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8-[(*E*)-4-Methoxybenzylidene]-4-(4-methoxyphenyl)-3-(3-phenyl-1,2,4-oxadiazol-5-yl)-5,6,7,8-tetrahydro-4*H*-chromen-2-ylamine

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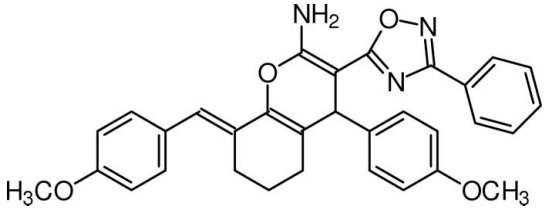
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.054; wR factor = 0.178; data-to-parameter ratio = 26.5.

The molecular conformation of the title compound, $C_{32}H_{29}N_3O_4$, is influenced by an intramolecular N—H···N hydrogen bond. Molecules are linked by N—H···O and C—H···O hydrogen bonds, generating sheets parallel to (001). In addition, a π–π stacking interaction (centroid–centroid distance = 4.038 Å) between the phenyl rings attached to the oxadiazole ring in adjacent molecules, and a C—H···π interaction involving the oxadiazole ring are also found to stabilize the crystal structure.

Related literature

For related literature, see: Yang *et al.* (2007); Cremer & Pople (1975).



Experimental

Crystal data

$C_{32}H_{29}N_3O_4$
M_r = 519.58
Triclinic, $P\overline{1}$
a = 10.5014 (3) Å
b = 11.3187 (4) Å
c = 11.6458 (4) Å
α = 91.07 (2)°
β = 98.45 (2)°
γ = 104.10 (3)°
V = 1325.8 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.30 × 0.20 × 0.15 mm

Data collection

Bruker Kappa APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.98, T_{max} = 0.99
36723 measured reflections
9546 independent reflections
6512 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.178
S = 1.03
9546 reflections
360 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

Cg is the centroid of the oxadiazole ring.

D—H···A	D—H	H···A	D···A	D—H···A
N21—H21*B*···N34	0.88 (2)	2.087 (19)	2.7650 (18)	133.7 (17)
C42—H42···O8^{i}	0.93	2.54	3.4594 (18)	170
N21—H21*A*···O4^{ii}	0.872 (19)	2.16 (2)	3.0291 (18)	171.8 (17)
C47—H47*A*···*Cg*^{iii}	0.96	2.59	3.489 (2)	156

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); 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: CF2131).

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  doi:10.1107/S1600536807038093

supplementary materials

Acta Cryst. (2007). E**63**, o3750 [**doi:10.1107/S1600536807038093**]

8-[(*E*)-4-Methoxybenzylidene]-4-(4-methoxyphenyl)-3-(3-phenyl-1,2,4-oxadiazol-5-yl)-5,6,7,8-tetrahydro-4*H*-chromen-2-ylamine

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Comment

The title compound, (I) (Fig. 1), belongs to an important class of organic molecules that have been found to display photoluminiscent properties (Yang *et al.*, 2007). Precise X-ray crystallographic investigations on such compounds are expected to provide insights for modelling and fabrication of organic light-emitting diodes.

An analysis of the puckering amplitudes (Cremer & Pople, 1975) indicates that the pyran ring adopts a boat conformation [Q = 0.164 (1) Å, θ = 103.8 (5)° and φ = 357.9 (5)°] and the six-membered ring fused to it adopts an envelope conformation [Q = 0.444 (2) Å, θ = 129.3 (2)° and φ = 56.0 (3)°], but deviations from planarity in this fused ring system are small.

The title molecule may be thought of as made up of four ring systems: the 5,6,7,8-tetrahydro-4*H*-chromen-2-ψlamine (A), 3-phenyl-1,2,4-oxadiazol-5-yl) (B), (4-methoxyphenyl)methylidene (C), and 4-(4-methoxyphenyl) (D). B, C and D make dihedral angles of 19.5 (1)°, 44.2 (1)° and 88.0 (1)°, respectively, with the mean plane of ring system A.

The conformation of the molecule is influenced by an intramolecular N—H···N hydrogen bond. Molecules are linked by N—H···O and C—H···O hydrogen bonds, generating sheets parallel to (001) (Fig.2). In addition, a π-π stacking interaction (centroid-centroid distance 4.038 Å) between phenyl rings attached to the oxadiazole ring in adjacent molecules, and a C—H···π interaction involving the oxadiazole ring are also found to stabilize the crystal structure.

Experimental

A mixture of 2-amino-4-(4-methoxyphenyl)-8-[(*E*)-(4-methoxyphenyl)methylidene]-5,6,7,8-tetrahydro-4*H*-chromene-3-carbonitrile (0.2 g, 5 mmol) and *N*-hydroxybenzenecarboximidoyl chloride (0.080 g, 5 mmol) was dissolved in benzene (15 ml). A solution of triethylamine (0.05 g, 5 mmol) in benzene (2 ml) was added dropwise to the above mixture, which was refluxed until the reaction was complete (about 5 h). Triethylamine hydrochloride was filtered off, the solvent removed *in vacuo* and the residue purified by column chromatography with silica gel, using petroleum ether:ethyl acetate (90:10 *v*/*v*) as eluant, to obtain the pure product as a pale-yellow solid (yield 52%). The compound was recrystallized from ethyl acetate to obtain suitable crystals for X-ray crystallographic study.

Refinement

H atoms bonded to N were located in a difference map and refined isotropically. Other H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95–0.99 Å and with U_{iso}(H) = 1.2 (1.5 for methyl groups) times U_{eq}(C).

Figures

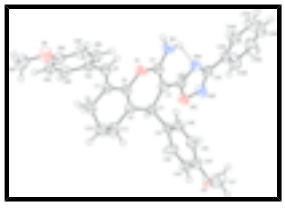

Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms. The intramolecular hydrogen bond is shown as a dashed line.

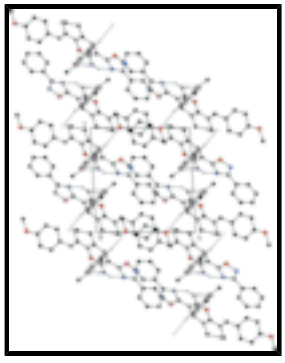

Fig. 2. The packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

8-[(*E*)-4-Methoxybenzylidene]-4-(4-Methoxyphenyl)-3- (3-phenyl-1,2,4-oxadiazol-5-yl)-5,6,7,8-tetrahydro-4*H*-chromen-2-amine

Crystal data

$C_{32}H_{29}N_3O_4$	$Z = 2$
$M_r = 519.58$	$F_{000} = 548$
Triclinic, $P\overline{1}$	$D_x = 1.301$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 10.5014$ (3) Å	Cell parameters from 4468 reflections
$b = 11.3187$ (4) Å	$\theta = 3$–29º
$c = 11.6458$ (4) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 91.07$ (2)º	$T = 298$ (2) K
$\beta = 98.45$ (2)º	Block, colourless
$\gamma = 104.10$ (3)º	0.30 × 0.20 × 0.15 mm
$V = 1325.8$ (2) Å^3	

Data collection

Bruker Kappa APEXII diffractometer	9546 independent reflections
Radiation source: fine-focus sealed tube	6512 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.025$
$T = 298(2)$ K	$\theta_{max} = 32.7$º
ω and φ scans	$\theta_{min} = 1.8$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$h = -15 \rightarrow 15$

T_{min} = 0.98, T_{max} = 0.99	k = −17→17
36723 measured reflections	l = −17→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.054	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.178	$w = 1/[\sigma^2(F_o^2) + (0.088P)^2 + 0.2805P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
9546 reflections	$\Delta\rho_{max}$ = 0.60 e Å^{-3}
360 parameters	$\Delta\rho_{min}$ = −0.32 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	0.44750 (9)	−0.06770 (9)	0.74873 (8)	0.0453 (2)
O31	0.77429 (10)	0.29001 (9)	0.69489 (8)	0.0445 (2)
O8	0.03663 (13)	−0.70140 (10)	1.00681 (11)	0.0603 (3)
O4	1.11417 (11)	−0.09930 (11)	0.60871 (11)	0.0586 (3)
N21	0.39565 (13)	0.05173 (14)	0.61084 (12)	0.0553 (4)
N32	0.78419 (13)	0.39013 (11)	0.62222 (11)	0.0476 (3)
N34	0.59051 (11)	0.25039 (10)	0.56538 (9)	0.0386 (2)
C2	0.49210 (13)	0.03321 (12)	0.69183 (11)	0.0389 (3)
C3	0.61886 (12)	0.10433 (11)	0.71726 (10)	0.0357 (2)
C4	0.72111 (12)	0.06893 (11)	0.80595 (10)	0.0336 (2)
H4	0.7668	0.1409	0.8575	0.040*
C5	0.65386 (12)	−0.02897 (11)	0.87854 (10)	0.0352 (2)
C6	0.52803 (12)	−0.09250 (11)	0.84756 (10)	0.0359 (2)
C7	0.45949 (13)	−0.19525 (12)	0.90757 (11)	0.0379 (3)
C8	0.33702 (13)	−0.26063 (12)	0.86451 (11)	0.0402 (3)

H8	0.2957	−0.2324	0.7982	0.048*
C81	0.25958 (13)	−0.37157 (12)	0.90901 (11)	0.0392 (3)
C82	0.25819 (14)	−0.39269 (13)	1.02612 (12)	0.0432 (3)
H82	0.3073	−0.3323	1.0816	0.052*
C83	0.18564 (14)	−0.50118 (13)	1.06275 (12)	0.0444 (3)
H83	0.1872	−0.5132	1.1417	0.053*
C84	0.11123 (14)	−0.59097 (12)	0.98113 (13)	0.0443 (3)
C85	0.10668 (17)	−0.57044 (14)	0.86384 (13)	0.0519 (3)
H85	0.0543	−0.6296	0.8086	0.062*
C86	0.17943 (15)	−0.46289 (13)	0.82911 (12)	0.0479 (3)
H86	0.1752	−0.4505	0.7501	0.058*
C87	0.0408 (2)	−0.72963 (17)	1.12512 (17)	0.0662 (5)
H87A	−0.0156	−0.8094	1.1304	0.099*
H87B	0.0104	−0.6704	1.1667	0.099*
H87C	0.1304	−0.7282	1.1585	0.099*
C33	0.67314 (13)	0.36061 (11)	0.54936 (11)	0.0380 (3)
C35	0.65582 (12)	0.21169 (11)	0.65603 (10)	0.0354 (2)
C331	0.64071 (14)	0.43903 (12)	0.45653 (11)	0.0415 (3)
C332	0.53368 (18)	0.39616 (16)	0.37008 (14)	0.0571 (4)
H332	0.4821	0.3166	0.3697	0.068*
C333	0.5029 (2)	0.4720 (2)	0.28346 (16)	0.0708 (5)
H333	0.4310	0.4428	0.2248	0.085*
C334	0.5776 (2)	0.58886 (19)	0.28403 (16)	0.0721 (6)
H334	0.5559	0.6396	0.2265	0.087*
C335	0.6838 (3)	0.63113 (17)	0.36871 (17)	0.0753 (6)
H335	0.7350	0.7107	0.3683	0.090*
C336	0.7166 (2)	0.55744 (14)	0.45519 (15)	0.0607 (4)
H336	0.7897	0.5872	0.5126	0.073*
C41	0.82502 (11)	0.02492 (10)	0.74932 (9)	0.0320 (2)
C42	0.96020 (12)	0.07784 (12)	0.78085 (12)	0.0401 (3)
H42	0.9883	0.1428	0.8363	0.048*
C43	1.05339 (13)	0.03552 (13)	0.73117 (13)	0.0444 (3)
H43	1.1433	0.0729	0.7525	0.053*
C44	1.01362 (13)	−0.06205 (12)	0.65001 (11)	0.0397 (3)
C45	0.87972 (13)	−0.11618 (13)	0.61681 (12)	0.0428 (3)
H45	0.8520	−0.1814	0.5616	0.051*
C46	0.78722 (12)	−0.07194 (12)	0.66695 (11)	0.0399 (3)
H46	0.6972	−0.1085	0.6445	0.048*
C47	1.0822 (2)	−0.2066 (2)	0.5367 (2)	0.0774 (6)
H47A	1.1619	−0.2209	0.5144	0.116*
H47B	1.0225	−0.1980	0.4684	0.116*
H47C	1.0404	−0.2742	0.5780	0.116*
C9	0.54074 (16)	−0.22266 (16)	1.01766 (13)	0.0523 (4)
H9A	0.5236	−0.1779	1.0831	0.063*
H9B	0.5124	−0.3090	1.0301	0.063*
C10	0.68687 (16)	−0.18972 (15)	1.01387 (15)	0.0548 (4)
H10A	0.7059	−0.2426	0.9557	0.066*
H10B	0.7340	−0.2031	1.0886	0.066*
C11	0.73658 (14)	−0.05845 (13)	0.98560 (11)	0.0449 (3)

H11A	0.7334	−0.0052	1.0508	0.054*
H11B	0.8284	−0.0439	0.9733	0.054*
H21A	0.3161 (19)	0.0034 (17)	0.6047 (16)	0.052 (5)*
H21B	0.4161 (19)	0.1154 (18)	0.5695 (17)	0.062 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0373 (5)	0.0480 (5)	0.0464 (5)	0.0050 (4)	0.0002 (4)	0.0211 (4)
O31	0.0475 (5)	0.0386 (5)	0.0433 (5)	0.0055 (4)	0.0015 (4)	0.0116 (4)
O8	0.0689 (7)	0.0408 (6)	0.0675 (7)	0.0025 (5)	0.0165 (6)	0.0148 (5)
O4	0.0440 (6)	0.0663 (7)	0.0713 (7)	0.0176 (5)	0.0213 (5)	−0.0014 (6)
N21	0.0359 (6)	0.0623 (8)	0.0633 (8)	0.0075 (6)	−0.0017 (5)	0.0321 (7)
N32	0.0554 (7)	0.0369 (6)	0.0473 (6)	0.0061 (5)	0.0052 (5)	0.0129 (5)
N34	0.0392 (5)	0.0367 (5)	0.0426 (5)	0.0127 (4)	0.0084 (4)	0.0121 (4)
C2	0.0370 (6)	0.0418 (6)	0.0407 (6)	0.0136 (5)	0.0074 (5)	0.0150 (5)
C3	0.0362 (6)	0.0361 (6)	0.0376 (5)	0.0128 (5)	0.0081 (4)	0.0105 (4)
C4	0.0340 (5)	0.0325 (5)	0.0345 (5)	0.0087 (4)	0.0043 (4)	0.0055 (4)
C5	0.0364 (6)	0.0373 (6)	0.0336 (5)	0.0114 (5)	0.0065 (4)	0.0086 (4)
C6	0.0357 (6)	0.0392 (6)	0.0348 (5)	0.0123 (5)	0.0060 (4)	0.0110 (4)
C7	0.0388 (6)	0.0394 (6)	0.0385 (5)	0.0122 (5)	0.0102 (5)	0.0129 (5)
C8	0.0410 (6)	0.0411 (6)	0.0400 (6)	0.0107 (5)	0.0086 (5)	0.0117 (5)
C81	0.0380 (6)	0.0387 (6)	0.0436 (6)	0.0112 (5)	0.0111 (5)	0.0089 (5)
C82	0.0454 (7)	0.0424 (7)	0.0417 (6)	0.0079 (5)	0.0118 (5)	0.0036 (5)
C83	0.0462 (7)	0.0469 (7)	0.0432 (6)	0.0121 (6)	0.0152 (5)	0.0107 (5)
C84	0.0442 (7)	0.0372 (6)	0.0549 (7)	0.0124 (5)	0.0140 (6)	0.0118 (5)
C85	0.0598 (9)	0.0421 (7)	0.0488 (7)	0.0047 (6)	0.0062 (6)	0.0024 (6)
C86	0.0530 (8)	0.0470 (8)	0.0417 (6)	0.0081 (6)	0.0073 (6)	0.0073 (5)
C87	0.0737 (11)	0.0546 (9)	0.0768 (11)	0.0158 (8)	0.0301 (9)	0.0286 (8)
C33	0.0454 (7)	0.0339 (6)	0.0386 (5)	0.0135 (5)	0.0121 (5)	0.0073 (4)
C35	0.0379 (6)	0.0349 (6)	0.0371 (5)	0.0128 (5)	0.0102 (4)	0.0068 (4)
C331	0.0538 (8)	0.0373 (6)	0.0392 (6)	0.0178 (6)	0.0138 (5)	0.0104 (5)
C332	0.0573 (9)	0.0574 (9)	0.0564 (8)	0.0152 (7)	0.0049 (7)	0.0186 (7)
C333	0.0736 (12)	0.0860 (14)	0.0563 (9)	0.0305 (10)	0.0006 (8)	0.0231 (9)
C334	0.1074 (16)	0.0712 (12)	0.0546 (9)	0.0462 (12)	0.0230 (10)	0.0308 (8)
C335	0.1195 (18)	0.0459 (9)	0.0628 (10)	0.0222 (10)	0.0159 (11)	0.0226 (8)
C336	0.0854 (12)	0.0396 (8)	0.0526 (8)	0.0092 (8)	0.0062 (8)	0.0120 (6)
C41	0.0304 (5)	0.0316 (5)	0.0330 (5)	0.0067 (4)	0.0032 (4)	0.0068 (4)
C42	0.0340 (6)	0.0356 (6)	0.0456 (6)	0.0016 (5)	0.0019 (5)	−0.0024 (5)
C43	0.0285 (6)	0.0460 (7)	0.0537 (7)	0.0013 (5)	0.0041 (5)	0.0018 (6)
C44	0.0355 (6)	0.0438 (7)	0.0421 (6)	0.0115 (5)	0.0100 (5)	0.0075 (5)
C45	0.0395 (6)	0.0431 (7)	0.0435 (6)	0.0079 (5)	0.0046 (5)	−0.0066 (5)
C46	0.0289 (5)	0.0435 (7)	0.0429 (6)	0.0035 (5)	0.0014 (4)	−0.0028 (5)
C47	0.0719 (12)	0.0823 (14)	0.0883 (14)	0.0314 (10)	0.0278 (11)	−0.0169 (11)
C9	0.0508 (8)	0.0572 (9)	0.0478 (7)	0.0111 (7)	0.0053 (6)	0.0230 (6)
C10	0.0484 (8)	0.0567 (9)	0.0569 (8)	0.0132 (7)	−0.0020 (6)	0.0207 (7)
C11	0.0419 (7)	0.0503 (8)	0.0391 (6)	0.0081 (6)	−0.0003 (5)	0.0131 (5)

Geometric parameters (Å, °)

O1—C2	1.3499 (15)	C87—H87A	0.960
O1—C6	1.4001 (14)	C87—H87B	0.960
O31—C35	1.3493 (16)	C87—H87C	0.960
O31—N32	1.4198 (14)	C33—C331	1.4655 (17)
O8—C84	1.3680 (17)	C331—C332	1.377 (2)
O8—C87	1.417 (2)	C331—C336	1.384 (2)
O4—C44	1.3721 (16)	C332—C333	1.389 (2)
O4—C47	1.404 (2)	C332—H332	0.930
N21—C2	1.3377 (17)	C333—C334	1.363 (3)
N21—H21A	0.872 (19)	C333—H333	0.930
N21—H21B	0.88 (2)	C334—C335	1.358 (3)
N32—C33	1.3038 (18)	C334—H334	0.930
N34—C35	1.3144 (16)	C335—C336	1.378 (2)
N34—C33	1.3686 (17)	C335—H335	0.930
C2—C3	1.3641 (18)	C336—H336	0.930
C3—C35	1.4213 (17)	C41—C46	1.3843 (17)
C3—C4	1.5097 (16)	C41—C42	1.3903 (16)
C4—C5	1.5070 (16)	C42—C43	1.3807 (19)
C4—C41	1.5290 (16)	C42—H42	0.930
C4—H4	0.980	C43—C44	1.3812 (19)
C5—C6	1.3344 (17)	C43—H43	0.930
C5—C11	1.5029 (17)	C44—C45	1.3834 (18)
C6—C7	1.4593 (16)	C45—C46	1.3885 (18)
C7—C8	1.3364 (19)	C45—H45	0.930
C7—C9	1.5134 (18)	C46—H46	0.930
C8—C81	1.4688 (18)	C47—H47A	0.960
C8—H8	0.930	C47—H47B	0.960
C81—C82	1.3907 (18)	C47—H47C	0.960
C81—C86	1.3972 (19)	C9—C10	1.496 (2)
C82—C83	1.3892 (19)	C9—H9A	0.970
C82—H82	0.930	C9—H9B	0.970
C83—C84	1.380 (2)	C10—C11	1.510 (2)
C83—H83	0.930	C10—H10A	0.970
C84—C85	1.386 (2)	C10—H10B	0.970
C85—C86	1.373 (2)	C11—H11A	0.970
C85—H85	0.930	C11—H11B	0.970
C86—H86	0.930		
C2—O1—C6	118.98 (10)	O31—C35—C3	117.70 (11)
C35—O31—N32	106.29 (10)	C332—C331—C336	119.16 (14)
C84—O8—C87	118.35 (14)	C332—C331—C33	120.40 (13)
C44—O4—C47	118.78 (13)	C336—C331—C33	120.43 (14)
C2—N21—H21A	119.3 (12)	C331—C332—C333	119.83 (17)
C2—N21—H21B	116.8 (13)	C331—C332—H332	120.1
H21A—N21—H21B	123.8 (18)	C333—C332—H332	120.1
C33—N32—O31	103.40 (10)	C334—C333—C332	120.35 (19)
C35—N34—C33	103.05 (11)	C334—C333—H333	119.8

N21—C2—O1	110.65 (12)	C332—C333—H333	119.8
N21—C2—C3	126.55 (12)	C335—C334—C333	119.95 (16)
O1—C2—C3	122.79 (11)	C335—C334—H334	120.0
C2—C3—C35	119.00 (11)	C333—C334—H334	120.0
C2—C3—C4	121.30 (10)	C334—C335—C336	120.72 (18)
C35—C3—C4	119.66 (11)	C334—C335—H335	119.6
C5—C4—C3	110.09 (10)	C336—C335—H335	119.6
C5—C4—C41	109.71 (9)	C335—C336—C331	119.98 (18)
C3—C4—C41	112.14 (9)	C335—C336—H336	120.0
C5—C4—H4	108.3	C331—C336—H336	120.0
C3—C4—H4	108.3	C46—C41—C42	117.73 (11)
C41—C4—H4	108.3	C46—C41—C4	120.73 (10)
C6—C5—C11	120.25 (11)	C42—C41—C4	121.51 (10)
C6—C5—C4	122.33 (10)	C43—C42—C41	121.06 (12)
C11—C5—C4	117.34 (11)	C43—C42—H42	119.5
C5—C6—O1	122.04 (11)	C41—C42—H42	119.5
C5—C6—C7	125.61 (11)	C42—C43—C44	120.28 (12)
O1—C6—C7	112.33 (10)	C42—C43—H43	119.9
C8—C7—C6	121.52 (11)	C44—C43—H43	119.9
C8—C7—C9	124.10 (12)	O4—C44—C43	115.64 (12)
C6—C7—C9	114.34 (11)	O4—C44—C45	124.51 (12)
C7—C8—C81	128.01 (12)	C43—C44—C45	119.84 (12)
C7—C8—H8	116.0	C44—C45—C46	119.15 (12)
C81—C8—H8	116.0	C44—C45—H45	120.4
C82—C81—C86	116.71 (12)	C46—C45—H45	120.4
C82—C81—C8	124.79 (12)	C41—C46—C45	121.92 (11)
C86—C81—C8	118.49 (12)	C41—C46—H46	119.0
C83—C82—C81	122.04 (13)	C45—C46—H46	119.0
C83—C82—H82	119.0	O4—C47—H47A	109.5
C81—C82—H82	119.0	O4—C47—H47B	109.5
C84—C83—C82	119.50 (13)	H47A—C47—H47B	109.5
C84—C83—H83	120.2	O4—C47—H47C	109.5
C82—C83—H83	120.2	H47A—C47—H47C	109.5
O8—C84—C83	124.69 (13)	H47B—C47—H47C	109.5
O8—C84—C85	115.64 (14)	C10—C9—C7	112.95 (12)
C83—C84—C85	119.65 (13)	C10—C9—H9A	109.0
C86—C85—C84	120.07 (14)	C7—C9—H9A	109.0
C86—C85—H85	120.0	C10—C9—H9B	109.0
C84—C85—H85	120.0	C7—C9—H9B	109.0
C85—C86—C81	121.94 (13)	H9A—C9—H9B	107.8
C85—C86—H86	119.0	C9—C10—C11	112.05 (13)
C81—C86—H86	119.0	C9—C10—H10A	109.2
O8—C87—H87A	109.5	C11—C10—H10A	109.2
O8—C87—H87B	109.5	C9—C10—H10B	109.2
H87A—C87—H87B	109.5	C11—C10—H10B	109.2
O8—C87—H87C	109.5	H10A—C10—H10B	107.9
H87A—C87—H87C	109.5	C5—C11—C10	111.05 (11)
H87B—C87—H87C	109.5	C5—C11—H11A	109.4
N32—C33—N34	114.88 (11)	C10—C11—H11A	109.4

N32—C33—C331	122.32 (12)	C5—C11—H11B	109.4
N34—C33—C331	122.79 (12)	C10—C11—H11B	109.4
N34—C35—O31	112.37 (11)	H11A—C11—H11B	108.0
N34—C35—C3	129.92 (12)		
C35—O31—N32—C33	−0.30 (14)	C35—N34—C33—C331	−179.71 (11)
C6—O1—C2—N21	−170.98 (12)	C33—N34—C35—O31	−1.31 (14)
C6—O1—C2—C3	8.5 (2)	C33—N34—C35—C3	177.36 (12)
N21—C2—C3—C35	1.9 (2)	N32—O31—C35—N34	1.06 (14)
O1—C2—C3—C35	−177.52 (12)	N32—O31—C35—C3	−177.79 (10)
N21—C2—C3—C4	−176.10 (14)	C2—C3—C35—N34	−8.7 (2)
O1—C2—C3—C4	4.5 (2)	C4—C3—C35—N34	169.25 (12)
C2—C3—C4—C5	−15.19 (16)	C2—C3—C35—O31	169.87 (11)
C35—C3—C4—C5	166.86 (11)	C4—C3—C35—O31	−12.13 (17)
C2—C3—C4—C41	107.26 (13)	N32—C33—C331—C332	169.18 (14)
C35—C3—C4—C41	−70.69 (14)	N34—C33—C331—C332	−9.9 (2)
C3—C4—C5—C6	14.83 (16)	N32—C33—C331—C336	−11.4 (2)
C41—C4—C5—C6	−109.04 (13)	N34—C33—C331—C336	169.55 (14)
C3—C4—C5—C11	−168.57 (11)	C336—C331—C332—C333	−0.3 (3)
C41—C4—C5—C11	67.56 (14)	C33—C331—C332—C333	179.13 (15)
C11—C5—C6—O1	179.78 (12)	C331—C332—C333—C334	−0.4 (3)
C4—C5—C6—O1	−3.72 (19)	C332—C333—C334—C335	0.9 (3)
C11—C5—C6—C7	−2.0 (2)	C333—C334—C335—C336	−0.6 (3)
C4—C5—C6—C7	174.50 (11)	C334—C335—C336—C331	−0.2 (3)
C2—O1—C6—C5	−8.98 (19)	C332—C331—C336—C335	0.6 (3)
C2—O1—C6—C7	172.59 (11)	C33—C331—C336—C335	−178.82 (16)
C5—C6—C7—C8	−173.88 (13)	C5—C4—C41—C46	65.67 (14)
O1—C6—C7—C8	4.48 (18)	C3—C4—C41—C46	−56.99 (15)
C5—C6—C7—C9	3.84 (19)	C5—C4—C41—C42	−112.39 (12)
O1—C6—C7—C9	−177.80 (12)	C3—C4—C41—C42	124.95 (12)
C6—C7—C8—C81	174.63 (12)	C46—C41—C42—C43	0.33 (19)
C9—C7—C8—C81	−2.9 (2)	C4—C41—C42—C43	178.45 (12)
C7—C8—C81—C82	38.2 (2)	C41—C42—C43—C44	−1.0 (2)
C7—C8—C81—C86	−142.75 (15)	C47—O4—C44—C43	172.53 (16)
C86—C81—C82—C83	2.8 (2)	C47—O4—C44—C45	−6.7 (2)
C8—C81—C82—C83	−178.13 (13)	C42—C43—C44—O4	−178.05 (13)
C81—C82—C83—C84	−0.7 (2)	C42—C43—C44—C45	1.2 (2)
C87—O8—C84—C83	−3.7 (2)	O4—C44—C45—C46	178.47 (13)
C87—O8—C84—C85	177.81 (15)	C43—C44—C45—C46	−0.7 (2)
C82—C83—C84—O8	179.65 (13)	C42—C41—C46—C45	0.16 (19)
C82—C83—C84—C85	−1.9 (2)	C4—C41—C46—C45	−177.97 (12)
O8—C84—C85—C86	−179.22 (14)	C44—C45—C46—C41	0.0 (2)
C83—C84—C85—C86	2.2 (2)	C8—C7—C9—C10	147.76 (15)
C84—C85—C86—C81	0.1 (2)	C6—C7—C9—C10	−29.89 (19)
C82—C81—C86—C85	−2.5 (2)	C7—C9—C10—C11	54.36 (19)
C8—C81—C86—C85	178.36 (14)	C6—C5—C11—C10	25.58 (18)
O31—N32—C33—N34	−0.52 (15)	C4—C5—C11—C10	−151.09 (12)
O31—N32—C33—C331	−179.68 (11)	C9—C10—C11—C5	−51.22 (18)
C35—N34—C33—N32	1.14 (15)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N21—H21B···N34	0.88 (2)	2.087 (19)	2.7650 (18)	133.7 (17)
C42—H42···O8^{i}	0.93	2.54	3.4594 (18)	170
N21—H21A···O4^{ii}	0.872 (19)	2.16 (2)	3.0291 (18)	171.8 (17)
C47—H47A···Cg^{iii}	0.96	2.59	3.489 (2)	156

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

Fig. 1

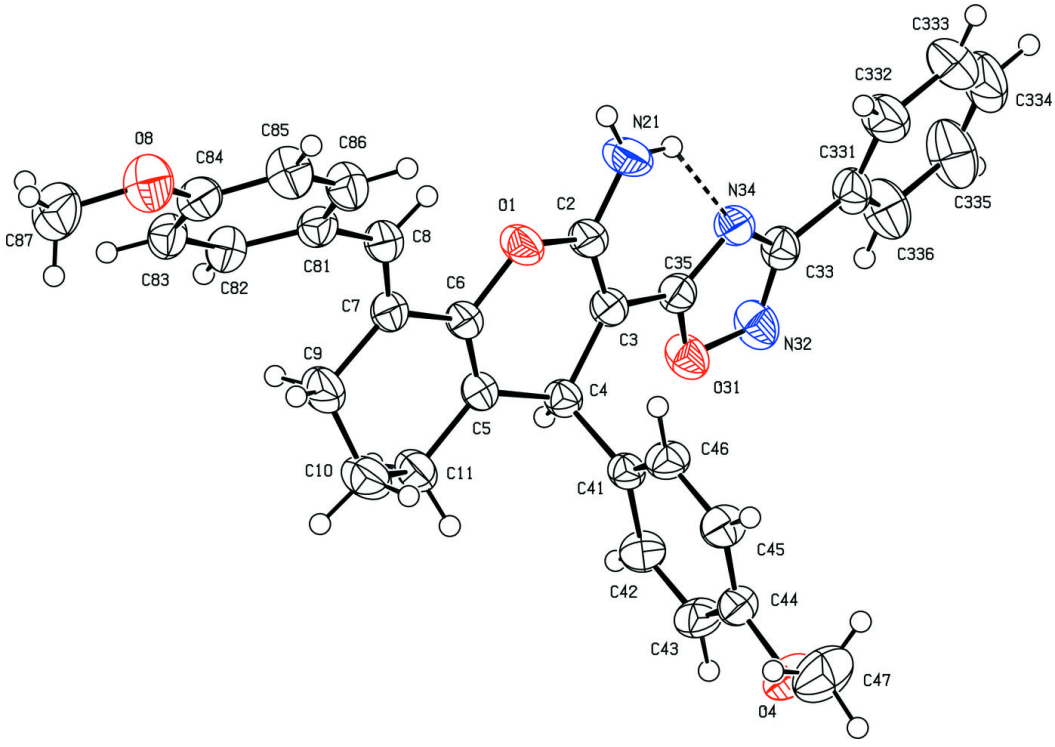
C333
C332
C334
N21
O8
C84
C85
C86
C331
N34
C335
O1
C2
C87
C8
C81
C35
C33
C336
C83
C82
C6
C7
C3
N32
C9
O31
C4
C5
C46
C10
C11
C41
C45
C42
C44
C43
C47
O4

Fig. 2