

2-Amino-4-(3,4-dimethoxyphenyl)-5,6-dihydrobenzo[*h*]quinoline-3-carbonitrile–3-amino-1-(3,4-dimethoxyphenyl)-9,10-dihydrophenanthrene-2,4-dicarbonitrile (1/19)

Abdullah M. Asiri,^{a,b} Abdulrahman O. Al-Youbi,^a
Hassan M. Faidallah^a and Seik Weng Ng^{c,a*}

^aChemistry Department, Faculty of Science, King Abdulaziz University, PO Box 80203 Jeddah, Saudi Arabia, ^bCenter of Excellence for Advanced Materials Research, King Abdulaziz University, PO Box 80203 Jeddah, Saudi Arabia, and ^cDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

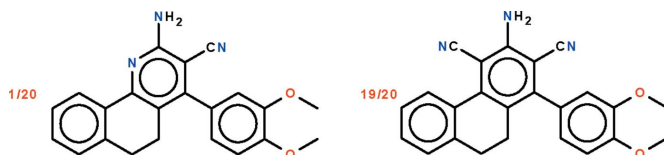
Received 11 September 2011; accepted 3 October 2011

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; disorder in main residue; R factor = 0.049; wR factor = 0.124; data-to-parameter ratio = 15.4.

The asymmetric unit of the 1:19 title co-crystal of 2-amino-4-(3,4-dimethoxyphenyl)-5,6-dihydrobenzo[*h*]quinoline-3-carbonitrile and 3-amino-1-(3,4-dimethoxyphenyl)-9,10-dihydrophenanthrene-2,4-dicarbonitrile, $0.05\text{C}_{22}\text{H}_{19}\text{N}_3\text{O}_2 \cdot 0.95\text{C}_{24}\text{H}_{19}\text{N}_3\text{O}_2$, has the atoms of the fused-ring system and those of the amino, cyano and dimethoxyphenyl substituents overlapped. The fused-ring system is buckled owing to the ethylene linkage in the central ring with the two flanking aromatic rings being twisted by $31.9(1)^\circ$. The ring of the dimethoxyphenyl substituent is twisted by $72.4(1)^\circ$ relative to the amino- and cyano-bearing aromatic ring. In the crystal, molecules are linked by duplex amine $\text{N}-\text{H} \cdots \text{O}(\text{methoxy})$ hydrogen bonds in a cyclic association [graph-set $R_2^2(7)$], generating a helical chain structure extending along [201].

Related literature

For a similar co-crystal, see: Asiri *et al.* (2011). For graph-set analysis, see: Etter *et al.* (1990).



Experimental

Crystal data

$0.05\text{C}_{22}\text{H}_{19}\text{N}_3\text{O}_2 \cdot 0.95\text{C}_{24}\text{H}_{19}\text{N}_3\text{O}_2$
 $M_r = 380.22$
Monoclinic, $P2_1/c$
 $a = 8.9347(3)$ Å
 $b = 14.4915(5)$ Å
 $c = 14.7818(6)$ Å
 $\beta = 103.446(4)^\circ$

$V = 1861.45(12)$ Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 100$ K
 $0.30 \times 0.25 \times 0.20$ mm

Data collection

Agilent SuperNova Dual
diffractometer with an Atlas
detector
Absorption correction: multi-scan
(*CrysAlis PRO*; Agilent, 2010)
 $T_{\min} = 0.974$, $T_{\max} = 0.983$

9240 measured reflections
4160 independent reflections
3146 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.124$
 $S = 1.04$
4160 reflections
270 parameters

H atoms treated by a mixture of
independent and constrained
refinement
 $\Delta\rho_{\max} = 0.30$ e Å⁻³
 $\Delta\rho_{\min} = -0.24$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{N3}-\text{H1} \cdots \text{O1}^i$	0.95 (2)	2.24 (2)	2.927 (2)	129 (2)
$\text{N3}-\text{H2} \cdots \text{O2}^i$	0.92 (2)	2.25 (2)	2.987 (2)	136 (2)

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank King Abdulaziz University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2146).

References

- Agilent (2010). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
Asiri, A. M., Al-Youbi, A. O., Faidallah, H. M. & Ng, S. W. (2011). *Acta Cryst. E* **67**, o2872.
Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst. B* **46**, 256–262.
Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supplementary materials

Acta Cryst. (2011). E67, o2873 [doi:10.1107/S1600536811040517]

2-Amino-4-(3,4-dimethoxyphenyl)-5,6-dihydrobenzo[*h*]quinoline-3-carbonitrile-3-amino-1-(3,4-dimethoxyphenyl)-9,10-dihydrophenanthrene-2,4-dicarbonitrile (1/19)

A. M. Asiri, A. O. Al-Youbi, H. M. Faidallah and S. W. Ng

Comment

2-Amino-5,6-dihydro-4-phenyl-benzoquinoline-3-carbonitrile is synthesized from the reaction of the α -substituted cinnamitrile, $C_6H_5CH=C(CN)_2$, with α -tetralone in a reaction that is catalyzed by ammonium acetate. The synthesis when conducted under microwave irradiation leads to an improved yield. In previous studies, we obtained instead di-carbonitrile substituted dihydrophenanthrenes (3-amino-1-(4-methoxyphenyl)-9,10-dihydrophenanthrene-2,4-dicarbonitrile and 3-amino-1-(2*H*-1,3-benzodioxol-5-yl)-9,10-dihydrophenanthrene-2,4-dicarbonitrile) with 4-methoxybenzaldehyde and piperonaldehyde in syntheses that differed slightly from the reported ones as we used substituted benzaldehydes, α -tetralone and ethyl cyanoacetate along with a molar excess of ammonium acetate.

In this study, the reaction of 3,4-dimethoxybenzaldehyde, α -tetralone and ethyl cyanoacetate yielded the co-crystal of 2-amino-4-(3,4-dimethoxyphenyl)-5,6-dihydrobenzoquinoline-3-carbonitrile ($C_{22}H_{19}N_3O_2$) and 3-amino-1-(3,4-dimethoxyphenyl)-9,10-dihydrophenanthrene-2,4-dicarbonitrile ($C_{24}H_{19}N_3O_2$) with the components present in a 1:19 molar ratio (Scheme I). The fused-ring system is buckled owing to the ethylene linkage in the central ring with the two flanking aromatic rings twisted by 31.9 (1)°. Relative to the amino- and cyano-bearing aromatic ring, the benzene ring is twisted by 72.4 (1)° (Figs. 1 and 2). Molecules are linked by duplex amine N–H...O (methoxy) hydrogen bonds (Table 1) in a cyclic association [graph set $R^2_2(7)$ (Etter *et al.*, 1990)], generating a helical chain structure extending along [2 0 1].

Experimental

A mixture of 3,4-dimethoxybenzaldehyde (1.66 g, 10 mmol), α -tetralone (1.46 g, 10 mmol), ethyl cyanoacetate (1.13 g, 10 mmol) and ammonium acetate (6.16 g, 80 mmol) in absolute ethanol (50 ml) was refluxed for 6 h. The mixture was allowed to cool and the precipitate that formed was filtered, washed with water, dried and recrystallized from DMF.

Refinement

Carbon-bound H-atoms were placed in calculated positions [$C-H = 0.95-0.99$ Å; $U_{iso}(H) 1.2-1.5U_{eq}(C)$] and were included in the refinement in the riding model approximation. The amino H-atoms were located in a difference Fourier map and were refined without restraint, including their temperature isotropic displacement parameters. The compound is a co-crystal of 2-amino-4-(3,4-dimethoxy)-5,6-dihydrobenzoquinoline-3-carbonitrile ($C_{22}H_{19}N_3O_2$) and 3-amino-1-(3,4-dimethoxyphenyl)-9,10-dihydrophenanthrene-2,4-dicarbonitrile ($C_{24}H_{19}N_3O_2$). The first component is a dihydrobenzoquinoline and has only one cyano substituent. The second component is a dihydrophenanthrene with two cyano substituents. The two-coordinate N atom of the first molecule occupies the same site as the three-coordinate C atom of the second molecule. As the occupancy refined to an almost 1:19 ratio, the occupancy was then fixed as this ratio.

Figures

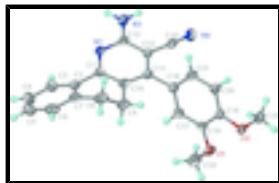


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{22}H_{19}N_3O_2$ (molecule 1) at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

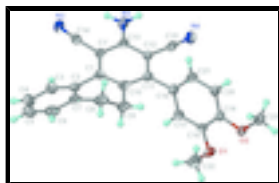


Fig. 2. Thermal ellipsoid plot (Barbour, 2001) of $C_{24}H_{19}N_3O_2$ (molecule 2) at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

2-Amino-4-(3,4-dimethoxyphenyl)-5,6-dihydrobenzo[*h*]quinoline-3- carbonitrile-3-amino-1-(3,4-dimethoxyphenyl)-9,10-dihydrophenanthrene-2,4- dicarbonitrile (1/19)

Crystal data

$0.05C_{22}H_{19}N_3O_2 \cdot 0.95C_{24}H_{19}N_3O_2$

$M_r = 380.22$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.9347$ (3) Å

$b = 14.4915$ (5) Å

$c = 14.7818$ (6) Å

$\beta = 103.446$ (4)°

$V = 1861.45$ (12) Å³

$Z = 4$

$F(000) = 797.6$

$D_x = 1.357$ Mg m⁻³

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

Cell parameters from 3220 reflections

$\theta = 2.3$ – 29.2 °

$\mu = 0.09$ mm⁻¹

$T = 100$ K

Block, orange

$0.30 \times 0.25 \times 0.20$ mm

Data collection

Agilent SuperNova Dual
diffractometer with an Atlas detector

Radiation source: SuperNova (Mo) X-ray Source

Mirror

Detector resolution: 10.4041 pixels mm⁻¹

ω scans

Absorption correction: multi-scan
(*Crys.Alis PRO*; Agilent, 2010)

$T_{\min} = 0.974$, $T_{\max} = 0.983$

9240 measured reflections

4160 independent reflections

3146 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 2.3$ °

$h = -11 \rightarrow 8$

$k = -18 \rightarrow 17$

$l = -19 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

$$R[F^2 > 2\sigma(F^2)] = 0.049$$

$$wR(F^2) = 0.124$$

$$S = 1.04$$

4160 reflections

270 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2 + 0.6446P]$$

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

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.30 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\min} = -0.24 \text{ e } \text{Å}^{-3}$$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.30588 (13)	0.63995 (9)	0.10804 (8)	0.0271 (3)	
O2	0.07648 (12)	0.69798 (8)	0.17421 (8)	0.0220 (3)	
N1	0.94620 (18)	0.95400 (11)	0.33260 (11)	0.0197 (3)	0.05
C1'	0.94620 (18)	0.95400 (11)	0.33260 (11)	0.0197 (3)	0.95
N2	1.21403 (18)	1.02734 (11)	0.39566 (11)	0.0279 (4)	0.95
N3	1.00292 (18)	0.87635 (11)	0.48043 (11)	0.0271 (4)	
H1	1.104 (3)	0.9012 (16)	0.4948 (16)	0.053 (7)*	
H2	0.973 (2)	0.8423 (15)	0.5258 (15)	0.035 (6)*	
N4	0.68515 (17)	0.74165 (11)	0.49326 (11)	0.0294 (4)	
C1	0.84721 (19)	0.97189 (11)	0.24628 (11)	0.0209 (4)	
C2	0.8945 (2)	1.02681 (12)	0.17292 (12)	0.0253 (4)	
C3	1.0457 (2)	1.02878 (13)	0.16227 (13)	0.0315 (4)	
H3	1.1232	0.9947	0.2038	0.038*	
C4	1.0837 (3)	1.07998 (14)	0.09164 (14)	0.0374 (5)	
H4	1.1867	1.0808	0.0847	0.045*	
C5	0.9711 (3)	1.12980 (14)	0.03145 (14)	0.0425 (5)	
H5	0.9976	1.1660	-0.0161	0.051*	
C6	0.8200 (3)	1.12739 (13)	0.03999 (13)	0.0382 (5)	
H6	0.7435	1.1618	-0.0019	0.046*	
C7	0.7794 (2)	1.07502 (12)	0.10951 (12)	0.0295 (4)	
C8	0.6159 (2)	1.06430 (13)	0.11720 (13)	0.0326 (4)	
H8A	0.5449	1.0839	0.0585	0.039*	
H8B	0.5970	1.1035	0.1682	0.039*	
C9	0.5876 (2)	0.96303 (13)	0.13680 (12)	0.0292 (4)	
H9A	0.4801	0.9547	0.1424	0.035*	
H9B	0.6036	0.9242	0.0848	0.035*	
C10	0.69749 (19)	0.93394 (12)	0.22619 (12)	0.0231 (4)	
C11	0.65552 (18)	0.87253 (11)	0.28827 (11)	0.0213 (4)	
C12	0.75689 (18)	0.85343 (11)	0.37374 (11)	0.0193 (3)	
C13	0.90408 (18)	0.89474 (11)	0.39830 (11)	0.0196 (3)	
C14	1.0941 (2)	0.99715 (12)	0.36327 (12)	0.0225 (4)	0.95
C15	0.71436 (18)	0.79079 (12)	0.43878 (12)	0.0224 (4)	
C16	0.50312 (18)	0.82492 (12)	0.26343 (11)	0.0215 (4)	
C17	0.47860 (19)	0.75520 (12)	0.19617 (12)	0.0224 (4)	
H17	0.5599	0.7375	0.1683	0.027*	

supplementary materials

C18	0.33741 (18)	0.71173 (11)	0.16982 (11)	0.0203 (4)
C19	0.21512 (18)	0.74044 (11)	0.20790 (11)	0.0193 (3)
C20	0.2414 (2)	0.80721 (12)	0.27631 (12)	0.0249 (4)
H20	0.1605	0.8251	0.3044	0.030*
C21	0.3855 (2)	0.84870 (13)	0.30454 (12)	0.0262 (4)
H21	0.4026	0.8937	0.3526	0.031*
C22	0.4368 (2)	0.59651 (14)	0.08517 (15)	0.0368 (5)
H22A	0.4020	0.5462	0.0409	0.055*
H22B	0.5043	0.5716	0.1418	0.055*
H22C	0.4934	0.6420	0.0571	0.055*
C23	-0.05031 (19)	0.73108 (12)	0.20937 (13)	0.0260 (4)
H23A	-0.1430	0.6957	0.1814	0.039*
H23B	-0.0678	0.7965	0.1937	0.039*
H23C	-0.0268	0.7236	0.2771	0.039*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0180 (6)	0.0316 (7)	0.0312 (7)	0.0003 (5)	0.0050 (5)	-0.0126 (6)
O2	0.0152 (6)	0.0253 (6)	0.0258 (6)	-0.0021 (5)	0.0051 (5)	-0.0061 (5)
N1	0.0175 (8)	0.0197 (8)	0.0218 (8)	-0.0008 (7)	0.0042 (7)	-0.0027 (7)
C1'	0.0175 (8)	0.0197 (8)	0.0218 (8)	-0.0008 (7)	0.0042 (7)	-0.0027 (7)
N2	0.0214 (8)	0.0270 (8)	0.0354 (9)	-0.0022 (7)	0.0065 (7)	0.0011 (7)
N3	0.0209 (8)	0.0325 (9)	0.0240 (8)	-0.0045 (7)	-0.0028 (6)	0.0063 (7)
N4	0.0257 (8)	0.0309 (8)	0.0315 (8)	-0.0031 (7)	0.0065 (7)	0.0030 (7)
C1	0.0233 (8)	0.0196 (8)	0.0192 (8)	0.0004 (7)	0.0036 (7)	-0.0026 (7)
C2	0.0347 (10)	0.0209 (8)	0.0199 (8)	-0.0056 (8)	0.0056 (8)	-0.0035 (7)
C3	0.0391 (11)	0.0267 (10)	0.0308 (10)	-0.0063 (8)	0.0126 (9)	-0.0034 (8)
C4	0.0523 (13)	0.0317 (10)	0.0342 (11)	-0.0115 (10)	0.0219 (10)	-0.0042 (9)
C5	0.0717 (16)	0.0323 (11)	0.0272 (10)	-0.0162 (11)	0.0190 (11)	-0.0001 (9)
C6	0.0593 (14)	0.0284 (10)	0.0228 (9)	-0.0058 (10)	0.0013 (9)	0.0015 (8)
C7	0.0434 (11)	0.0237 (9)	0.0191 (9)	-0.0057 (8)	0.0026 (8)	-0.0020 (7)
C8	0.0400 (11)	0.0293 (10)	0.0217 (9)	0.0011 (9)	-0.0065 (8)	0.0023 (8)
C9	0.0298 (10)	0.0303 (10)	0.0225 (9)	-0.0031 (8)	-0.0041 (8)	0.0001 (8)
C10	0.0217 (8)	0.0244 (9)	0.0205 (8)	-0.0004 (7)	-0.0006 (7)	-0.0016 (7)
C11	0.0175 (8)	0.0229 (8)	0.0231 (8)	0.0010 (7)	0.0037 (7)	-0.0042 (7)
C12	0.0174 (8)	0.0204 (8)	0.0205 (8)	-0.0001 (7)	0.0051 (7)	-0.0015 (7)
C13	0.0180 (8)	0.0185 (8)	0.0216 (8)	0.0019 (7)	0.0031 (7)	-0.0025 (7)
C14	0.0246 (9)	0.0218 (9)	0.0218 (9)	0.0016 (8)	0.0068 (8)	0.0016 (7)
C15	0.0161 (8)	0.0247 (9)	0.0243 (9)	-0.0003 (7)	0.0007 (7)	-0.0036 (7)
C16	0.0159 (8)	0.0251 (8)	0.0210 (8)	0.0015 (7)	-0.0006 (7)	0.0023 (7)
C17	0.0164 (8)	0.0281 (9)	0.0225 (8)	0.0019 (7)	0.0041 (7)	-0.0008 (7)
C18	0.0198 (8)	0.0219 (8)	0.0184 (8)	0.0010 (7)	0.0026 (7)	-0.0027 (7)
C19	0.0151 (8)	0.0210 (8)	0.0203 (8)	-0.0001 (6)	0.0013 (7)	0.0029 (7)
C20	0.0195 (8)	0.0294 (9)	0.0266 (9)	-0.0019 (7)	0.0072 (7)	-0.0068 (7)
C21	0.0230 (9)	0.0300 (10)	0.0247 (9)	-0.0023 (8)	0.0035 (7)	-0.0072 (8)
C22	0.0242 (10)	0.0402 (12)	0.0475 (12)	0.0015 (9)	0.0115 (9)	-0.0201 (10)
C23	0.0168 (8)	0.0287 (9)	0.0334 (10)	-0.0011 (7)	0.0079 (7)	-0.0061 (8)

Geometric parameters (Å, °)

O1—C18	1.370 (2)	C8—H8A	0.9900
O1—C22	1.436 (2)	C8—H8B	0.9900
O2—C19	1.3690 (19)	C9—C10	1.512 (2)
O2—C23	1.434 (2)	C9—H9A	0.9900
N1—C1	1.397 (2)	C9—H9B	0.9900
N1—C13	1.411 (2)	C10—C11	1.391 (2)
N2—C14	1.153 (2)	C11—C12	1.400 (2)
N3—C13	1.351 (2)	C11—C16	1.494 (2)
N3—H1	0.95 (2)	C12—C13	1.413 (2)
N3—H2	0.92 (2)	C12—C15	1.436 (2)
N4—C15	1.150 (2)	C16—C21	1.374 (2)
C1—C10	1.413 (2)	C16—C17	1.399 (2)
C1—C2	1.484 (2)	C17—C18	1.383 (2)
C2—C3	1.396 (3)	C17—H17	0.9500
C2—C7	1.405 (3)	C18—C19	1.403 (2)
C3—C4	1.386 (3)	C19—C20	1.380 (2)
C3—H3	0.9500	C20—C21	1.394 (2)
C4—C5	1.381 (3)	C20—H20	0.9500
C4—H4	0.9500	C21—H21	0.9500
C5—C6	1.386 (3)	C22—H22A	0.9800
C5—H5	0.9500	C22—H22B	0.9800
C6—C7	1.392 (3)	C22—H22C	0.9800
C6—H6	0.9500	C23—H23A	0.9800
C7—C8	1.499 (3)	C23—H23B	0.9800
C8—C9	1.528 (3)	C23—H23C	0.9800
C18—O1—C22	115.94 (13)	C1—C10—C9	117.78 (15)
C19—O2—C23	116.23 (13)	C10—C11—C12	120.31 (15)
C1—N1—C13	121.85 (15)	C10—C11—C16	120.23 (15)
C13—N3—H1	121.0 (15)	C12—C11—C16	119.45 (15)
C13—N3—H2	121.2 (13)	C11—C12—C13	121.05 (15)
H1—N3—H2	117.7 (19)	C11—C12—C15	120.84 (14)
N1—C1—C10	119.15 (15)	C13—C12—C15	118.10 (14)
N1—C1—C2	122.66 (15)	N3—C13—N1	121.00 (15)
C10—C1—C2	118.16 (15)	N3—C13—C12	121.45 (16)
C3—C2—C7	119.44 (17)	N1—C13—C12	117.52 (14)
C3—C2—C1	122.73 (17)	N4—C15—C12	177.44 (18)
C7—C2—C1	117.76 (16)	C21—C16—C17	119.08 (15)
C4—C3—C2	120.60 (19)	C21—C16—C11	121.48 (15)
C4—C3—H3	119.7	C17—C16—C11	119.44 (15)
C2—C3—H3	119.7	C18—C17—C16	120.75 (15)
C5—C4—C3	119.7 (2)	C18—C17—H17	119.6
C5—C4—H4	120.1	C16—C17—H17	119.6
C3—C4—H4	120.1	O1—C18—C17	124.52 (15)
C4—C5—C6	120.47 (19)	O1—C18—C19	115.72 (14)
C4—C5—H5	119.8	C17—C18—C19	119.76 (15)
C6—C5—H5	119.8	O2—C19—C20	124.56 (15)

supplementary materials

C5—C6—C7	120.5 (2)	O2—C19—C18	116.35 (14)
C5—C6—H6	119.7	C20—C19—C18	119.08 (15)
C7—C6—H6	119.7	C19—C20—C21	120.66 (16)
C6—C7—C2	119.19 (19)	C19—C20—H20	119.7
C6—C7—C8	122.56 (18)	C21—C20—H20	119.7
C2—C7—C8	118.19 (16)	C16—C21—C20	120.48 (16)
C7—C8—C9	108.73 (16)	C16—C21—H21	119.8
C7—C8—H8A	109.9	C20—C21—H21	119.8
C9—C8—H8A	109.9	O1—C22—H22A	109.5
C7—C8—H8B	109.9	O1—C22—H22B	109.5
C9—C8—H8B	109.9	H22A—C22—H22B	109.5
H8A—C8—H8B	108.3	O1—C22—H22C	109.5
C10—C9—C8	109.33 (14)	H22A—C22—H22C	109.5
C10—C9—H9A	109.8	H22B—C22—H22C	109.5
C8—C9—H9A	109.8	O2—C23—H23A	109.5
C10—C9—H9B	109.8	O2—C23—H23B	109.5
C8—C9—H9B	109.8	H23A—C23—H23B	109.5
H9A—C9—H9B	108.3	O2—C23—H23C	109.5
C11—C10—C1	119.86 (15)	H23A—C23—H23C	109.5
C11—C10—C9	122.36 (15)	H23B—C23—H23C	109.5

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H1 \cdots O1 ⁱ	0.95 (2)	2.24 (2)	2.927 (2)	129 (2)
N3—H2 \cdots O2 ⁱ	0.92 (2)	2.25 (2)	2.987 (2)	136 (2)

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

Fig. 1

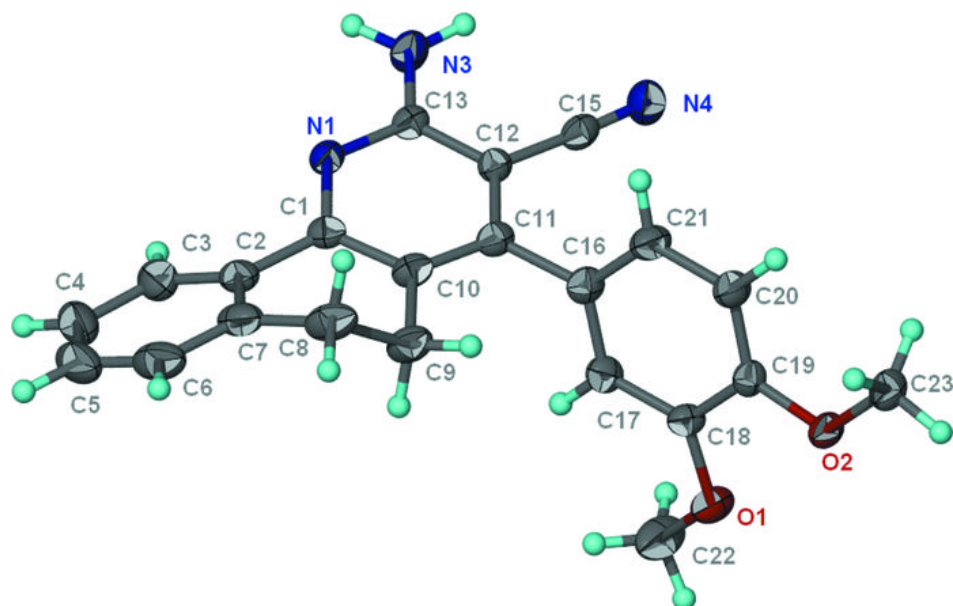


Fig. 2

