

3'-Benzoyl-1'-methyl-4'-phenylspiro[acenaphthylene-1(2H),2'-pyrrolidin]-2-one

T. Augustine,^{a*} Scholastica Mary Vithiya,^b
S. Ignacimuthu^c and V. Ramkumar^d

^aDepartment of Chemistry, D. G. Vaishnav College, Chennai 106, Tamil Nadu, India, ^bDepartment of Chemistry, Auxilium College, Vellore, Tamil Nadu, India, ^cEntomology Research Institute, Loyola College, Chennai 34, Tamil Nadu, India, and ^dDepartment of Chemistry, Indian Institute of Technology Madras, Chennai 36, Tamil Nadu, India

Correspondence e-mail: augustineap@gmail.com

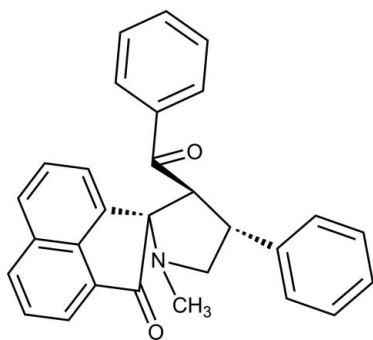
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.052; wR factor = 0.142; data-to-parameter ratio = 19.1.

In the title compound, $\text{C}_{29}\text{H}_{23}\text{NO}_2$, the pyrrolidine ring adopts a twisted conformation about one of the C–N bonds. The acenaphthone ring (r.m.s. deviation = 0.025 Å) lies almost perpendicular to the pyrrolidine ring [dihedral angle = 88.08 (8°)]. The dihedral angle between the phenyl rings is 88.12 (11°). In the crystal structure, weak C–H $\cdots\pi$ interactions connect the molecules.

Related literature

For background on 1,3-dipolar cycloadditions, see: Grigg (1987); Huisgen (1988); Bridges *et al.* (1993); Padwa (1984). For a related structure, see: Augustine *et al.* (2007). For ring conformation analysis, see: Cremer & Pople (1975); Rao *et al.* (1981).



Experimental

Crystal data

$\text{C}_{29}\text{H}_{23}\text{NO}_2$
 $M_r = 417.48$
Monoclinic, $P2_1/c$
 $a = 8.6462$ (4) Å
 $b = 15.8352$ (8) Å
 $c = 16.7174$ (8) Å
 $\beta = 99.827$ (2°)
 $V = 2255.27$ (19) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 298$ K
 $0.42 \times 0.34 \times 0.22$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2004)
 $T_{\min} = 0.969$, $T_{\max} = 0.983$
16782 measured reflections
5549 independent reflections
2944 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.142$
 $S = 1.02$
5549 reflections
290 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.18$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg5 is the centroid of the C17–C22 ring.

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|-------|-------------|-------------|---------------|
| $\text{C3}-\text{H3}\cdots\text{Cg5}^i$ | 0.93 | 2.85 | 3.638 (2) | 144 |

Symmetry code: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$.

Data collection: APEX2 (Bruker, 2004); cell refinement: APEX2 and SAINT (Bruker, 2004); data reduction: SAINT and XPREP (Bruker, 2004); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

The authors acknowledge the Department of Chemistry, IIT Madras, for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5706).

References

- Augustine, T., Ramkumar, V., Arul Antony, S. & Kanakam, C. C. (2007). *Acta Cryst.* **E63**, o4412.
Bridges, R. J., Lovering, F. E., Humphrey, J. M., Stanley, M. S., Blakely, T. N., Cristofaro, M. F. & Chamberlin, A. R. (1993). *Bioorg. Med. Chem. Lett.* **3**, 115–121.
Bruker (2004). SADABS, APEX2 and SAINT-Plus. Bruker AXS Inc., Madison, Wisconsin, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Grigg, G. (1987). *Chem. Soc. Rev.* **16**, 89–121.
Huisgen, R. (1988). *Adv. Cycloaddition*, **1**, 1–31.
Padwa, A. (1984). *1,3-Dipolar Addition Chemistry*. New York: John Wiley.
Rao, S. T., Westhof, E. & Sundaralingam, M. (1981). *Acta Cryst.* **A37**, 421–425.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

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3'-Benzoyl-1'-methyl-4'-phenylspiro[acenaphthylene-1(2*H*),2'-pyrrolidin]-2-one

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Comment

1,3-dipolar cycloadditions using azomethine ylides is conceptually the most simple and efficient method for the construction of saturated, nitrogen containing five-membered heterocycles (Padwa, 1984). Ylide generation, often performed *in situ* (Grigg, 1987), followed by cycloaddition with suitable dipolarophiles furnishes pyrrolidines and pyrroles in only one step from simple starting materials (Huisgen, 1988). 1,3-dipolar cycloadditions of azomethine ylides with olefinic and acetylenic dipolarophiles represent an important approach for the formation of pyrrolidines and pyrrolizines which are prevalent in a variety of biologically active compounds (Bridges *et al.*, 1993). In view of this we have determined the structure of the title compound.

In the title compound C₂₉H₂₃NO₂, the C—O bond distance (1.21 Å) of the carbonyl group in the benzoyl moiety indicates n-p overlap. The bond angles and dihedral angle of C9—C12—C1 (101.69 Å) of the acenaphthone ring indicate it to be in a plane nearly perpendicular to the pyrrolidine ring. The sum of the angles around N-atom of the pyrrolidine ring accounts for 338.78°. This indicates that the structure approaches pyramidal shape. The study of torsion angle, asymmetry parameters and least-square plane calculation shows that the pyrrolidine ring adopts a envelope conformation and puckered, Q₂ = 0.4030 (18) Å, φ = 333.0 (3)° (Cremer & Pople, 1975). The Pseudorotation parameter P and τ are 136.4 (1)° and 43.71 (1)° respectively (Rao *et al.*, 1981) showing that C15 and N1 are twisted and puckered.

The crystal structure is stabilized by weak C—H···π interactions.

Experimental

A mixture of chalcone [1,3-diphenyl-2-propen-1-one] (0.40 g, 2 mmol), acenaphthenequinone (0.36 g, 2 mmol), sarcosine (0.17 g, 2 mmol) and methanol (25 ml) was heated for four hours using oil bath using a dimmerstat at a temperature of 40° C. The reaction mixture was cooled to room temperature and poured into ice-cold water. The solid mass obtained was filtered, washed with water, dried and colourless blocks of (I) were obtained by recrystallization using acetone as solvent by slow evaporation method.

Refinement

H atoms were positioned geometrically and refined using riding model, with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic C—H, C—H = 0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for CH₂, C—H = 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{iso}}(\text{C})$ for CH₃.

Figures

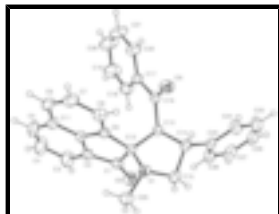


Fig. 1. View of (I) with atoms represented as 30% probability ellipsoids.

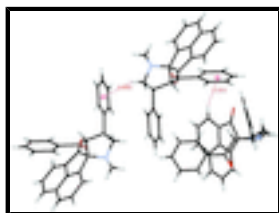


Fig. 2. Packing diagram showing the C—H... π interactions.

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Crystal data

$C_{29}H_{23}NO_2$

$M_r = 417.48$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.6462$ (4) Å

$b = 15.8352$ (8) Å

$c = 16.7174$ (8) Å

$\beta = 99.827$ (2)°

$V = 2255.27$ (19) Å³

$Z = 4$

$F(000) = 880$

$D_x = 1.230$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3350 reflections

$\theta = 2.5$ – 25.3 °

$\mu = 0.08$ mm⁻¹

$T = 298$ K

Block, colourless

$0.42 \times 0.34 \times 0.22$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2004)

$T_{\min} = 0.969$, $T_{\max} = 0.983$

16782 measured reflections

5549 independent reflections

2944 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\text{max}} = 28.3$ °, $\theta_{\text{min}} = 1.8$ °

$h = -9 \rightarrow 11$

$k = -18 \rightarrow 20$

$l = -22 \rightarrow 22$

Refinement

Refinement on F^2

Least-squares matrix: full

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

| | |
|---------------------------------|--|
| $R[F^2 > 2\sigma(F^2)] = 0.052$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.142$ | H-atom parameters constrained |
| $S = 1.01$ | $w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 0.296P]$ |
| 5549 reflections | where $P = (F_o^2 + 2F_c^2)/3$ |
| 290 parameters | $(\Delta/\sigma)_{\max} < 0.001$ |
| 0 restraints | $\Delta\rho_{\max} = 0.18 \text{ e } \text{\AA}^{-3}$ |
| | $\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$ |

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|--------------|----------------------------------|
| C1 | 0.2530 (2) | 0.32056 (11) | 0.28710 (10) | 0.0505 (5) |
| C2 | 0.3044 (2) | 0.27558 (11) | 0.21910 (10) | 0.0523 (5) |
| C3 | 0.4124 (3) | 0.21283 (14) | 0.21511 (14) | 0.0848 (7) |
| H3 | 0.4681 | 0.1887 | 0.2621 | 0.102* |
| C4 | 0.4365 (4) | 0.18600 (16) | 0.13784 (16) | 0.1013 (9) |
| H4 | 0.5112 | 0.1445 | 0.1344 | 0.122* |
| C5 | 0.3546 (3) | 0.21865 (15) | 0.06826 (14) | 0.0845 (7) |
| H5 | 0.3733 | 0.1985 | 0.0185 | 0.101* |
| C6 | 0.1491 (3) | 0.32230 (14) | 0.00346 (11) | 0.0643 (6) |
| H6 | 0.1588 | 0.3072 | -0.0492 | 0.077* |
| C7 | 0.0454 (2) | 0.38280 (14) | 0.01609 (11) | 0.0631 (6) |
| H7 | -0.0157 | 0.4080 | -0.0288 | 0.076* |
| C8 | 0.0257 (2) | 0.40942 (12) | 0.09475 (11) | 0.0549 (5) |
| H8 | -0.0478 | 0.4506 | 0.1012 | 0.066* |
| C9 | 0.11632 (19) | 0.37362 (10) | 0.16061 (9) | 0.0413 (4) |
| C10 | 0.2211 (2) | 0.31009 (10) | 0.14725 (9) | 0.0430 (4) |
| C11 | 0.2423 (2) | 0.28227 (12) | 0.06998 (10) | 0.0555 (5) |
| C12 | 0.13015 (19) | 0.38914 (10) | 0.25123 (9) | 0.0414 (4) |
| C13 | 0.19337 (19) | 0.47786 (10) | 0.28373 (9) | 0.0402 (4) |
| H13 | 0.2961 | 0.4699 | 0.3181 | 0.048* |
| C14 | 0.0763 (2) | 0.50907 (11) | 0.33801 (10) | 0.0497 (5) |
| H14 | -0.0009 | 0.5458 | 0.3051 | 0.060* |
| C15 | -0.0067 (2) | 0.42866 (13) | 0.35599 (12) | 0.0637 (6) |
| H15A | 0.0552 | 0.3972 | 0.4000 | 0.076* |

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|------|---------------|--------------|--------------|------------|
| H15B | -0.1090 | 0.4407 | 0.3697 | 0.076* |
| C16 | 0.2127 (2) | 0.53926 (11) | 0.21632 (10) | 0.0447 (4) |
| C17 | 0.35855 (19) | 0.53647 (11) | 0.17984 (9) | 0.0437 (4) |
| C18 | 0.4852 (2) | 0.48560 (13) | 0.20867 (12) | 0.0612 (5) |
| H18 | 0.4812 | 0.4498 | 0.2524 | 0.073* |
| C19 | 0.6179 (3) | 0.48749 (17) | 0.17315 (16) | 0.0906 (8) |
| H19 | 0.7031 | 0.4531 | 0.1929 | 0.109* |
| C20 | 0.6240 (3) | 0.53984 (19) | 0.10893 (17) | 0.1026 (9) |
| H20 | 0.7140 | 0.5412 | 0.0854 | 0.123* |
| C21 | 0.5004 (3) | 0.58984 (18) | 0.07900 (14) | 0.0889 (8) |
| H21 | 0.5055 | 0.6248 | 0.0348 | 0.107* |
| C22 | 0.3676 (2) | 0.58877 (13) | 0.11399 (11) | 0.0625 (5) |
| H22 | 0.2831 | 0.6233 | 0.0934 | 0.075* |
| C23 | 0.1560 (2) | 0.55919 (11) | 0.41019 (11) | 0.0495 (5) |
| C24 | 0.1259 (3) | 0.64403 (13) | 0.41745 (13) | 0.0690 (6) |
| H24 | 0.0502 | 0.6700 | 0.3791 | 0.083* |
| C25 | 0.2064 (3) | 0.69126 (16) | 0.48084 (18) | 0.0879 (8) |
| H25 | 0.1839 | 0.7484 | 0.4845 | 0.105* |
| C26 | 0.3172 (3) | 0.65544 (18) | 0.53761 (16) | 0.0827 (7) |
| H26 | 0.3726 | 0.6878 | 0.5794 | 0.099* |
| C27 | 0.3463 (3) | 0.57130 (17) | 0.53253 (13) | 0.0767 (7) |
| H27 | 0.4204 | 0.5456 | 0.5719 | 0.092* |
| C28 | 0.2670 (2) | 0.52382 (14) | 0.46946 (12) | 0.0649 (6) |
| H28 | 0.2890 | 0.4665 | 0.4669 | 0.078* |
| C29 | -0.0869 (3) | 0.29814 (14) | 0.28011 (14) | 0.0825 (7) |
| H29A | -0.0220 | 0.2644 | 0.3202 | 0.124* |
| H29B | -0.0912 | 0.2728 | 0.2276 | 0.124* |
| H29C | -0.1908 | 0.3015 | 0.2928 | 0.124* |
| N1 | -0.02068 (18) | 0.38299 (10) | 0.27962 (9) | 0.0565 (4) |
| O2 | 0.29920 (19) | 0.30958 (9) | 0.35906 (7) | 0.0752 (4) |
| O3 | 0.10953 (17) | 0.58979 (9) | 0.19095 (8) | 0.0704 (4) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C1 | 0.0700 (12) | 0.0418 (10) | 0.0407 (10) | 0.0002 (9) | 0.0121 (8) | 0.0050 (8) |
| C2 | 0.0703 (12) | 0.0418 (10) | 0.0463 (10) | 0.0078 (10) | 0.0138 (9) | 0.0029 (8) |
| C3 | 0.1170 (19) | 0.0658 (15) | 0.0715 (14) | 0.0422 (14) | 0.0160 (13) | 0.0069 (12) |
| C4 | 0.135 (2) | 0.0850 (18) | 0.0903 (19) | 0.0531 (17) | 0.0388 (17) | -0.0077 (15) |
| C5 | 0.1157 (19) | 0.0786 (16) | 0.0668 (15) | 0.0179 (15) | 0.0375 (14) | -0.0189 (13) |
| C6 | 0.0806 (15) | 0.0741 (15) | 0.0382 (10) | -0.0217 (12) | 0.0106 (10) | -0.0067 (10) |
| C7 | 0.0625 (13) | 0.0805 (15) | 0.0414 (10) | -0.0169 (12) | -0.0050 (9) | 0.0092 (10) |
| C8 | 0.0464 (10) | 0.0618 (12) | 0.0555 (11) | -0.0014 (9) | 0.0062 (8) | 0.0051 (9) |
| C9 | 0.0441 (9) | 0.0410 (10) | 0.0398 (9) | -0.0050 (8) | 0.0098 (7) | 0.0027 (7) |
| C10 | 0.0528 (10) | 0.0395 (10) | 0.0386 (9) | -0.0040 (8) | 0.0129 (7) | -0.0022 (7) |
| C11 | 0.0707 (12) | 0.0539 (12) | 0.0455 (10) | -0.0071 (10) | 0.0198 (9) | -0.0088 (9) |
| C12 | 0.0501 (10) | 0.0378 (9) | 0.0396 (8) | 0.0014 (8) | 0.0175 (7) | 0.0017 (7) |
| C13 | 0.0441 (9) | 0.0386 (9) | 0.0415 (8) | 0.0031 (7) | 0.0172 (7) | 0.0002 (7) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C14 | 0.0521 (10) | 0.0487 (11) | 0.0541 (10) | 0.0063 (9) | 0.0252 (8) | -0.0007 (8) |
| C15 | 0.0715 (13) | 0.0652 (13) | 0.0648 (12) | -0.0122 (11) | 0.0412 (10) | -0.0122 (10) |
| C16 | 0.0509 (10) | 0.0371 (10) | 0.0484 (10) | 0.0014 (9) | 0.0153 (8) | -0.0006 (8) |
| C17 | 0.0460 (10) | 0.0441 (10) | 0.0435 (9) | -0.0115 (8) | 0.0149 (7) | -0.0033 (8) |
| C18 | 0.0554 (12) | 0.0624 (13) | 0.0707 (12) | 0.0027 (10) | 0.0244 (10) | 0.0129 (10) |
| C19 | 0.0621 (14) | 0.1001 (19) | 0.119 (2) | 0.0144 (13) | 0.0434 (14) | 0.0281 (16) |
| C20 | 0.0779 (17) | 0.128 (2) | 0.118 (2) | 0.0117 (18) | 0.0625 (16) | 0.0354 (19) |
| C21 | 0.0830 (17) | 0.117 (2) | 0.0747 (15) | -0.0113 (16) | 0.0368 (13) | 0.0311 (15) |
| C22 | 0.0587 (12) | 0.0762 (14) | 0.0537 (11) | -0.0109 (11) | 0.0126 (9) | 0.0105 (10) |
| C23 | 0.0562 (11) | 0.0480 (11) | 0.0510 (10) | 0.0038 (9) | 0.0284 (9) | -0.0048 (8) |
| C24 | 0.0813 (15) | 0.0529 (13) | 0.0771 (14) | 0.0102 (11) | 0.0261 (11) | -0.0052 (11) |
| C25 | 0.106 (2) | 0.0577 (15) | 0.109 (2) | -0.0035 (14) | 0.0435 (17) | -0.0292 (15) |
| C26 | 0.0799 (17) | 0.096 (2) | 0.0821 (16) | -0.0169 (15) | 0.0407 (14) | -0.0399 (15) |
| C27 | 0.0698 (14) | 0.106 (2) | 0.0576 (13) | 0.0073 (13) | 0.0194 (11) | -0.0129 (13) |
| C28 | 0.0769 (14) | 0.0605 (13) | 0.0610 (12) | 0.0120 (11) | 0.0228 (11) | -0.0084 (10) |
| C29 | 0.0991 (17) | 0.0744 (16) | 0.0854 (16) | -0.0376 (14) | 0.0483 (13) | -0.0165 (12) |
| N1 | 0.0604 (10) | 0.0557 (10) | 0.0611 (10) | -0.0151 (8) | 0.0322 (8) | -0.0083 (7) |
| O2 | 0.1194 (12) | 0.0654 (9) | 0.0392 (7) | 0.0163 (8) | 0.0087 (7) | 0.0095 (6) |
| O3 | 0.0751 (9) | 0.0600 (9) | 0.0827 (10) | 0.0249 (8) | 0.0324 (7) | 0.0237 (8) |

Geometric parameters (Å, °)

| | | | |
|---------|-------------|----------|-----------|
| C1—O2 | 1.2136 (19) | C15—H15A | 0.9700 |
| C1—C2 | 1.473 (2) | C15—H15B | 0.9700 |
| C1—C12 | 1.565 (2) | C16—O3 | 1.219 (2) |
| C2—C3 | 1.373 (3) | C16—C17 | 1.492 (2) |
| C2—C10 | 1.402 (2) | C17—C18 | 1.379 (3) |
| C3—C4 | 1.409 (3) | C17—C22 | 1.390 (2) |
| C3—H3 | 0.9300 | C18—C19 | 1.379 (3) |
| C4—C5 | 1.357 (3) | C18—H18 | 0.9300 |
| C4—H4 | 0.9300 | C19—C20 | 1.364 (3) |
| C5—C11 | 1.403 (3) | C19—H19 | 0.9300 |
| C5—H5 | 0.9300 | C20—C21 | 1.355 (3) |
| C6—C7 | 1.353 (3) | C20—H20 | 0.9300 |
| C6—C11 | 1.408 (3) | C21—C22 | 1.375 (3) |
| C6—H6 | 0.9300 | C21—H21 | 0.9300 |
| C7—C8 | 1.418 (3) | C22—H22 | 0.9300 |
| C7—H7 | 0.9300 | C23—C28 | 1.375 (3) |
| C8—C9 | 1.361 (2) | C23—C24 | 1.378 (3) |
| C8—H8 | 0.9300 | C24—C25 | 1.384 (3) |
| C9—C10 | 1.397 (2) | C24—H24 | 0.9300 |
| C9—C12 | 1.519 (2) | C25—C26 | 1.353 (3) |
| C10—C11 | 1.406 (2) | C25—H25 | 0.9300 |
| C12—N1 | 1.465 (2) | C26—C27 | 1.361 (3) |
| C12—C13 | 1.571 (2) | C26—H26 | 0.9300 |
| C13—C16 | 1.519 (2) | C27—C28 | 1.379 (3) |
| C13—C14 | 1.551 (2) | C27—H27 | 0.9300 |
| C13—H13 | 0.9800 | C28—H28 | 0.9300 |
| C14—C23 | 1.509 (2) | C29—N1 | 1.461 (2) |

supplementary materials

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|-------------|-------------|---------------|-------------|
| C14—C15 | 1.516 (3) | C29—H29A | 0.9600 |
| C14—H14 | 0.9800 | C29—H29B | 0.9600 |
| C15—N1 | 1.454 (2) | C29—H29C | 0.9600 |
| O2—C1—C2 | 127.16 (17) | C14—C15—H15A | 111.3 |
| O2—C1—C12 | 124.49 (16) | N1—C15—H15B | 111.3 |
| C2—C1—C12 | 108.31 (14) | C14—C15—H15B | 111.3 |
| C3—C2—C10 | 119.66 (17) | H15A—C15—H15B | 109.2 |
| C3—C2—C1 | 133.22 (18) | O3—C16—C17 | 119.76 (15) |
| C10—C2—C1 | 107.11 (15) | O3—C16—C13 | 120.78 (15) |
| C2—C3—C4 | 118.1 (2) | C17—C16—C13 | 119.45 (15) |
| C2—C3—H3 | 120.9 | C18—C17—C22 | 118.44 (17) |
| C4—C3—H3 | 120.9 | C18—C17—C16 | 123.43 (15) |
| C5—C4—C3 | 122.3 (2) | C22—C17—C16 | 118.12 (16) |
| C5—C4—H4 | 118.9 | C17—C18—C19 | 120.42 (19) |
| C3—C4—H4 | 118.9 | C17—C18—H18 | 119.8 |
| C4—C5—C11 | 121.20 (19) | C19—C18—H18 | 119.8 |
| C4—C5—H5 | 119.4 | C20—C19—C18 | 119.9 (2) |
| C11—C5—H5 | 119.4 | C20—C19—H19 | 120.0 |
| C7—C6—C11 | 120.06 (17) | C18—C19—H19 | 120.0 |
| C7—C6—H6 | 120.0 | C21—C20—C19 | 120.8 (2) |
| C11—C6—H6 | 120.0 | C21—C20—H20 | 119.6 |
| C6—C7—C8 | 122.84 (18) | C19—C20—H20 | 119.6 |
| C6—C7—H7 | 118.6 | C20—C21—C22 | 119.9 (2) |
| C8—C7—H7 | 118.6 | C20—C21—H21 | 120.0 |
| C9—C8—C7 | 118.87 (18) | C22—C21—H21 | 120.0 |
| C9—C8—H8 | 120.6 | C21—C22—C17 | 120.5 (2) |
| C7—C8—H8 | 120.6 | C21—C22—H22 | 119.7 |
| C8—C9—C10 | 118.02 (15) | C17—C22—H22 | 119.7 |
| C8—C9—C12 | 132.51 (16) | C28—C23—C24 | 116.91 (19) |
| C10—C9—C12 | 109.46 (13) | C28—C23—C14 | 121.96 (17) |
| C9—C10—C2 | 113.33 (14) | C24—C23—C14 | 121.06 (18) |
| C9—C10—C11 | 124.21 (16) | C23—C24—C25 | 121.1 (2) |
| C2—C10—C11 | 122.45 (16) | C23—C24—H24 | 119.4 |
| C5—C11—C10 | 116.29 (18) | C25—C24—H24 | 119.4 |
| C5—C11—C6 | 127.74 (18) | C26—C25—C24 | 120.9 (2) |
| C10—C11—C6 | 115.96 (18) | C26—C25—H25 | 119.5 |
| N1—C12—C9 | 112.92 (13) | C24—C25—H25 | 119.5 |
| N1—C12—C1 | 114.40 (13) | C25—C26—C27 | 118.9 (2) |
| C9—C12—C1 | 101.69 (12) | C25—C26—H26 | 120.6 |
| N1—C12—C13 | 102.93 (12) | C27—C26—H26 | 120.6 |
| C9—C12—C13 | 116.96 (13) | C26—C27—C28 | 120.5 (2) |
| C1—C12—C13 | 108.34 (13) | C26—C27—H27 | 119.7 |
| C16—C13—C14 | 113.29 (13) | C28—C27—H27 | 119.7 |
| C16—C13—C12 | 113.06 (13) | C23—C28—C27 | 121.6 (2) |
| C14—C13—C12 | 105.31 (13) | C23—C28—H28 | 119.2 |
| C16—C13—H13 | 108.3 | C27—C28—H28 | 119.2 |
| C14—C13—H13 | 108.3 | N1—C29—H29A | 109.5 |
| C12—C13—H13 | 108.3 | N1—C29—H29B | 109.5 |
| C23—C14—C15 | 116.74 (15) | H29A—C29—H29B | 109.5 |

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|----------------|--------------|-----------------|--------------|
| C23—C14—C13 | 112.37 (14) | N1—C29—H29C | 109.5 |
| C15—C14—C13 | 103.08 (14) | H29A—C29—H29C | 109.5 |
| C23—C14—H14 | 108.1 | H29B—C29—H29C | 109.5 |
| C15—C14—H14 | 108.1 | C15—N1—C29 | 115.24 (15) |
| C13—C14—H14 | 108.1 | C15—N1—C12 | 107.82 (14) |
| N1—C15—C14 | 102.41 (14) | C29—N1—C12 | 115.72 (15) |
| N1—C15—H15A | 111.3 | | |
| O2—C1—C2—C3 | -0.6 (4) | C9—C12—C13—C14 | 131.26 (14) |
| C12—C1—C2—C3 | 177.3 (2) | C1—C12—C13—C14 | -114.66 (14) |
| O2—C1—C2—C10 | -179.13 (18) | C16—C13—C14—C23 | -90.89 (18) |
| C12—C1—C2—C10 | -1.20 (19) | C12—C13—C14—C23 | 145.06 (14) |
| C10—C2—C3—C4 | 0.7 (3) | C16—C13—C14—C15 | 142.59 (16) |
| C1—C2—C3—C4 | -177.7 (2) | C12—C13—C14—C15 | 18.54 (17) |
| C2—C3—C4—C5 | -1.5 (4) | C23—C14—C15—N1 | -161.09 (15) |
| C3—C4—C5—C11 | 0.9 (4) | C13—C14—C15—N1 | -37.41 (19) |
| C11—C6—C7—C8 | 0.6 (3) | C14—C13—C16—O3 | -24.2 (2) |
| C6—C7—C8—C9 | 0.9 (3) | C12—C13—C16—O3 | 95.49 (18) |
| C7—C8—C9—C10 | -2.1 (2) | C14—C13—C16—C17 | 156.88 (14) |
| C7—C8—C9—C12 | 177.34 (17) | C12—C13—C16—C17 | -83.41 (18) |
| C8—C9—C10—C2 | -177.88 (16) | O3—C16—C17—C18 | 175.23 (18) |
| C12—C9—C10—C2 | 2.6 (2) | C13—C16—C17—C18 | -5.9 (2) |
| C8—C9—C10—C11 | 1.9 (3) | O3—C16—C17—C22 | -3.9 (2) |
| C12—C9—C10—C11 | -177.67 (16) | C13—C16—C17—C22 | 174.97 (16) |
| C3—C2—C10—C9 | -179.55 (18) | C22—C17—C18—C19 | 0.6 (3) |
| C1—C2—C10—C9 | -0.8 (2) | C16—C17—C18—C19 | -178.6 (2) |
| C3—C2—C10—C11 | 0.7 (3) | C17—C18—C19—C20 | -0.1 (4) |
| C1—C2—C10—C11 | 179.43 (16) | C18—C19—C20—C21 | -0.5 (4) |
| C4—C5—C11—C10 | 0.5 (3) | C19—C20—C21—C22 | 0.7 (4) |
| C4—C5—C11—C6 | 179.7 (2) | C20—C21—C22—C17 | -0.2 (4) |
| C9—C10—C11—C5 | 179.02 (18) | C18—C17—C22—C21 | -0.4 (3) |
| C2—C10—C11—C5 | -1.3 (3) | C16—C17—C22—C21 | 178.81 (19) |
| C9—C10—C11—C6 | -0.4 (3) | C15—C14—C23—C28 | 55.7 (2) |
| C2—C10—C11—C6 | 179.36 (17) | C13—C14—C23—C28 | -63.1 (2) |
| C7—C6—C11—C5 | 179.8 (2) | C15—C14—C23—C24 | -127.57 (19) |
| C7—C6—C11—C10 | -0.9 (3) | C13—C14—C23—C24 | 113.66 (18) |
| C8—C9—C12—N1 | 54.5 (2) | C28—C23—C24—C25 | 1.2 (3) |
| C10—C9—C12—N1 | -126.08 (15) | C14—C23—C24—C25 | -175.76 (18) |
| C8—C9—C12—C1 | 177.53 (18) | C23—C24—C25—C26 | 0.0 (3) |
| C10—C9—C12—C1 | -3.02 (17) | C24—C25—C26—C27 | -1.4 (4) |
| C8—C9—C12—C13 | -64.7 (2) | C25—C26—C27—C28 | 1.6 (3) |
| C10—C9—C12—C13 | 114.74 (16) | C24—C23—C28—C27 | -1.0 (3) |
| O2—C1—C12—N1 | -57.4 (2) | C14—C23—C28—C27 | 175.89 (17) |
| C2—C1—C12—N1 | 124.57 (15) | C26—C27—C28—C23 | -0.4 (3) |
| O2—C1—C12—C9 | -179.48 (18) | C14—C15—N1—C29 | 175.49 (17) |
| C2—C1—C12—C9 | 2.53 (17) | C14—C15—N1—C12 | 44.53 (19) |
| O2—C1—C12—C13 | 56.7 (2) | C9—C12—N1—C15 | -158.77 (15) |
| C2—C1—C12—C13 | -121.27 (15) | C1—C12—N1—C15 | 85.56 (17) |
| N1—C12—C13—C16 | -117.34 (15) | C13—C12—N1—C15 | -31.75 (17) |
| C9—C12—C13—C16 | 7.1 (2) | C9—C12—N1—C29 | 70.5 (2) |

supplementary materials

| | | | |
|----------------|-------------|----------------|--------------|
| C1—C12—C13—C16 | 121.15 (15) | C1—C12—N1—C29 | -45.1 (2) |
| N1—C12—C13—C14 | 6.86 (16) | C13—C12—N1—C29 | -162.45 (16) |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|---------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| C3—H3 \cdots Cg5 ⁱ | 0.93 | 2.85 | 3.638 (2) | 144 |

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

Fig. 1

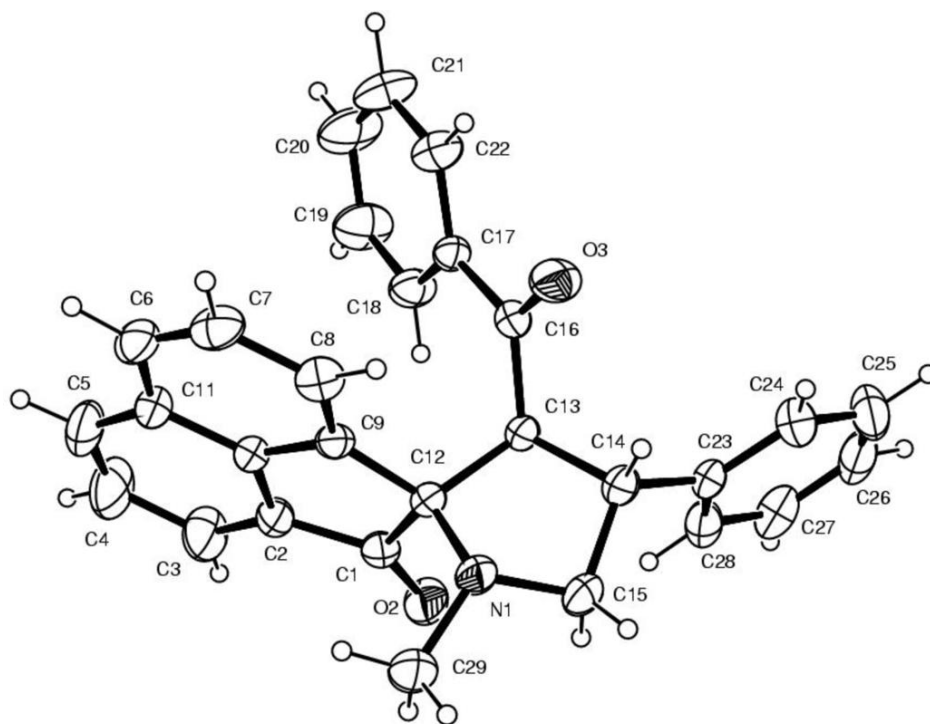


Fig. 2

