A	120.4	C40—C41—H41A	119.8
C18—C17—H17A	120.4	C42—C41—H41A	119.8
C13—C18—O4	115.7 (3)	C43—C42—C41	120.4 (4)
C13—C18—C17	121.8 (3)	C43—C42—H42A	119.8
O4—C18—C17	122.5 (3)	C41—C42—H42A	119.8
C20—C19—C14	120.3 (4)	C42—C43—C38	121.3 (4)
C20—C19—H19A	119.8	C42—C43—H43A	119.3

C14—C19—H19A	119.8	C38—C43—H43A	119.3
C19—C20—C21	121.6 (4)	C37—C44—O10	119.5 (3)
C19—C20—H20A	119.2	C37—C44—C45	121.9 (3)
C21—C20—H20A	119.2	O10—C44—C45	118.6 (3)
C22—C21—C20	119.6 (3)	C46—C45—C44	119.9 (3)
C22—C21—H21A	120.2	C46—C45—H45A	120.1
C20—C21—H21A	120.2	C44—C45—H45A	120.1
C21—C22—C15	121.0 (4)	C45—C46—C39	121.3 (3)
C21—C22—H22A	119.5	C45—C46—H46A	119.3
C15—C22—H22A	119.5	C39—C46—H46A	119.3
O4—C23—C24	107.1 (3)	O10—C47—C48	111.8 (3)
O4—C23—H23A	110.3	O10—C47—H47A	109.3
C24—C23—H23A	110.3	C48—C47—H47A	109.3
O4—C23—H23B	110.3	O10—C47—H47B	109.3
C24—C23—H23B	110.3	C48—C47—H47B	109.3
H23A—C23—H23B	108.5	H47A—C47—H47B	107.9
O5—C24—O6	122.8 (3)	O11—C48—O12	124.2 (3)
O5—C24—C23	125.8 (3)	O11—C48—C47	122.4 (3)
O6—C24—C23	111.4 (3)	O12—C48—C47	113.4 (3)
C3—O3—C2—C1	171.3 (3)	C27—O9—C26—C25	−72.3 (4)
O2—C1—C2—O3	−1.7 (6)	O7—C25—C26—O9	−21.7 (5)
O1—C1—C2—O3	178.8 (3)	O8—C25—C26—O9	160.7 (3)
C2—O3—C3—C4	−135.6 (3)	C26—O9—C27—C32	177.6 (3)
C2—O3—C3—C8	47.3 (4)	C26—O9—C27—C28	−2.6 (5)
O3—C3—C4—C5	−180.0 (3)	O9—C27—C28—C29	−177.5 (3)
C8—C3—C4—C5	−2.9 (5)	C32—C27—C28—C29	2.3 (5)
O3—C3—C4—C13	2.5 (4)	C27—C28—C29—C30	0.1 (6)
C8—C3—C4—C13	179.6 (3)	C28—C29—C30—C33	177.2 (4)
C3—C4—C5—C9	−179.3 (3)	C28—C29—C30—C31	−1.5 (5)
C13—C4—C5—C9	−1.7 (4)	C29—C30—C31—C36	177.7 (3)
C3—C4—C5—C6	0.5 (4)	C33—C30—C31—C36	−1.1 (5)
C13—C4—C5—C6	178.2 (3)	C29—C30—C31—C32	0.6 (5)
C9—C5—C6—C7	−178.7 (3)	C33—C30—C31—C32	−178.1 (3)
C4—C5—C6—C7	1.5 (5)	O9—C27—C32—C31	176.7 (3)
C9—C5—C6—C12	1.7 (5)	C28—C27—C32—C31	−3.1 (5)
C4—C5—C6—C12	−178.1 (3)	O9—C27—C32—C37	−1.5 (4)
C5—C6—C7—C8	−1.2 (5)	C28—C27—C32—C37	178.8 (3)
C12—C6—C7—C8	178.3 (3)	C36—C31—C32—C27	−175.3 (3)
C6—C7—C8—C3	−1.1 (5)	C30—C31—C32—C27	1.6 (5)
C4—C3—C8—C7	3.2 (5)	C36—C31—C32—C37	2.9 (5)
O3—C3—C8—C7	−179.8 (3)	C30—C31—C32—C37	179.8 (3)
C6—C5—C9—C10	−2.3 (5)	C29—C30—C33—C34	−176.3 (4)
C4—C5—C9—C10	177.5 (3)	C31—C30—C33—C34	2.4 (6)
C5—C9—C10—C11	2.6 (6)	C30—C33—C34—C35	−1.5 (7)
C9—C10—C11—C12	−2.2 (7)	C33—C34—C35—C36	−0.7 (7)
C10—C11—C12—C6	1.6 (7)	C34—C35—C36—C31	2.0 (6)
C7—C6—C12—C11	179.1 (4)	C30—C31—C36—C35	−1.1 (5)
C5—C6—C12—C11	−1.4 (6)	C32—C31—C36—C35	175.9 (4)
C3—C4—C13—C18	72.8 (4)	C27—C32—C37—C44	−93.5 (4)

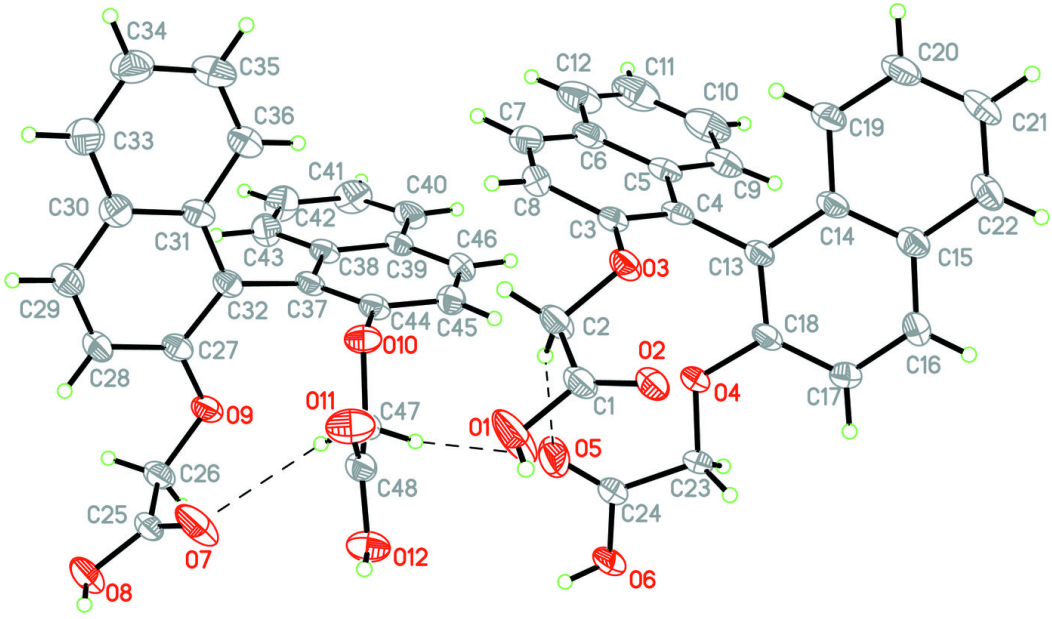
C5—C4—C13—C18	−104.8 (4)	C31—C32—C37—C44	88.3 (4)
C3—C4—C13—C14	−110.4 (3)	C27—C32—C37—C38	94.2 (4)
C5—C4—C13—C14	72.0 (4)	C31—C32—C37—C38	−83.9 (4)
C18—C13—C14—C15	4.7 (5)	C44—C37—C38—C43	177.9 (3)
C4—C13—C14—C15	−172.1 (3)	C32—C37—C38—C43	−9.7 (5)
C18—C13—C14—C19	−173.9 (3)	C44—C37—C38—C39	0.1 (5)
C4—C13—C14—C19	9.2 (5)	C32—C37—C38—C39	172.6 (3)
C19—C14—C15—C16	177.5 (3)	C43—C38—C39—C40	1.2 (5)
C13—C14—C15—C16	−1.2 (5)	C37—C38—C39—C40	179.1 (3)
C19—C14—C15—C22	−2.0 (5)	C43—C38—C39—C46	−178.3 (3)
C13—C14—C15—C22	179.3 (3)	C37—C38—C39—C46	−0.5 (5)
C14—C15—C16—C17	−1.3 (6)	C38—C39—C40—C41	−1.6 (5)
C22—C15—C16—C17	178.2 (4)	C46—C39—C40—C41	177.9 (4)
C15—C16—C17—C18	0.2 (6)	C39—C40—C41—C42	1.4 (6)
C14—C13—C18—O4	176.1 (3)	C40—C41—C42—C43	−0.8 (7)
C4—C13—C18—O4	−7.1 (5)	C41—C42—C43—C38	0.5 (6)
C14—C13—C18—C17	−5.9 (5)	C39—C38—C43—C42	−0.7 (5)
C4—C13—C18—C17	171.0 (3)	C37—C38—C43—C42	−178.5 (4)
C23—O4—C18—C13	172.5 (3)	C38—C37—C44—O10	178.1 (3)
C23—O4—C18—C17	−5.5 (5)	C32—C37—C44—O10	5.7 (5)
C16—C17—C18—C13	3.5 (6)	C38—C37—C44—C45	−1.3 (5)
C16—C17—C18—O4	−178.6 (3)	C32—C37—C44—C45	−173.7 (3)
C15—C14—C19—C20	1.1 (5)	C47—O10—C44—C37	98.8 (3)
C13—C14—C19—C20	179.8 (3)	C47—O10—C44—C45	−81.7 (4)
C14—C19—C20—C21	0.5 (6)	C37—C44—C45—C46	2.9 (5)
C19—C20—C21—C22	−1.2 (6)	O10—C44—C45—C46	−176.5 (3)
C20—C21—C22—C15	0.2 (6)	C44—C45—C46—C39	−3.2 (5)
C16—C15—C22—C21	−178.1 (4)	C40—C39—C46—C45	−177.5 (3)
C14—C15—C22—C21	1.3 (6)	C38—C39—C46—C45	2.1 (5)
C18—O4—C23—C24	175.2 (3)	C44—O10—C47—C48	174.6 (3)
O4—C23—C24—O5	−11.5 (6)	O10—C47—C48—O11	4.3 (5)
O4—C23—C24—O6	168.9 (3)	O10—C47—C48—O12	−176.2 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1A···O7^{i}	0.82	1.78	2.598 (4)	174
O6—H6A···O2^{ii}	0.82	1.96	2.760 (3)	167
O8—H8B···O2^{i}	0.82	1.95	2.770 (4)	179
O12—H12B···O11^{i}	0.82	1.87	2.671 (4)	166
C2—H2A···O5	0.97	2.42	3.360 (5)	162
C47—H47A···O5	0.97	2.39	3.139 (4)	134
C47—H47B···O7	0.97	2.44	3.236 (4)	139

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

Fig. 1


C34
C35
C36
C33
C30
C31
C29
C32
C28
C27
C41
C40
C42
C43
C38
C39
C46
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C44
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O10
O11
C47
O9
C26
C25
O7
O8
C48
O12
C12
C11
C10
C7
C6
C5
C9
C8
C4
C3
O3
C13
C2
O2
C1
O1
O5
O4
C18
C17
C16
C19
C20
C21
C14
C15
C22
C23
C24
O6

Fig. 2