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2-Amino-2′,5-dioxo-5,6,7,8-tetrahydrospiro[chromene-4,3′-indoline]-3-carbonitrile *N*,*N*-dimethylformamide solvate

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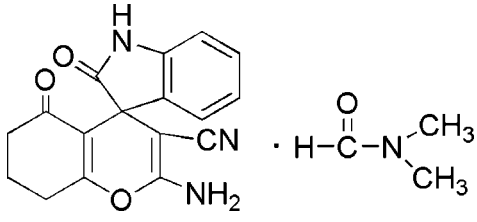
Key indicators: single-crystal X-ray study; T = 193 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.114; data-to-parameter ratio = 13.1.

The title compound, $C_{17}H_{13}N_3O_3\cdot C_3H_7NO$, was synthesized by the one-pot reaction of isatin, malononitrile and cyclohexane-1,3-dione in water. The cyclohexene ring adopts a half-chair conformation and the five-membered ring of the indolinone ring system is in a twist conformation. N—H···O and N—H···N intermolecular hydrogen bonds link the spirooxindole molecules into a chain along the *b* axis, and the *N*,*N*-dimethylformamide solvent molecules are linked to the chain *via* N—H···O and C—H···O hydrogen bonds.

Related literature

For general background, see: Da-Silva *et al.* (2001); Joshi & Chand (1982); Abdel-Rahman *et al.* (2004); Zhu *et al.* (2007).



Experimental

Crystal data

$C_{17}H_{13}N_3O_3\cdot C_3H_7NO$
M_r = 380.40
Triclinic, $P\overline{1}$
a = 7.1833 (14) Å
b = 8.8173 (18) Å
c = 15.351 (3) Å
α = 77.907 (13)°
β = 77.691 (13)°
γ = 81.392 (14)°
V = 923.3 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 193 (2) K
0.60 × 0.30 × 0.30 mm

Data collection

Rigaku Mercury diffractometer
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.802, T_{max} = 0.971
9034 measured reflections
3358 independent reflections
2961 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.114
S = 1.09
3358 reflections
256 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O4^{i}	0.88	1.99	2.767 (2)	147
N3—H3A···N2^{ii}	0.88	2.31	3.130 (2)	155
N3—H3B···O3^{iii}	0.88	2.06	2.897 (2)	158
C20—H20A···O2	0.98	2.50	3.464 (3)	167

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

Data collection: *CrystalClear* (Rigaku/MSC, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); 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: CI2470).

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supplementary materials

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2-Amino-2',5-dioxo-5,6,7,8-tetrahydrospiro[chromene-4,3'-indoline]-3-carbonitrile *N*,*N*-dimethylformamide solvate

J. Wang and S.-L. Zhu

Comment

The indole nucleus is a well known heterocycle (Da-Silva *et al.*, 2001). Compounds carrying the indole moiety exhibit antibacterial and antifungal activities (Joshi & Chand, 1982). Spirooxindole ring systems are found in a number of alkaloids like horsifiline, spirotryprostatin and elacomine (Abdel-Rahman *et al.*, 2004). As a part of our program devoted to the preparation of heterocyclic compounds involving indole derivatives (Zhu *et al.*, 2007), we have synthesized a series of spirooxindoles *via* reactions of isatins together with malononitrile and cyclohexane-1,3-dione in water. We report here the crystal structure of the title compound.

In the title molecule (Fig.1), the atoms of the pyran ring (C1—C5/O1) are almost coplanar with the largest deviation being 0.029 (2) Å for atom C2. The five-membered ring of the dihydroindolone ring system adopts a twist conformation. The cyclohexene ring adopts a half-chair conformation, with atoms C7 and C8 deviating from the C1/C2/C6/C9 plane by −0.222 (3) and 0.453 (3) Å, respectively.

The N—H···O and N—H···N hydrogen bonds link the spirooxindole molecules into a chain along the *b* axis. The *N*,*N*-dimethylformamide solvent molecules are linked to the chain *via* N—H···O and C—H···O hydrogen bonds (Table 1 and Fig. 2).

Experimental

The title compound was prepared by the reaction of isatin (1 mmol), malononitrile (1 mmol) and cyclohexane-1,3-dione (1 mmol) in water. The reaction was catalyzed by TEBA (triethylbenzylammonium chloride, 1 mmol). After stirring at 333 K for 3 h, the reaction mixture was cooled and washed with small amount ethanol. The crude product was filtered and single crystals of the title compound, suitable for X-ray diffraction, were obtained from *N*,*N*-dimethylformamide solution by slow evaporation (yield 88%; m.p. 573–575 K). Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 33372, 3287, 3133, 2955, 2191, 1698, 1613, 1466, 1350, 1211, 1011, 933, 764, 679 ^{1}H NMR (400 MHz, DMSO-d_6): 10.39 (s, 1H, NH), 7.21 (br s, 2H, NH_2), 7.13 (t, 1H, J = 7.6 Hz, ArH), 7.01(d, 1H, J = 7.6 Hz, ArH), 6.88 (t, 1H, J = 7.6 Hz, ArH), 6.77(d, 1H, J = 8.0 Hz, ArH), 2.63–2.67 (m, 2H, CH_2), 2.30–2.37 (m, 2H, CH_2), 1.90–1.93 (m, 2H, CH_2).

Refinement

H atoms were placed in the idealized positions and allowed to ride on their parent atoms, with N—H = 0.88 Å, C—H = 0.95–0.99 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C,*N*) for others.

Figures

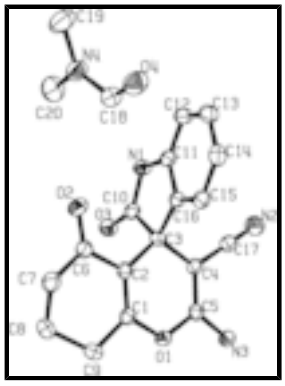

Fig. 1. The molecular structure of the title compound, showing 40% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

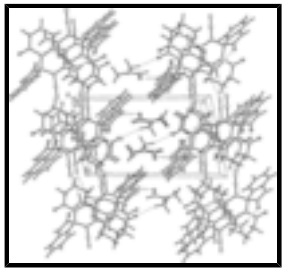

Fig. 2. The crystal packing of the title compound. Hydrogen bonds are shown as dashed lines.

2-Amino-2',5-dioxo-5,6,7,8-tetrahydrospiro[chromene-4,3'-indoline]- 3-carbonitrile *N*,*N*-dimethylformamide solvate

Crystal data

$C_{17}H_{13}N_3O_3 \cdot C_3H_7NO$	$Z = 2$
$M_r = 380.40$	$F_{000} = 400$
Triclinic, $P\overline{1}$	$D_x = 1.368$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 573-575 K
$a = 7.1833$ (14) Å	Mo $K\alpha$ radiation $\lambda = 0.71070$ Å
$b = 8.8173$ (18) Å	Cell parameters from 3696 reflections
$c = 15.351$ (3) Å	$\theta = 3.3$–25.3º
$\alpha = 77.907$ (13)º	$\mu = 0.10$ mm^{-1}
$\beta = 77.691$ (13)º	$T = 193$ (2) K
$\gamma = 81.392$ (14)º	Block, colourless
$V = 923.3$ (3) Å^3	0.60 × 0.30 × 0.30 mm

Data collection

Rigaku Mercury diffractometer	3358 independent reflections
Radiation source: fine-focus sealed tube	2961 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.023$
$T = 193$(2) K	$\theta_{max} = 25.4$º
ω scans	$\theta_{min} = 3.3$º

Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.802, T_{max} = 0.971
9034 measured reflections

h = −8→8
k = −10→10
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.114
S = 1.09
3358 reflections
256 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0535P)^2 + 0.3215P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-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 $F^2 > 2$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}
O1	0.75602 (16)	0.52904 (12)	−0.00607 (7)	0.0249 (3)
O2	0.50301 (19)	0.52756 (14)	0.30435 (8)	0.0367 (3)
O3	0.98417 (17)	0.42893 (14)	0.22839 (8)	0.0293 (3)
O4	−0.06193 (19)	0.22731 (16)	0.47536 (9)	0.0418 (4)
N1	0.8164 (2)	0.23025 (16)	0.31563 (9)	0.0254 (3)
H1	0.8864	0.1995	0.3580	0.030*
N2	0.9859 (2)	0.00722 (18)	0.10933 (11)	0.0399 (4)
N3	0.9162 (2)	0.33299 (16)	−0.07013 (9)	0.0272 (3)
H3A	0.9712	0.2370	−0.0710	0.033*
H3B	0.9163	0.4006	−0.1212	0.033*
N4	0.1442 (2)	0.31623 (18)	0.54114 (10)	0.0338 (4)
C1	0.6547 (2)	0.59323 (18)	0.06719 (11)	0.0217 (4)
C2	0.6263 (2)	0.51357 (18)	0.15212 (11)	0.0212 (4)
C3	0.7133 (2)	0.34806 (18)	0.17859 (11)	0.0214 (4)

C4	0.8191 (2)	0.28850 (18)	0.09332 (11)	0.0219 (4)
C5	0.8330 (2)	0.37603 (18)	0.00864 (11)	0.0213 (4)
C6	0.5133 (2)	0.59234 (19)	0.22550 (11)	0.0256 (4)
C7	0.4122 (3)	0.7513 (2)	0.19830 (12)	0.0327 (4)
H7A	0.3859	0.8074	0.2499	0.039*
H7B	0.2877	0.7405	0.1836	0.039*
C8	0.5300 (3)	0.8459 (2)	0.11697 (13)	0.0348 (4)
H8A	0.6477	0.8673	0.1339	0.042*
H8B	0.4553	0.9471	0.0987	0.042*
C9	0.5850 (2)	0.75999 (19)	0.03733 (11)	0.0265 (4)
H9A	0.4723	0.7661	0.0087	0.032*
H9B	0.6868	0.8110	−0.0086	0.032*
C10	0.8564 (2)	0.34412 (18)	0.24291 (11)	0.0230 (4)
C11	0.6486 (2)	0.16690 (19)	0.31519 (11)	0.0236 (4)
C12	0.5570 (3)	0.0556 (2)	0.38021 (12)	0.0291 (4)
H12	0.6091	0.0065	0.4324	0.035*
C13	0.3851 (3)	0.0174 (2)	0.36679 (12)	0.0312 (4)
H13	0.3189	−0.0592	0.4105	0.037*
C14	0.3093 (2)	0.0893 (2)	0.29071 (12)	0.0292 (4)
H14	0.1905	0.0632	0.2836	0.035*
C15	0.4053 (2)	0.19930 (19)	0.22472 (11)	0.0252 (4)
H15	0.3544	0.2474	0.1721	0.030*
C16	0.5758 (2)	0.23715 (18)	0.23728 (11)	0.0215 (4)
C17	0.9110 (2)	0.1332 (2)	0.10209 (11)	0.0266 (4)
C18	0.0839 (3)	0.2885 (2)	0.47086 (12)	0.0325 (4)
H18	0.1590	0.3182	0.4124	0.039*
C19	0.0350 (3)	0.2783 (3)	0.63242 (14)	0.0517 (6)
H19A	−0.0873	0.2444	0.6299	0.077*
H19B	0.0106	0.3708	0.6609	0.077*
H19C	0.1079	0.1942	0.6681	0.077*
C20	0.3197 (3)	0.3875 (3)	0.52988 (16)	0.0454 (5)
H20A	0.3779	0.4090	0.4654	0.068*
H20B	0.4093	0.3162	0.5634	0.068*
H20C	0.2900	0.4853	0.5532	0.068*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0310 (6)	0.0231 (6)	0.0189 (6)	−0.0024 (5)	−0.0024 (5)	−0.0025 (5)
O2	0.0511 (8)	0.0345 (7)	0.0211 (7)	0.0021 (6)	−0.0031 (6)	−0.0063 (5)
O3	0.0283 (6)	0.0318 (7)	0.0287 (7)	−0.0089 (5)	−0.0069 (5)	−0.0026 (5)
O4	0.0434 (8)	0.0536 (9)	0.0340 (8)	−0.0151 (7)	−0.0133 (6)	−0.0074 (6)
N1	0.0259 (7)	0.0292 (8)	0.0208 (7)	−0.0042 (6)	−0.0076 (6)	−0.0001 (6)
N2	0.0497 (10)	0.0302 (9)	0.0351 (9)	0.0039 (8)	−0.0022 (8)	−0.0071 (7)
N3	0.0333 (8)	0.0256 (7)	0.0209 (7)	−0.0034 (6)	−0.0001 (6)	−0.0052 (6)
N4	0.0357 (8)	0.0396 (9)	0.0281 (8)	−0.0114 (7)	−0.0049 (7)	−0.0070 (7)
C1	0.0208 (8)	0.0237 (8)	0.0219 (8)	−0.0039 (6)	−0.0043 (6)	−0.0057 (6)
C2	0.0214 (8)	0.0211 (8)	0.0221 (8)	−0.0027 (6)	−0.0052 (7)	−0.0051 (6)

C3	0.0226 (8)	0.0212 (8)	0.0196 (8)	−0.0033 (7)	−0.0022 (6)	−0.0031 (6)
C4	0.0221 (8)	0.0226 (8)	0.0208 (8)	−0.0026 (7)	−0.0025 (6)	−0.0048 (6)
C5	0.0196 (8)	0.0216 (8)	0.0239 (9)	−0.0057 (6)	−0.0029 (6)	−0.0059 (7)
C6	0.0273 (9)	0.0263 (9)	0.0241 (9)	−0.0035 (7)	−0.0041 (7)	−0.0069 (7)
C7	0.0372 (10)	0.0304 (10)	0.0292 (10)	0.0043 (8)	−0.0048 (8)	−0.0097 (8)
C8	0.0466 (11)	0.0217 (9)	0.0353 (10)	0.0013 (8)	−0.0097 (9)	−0.0053 (7)
C9	0.0282 (9)	0.0235 (9)	0.0261 (9)	−0.0026 (7)	−0.0068 (7)	0.0009 (7)
C10	0.0228 (8)	0.0236 (8)	0.0214 (8)	−0.0004 (7)	−0.0021 (7)	−0.0052 (7)
C11	0.0245 (8)	0.0239 (8)	0.0214 (9)	−0.0012 (7)	−0.0010 (7)	−0.0063 (7)
C12	0.0351 (10)	0.0273 (9)	0.0210 (9)	−0.0029 (8)	−0.0017 (7)	−0.0002 (7)
C13	0.0337 (10)	0.0273 (9)	0.0291 (10)	−0.0089 (8)	0.0042 (8)	−0.0039 (7)
C14	0.0242 (9)	0.0302 (9)	0.0343 (10)	−0.0063 (7)	0.0006 (7)	−0.0125 (8)
C15	0.0246 (8)	0.0262 (9)	0.0245 (9)	0.0000 (7)	−0.0024 (7)	−0.0082 (7)
C16	0.0234 (8)	0.0198 (8)	0.0194 (8)	−0.0006 (7)	−0.0006 (6)	−0.0042 (6)
C17	0.0285 (9)	0.0286 (10)	0.0218 (9)	−0.0036 (8)	−0.0015 (7)	−0.0057 (7)
C18	0.0370 (10)	0.0337 (10)	0.0264 (10)	−0.0039 (8)	−0.0054 (8)	−0.0050 (7)
C19	0.0602 (14)	0.0711 (16)	0.0292 (11)	−0.0247 (12)	−0.0036 (10)	−0.0141 (10)
C20	0.0410 (11)	0.0483 (12)	0.0520 (13)	−0.0158 (10)	−0.0110 (10)	−0.0109 (10)

Geometric parameters (Å, °)

O1—C5	1.3708 (19)	C7—C8	1.515 (3)
O1—C1	1.3818 (19)	C7—H7A	0.99
O2—C6	1.217 (2)	C7—H7B	0.99
O3—C10	1.224 (2)	C8—C9	1.522 (2)
O4—C18	1.232 (2)	C8—H8A	0.99
N1—C10	1.348 (2)	C8—H8B	0.99
N1—C11	1.404 (2)	C9—H9A	0.99
N1—H1	0.88	C9—H9B	0.99
N2—C17	1.154 (2)	C11—C12	1.377 (2)
N3—C5	1.334 (2)	C11—C16	1.395 (2)
N3—H3A	0.88	C12—C13	1.394 (3)
N3—H3B	0.88	C12—H12	0.95
N4—C18	1.324 (2)	C13—C14	1.386 (3)
N4—C19	1.451 (3)	C13—H13	0.95
N4—C20	1.453 (2)	C14—C15	1.390 (2)
C1—C2	1.334 (2)	C14—H14	0.95
C1—C9	1.489 (2)	C15—C16	1.378 (2)
C2—C6	1.476 (2)	C15—H15	0.95
C2—C3	1.507 (2)	C18—H18	0.95
C3—C16	1.512 (2)	C19—H19A	0.98
C3—C4	1.520 (2)	C19—H19B	0.98
C3—C10	1.562 (2)	C19—H19C	0.98
C4—C5	1.358 (2)	C20—H20A	0.98
C4—C17	1.421 (2)	C20—H20B	0.98
C6—C7	1.499 (2)	C20—H20C	0.98
C5—O1—C1	118.78 (12)	C1—C9—C8	111.18 (14)
C10—N1—C11	111.37 (13)	C1—C9—H9A	109.4
C10—N1—H1	124.3	C8—C9—H9A	109.4

C11—N1—H1	124.3	C1—C9—H9B	109.4
C5—N3—H3A	120.0	C8—C9—H9B	109.4
C5—N3—H3B	120.0	H9A—C9—H9B	108.0
H3A—N3—H3B	120.0	O3—C10—N1	126.42 (15)
C18—N4—C19	120.87 (16)	O3—C10—C3	125.40 (14)
C18—N4—C20	121.55 (16)	N1—C10—C3	108.17 (13)
C19—N4—C20	117.58 (16)	C12—C11—C16	121.73 (16)
C2—C1—O1	123.37 (14)	C12—C11—N1	128.21 (15)
C2—C1—C9	126.10 (15)	C16—C11—N1	110.03 (14)
O1—C1—C9	110.52 (13)	C11—C12—C13	117.63 (16)
C1—C2—C6	119.15 (14)	C11—C12—H12	121.2
C1—C2—C3	123.28 (14)	C13—C12—H12	121.2
C6—C2—C3	117.47 (14)	C14—C13—C12	121.03 (16)
C2—C3—C16	115.35 (13)	C14—C13—H13	119.5
C2—C3—C4	108.87 (13)	C12—C13—H13	119.5
C16—C3—C4	111.96 (13)	C13—C14—C15	120.71 (16)
C2—C3—C10	109.40 (13)	C13—C14—H14	119.6
C16—C3—C10	101.15 (12)	C15—C14—H14	119.6
C4—C3—C10	109.80 (13)	C16—C15—C14	118.60 (16)
C5—C4—C17	117.66 (15)	C16—C15—H15	120.7
C5—C4—C3	123.70 (14)	C14—C15—H15	120.7
C17—C4—C3	118.63 (14)	C15—C16—C11	120.26 (15)
N3—C5—C4	128.14 (15)	C15—C16—C3	131.25 (15)
N3—C5—O1	110.07 (13)	C11—C16—C3	108.49 (14)
C4—C5—O1	121.79 (14)	N2—C17—C4	179.8 (2)
O2—C6—C2	120.16 (15)	O4—C18—N4	125.09 (17)
O2—C6—C7	122.53 (15)	O4—C18—H18	117.5
C2—C6—C7	117.30 (14)	N4—C18—H18	117.5
C6—C7—C8	111.72 (14)	N4—C19—H19A	109.5
C6—C7—H7A	109.3	N4—C19—H19B	109.5
C8—C7—H7A	109.3	H19A—C19—H19B	109.5
C6—C7—H7B	109.3	N4—C19—H19C	109.5
C8—C7—H7B	109.3	H19A—C19—H19C	109.5
H7A—C7—H7B	107.9	H19B—C19—H19C	109.5
C7—C8—C9	111.41 (14)	N4—C20—H20A	109.5
C7—C8—H8A	109.3	N4—C20—H20B	109.5
C9—C8—H8A	109.3	H20A—C20—H20B	109.5
C7—C8—H8B	109.3	N4—C20—H20C	109.5
C9—C8—H8B	109.3	H20A—C20—H20C	109.5
H8A—C8—H8B	108.0	H20B—C20—H20C	109.5
C5—O1—C1—C2	−0.4 (2)	O1—C1—C9—C8	−162.11 (14)
C5—O1—C1—C9	179.39 (13)	C7—C8—C9—C1	−45.1 (2)
O1—C1—C2—C6	−179.06 (14)	C11—N1—C10—O3	173.86 (15)
C9—C1—C2—C6	1.1 (2)	C11—N1—C10—C3	−7.13 (18)
O1—C1—C2—C3	4.5 (2)	C2—C3—C10—O3	−50.0 (2)
C9—C1—C2—C3	−175.34 (15)	C16—C3—C10—O3	−172.14 (15)
C1—C2—C3—C16	−131.48 (16)	C4—C3—C10—O3	69.4 (2)
C6—C2—C3—C16	52.0 (2)	C2—C3—C10—N1	130.99 (14)
C1—C2—C3—C4	−4.7 (2)	C16—C3—C10—N1	8.84 (16)

C6—C2—C3—C4	178.81 (13)	C4—C3—C10—N1	−109.59 (14)
C1—C2—C3—C10	115.34 (17)	C10—N1—C11—C12	−176.03 (16)
C6—C2—C3—C10	−61.19 (17)	C10—N1—C11—C16	2.15 (19)
C2—C3—C4—C5	1.5 (2)	C16—C11—C12—C13	−1.5 (2)
C16—C3—C4—C5	130.23 (16)	N1—C11—C12—C13	176.47 (16)
C10—C3—C4—C5	−118.26 (17)	C11—C12—C13—C14	−0.1 (3)
C2—C3—C4—C17	−179.28 (14)	C12—C13—C14—C15	1.4 (3)
C16—C3—C4—C17	−50.54 (19)	C13—C14—C15—C16	−1.1 (2)
C10—C3—C4—C17	60.97 (18)	C14—C15—C16—C11	−0.5 (2)
C17—C4—C5—N3	3.3 (3)	C14—C15—C16—C3	178.92 (15)
C3—C4—C5—N3	−177.44 (15)	C12—C11—C16—C15	1.9 (2)
C17—C4—C5—O1	−177.17 (14)	N1—C11—C16—C15	−176.44 (14)
C3—C4—C5—O1	2.1 (2)	C12—C11—C16—C3	−177.70 (14)
C1—O1—C5—N3	176.78 (13)	N1—C11—C16—C3	3.99 (18)
C1—O1—C5—C4	−2.8 (2)	C2—C3—C16—C15	55.1 (2)
C1—C2—C6—O2	−172.10 (16)	C4—C3—C16—C15	−70.2 (2)
C3—C2—C6—O2	4.6 (2)	C10—C3—C16—C15	172.96 (17)
C1—C2—C6—C7	8.8 (2)	C2—C3—C16—C11	−125.43 (15)
C3—C2—C6—C7	−174.53 (14)	C4—C3—C16—C11	109.32 (15)
O2—C6—C7—C8	143.83 (18)	C10—C3—C16—C11	−7.52 (16)
C2—C6—C7—C8	−37.1 (2)	C19—N4—C18—O4	−1.7 (3)
C6—C7—C8—C9	55.4 (2)	C20—N4—C18—O4	179.26 (18)
C2—C1—C9—C8	17.7 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O4^{i}	0.88	1.99	2.767 (2)	147
N3—H3A···N2^{ii}	0.88	2.31	3.130 (2)	155
N3—H3B···O3^{iii}	0.88	2.06	2.897 (2)	158
C20—H20A···O2	0.98	2.50	3.464 (3)	167

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+2, −*y*, −*z*; (iii) −*x*+2, −*y*+1, −*z*.

Fig. 1

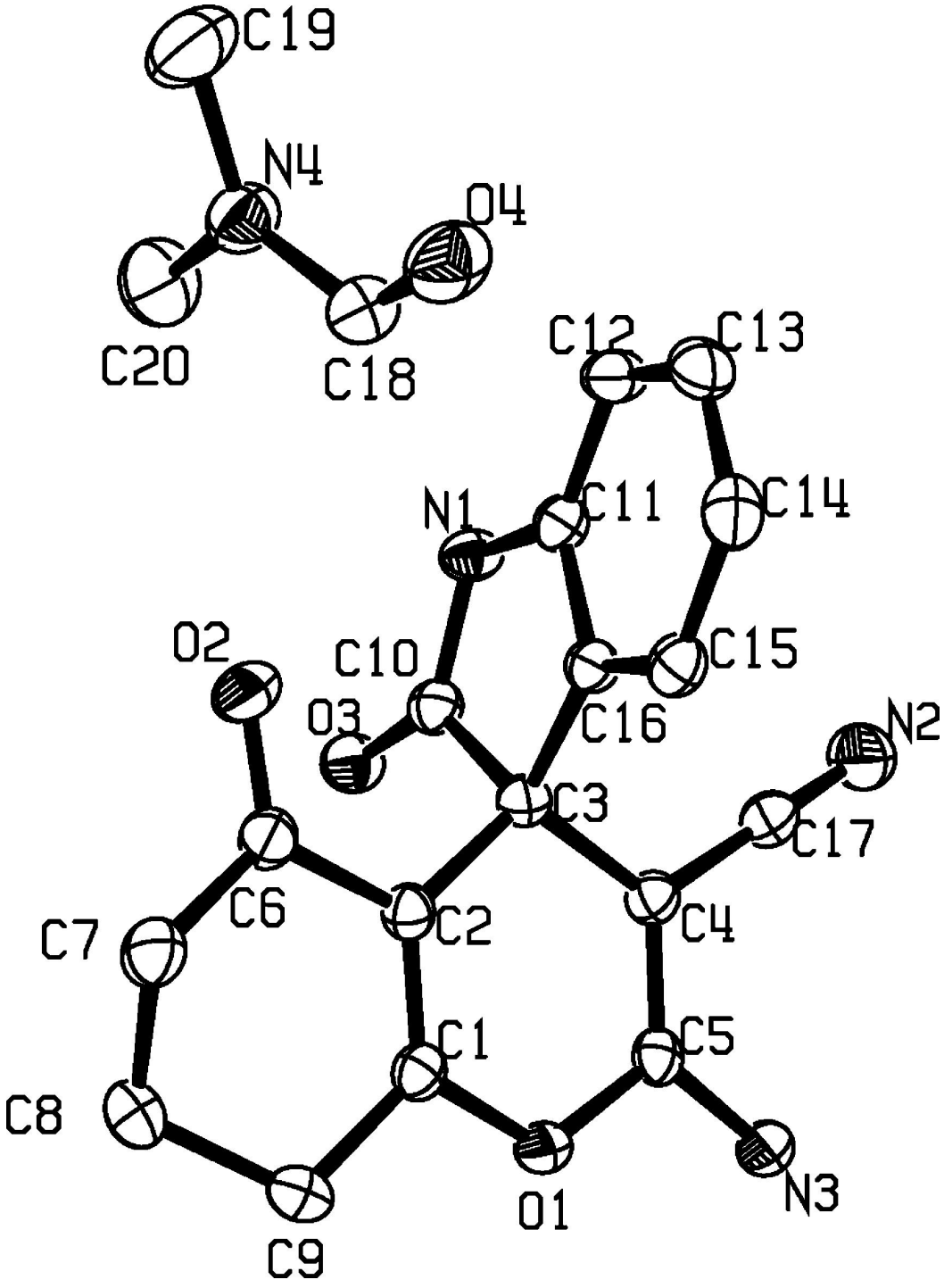
C19
N4
O4
C20
C18
C12
C13
N1
C11
C14
O2
C10
C15
O3
C16
N2
C3
C17
C6
C2
C4
C7
C1
C5
C8
O1
N3
C9

Fig. 2