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Structure Reports

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2-Amino-4-(2-chlorophenyl)-5,10-dioxo-5,10-dihydro-4H-benzo[g]chromene-3-carbonitrile

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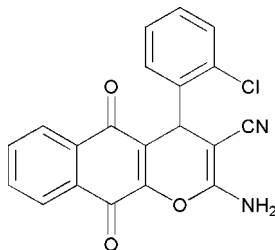
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.053; wR factor = 0.100; data-to-parameter ratio = 11.7.

In the molecule of the title compound, $\text{C}_{20}\text{H}_{11}\text{ClN}_2\text{O}_3$, the pyran ring adopts a flattened-boat conformation. In the crystal structure, intermolecular $\text{N}-\text{H}\cdots\text{N}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds generate edge-fused $R_2^2(12)$ and $R_2^2(14)$ ring motifs; the hydrogen-bonded motifs are linked to each other, forming a three-dimensional network. A $\pi-\pi$ contact [centroid-to-centroid distance = $3.879(3)$ Å] between the chlorophenyl rings may further stabilize the structure.

Related literature

For background to the biological activity of pyran and naphthoquinone compounds, see: El-Agrody *et al.* (2000); Mohr *et al.* (1975); Banzatti *et al.* (1984); Hatakeyama *et al.* (1988); Tandon *et al.* (1991); Kongkathip *et al.* (2003). For bond-length data, see: Allen *et al.* (1987). For puckering parameters, see: Cremer & Pople (1975). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_{20}\text{H}_{11}\text{ClN}_2\text{O}_3$
 $M_r = 362.76$
Triclinic, $P\bar{1}$
 $a = 8.3201(10)$ Å

$b = 9.3729(12)$ Å
 $c = 11.0081(16)$ Å
 $\alpha = 93.015(1)^\circ$
 $\beta = 96.393(1)^\circ$

$\gamma = 110.732(2)^\circ$
 $V = 793.95(18)$ Å³
 $Z = 2$
Mo $K\alpha$ radiation

$\mu = 0.27$ mm⁻¹
 $T = 298(2)$ K
 $0.17 \times 0.15 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.956$, $T_{\max} = 0.974$
4207 measured reflections
2746 independent reflections
1566 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.100$
 $S = 1.04$
2746 reflections
235 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.40$ e Å⁻³
 $\Delta\rho_{\min} = -0.26$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{N2}^{\text{i}}$	0.86	2.26	3.080 (4)	159
$\text{N1}-\text{H1B}\cdots\text{O3}^{\text{ii}}$	0.86	2.22	2.889 (3)	134

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

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008) and PLATON (Spek, 2003); software used to prepare material for publication: SHELXTL and PLATON.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2585).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Banzatti, C., Branzoli, U., Lovisolò, M. P., Melloni, P. & Salvador, P. (1984). *Arzneim. Forsch.* **34**, 864–869.
- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 155–1573.
- Bruker (1997). SMART and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- El-Agrody, A. M., El-Hakim, M. H., Abd El-Latif, M. S., Fakery, A. H., El-Sayed, E. S. M. & El-Ghareab, K. A. (2000). *Acta Pharm.* **50**, 111–120.
- Hatakeyama, S., Ochi, N., Numata, H. & Takano, S. (1988). *J. Chem. Soc. Chem. Commun.* pp. 1202–1204.
- Kongkathip, N., Kongkathip, B., Siripong, P., Sangma, C., Luangkamin, S., Niyomdech, M., Pattanapa, S., Piyaviriyagul, S. & Kongsaeer, P. (2003). *Bioorg. Med. Chem.* **11**, 3179–3191.
- Mohr, S. J., Chirigos, M. A., Fuhrman, F. S. & Pryor, J. W. (1975). *Cancer Res.* **35**, 3750–3754.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Tandon, V. K., Vaish, M., Jain, S., Bhakuni, D. S. & Srimal, R. C. (1991). *Indian J. Pharm. Sci.* **53**, 22–23.

supplementary materials

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2-Amino-4-(2-chlorophenyl)-5,10-dioxo-5,10-dihydro-4*H*-benzo[*g*]chromene-3-carbonitrile

J. Zhang, X. Zhang, S. Yan, N. Ma and S. Tu

Comment

Pyrans and their derivatives are important compounds, which are found to possess antibacterial (El-Agrody *et al.*, 2000) and antitumor (Mohr *et al.*, 1975) activities and antiallergic (Banzatti *et al.*, 1984; Hatakeyama *et al.*, 1988) and hypotensive (Tandon *et al.*, 1991) effects. Compounds of 1,4-naphthoquinone series possess potent and versatile biological activities, such as antiallergic and anticancer activities (Kongkathip *et al.*, 2003). For these reasons, 1,4-pyranonaphthoquinone derivatives possessing both pyran ring and 1,4-naphthoquinone motif are strongly desired. We report herein the crystal structure of the title compound.

In the molecule of the title compound (Fig. 1) the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Rings B (C4-C6/C11-C13), C (C6-C11) and D (C15-C20) are, of course, planar and the dihedral angles between them are B/C = 1.33 (3)°, B/D = 83.55 (3)° and C/D = 82.65 (3)°. So, rings B and C are nearly coplanar. Ring A (O1/C1-C4/C13) is not planar, having total puckering amplitude, Q_T , of 0.172 (3) and flattened-boat conformation [$\varphi = -22.99$ (3)° and $\theta = 105.077$ (4)°] (Cremer & Pople, 1975).

In the crystal structure, intermolecular N-H...N and N-H...O hydrogen bonds (Table 1) generate edge-fused $R_2^2(12)$ and $R_2^2(14)$ ring motifs (Fig. 2) (Bernstein *et al.*, 1995). The hydrogen bonded motifs are linked to each other to form a three dimensional network, in which they may be effective in the stabilization of the structure. The π - π contact between the chlorophenyl rings, Cg4—Cg4ⁱ [symmetry code: (i) -x, -y, 2 - z, where Cg4 is centroid of the ring D (C15-C20)] may further stabilize the structure, with centroid-centroid distance of 3.879 (3) Å.

Experimental

The title compound was prepared by the reaction of 2-(2-chlorobenzylidene)- malononitrile (1 mmol) and 2-hydroxynaphthalene-1,4-dione (1 mmol) in glacial acetic acid without catalyst. Crystals suitable for X-ray analysis were obtained by slow evaporation of an aqueous ethanol solution (95%) (yield; 90%; m.p. > 573 K).

Refinement

H atoms were positioned geometrically, with N-H = 0.86 Å (for NH₂) and C-H = 0.93 and 0.98 Å for aromatic and methine H, respectively, and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

Figures

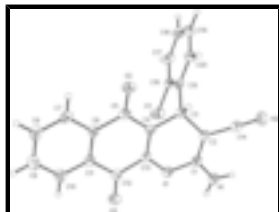


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

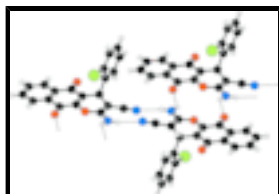


Fig. 2. A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

2-Amino-4-(2-chlorophenyl)-5,10-dioxo-5,10-dihydro-4H- benzo[g]chromene-3-carbonitrile

Crystal data

$C_{20}H_{11}ClN_2O_3$

$M_r = 362.76$

Triclinic, PT

Hall symbol: $-P\ 1$

$a = 8.3201\ (10)\ \text{\AA}$

$b = 9.3729\ (12)\ \text{\AA}$

$c = 11.0081\ (16)\ \text{\AA}$

$\alpha = 93.015\ (1)^\circ$

$\beta = 96.393\ (1)^\circ$

$\gamma = 110.732\ (2)^\circ$

$V = 793.95\ (18)\ \text{\AA}^3$

$Z = 2$

$F_{000} = 372$

$D_x = 1.517\ \text{Mg m}^{-3}$

Melting point $> 573\ \text{K}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 959 reflections

$\theta = 2.8\text{--}25.1^\circ$

$\mu = 0.27\ \text{mm}^{-1}$

$T = 298\ (2)\ \text{K}$

Block, orange

$0.17 \times 0.15 \times 0.10\ \text{mm}$

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298\ (2)\ \text{K}$

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.956$, $T_{\max} = 0.974$

4207 measured reflections

2746 independent reflections

1566 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 25.0^\circ$

$\theta_{\min} = 1.9^\circ$

$h = -9 \rightarrow 9$

$k = -11 \rightarrow 10$

$l = -13 \rightarrow 8$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.053$	H-atom parameters constrained
$wR(F^2) = 0.100$	$w = 1/[\sigma^2(F_o^2) + (0.0295P)^2]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
2746 reflections	$(\Delta/\sigma)_{\max} < 0.001$
235 parameters	$\Delta\rho_{\max} = 0.40 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.26 \text{ e } \text{\AA}^{-3}$
	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 $F^2 > \sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	-0.01619 (12)	0.04001 (10)	0.69113 (9)	0.0587 (3)
O1	0.1440 (3)	0.3890 (2)	0.6133 (2)	0.0405 (6)
O2	0.5886 (3)	0.2063 (3)	0.7464 (2)	0.0541 (7)
O3	0.2251 (3)	0.3753 (3)	0.3898 (2)	0.0536 (7)
N1	-0.0034 (3)	0.4822 (3)	0.7313 (2)	0.0471 (8)
H1A	-0.0304	0.5099	0.7994	0.057*
H1B	-0.0556	0.4936	0.6626	0.057*
N2	0.1921 (4)	0.4871 (4)	1.0491 (3)	0.0578 (9)
C1	0.1196 (4)	0.4216 (3)	0.7320 (3)	0.0369 (8)
C2	0.2135 (4)	0.3957 (3)	0.8299 (3)	0.0344 (8)
C3	0.3297 (4)	0.3028 (3)	0.8197 (3)	0.0336 (8)
H3	0.4412	0.3622	0.8696	0.040*
C4	0.3646 (4)	0.2940 (3)	0.6883 (3)	0.0309 (8)
C5	0.5061 (4)	0.2424 (3)	0.6626 (3)	0.0360 (8)
C6	0.5448 (4)	0.2361 (3)	0.5342 (3)	0.0339 (8)
C7	0.6774 (4)	0.1889 (4)	0.5076 (3)	0.0466 (9)
H7	0.7416	0.1604	0.5699	0.056*
C8	0.7152 (5)	0.1837 (4)	0.3890 (3)	0.0538 (10)

supplementary materials

H8	0.8064	0.1540	0.3718	0.065*
C9	0.6180 (4)	0.2225 (4)	0.2961 (3)	0.0530 (10)
H9	0.6426	0.2170	0.2160	0.064*
C10	0.4851 (4)	0.2690 (4)	0.3207 (3)	0.0461 (9)
H10	0.4202	0.2951	0.2574	0.055*
C11	0.4476 (4)	0.2770 (3)	0.4401 (3)	0.0346 (8)
C12	0.3083 (4)	0.3326 (3)	0.4679 (3)	0.0350 (8)
C13	0.2762 (4)	0.3374 (3)	0.5983 (3)	0.0337 (8)
C14	0.1989 (4)	0.4470 (4)	0.9505 (3)	0.0405 (9)
C15	0.2617 (4)	0.1487 (3)	0.8721 (3)	0.0322 (8)
C16	0.1126 (4)	0.0269 (4)	0.8235 (3)	0.0374 (9)
C17	0.0573 (4)	-0.1101 (4)	0.8767 (3)	0.0450 (9)
H17	-0.0427	-0.1903	0.8412	0.054*
C18	0.1515 (5)	-0.1260 (4)	0.9819 (3)	0.0538 (10)
H18	0.1157	-0.2177	1.0179	0.065*
C19	0.2985 (5)	-0.0068 (4)	1.0343 (3)	0.0514 (10)
H19	0.3613	-0.0172	1.1065	0.062*
C20	0.3530 (4)	0.1287 (4)	0.9798 (3)	0.0433 (9)
H20	0.4532	0.2084	1.0158	0.052*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0512 (6)	0.0542 (6)	0.0649 (7)	0.0171 (4)	-0.0099 (5)	0.0082 (5)
O1	0.0409 (14)	0.0529 (14)	0.0391 (14)	0.0302 (12)	0.0069 (11)	0.0067 (12)
O2	0.0534 (16)	0.0759 (17)	0.0500 (16)	0.0423 (14)	0.0080 (13)	0.0191 (14)
O3	0.0643 (17)	0.0733 (17)	0.0398 (15)	0.0459 (14)	0.0020 (13)	0.0114 (13)
N1	0.0525 (19)	0.0636 (19)	0.0407 (18)	0.0403 (16)	0.0056 (15)	0.0031 (16)
N2	0.065 (2)	0.066 (2)	0.046 (2)	0.0297 (18)	0.0090 (18)	-0.0059 (18)
C1	0.037 (2)	0.0340 (19)	0.044 (2)	0.0177 (16)	0.0102 (18)	0.0047 (17)
C2	0.037 (2)	0.0356 (19)	0.035 (2)	0.0183 (16)	0.0072 (17)	0.0027 (17)
C3	0.0340 (19)	0.0366 (19)	0.033 (2)	0.0165 (15)	0.0015 (16)	0.0036 (17)
C4	0.0328 (19)	0.0285 (17)	0.034 (2)	0.0135 (15)	0.0057 (16)	0.0044 (16)
C5	0.0319 (19)	0.0350 (19)	0.044 (2)	0.0162 (16)	0.0015 (18)	0.0081 (18)
C6	0.033 (2)	0.0334 (18)	0.036 (2)	0.0121 (15)	0.0061 (17)	0.0041 (17)
C7	0.043 (2)	0.053 (2)	0.054 (3)	0.0278 (18)	0.0078 (19)	0.008 (2)
C8	0.049 (2)	0.065 (3)	0.055 (3)	0.028 (2)	0.016 (2)	-0.002 (2)
C9	0.051 (2)	0.065 (3)	0.044 (2)	0.021 (2)	0.010 (2)	-0.001 (2)
C10	0.043 (2)	0.054 (2)	0.044 (2)	0.0207 (18)	0.0074 (19)	0.003 (2)
C11	0.035 (2)	0.0324 (19)	0.035 (2)	0.0108 (15)	0.0047 (17)	-0.0009 (17)
C12	0.035 (2)	0.0325 (19)	0.039 (2)	0.0132 (15)	0.0047 (17)	0.0037 (17)
C13	0.0346 (19)	0.0297 (18)	0.040 (2)	0.0158 (15)	0.0061 (17)	0.0012 (17)
C14	0.039 (2)	0.037 (2)	0.048 (2)	0.0164 (16)	0.0055 (19)	0.008 (2)
C15	0.0331 (19)	0.0377 (19)	0.0338 (19)	0.0207 (16)	0.0098 (16)	0.0064 (17)
C16	0.036 (2)	0.042 (2)	0.042 (2)	0.0234 (16)	0.0058 (17)	0.0035 (18)
C17	0.041 (2)	0.036 (2)	0.062 (3)	0.0163 (16)	0.0148 (19)	0.0048 (19)
C18	0.062 (3)	0.050 (2)	0.063 (3)	0.031 (2)	0.021 (2)	0.023 (2)
C19	0.059 (3)	0.059 (3)	0.046 (2)	0.032 (2)	0.009 (2)	0.015 (2)

C20 0.045 (2) 0.047 (2) 0.040 (2) 0.0190 (17) 0.0035 (18) 0.0086 (18)

Geometric parameters (Å, °)

C11—C16	1.747 (3)	C7—C8	1.379 (5)
O1—C1	1.380 (4)	C7—H7	0.9300
O1—C13	1.370 (3)	C8—C9	1.377 (4)
O2—C5	1.223 (3)	C8—H8	0.9300
O3—C12	1.216 (3)	C9—C10	1.372 (4)
N1—C1	1.334 (3)	C9—H9	0.9300
N1—H1A	0.8600	C10—C11	1.389 (4)
N1—H1B	0.8600	C10—H10	0.9300
N2—C14	1.144 (4)	C11—C12	1.482 (4)
C1—C2	1.343 (4)	C12—C13	1.490 (4)
C2—C14	1.420 (5)	C15—C16	1.383 (4)
C2—C3	1.522 (4)	C15—C20	1.393 (4)
C3—C4	1.510 (4)	C16—C17	1.387 (4)
C3—C15	1.524 (4)	C17—C18	1.370 (4)
C3—H3	0.9800	C17—H17	0.9300
C4—C13	1.335 (4)	C18—C19	1.373 (4)
C4—C5	1.470 (4)	C18—H18	0.9300
C5—C6	1.487 (4)	C19—C20	1.383 (4)
C6—C7	1.380 (4)	C19—H19	0.9300
C6—C11	1.396 (4)	C20—H20	0.9300
C13—O1—C1	117.4 (2)	C8—C9—H9	119.7
C1—N1—H1A	120.0	C9—C10—C11	120.0 (3)
C1—N1—H1B	120.0	C9—C10—H10	120.0
H1A—N1—H1B	120.0	C11—C10—H10	120.0
N1—C1—C2	127.8 (3)	C10—C11—C6	119.6 (3)
N1—C1—O1	110.1 (3)	C10—C11—C12	120.2 (3)
C2—C1—O1	122.1 (3)	C6—C11—C12	120.2 (3)
C1—C2—C14	120.4 (3)	O3—C12—C11	122.6 (3)
C1—C2—C3	122.9 (3)	O3—C12—C13	120.7 (3)
C14—C2—C3	116.6 (3)	C11—C12—C13	116.8 (3)
C4—C3—C2	108.4 (2)	C4—C13—O1	124.6 (3)
C4—C3—C15	114.8 (2)	C4—C13—C12	123.5 (3)
C2—C3—C15	113.2 (3)	O1—C13—C12	111.9 (3)
C4—C3—H3	106.6	N2—C14—C2	177.6 (4)
C2—C3—H3	106.6	C16—C15—C20	116.6 (3)
C15—C3—H3	106.6	C16—C15—C3	125.4 (3)
C13—C4—C5	120.3 (3)	C20—C15—C3	118.0 (3)
C13—C4—C3	121.8 (3)	C15—C16—C17	122.3 (3)
C5—C4—C3	117.8 (3)	C15—C16—C11	120.9 (2)
O2—C5—C4	119.6 (3)	C17—C16—C11	116.8 (3)
O2—C5—C6	121.8 (3)	C18—C17—C16	119.4 (3)
C4—C5—C6	118.6 (3)	C18—C17—H17	120.3
C7—C6—C11	119.7 (3)	C16—C17—H17	120.3
C7—C6—C5	119.8 (3)	C17—C18—C19	120.1 (3)
C11—C6—C5	120.6 (3)	C17—C18—H18	120.0

supplementary materials

C8—C7—C6	120.2 (3)	C19—C18—H18	120.0
C8—C7—H7	119.9	C18—C19—C20	119.9 (3)
C6—C7—H7	119.9	C18—C19—H19	120.0
C9—C8—C7	120.0 (4)	C20—C19—H19	120.0
C9—C8—H8	120.0	C19—C20—C15	121.6 (3)
C7—C8—H8	120.0	C19—C20—H20	119.2
C10—C9—C8	120.5 (4)	C15—C20—H20	119.2
C10—C9—H9	119.7		
C13—O1—C1—N1	174.8 (2)	C7—C6—C11—C12	-178.1 (3)
C13—O1—C1—C2	-3.9 (4)	C5—C6—C11—C12	2.1 (4)
N1—C1—C2—C14	-5.9 (6)	C10—C11—C12—O3	-1.4 (5)
O1—C1—C2—C14	172.6 (3)	C6—C11—C12—O3	177.0 (3)
N1—C1—C2—C3	169.9 (3)	C10—C11—C12—C13	179.9 (3)
O1—C1—C2—C3	-11.5 (5)	C6—C11—C12—C13	-1.7 (4)
C1—C2—C3—C4	18.2 (4)	C5—C4—C13—O1	-179.1 (3)
C14—C2—C3—C4	-165.8 (3)	C3—C4—C13—O1	-2.0 (5)
C1—C2—C3—C15	-110.3 (4)	C5—C4—C13—C12	2.0 (5)
C14—C2—C3—C15	65.6 (4)	C3—C4—C13—C12	179.1 (3)
C2—C3—C4—C13	-11.5 (4)	C1—O1—C13—C4	11.0 (4)
C15—C3—C4—C13	116.1 (3)	C1—O1—C13—C12	-170.1 (3)
C2—C3—C4—C5	165.6 (3)	O3—C12—C13—C4	-179.1 (3)
C15—C3—C4—C5	-66.7 (3)	C11—C12—C13—C4	-0.4 (4)
C13—C4—C5—O2	178.4 (3)	O3—C12—C13—O1	1.9 (4)
C3—C4—C5—O2	1.1 (4)	C11—C12—C13—O1	-179.4 (2)
C13—C4—C5—C6	-1.6 (4)	C4—C3—C15—C16	-57.8 (4)
C3—C4—C5—C6	-178.8 (3)	C2—C3—C15—C16	67.4 (4)
O2—C5—C6—C7	-0.2 (5)	C4—C3—C15—C20	124.2 (3)
C4—C5—C6—C7	179.7 (3)	C2—C3—C15—C20	-110.6 (3)
O2—C5—C6—C11	179.5 (3)	C20—C15—C16—C17	-1.4 (5)
C4—C5—C6—C11	-0.6 (4)	C3—C15—C16—C17	-179.4 (3)
C11—C6—C7—C8	0.7 (5)	C20—C15—C16—C11	178.3 (2)
C5—C6—C7—C8	-179.5 (3)	C3—C15—C16—C11	0.2 (4)
C6—C7—C8—C9	-1.4 (5)	C15—C16—C17—C18	0.8 (5)
C7—C8—C9—C10	1.1 (5)	C11—C16—C17—C18	-178.8 (3)
C8—C9—C10—C11	-0.1 (5)	C16—C17—C18—C19	0.4 (5)
C9—C10—C11—C6	-0.6 (5)	C17—C18—C19—C20	-1.0 (5)
C9—C10—C11—C12	177.8 (3)	C18—C19—C20—C15	0.4 (5)
C7—C6—C11—C10	0.3 (5)	C16—C15—C20—C19	0.8 (5)
C5—C6—C11—C10	-179.5 (3)	C3—C15—C20—C19	179.0 (3)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots N2 ⁱ	0.86	2.26	3.080 (4)	159
N1—H1B \cdots O3 ⁱⁱ	0.86	2.22	2.889 (3)	134

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

Fig. 1

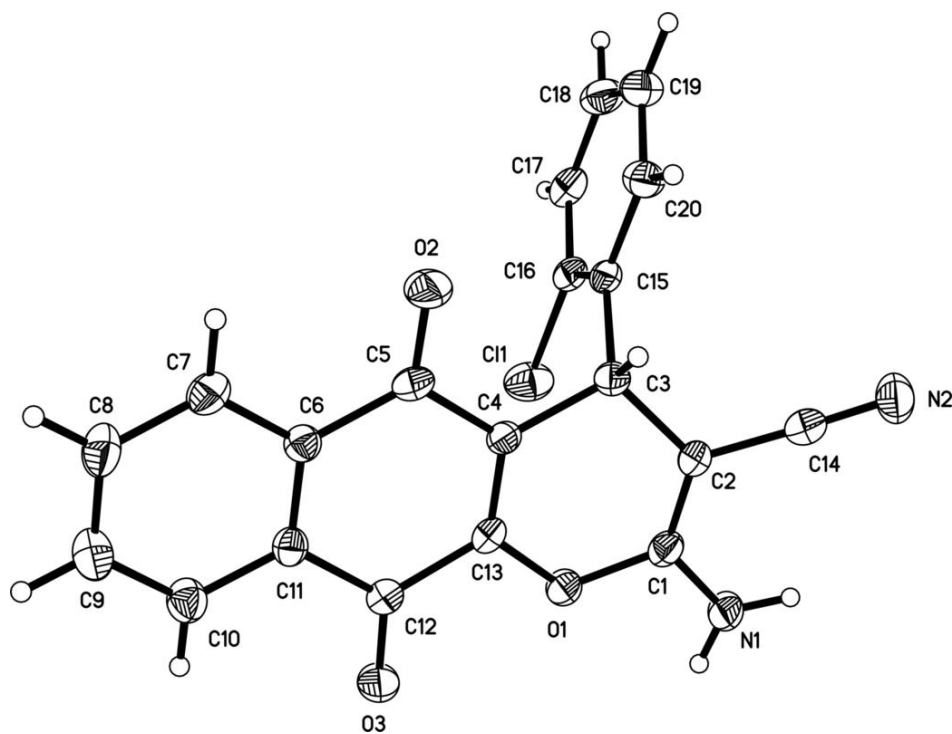


Fig. 2

