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

**Jinpeng Zhang,[a] Xiaohong Zhang,[b] Shu Yan,[b] Ning Ma[b] and Shujiang Tu[b]***

[a]Department of Public Health, Xuzhou Medical College, Xuzhou 221000, People's Republic of China, and [b]School of Chemistry and Chemical Engineering, Xuzhou Normal University, Xuzhou 221116, People's Republic of China
Correspondence e-mail: laotu2001@263.net

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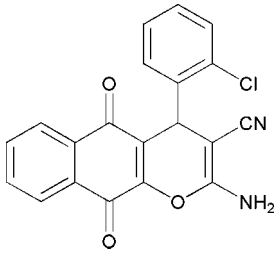
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.053; $wR$ factor = 0.100; data-to-parameter ratio = 11.7.

In the molecule of the title compound, $C_{20}H_{11}ClN_2O_3$, the pyran ring adopts a flattened-boat conformation. In the crystal structure, intermolecular N—H···N and N—H···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

$C_{20}H_{11}ClN_2O_3$
$M_r$ = 362.76
Triclinic, $P\overline{1}$
$a$ = 8.3201 (10) Å
$b$ = 9.3729 (12) Å
$c$ = 11.0081 (16) Å
$\alpha$ = 93.015 (1)°
$\beta$ = 96.393 (1)°
$\gamma$ = 110.732 (2)°
$V$ = 793.95 (18) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.27 mm$^{-1}$
$T$ = 298 (2) K
0.17 × 0.15 × 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 $I > 2\sigma(I)$
$R_{int}$ = 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···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$A$···N2$^{i}$ | 0.86 | 2.26 | 3.080 (4) | 159 |
| N1—H1$B$···O3$^{ii}$ | 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).

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# 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 [φ = -22.99 (3)° and θ = 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—$Cg4^i$ [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 $NH_2$) 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

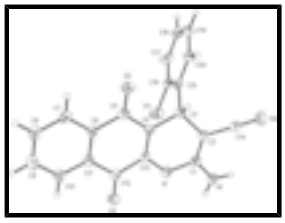

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

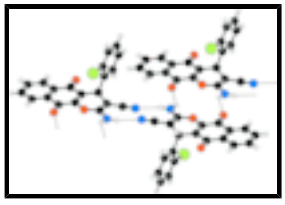

Fig. 2. A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

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

*Crystal data*

| | |
|---|---|
| $C_{20}H_{11}ClN_2O_3$ | $Z = 2$ |
| $M_r = 362.76$ | $F_{000} = 372$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.517$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Melting point > 573 K |
| $a = 8.3201$ (10) Å | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $b = 9.3729$ (12) Å | Cell parameters from 959 reflections |
| $c = 11.0081$ (16) Å | $\theta = 2.8$–$25.1^\circ$ |
| $\alpha = 93.015$ (1)° | $\mu = 0.27$ mm$^{-1}$ |
| $\beta = 96.393$ (1)° | $T = 298$ (2) K |
| $\gamma = 110.732$ (2)° | Block, orange |
| $V = 793.95$ (18) Å$^3$ | 0.17 × 0.15 × 0.10 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 2746 independent reflections |
| Radiation source: fine-focus sealed tube | 1566 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.025$ |
| $T = 298$(2) K | $\theta_{max} = 25.0^\circ$ |
| φ and ω scans | $\theta_{min} = 1.9^\circ$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $h = -9 \rightarrow 9$ |
| $T_{min} = 0.956$, $T_{max} = 0.974$ | $k = -11 \rightarrow 10$ |
| 4207 measured reflections | $l = -13 \rightarrow 8$ |

*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 |
| $wR(F^2) = 0.100$ | $w = 1/[\sigma^2(F_o^2) + (0.0295P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.04$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 2746 reflections | $\Delta\rho_{max} = 0.40$ e Å$^{-3}$ |
| 235 parameters | $\Delta\rho_{min} = -0.26$ 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 $F^2 > \sigma(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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) |

| | | | | |
|---|---|---|---|---|
| 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 (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 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, °)*

| | | | |
|---|---|---|---|
| Cl1—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—Cl1 | 120.9 (2) |
| O2—C5—C4 | 119.6 (3) | C17—C16—Cl1 | 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 |

| | | | |
|---|---|---|---|
| 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—Cl1 | 178.3 (2) |
| C5—C6—C7—C8 | −179.5 (3) | C3—C15—C16—Cl1 | 0.2 (4) |
| C6—C7—C8—C9 | −1.4 (5) | C15—C16—C17—C18 | 0.8 (5) |
| C7—C8—C9—C10 | 1.1 (5) | Cl1—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 (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1A···N2$^{i}$ | 0.86 | 2.26 | 3.080 (4) | 159 |
| N1—H1B···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**

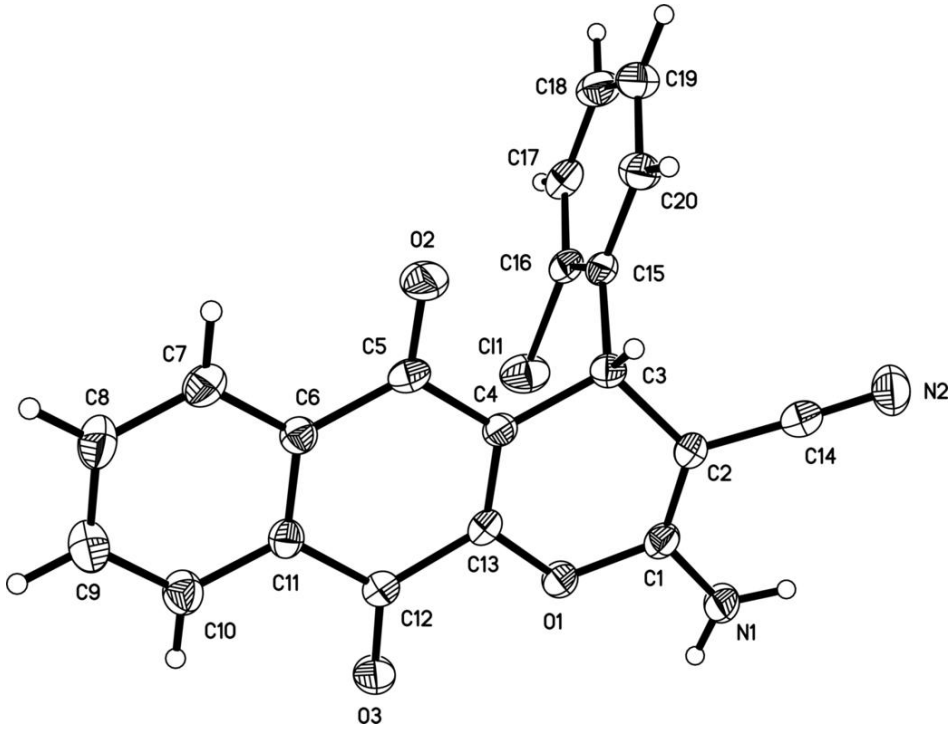
C18
C19
C17
C20
O2
C16
C15
C7
C5
Cl1
C3
N2
C8
C6
C4
C14
C2
C13
C1
C9
C11
C12
O1
N1
C10
O3

**Fig. 2**