Ethyl 3-phenyl-4-[(*Z*)-3-phenylacryloyl] 5-(3,4,5-trimethoxyphenyl)pyrrolidine-2-carboxylate

P. Ramesh,[a] S. Murugavel,[b] A. Subbiah Pandi,[a]* Ramalingam Murugan[c] and S. Sriman Narayanan[c]

[a]Department of Physics, Presidency College (Autonomous), Chennai - 600 005, India, [b]Department of Physics, Government College of Engineering, Bargur - 635 104, India, and [c]Department of Analytical Chemistry, University of Madras, Guindy Campus, Chennai - 600 025, India
Correspondence e-mail: a_spandian@yahoo.com

In the title compound, $C_{31}H_{33}NO_6$, the pyrrolidine ring adopts an envelope conformation. The molecular structure is stabilized by an intramolecular N—H···O hydrogen bond which generates an $S(5)$ motif. The crystal packing is stabilized by C—H···O intermolecular hydrogen bonds and C—H···π interactions. Intermolecular C—H···O interactions are involved in the formation of dimers which are connected into a zigzag chain running along the *b* axis.

Related literature

For related literature, see: Bernstein *et al.* (1995); Bowman *et al.* (1996); Cremer & Pople (1975); Esker & Newcomb (1993); Fallis & Brinza (1997); Guindon *et al.* (2001); Nardelli (1983, 1995).
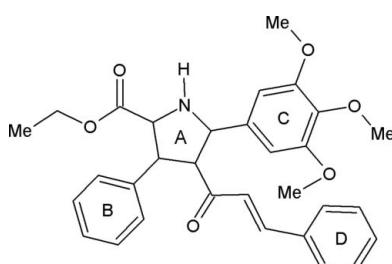
Experimental

Crystal data

$C_{31}H_{33}NO_6$	$V = 2720.29$ (16) Å³
$M_r = 515.58$	$Z = 4$
Orthorhombic, $P2_12_12$	Mo $K\alpha$ radiation
$a = 13.7016$ (5) Å	$\mu = 0.09$ mm⁻¹
$b = 23.1680$ (9) Å	$T = 293$ (2) K
$c = 8.5695$ (2) Å	$0.26 \times 0.15 \times 0.15$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer	16299 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	3169 independent reflections
$T_{min} = 0.984$, $T_{max} = 0.987$	2587 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.099$	
$S = 1.03$	
3169 reflections	$\Delta\rho_{max} = 0.14$ e Å⁻³
347 parameters	$\Delta\rho_{min} = -0.16$ e Å⁻³

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.74 (4)	2.44 (4)	2.813 (3)	113 (4)
C11—H11···O4^i	0.93	2.50	3.374 (3)	157
C12—H12···O3^ii	0.93	2.57	3.370 (3)	144
C7—H7A···Cg2^iii	0.97	2.85	3.683 (4)	145
C10—H10···Cg3^i	0.93	2.92	3.701 (3)	142

Symmetry codes: (i) $-x+2, -y+1, z$; (ii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+1$; (iii) $x, y, z+1$. $Cg2$ and $Cg3$ are the centroids of the C9–C14 and C15–C20 rings, respectively.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N. L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bowman, W. R., Stephenson, P. T. & Young, A. R. (1996). *Tetrahedron*, **52**, 11445–11462.
Bruker (2004). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Esker, J. L. & Newcomb, M. (1993). *Adv. Heterocycl. Chem.* **58**, 1–45.
Fallis, A. G. & Brinza, I. M. (1997). *Tetrahedron*, **53**, 17543–17594.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Guindon, I., Guerin, B. & Landry, S. R. (2001). *Org. Lett.* **3**, 2293–2296.
Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Zsolnai, L. (1997). *ZORTEP*. University of Heidelberg, Germany.

supplementary materials

Ethyl 3-phenyl-4-[(*Z*)-3-phenylacryloyl] 5-(3,4,5-trimethoxyphenyl)pyrrolidine-2-carboxylate

P. Ramesh, S. Murugavel, A. S. Pandi, R. Murugan and **S. S. Narayanan**

Comment

Pyrrolidine derivatives are present in a large number of biologically active natural products and numerous therapeutic agents. Radical cyclizations have emerged as a useful synthetic tool and have been reported in the synthesis of alkaloids and related pyrrolidinic compounds *via* the generation and trapping of nitrogen-centred radicals *e.g.* aminyl, iminyl, amidyl radicals *etc*. (Esker & Newcomb, 1993; Fallis & Brinza, 1997; Bowman *et al.*, 1996; Guindon *et al.*, 2001). However, no reports about the capture of neutral alkyl-oxyaminyl radicals by a multiple function have been published up to date for the preparation of fused pyrrolidine derivatives. In view of this biological importance, the crystal structure of the title compound has been determined and the results are presented here.

In the molecule of the title compound (Fig.1), the least-squares plane of the phenyl ring B lies almost perpendicular (86.4 (2)°) to the least-squares plane of the pyrrolidine ring A, whereas the least-squares plane of the trimethoxy phenyl ring C makes an angle of [60.0 (1)°]with the least-squares plane of the pyrrolidine ring A. The pyrrolidine ring A adopts an envelope conformation, with an approxymate C2 axis running through N1 and the centre of the C3—C4 bond. The puckering parameters (Cremer & Pople, 1975) q_2=0.395 (3)Å and φ=30.2 (4)°, and the smallest displacement asymmetry parameter(Nardelli, 1983) Δ_s(C2)=7.7 (2)°. The C16—C17—O3—C21 [−7.3 (3)] and C20—C19—O5—C23 [−0.5 (4)] torsional angles indicate that the two methoxy substitutions are essentially coplanor with the attached ring.

The molecular structure is stabilized by intramolecular N—H⋯O hydrogen bond which generates an S(5) motif (Fig. 2) (Bernstein *et al.*, 1995). The refined N1—H1 distance is to short and its closure to the O1 atom which generates an hydrogen bond. The sum of the angles at donar atom N1 (335.7°) of the pyrrolidine ring is accordance with sp^3 hybridization. The crystal packing is stabilized by C—H⋯O intermolecular hydrogen bonds and the C—H⋯π interactions. The molecules at (x, y, z) and $(2 − x, 1 − y, z)$ are linked by C11—H11⋯O4 hydrogen bonds into cyclic centrosymmetric R_2^2(24) dimers. The dimers are linked by the C12—H12⋯O3 hydrogen bond (Table 2.) forming C(12) zigzag chains running along the *b* axis directions. The dimers chains are cross-linked *via* C—H⋯π interactions involving the C9/C10/C11/C12/C13/C14 (centroid *Cg*2) and C15/C16/C17/C18/C19/C20 ring (*Cg*3) respectively (Table 2.).

Experimental

To a solution of ethyl {[(1E)-(3,4,5-trimethoxyphenyl)methylene]amino} acetate (1 mmol) in dry acetonitrile (10 ml), triethylamine (1 mmol), (1E,4E,6Z)-4-benzylidene-1,7-diphenylhepta-1,6-diene-3,5-diones and then lithium bromide (0.1 equiv) were added. After the completion of the reaction as determined by TLC, the reaction mixture was filtered through a celite pad, washed with saturated aqueous solution of NH_4Cl and then extracted with CH_2Cl_2 (2x20 ml). The combined organic layers were washed with brine, dried ($MgSO_4$) filtered and the solvent evaporated *in vacuo*. The residue was purified by column chromatography on silica gel (100–200 mesh) with petroleum ether/ethyl acetate (4:1) to afford the cycloadduct, which was recrystallized in etanol.

supplementary materials

Refinement

The authors have located and refined the N—H hydrogen atom whereas the remaining three types of carbons, namely methane, methylene and methyl atoms are fixed.

Figures
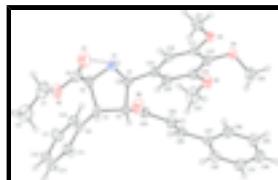
Fig. 1. The molecular configuration and atom numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

Fig. 2 Intramolecular interaction and atom numbering scheme for (I). Displacement ellipsoids are drawn at 30% probability level.

Ethyl 3-phenyl-4-[(*Z*)-3-phenylacryloyl]5-(3,4,5-trimethoxyphenyl)-\ pyrrolidine-2-carboxylate

Crystal data

$C_{31}H_{33}NO_6$	$F_{000} = 1096$
$M_r = 515.58$	$D_x = 1.259$ Mg m^{-3}
Orthorhombic, $p2_12_12$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: p 2 2ab	Cell parameters from 10259 reflections
$a = 13.7016 (5)$ Å	$\theta = 1.7–26.4°$
$b = 23.1680 (9)$ Å	$\mu = 0.09$ mm^{-1}
$c = 8.5695 (2)$ Å	$T = 293 (2)$ K
$V = 2720.29 (16)$ Å^3	Block, colourless
$Z = 4$	$0.26 \times 0.15 \times 0.15$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer	3169 independent reflections
Radiation source: fine-focus sealed tube	2587 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.026$
$T = 293(2)$ K	$\theta_{max} = 26.4°$
ω scans	$\theta_{min} = 1.7°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -17 \rightarrow 17$
$T_{min} = 0.984$, $T_{max} = 0.987$	$k = -28 \rightarrow 23$
16299 measured reflections	$l = -10 \rightarrow 6$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 0.2508P]$

where $P = (F_o{}^2 + 2F_c{}^2)/3$

$R[F^2 > 2\sigma(F^2)] = 0.036$

$(\Delta/\sigma)_{max} = 0.003$

$wR(F^2) = 0.099$

$\Delta\rho_{max} = 0.14$ e Å^{-3}

$S = 1.04$

$\Delta\rho_{min} = -0.16$ e Å^{-3}

3169 reflections

Extinction correction: none

347 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

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 > 2sigma(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	0.7792 (2)	0.47852 (9)	1.0428 (2)	0.0840 (7)
O2	0.68258 (16)	0.54921 (9)	0.9537 (2)	0.0722 (6)
O3	1.02935 (12)	0.26874 (7)	0.6449 (2)	0.0499 (4)
O4	1.08272 (11)	0.26970 (8)	0.3478 (2)	0.0539 (4)
O5	1.04771 (14)	0.36179 (8)	0.1643 (2)	0.0598 (5)
O6	0.69787 (16)	0.38770 (8)	0.7157 (2)	0.0675 (5)
N1	0.87654 (17)	0.47263 (11)	0.7542 (3)	0.0509 (5)
C2	0.87626 (16)	0.46216 (10)	0.5862 (3)	0.0421 (5)
H2	0.9017	0.4970	0.5356	0.051*
C3	0.76499 (16)	0.45786 (9)	0.5451 (3)	0.0383 (5)
H3	0.7538	0.4671	0.4349	0.046*
C4	0.71835 (16)	0.50404 (10)	0.6518 (2)	0.0400 (5)
H4	0.6607	0.4871	0.7015	0.048*
C5	0.79778 (17)	0.51404 (10)	0.7806 (3)	0.0433 (5)
H5	0.8244	0.5530	0.7668	0.052*
C6	0.7546 (2)	0.51027 (11)	0.9420 (3)	0.0546 (6)
C7	0.6255 (3)	0.55091 (17)	1.0957 (4)	0.0875 (11)
H7A	0.6635	0.5672	1.1803	0.105*
H7B	0.6051	0.5123	1.1251	0.105*
C8	0.5384 (3)	0.5879 (2)	1.0622 (5)	0.1124 (15)
H8A	0.4983	0.5904	1.1538	0.169*

H8B	0.5014	0.5712	0.9784	0.169*
H8C	0.5596	0.6258	1.0327	0.169*
C9	0.68751 (16)	0.55851 (10)	0.5691 (3)	0.0410 (5)
C10	0.75296 (18)	0.59451 (10)	0.4952 (3)	0.0466 (5)
H10	0.8189	0.5850	0.4963	0.056*
C11	0.7239 (2)	0.64415 (11)	0.4199 (3)	0.0574 (7)
H11	0.7698	0.6675	0.3708	0.069*
C12	0.6273 (2)	0.65901 (12)	0.4174 (4)	0.0659 (8)
H12	0.6073	0.6926	0.3674	0.079*
C13	0.5600 (2)	0.62392 (13)	0.4894 (4)	0.0744 (9)
H13	0.4943	0.6338	0.4877	0.089*
C14	0.58979 (18)	0.57397 (12)	0.5644 (4)	0.0599 (7)
H14	0.5436	0.5505	0.6122	0.072*
C15	0.93748 (15)	0.41216 (9)	0.5320 (3)	0.0401 (5)
C16	0.95837 (15)	0.36506 (9)	0.6259 (3)	0.0407 (5)
H16	0.9406	0.3653	0.7307	0.049*
C17	1.00602 (15)	0.31734 (10)	0.5633 (3)	0.0412 (5)
C18	1.03315 (15)	0.31654 (10)	0.4065 (3)	0.0429 (5)
C19	1.01529 (16)	0.36484 (11)	0.3146 (3)	0.0454 (6)
C20	0.96761 (15)	0.41252 (10)	0.3775 (3)	0.0435 (5)
H20	0.9559	0.4448	0.3156	0.052*
C21	1.0141 (2)	0.26865 (12)	0.8091 (3)	0.0572 (7)
H21A	1.0337	0.2321	0.8513	0.086*
H21B	0.9462	0.2750	0.8308	0.086*
H21C	1.0522	0.2988	0.8560	0.086*
C22	1.0267 (2)	0.23402 (11)	0.2476 (4)	0.0610 (7)
H22A	1.0663	0.2027	0.2107	0.091*
H22B	1.0040	0.2563	0.1604	0.091*
H22C	0.9717	0.2189	0.3039	0.091*
C23	1.0310 (3)	0.40979 (14)	0.0675 (3)	0.0833 (10)
H23A	1.0574	0.4024	−0.0343	0.125*
H23B	1.0621	0.4432	0.1114	0.125*
H23C	0.9621	0.4166	0.0593	0.125*
C24	0.72643 (16)	0.39775 (10)	0.5840 (3)	0.0433 (5)
C25	0.72722 (17)	0.35096 (10)	0.4693 (3)	0.0473 (6)
H25	0.7119	0.3143	0.5061	0.057*
C26	0.74747 (17)	0.35534 (10)	0.3189 (3)	0.0450 (5)
H26	0.7599	0.3920	0.2796	0.054*
C27	0.75193 (16)	0.30645 (10)	0.2085 (3)	0.0438 (5)
C28	0.75992 (19)	0.24969 (11)	0.2593 (3)	0.0526 (6)
H28	0.7620	0.2419	0.3657	0.063*
C29	0.7649 (2)	0.20470 (12)	0.1539 (4)	0.0616 (7)
H29	0.7715	0.1669	0.1893	0.074*
C30	0.7601 (2)	0.21589 (12)	−0.0037 (4)	0.0605 (7)
H30	0.7620	0.1856	−0.0748	0.073*
C31	0.7524 (2)	0.27172 (14)	−0.0558 (3)	0.0618 (7)
H31	0.7494	0.2793	−0.1622	0.074*
C32	0.74900 (19)	0.31668 (12)	0.0500 (3)	0.0524 (6)
H32	0.7447	0.3544	0.0138	0.063*

H1	0.862 (3)	0.4483 (17)	0.805 (5)	0.110 (17)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.129 (2)	0.0714 (15)	0.0512 (11)	0.0143 (14)	0.0021 (13)	0.0141 (11)
O2	0.0975 (15)	0.0689 (13)	0.0503 (11)	0.0204 (12)	0.0185 (11)	−0.0080 (10)
O3	0.0598 (9)	0.0385 (9)	0.0515 (9)	0.0078 (8)	−0.0067 (8)	0.0020 (8)
O4	0.0515 (9)	0.0497 (10)	0.0606 (10)	0.0134 (8)	−0.0014 (8)	−0.0164 (9)
O5	0.0742 (11)	0.0552 (11)	0.0500 (10)	0.0065 (9)	0.0178 (9)	0.0007 (9)
O6	0.1046 (15)	0.0447 (10)	0.0531 (11)	−0.0130 (10)	0.0171 (11)	0.0020 (9)
N1	0.0611 (12)	0.0451 (13)	0.0466 (12)	0.0108 (11)	−0.0109 (11)	−0.0087 (11)
C2	0.0440 (11)	0.0335 (12)	0.0489 (13)	−0.0003 (10)	−0.0007 (10)	−0.0027 (11)
C3	0.0463 (11)	0.0329 (11)	0.0357 (11)	0.0016 (9)	0.0004 (9)	−0.0036 (10)
C4	0.0457 (11)	0.0334 (11)	0.0409 (11)	−0.0011 (9)	0.0052 (10)	−0.0031 (11)
C5	0.0572 (13)	0.0330 (12)	0.0397 (11)	0.0023 (10)	−0.0045 (10)	−0.0042 (10)
C6	0.0824 (17)	0.0386 (14)	0.0429 (12)	−0.0047 (14)	−0.0018 (14)	−0.0071 (12)
C7	0.108 (3)	0.094 (3)	0.0614 (18)	−0.008 (2)	0.0295 (19)	−0.0241 (19)
C8	0.090 (2)	0.137 (4)	0.110 (3)	0.004 (3)	0.031 (2)	−0.039 (3)
C9	0.0434 (11)	0.0353 (12)	0.0444 (12)	0.0047 (10)	−0.0033 (10)	−0.0099 (10)
C10	0.0427 (10)	0.0439 (13)	0.0532 (13)	0.0063 (11)	−0.0013 (11)	0.0035 (11)
C11	0.0661 (15)	0.0476 (15)	0.0585 (16)	0.0043 (13)	−0.0100 (14)	0.0046 (13)
C12	0.0730 (17)	0.0481 (16)	0.0764 (19)	0.0198 (14)	−0.0202 (16)	0.0020 (15)
C13	0.0505 (14)	0.068 (2)	0.105 (2)	0.0266 (15)	−0.0127 (16)	−0.0096 (19)
C14	0.0446 (12)	0.0558 (16)	0.079 (2)	0.0034 (12)	0.0020 (13)	−0.0058 (16)
C15	0.0365 (10)	0.0331 (12)	0.0509 (13)	−0.0020 (9)	−0.0006 (10)	−0.0064 (11)
C16	0.0419 (10)	0.0386 (12)	0.0416 (12)	−0.0003 (10)	−0.0009 (9)	−0.0041 (11)
C17	0.0374 (10)	0.0373 (12)	0.0488 (13)	−0.0004 (9)	−0.0056 (10)	−0.0034 (11)
C18	0.0391 (10)	0.0384 (12)	0.0510 (13)	0.0030 (9)	0.0005 (10)	−0.0082 (11)
C19	0.0423 (11)	0.0469 (14)	0.0471 (13)	−0.0012 (11)	0.0022 (10)	−0.0047 (12)
C20	0.0431 (11)	0.0378 (12)	0.0497 (13)	−0.0008 (10)	0.0026 (10)	0.0032 (11)
C21	0.0624 (15)	0.0550 (16)	0.0542 (15)	0.0077 (13)	−0.0009 (13)	0.0077 (13)
C22	0.0674 (16)	0.0441 (14)	0.0714 (17)	−0.0020 (13)	0.0028 (14)	−0.0134 (15)
C23	0.131 (3)	0.065 (2)	0.0543 (17)	−0.003 (2)	0.0280 (19)	0.0057 (16)
C24	0.0512 (12)	0.0355 (12)	0.0431 (13)	0.0000 (10)	−0.0023 (10)	−0.0036 (11)
C25	0.0560 (13)	0.0320 (12)	0.0539 (15)	−0.0063 (10)	0.0016 (11)	−0.0008 (11)
C26	0.0469 (11)	0.0387 (13)	0.0492 (13)	−0.0035 (11)	−0.0061 (11)	−0.0010 (11)
C27	0.0395 (10)	0.0443 (13)	0.0476 (13)	−0.0002 (10)	−0.0058 (10)	−0.0084 (11)
C28	0.0613 (14)	0.0509 (15)	0.0457 (13)	0.0019 (12)	−0.0031 (12)	−0.0061 (12)
C29	0.0691 (16)	0.0427 (14)	0.0731 (18)	0.0015 (13)	−0.0032 (15)	−0.0092 (14)
C30	0.0605 (15)	0.0591 (18)	0.0619 (16)	−0.0034 (14)	0.0045 (13)	−0.0246 (15)
C31	0.0634 (15)	0.078 (2)	0.0441 (13)	−0.0018 (15)	0.0003 (13)	−0.0139 (14)
C32	0.0569 (13)	0.0491 (14)	0.0513 (14)	−0.0013 (12)	0.0010 (12)	−0.0028 (12)

Geometric parameters (Å, °)

O1—C6	1.183 (3)	C12—H12	0.9300
O2—C6	1.341 (3)	C13—C14	1.385 (4)
O2—C7	1.447 (4)	C13—H13	0.9300

O3—C17	1.363 (3)	C14—H14	0.9300
O3—C21	1.422 (3)	C15—C16	1.386 (3)
O4—C18	1.375 (3)	C15—C20	1.387 (3)
O4—C22	1.418 (3)	C16—C17	1.392 (3)
O5—C19	1.364 (3)	C16—H16	0.9300
O5—C23	1.406 (3)	C17—C18	1.395 (3)
O6—C24	1.217 (3)	C18—C19	1.390 (3)
N1—C2	1.460 (3)	C19—C20	1.392 (3)
N1—C5	1.462 (3)	C20—H20	0.9300
N1—H1	0.74 (4)	C21—H21A	0.9600
C2—C15	1.504 (3)	C21—H21B	0.9600
C2—C3	1.568 (3)	C21—H21C	0.9600
C2—H2	0.9800	C22—H22A	0.9600
C3—C24	1.526 (3)	C22—H22B	0.9600
C3—C4	1.546 (3)	C22—H22C	0.9600
C3—H3	0.9800	C23—H23A	0.9600
C4—C9	1.508 (3)	C23—H23B	0.9600
C4—C5	1.567 (3)	C23—H23C	0.9600
C4—H4	0.9800	C24—C25	1.463 (3)
C5—C6	1.508 (3)	C25—C26	1.322 (3)
C5—H5	0.9800	C25—H25	0.9300
C6—O1	1.183 (3)	C26—C27	1.477 (3)
C7—C8	1.497 (5)	C26—H26	0.9300
C7—H7A	0.9700	C27—C32	1.379 (3)
C7—H7B	0.9700	C27—C28	1.390 (3)
C8—H8A	0.9600	C28—C29	1.381 (4)
C8—H8B	0.9600	C28—H28	0.9300
C8—H8C	0.9600	C29—C30	1.376 (4)
C9—C10	1.379 (3)	C29—H29	0.9300
C9—C14	1.387 (3)	C30—C31	1.372 (4)
C10—C11	1.378 (3)	C30—H30	0.9300
C10—H10	0.9300	C31—C32	1.382 (4)
C11—C12	1.368 (4)	C31—H31	0.9300
C11—H11	0.9300	C32—H32	0.9300
C12—C13	1.375 (4)		
C6—O2—C7	118.6 (3)	C9—C14—H14	119.5
C17—O3—C21	118.31 (19)	C16—C15—C20	119.9 (2)
C18—O4—C22	114.44 (18)	C16—C15—C2	122.8 (2)
C19—O5—C23	117.6 (2)	C20—C15—C2	117.1 (2)
C2—N1—C5	105.1 (2)	C15—C16—C17	119.9 (2)
C2—N1—H1	117 (3)	C15—C16—H16	120.1
C5—N1—H1	103 (3)	C17—C16—H16	120.1
N1—C2—C15	115.5 (2)	O3—C17—C16	124.6 (2)
N1—C2—C3	103.58 (19)	O3—C17—C18	114.9 (2)
C15—C2—C3	115.09 (18)	C16—C17—C18	120.5 (2)
N1—C2—H2	107.4	O4—C18—C19	121.0 (2)
C15—C2—H2	107.4	O4—C18—C17	119.6 (2)
C3—C2—H2	107.4	C19—C18—C17	119.2 (2)
C24—C3—C4	111.05 (18)	O5—C19—C18	115.8 (2)

C24—C3—C2	110.20 (18)	O5—C19—C20	124.0 (2)
C4—C3—C2	103.00 (17)	C18—C19—C20	120.1 (2)
C24—C3—H3	110.8	C15—C20—C19	120.3 (2)
C4—C3—H3	110.8	C15—C20—H20	119.8
C2—C3—H3	110.8	C19—C20—H20	119.8
C9—C4—C3	114.66 (17)	O3—C21—H21A	109.5
C9—C4—C5	113.70 (19)	O3—C21—H21B	109.5
C3—C4—C5	103.41 (17)	H21A—C21—H21B	109.5
C9—C4—H4	108.3	O3—C21—H21C	109.5
C3—C4—H4	108.3	H21A—C21—H21C	109.5
C5—C4—H4	108.3	H21B—C21—H21C	109.5
N1—C5—C6	113.2 (2)	O4—C22—H22A	109.5
N1—C5—C4	107.86 (18)	O4—C22—H22B	109.5
C6—C5—C4	111.4 (2)	H22A—C22—H22B	109.5
N1—C5—H5	108.1	O4—C22—H22C	109.5
C6—C5—H5	108.1	H22A—C22—H22C	109.5
C4—C5—H5	108.1	H22B—C22—H22C	109.5
O1—C6—O2	125.1 (3)	O5—C23—H23A	109.5
O1—C6—O2	125.1 (3)	O5—C23—H23B	109.5
O1—C6—C5	126.4 (3)	H23A—C23—H23B	109.5
O1—C6—C5	126.4 (3)	O5—C23—H23C	109.5
O2—C6—C5	108.6 (2)	H23A—C23—H23C	109.5
O2—C7—C8	106.6 (3)	H23B—C23—H23C	109.5
O2—C7—H7A	110.4	O6—C24—C25	118.9 (2)
C8—C7—H7A	110.4	O6—C24—C3	119.2 (2)
O2—C7—H7B	110.4	C25—C24—C3	121.8 (2)
C8—C7—H7B	110.4	C26—C25—C24	126.8 (2)
H7A—C7—H7B	108.6	C26—C25—H25	116.6
C7—C8—H8A	109.5	C24—C25—H25	116.6
C7—C8—H8B	109.5	C25—C26—C27	125.0 (2)
H8A—C8—H8B	109.5	C25—C26—H26	117.5
C7—C8—H8C	109.5	C27—C26—H26	117.5
H8A—C8—H8C	109.5	C32—C27—C28	118.3 (2)
H8B—C8—H8C	109.5	C32—C27—C26	119.9 (2)
C10—C9—C14	117.3 (2)	C28—C27—C26	121.9 (2)
C10—C9—C4	122.7 (2)	C29—C28—C27	120.9 (2)
C14—C9—C4	120.0 (2)	C29—C28—H28	119.6
C11—C10—C9	122.2 (2)	C27—C28—H28	119.6
C11—C10—H10	118.9	C30—C29—C28	119.8 (3)
C9—C10—H10	118.9	C30—C29—H29	120.1
C12—C11—C10	119.8 (3)	C28—C29—H29	120.1
C12—C11—H11	120.1	C31—C30—C29	120.0 (3)
C10—C11—H11	120.1	C31—C30—H30	120.0
C11—C12—C13	119.5 (3)	C29—C30—H30	120.0
C11—C12—H12	120.2	C30—C31—C32	120.0 (3)
C13—C12—H12	120.2	C30—C31—H31	120.0
C12—C13—C14	120.3 (2)	C32—C31—H31	120.0
C12—C13—H13	119.8	C27—C32—C31	121.0 (3)
C14—C13—H13	119.8	C27—C32—H32	119.5

C13—C14—C9	120.9 (3)	C31—C32—H32	119.5
C13—C14—H14	119.5		
C5—N1—C2—C15	−168.34 (19)	N1—C2—C15—C20	−157.7 (2)
C5—N1—C2—C3	−41.5 (2)	C3—C2—C15—C20	81.6 (3)
N1—C2—C3—C24	−80.9 (2)	C20—C15—C16—C17	−2.6 (3)
C15—C2—C3—C24	46.2 (3)	C2—C15—C16—C17	172.7 (2)
N1—C2—C3—C4	37.6 (2)	C21—O3—C17—C16	−7.3 (3)
C15—C2—C3—C4	164.70 (19)	C21—O3—C17—C18	173.1 (2)
C24—C3—C4—C9	−136.9 (2)	C15—C16—C17—O3	−179.51 (19)
C2—C3—C4—C9	105.1 (2)	C15—C16—C17—C18	0.1 (3)
C24—C3—C4—C5	98.7 (2)	C22—O4—C18—C19	−76.5 (3)
C2—C3—C4—C5	−19.2 (2)	C22—O4—C18—C17	107.8 (3)
C2—N1—C5—C6	153.2 (2)	O3—C17—C18—O4	−2.2 (3)
C2—N1—C5—C4	29.4 (3)	C16—C17—C18—O4	178.11 (19)
C9—C4—C5—N1	−130.0 (2)	O3—C17—C18—C19	−177.99 (19)
C3—C4—C5—N1	−5.0 (2)	C16—C17—C18—C19	2.4 (3)
C9—C4—C5—C6	105.2 (2)	C23—O5—C19—C18	180.0 (2)
C3—C4—C5—C6	−129.9 (2)	C23—O5—C19—C20	−0.5 (4)
O1—O1—C6—O2	0.0 (7)	O4—C18—C19—O5	1.5 (3)
O1—O1—C6—C5	0.0 (7)	C17—C18—C19—O5	177.2 (2)
C7—O2—C6—O1	−4.2 (4)	O4—C18—C19—C20	−178.04 (19)
C7—O2—C6—O1	−4.2 (4)	C17—C18—C19—C20	−2.4 (3)
C7—O2—C6—C5	175.8 (2)	C16—C15—C20—C19	2.6 (3)
N1—C5—C6—O1	0.8 (4)	C2—C15—C20—C19	−173.0 (2)
C4—C5—C6—O1	122.6 (3)	O5—C19—C20—C15	−179.6 (2)
N1—C5—C6—O1	0.8 (4)	C18—C19—C20—C15	−0.1 (3)
C4—C5—C6—O1	122.6 (3)	C4—C3—C24—O6	−27.1 (3)
N1—C5—C6—O2	−179.2 (2)	C2—C3—C24—O6	86.3 (3)
C4—C5—C6—O2	−57.4 (3)	C4—C3—C24—C25	155.9 (2)
C6—O2—C7—C8	−167.3 (3)	C2—C3—C24—C25	−90.7 (3)
C3—C4—C9—C10	−63.0 (3)	O6—C24—C25—C26	173.6 (3)
C5—C4—C9—C10	55.7 (3)	C3—C24—C25—C26	−9.4 (4)
C3—C4—C9—C14	116.6 (2)	C24—C25—C26—C27	177.0 (2)
C5—C4—C9—C14	−124.7 (2)	C25—C26—C27—C32	163.6 (3)
C14—C9—C10—C11	0.3 (4)	C25—C26—C27—C28	−17.0 (4)
C4—C9—C10—C11	179.9 (2)	C32—C27—C28—C29	0.1 (4)
C9—C10—C11—C12	0.2 (4)	C26—C27—C28—C29	−179.4 (2)
C10—C11—C12—C13	−0.5 (5)	C27—C28—C29—C30	−1.2 (4)
C11—C12—C13—C14	0.2 (5)	C28—C29—C30—C31	1.3 (5)
C12—C13—C14—C9	0.3 (5)	C29—C30—C31—C32	−0.3 (5)
C10—C9—C14—C13	−0.5 (4)	C28—C27—C32—C31	1.0 (4)
C4—C9—C14—C13	179.8 (2)	C26—C27—C32—C31	−179.5 (2)
N1—C2—C15—C16	26.9 (3)	C30—C31—C32—C27	−0.9 (4)
C3—C2—C15—C16	−93.9 (3)		

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O1	0.74 (4)	2.44 (4)	2.813 (3)	113 (4)

C11—H11···O4^{i}	0.93	2.50	3.374 (3)	157
C12—H12···O3^{ii}	0.93	2.57	3.370 (3)	144
C7—H7A···Cg2^{iii}	0.97	2.85	3.683 (4)	145
C10—H10···Cg3^{i}	0.93	2.92	3.701 (3)	142

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

Fig. 1