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# 2-Benzylidene-2,3-dihydro-1*H*-benzo[*f*]-chromen-1-one

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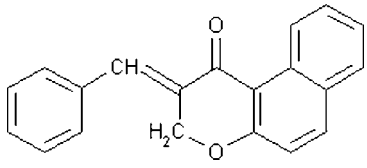
Key indicators: single-crystal X-ray study; $T$ = 150 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.032; $wR$ factor = 0.061; data-to-parameter ratio = 7.2.

The dihedral angles between the phenyl ring and the naphthalene part of the chromanone unit in the two independent molecules of the title compound, $C_{20}H_{14}O_2$, are 56.17 (4) and 55.62 (7)°. The molecular structure is stabilized by weak intramolecular C—H···O interactions and the crystal packing is stabilized by weak C—H···$\pi$ interactions.

## Related literature

For related literature see: Puviarasan *et al.* (1998); Tillekeratne *et al.* (2001); Nissa *et al.* (2001); Kang *et al.* (2004); Wu *et al.* (2005*a*,*b*); Schollmeyer *et al.* (2005).




## Experimental

### Crystal data

$C_{20}H_{14}O_2$
$M_r$ = 286.31
Orthorhombic, $Pca2_1$
$a$ = 12.3567 (6) Å
$b$ = 11.2675 (5) Å
$c$ = 20.4103 (12) Å
$V$ = 2841.7 (3) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 150 (2) K
0.32 × 0.23 × 0.18 mm

### Data collection

Stoe IPDS2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min}$ = 0.973, $T_{max}$ = 0.985
33648 measured reflections
2881 independent reflections
2120 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.105

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.061
$S$ = 0.94
2881 reflections
398 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.14 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C7—H7···O1 | 0.95 | 2.38 | 2.770 (4) | 104 |
| C12—H12···O1 | 0.95 | 2.22 | 2.856 (4) | 123 |
| C27—H27···O3 | 0.95 | 2.35 | 2.755 (4) | 105 |
| C32—H32···O3 | 0.95 | 2.25 | 2.857 (4) | 121 |
| C20—H20$A$···$Cg2^{i}$ | 0.99 | 2.90 | 3.735 (3) | 143 |
| C38—H38···$Cg1$ | 0.95 | 2.89 | 3.739 (3) | 149 |

Symmetry code: (i) $x-\frac{1}{2}, -y, z$. $Cg1$ and $Cg2$ are the centroids of the C1–C6 and C11–C16 rings, respectively.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2219).

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# supplementary materials

*Acta Cryst.* (2007). E**63**, o4383 [ **doi:10.1107/S1600536807050258** ]

# 2-Benzylidene-2,3-dihydro-1*H*-benzo[*f*]chromen-1-one

**A. Marx, V. Manivannan, R. Suresh, C. C. Kanagam and O. Büyükgüngör**

## Comment

The chromanone moiety present in the title compound consisting of the pyrone and benzene rings, which plays an important role in many areas of medicines such as inhibition of HIV replication (Tillekeratne *et al.*, 2001). The naturally occurring classes of compounds to which they belong, the benzylidene chroman-4-ones, have identified as a potential source of new anti-fungal agents (Kang *et al.*, 2004).

The geometric parameters in the title compound agree with the reported values of similar structure (Puviarasan *et al.*, 1998; Wu *et al.*, 2005*a*,b; Schollmeyer *et al.*, 2005; Nissa *et al.*, 2001). The dihedral angles between the phenyl ring and the benzene ring of chromanone unit in the two crystallographically independent molecules are 56.17 (4) and 55.62 (7)°.

The molecular structure is stabilized by weak intramolecular C—H···O interactions and the crystal packing is stabilized by weak C—H···π interactions.

## Experimental

Bromomethyl-3-phenyl-propenoate (10 mmol) was treated with beta-napthanol (10 mmol) in the presence of potassium carbonate in acetone at reflux temperature for 3 h. The napthoxy ester of methyl- 3-phenyl-2-(2-methoxy-4-prop-2-enyl) phenoxy methyl-prop-2-enoate was obtained after silica gel column chromatography (3% EtOAc-hexane). Hydrolysis of this ester was carried out with KOH in aqueous 1,4-dioxane at room temperature. The reaction mixture was acidified and the precipitated acid was purified by recrystalization. Finally the acid was treated with TFAA and the reaction mixture refluxed in dichloromethane for 1 h. It was further purified by column chromatography (silica gel − 3% EtOAc-hexane).

## Refinement

H atoms were positioned geometrically (C—H = 0.95 or 0.99 Å) and refined using riding model, with $U_{iso}$(H) = 1.2$U_{eq}$(C). In the absence of significant anomalous scattering effects, Friedel pairs have been merged.

## Figures

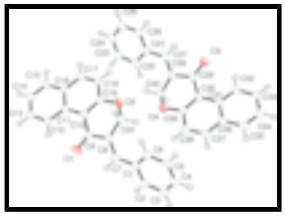


Fig. 1. The asymmetric unit of the title compound, with atom labels and 50% probability displacement ellipsoids for non-H atoms.

## 2-Benzylidene-2,3-dihydro-1*H*-benzo[*f*]chromen-1-one

### *Crystal data*

| | |
|---|---|
| $C_{20}H_{14}O_2$ | $F_{000}$ = 1200 |
| $M_r$ = 286.31 | $D_x$ = 1.338 Mg m$^{-3}$ |
| Orthorhombic, *Pca*2$_1$ | Mo *K*α radiation<br>λ = 0.71073 Å |
| Hall symbol: P 2c -2ac | Cell parameters from 30955 reflections |
| *a* = 12.3567 (6) Å | θ = 1.7–27.2º |
| *b* = 11.2675 (5) Å | μ = 0.09 mm$^{-1}$ |
| *c* = 20.4103 (12) Å | *T* = 150 (2) K |
| *V* = 2841.7 (3) Å$^3$ | Prism, yellow |
| *Z* = 8 | 0.32 × 0.23 × 0.18 mm |

### *Data collection*

| | |
|---|---|
| Stoe IPDS2<br>diffractometer | 2881 independent reflections |
| Radiation source: fine-focus sealed tube | 2120 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.105 |
| Detector resolution: 6.67 pixels mm$^{-1}$ | $\theta_{max}$ = 26.0º |
| *T* = 150(2) K | $\theta_{min}$ = 1.8º |
| rotation method scans | *h* = −15→15 |
| Absorption correction: integration<br>(*X-RED32*; Stoe & Cie, 2002) | *k* = −13→13 |
| $T_{min}$ = 0.973, $T_{max}$ = 0.985 | *l* = −25→25 |
| 33648 measured reflections | |

### *Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)]$ = 0.032 | $w = 1/[\sigma^2(F_o^2) + (0.0267P)^2]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2)$ = 0.061 | $(\Delta/\sigma)_{max}$ = 0.001 |
| *S* = 0.94 | $\Delta\rho_{max}$ = 0.12 e Å$^{-3}$ |
| 2881 reflections | $\Delta\rho_{min}$ = −0.14 e Å$^{-3}$ |
| 398 parameters | Extinction correction: none |
| 1 restraint | |
| Primary atom site location: structure-invariant direct methods | |
| 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 $F^2 > 2\text{sigma}(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.1622 (2) | −0.0440 (3) | 0.23909 (15) | 0.0308 (6) |
| C2 | 0.1523 (2) | −0.1118 (3) | 0.29624 (14) | 0.0377 (7) |
| H2 | 0.2018 | −0.1746 | 0.3042 | 0.045* |
| C3 | 0.0716 (2) | −0.0885 (3) | 0.34113 (16) | 0.0444 (8) |
| H3 | 0.0661 | −0.1352 | 0.3797 | 0.053* |
| C4 | −0.0014 (3) | 0.0026 (2) | 0.3303 (2) | 0.0384 (10) |
| H4 | −0.0560 | 0.0195 | 0.3617 | 0.046* |
| C5 | 0.0056 (2) | 0.0686 (3) | 0.27360 (17) | 0.0380 (7) |
| H5 | −0.0453 | 0.1301 | 0.2657 | 0.046* |
| C6 | 0.0866 (2) | 0.0461 (3) | 0.22768 (16) | 0.0334 (7) |
| H6 | 0.0905 | 0.0919 | 0.1887 | 0.040* |
| C7 | 0.2507 (2) | −0.0713 (2) | 0.19390 (15) | 0.0322 (6) |
| H7 | 0.2714 | −0.1524 | 0.1925 | 0.039* |
| C8 | 0.3068 (3) | 0.0006 (2) | 0.1543 (2) | 0.0292 (9) |
| C9 | 0.3951 (2) | −0.0516 (2) | 0.11341 (15) | 0.0317 (7) |
| C10 | 0.4700 (2) | 0.0332 (3) | 0.08277 (15) | 0.0307 (7) |
| C11 | 0.5574 (3) | −0.0012 (2) | 0.0393 (2) | 0.0283 (9) |
| C12 | 0.5704 (2) | −0.1172 (3) | 0.01372 (14) | 0.0341 (7) |
| H12 | 0.5198 | −0.1770 | 0.0255 | 0.041* |
| C13 | 0.6539 (2) | −0.1451 (3) | −0.02748 (15) | 0.0384 (7) |
| H13 | 0.6595 | −0.2233 | −0.0446 | 0.046* |
| C14 | 0.7310 (3) | −0.0600 (3) | −0.04466 (16) | 0.0406 (8) |
| H14 | 0.7897 | −0.0804 | −0.0726 | 0.049* |
| C15 | 0.7213 (3) | 0.0530 (3) | −0.02093 (17) | 0.0395 (9) |
| H15 | 0.7739 | 0.1107 | −0.0326 | 0.047* |
| C16 | 0.6352 (2) | 0.0857 (2) | 0.02037 (14) | 0.0325 (7) |
| C17 | 0.6268 (2) | 0.2029 (3) | 0.04455 (14) | 0.0372 (7) |
| H17 | 0.6802 | 0.2598 | 0.0330 | 0.045* |
| C18 | 0.5439 (2) | 0.2354 (2) | 0.08387 (15) | 0.0362 (7) |
| H18 | 0.5388 | 0.3149 | 0.0991 | 0.043* |
| C19 | 0.4647 (2) | 0.1511 (3) | 0.10228 (15) | 0.0319 (7) |
| C20 | 0.2890 (2) | 0.1306 (2) | 0.14782 (16) | 0.0354 (7) |
| H20A | 0.2442 | 0.1460 | 0.1085 | 0.043* |

| | | | | |
|---|---|---|---|---|
| H20B | 0.2484 | 0.1593 | 0.1865 | 0.043* |
| C21 | 0.5995 (2) | 0.5378 (3) | 0.18920 (15) | 0.0312 (7) |
| C22 | 0.6709 (2) | 0.4432 (3) | 0.19898 (16) | 0.0357 (7) |
| H22 | 0.6618 | 0.3924 | 0.2357 | 0.043* |
| C23 | 0.7547 (2) | 0.4230 (3) | 0.15543 (17) | 0.0390 (7) |
| H23 | 0.8018 | 0.3574 | 0.1619 | 0.047* |
| C24 | 0.7703 (3) | 0.4972 (2) | 0.1031 (2) | 0.0413 (10) |
| H24 | 0.8287 | 0.4834 | 0.0738 | 0.050* |
| C25 | 0.7012 (2) | 0.5924 (3) | 0.09264 (16) | 0.0408 (7) |
| H25 | 0.7112 | 0.6430 | 0.0559 | 0.049* |
| C26 | 0.6172 (2) | 0.6127 (2) | 0.13634 (14) | 0.0358 (7) |
| H26 | 0.5710 | 0.6792 | 0.1300 | 0.043* |
| C27 | 0.5093 (2) | 0.5654 (3) | 0.23332 (14) | 0.0323 (7) |
| H27 | 0.4904 | 0.6470 | 0.2354 | 0.039* |
| C28 | 0.4494 (3) | 0.4940 (2) | 0.2711 (2) | 0.0311 (10) |
| C29 | 0.3629 (2) | 0.5486 (3) | 0.31194 (14) | 0.0310 (7) |
| C30 | 0.2851 (2) | 0.4663 (3) | 0.34178 (15) | 0.0290 (6) |
| C31 | 0.2010 (3) | 0.5032 (2) | 0.3862 (2) | 0.0285 (9) |
| C32 | 0.1977 (2) | 0.6160 (3) | 0.41633 (14) | 0.0335 (7) |
| H32 | 0.2536 | 0.6717 | 0.4076 | 0.040* |
| C33 | 0.1151 (2) | 0.6464 (3) | 0.45793 (14) | 0.0378 (7) |
| H33 | 0.1158 | 0.7222 | 0.4783 | 0.045* |
| C34 | 0.0293 (3) | 0.5679 (3) | 0.47106 (17) | 0.0391 (8) |
| H34 | −0.0288 | 0.5914 | 0.4986 | 0.047* |
| C35 | 0.0308 (3) | 0.4586 (3) | 0.44398 (16) | 0.0363 (8) |
| H35 | −0.0261 | 0.4046 | 0.4535 | 0.044* |
| C36 | 0.1155 (2) | 0.4227 (3) | 0.40164 (14) | 0.0326 (7) |
| C37 | 0.1164 (2) | 0.3071 (2) | 0.37394 (14) | 0.0354 (7) |
| H37 | 0.0585 | 0.2542 | 0.3833 | 0.042* |
| C38 | 0.1981 (2) | 0.2705 (3) | 0.33438 (14) | 0.0353 (7) |
| H38 | 0.1981 | 0.1923 | 0.3170 | 0.042* |
| C39 | 0.2830 (2) | 0.3498 (3) | 0.31937 (15) | 0.0310 (7) |
| C40 | 0.4609 (2) | 0.3623 (2) | 0.27652 (16) | 0.0354 (7) |
| H40A | 0.5029 | 0.3326 | 0.2386 | 0.042* |
| H40B | 0.5020 | 0.3431 | 0.3168 | 0.042* |
| O1 | 0.40678 (16) | −0.16006 (19) | 0.10971 (12) | 0.0423 (6) |
| O2 | 0.38869 (16) | 0.19617 (18) | 0.14242 (10) | 0.0380 (5) |
| O3 | 0.35498 (16) | 0.65771 (18) | 0.31632 (12) | 0.0381 (5) |
| O4 | 0.35807 (14) | 0.30302 (19) | 0.27852 (10) | 0.0381 (5) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0286 (15) | 0.0270 (16) | 0.0369 (18) | −0.0049 (13) | −0.0027 (14) | −0.0051 (15) |
| C2 | 0.0379 (17) | 0.0339 (16) | 0.0413 (18) | 0.0000 (13) | −0.0016 (14) | 0.0032 (14) |
| C3 | 0.0447 (18) | 0.046 (2) | 0.0428 (19) | −0.0055 (15) | 0.0031 (15) | 0.0042 (16) |
| C4 | 0.0350 (18) | 0.039 (2) | 0.041 (2) | −0.0061 (13) | 0.0028 (17) | −0.0058 (13) |
| C5 | 0.0272 (15) | 0.0331 (16) | 0.054 (2) | −0.0025 (12) | −0.0041 (15) | −0.0053 (15) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C6 | 0.0317 (16) | 0.0319 (18) | 0.0367 (18) | −0.0046 (13) | −0.0026 (14) | 0.0014 (14) |
| C7 | 0.0293 (14) | 0.0276 (14) | 0.0398 (17) | −0.0014 (12) | −0.0023 (13) | −0.0023 (13) |
| C8 | 0.0285 (18) | 0.026 (2) | 0.033 (2) | 0.0008 (11) | −0.0053 (16) | −0.0023 (11) |
| C9 | 0.0310 (16) | 0.0253 (19) | 0.0390 (18) | −0.0029 (12) | −0.0047 (13) | −0.0002 (14) |
| C10 | 0.0331 (17) | 0.0302 (15) | 0.0289 (17) | −0.0031 (14) | −0.0063 (14) | −0.0002 (15) |
| C11 | 0.0317 (19) | 0.024 (2) | 0.029 (2) | −0.0013 (11) | −0.0034 (15) | 0.0002 (11) |
| C12 | 0.0392 (17) | 0.0302 (17) | 0.0329 (17) | −0.0038 (13) | −0.0001 (13) | −0.0030 (13) |
| C13 | 0.0442 (17) | 0.0337 (17) | 0.0372 (17) | −0.0009 (14) | −0.0015 (15) | −0.0044 (14) |
| C14 | 0.0405 (18) | 0.0408 (19) | 0.040 (2) | −0.0040 (15) | 0.0070 (15) | −0.0032 (16) |
| C15 | 0.0437 (19) | 0.034 (2) | 0.041 (2) | −0.0082 (15) | 0.0055 (16) | −0.0024 (16) |
| C16 | 0.0343 (16) | 0.0326 (17) | 0.0305 (16) | −0.0068 (13) | −0.0044 (13) | −0.0010 (12) |
| C17 | 0.0395 (17) | 0.0301 (16) | 0.0419 (17) | −0.0087 (13) | 0.0006 (14) | −0.0025 (13) |
| C18 | 0.0447 (17) | 0.0230 (15) | 0.0409 (17) | −0.0063 (13) | 0.0019 (15) | −0.0041 (13) |
| C19 | 0.0340 (15) | 0.0295 (15) | 0.0323 (17) | 0.0000 (13) | −0.0036 (14) | −0.0015 (13) |
| C20 | 0.0322 (16) | 0.0315 (17) | 0.0426 (18) | −0.0022 (12) | −0.0007 (14) | 0.0005 (13) |
| C21 | 0.0322 (17) | 0.0298 (16) | 0.0315 (17) | −0.0020 (14) | −0.0035 (14) | −0.0061 (15) |
| C22 | 0.0303 (16) | 0.0330 (18) | 0.0439 (19) | −0.0034 (13) | −0.0071 (14) | −0.0030 (14) |
| C23 | 0.0307 (15) | 0.0337 (16) | 0.053 (2) | 0.0004 (13) | −0.0015 (15) | −0.0083 (15) |
| C24 | 0.0343 (18) | 0.045 (2) | 0.045 (2) | −0.0030 (13) | 0.0066 (17) | −0.0139 (15) |
| C25 | 0.0446 (17) | 0.0393 (18) | 0.0384 (18) | −0.0059 (15) | 0.0011 (15) | −0.0025 (14) |
| C26 | 0.0333 (15) | 0.0339 (15) | 0.0401 (16) | 0.0003 (13) | −0.0018 (14) | −0.0004 (14) |
| C27 | 0.0335 (16) | 0.0285 (15) | 0.0349 (17) | 0.0014 (12) | −0.0082 (14) | −0.0036 (13) |
| C28 | 0.0295 (18) | 0.030 (2) | 0.033 (2) | 0.0011 (12) | −0.0031 (16) | −0.0040 (12) |
| C29 | 0.0320 (17) | 0.0292 (19) | 0.0317 (16) | 0.0006 (12) | −0.0043 (13) | −0.0036 (14) |
| C30 | 0.0288 (16) | 0.0269 (15) | 0.0311 (17) | 0.0020 (13) | −0.0020 (13) | −0.0004 (15) |
| C31 | 0.0304 (19) | 0.031 (2) | 0.024 (2) | 0.0022 (11) | −0.0061 (15) | 0.0011 (11) |
| C32 | 0.0327 (16) | 0.0326 (16) | 0.0353 (17) | 0.0001 (12) | −0.0014 (13) | −0.0003 (14) |
| C33 | 0.0413 (16) | 0.0329 (16) | 0.0392 (17) | 0.0021 (13) | 0.0041 (14) | −0.0030 (13) |
| C34 | 0.0342 (16) | 0.046 (2) | 0.037 (2) | 0.0023 (15) | 0.0035 (14) | 0.0011 (16) |
| C35 | 0.0310 (16) | 0.043 (2) | 0.035 (2) | −0.0028 (15) | 0.0002 (14) | 0.0057 (16) |
| C36 | 0.0360 (16) | 0.0308 (16) | 0.0312 (16) | −0.0001 (14) | −0.0057 (14) | 0.0029 (13) |
| C37 | 0.0376 (16) | 0.0309 (16) | 0.0377 (16) | −0.0068 (13) | −0.0062 (14) | 0.0053 (13) |
| C38 | 0.0378 (17) | 0.0299 (16) | 0.0382 (17) | −0.0036 (13) | −0.0030 (14) | 0.0015 (13) |
| C39 | 0.0357 (17) | 0.0293 (15) | 0.0278 (16) | 0.0021 (13) | −0.0011 (14) | −0.0025 (13) |
| C40 | 0.0335 (15) | 0.0323 (16) | 0.0403 (17) | −0.0003 (12) | 0.0010 (14) | −0.0028 (14) |
| O1 | 0.0428 (13) | 0.0256 (13) | 0.0584 (15) | −0.0023 (10) | 0.0090 (12) | −0.0049 (11) |
| O2 | 0.0378 (11) | 0.0292 (12) | 0.0469 (14) | −0.0042 (9) | 0.0059 (10) | −0.0066 (11) |
| O3 | 0.0386 (12) | 0.0240 (13) | 0.0519 (15) | 0.0005 (9) | 0.0078 (11) | −0.0032 (10) |
| O4 | 0.0379 (12) | 0.0319 (12) | 0.0446 (14) | −0.0030 (9) | 0.0061 (10) | −0.0061 (10) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C6 | 1.398 (5) | C21—C26 | 1.387 (4) |
| C1—C2 | 1.400 (4) | C21—C22 | 1.398 (4) |
| C1—C7 | 1.463 (4) | C21—C27 | 1.466 (4) |
| C2—C3 | 1.380 (4) | C22—C23 | 1.383 (4) |
| C2—H2 | 0.9500 | C22—H22 | 0.9500 |
| C3—C4 | 1.383 (4) | C23—C24 | 1.370 (5) |
| C3—H3 | 0.9500 | C23—H23 | 0.9500 |

| | | | |
|---|---|---|---|
| C4—C5 | 1.378 (5) | C24—C25 | 1.387 (4) |
| C4—H4 | 0.9500 | C24—H24 | 0.9500 |
| C5—C6 | 1.394 (4) | C25—C26 | 1.387 (4) |
| C5—H5 | 0.9500 | C25—H25 | 0.9500 |
| C6—H6 | 0.9500 | C26—H26 | 0.9500 |
| C7—C8 | 1.338 (4) | C27—C28 | 1.338 (4) |
| C7—H7 | 0.9500 | C27—H27 | 0.9500 |
| C8—C20 | 1.487 (3) | C28—C29 | 1.488 (5) |
| C8—C9 | 1.493 (5) | C28—C40 | 1.494 (4) |
| C9—O1 | 1.233 (3) | C29—O3 | 1.237 (3) |
| C9—C10 | 1.470 (4) | C29—C30 | 1.467 (4) |
| C10—C19 | 1.388 (4) | C30—C39 | 1.391 (4) |
| C10—C11 | 1.451 (5) | C30—C31 | 1.440 (5) |
| C11—C12 | 1.416 (4) | C31—C32 | 1.412 (4) |
| C11—C16 | 1.426 (4) | C31—C36 | 1.429 (5) |
| C12—C13 | 1.368 (4) | C32—C33 | 1.372 (4) |
| C12—H12 | 0.9500 | C32—H32 | 0.9500 |
| C13—C14 | 1.396 (4) | C33—C34 | 1.406 (4) |
| C13—H13 | 0.9500 | C33—H33 | 0.9500 |
| C14—C15 | 1.368 (5) | C34—C35 | 1.350 (5) |
| C14—H14 | 0.9500 | C34—H34 | 0.9500 |
| C15—C16 | 1.407 (4) | C35—C36 | 1.416 (4) |
| C15—H15 | 0.9500 | C35—H35 | 0.9500 |
| C16—C17 | 1.413 (4) | C36—C37 | 1.420 (4) |
| C17—C18 | 1.352 (4) | C37—C38 | 1.357 (4) |
| C17—H17 | 0.9500 | C37—H37 | 0.9500 |
| C18—C19 | 1.415 (4) | C38—C39 | 1.412 (4) |
| C18—H18 | 0.9500 | C38—H38 | 0.9500 |
| C19—O2 | 1.346 (3) | C39—O4 | 1.354 (3) |
| C20—O2 | 1.440 (3) | C40—O4 | 1.437 (3) |
| C20—H20A | 0.9900 | C40—H40A | 0.9900 |
| C20—H20B | 0.9900 | C40—H40B | 0.9900 |
| C6—C1—C2 | 118.4 (3) | C26—C21—C27 | 117.9 (3) |
| C6—C1—C7 | 123.1 (3) | C22—C21—C27 | 123.6 (3) |
| C2—C1—C7 | 118.4 (3) | C23—C22—C21 | 120.4 (3) |
| C3—C2—C1 | 120.8 (3) | C23—C22—H22 | 119.8 |
| C3—C2—H2 | 119.6 | C21—C22—H22 | 119.8 |
| C1—C2—H2 | 119.6 | C24—C23—C22 | 120.4 (3) |
| C2—C3—C4 | 120.4 (3) | C24—C23—H23 | 119.8 |
| C2—C3—H3 | 119.8 | C22—C23—H23 | 119.8 |
| C4—C3—H3 | 119.8 | C23—C24—C25 | 120.4 (4) |
| C5—C4—C3 | 119.6 (4) | C23—C24—H24 | 119.8 |
| C5—C4—H4 | 120.2 | C25—C24—H24 | 119.8 |
| C3—C4—H4 | 120.2 | C24—C25—C26 | 119.3 (3) |
| C4—C5—C6 | 120.7 (3) | C24—C25—H25 | 120.4 |
| C4—C5—H5 | 119.6 | C26—C25—H25 | 120.4 |
| C6—C5—H5 | 119.6 | C25—C26—C21 | 121.1 (3) |
| C5—C6—C1 | 120.0 (3) | C25—C26—H26 | 119.4 |
| C5—C6—H6 | 120.0 | C21—C26—H26 | 119.4 |

| | | | |
|---|---|---|---|
| C1—C6—H6 | 120.0 | C28—C27—C21 | 130.3 (3) |
| C8—C7—C1 | 129.9 (3) | C28—C27—H27 | 114.8 |
| C8—C7—H7 | 115.1 | C21—C27—H27 | 114.8 |
| C1—C7—H7 | 115.1 | C27—C28—C29 | 118.1 (3) |
| C7—C8—C20 | 125.0 (3) | C27—C28—C40 | 125.9 (3) |
| C7—C8—C9 | 118.6 (2) | C29—C28—C40 | 115.9 (3) |
| C20—C8—C9 | 116.5 (3) | O3—C29—C30 | 123.1 (3) |
| O1—C9—C10 | 123.0 (3) | O3—C29—C28 | 120.6 (3) |
| O1—C9—C8 | 120.7 (3) | C30—C29—C28 | 116.2 (3) |
| C10—C9—C8 | 116.2 (3) | C39—C30—C31 | 117.8 (3) |
| C19—C10—C11 | 117.8 (3) | C39—C30—C29 | 118.2 (3) |
| C19—C10—C9 | 118.1 (3) | C31—C30—C29 | 123.5 (3) |
| C11—C10—C9 | 123.7 (3) | C32—C31—C36 | 117.0 (3) |
| C12—C11—C16 | 117.3 (3) | C32—C31—C30 | 123.7 (3) |
| C12—C11—C10 | 123.8 (3) | C36—C31—C30 | 119.3 (3) |
| C16—C11—C10 | 118.9 (3) | C33—C32—C31 | 121.1 (3) |
| C13—C12—C11 | 121.6 (3) | C33—C32—H32 | 119.4 |
| C13—C12—H12 | 119.2 | C31—C32—H32 | 119.4 |
| C11—C12—H12 | 119.2 | C32—C33—C34 | 121.4 (3) |
| C12—C13—C14 | 120.7 (3) | C32—C33—H33 | 119.3 |
| C12—C13—H13 | 119.6 | C34—C33—H33 | 119.3 |
| C14—C13—H13 | 119.6 | C35—C34—C33 | 119.1 (3) |
| C15—C14—C13 | 119.4 (3) | C35—C34—H34 | 120.5 |
| C15—C14—H14 | 120.3 | C33—C34—H34 | 120.5 |
| C13—C14—H14 | 120.3 | C34—C35—C36 | 121.4 (3) |
| C14—C15—C16 | 121.5 (3) | C34—C35—H35 | 119.3 |
| C14—C15—H15 | 119.2 | C36—C35—H35 | 119.3 |
| C16—C15—H15 | 119.2 | C35—C36—C37 | 120.7 (3) |
| C15—C16—C17 | 120.7 (3) | C35—C36—C31 | 120.0 (3) |
| C15—C16—C11 | 119.4 (3) | C37—C36—C31 | 119.3 (3) |
| C17—C16—C11 | 119.9 (3) | C38—C37—C36 | 121.4 (3) |
| C18—C17—C16 | 121.1 (3) | C38—C37—H37 | 119.3 |
| C18—C17—H17 | 119.5 | C36—C37—H37 | 119.3 |
| C16—C17—H17 | 119.5 | C37—C38—C39 | 119.3 (3) |
| C17—C18—C19 | 120.0 (3) | C37—C38—H38 | 120.3 |
| C17—C18—H18 | 120.0 | C39—C38—H38 | 120.3 |
| C19—C18—H18 | 120.0 | O4—C39—C30 | 123.8 (3) |
| O2—C19—C10 | 124.6 (3) | O4—C39—C38 | 113.4 (2) |
| O2—C19—C18 | 113.0 (2) | C30—C39—C38 | 122.7 (3) |
| C10—C19—C18 | 122.3 (3) | O4—C40—C28 | 112.3 (2) |
| O2—C20—C8 | 112.7 (2) | O4—C40—H40A | 109.1 |
| O2—C20—H20A | 109.1 | C28—C40—H40A | 109.1 |
| C8—C20—H20A | 109.1 | O4—C40—H40B | 109.1 |
| O2—C20—H20B | 109.1 | C28—C40—H40B | 109.1 |
| C8—C20—H20B | 109.1 | H40A—C40—H40B | 107.9 |
| H20A—C20—H20B | 107.8 | C19—O2—C20 | 116.7 (2) |
| C26—C21—C22 | 118.4 (3) | C39—O4—C40 | 116.3 (2) |
| C6—C1—C2—C3 | −1.6 (4) | C22—C23—C24—C25 | −0.9 (5) |
| C7—C1—C2—C3 | 178.8 (3) | C23—C24—C25—C26 | 1.0 (5) |

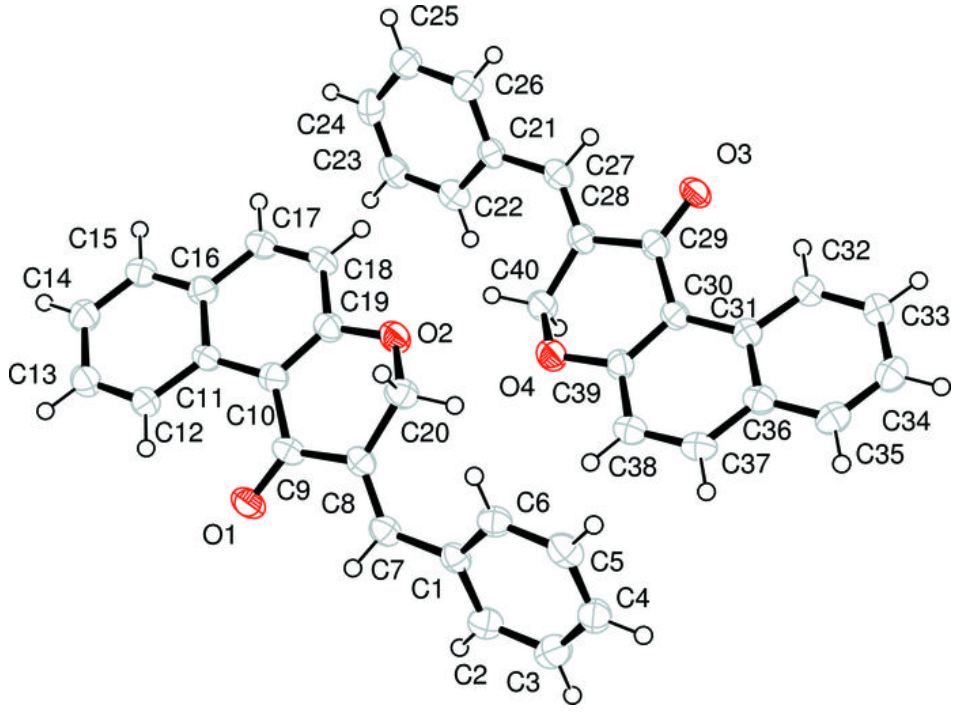
| | | | |
|---|---|---|---|
| C1—C2—C3—C4 | 0.1 (5) | C24—C25—C26—C21 | −1.8 (4) |
| C2—C3—C4—C5 | 1.3 (5) | C22—C21—C26—C25 | 2.3 (4) |
| C3—C4—C5—C6 | −1.2 (5) | C27—C21—C26—C25 | 179.9 (3) |
| C4—C5—C6—C1 | −0.3 (5) | C26—C21—C27—C28 | 152.6 (3) |
| C2—C1—C6—C5 | 1.7 (5) | C22—C21—C27—C28 | −30.0 (5) |
| C7—C1—C6—C5 | −178.8 (3) | C21—C27—C28—C29 | 179.2 (3) |
| C6—C1—C7—C8 | 32.2 (5) | C21—C27—C28—C40 | −0.7 (6) |
| C2—C1—C7—C8 | −148.3 (4) | C27—C28—C29—O3 | −7.9 (5) |
| C1—C7—C8—C20 | −0.2 (6) | C40—C28—C29—O3 | 172.0 (3) |
| C1—C7—C8—C9 | 179.2 (3) | C27—C28—C29—C30 | 167.8 (3) |
| C7—C8—C9—O1 | 9.3 (5) | C40—C28—C29—C30 | −12.3 (4) |
| C20—C8—C9—O1 | −171.2 (3) | O3—C29—C30—C39 | 162.2 (3) |
| C7—C8—C9—C10 | −166.3 (3) | C28—C29—C30—C39 | −13.4 (4) |
| C20—C8—C9—C10 | 13.1 (4) | O3—C29—C30—C31 | −8.8 (5) |
| O1—C9—C10—C19 | −165.1 (3) | C28—C29—C30—C31 | 175.6 (3) |
| C8—C9—C10—C19 | 10.4 (4) | C39—C30—C31—C32 | 175.3 (3) |
| O1—C9—C10—C11 | 7.2 (5) | C29—C30—C31—C32 | −13.7 (5) |
| C8—C9—C10—C11 | −177.2 (3) | C39—C30—C31—C36 | −4.4 (5) |
| C19—C10—C11—C12 | −177.7 (3) | C29—C30—C31—C36 | 166.6 (3) |
| C9—C10—C11—C12 | 10.0 (5) | C36—C31—C32—C33 | −0.7 (5) |
| C19—C10—C11—C16 | 1.9 (5) | C30—C31—C32—C33 | 179.6 (3) |
| C9—C10—C11—C16 | −170.4 (3) | C31—C32—C33—C34 | −1.5 (5) |
| C16—C11—C12—C13 | 0.0 (5) | C32—C33—C34—C35 | 2.5 (5) |
| C10—C11—C12—C13 | 179.6 (3) | C33—C34—C35—C36 | −1.4 (5) |
| C11—C12—C13—C14 | 1.4 (5) | C34—C35—C36—C37 | 179.5 (3) |
| C12—C13—C14—C15 | −1.3 (5) | C34—C35—C36—C31 | −0.7 (5) |
| C13—C14—C15—C16 | −0.1 (5) | C32—C31—C36—C35 | 1.7 (5) |
| C14—C15—C16—C17 | 179.8 (3) | C30—C31—C36—C35 | −178.5 (3) |
| C14—C15—C16—C11 | 1.5 (5) | C32—C31—C36—C37 | −178.5 (3) |
| C12—C11—C16—C15 | −1.4 (5) | C30—C31—C36—C37 | 1.3 (5) |
| C10—C11—C16—C15 | 179.0 (3) | C35—C36—C37—C38 | −178.6 (3) |
| C12—C11—C16—C17 | −179.7 (3) | C31—C36—C37—C38 | 1.6 (4) |
| C10—C11—C16—C17 | 0.7 (5) | C36—C37—C38—C39 | −1.3 (4) |
| C15—C16—C17—C18 | 179.5 (3) | C31—C30—C39—O4 | −178.7 (3) |
| C11—C16—C17—C18 | −2.2 (5) | C29—C30—C39—O4 | 9.8 (4) |
| C16—C17—C18—C19 | 1.0 (4) | C31—C30—C39—C38 | 5.0 (4) |
| C11—C10—C19—O2 | −179.9 (3) | C29—C30—C39—C38 | −166.5 (3) |
| C9—C10—C19—O2 | −7.1 (4) | C37—C38—C39—O4 | −178.8 (3) |
| C11—C10—C19—C18 | −3.2 (4) | C37—C38—C39—C30 | −2.1 (4) |
| C9—C10—C19—C18 | 169.6 (3) | C27—C28—C40—O4 | −138.8 (3) |
| C17—C18—C19—O2 | 178.8 (3) | C29—C28—C40—O4 | 41.4 (4) |
| C17—C18—C19—C10 | 1.8 (4) | C10—C19—O2—C20 | −21.3 (4) |
| C7—C8—C20—O2 | 139.9 (3) | C18—C19—O2—C20 | 161.7 (2) |
| C9—C8—C20—O2 | −39.5 (4) | C8—C20—O2—C19 | 43.8 (4) |
| C26—C21—C22—C23 | −2.2 (4) | C30—C39—O4—C40 | 21.4 (4) |
| C27—C21—C22—C23 | −179.6 (3) | C38—C39—O4—C40 | −161.9 (2) |
| C21—C22—C23—C24 | 1.5 (5) | C28—C40—O4—C39 | −46.2 (4) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C7—H7···O1 | 0.95 | 2.38 | 2.770 (4) | 104 |
| C12—H12···O1 | 0.95 | 2.22 | 2.856 (4) | 123 |
| C27—H27···O3 | 0.95 | 2.35 | 2.755 (4) | 105 |
| C32—H32···O3 | 0.95 | 2.25 | 2.857 (4) | 121 |
| C20—H20A···Cg2$^{i}$ | 0.99 | 2.90 | 3.735 (3) | 143 |
| C38—H38···Cg1 | 0.95 | 2.89 | 3.739 (3) | 149 |

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

**Fig. 1**


C25
C26
C24
C21
C23
C27
O3
C28
C22
C29
C17
C15
C16
C18
C32
C40
C14
C30
C19
C31
C33
O2
C13
O4
C39
C11
C10
C20
C34
C12
C36
C38
C37
C35
C9
C8
C6
O1
C5
C7
C1
C4
C2
C3