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1′-Methyl-4′-phenylacenaphthene-1-spiro-2′-pyrrolidine-3′-spiro-3″-benzo[*g*]-chroman-2,4″-dione

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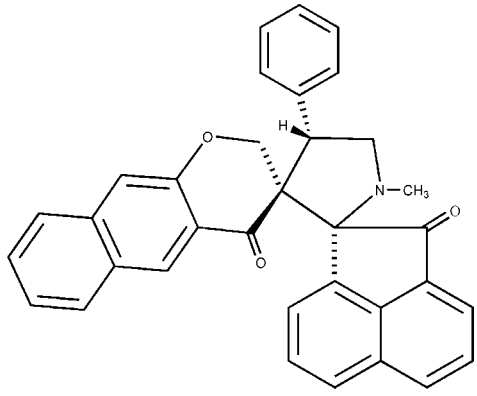
Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.155; data-to-parameter ratio = 17.5.

In the crystal structure of the title compound, $C_{34}H_{25}NO_3$, the dihydropyrone ring is in a half-chair conformation and the five-membered heterocyclic ring is in an envelope conformation.

Related literature

For related literature, see: Augustine *et al.* (2007); Deshong & Leginus (1983); Fujimori (1990); Henrickson & Silva (1962); James *et al.* (1991).



Experimental

Crystal data

$C_{34}H_{25}NO_3$
M_r = 495.55
Monoclinic, $P2_1/c$
a = 12.1286 (4) Å
b = 10.2211 (4) Å
c = 20.0589 (7) Å
β = 95.1590 (10)°
V = 2476.58 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 297 (2) K
0.45 × 0.35 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.944, T_{max} = 0.982
17928 measured reflections
6036 independent reflections
4156 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.155
S = 0.92
6036 reflections
344 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *SAINT-Plus* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); 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: NC2069).

References

Augustine, T., Ramkumar, V., Arul Antony, S. & Kanakam, C. C. (2007). *Acta Cryst.* E**63**, o4412.
Bruker (1999). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2004). *APEX2* (Version 2.0-2) and *SAINT-Plus* (Version 7.06a). Bruker AXS Inc., Madison, Wisconsin, USA.
Deshong, P. & Leginus, J. M. (1983). *J. Am. Chem. Soc.* **105**, 1686–1688.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Fujimori, S. (1990). *Chem. Abs.* **112**, 98409.
Henrickson, J. B. & Silva, R. A. (1962). *J. Am. Chem. Soc.* **34**, 643–650.
James, D. M., Kunze, H. B. & Faul Kner, D. J. (1991). *J. Nat. Prod.* **54**, 1137–1140.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

Acta Cryst. (2007). E**63**, o4717 [**doi:10.1107/S1600536807056796**]

1'-Methyl-4'-phenylacenaphthene-1-spiro-2'-pyrrolidine-3'-spiro-3''-benzo[*g*]chroman-2,4''-dione

T. Augustine, V. Ramkumar and C. C. Kanakam

Comment

An important class of naturally occurring substances like spiroheterocycles are characterized by their highly pronounced biological activities (James *et al.* 1991). The pyrrolidines which form the central skeleton of many pharmacologically active compounds and natural products have gained much prominence in recent days·(Deshong *et al.* 1983). The other class of compounds with significant biological activity are pyrrolidine and oxindole alkaloids (Fujimori 1990) which are normally found in rhyncophylline, corynoxeine, nitraphylline, vincatine and horsifiline. (Henrickson & Silva, 1962). As a part of our ongoing investigations on pharmacologically spiro compounds we report here the crystal structure of the title compound.

In the crystal structure of the title compound (I) the chromanone moiety consists of a naphthalene ring fused with a six membered heterocyclic ring. The heterocyclic ring adopts an envelope conformation that is also observed in the structure of a a similar compound reported previously (Augustine *et al.*, 2007).

The dihydropyrone ring is in half chair conformation. The nitrogen atom of the pyrrolidine ring is slightly pyramidal,the sum of the angles amount to 342.51 °.

Experimental

A mixture of acenaphthenequinone (acenaphthylene-1,2-dione) (1 mmol, 0.095 g), sarcosine (2-methylaminoacetic acid)(1 mmol, 0.043 g) and 3-benzylidene-6, 7-benz-chroman-4-one (1 mmol, 0.2 g) in aqueous methanol (20 ml) was refluxed for 4 h and the reaction was subsequently monitored by TLC for the disappearance of the starting materials. Afterwards the solvent was removed under reduced pressure and the crude product was purified by column chromatography using silica gel and petroleum ether-ethyl acetate (9:1) as eluent. Single crystals of the title compound were grown by slow evaporation of the solvent from a solution of I in methanol.

Refinement

All hydrogen atoms were placed in ideal positions and allowed to ride on the parent atom with C—H =0.93 Å for aromatic, C—H = 0.97Å for methylen, C—H = 0.98Å for methin and C—H = 0.96 Å for methyl H atoms with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and U_{iso}(H) = 1.2U_{eq}(C) for all other H atoms.

Figures

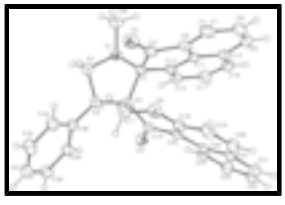


Fig. 1. *ORTEP* representation of I with the atom numbering scheme and displacement ellipsoids drawn with 30% probability.

1'-Methyl-4'-phenylacenaphthene-1-spiro-2'-pyrrolidine-3'-spiro-3''-\ benzo[*g*]chroman-2,4''-dione

Crystal data

$C_{34}H_{25}NO_3$	F_{000} = 1040
M_r = 495.55	D_x = 1.329 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5912 reflections
a = 12.1286 (4) Å	θ = 2.6–28.0º
b = 10.2211 (4) Å	μ = 0.09 mm^{-1}
c = 20.0589 (7) Å	T = 297 (2) K
β = 95.1590 (10)º	Hexagonal, colourless
V = 2476.58 (15) Å^3	0.45 × 0.35 × 0.22 mm
Z = 4	

Data collection

BRUKER APEXII CCD area-detector diffractometer	6036 independent reflections
Radiation source: fine-focus sealed tube	4156 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.024
T = 297(2) K	θ_{max} = 28.3º
φ and ω scans	θ_{min} = 2.8º
Absorption correction: multi-scan (SADABS; Bruker, 1999)	h = −10→16
T_{min} = 0.944, T_{max} = 0.982	k = −12→13
17928 measured reflections	l = −26→26

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.045	H-atom parameters constrained
$wR(F^2)$ = 0.155	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.3555P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.92	$(\Delta/\sigma)_{max} < 0.001$
6036 reflections	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
344 parameters	$\Delta\rho_{min}$ = −0.18 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}
C1	0.35562 (12)	0.79445 (14)	0.14386 (7)	0.0373 (3)
C2	0.27193 (12)	0.67730 (13)	0.12556 (6)	0.0342 (3)
C3	0.33599 (12)	0.58849 (15)	0.07938 (7)	0.0381 (3)
H3	0.3842	0.5325	0.1090	0.046*
C4	0.41132 (14)	0.68527 (17)	0.04820 (8)	0.0473 (4)
H4A	0.4720	0.6413	0.0292	0.057*
H4B	0.3708	0.7376	0.0139	0.057*
C5	0.30365 (14)	0.93248 (15)	0.12540 (7)	0.0436 (4)
C6	0.29813 (13)	1.00698 (15)	0.18812 (8)	0.0434 (4)
C7	0.25714 (15)	1.12784 (17)	0.20190 (10)	0.0552 (4)
H7	0.2224	1.1795	0.1681	0.066*
C8	0.26905 (18)	1.17163 (19)	0.26861 (11)	0.0653 (5)
H8	0.2406	1.2531	0.2787	0.078*
C9	0.32117 (16)	1.0982 (2)	0.31930 (10)	0.0602 (5)
H9	0.3285	1.1315	0.3626	0.072*
C10	0.42041 (15)	0.8861 (2)	0.35297 (8)	0.0536 (4)
H10	0.4306	0.9083	0.3981	0.064*
C11	0.45993 (14)	0.77016 (19)	0.33169 (8)	0.0520 (4)
H11	0.4977	0.7153	0.3629	0.062*
C12	0.44582 (13)	0.72961 (16)	0.26388 (8)	0.0441 (4)
H12	0.4743	0.6502	0.2507	0.053*
C13	0.38930 (12)	0.81025 (14)	0.21833 (7)	0.0369 (3)
C14	0.35039 (12)	0.93059 (15)	0.24003 (7)	0.0388 (3)
C15	0.36389 (14)	0.97312 (16)	0.30686 (8)	0.0461 (4)

C16	0.24100 (11)	0.60543 (14)	0.18841 (7)	0.0347 (3)
C17	0.16132 (13)	0.72197 (15)	0.09048 (7)	0.0419 (3)
H17A	0.1741	0.7637	0.0484	0.050*
H17B	0.1146	0.6462	0.0805	0.050*
C18	0.09846 (12)	0.77554 (15)	0.19539 (8)	0.0398 (3)
C19	0.02184 (14)	0.84872 (18)	0.22866 (9)	0.0525 (4)
H19	−0.0164	0.9177	0.2070	0.063*
C20	0.00443 (15)	0.81791 (18)	0.29231 (10)	0.0571 (5)
H20	−0.0485	0.8643	0.3134	0.069*
C21	0.04502 (16)	0.6857 (2)	0.39448 (9)	0.0595 (5)
H21	−0.0099	0.7297	0.4150	0.071*
C22	0.10542 (17)	0.5923 (2)	0.42892 (8)	0.0606 (5)
H22	0.0900	0.5701	0.4721	0.073*
C23	0.19058 (16)	0.52959 (19)	0.39951 (8)	0.0552 (4)
H23	0.2350	0.4697	0.4243	0.066*
C24	0.20999 (14)	0.55504 (16)	0.33427 (8)	0.0448 (4)
H24	0.2664	0.5108	0.3153	0.054*
C25	0.16043 (11)	0.67574 (14)	0.22682 (7)	0.0349 (3)
C26	0.14573 (12)	0.64684 (14)	0.29596 (7)	0.0371 (3)
C27	0.06414 (13)	0.71733 (17)	0.32784 (8)	0.0466 (4)
C28	0.26798 (13)	0.49805 (15)	0.03250 (7)	0.0395 (3)
C29	0.26325 (14)	0.36576 (16)	0.04875 (8)	0.0466 (4)
H29	0.3029	0.3356	0.0876	0.056*
C30	0.20093 (16)	0.27821 (18)	0.00855 (9)	0.0545 (4)
H30	0.1996	0.1902	0.0202	0.065*
C31	0.14078 (15)	0.32166 (19)	−0.04877 (9)	0.0564 (5)
H31	0.0975	0.2635	−0.0755	0.068*
C32	0.14520 (16)	0.45172 (19)	−0.06622 (9)	0.0577 (5)
H32	0.1051	0.4812	−0.1050	0.069*
C33	0.20885 (15)	0.53873 (17)	−0.02653 (8)	0.0509 (4)
H33	0.2122	0.6259	−0.0395	0.061*
C34	0.52510 (17)	0.8703 (2)	0.09401 (10)	0.0645 (5)
H34A	0.5890	0.8363	0.0750	0.097*
H34B	0.5477	0.9130	0.1356	0.097*
H34C	0.4882	0.9322	0.0636	0.097*
N1	0.45024 (11)	0.76404 (13)	0.10573 (6)	0.0442 (3)
O1	0.28162 (12)	0.97051 (12)	0.06845 (6)	0.0613 (4)
O2	0.27806 (10)	0.49797 (11)	0.20330 (5)	0.0510 (3)
O3	0.10534 (9)	0.81144 (10)	0.13061 (5)	0.0452 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0406 (8)	0.0339 (8)	0.0363 (7)	−0.0031 (6)	−0.0016 (6)	0.0033 (6)
C2	0.0363 (7)	0.0312 (7)	0.0343 (7)	0.0015 (6)	−0.0016 (6)	0.0013 (5)
C3	0.0403 (7)	0.0374 (8)	0.0363 (7)	0.0030 (7)	0.0024 (6)	0.0006 (6)
C4	0.0518 (9)	0.0499 (10)	0.0411 (8)	−0.0036 (8)	0.0083 (7)	−0.0016 (7)
C5	0.0487 (9)	0.0345 (8)	0.0460 (8)	−0.0043 (7)	−0.0041 (7)	0.0073 (6)

C6	0.0446 (8)	0.0328 (8)	0.0526 (8)	−0.0050 (7)	0.0024 (7)	0.0039 (6)
C7	0.0551 (10)	0.0378 (9)	0.0730 (11)	0.0007 (8)	0.0066 (9)	0.0036 (8)
C8	0.0660 (12)	0.0420 (10)	0.0901 (15)	−0.0002 (9)	0.0189 (11)	−0.0149 (9)
C9	0.0608 (11)	0.0584 (12)	0.0636 (11)	−0.0127 (10)	0.0176 (9)	−0.0196 (9)
C10	0.0521 (10)	0.0697 (12)	0.0385 (8)	−0.0162 (9)	0.0017 (7)	−0.0062 (7)
C11	0.0467 (9)	0.0639 (12)	0.0433 (8)	−0.0061 (9)	−0.0080 (7)	0.0088 (8)
C12	0.0397 (8)	0.0455 (9)	0.0457 (8)	−0.0002 (7)	−0.0033 (7)	0.0047 (7)
C13	0.0351 (7)	0.0358 (8)	0.0390 (7)	−0.0049 (6)	−0.0011 (6)	0.0010 (6)
C14	0.0360 (7)	0.0377 (8)	0.0425 (7)	−0.0086 (6)	0.0020 (6)	−0.0006 (6)
C15	0.0436 (8)	0.0474 (10)	0.0479 (8)	−0.0128 (7)	0.0082 (7)	−0.0074 (7)
C16	0.0332 (7)	0.0311 (7)	0.0387 (7)	0.0001 (6)	−0.0014 (6)	0.0011 (5)
C17	0.0434 (8)	0.0392 (8)	0.0413 (7)	0.0031 (7)	−0.0065 (6)	−0.0003 (6)
C18	0.0346 (7)	0.0344 (8)	0.0491 (8)	0.0004 (6)	−0.0036 (6)	−0.0045 (6)
C19	0.0429 (9)	0.0471 (10)	0.0664 (11)	0.0141 (8)	−0.0012 (8)	−0.0087 (8)
C20	0.0438 (9)	0.0564 (11)	0.0719 (12)	0.0084 (9)	0.0094 (8)	−0.0202 (9)
C21	0.0553 (10)	0.0700 (13)	0.0555 (10)	−0.0084 (10)	0.0178 (9)	−0.0203 (9)
C22	0.0698 (12)	0.0723 (13)	0.0408 (8)	−0.0168 (11)	0.0105 (8)	−0.0088 (8)
C23	0.0640 (11)	0.0556 (11)	0.0448 (8)	−0.0097 (9)	−0.0009 (8)	0.0030 (7)
C24	0.0464 (8)	0.0429 (9)	0.0451 (8)	−0.0044 (7)	0.0038 (7)	0.0000 (6)
C25	0.0306 (7)	0.0317 (8)	0.0420 (7)	−0.0030 (6)	0.0006 (6)	−0.0031 (6)
C26	0.0333 (7)	0.0357 (8)	0.0420 (7)	−0.0082 (6)	0.0019 (6)	−0.0063 (6)
C27	0.0411 (8)	0.0492 (10)	0.0501 (8)	−0.0062 (8)	0.0065 (7)	−0.0130 (7)
C28	0.0423 (8)	0.0385 (8)	0.0380 (7)	0.0030 (7)	0.0054 (6)	−0.0029 (6)
C29	0.0533 (9)	0.0414 (9)	0.0450 (8)	0.0024 (8)	0.0034 (7)	0.0004 (7)
C30	0.0625 (11)	0.0397 (9)	0.0622 (10)	−0.0056 (8)	0.0110 (9)	−0.0062 (8)
C31	0.0517 (10)	0.0558 (11)	0.0611 (10)	−0.0044 (9)	0.0019 (8)	−0.0184 (8)
C32	0.0595 (11)	0.0628 (12)	0.0481 (9)	0.0091 (9)	−0.0104 (8)	−0.0089 (8)
C33	0.0646 (11)	0.0423 (9)	0.0446 (8)	0.0053 (8)	−0.0024 (8)	−0.0013 (7)
C34	0.0606 (11)	0.0632 (13)	0.0715 (12)	−0.0205 (10)	0.0154 (10)	0.0014 (9)
N1	0.0429 (7)	0.0463 (8)	0.0437 (7)	−0.0080 (6)	0.0061 (6)	0.0004 (6)
O1	0.0836 (9)	0.0477 (7)	0.0498 (7)	0.0006 (7)	−0.0093 (6)	0.0156 (5)
O2	0.0661 (8)	0.0362 (6)	0.0525 (6)	0.0141 (6)	0.0155 (5)	0.0099 (5)
O3	0.0451 (6)	0.0404 (6)	0.0484 (6)	0.0109 (5)	−0.0052 (5)	0.0025 (5)

Geometric parameters (Å, °)

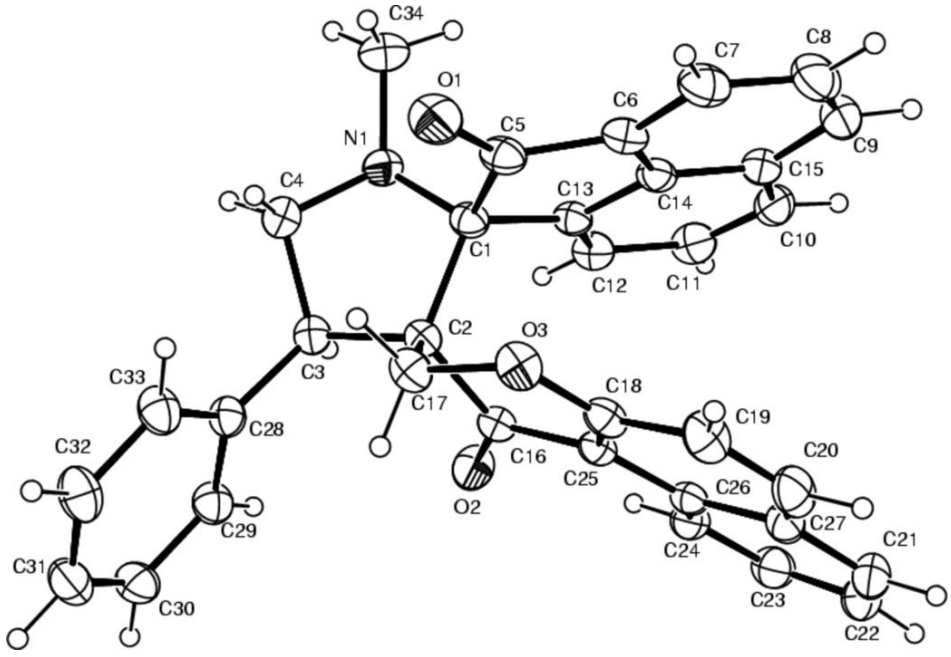
C1—N1	1.468 (2)	C17—H17A	0.9700
C1—C13	1.5214 (19)	C17—H17B	0.9700
C1—C5	1.576 (2)	C18—O3	1.3599 (18)
C1—C2	1.591 (2)	C18—C25	1.385 (2)
C2—C17	1.528 (2)	C18—C19	1.407 (2)
C2—C16	1.5345 (19)	C19—C20	1.350 (3)
C2—C3	1.554 (2)	C19—H19	0.9300
C3—C28	1.510 (2)	C20—C27	1.413 (3)
C3—C4	1.519 (2)	C20—H20	0.9300
C3—H3	0.9800	C21—C22	1.355 (3)
C4—N1	1.451 (2)	C21—C27	1.415 (2)
C4—H4A	0.9700	C21—H21	0.9300
C4—H4B	0.9700	C22—C23	1.391 (3)

C5—O1	1.2135 (17)	C22—H22	0.9300
C5—C6	1.477 (2)	C23—C24	1.375 (2)
C6—C7	1.369 (2)	C23—H23	0.9300
C6—C14	1.406 (2)	C24—C26	1.404 (2)
C7—C8	1.406 (3)	C24—H24	0.9300
C7—H7	0.9300	C25—C26	1.444 (2)
C8—C9	1.371 (3)	C26—C27	1.422 (2)
C8—H8	0.9300	C28—C33	1.392 (2)
C9—C15	1.410 (3)	C28—C29	1.393 (2)
C9—H9	0.9300	C29—C30	1.383 (2)
C10—C11	1.361 (3)	C29—H29	0.9300
C10—C15	1.415 (2)	C30—C31	1.379 (3)
C10—H10	0.9300	C30—H30	0.9300
C11—C12	1.418 (2)	C31—C32	1.377 (3)
C11—H11	0.9300	C31—H31	0.9300
C12—C13	1.368 (2)	C32—C33	1.382 (2)
C12—H12	0.9300	C32—H32	0.9300
C13—C14	1.401 (2)	C33—H33	0.9300
C14—C15	1.405 (2)	C34—N1	1.449 (2)
C16—O2	1.2140 (17)	C34—H34A	0.9600
C16—C25	1.4834 (19)	C34—H34B	0.9600
C17—O3	1.4295 (18)	C34—H34C	0.9600
N1—C1—C13	112.36 (12)	C2—C17—H17A	109.2
N1—C1—C5	112.46 (12)	O3—C17—H17B	109.2
C13—C1—C5	101.66 (11)	C2—C17—H17B	109.2
N1—C1—C2	103.46 (11)	H17A—C17—H17B	107.9
C13—C1—C2	114.57 (11)	O3—C18—C25	123.80 (13)
C5—C1—C2	112.72 (11)	O3—C18—C19	114.18 (14)
C17—C2—C16	104.60 (11)	C25—C18—C19	122.01 (15)
C17—C2—C3	111.43 (11)	C20—C19—C18	119.39 (16)
C16—C2—C3	112.48 (11)	C20—C19—H19	120.3
C17—C2—C1	113.39 (12)	C18—C19—H19	120.3
C16—C2—C1	111.64 (10)	C19—C20—C27	122.01 (15)
C3—C2—C1	103.55 (11)	C19—C20—H20	119.0
C28—C3—C4	117.22 (12)	C27—C20—H20	119.0
C28—C3—C2	117.03 (12)	C22—C21—C27	121.07 (17)
C4—C3—C2	102.50 (12)	C22—C21—H21	119.5
C28—C3—H3	106.4	C27—C21—H21	119.5
C4—C3—H3	106.4	C21—C22—C23	119.77 (16)
C2—C3—H3	106.4	C21—C22—H22	120.1
N1—C4—C3	101.45 (12)	C23—C22—H22	120.1
N1—C4—H4A	111.5	C24—C23—C22	120.91 (18)
C3—C4—H4A	111.5	C24—C23—H23	119.5
N1—C4—H4B	111.5	C22—C23—H23	119.5
C3—C4—H4B	111.5	C23—C24—C26	120.88 (16)
H4A—C4—H4B	109.3	C23—C24—H24	119.6
O1—C5—C6	127.85 (15)	C26—C24—H24	119.6
O1—C5—C1	123.88 (14)	C18—C25—C26	118.62 (13)
C6—C5—C1	108.02 (12)	C18—C25—C16	118.44 (13)

C7—C6—C14	119.94 (15)	C26—C25—C16	122.93 (13)
C7—C6—C5	133.02 (15)	C24—C26—C27	117.85 (14)
C14—C6—C5	107.01 (14)	C24—C26—C25	123.49 (13)
C6—C7—C8	118.12 (17)	C27—C26—C25	118.64 (14)
C6—C7—H7	120.9	C20—C27—C21	121.53 (16)
C8—C7—H7	120.9	C20—C27—C26	119.19 (15)
C9—C8—C7	122.18 (18)	C21—C27—C26	119.28 (16)
C9—C8—H8	118.9	C33—C28—C29	117.40 (15)
C7—C8—H8	118.9	C33—C28—C3	123.91 (14)
C8—C9—C15	121.13 (17)	C29—C28—C3	118.69 (13)
C8—C9—H9	119.4	C30—C29—C28	121.55 (16)
C15—C9—H9	119.4	C30—C29—H29	119.2
C11—C10—C15	120.39 (15)	C28—C29—H29	119.2
C11—C10—H10	119.8	C31—C30—C29	119.89 (17)
C15—C10—H10	119.8	C31—C30—H30	120.1
C10—C11—C12	122.67 (16)	C29—C30—H30	120.1
C10—C11—H11	118.7	C32—C31—C30	119.56 (16)
C12—C11—H11	118.7	C32—C31—H31	120.2
C13—C12—C11	118.34 (16)	C30—C31—H31	120.2
C13—C12—H12	120.8	C31—C32—C33	120.48 (16)
C11—C12—H12	120.8	C31—C32—H32	119.8
C12—C13—C14	118.96 (14)	C33—C32—H32	119.8
C12—C13—C1	131.76 (14)	C32—C33—C28	121.09 (16)
C14—C13—C1	109.28 (12)	C32—C33—H33	119.5
C13—C14—C15	123.69 (14)	C28—C33—H33	119.5
C13—C14—C6	113.55 (13)	N1—C34—H34A	109.5
C15—C14—C6	122.75 (15)	N1—C34—H34B	109.5
C14—C15—C9	115.86 (16)	H34A—C34—H34B	109.5
C14—C15—C10	115.93 (15)	N1—C34—H34C	109.5
C9—C15—C10	128.20 (16)	H34A—C34—H34C	109.5
O2—C16—C25	123.86 (13)	H34B—C34—H34C	109.5
O2—C16—C2	121.52 (13)	C34—N1—C4	116.80 (13)
C25—C16—C2	114.58 (12)	C34—N1—C1	117.15 (14)
O3—C17—C2	111.95 (11)	C4—N1—C1	108.55 (12)
O3—C17—H17A	109.2	C18—O3—C17	116.01 (11)
N1—C1—C2—C17	−118.51 (12)	C1—C2—C16—O2	−105.89 (16)
C13—C1—C2—C17	118.84 (13)	C17—C2—C16—C25	−46.79 (15)
C5—C1—C2—C17	3.23 (16)	C3—C2—C16—C25	−167.88 (11)
N1—C1—C2—C16	123.65 (12)	C1—C2—C16—C25	76.21 (14)
C13—C1—C2—C16	1.00 (17)	C16—C2—C17—O3	62.52 (15)
C5—C1—C2—C16	−114.60 (13)	C3—C2—C17—O3	−175.70 (12)
N1—C1—C2—C3	2.40 (13)	C1—C2—C17—O3	−59.33 (16)
C13—C1—C2—C3	−120.25 (13)	O3—C18—C19—C20	177.09 (15)
C5—C1—C2—C3	124.15 (12)	C25—C18—C19—C20	−2.7 (3)
C17—C2—C3—C28	−35.15 (17)	C18—C19—C20—C27	2.6 (3)
C16—C2—C3—C28	81.94 (15)	C27—C21—C22—C23	2.5 (3)
C1—C2—C3—C28	−157.38 (12)	C21—C22—C23—C24	−4.2 (3)
C17—C2—C3—C4	94.57 (14)	C22—C23—C24—C26	1.2 (3)
C16—C2—C3—C4	−148.34 (12)	O3—C18—C25—C26	179.96 (13)

C1—C2—C3—C4	−27.65 (13)	C19—C18—C25—C26	−0.2 (2)
C28—C3—C4—N1	172.86 (13)	O3—C18—C25—C16	1.4 (2)
C2—C3—C4—N1	43.25 (14)	C19—C18—C25—C16	−178.75 (14)
N1—C1—C5—O1	47.4 (2)	O2—C16—C25—C18	−160.19 (15)
C13—C1—C5—O1	167.78 (16)	C2—C16—C25—C18	17.65 (18)
C2—C1—C5—O1	−69.1 (2)	O2—C16—C25—C26	21.3 (2)
N1—C1—C5—C6	−127.24 (13)	C2—C16—C25—C26	−160.81 (12)
C13—C1—C5—C6	−6.87 (15)	C23—C24—C26—C27	3.3 (2)
C2—C1—C5—C6	116.26 (13)	C23—C24—C26—C25	−178.59 (15)
O1—C5—C6—C7	8.7 (3)	C18—C25—C26—C24	−174.88 (14)
C1—C5—C6—C7	−176.93 (18)	C16—C25—C26—C24	3.6 (2)
O1—C5—C6—C14	−169.31 (17)	C18—C25—C26—C27	3.3 (2)
C1—C5—C6—C14	5.06 (17)	C16—C25—C26—C27	−178.28 (13)
C14—C6—C7—C8	0.0 (3)	C19—C20—C27—C21	−179.86 (18)
C5—C6—C7—C8	−177.83 (18)	C19—C20—C27—C26	0.5 (3)
C6—C7—C8—C9	0.9 (3)	C22—C21—C27—C20	−177.63 (17)
C7—C8—C9—C15	−1.2 (3)	C22—C21—C27—C26	2.0 (3)
C15—C10—C11—C12	−0.9 (3)	C24—C26—C27—C20	174.83 (15)
C10—C11—C12—C13	−0.6 (3)	C25—C26—C27—C20	−3.4 (2)
C11—C12—C13—C14	1.7 (2)	C24—C26—C27—C21	−4.8 (2)
C11—C12—C13—C1	−176.97 (15)	C25—C26—C27—C21	176.92 (14)
N1—C1—C13—C12	−54.4 (2)	C4—C3—C28—C33	−45.5 (2)
C5—C1—C13—C12	−174.88 (16)	C2—C3—C28—C33	76.87 (19)
C2—C1—C13—C12	63.3 (2)	C4—C3—C28—C29	134.60 (16)
N1—C1—C13—C14	126.81 (13)	C2—C3—C28—C29	−103.01 (16)
C5—C1—C13—C14	6.37 (15)	C33—C28—C29—C30	−1.0 (2)
C2—C1—C13—C14	−115.49 (14)	C3—C28—C29—C30	178.86 (15)
C12—C13—C14—C15	−1.4 (2)	C28—C29—C30—C31	−0.6 (3)
C1—C13—C14—C15	177.51 (14)	C29—C30—C31—C32	1.4 (3)
C12—C13—C14—C6	177.28 (14)	C30—C31—C32—C33	−0.4 (3)
C1—C13—C14—C6	−3.78 (17)	C31—C32—C33—C28	−1.4 (3)
C7—C6—C14—C13	−179.24 (15)	C29—C28—C33—C32	2.0 (2)
C5—C6—C14—C13	−0.92 (18)	C3—C28—C33—C32	−177.86 (16)
C7—C6—C14—C15	−0.5 (2)	C3—C4—N1—C34	−179.27 (14)
C5—C6—C14—C15	177.80 (14)	C3—C4—N1—C1	−44.13 (15)
C13—C14—C15—C9	178.84 (15)	C13—C1—N1—C34	−75.17 (17)
C6—C14—C15—C9	0.3 (2)	C5—C1—N1—C34	38.81 (18)
C13—C14—C15—C10	0.0 (2)	C2—C1—N1—C34	160.73 (13)
C6—C14—C15—C10	−178.63 (14)	C13—C1—N1—C4	149.87 (12)
C8—C9—C15—C14	0.6 (3)	C5—C1—N1—C4	−96.14 (14)
C8—C9—C15—C10	179.29 (17)	C2—C1—N1—C4	25.77 (15)
C11—C10—C15—C14	1.2 (2)	C25—C18—O3—C17	14.0 (2)
C11—C10—C15—C9	−177.53 (17)	C19—C18—O3—C17	−165.81 (13)
C17—C2—C16—O2	131.11 (14)	C2—C17—O3—C18	−48.00 (17)
C3—C2—C16—O2	10.03 (19)		

Fig. 1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