

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

(R)-1,1'-Binaphthalene-2,2'-diyl dicinnamate

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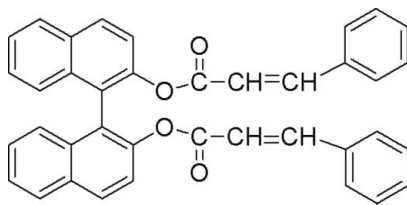
Received 26 February 2008; accepted 2 April 2008

 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.044; wR factor = 0.144; data-to-parameter ratio = 7.6.

In the title compound, $\text{C}_{38}\text{H}_{26}\text{O}_4$, two cinnamoyloxy groups are linked in a *trans* fashion to the two O atoms of optically active (*R*)-1,10-bi-2-naphthol. The dihedral angle between the mean planes of the two naphthyl groups is $71.8(1)^\circ$. The crystal structure contains intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For related literature, see: Chu *et al.* (2001); Goldberg (1980); Horikoshi *et al.* (2004); Lee & Lin (2002); Luo *et al.* (2002); Noyori (2002); Pu (1998).



Experimental

Crystal data

| | |
|--|-----------------------------------|
| $\text{C}_{38}\text{H}_{26}\text{O}_4$ | $V = 2803.1(8)$ Å ³ |
| $M_r = 546.59$ | $Z = 4$ |
| Orthorhombic, $P2_12_12_1$ | Mo $K\alpha$ radiation |
| $a = 10.3391(17)$ Å | $\mu = 0.08$ mm ⁻¹ |
| $b = 15.352(2)$ Å | $T = 293(2)$ K |
| $c = 17.660(3)$ Å | $0.52 \times 0.43 \times 0.38$ mm |

Data collection

| | |
|---|--|
| Siemens SMART CCD diffractometer | 27455 measured reflections |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | 2894 independent reflections |
| $T_{\min} = 0.936$, $T_{\max} = 0.969$ | 2753 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.037$ |

Refinement

| | |
|---------------------------------|---|
| $R[F^2 > 2\sigma(F^2)] = 0.043$ | 381 parameters |
| $wR(F^2) = 0.143$ | H-atom parameters constrained |
| $S = 1.02$ | $\Delta\rho_{\max} = 0.14$ e Å ⁻³ |
| 2894 reflections | $\Delta\rho_{\min} = -0.15$ e Å ⁻³ |

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{C}23-\text{H}23\text{A}\cdots\text{O}1$ | 0.93 | 2.38 | 2.736 (3) | 103 |
| $\text{C}32-\text{H}32\text{A}\cdots\text{O}4$ | 0.93 | 2.49 | 2.833 (4) | 102 |
| $\text{C}11-\text{C}11\text{A}\cdots\text{C}g1^{\text{i}}$ | 0.93 | 2.85 | 3.746 (3) | 162 |
| $\text{C}2-\text{H}2\text{A}\cdots\text{C}g2^{\text{ii}}$ | 0.93 | 2.74 | 3.507 (3) | 140 |

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

Data collection: SMART (Siemens, 1994); cell refinement: SAINT (Siemens, 1994); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXL97; software used to prepare material for publication: SHELXL97.

This work was supported by the National Natural Science Foundation of China (20705031) and the Project of Fujian Science & Technology Committee (2006 F5067).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2285).

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supplementary materials

Acta Cryst. (2008). E64, o819 [doi:10.1107/S1600536808008878]

(*R*)-1,1'-Binaphthalene-2,2'-diyl dicinnamate

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Comment

Optically active 1,10-bi-2-naphthol (BINOL) derivatives have been used successfully in asymmetric catalysis, molecular recognition and optical materials (Pu, 1998; Chu *et al.*, 2001; Luo *et al.*, 2002; Lee & Lin, 2002; Noyori, 2002). Their success is due to the fact that the axial chirality of the ligands can be well expressed in the steric environment of the active sites, and the chiral configuration of BINOL molecules is known to be stable at high temperature over extended periods of time. Thus, BINOL may be used as a preferred starting material or auxiliary for the synthesis of homochiral functional supramolecular complexes (Horikoshi *et al.*, 2004). Here we report the synthesis and crystal structure of the homochiral title compound.

The compound is composed of two cinnamoyloxy units linked in a *trans* fashion to the two O atoms (2,2'-) of the optically active (*R*)-BINOL (Fig. 1). The bond distances C6—O1 and C16—O3 are 1.399 (3) and 1.403 (4) Å, respectively. The separation between atoms O1 and O3 is 4.230 (3) Å, which is longer than that reported in other 2,2'-O-substituted complexes (Goldberg, 1980). Considerable twisting between the two naphthyl groups in the compound produces a dihedral angle 71.8 (1)°, much less than the angle of 101.7° found in (*R*)-BINOL itself. The naphthyl groups are also highly twisted with respect to their covalently linked phenyl groups, with dihedral angles of 28.6 (1) and 74.2 (1)°, respectively. These twists may be ascribed to steric repulsion, resulting in the two cinnamoyloxy units lying on opposite sides of the binaphthyl backbone.

The crystal structure contains C—H \cdots O and C—H \cdots π interactions (Fig. 2 and Table 1). Denoting the centroids of rings [C1—C4, C9, C10], [C11—C14, C19, C20], [C24—C29] and [C33—C38] as Cg1, Cg2, Cg3 and Cg4, respectively, the centroid-centroid distances are: Cg2 \cdots Cg1ⁱ = 4.749 (2) Å, Cg3 \cdots Cg4ⁱⁱ = 4.716 (2) Å [symmetry codes (i): 1/2 + x, 3/2 - y, -z; (ii) 1 + x, y, z].

Experimental

To a 50 ml round-bottom flask was added 2.0 g (7.0 mmol) of (*R*)-1,1'-bi-2-naphthol, 20 ml THF and 6.6 ml pyridine. Then, 5.0 ml *trans*-cinnamoyl chloride (25.9 mmol) was added in an ice bath. The mixture was stirred at ambient temperature for 24 h, and then poured onto ice. The resulting solid was filtered and washed with hot water. The crude product was soaked with absolute methanol twice to afford the target compound, which was recrystallized from THF/MeOH to afford colourless blocks.

Refinement

H atoms were positioned geometrically (C—H = 0.93 Å) and allowed to ride on their respective parent C atoms with $U_{iso}(H) = 1.2 U_{eq}(C)$. In the absence of significant anomalous scattering effects, 2231 Friedel pairs have been merged.

Figures

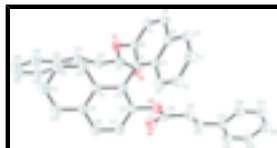


Fig. 1. The molecular structure with displacement ellipsoids drawn at the 50% probability level for non-H atoms.

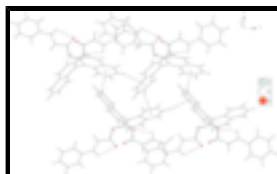


Fig. 2. View of the hydrogen-bond packing for compound (I), showing C—H...O and C—H... π contact between molecules as dashed lines.

(R)-1,1'-Binaphthalene-2,2'-diyl dicinnamate

Crystal data

| | |
|--------------------------------|---|
| $C_{38}H_{26}O_4$ | $F_{000} = 1144$ |
| $M_r = 546.59$ | $D_x = 1.295 \text{ Mg m}^{-3}$ |
| Orthorhombic, $P2_12_12_1$ | Mo $K\alpha$ radiation |
| Hall symbol: P 2ac 2ab | $\lambda = 0.71070 \text{ \AA}$ |
| $a = 10.3391 (17) \text{ \AA}$ | Cell parameters from 27455 reflections |
| $b = 15.352 (2) \text{ \AA}$ | $\theta = 3.0\text{--}25.4^\circ$ |
| $c = 17.660 (3) \text{ \AA}$ | $\mu = 0.08 \text{ mm}^{-1}$ |
| $V = 2803.1 (8) \text{ \AA}^3$ | $T = 293 (2) \text{ K}$ |
| $Z = 4$ | Block, colourless |
| | $0.52 \times 0.43 \times 0.38 \text{ mm}$ |

Data collection

| | |
|---|--|
| Siemens SMART CCD diffractometer | 2894 independent reflections |
| Radiation source: fine-focus sealed tube | 2753 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.037$ |
| $T = 293(2) \text{ K}$ | $\theta_{\text{max}} = 25.4^\circ$ |
| ω scans | $\theta_{\text{min}} = 3.0^\circ$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $h = -12 \rightarrow 12$ |
| $T_{\text{min}} = 0.936$, $T_{\text{max}} = 0.969$ | $k = -18 \rightarrow 18$ |
| 27455 measured reflections | $l = -18 \rightarrow 21$ |

Refinement

| | |
|---------------------------------|--|
| Refinement on F^2 | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.043$ | $w = 1/[\sigma^2(F_o^2) + (0.12P)^2]$ |
| | where $P = (F_o^2 + 2F_c^2)/3$ |

| | |
|--|--|
| $wR(F^2) = 0.144$ | $(\Delta/\sigma)_{\max} < 0.001$ |
| $S = 1.02$ | $\Delta\rho_{\max} = 0.14 \text{ e } \text{\AA}^{-3}$ |
| 2894 reflections | $\Delta\rho_{\min} = -0.15 \text{ e } \text{\AA}^{-3}$ |
| 381 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: 0.013 (3) |
| Secondary atom site location: difference Fourier map | |

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 | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|------------|--------------|---------------|----------------------------------|
| C1 | 0.6025 (3) | 0.8638 (3) | -0.04126 (18) | 0.0563 (8) |
| H1A | 0.5442 | 0.8445 | -0.0777 | 0.068* |
| C2 | 0.6764 (3) | 0.8040 (2) | -0.00129 (17) | 0.0535 (8) |
| H2A | 0.6671 | 0.7448 | -0.0114 | 0.064* |
| C3 | 0.7621 (3) | 0.8302 (2) | 0.05227 (17) | 0.0477 (7) |
| H3A | 0.8100 | 0.7889 | 0.0787 | 0.057* |
| C4 | 0.7794 (3) | 0.91986 (18) | 0.06838 (15) | 0.0380 (6) |
| C5 | 0.8758 (3) | 0.95009 (17) | 0.12052 (14) | 0.0373 (6) |
| C6 | 0.8883 (3) | 1.03778 (19) | 0.13057 (15) | 0.0394 (6) |
| C7 | 0.8090 (3) | 1.09864 (19) | 0.09384 (16) | 0.0450 (7) |
| H7A | 0.8187 | 1.1578 | 0.1037 | 0.054* |
| C8 | 0.7186 (3) | 1.0711 (2) | 0.04412 (17) | 0.0454 (7) |
| H8A | 0.6661 | 1.1116 | 0.0198 | 0.055* |
| C9 | 0.7027 (3) | 0.9807 (2) | 0.02847 (15) | 0.0421 (6) |
| C10 | 0.6151 (3) | 0.9511 (2) | -0.02713 (17) | 0.0511 (8) |
| H10A | 0.5658 | 0.9910 | -0.0543 | 0.061* |
| C11 | 1.3620 (3) | 0.8723 (3) | 0.1022 (2) | 0.0603 (9) |
| H11A | 1.4498 | 0.8707 | 0.0910 | 0.072* |
| C12 | 1.2803 (3) | 0.9291 (2) | 0.0633 (2) | 0.0586 (9) |
| H12A | 1.3144 | 0.9643 | 0.0254 | 0.070* |
| C13 | 1.1519 (3) | 0.9341 (2) | 0.07945 (18) | 0.0477 (7) |
| H13A | 1.0997 | 0.9725 | 0.0525 | 0.057* |
| C14 | 1.0972 (3) | 0.88157 (17) | 0.13694 (15) | 0.0386 (6) |
| C15 | 0.9642 (3) | 0.88757 (17) | 0.15923 (15) | 0.0371 (6) |

supplementary materials

| | | | | |
|------|--------------|--------------|--------------|-------------|
| C16 | 0.9218 (3) | 0.83490 (18) | 0.21667 (16) | 0.0399 (6) |
| C17 | 1.0014 (3) | 0.77282 (19) | 0.25182 (18) | 0.0469 (7) |
| H17A | 0.9679 | 0.7362 | 0.2889 | 0.056* |
| C18 | 1.1267 (3) | 0.7668 (2) | 0.23135 (17) | 0.0493 (7) |
| H18A | 1.1791 | 0.7255 | 0.2547 | 0.059* |
| C19 | 1.1804 (3) | 0.82197 (18) | 0.17505 (17) | 0.0431 (6) |
| C20 | 1.3128 (3) | 0.8194 (2) | 0.15641 (19) | 0.0537 (8) |
| H20A | 1.3672 | 0.7809 | 0.1817 | 0.064* |
| C21 | 0.9902 (3) | 1.05583 (19) | 0.24984 (17) | 0.0431 (7) |
| C22 | 1.1166 (3) | 1.0709 (2) | 0.28411 (18) | 0.0475 (7) |
| H22A | 1.1209 | 1.0753 | 0.3366 | 0.057* |
| C23 | 1.2250 (3) | 1.0785 (2) | 0.24563 (18) | 0.0498 (8) |
| H23A | 1.2183 | 1.0785 | 0.1931 | 0.060* |
| C24 | 1.3556 (3) | 1.0870 (2) | 0.27757 (19) | 0.0518 (8) |
| C25 | 1.4610 (3) | 1.0705 (3) | 0.2321 (2) | 0.0665 (10) |
| H25A | 1.4485 | 1.0593 | 0.1809 | 0.080* |
| C26 | 1.5852 (4) | 1.0704 (3) | 0.2613 (3) | 0.0771 (12) |
| H26A | 1.6550 | 1.0573 | 0.2301 | 0.093* |
| C27 | 1.6053 (4) | 1.0894 (3) | 0.3354 (3) | 0.0697 (10) |
| H27A | 1.6888 | 1.0894 | 0.3549 | 0.084* |
| C28 | 1.5032 (4) | 1.1086 (3) | 0.3813 (2) | 0.0729 (11) |
| H28A | 1.5174 | 1.1222 | 0.4319 | 0.087* |
| C29 | 1.3775 (3) | 1.1078 (3) | 0.3526 (2) | 0.0654 (10) |
| H29A | 1.3083 | 1.1214 | 0.3841 | 0.078* |
| C30 | 0.7596 (3) | 0.8456 (2) | 0.31233 (16) | 0.0476 (7) |
| C31 | 0.6190 (3) | 0.8482 (2) | 0.3223 (2) | 0.0542 (8) |
| H31A | 0.5650 | 0.8516 | 0.2803 | 0.065* |
| C32 | 0.5688 (3) | 0.8457 (2) | 0.3910 (2) | 0.0526 (8) |
| H32A | 0.6282 | 0.8421 | 0.4305 | 0.063* |
| C33 | 0.4326 (3) | 0.8477 (2) | 0.41384 (18) | 0.0499 (7) |
| C34 | 0.4012 (3) | 0.8251 (2) | 0.4876 (2) | 0.0591 (8) |
| H34A | 0.4665 | 0.8079 | 0.5205 | 0.071* |
| C35 | 0.2755 (4) | 0.8276 (3) | 0.5130 (2) | 0.0668 (10) |
| H35A | 0.2562 | 0.8129 | 0.5628 | 0.080* |
| C36 | 0.1783 (4) | 0.8520 (2) | 0.4639 (3) | 0.0681 (10) |
| H36A | 0.0929 | 0.8528 | 0.4804 | 0.082* |
| C37 | 0.2072 (4) | 0.8749 (3) | 0.3909 (2) | 0.0661 (10) |
| H37A | 0.1413 | 0.8917 | 0.3581 | 0.079* |
| C38 | 0.3337 (4) | 0.8733 (2) | 0.3654 (2) | 0.0616 (9) |
| H38A | 0.3526 | 0.8893 | 0.3159 | 0.074* |
| O1 | 0.99096 (19) | 1.07037 (12) | 0.17371 (12) | 0.0440 (5) |
| O2 | 0.8952 (2) | 1.03255 (17) | 0.28368 (13) | 0.0579 (6) |
| O3 | 0.79103 (19) | 0.84135 (14) | 0.23723 (11) | 0.0462 (5) |
| O4 | 0.8384 (2) | 0.8467 (2) | 0.36224 (13) | 0.0664 (7) |

Atomic displacement parameters (\AA^2)

U^{11}

U^{22}

U^{33}

U^{12}

U^{13}

U^{23}

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C1 | 0.0468 (16) | 0.081 (2) | 0.0407 (15) | -0.0084 (17) | -0.0064 (14) | -0.0052 (15) |
| C2 | 0.0578 (18) | 0.0568 (18) | 0.0458 (16) | -0.0116 (15) | -0.0009 (15) | -0.0114 (14) |
| C3 | 0.0511 (16) | 0.0498 (16) | 0.0424 (15) | -0.0003 (14) | -0.0014 (14) | -0.0005 (13) |
| C4 | 0.0367 (14) | 0.0461 (14) | 0.0313 (12) | -0.0033 (11) | 0.0013 (11) | 0.0017 (11) |
| C5 | 0.0392 (14) | 0.0432 (14) | 0.0294 (12) | -0.0002 (12) | 0.0014 (11) | 0.0024 (11) |
| C6 | 0.0373 (14) | 0.0422 (14) | 0.0388 (14) | 0.0004 (12) | -0.0023 (12) | 0.0007 (11) |
| C7 | 0.0480 (16) | 0.0396 (14) | 0.0474 (15) | 0.0043 (12) | 0.0003 (14) | 0.0030 (12) |
| C8 | 0.0405 (14) | 0.0503 (16) | 0.0455 (15) | 0.0072 (13) | -0.0025 (13) | 0.0047 (12) |
| C9 | 0.0362 (13) | 0.0574 (17) | 0.0326 (13) | -0.0003 (12) | 0.0034 (11) | 0.0021 (12) |
| C10 | 0.0425 (16) | 0.069 (2) | 0.0416 (16) | 0.0001 (15) | -0.0030 (13) | 0.0034 (14) |
| C11 | 0.0436 (17) | 0.074 (2) | 0.063 (2) | 0.0080 (16) | 0.0087 (16) | -0.0057 (18) |
| C12 | 0.0595 (19) | 0.0611 (19) | 0.0552 (18) | -0.0013 (16) | 0.0182 (16) | 0.0022 (16) |
| C13 | 0.0498 (17) | 0.0451 (15) | 0.0483 (16) | 0.0018 (13) | 0.0078 (14) | 0.0020 (13) |
| C14 | 0.0427 (14) | 0.0372 (13) | 0.0359 (13) | 0.0021 (11) | -0.0020 (12) | -0.0041 (11) |
| C15 | 0.0410 (14) | 0.0359 (12) | 0.0344 (13) | -0.0011 (11) | -0.0034 (11) | -0.0010 (11) |
| C16 | 0.0394 (14) | 0.0413 (14) | 0.0389 (14) | 0.0002 (11) | -0.0003 (12) | -0.0007 (12) |
| C17 | 0.0532 (17) | 0.0441 (15) | 0.0432 (15) | -0.0021 (14) | -0.0017 (13) | 0.0067 (12) |
| C18 | 0.0564 (18) | 0.0435 (15) | 0.0480 (16) | 0.0107 (14) | -0.0074 (15) | 0.0074 (13) |
| C19 | 0.0436 (15) | 0.0426 (14) | 0.0432 (14) | 0.0049 (12) | -0.0017 (13) | -0.0073 (12) |
| C20 | 0.0480 (16) | 0.0589 (17) | 0.0542 (18) | 0.0118 (15) | 0.0002 (15) | -0.0009 (15) |
| C21 | 0.0423 (15) | 0.0469 (15) | 0.0401 (15) | 0.0055 (13) | -0.0008 (12) | -0.0054 (12) |
| C22 | 0.0436 (16) | 0.0573 (18) | 0.0415 (15) | 0.0022 (13) | -0.0046 (13) | -0.0061 (14) |
| C23 | 0.0408 (16) | 0.0619 (19) | 0.0466 (16) | 0.0031 (14) | -0.0057 (13) | -0.0020 (14) |
| C24 | 0.0412 (16) | 0.0604 (18) | 0.0536 (18) | -0.0027 (14) | -0.0008 (14) | 0.0010 (15) |
| C25 | 0.0461 (18) | 0.092 (3) | 0.061 (2) | -0.0067 (18) | -0.0005 (16) | -0.007 (2) |
| C26 | 0.0386 (17) | 0.107 (3) | 0.086 (3) | -0.0026 (19) | 0.0075 (18) | -0.011 (3) |
| C27 | 0.0407 (17) | 0.085 (3) | 0.084 (3) | -0.0079 (17) | -0.0131 (18) | 0.003 (2) |
| C28 | 0.054 (2) | 0.100 (3) | 0.065 (2) | -0.007 (2) | -0.0151 (18) | -0.010 (2) |
| C29 | 0.0471 (17) | 0.087 (3) | 0.062 (2) | -0.0015 (18) | -0.0020 (16) | -0.0150 (19) |
| C30 | 0.0485 (16) | 0.0566 (17) | 0.0376 (14) | -0.0043 (14) | 0.0049 (14) | 0.0043 (13) |
| C31 | 0.0452 (16) | 0.069 (2) | 0.0489 (17) | -0.0060 (15) | 0.0009 (14) | 0.0040 (15) |
| C32 | 0.0481 (16) | 0.0616 (18) | 0.0481 (17) | 0.0003 (15) | 0.0016 (14) | 0.0032 (15) |
| C33 | 0.0492 (16) | 0.0509 (16) | 0.0495 (17) | -0.0027 (14) | 0.0012 (14) | -0.0018 (14) |
| C34 | 0.0522 (18) | 0.070 (2) | 0.0548 (19) | 0.0006 (17) | 0.0037 (16) | 0.0054 (16) |
| C35 | 0.060 (2) | 0.070 (2) | 0.070 (2) | -0.0032 (18) | 0.0194 (19) | 0.0031 (18) |
| C36 | 0.054 (2) | 0.059 (2) | 0.091 (3) | -0.0054 (17) | 0.018 (2) | -0.0091 (19) |
| C37 | 0.057 (2) | 0.065 (2) | 0.077 (2) | 0.0075 (18) | -0.0123 (19) | -0.012 (2) |
| C38 | 0.064 (2) | 0.065 (2) | 0.0564 (19) | 0.0058 (17) | -0.0020 (17) | -0.0009 (16) |
| O1 | 0.0415 (10) | 0.0474 (10) | 0.0432 (10) | -0.0053 (9) | -0.0061 (9) | -0.0015 (9) |
| O2 | 0.0426 (12) | 0.0834 (16) | 0.0478 (12) | -0.0039 (11) | 0.0047 (10) | -0.0116 (11) |
| O3 | 0.0438 (11) | 0.0553 (11) | 0.0395 (10) | -0.0036 (9) | -0.0010 (9) | 0.0037 (9) |
| O4 | 0.0557 (13) | 0.102 (2) | 0.0418 (12) | 0.0007 (13) | -0.0019 (11) | -0.0032 (12) |

Geometric parameters (Å, °)

| | | | |
|--------|-----------|---------|-----------|
| C1—C10 | 1.371 (5) | C21—O2 | 1.204 (4) |
| C1—C2 | 1.388 (5) | C21—O1 | 1.363 (4) |
| C1—H1A | 0.930 | C21—C22 | 1.459 (4) |
| C2—C3 | 1.357 (4) | C22—C23 | 1.316 (4) |

supplementary materials

| | | | |
|------------|-----------|--------------|-----------|
| C2—H2A | 0.930 | C22—H22A | 0.930 |
| C3—C4 | 1.417 (4) | C23—C24 | 1.469 (4) |
| C3—H3A | 0.930 | C23—H23A | 0.930 |
| C4—C9 | 1.414 (4) | C24—C25 | 1.376 (5) |
| C4—C5 | 1.434 (4) | C24—C29 | 1.382 (5) |
| C5—C6 | 1.364 (4) | C25—C26 | 1.384 (5) |
| C5—C15 | 1.491 (4) | C25—H25A | 0.930 |
| C6—O1 | 1.399 (3) | C26—C27 | 1.355 (6) |
| C6—C7 | 1.402 (4) | C26—H26A | 0.930 |
| C7—C8 | 1.350 (4) | C27—C28 | 1.363 (6) |
| C7—H7A | 0.930 | C27—H27A | 0.930 |
| C8—C9 | 1.425 (4) | C28—C29 | 1.395 (5) |
| C8—H8A | 0.930 | C28—H28A | 0.930 |
| C9—C10 | 1.411 (4) | C29—H29A | 0.930 |
| C10—H10A | 0.930 | C30—O4 | 1.201 (4) |
| C11—C20 | 1.354 (5) | C30—O3 | 1.367 (3) |
| C11—C12 | 1.396 (5) | C30—C31 | 1.465 (5) |
| C11—H11A | 0.930 | C31—C32 | 1.320 (5) |
| C12—C13 | 1.359 (5) | C31—H31A | 0.930 |
| C12—H12A | 0.930 | C32—C33 | 1.466 (5) |
| C13—C14 | 1.415 (4) | C32—H32A | 0.930 |
| C13—H13A | 0.930 | C33—C34 | 1.386 (5) |
| C14—C19 | 1.425 (4) | C33—C38 | 1.389 (5) |
| C14—C15 | 1.433 (4) | C34—C35 | 1.376 (5) |
| C15—C16 | 1.369 (4) | C34—H34A | 0.930 |
| C16—O3 | 1.403 (4) | C35—C36 | 1.379 (6) |
| C16—C17 | 1.404 (4) | C35—H35A | 0.930 |
| C17—C18 | 1.348 (5) | C36—C37 | 1.370 (6) |
| C17—H17A | 0.930 | C36—H36A | 0.930 |
| C18—C19 | 1.420 (4) | C37—C38 | 1.383 (5) |
| C18—H18A | 0.930 | C37—H37A | 0.930 |
| C19—C20 | 1.409 (5) | C38—H38A | 0.930 |
| C20—H20A | 0.930 | | |
| C10—C1—C2 | 120.2 (3) | C11—C20—H20A | 119.6 |
| C10—C1—H1A | 119.9 | C19—C20—H20A | 119.6 |
| C2—C1—H1A | 119.9 | O2—C21—O1 | 122.9 (3) |
| C3—C2—C1 | 121.2 (3) | O2—C21—C22 | 124.9 (3) |
| C3—C2—H2A | 119.4 | O1—C21—C22 | 112.2 (3) |
| C1—C2—H2A | 119.4 | C23—C22—C21 | 124.3 (3) |
| C2—C3—C4 | 120.7 (3) | C23—C22—H22A | 117.9 |
| C2—C3—H3A | 119.6 | C21—C22—H22A | 117.9 |
| C4—C3—H3A | 119.6 | C22—C23—C24 | 126.3 (3) |
| C9—C4—C3 | 118.1 (3) | C22—C23—H23A | 116.8 |
| C9—C4—C5 | 119.8 (2) | C24—C23—H23A | 116.8 |
| C3—C4—C5 | 122.1 (3) | C25—C24—C29 | 118.1 (3) |
| C6—C5—C4 | 117.9 (3) | C25—C24—C23 | 119.2 (3) |
| C6—C5—C15 | 121.2 (3) | C29—C24—C23 | 122.7 (3) |
| C4—C5—C15 | 120.8 (2) | C24—C25—C26 | 121.2 (4) |
| C5—C6—O1 | 119.7 (2) | C24—C25—H25A | 119.4 |

| | | | |
|--------------|------------|-----------------|------------|
| C5—C6—C7 | 122.9 (3) | C26—C25—H25A | 119.4 |
| O1—C6—C7 | 117.2 (3) | C27—C26—C25 | 120.1 (4) |
| C8—C7—C6 | 119.8 (3) | C27—C26—H26A | 120.0 |
| C8—C7—H7A | 120.1 | C25—C26—H26A | 120.0 |
| C6—C7—H7A | 120.1 | C26—C27—C28 | 120.1 (3) |
| C7—C8—C9 | 120.7 (3) | C26—C27—H27A | 119.9 |
| C7—C8—H8A | 119.6 | C28—C27—H27A | 119.9 |
| C9—C8—H8A | 119.6 | C27—C28—C29 | 120.2 (4) |
| C10—C9—C4 | 119.6 (3) | C27—C28—H28A | 119.9 |
| C10—C9—C8 | 121.5 (3) | C29—C28—H28A | 119.9 |
| C4—C9—C8 | 118.8 (3) | C24—C29—C28 | 120.2 (4) |
| C1—C10—C9 | 120.2 (3) | C24—C29—H29A | 119.9 |
| C1—C10—H10A | 119.9 | C28—C29—H29A | 119.9 |
| C9—C10—H10A | 119.9 | O4—C30—O3 | 123.5 (3) |
| C20—C11—C12 | 119.7 (3) | O4—C30—C31 | 125.8 (3) |
| C20—C11—H11A | 120.1 | O3—C30—C31 | 110.7 (3) |
| C12—C11—H11A | 120.1 | C32—C31—C30 | 120.0 (3) |
| C13—C12—C11 | 121.5 (3) | C32—C31—H31A | 120.0 |
| C13—C12—H12A | 119.2 | C30—C31—H31A | 120.0 |
| C11—C12—H12A | 119.2 | C31—C32—C33 | 129.0 (3) |
| C12—C13—C14 | 120.6 (3) | C31—C32—H32A | 115.5 |
| C12—C13—H13A | 119.7 | C33—C32—H32A | 115.5 |
| C14—C13—H13A | 119.7 | C34—C33—C38 | 118.5 (3) |
| C13—C14—C19 | 117.6 (3) | C34—C33—C32 | 118.5 (3) |
| C13—C14—C15 | 123.0 (3) | C38—C33—C32 | 123.0 (3) |
| C19—C14—C15 | 119.4 (3) | C35—C34—C33 | 121.3 (4) |
| C16—C15—C14 | 118.2 (3) | C35—C34—H34A | 119.3 |
| C16—C15—C5 | 121.6 (2) | C33—C34—H34A | 119.3 |
| C14—C15—C5 | 120.2 (2) | C34—C35—C36 | 119.5 (4) |
| C15—C16—O3 | 117.3 (2) | C34—C35—H35A | 120.3 |
| C15—C16—C17 | 122.7 (3) | C36—C35—H35A | 120.3 |
| O3—C16—C17 | 119.9 (3) | C37—C36—C35 | 120.1 (3) |
| C18—C17—C16 | 119.4 (3) | C37—C36—H36A | 119.9 |
| C18—C17—H17A | 120.3 | C35—C36—H36A | 119.9 |
| C16—C17—H17A | 120.3 | C36—C37—C38 | 120.5 (4) |
| C17—C18—C19 | 121.5 (3) | C36—C37—H37A | 119.7 |
| C17—C18—H18A | 119.2 | C38—C37—H37A | 119.7 |
| C19—C18—H18A | 119.2 | C37—C38—C33 | 120.0 (3) |
| C20—C19—C18 | 121.8 (3) | C37—C38—H38A | 120.0 |
| C20—C19—C14 | 119.6 (3) | C33—C38—H38A | 120.0 |
| C18—C19—C14 | 118.5 (3) | C21—O1—C6 | 118.3 (2) |
| C11—C20—C19 | 120.9 (3) | C30—O3—C16 | 118.9 (2) |
| C10—C1—C2—C3 | 0.0 (5) | C17—C18—C19—C20 | 176.3 (3) |
| C1—C2—C3—C4 | -0.6 (5) | C17—C18—C19—C14 | -3.4 (5) |
| C2—C3—C4—C9 | 1.6 (4) | C13—C14—C19—C20 | 2.1 (4) |
| C2—C3—C4—C5 | -175.3 (3) | C15—C14—C19—C20 | -176.3 (3) |
| C9—C4—C5—C6 | 1.1 (4) | C13—C14—C19—C18 | -178.2 (3) |
| C3—C4—C5—C6 | 177.9 (3) | C15—C14—C19—C18 | 3.4 (4) |
| C9—C4—C5—C15 | -175.7 (2) | C12—C11—C20—C19 | -1.1 (5) |

supplementary materials

| | | | |
|-----------------|------------|-----------------|------------|
| C3—C4—C5—C15 | 1.1 (4) | C18—C19—C20—C11 | 179.6 (3) |
| C4—C5—C6—O1 | -172.3 (2) | C14—C19—C20—C11 | -0.7 (5) |
| C15—C5—C6—O1 | 4.4 (4) | O2—C21—C22—C23 | 165.2 (3) |
| C4—C5—C6—C7 | 1.7 (4) | O1—C21—C22—C23 | -14.2 (4) |
| C15—C5—C6—C7 | 178.5 (3) | C21—C22—C23—C24 | -175.0 (3) |
| C5—C6—C7—C8 | -2.3 (4) | C22—C23—C24—C25 | 162.3 (4) |
| O1—C6—C7—C8 | 171.9 (3) | C22—C23—C24—C29 | -15.3 (6) |
| C6—C7—C8—C9 | 0.0 (4) | C29—C24—C25—C26 | 3.2 (6) |
| C3—C4—C9—C10 | -1.9 (4) | C23—C24—C25—C26 | -174.4 (4) |
| C5—C4—C9—C10 | 175.0 (2) | C24—C25—C26—C27 | -2.1 (7) |
| C3—C4—C9—C8 | 179.8 (3) | C25—C26—C27—C28 | 0.1 (7) |
| C5—C4—C9—C8 | -3.3 (4) | C26—C27—C28—C29 | 0.7 (7) |
| C7—C8—C9—C10 | -175.5 (3) | C25—C24—C29—C28 | -2.4 (6) |
| C7—C8—C9—C4 | 2.7 (4) | C23—C24—C29—C28 | 175.1 (4) |
| C2—C1—C10—C9 | -0.4 (5) | C27—C28—C29—C24 | 0.5 (7) |
| C4—C9—C10—C1 | 1.4 (4) | O4—C30—C31—C32 | -4.8 (6) |
| C8—C9—C10—C1 | 179.6 (3) | O3—C30—C31—C32 | 174.8 (3) |
| C20—C11—C12—C13 | 1.3 (6) | C30—C31—C32—C33 | 179.8 (3) |
| C11—C12—C13—C14 | 0.2 (5) | C31—C32—C33—C34 | 165.9 (4) |
| C12—C13—C14—C19 | -1.9 (4) | C31—C32—C33—C38 | -15.7 (6) |
| C12—C13—C14—C15 | 176.5 (3) | C38—C33—C34—C35 | 0.0 (6) |
| C13—C14—C15—C16 | -178.6 (3) | C32—C33—C34—C35 | 178.5 (3) |
| C19—C14—C15—C16 | -0.3 (4) | C33—C34—C35—C36 | 0.9 (6) |
| C13—C14—C15—C5 | 1.1 (4) | C34—C35—C36—C37 | -1.1 (6) |
| C19—C14—C15—C5 | 179.5 (2) | C35—C36—C37—C38 | 0.4 (6) |
| C6—C5—C15—C16 | 109.7 (3) | C36—C37—C38—C33 | 0.4 (6) |
| C4—C5—C15—C16 | -73.6 (4) | C34—C33—C38—C37 | -0.6 (5) |
| C6—C5—C15—C14 | -70.1 (4) | C32—C33—C38—C37 | -179.1 (3) |
| C4—C5—C15—C14 | 106.6 (3) | O2—C21—O1—C6 | -15.5 (4) |
| C14—C15—C16—O3 | -179.6 (2) | C22—C21—O1—C6 | 163.9 (2) |
| C5—C15—C16—O3 | 0.7 (4) | C5—C6—O1—C21 | -69.4 (3) |
| C14—C15—C16—C17 | -3.1 (4) | C7—C6—O1—C21 | 116.2 (3) |
| C5—C15—C16—C17 | 177.1 (3) | O4—C30—O3—C16 | 2.2 (5) |
| C15—C16—C17—C18 | 3.3 (5) | C31—C30—O3—C16 | -177.5 (2) |
| O3—C16—C17—C18 | 179.6 (3) | C15—C16—O3—C30 | -134.4 (3) |
| C16—C17—C18—C19 | 0.1 (5) | C17—C16—O3—C30 | 49.0 (4) |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|-------|-------------|-------------|---------------|
| C23—H23A \cdots O1 | 0.93 | 2.38 | 2.736 (3) | 103 |
| C32—H32A \cdots O4 | 0.93 | 2.49 | 2.833 (4) | 102 |
| C11—C11A \cdots Cg1 ⁱ | 0.93 | 2.85 | 3.746 (3) | 162 |
| C2—H2A \cdots Cg2 ⁱⁱ | 0.93 | 2.74 | 3.507 (3) | 140 |

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

Fig. 1

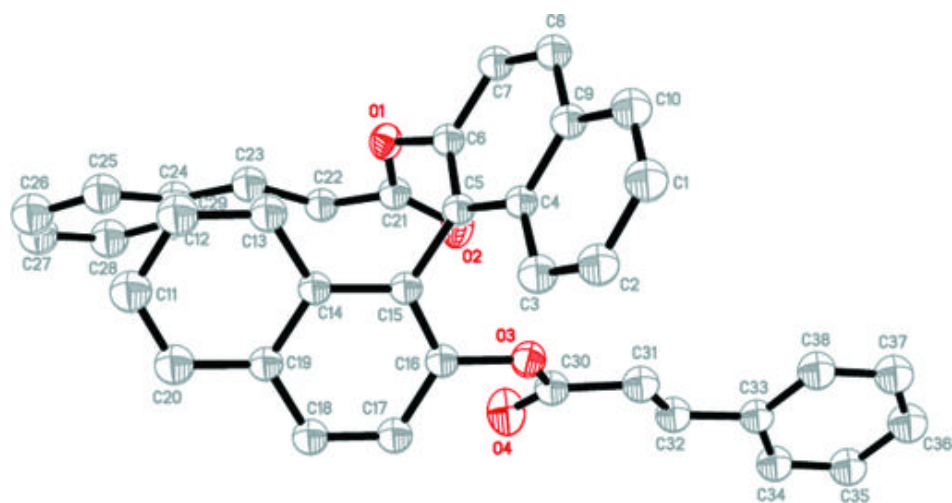


Fig. 2

