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6″-Methoxy-1′-methyl-4′-phenylacenaphthene-1-spiro-2′-pyrrolidine-3′-spiro-3″-chroman-2,4″-dione

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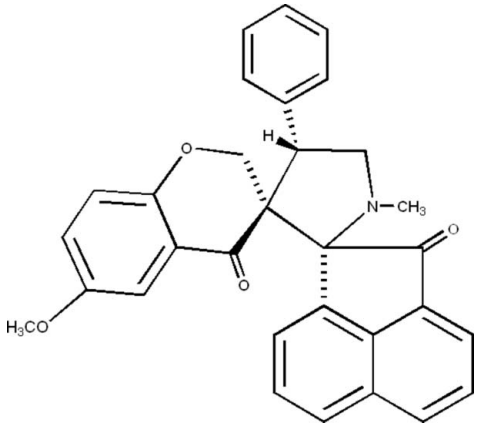
Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.003 Å; R factor = 0.055; wR factor = 0.160; data-to-parameter ratio = 17.6.

In the crystal structure of the title compound, $C_{31}H_{25}NO_4$, the five-membered heterocyclic ring is in an envelope conformation and the dihydropyrone ring is in a half-chair conformation.

Related literature

For related literature, see: Fujimori (1990), James *et al.* (1991), Deshong *et al.* (1983) and Henrickson *et al.* (1962).



Experimental

Crystal data

$C_{31}H_{25}NO_4$
M_r = 475.52
Triclinic, $P\overline{1}$
a = 8.3233 (6) Å
b = 9.2393 (7) Å
c = 15.9107 (12) Å
α = 79.969 (4)°
β = 88.820 (4)°
γ = 86.311 (4)°
V = 1202.30 (16) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 297 (2) K
0.25 × 0.22 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.969, T_{max} = 0.983
14955 measured reflections
5771 independent reflections
3881 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.160
S = 1.03
5771 reflections
327 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 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: NC2063).

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supplementary materials

Acta Cryst. (2007). E**63**, o4412 [**doi:10.1107/S1600536807051264**]

6''-Methoxy-1'-methyl-4'-phenylacenaphthene-1-spiro-2'-pyrrolidine-3'-spiro-3''-chroman-2,4''-dione

T. Augustine, V. Ramkumar, S. Arul Antony and C. C. Kanakam

Comment

Spiroheterocycles belong to an important class of compounds with highly pronounced biological activities·(James *et al.* 1991) The central skeleton of many natural products and pharmacologically active compounds contain highly substituted pyrrolidines which are of much prominence (Deshong *et al.*, 1983). Some of the biological active compounds such as rhyncophylline, corynoxeine,nitraphylline,vincatine, horsifiline,*etc* (Henrickson *et al.* 1962) contain pyrrolidine and oxindole moiety which forms a separate class of alkaloids (Fujimori 1990). In view of this we have determined the structure of the title compound.

The chromanone moiety consists of a methoxy benzene ring fused with a five membered heterocyclic ring which adopts a sofa conformation. The dihydropyrone ring is in an half chair conformation. The nitrogen atom of the pyrrolidine ring is slightly pyramidal, the sum of the angles amount to 342.04 °.

Experimental

A mixture of acenapthenequinone (acenaphthylene-1,2-dione) (1 mmol, 0.102 g), sarcosine (2-methylaminoacetic acid)(1 mmol, 0.046 g) and the dipolarophile (3-arylidene-4-chromanone) (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. 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 to give the cycloadduct.

Refinement

All H atoms were positioned with idealized geometry and were refined isotropic with U_{iso}(H) = 1.5 U_{eq}(C) for methyl H atoms and 1.2 U_{eq}(C) for all other H atoms using a riding model with C—H = 0.93 − 0.98 Å.

Figures

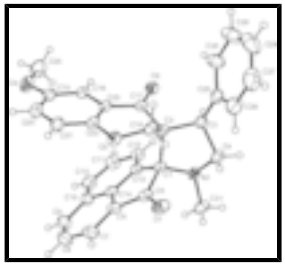


Fig. 1. *ORTEP* representation of the molecule showing the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability.

6''-Methoxy-1'-methyl-4'-phenylacenaphthene-1-spiro-2'-pyrrolidine-3'- spiro-3''-chroman-2,4''-dione

Crystal data

$C_{31}H_{25}NO_4$	$Z = 2$
$M_r = 475.52$	$F_{000} = 500$
Triclinic, $P\overline{1}$	$D_x = 1.314$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.3233$ (6) Å	Cell parameters from 6077 reflections
$b = 9.2393$ (7) Å	$\theta = 2.5$–27.7º
$c = 15.9107$ (12) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 79.969$ (4)º	$T = 297$ (2) K
$\beta = 88.820$ (4)º	Cubic, colourless
$\gamma = 86.311$ (4)º	0.25 × 0.22 × 0.20 mm
$V = 1202.30$ (16) Å^3	

Data collection

Bruker APEXII CCD area-detector diffractometer	5771 independent reflections
Radiation source: fine-focus sealed tube	3881 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.038$
$T = 297$(2) K	$\theta_{max} = 28.4$º
φ and ω scans	$\theta_{min} = 1.3$º
Absorption correction: multi-scan (Blessing, 1995)	$h = -10 \rightarrow 11$
$T_{min} = 0.969$, $T_{max} = 0.983$	$k = -9 \rightarrow 12$
14955 measured reflections	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.055$	H-atom parameters constrained
$wR(F^2) = 0.160$	$w = 1/[\sigma^2(F_o^2) + (0.0754P)^2 + 0.2718P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
5771 reflections	$\Delta\rho_{max} = 0.28$ e Å^{-3}
327 parameters	$\Delta\rho_{min} = -0.28$ 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 $F^2 > 2\mathrm{sigma}(F^2)$ 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	0.57850 (18)	0.03937 (18)	0.27098 (11)	0.0360 (4)
C2	0.40922 (18)	−0.00749 (17)	0.31375 (10)	0.0346 (3)
C3	0.45746 (19)	−0.09347 (19)	0.40289 (11)	0.0408 (4)
H3	0.4611	−0.0200	0.4403	0.049*
C4	0.6312 (2)	−0.1479 (2)	0.38993 (12)	0.0462 (4)
H4A	0.6873	−0.1749	0.4438	0.055*
H4B	0.6376	−0.2312	0.3601	0.055*
C5	0.61230 (19)	−0.0249 (2)	0.18623 (12)	0.0421 (4)
C6	0.6259 (2)	0.0998 (2)	0.11601 (11)	0.0432 (4)
C7	0.6504 (2)	0.1087 (3)	0.02958 (13)	0.0577 (5)
H7	0.6568	0.0244	0.0048	0.069*
C8	0.6655 (3)	0.2480 (3)	−0.02011 (14)	0.0674 (6)
H8	0.6792	0.2555	−0.0788	0.081*
C9	0.6607 (3)	0.3738 (3)	0.01491 (14)	0.0644 (6)
H9	0.6743	0.4639	−0.0201	0.077*
C10	0.6304 (2)	0.4866 (2)	0.14943 (14)	0.0565 (5)
H10	0.6401	0.5823	0.1205	0.068*
C11	0.6114 (2)	0.4608 (2)	0.23585 (15)	0.0557 (5)
H11	0.6100	0.5400	0.2648	0.067*
C12	0.5937 (2)	0.31861 (19)	0.28313 (12)	0.0444 (4)

H12	0.5827	0.3043	0.3422	0.053*
C13	0.59326 (18)	0.20230 (18)	0.24059 (11)	0.0364 (4)
C14	0.61716 (19)	0.22864 (19)	0.15170 (11)	0.0395 (4)
C15	0.6353 (2)	0.3685 (2)	0.10360 (12)	0.0484 (4)
C16	0.3152 (2)	−0.10079 (18)	0.26368 (11)	0.0407 (4)
H16A	0.3783	−0.1918	0.2606	0.049*
H16B	0.2161	−0.1260	0.2942	0.049*
C17	0.30017 (18)	0.13009 (18)	0.31895 (11)	0.0363 (4)
C18	0.1461 (2)	0.33730 (19)	0.22831 (12)	0.0427 (4)
H18	0.1476	0.3909	0.2726	0.051*
C19	0.0768 (2)	0.3996 (2)	0.15207 (13)	0.0484 (4)
C20	0.0703 (2)	0.3165 (2)	0.08699 (13)	0.0558 (5)
H20	0.0214	0.3583	0.0357	0.067*
C21	0.1346 (2)	0.1749 (2)	0.09756 (13)	0.0518 (5)
H21	0.1281	0.1203	0.0540	0.062*
C22	0.20975 (19)	0.11263 (19)	0.17369 (11)	0.0396 (4)
C23	0.21491 (18)	0.19285 (18)	0.23951 (11)	0.0369 (4)
C24	0.0114 (3)	0.6291 (3)	0.19620 (18)	0.0801 (7)
H24A	−0.0518	0.5853	0.2440	0.120*
H24B	−0.0346	0.7258	0.1741	0.120*
H24C	0.1196	0.6360	0.2143	0.120*
C25	0.3443 (2)	−0.2068 (2)	0.44581 (12)	0.0490 (5)
C26	0.3616 (3)	−0.3527 (3)	0.43840 (17)	0.0727 (7)
H26	0.4423	−0.3846	0.4034	0.087*
C27	0.2594 (4)	−0.4537 (3)	0.4829 (2)	0.0962 (10)
H27	0.2717	−0.5523	0.4772	0.115*
C28	0.1417 (4)	−0.4081 (4)	0.53457 (19)	0.0990 (11)
H28	0.0759	−0.4761	0.5658	0.119*
C29	0.1200 (3)	−0.2623 (4)	0.54074 (17)	0.0932 (10)
H29	0.0372	−0.2309	0.5747	0.112*
C30	0.2205 (3)	−0.1618 (3)	0.49687 (14)	0.0681 (6)
H30	0.2051	−0.0629	0.5015	0.082*
C31	0.8618 (2)	−0.0306 (3)	0.31296 (15)	0.0604 (6)
H31A	0.9278	−0.0529	0.3629	0.091*
H31B	0.8901	0.0612	0.2795	0.091*
H31C	0.8787	−0.1075	0.2797	0.091*
N1	0.69349 (16)	−0.01929 (16)	0.33844 (10)	0.0426 (4)
O1	0.63514 (17)	−0.15490 (15)	0.18358 (10)	0.0584 (4)
O2	0.27733 (15)	−0.02802 (13)	0.17924 (8)	0.0454 (3)
O3	0.28659 (16)	0.18571 (15)	0.38289 (8)	0.0512 (3)
O4	0.01298 (19)	0.54128 (16)	0.13208 (10)	0.0658 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0284 (7)	0.0367 (9)	0.0444 (9)	−0.0016 (6)	0.0011 (6)	−0.0117 (7)
C2	0.0284 (7)	0.0340 (8)	0.0416 (9)	−0.0021 (6)	−0.0004 (6)	−0.0074 (7)
C3	0.0328 (8)	0.0427 (10)	0.0457 (10)	−0.0018 (7)	−0.0032 (7)	−0.0041 (7)

C4	0.0353 (9)	0.0444 (10)	0.0573 (11)	0.0005 (7)	−0.0079 (8)	−0.0041 (8)
C5	0.0293 (8)	0.0431 (10)	0.0576 (11)	−0.0022 (7)	0.0030 (7)	−0.0198 (8)
C6	0.0320 (8)	0.0520 (11)	0.0485 (10)	−0.0047 (7)	0.0056 (7)	−0.0173 (8)
C7	0.0466 (11)	0.0790 (15)	0.0534 (12)	−0.0086 (10)	0.0081 (9)	−0.0276 (11)
C8	0.0612 (13)	0.0972 (19)	0.0441 (12)	−0.0118 (12)	0.0066 (9)	−0.0112 (12)
C9	0.0566 (13)	0.0773 (16)	0.0537 (13)	−0.0105 (11)	0.0020 (10)	0.0064 (11)
C10	0.0549 (12)	0.0399 (11)	0.0723 (15)	−0.0078 (8)	−0.0025 (10)	−0.0015 (10)
C11	0.0568 (12)	0.0395 (11)	0.0752 (15)	−0.0086 (8)	−0.0005 (10)	−0.0200 (10)
C12	0.0405 (9)	0.0460 (10)	0.0507 (11)	−0.0061 (7)	0.0012 (8)	−0.0184 (8)
C13	0.0285 (8)	0.0377 (9)	0.0445 (9)	−0.0022 (6)	0.0004 (6)	−0.0117 (7)
C14	0.0289 (8)	0.0445 (10)	0.0464 (10)	−0.0025 (6)	0.0010 (7)	−0.0113 (8)
C15	0.0395 (9)	0.0508 (11)	0.0526 (11)	−0.0061 (8)	−0.0004 (8)	−0.0019 (9)
C16	0.0323 (8)	0.0385 (9)	0.0519 (10)	−0.0024 (6)	−0.0034 (7)	−0.0089 (8)
C17	0.0272 (7)	0.0407 (9)	0.0418 (9)	−0.0019 (6)	0.0031 (6)	−0.0095 (7)
C18	0.0348 (9)	0.0434 (10)	0.0498 (10)	0.0026 (7)	0.0026 (7)	−0.0102 (8)
C19	0.0381 (9)	0.0463 (11)	0.0575 (12)	0.0059 (7)	0.0003 (8)	−0.0032 (9)
C20	0.0514 (11)	0.0614 (13)	0.0509 (11)	0.0085 (9)	−0.0113 (9)	−0.0028 (10)
C21	0.0497 (11)	0.0592 (12)	0.0479 (11)	0.0053 (9)	−0.0107 (8)	−0.0147 (9)
C22	0.0301 (8)	0.0424 (10)	0.0469 (10)	0.0002 (7)	−0.0018 (7)	−0.0096 (8)
C23	0.0263 (7)	0.0419 (9)	0.0425 (9)	0.0008 (6)	0.0009 (6)	−0.0083 (7)
C24	0.0880 (18)	0.0550 (14)	0.0968 (19)	0.0202 (12)	−0.0077 (14)	−0.0201 (13)
C25	0.0379 (9)	0.0572 (12)	0.0482 (11)	−0.0075 (8)	−0.0071 (8)	0.0040 (9)
C26	0.0552 (13)	0.0555 (14)	0.1002 (19)	−0.0099 (10)	0.0027 (12)	0.0087 (12)
C27	0.0789 (19)	0.0747 (18)	0.123 (3)	−0.0311 (14)	−0.0210 (18)	0.0269 (17)
C28	0.079 (2)	0.135 (3)	0.0760 (18)	−0.064 (2)	−0.0102 (15)	0.0235 (18)
C29	0.0667 (16)	0.154 (3)	0.0644 (16)	−0.0524 (18)	0.0146 (12)	−0.0203 (17)
C30	0.0509 (12)	0.0996 (18)	0.0568 (13)	−0.0224 (11)	0.0060 (10)	−0.0164 (12)
C31	0.0271 (9)	0.0734 (14)	0.0803 (15)	−0.0004 (8)	−0.0001 (9)	−0.0134 (11)
N1	0.0266 (7)	0.0465 (9)	0.0537 (9)	−0.0015 (6)	−0.0034 (6)	−0.0054 (7)
O1	0.0538 (8)	0.0449 (8)	0.0815 (10)	0.0008 (6)	0.0071 (7)	−0.0273 (7)
O2	0.0452 (7)	0.0445 (7)	0.0488 (7)	0.0040 (5)	−0.0088 (5)	−0.0155 (6)
O3	0.0511 (8)	0.0594 (8)	0.0442 (7)	0.0100 (6)	0.0001 (6)	−0.0173 (6)
O4	0.0684 (10)	0.0510 (9)	0.0729 (10)	0.0179 (7)	−0.0084 (7)	−0.0033 (7)

Geometric parameters (Å, °)

C1—N1	1.461 (2)	C16—H16B	0.9700
C1—C13	1.511 (2)	C17—O3	1.2173 (19)
C1—C5	1.579 (2)	C17—C23	1.472 (2)
C1—C2	1.604 (2)	C18—C19	1.371 (3)
C2—C17	1.527 (2)	C18—C23	1.401 (2)
C2—C16	1.530 (2)	C18—H18	0.9300
C2—C3	1.547 (2)	C19—O4	1.367 (2)
C3—C25	1.517 (2)	C19—C20	1.396 (3)
C3—C4	1.522 (2)	C20—C21	1.365 (3)
C3—H3	0.9800	C20—H20	0.9300
C4—N1	1.440 (2)	C21—C22	1.390 (2)
C4—H4A	0.9700	C21—H21	0.9300
C4—H4B	0.9700	C22—O2	1.371 (2)

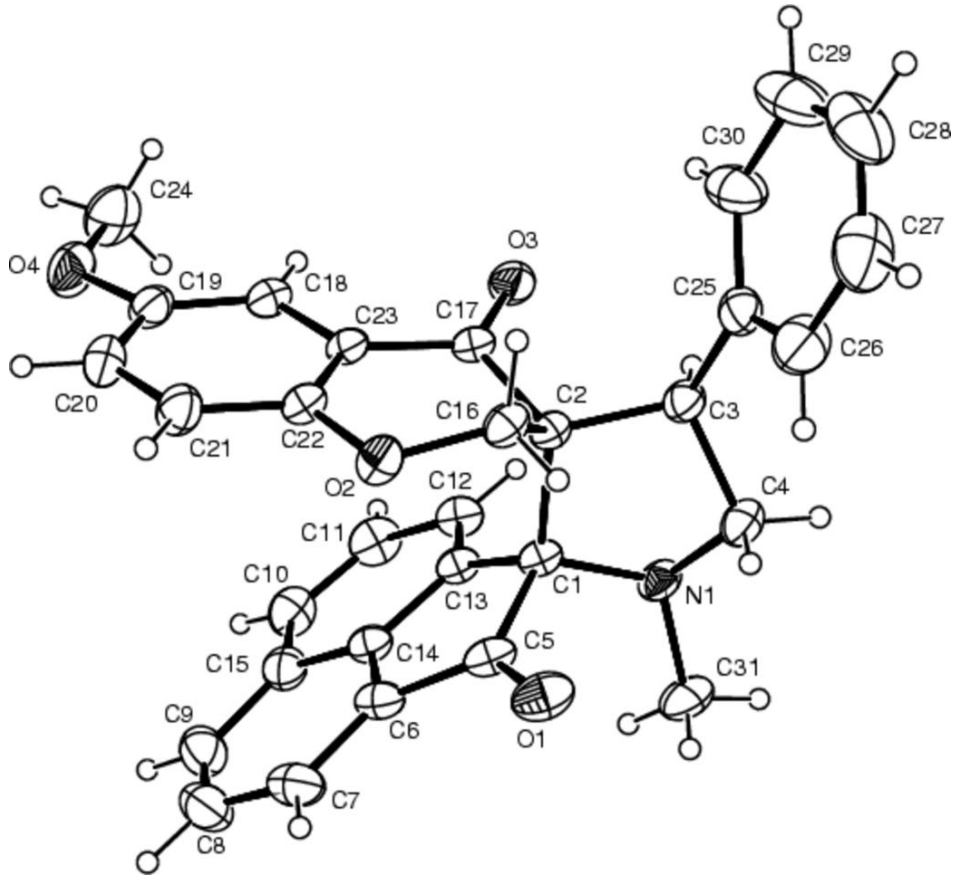
C5—O1	1.212 (2)	C22—C23	1.387 (2)
C5—C6	1.467 (3)	C24—O4	1.409 (3)
C6—C7	1.375 (3)	C24—H24A	0.9600
C6—C14	1.403 (2)	C24—H24B	0.9600
C7—C8	1.400 (3)	C24—H24C	0.9600
C7—H7	0.9300	C25—C26	1.372 (3)
C8—C9	1.372 (3)	C25—C30	1.387 (3)
C8—H8	0.9300	C26—C27	1.394 (3)
C9—C15	1.415 (3)	C26—H26	0.9300
C9—H9	0.9300	C27—C28	1.359 (5)
C10—C11	1.362 (3)	C27—H27	0.9300
C10—C15	1.412 (3)	C28—C29	1.368 (5)
C10—H10	0.9300	C28—H28	0.9300
C11—C12	1.410 (3)	C29—C30	1.378 (3)
C11—H11	0.9300	C29—H29	0.9300
C12—C13	1.366 (2)	C30—H30	0.9300
C12—H12	0.9300	C31—N1	1.453 (2)
C13—C14	1.405 (2)	C31—H31A	0.9600
C14—C15	1.398 (3)	C31—H31B	0.9600
C16—O2	1.426 (2)	C31—H31C	0.9600
C16—H16A	0.9700		

N1—C1—C13	111.49 (13)	O2—C16—H16B	109.0
N1—C1—C5	113.14 (13)	C2—C16—H16B	109.0
C13—C1—C5	101.98 (13)	H16A—C16—H16B	107.8
N1—C1—C2	102.92 (13)	O3—C17—C23	122.31 (15)
C13—C1—C2	116.02 (13)	O3—C17—C2	123.07 (15)
C5—C1—C2	111.71 (12)	C23—C17—C2	114.61 (14)
C17—C2—C16	105.91 (13)	C19—C18—C23	120.01 (17)
C17—C2—C3	112.42 (13)	C19—C18—H18	120.0
C16—C2—C3	111.65 (13)	C23—C18—H18	120.0
C17—C2—C1	109.67 (13)	O4—C19—C18	125.42 (18)
C16—C2—C1	114.13 (13)	O4—C19—C20	115.01 (17)
C3—C2—C1	103.22 (12)	C18—C19—C20	119.56 (17)
C25—C3—C4	116.79 (15)	C21—C20—C19	121.00 (18)
C25—C3—C2	117.08 (14)	C21—C20—H20	119.5
C4—C3—C2	103.40 (13)	C19—C20—H20	119.5
C25—C3—H3	106.2	C20—C21—C22	119.74 (18)
C4—C3—H3	106.2	C20—C21—H21	120.1
C2—C3—H3	106.2	C22—C21—H21	120.1
N1—C4—C3	100.96 (13)	O2—C22—C23	123.02 (15)
N1—C4—H4A	111.6	O2—C22—C21	117.01 (16)
C3—C4—H4A	111.6	C23—C22—C21	119.97 (16)
N1—C4—H4B	111.6	C22—C23—C18	119.67 (15)
C3—C4—H4B	111.6	C22—C23—C17	120.31 (15)
H4A—C4—H4B	109.4	C18—C23—C17	119.93 (15)
O1—C5—C6	127.80 (17)	O4—C24—H24A	109.5
O1—C5—C1	124.22 (17)	O4—C24—H24B	109.5
C6—C5—C1	107.73 (14)	H24A—C24—H24B	109.5
C7—C6—C14	119.71 (18)	O4—C24—H24C	109.5

C7—C6—C5	132.76 (17)	H24A—C24—H24C	109.5
C14—C6—C5	107.47 (15)	H24B—C24—H24C	109.5
C6—C7—C8	118.2 (2)	C26—C25—C30	118.4 (2)
C6—C7—H7	120.9	C26—C25—C3	123.00 (19)
C8—C7—H7	120.9	C30—C25—C3	118.56 (19)
C9—C8—C7	122.2 (2)	C25—C26—C27	120.6 (3)
C9—C8—H8	118.9	C25—C26—H26	119.7
C7—C8—H8	118.9	C27—C26—H26	119.7
C8—C9—C15	121.0 (2)	C28—C27—C26	120.0 (3)
C8—C9—H9	119.5	C28—C27—H27	120.0
C15—C9—H9	119.5	C26—C27—H27	120.0
C11—C10—C15	120.14 (18)	C27—C28—C29	120.0 (2)
C11—C10—H10	119.9	C27—C28—H28	120.0
C15—C10—H10	119.9	C29—C28—H28	120.0
C10—C11—C12	122.58 (18)	C28—C29—C30	120.3 (3)
C10—C11—H11	118.7	C28—C29—H29	119.9
C12—C11—H11	118.7	C30—C29—H29	119.9
C13—C12—C11	118.85 (18)	C29—C30—C25	120.6 (3)
C13—C12—H12	120.6	C29—C30—H30	119.7
C11—C12—H12	120.6	C25—C30—H30	119.7
C12—C13—C14	118.45 (16)	N1—C31—H31A	109.5
C12—C13—C1	132.26 (16)	N1—C31—H31B	109.5
C14—C13—C1	109.23 (14)	H31A—C31—H31B	109.5
C15—C14—C6	123.07 (17)	N1—C31—H31C	109.5
C15—C14—C13	123.62 (16)	H31A—C31—H31C	109.5
C6—C14—C13	113.28 (16)	H31B—C31—H31C	109.5
C14—C15—C10	116.31 (17)	C4—N1—C31	116.72 (15)
C14—C15—C9	115.80 (19)	C4—N1—C1	108.78 (13)
C10—C15—C9	127.87 (19)	C31—N1—C1	116.55 (15)
O2—C16—C2	113.12 (14)	C22—O2—C16	114.83 (13)
O2—C16—H16A	109.0	C19—O4—C24	117.59 (17)
C2—C16—H16A	109.0		
N1—C1—C2—C17	118.59 (14)	C11—C10—C15—C9	−177.2 (2)
C13—C1—C2—C17	−3.43 (19)	C8—C9—C15—C14	0.5 (3)
C5—C1—C2—C17	−119.72 (15)	C8—C9—C15—C10	178.6 (2)
N1—C1—C2—C16	−122.79 (14)	C17—C2—C16—O2	60.91 (17)
C13—C1—C2—C16	115.19 (15)	C3—C2—C16—O2	−176.41 (13)
C5—C1—C2—C16	−1.10 (19)	C1—C2—C16—O2	−59.83 (18)
N1—C1—C2—C3	−1.42 (15)	C16—C2—C17—O3	139.69 (16)
C13—C1—C2—C3	−123.44 (15)	C3—C2—C17—O3	17.5 (2)
C5—C1—C2—C3	120.27 (14)	C1—C2—C17—O3	−96.72 (18)
C17—C2—C3—C25	87.35 (18)	C16—C2—C17—C23	−41.67 (18)
C16—C2—C3—C25	−31.5 (2)	C3—C2—C17—C23	−163.86 (13)
C1—C2—C3—C25	−154.56 (15)	C1—C2—C17—C23	81.92 (16)
C17—C2—C3—C4	−142.70 (14)	C23—C18—C19—O4	−176.67 (17)
C16—C2—C3—C4	98.42 (15)	C23—C18—C19—C20	2.2 (3)
C1—C2—C3—C4	−24.60 (16)	O4—C19—C20—C21	177.81 (19)
C25—C3—C4—N1	172.33 (15)	C18—C19—C20—C21	−1.2 (3)
C2—C3—C4—N1	42.21 (17)	C19—C20—C21—C22	−0.9 (3)

N1—C1—C5—O1	49.1 (2)	C20—C21—C22—O2	−177.58 (17)
C13—C1—C5—O1	168.97 (16)	C20—C21—C22—C23	1.9 (3)
C2—C1—C5—O1	−66.5 (2)	O2—C22—C23—C18	178.58 (15)
N1—C1—C5—C6	−125.49 (15)	C21—C22—C23—C18	−0.9 (2)
C13—C1—C5—C6	−5.63 (16)	O2—C22—C23—C17	2.1 (2)
C2—C1—C5—C6	118.92 (14)	C21—C22—C23—C17	−177.41 (16)
O1—C5—C6—C7	7.5 (3)	C19—C18—C23—C22	−1.2 (3)
C1—C5—C6—C7	−178.13 (18)	C19—C18—C23—C17	175.33 (15)
O1—C5—C6—C14	−169.53 (17)	O3—C17—C23—C22	−168.08 (16)
C1—C5—C6—C14	4.81 (17)	C2—C17—C23—C22	13.3 (2)
C14—C6—C7—C8	−0.1 (3)	O3—C17—C23—C18	15.4 (2)
C5—C6—C7—C8	−176.89 (19)	C2—C17—C23—C18	−163.22 (14)
C6—C7—C8—C9	1.7 (3)	C4—C3—C25—C26	−30.5 (3)
C7—C8—C9—C15	−1.9 (3)	C2—C3—C25—C26	92.9 (2)
C15—C10—C11—C12	−0.9 (3)	C4—C3—C25—C30	147.63 (19)
C10—C11—C12—C13	−1.0 (3)	C2—C3—C25—C30	−89.0 (2)
C11—C12—C13—C14	2.6 (2)	C30—C25—C26—C27	−1.6 (3)
C11—C12—C13—C1	179.43 (16)	C3—C25—C26—C27	176.5 (2)
N1—C1—C13—C12	−51.5 (2)	C25—C26—C27—C28	−0.3 (4)
C5—C1—C13—C12	−172.49 (17)	C26—C27—C28—C29	2.2 (4)
C2—C1—C13—C12	65.9 (2)	C27—C28—C29—C30	−2.1 (4)
N1—C1—C13—C14	125.54 (14)	C28—C29—C30—C25	0.2 (4)
C5—C1—C13—C14	4.52 (16)	C26—C25—C30—C29	1.6 (3)
C2—C1—C13—C14	−117.10 (15)	C3—C25—C30—C29	−176.5 (2)
C7—C6—C14—C15	−1.3 (3)	C3—C4—N1—C31	179.88 (16)
C5—C6—C14—C15	176.24 (15)	C3—C4—N1—C1	−45.72 (17)
C7—C6—C14—C13	−179.48 (16)	C13—C1—N1—C4	154.45 (14)
C5—C6—C14—C13	−1.97 (19)	C5—C1—N1—C4	−91.30 (16)
C12—C13—C14—C15	−2.6 (2)	C2—C1—N1—C4	29.42 (17)
C1—C13—C14—C15	179.92 (15)	C13—C1—N1—C31	−71.06 (19)
C12—C13—C14—C6	175.61 (15)	C5—C1—N1—C31	43.2 (2)
C1—C13—C14—C6	−1.88 (19)	C2—C1—N1—C31	163.90 (15)
C6—C14—C15—C10	−177.29 (16)	C23—C22—O2—C16	16.0 (2)
C13—C14—C15—C10	0.7 (3)	C21—C22—O2—C16	−164.50 (15)
C6—C14—C15—C9	1.1 (3)	C2—C16—O2—C22	−49.12 (18)
C13—C14—C15—C9	179.11 (16)	C18—C19—O4—C24	−3.1 (3)
C11—C10—C15—C14	1.0 (3)	C20—C19—O4—C24	178.0 (2)

Fig. 1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