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3-Benzylidene-1′-methyl-4′-phenyldispiro[cyclopentane-1,3′-pyrrolidine-2′,1″-acenaphthene]-2,2″-dione

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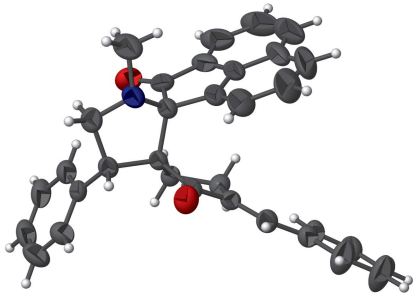
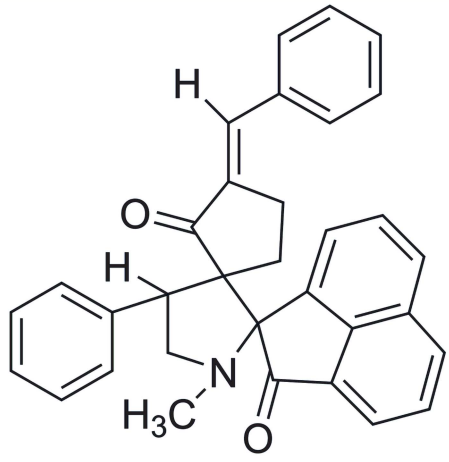
In the title compound, $C_{33}H_{27}NO_2$, the pyrrolidine ring adopts an envelope conformation, with the N atom as the flap. The cyclopentanone ring adopts a twisted conformation about the $C_s—CH_2$ bond (s = spiro link). The dihedral angle between these rings (all atoms) is 82.02 (15)°. The packing features weak C—H···O interactions.

3D view



Chemical scheme



Structure description

This work is part of a synthetic project aimed at solvent-free Claisen–Schmidt reactions of cycloalkanones with various substituted benzaldehyde (aryl aldehydes) using solid NaOH and applying a grinding technique to synthesize quantitative yields of α,α′-bis-(substituted benzylidene)cycloalkanones (Augustine *et al.*, 2009; Prasad *et al.* 2011). The carbonyl group of a ketone has less additive power than that of an aldehyde: when an aldehyde condenses with ketones it serves as a substrate for an enolized ketone. When bisbenzylidene cycloalkanones were subjected to 1,3-dipolar addition with azomethine ylides generated through decarboxylative route by reacting a secondary amino acid with a diketone using a solvent-free microwave approach, the synthesis yielded new dispiroheterocycles, which are being further subjected to biological study. Here, we present the crystal structure of the title compound.

The molecular structure consists of a pyrrolidine ring with a phenyl ring substituted in the 14 position, an acenaphthene ring in the 12 position and a benzelydine cyclopentanone ring in the 13 position (Fig. 1). The pyrrolidine (N1/C12/C13/C14/C15) ring adopts an envelope conformation with the N atom deviating from the plane by 0.581 (2) Å. The

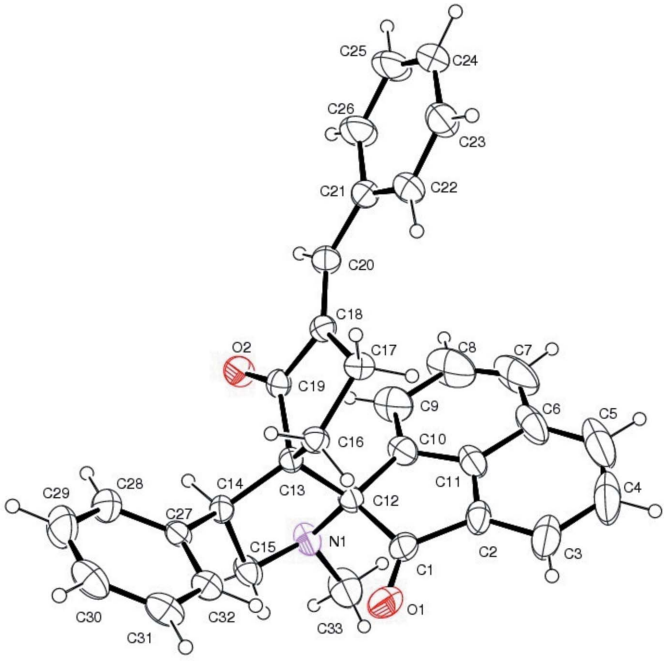


Figure 1
The molecular structure of the title compound with atoms represented as 50% probability ellipsoids.

cyclopentanone ring (C13/C16/C17/C18/C19) is twisted about the C13—C16 bond: the dihedral angle between these rings (all atoms) is 82.02 (15)°. An intramolecular C—H···O interaction closes an *S*(9) ring. The packing features weak C—H···O interactions (Table 1, Fig. 2).

Synthesis and crystallization

Acenaphthenequinone (0.37 g, 2 mmol) was added to a mixture of sarcosine (0.23 g, 2 mmol), bisbenzylidene cyclopentanone (0.53 g, 2 mmol) in methanol (20 ml) and refluxed for four h at a temperature of 100°C in an oil bath. The reaction was monitored by TLC and once the reaction was complete, the reaction mixture was cooled gradually and filtered. The yellow-coloured solution was kept undisturbed for slow evaporation and the yellow solid obtained was washed with water and dried. It was recrystallized from acetone solution to give the title compound.

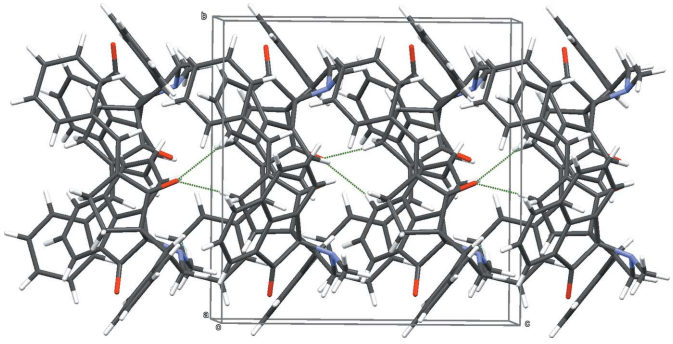


Figure 2
The packing of the title compound.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C32—H32···O1	0.93	2.58	3.436 (4)	152
C5—H5···O2^{i}	0.93	2.52	3.348 (8)	149
C17—H17*A*···O2^{ii}	0.97	2.40	3.372 (3)	174

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{33}H_{27}NO_2$
M_r	469.55
Crystal system, space group	Orthorhombic, $Pca2_1$
Temperature (K)	296
a, *b*, *c* (Å)	17.5297 (8), 12.1853 (6), 12.0781 (4)
V (Å^3)	2579.94 (19)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.08
Crystal size (mm)	0.25 × 0.22 × 0.15
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.982, 0.989
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10708, 3713, 3171
R_{int}	0.019
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.034, 0.084, 1.04
No. of reflections	3713
No. of parameters	327
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.11, −0.12

Computer programs: *APEX2* and *SAINT* (Bruker, 2004), *SHELXS97* and *SHELXTL* (Sheldrick, 2008) and *SHELXL2014* (Sheldrick, 2015).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x170581 [https://doi.org/10.1107/S2414314617005818]

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3-Benzylidene-1′-methyl-4′-phenyldispiro[cyclopentane-1,3′-pyrrolidine-2′,1′′-acenaphthene]-2,2′′-dione

Crystal data

$C_{33}H_{27}NO_2$
M_r = 469.55
Orthorhombic, $Pca2_1$
a = 17.5297 (8) Å
b = 12.1853 (6) Å
c = 12.0781 (4) Å
V = 2579.94 (19) Å^3
Z = 4
F(000) = 992
D_x = 1.209 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4328 reflections
θ = 2.3–24.2°
μ = 0.08 mm^{-1}
T = 296 K
Block, brown
0.25 × 0.22 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.982, T_{max} = 0.989
10708 measured reflections
3713 independent reflections
3171 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 25.0°, θ_{min} = 2.0°
h = −20→15
k = −14→13
l = −9→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.084
S = 1.04
3713 reflections
327 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0381P)^2 + 0.3666P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0048 (8)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Nitrogen H atoms were located in a difference Fourier map and refined isotropically. Other hydrogen atoms were fixed geometrically and allowed to ride on the parent carbon atoms,with aromatic C—H =0.93 Å, aliphatic C—H = 0.98 Å and methyl C—H = 0.96 Å. The displacement parameters were set for phenyl and aliphatic H atoms at U_{iso}(H) = 1.2U_{eq}(C) and for methyl H atoms at 1.5U_{eq}(C).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.93571 (16)	0.8127 (2)	0.6740 (3)	0.0534 (7)
C2	0.99936 (17)	0.7622 (3)	0.6124 (3)	0.0644 (10)
C3	1.0435 (2)	0.7980 (4)	0.5260 (3)	0.0979 (14)
H3	1.0345	0.8652	0.4920	0.117*
C4	1.1031 (3)	0.7283 (7)	0.4908 (5)	0.141 (3)
H4	1.1346	0.7512	0.4334	0.169*
C5	1.1157 (3)	0.6286 (7)	0.5380 (6)	0.144 (3)
H5	1.1542	0.5843	0.5093	0.173*
C6	1.0732 (2)	0.5901 (5)	0.6282 (4)	0.0980 (16)
C7	1.0820 (3)	0.4918 (5)	0.6910 (6)	0.127 (2)
H7	1.1192	0.4411	0.6713	0.153*
C8	1.0367 (3)	0.4721 (4)	0.7787 (6)	0.123 (2)
H8	1.0448	0.4085	0.8196	0.147*
C9	0.9774 (2)	0.5434 (3)	0.8115 (4)	0.0817 (11)
H9	0.9474	0.5274	0.8730	0.098*
C10	0.96486 (15)	0.6368 (2)	0.7509 (3)	0.0565 (8)
C11	1.01347 (16)	0.6597 (3)	0.6619 (3)	0.0663 (10)
C12	0.90674 (14)	0.7280 (2)	0.7623 (2)	0.0438 (6)
C13	0.82020 (13)	0.6964 (2)	0.7387 (2)	0.0364 (6)
C14	0.77458 (14)	0.7685 (2)	0.8232 (2)	0.0418 (6)
H14	0.7537	0.7180	0.8786	0.050*
C15	0.83459 (16)	0.8372 (2)	0.8822 (3)	0.0551 (8)
H15A	0.8415	0.9074	0.8457	0.066*
H15B	0.8206	0.8498	0.9589	0.066*
C16	0.79650 (16)	0.7002 (2)	0.6165 (2)	0.0411 (6)
H16A	0.7416	0.7082	0.6099	0.049*
H16B	0.8208	0.7615	0.5793	0.049*
C17	0.82258 (16)	0.5913 (2)	0.5654 (2)	0.0433 (6)
H17A	0.7896	0.5702	0.5045	0.052*
H17B	0.8746	0.5966	0.5386	0.052*
C18	0.81684 (13)	0.5107 (2)	0.6590 (2)	0.0396 (6)
C19	0.81010 (14)	0.5735 (2)	0.7635 (2)	0.0402 (6)
C20	0.81731 (15)	0.4010 (2)	0.6616 (3)	0.0479 (7)
H20	0.8139	0.3711	0.7322	0.057*
C21	0.82230 (16)	0.3197 (2)	0.5728 (3)	0.0508 (7)
C22	0.82514 (16)	0.3441 (2)	0.4606 (3)	0.0554 (8)
H22	0.8268	0.4171	0.4383	0.066*
C23	0.82559 (18)	0.2628 (3)	0.3820 (3)	0.0674 (9)
H23	0.8258	0.2812	0.3072	0.081*
C24	0.8257 (2)	0.1547 (3)	0.4131 (4)	0.0839 (12)

H24	0.8261	0.0996	0.3598	0.101*
C25	0.8253 (3)	0.1285 (3)	0.5224 (4)	0.0994 (15)
H25	0.8266	0.0552	0.5437	0.119*
C26	0.8230 (2)	0.2089 (3)	0.6015 (3)	0.0809 (12)
H26	0.8218	0.1892	0.6758	0.097*
C27	0.70803 (15)	0.8330 (2)	0.7781 (2)	0.0419 (6)
C28	0.63472 (17)	0.8051 (3)	0.8076 (3)	0.0578 (8)
H28	0.6266	0.7474	0.8567	0.069*
C29	0.57249 (19)	0.8620 (3)	0.7648 (3)	0.0754 (10)
H29	0.5232	0.8410	0.7841	0.091*
C30	0.5834 (2)	0.9485 (3)	0.6946 (3)	0.0693 (10)
H30	0.5418	0.9867	0.6663	0.083*
C31	0.6562 (2)	0.9785 (2)	0.6660 (3)	0.0652 (9)
H31	0.6641	1.0378	0.6188	0.078*
C32	0.71770 (17)	0.9211 (2)	0.7069 (3)	0.0532 (8)
H32	0.7667	0.9419	0.6864	0.064*
C33	0.97241 (19)	0.8233 (3)	0.9159 (3)	0.0843 (12)
H33A	0.9665	0.8398	0.9932	0.126*
H33B	0.9814	0.8900	0.8756	0.126*
H33C	1.0149	0.7746	0.9062	0.126*
N1	0.90293 (13)	0.7708 (2)	0.8745 (2)	0.0535 (6)
O1	0.91109 (12)	0.90536 (17)	0.6635 (2)	0.0719 (7)
O2	0.79755 (12)	0.53556 (15)	0.85476 (16)	0.0554 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0431 (16)	0.0577 (18)	0.0594 (19)	−0.0134 (14)	0.0008 (15)	−0.0130 (16)
C2	0.0444 (18)	0.091 (3)	0.058 (2)	−0.0234 (17)	0.0139 (16)	−0.0315 (18)
C3	0.074 (3)	0.147 (4)	0.072 (3)	−0.048 (3)	0.027 (2)	−0.039 (3)
C4	0.085 (4)	0.232 (7)	0.106 (5)	−0.066 (5)	0.055 (3)	−0.092 (5)
C5	0.058 (3)	0.224 (7)	0.151 (6)	−0.016 (4)	0.025 (3)	−0.129 (6)
C6	0.0384 (19)	0.135 (4)	0.120 (4)	0.006 (2)	−0.001 (2)	−0.081 (3)
C7	0.062 (3)	0.119 (4)	0.201 (7)	0.045 (3)	−0.025 (4)	−0.076 (4)
C8	0.077 (3)	0.097 (3)	0.194 (6)	0.048 (3)	−0.049 (4)	−0.033 (4)
C9	0.056 (2)	0.076 (2)	0.112 (3)	0.0186 (17)	−0.028 (2)	−0.005 (2)
C10	0.0371 (16)	0.0611 (19)	0.071 (2)	0.0054 (13)	−0.0108 (16)	−0.0189 (17)
C11	0.0342 (16)	0.086 (2)	0.079 (2)	−0.0023 (15)	−0.0017 (17)	−0.046 (2)
C12	0.0372 (16)	0.0470 (15)	0.0470 (16)	0.0022 (11)	0.0013 (12)	−0.0076 (13)
C13	0.0350 (14)	0.0370 (13)	0.0371 (14)	0.0017 (11)	0.0027 (11)	−0.0002 (11)
C14	0.0395 (15)	0.0442 (15)	0.0416 (15)	0.0015 (12)	0.0075 (13)	0.0003 (12)
C15	0.0541 (18)	0.0648 (19)	0.0465 (17)	0.0049 (15)	0.0007 (14)	−0.0191 (15)
C16	0.0447 (15)	0.0361 (14)	0.0425 (15)	0.0009 (12)	−0.0001 (12)	0.0020 (11)
C17	0.0496 (16)	0.0405 (14)	0.0398 (15)	−0.0002 (12)	−0.0049 (12)	0.0014 (12)
C18	0.0358 (13)	0.0397 (14)	0.0434 (15)	0.0012 (11)	−0.0023 (12)	0.0016 (13)
C19	0.0335 (14)	0.0443 (15)	0.0429 (17)	0.0050 (11)	−0.0012 (12)	0.0037 (14)
C20	0.0503 (16)	0.0417 (15)	0.0516 (17)	0.0034 (12)	0.0024 (14)	0.0045 (15)
C21	0.0454 (17)	0.0412 (15)	0.066 (2)	0.0016 (13)	0.0075 (14)	−0.0016 (15)

C22	0.0570 (19)	0.0480 (17)	0.061 (2)	0.0102 (14)	0.0029 (16)	−0.0055 (16)
C23	0.064 (2)	0.069 (2)	0.069 (2)	0.0068 (17)	0.0054 (17)	−0.0149 (19)
C24	0.085 (3)	0.062 (2)	0.104 (3)	−0.0154 (18)	0.032 (2)	−0.036 (2)
C25	0.152 (4)	0.0392 (19)	0.107 (4)	−0.009 (2)	0.050 (3)	−0.012 (2)
C26	0.121 (3)	0.0409 (19)	0.081 (3)	−0.0010 (19)	0.028 (2)	0.0016 (18)
C27	0.0426 (16)	0.0373 (14)	0.0457 (16)	0.0053 (11)	0.0030 (12)	−0.0080 (12)
C28	0.0470 (18)	0.0608 (19)	0.066 (2)	0.0016 (14)	0.0020 (15)	0.0001 (16)
C29	0.0418 (19)	0.098 (3)	0.087 (3)	0.0081 (17)	−0.0003 (18)	−0.008 (2)
C30	0.067 (2)	0.074 (2)	0.067 (2)	0.0315 (18)	−0.0118 (17)	−0.0145 (19)
C31	0.080 (2)	0.0495 (18)	0.066 (2)	0.0205 (16)	−0.0039 (19)	−0.0029 (17)
C32	0.0540 (18)	0.0440 (16)	0.0617 (19)	0.0068 (14)	0.0035 (14)	−0.0021 (14)
C33	0.053 (2)	0.120 (3)	0.080 (3)	0.001 (2)	−0.0162 (19)	−0.041 (2)
N1	0.0401 (13)	0.0727 (16)	0.0476 (14)	0.0061 (12)	−0.0071 (11)	−0.0186 (12)
O1	0.0697 (15)	0.0557 (13)	0.0903 (17)	−0.0123 (11)	0.0091 (13)	0.0087 (13)
O2	0.0716 (13)	0.0508 (12)	0.0438 (12)	0.0039 (10)	0.0067 (10)	0.0085 (9)

Geometric parameters (Å, º)

C1—O1	1.216 (3)	C17—C18	1.501 (4)
C1—C2	1.476 (4)	C17—H17A	0.9700
C1—C12	1.568 (4)	C17—H17B	0.9700
C2—C3	1.370 (5)	C18—C20	1.338 (3)
C2—C11	1.406 (5)	C18—C19	1.480 (4)
C3—C4	1.412 (8)	C19—O2	1.216 (3)
C3—H3	0.9300	C20—C21	1.462 (4)
C4—C5	1.360 (9)	C20—H20	0.9300
C4—H4	0.9300	C21—C22	1.388 (4)
C5—C6	1.401 (9)	C21—C26	1.394 (4)
C5—H5	0.9300	C22—C23	1.372 (4)
C6—C11	1.408 (5)	C22—H22	0.9300
C6—C7	1.426 (8)	C23—C24	1.371 (5)
C7—C8	1.345 (8)	C23—H23	0.9300
C7—H7	0.9300	C24—C25	1.357 (6)
C8—C9	1.412 (6)	C24—H24	0.9300
C8—H8	0.9300	C25—C26	1.369 (5)
C9—C10	1.370 (5)	C25—H25	0.9300
C9—H9	0.9300	C26—H26	0.9300
C10—C11	1.400 (5)	C27—C28	1.376 (4)
C10—C12	1.514 (4)	C27—C32	1.386 (4)
C12—N1	1.454 (3)	C28—C29	1.392 (4)
C12—C13	1.591 (3)	C28—H28	0.9300
C13—C16	1.534 (4)	C29—C30	1.366 (5)
C13—C19	1.538 (4)	C29—H29	0.9300
C13—C14	1.567 (4)	C30—C31	1.372 (5)
C14—C27	1.508 (4)	C30—H30	0.9300
C14—C15	1.522 (4)	C31—C32	1.376 (4)
C14—H14	0.9800	C31—H31	0.9300
C15—N1	1.449 (4)	C32—H32	0.9300

C15—H15A	0.9700	C33—N1	1.464 (4)
C15—H15B	0.9700	C33—H33A	0.9600
C16—C17	1.533 (4)	C33—H33B	0.9600
C16—H16A	0.9700	C33—H33C	0.9600
C16—H16B	0.9700		

O1—C1—C2	127.1 (3)	H16A—C16—H16B	108.7
O1—C1—C12	124.6 (3)	C18—C17—C16	104.1 (2)
C2—C1—C12	108.2 (3)	C18—C17—H17A	110.9
C3—C2—C11	120.5 (4)	C16—C17—H17A	110.9
C3—C2—C1	132.7 (4)	C18—C17—H17B	110.9
C11—C2—C1	106.8 (3)	C16—C17—H17B	110.9
C2—C3—C4	117.1 (5)	H17A—C17—H17B	109.0
C2—C3—H3	121.4	C20—C18—C19	119.8 (3)
C4—C3—H3	121.4	C20—C18—C17	132.1 (3)
C5—C4—C3	122.1 (6)	C19—C18—C17	108.0 (2)
C5—C4—H4	119.0	O2—C19—C18	126.2 (2)
C3—C4—H4	119.0	O2—C19—C13	124.6 (2)
C4—C5—C6	122.6 (5)	C18—C19—C13	109.2 (2)
C4—C5—H5	118.7	C18—C20—C21	131.3 (3)
C6—C5—H5	118.7	C18—C20—H20	114.3
C5—C6—C11	114.8 (6)	C21—C20—H20	114.3
C5—C6—C7	129.6 (5)	C22—C21—C26	116.7 (3)
C11—C6—C7	115.6 (5)	C22—C21—C20	125.0 (3)
C8—C7—C6	120.3 (4)	C26—C21—C20	118.3 (3)
C8—C7—H7	119.8	C23—C22—C21	121.5 (3)
C6—C7—H7	119.8	C23—C22—H22	119.3
C7—C8—C9	123.1 (5)	C21—C22—H22	119.3
C7—C8—H8	118.5	C24—C23—C22	120.2 (4)
C9—C8—H8	118.5	C24—C23—H23	119.9
C10—C9—C8	118.6 (5)	C22—C23—H23	119.9
C10—C9—H9	120.7	C25—C24—C23	119.5 (3)
C8—C9—H9	120.7	C25—C24—H24	120.2
C9—C10—C11	118.6 (3)	C23—C24—H24	120.2
C9—C10—C12	132.0 (3)	C24—C25—C26	120.7 (4)
C11—C10—C12	109.4 (3)	C24—C25—H25	119.7
C10—C11—C2	113.3 (3)	C26—C25—H25	119.7
C10—C11—C6	123.7 (4)	C25—C26—C21	121.4 (4)
C2—C11—C6	122.9 (4)	C25—C26—H26	119.3
N1—C12—C10	112.2 (2)	C21—C26—H26	119.3
N1—C12—C1	114.4 (2)	C28—C27—C32	117.8 (3)
C10—C12—C1	101.7 (2)	C28—C27—C14	120.0 (2)
N1—C12—C13	102.1 (2)	C32—C27—C14	122.2 (2)
C10—C12—C13	116.6 (2)	C27—C28—C29	120.8 (3)
C1—C12—C13	110.3 (2)	C27—C28—H28	119.6
C16—C13—C19	100.7 (2)	C29—C28—H28	119.6
C16—C13—C14	118.2 (2)	C30—C29—C28	120.3 (3)
C19—C13—C14	111.1 (2)	C30—C29—H29	119.8

C16—C13—C12	115.1 (2)	C28—C29—H29	119.8
C19—C13—C12	108.09 (19)	C29—C30—C31	119.5 (3)
C14—C13—C12	103.56 (19)	C29—C30—H30	120.2
C27—C14—C15	114.7 (2)	C31—C30—H30	120.2
C27—C14—C13	116.8 (2)	C30—C31—C32	120.1 (3)
C15—C14—C13	105.1 (2)	C30—C31—H31	119.9
C27—C14—H14	106.5	C32—C31—H31	119.9
C15—C14—H14	106.5	C31—C32—C27	121.4 (3)
C13—C14—H14	106.5	C31—C32—H32	119.3
N1—C15—C14	103.5 (2)	C27—C32—H32	119.3
N1—C15—H15A	111.1	N1—C33—H33A	109.5
C14—C15—H15A	111.1	N1—C33—H33B	109.5
N1—C15—H15B	111.1	H33A—C33—H33B	109.5
C14—C15—H15B	111.1	N1—C33—H33C	109.5
H15A—C15—H15B	109.0	H33A—C33—H33C	109.5
C17—C16—C13	106.3 (2)	H33B—C33—H33C	109.5
C17—C16—H16A	110.5	C15—N1—C12	107.4 (2)
C13—C16—H16A	110.5	C15—N1—C33	114.9 (2)
C17—C16—H16B	110.5	C12—N1—C33	115.9 (2)
C13—C16—H16B	110.5		

O1—C1—C2—C3	−5.0 (6)	C19—C13—C14—C15	118.6 (2)
C12—C1—C2—C3	178.1 (3)	C12—C13—C14—C15	2.7 (3)
O1—C1—C2—C11	172.8 (3)	C27—C14—C15—N1	−156.4 (2)
C12—C1—C2—C11	−4.1 (3)	C13—C14—C15—N1	−26.7 (3)
C11—C2—C3—C4	−0.6 (5)	C19—C13—C16—C17	−34.0 (3)
C1—C2—C3—C4	176.9 (4)	C14—C13—C16—C17	−155.1 (2)
C2—C3—C4—C5	1.5 (8)	C12—C13—C16—C17	82.0 (3)
C3—C4—C5—C6	−3.1 (10)	C13—C16—C17—C18	31.4 (3)
C4—C5—C6—C11	3.6 (8)	C16—C17—C18—C20	165.0 (3)
C4—C5—C6—C7	−175.9 (6)	C16—C17—C18—C19	−15.4 (3)
C5—C6—C7—C8	177.1 (5)	C20—C18—C19—O2	−7.3 (4)
C11—C6—C7—C8	−2.4 (7)	C17—C18—C19—O2	173.0 (3)
C6—C7—C8—C9	2.1 (8)	C20—C18—C19—C13	173.6 (2)
C7—C8—C9—C10	0.5 (7)	C17—C18—C19—C13	−6.1 (3)
C8—C9—C10—C11	−2.6 (5)	C16—C13—C19—O2	−154.5 (2)
C8—C9—C10—C12	179.1 (3)	C14—C13—C19—O2	−28.4 (3)
C9—C10—C11—C2	−174.6 (3)	C12—C13—C19—O2	84.5 (3)
C12—C10—C11—C2	4.1 (3)	C16—C13—C19—C18	24.6 (2)
C9—C10—C11—C6	2.3 (5)	C14—C13—C19—C18	150.6 (2)
C12—C10—C11—C6	−179.1 (3)	C12—C13—C19—C18	−96.4 (2)
C3—C2—C11—C10	178.3 (3)	C19—C18—C20—C21	179.2 (3)
C1—C2—C11—C10	0.2 (3)	C17—C18—C20—C21	−1.3 (5)
C3—C2—C11—C6	1.3 (5)	C18—C20—C21—C22	−2.4 (5)
C1—C2—C11—C6	−176.7 (3)	C18—C20—C21—C26	178.8 (3)
C5—C6—C11—C10	−179.3 (4)	C26—C21—C22—C23	2.4 (4)
C7—C6—C11—C10	0.2 (5)	C20—C21—C22—C23	−176.4 (3)
C5—C6—C11—C2	−2.7 (5)	C21—C22—C23—C24	−2.1 (5)

C7—C6—C11—C2	176.8 (3)	C22—C23—C24—C25	0.1 (6)
C9—C10—C12—N1	49.6 (4)	C23—C24—C25—C26	1.5 (7)
C11—C10—C12—N1	−128.8 (3)	C24—C25—C26—C21	−1.1 (7)
C9—C10—C12—C1	172.3 (3)	C22—C21—C26—C25	−0.8 (5)
C11—C10—C12—C1	−6.1 (3)	C20—C21—C26—C25	178.1 (4)
C9—C10—C12—C13	−67.7 (4)	C15—C14—C27—C28	−125.1 (3)
C11—C10—C12—C13	113.9 (3)	C13—C14—C27—C28	111.3 (3)
O1—C1—C12—N1	−49.6 (4)	C15—C14—C27—C32	54.9 (4)
C2—C1—C12—N1	127.4 (2)	C13—C14—C27—C32	−68.8 (3)
O1—C1—C12—C10	−170.9 (3)	C32—C27—C28—C29	1.6 (4)
C2—C1—C12—C10	6.1 (3)	C14—C27—C28—C29	−178.5 (3)
O1—C1—C12—C13	64.8 (4)	C27—C28—C29—C30	−1.5 (5)
C2—C1—C12—C13	−118.2 (2)	C28—C29—C30—C31	0.4 (5)
N1—C12—C13—C16	152.4 (2)	C29—C30—C31—C32	0.7 (5)
C10—C12—C13—C16	−84.9 (3)	C30—C31—C32—C27	−0.6 (5)
C1—C12—C13—C16	30.5 (3)	C28—C27—C32—C31	−0.6 (4)
N1—C12—C13—C19	−96.0 (2)	C14—C27—C32—C31	179.5 (3)
C10—C12—C13—C19	26.7 (3)	C14—C15—N1—C12	43.6 (3)
C1—C12—C13—C19	142.0 (2)	C14—C15—N1—C33	174.2 (3)
N1—C12—C13—C14	21.9 (2)	C10—C12—N1—C15	−166.6 (2)
C10—C12—C13—C14	144.7 (2)	C1—C12—N1—C15	78.1 (3)
C1—C12—C13—C14	−100.0 (2)	C13—C12—N1—C15	−41.0 (3)
C16—C13—C14—C27	2.6 (3)	C10—C12—N1—C33	63.3 (3)
C19—C13—C14—C27	−113.1 (2)	C1—C12—N1—C33	−51.9 (3)
C12—C13—C14—C27	131.1 (2)	C13—C12—N1—C33	−171.0 (3)
C16—C13—C14—C15	−125.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C32—H32···O1	0.93	2.58	3.436 (4)	152
C5—H5···O2^{i}	0.93	2.52	3.348 (8)	149
C17—H17*A*···O2^{ii}	0.97	2.40	3.372 (3)	174

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