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10-(4-Hydroxy-3-methoxy-5-nitrobenzylidene)anthrone

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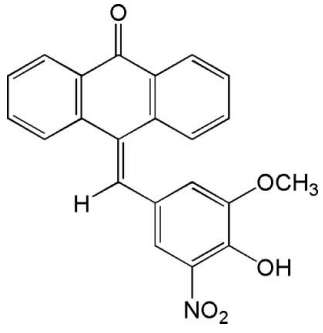
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.056; wR factor = 0.116; data-to-parameter ratio = 14.3.

In the title compound, $C_{22}H_{15}NO_5$, prepared from anthrone and 4-hydroxy-3-methoxy-5-nitrobenzaldehyde, the anthracene fragment is non-planar. The central six-membered ring assumes an asymmetric boat conformation, while the two outer benzene rings make a dihedral angle of 28.08 (10)°. The hydroxy group is involved in an intramolecular hydrogen bond. In the crystal structure, a weak intermolecular C—H···O hydrogen bond links the molecules into ribbons along the c axis.

Related literature

For related literature, see: Day (1963); Helge *et al.* (2003); Hu & Zhou (2004); Paull *et al.* (1992).



Experimental

Crystal data

$C_{22}H_{15}NO_5$
M_r = 373.35
Monoclinic, $P2_1/c$
a = 7.542 (3) Å
b = 27.810 (10) Å
c = 8.111 (3) Å
β = 97.257 (5)°
V = 1687.7 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
0.30 × 0.15 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.979, T_{max} = 0.988
8413 measured reflections
3691 independent reflections
2125 reflections with $I > 2\sigma(I)$
R_{int} = 0.060

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.116
S = 0.92
3691 reflections
258 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4X···O3	0.86 (3)	1.78 (3)	2.571 (3)	152 (3)
C22—H22C···O5^{i}	0.96	2.56	3.518 (3)	173

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); 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: CV2325).

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supplementary materials

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10-(4-Hydroxy-3-methoxy-5-nitrobenzylidene)anthrone

W. Zhou, Z. Wu, W. Hu and C. Xia

Comment

10-Substituted benzylideneanthrones have been known for a long time for their widespread use as functional dye and disperse dye (Day, 1963). Recently, some 10-substituted benzylideneanthrones have been found to possess a high antitumor activity (Paull *et al.*, 1992; Helge *et al.*, 2003). Owing to our interest in this area, we have prepared a series of 10-substituted benzylideneanthrones, and evaluated their anticancer activity. Our study on the structure-activity relationship (SAR) showed that the substitutent in phenyl moiety of the molecule affects its antitumor activity (Hu & Zhou, 2004). In continuation of our research work on SAR, we present here the crystal structure of the title compound, (I) (Fig. 1).

The three rings of anthraquinone moiety in (I) are not coplanar. The two outer benzene rings form a dihedral angle of 28.08 (10)°. The central six-membered ring assumes an asymmetric boat conformation with atoms C5 and C10 deviating from the plane at 0.322 (3) and 0.208 (3) Å, respectively. The plane of benzylidene moiety (C15—C21) forms a dihedral angle of 8.58 (11)° and 37.30 (8)° with the planes C1—C5/C11/C14 and C5—C9/C12/C13, respectively. The hydroxy and nitro groups are involved in intramolecluar hydrogen bond (Table 1).

In the crystal, obtained as a racemic mixture, the weak intermolecular C—H···O hydrogen bond (Table 1) links the molecules into ribbons along the *c* axis (Fig. 2).

Experimental

To a mixture of anthrone (1.60 g, 8.25 mmol) and 4-hydroxy-3-methoxy-5-nitrobenzaldehyde (1.70 g, 8.63 mmol) in 25 ml of absolute alcohol was slowly bubbled anhydrous hydrogen chloride. The mixture was refluxed until the TLC test showed that the reaction was complete. Then the mixture was cooled down to the room temperature. The precipitate was filtrated and washed with absolute alcohol, recrystallized with absolute alcohol to give red crystals of the title compound (2.5 g, yield 79.8%, m.p. 481–484 K). Since the crystal product was not found to be suitable for X-ray diffraction studies, a few crystals were dissolved in absolute ethanol, which was allowed to evaporate slowly to give red crystals of (I) suitable for X-ray diffraction studies.

Refinement

C-bound H atoms were placed in calculated positions (C—H 0.93–0.96 Å) and refined as riding, with U_{iso}(H) = 1.2–1.5Ueq of the parent atom. The hydroxy H atom was located in a difference map and refined isotropically with restaint O—H=0.86 (3) Å.

Figures

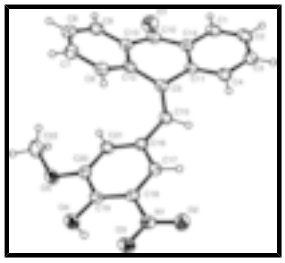

Fig. 1. The structure of (I) with 30% probability displacement ellipsoids.

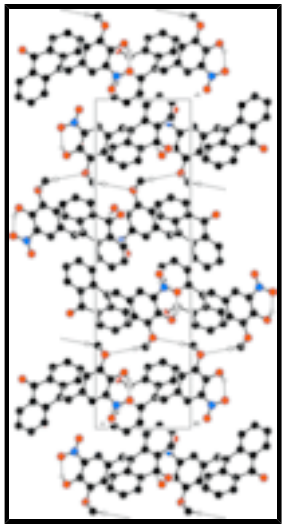

Fig. 2. Packing diagram of (I), viewed along the *a* axis. Dashed lines denote hydrogen bonds. For clarity, H atoms have been omitted except for those involved in hydrogen bonding.

10-(4-Hydroxy-3-methoxy-5-nitrobenzylidene)anthrone

Crystal data

$C_{22}H_{15}NO_5$	F_{000} = 776
M_r = 373.35	D_x = 1.469 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 645 reflections
a = 7.542 (3) Å	θ = 2.6–20.2°
b = 27.810 (10) Å	μ = 0.11 mm^{-1}
c = 8.111 (3) Å	T = 298 (2) K
β = 97.257 (5)°	Needle, red
V = 1687.7 (10) Å^3	0.30 × 0.15 × 0.10 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3691 independent reflections
Radiation source: fine-focus sealed tube	2125 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.060

T = 298(2) K	θ_{max} = 27.1º
φ and ω scans	θ_{min} = 1.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	h = −9→5
T_{min} = 0.979, T_{max} = 0.988	k = −33→35
8413 measured reflections	l = −9→10

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.056	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.116	$w = 1/[\sigma^2(F_o^2) + (0.037P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.92	$(\Delta/\sigma)_{max} < 0.001$
3691 reflections	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
258 parameters	$\Delta\rho_{min}$ = −0.21 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 $F^2 > 2\text{sigma}(F^2)$ 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	1.0555 (3)	0.64984 (6)	−0.2655 (2)	0.0685 (6)
O2	0.3735 (2)	0.53073 (6)	0.6703 (2)	0.0614 (5)
O3	0.3936 (2)	0.58518 (6)	0.8596 (2)	0.0580 (5)
O4	0.5069 (3)	0.66996 (7)	0.8018 (2)	0.0679 (6)
H4X	0.459 (4)	0.6465 (11)	0.850 (4)	0.107 (13)*
O5	0.6530 (2)	0.72449 (6)	0.6013 (2)	0.0636 (5)
N1	0.4187 (2)	0.57080 (7)	0.7197 (2)	0.0455 (5)
C1	0.9347 (3)	0.55343 (9)	−0.2948 (3)	0.0486 (6)
H1	0.9871	0.5657	−0.3837	0.058*
C2	0.8652 (3)	0.50819 (10)	−0.3044 (3)	0.0556 (7)
H2	0.8717	0.4895	−0.3985	0.067*

C3	0.7850 (3)	0.49035 (9)	−0.1734 (3)	0.0514 (6)
H3	0.7374	0.4594	−0.1794	0.062*
C4	0.7748 (3)	0.51779 (8)	−0.0342 (3)	0.0455 (6)
H4	0.7194	0.5053	0.0526	0.055*
C5	0.8392 (3)	0.59429 (7)	0.1292 (2)	0.0367 (5)
C6	1.0506 (3)	0.64585 (8)	0.3191 (3)	0.0446 (6)
H6	1.0080	0.6322	0.4111	0.054*
C7	1.1798 (3)	0.68127 (9)	0.3426 (3)	0.0529 (6)
H7	1.2221	0.6913	0.4497	0.064*
C8	1.2465 (3)	0.70188 (9)	0.2083 (3)	0.0570 (7)
H8	1.3309	0.7264	0.2240	0.068*
C9	1.1865 (3)	0.68571 (8)	0.0514 (3)	0.0514 (6)
H9	1.2320	0.6992	−0.0393	0.062*
C10	1.0131 (3)	0.62950 (8)	−0.1433 (3)	0.0452 (6)
C11	0.8460 (3)	0.56394 (8)	−0.0213 (3)	0.0369 (5)
C12	0.9829 (3)	0.63023 (7)	0.1605 (3)	0.0368 (5)
C13	1.0589 (3)	0.64947 (8)	0.0255 (3)	0.0397 (5)
C14	0.9285 (3)	0.58177 (8)	−0.1537 (3)	0.0402 (5)
C15	0.7018 (3)	0.58748 (8)	0.2178 (3)	0.0422 (6)
H15	0.6264	0.5625	0.1774	0.051*
C16	0.6457 (3)	0.61121 (8)	0.3653 (3)	0.0386 (5)
C17	0.5543 (3)	0.58345 (8)	0.4672 (3)	0.0398 (5)
H17	0.5247	0.5519	0.4378	0.048*
C18	0.5060 (3)	0.60242 (8)	0.6135 (3)	0.0379 (5)
C19	0.5437 (3)	0.64949 (8)	0.6604 (3)	0.0428 (6)
C20	0.6275 (3)	0.67848 (8)	0.5509 (3)	0.0427 (6)
C21	0.6749 (3)	0.65947 (8)	0.4073 (3)	0.0423 (6)
H21	0.7282	0.6793	0.3355	0.051*
C22	0.7791 (4)	0.75190 (9)	0.5260 (3)	0.0738 (9)
H22A	0.8903	0.7347	0.5336	0.111*
H22B	0.7976	0.7822	0.5822	0.111*
H22C	0.7350	0.7573	0.4113	0.111*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0878 (14)	0.0813 (13)	0.0387 (10)	−0.0103 (10)	0.0174 (10)	0.0143 (9)
O2	0.0745 (13)	0.0505 (11)	0.0633 (12)	−0.0172 (9)	0.0249 (10)	−0.0054 (9)
O3	0.0698 (12)	0.0653 (12)	0.0444 (11)	−0.0045 (9)	0.0287 (9)	−0.0022 (8)
O4	0.0867 (15)	0.0637 (13)	0.0617 (13)	−0.0198 (11)	0.0424 (11)	−0.0203 (10)
O5	0.0831 (13)	0.0455 (10)	0.0699 (13)	−0.0168 (9)	0.0395 (11)	−0.0149 (9)
N1	0.0433 (12)	0.0510 (13)	0.0441 (13)	0.0001 (9)	0.0133 (10)	0.0020 (10)
C1	0.0468 (15)	0.0680 (18)	0.0317 (13)	0.0102 (13)	0.0082 (11)	−0.0013 (12)
C2	0.0561 (17)	0.0702 (18)	0.0397 (15)	0.0116 (14)	0.0037 (13)	−0.0161 (13)
C3	0.0461 (15)	0.0538 (15)	0.0535 (16)	0.0043 (12)	0.0027 (13)	−0.0109 (12)
C4	0.0450 (15)	0.0515 (15)	0.0408 (14)	0.0029 (11)	0.0084 (12)	−0.0039 (11)
C5	0.0415 (13)	0.0411 (13)	0.0274 (12)	0.0043 (10)	0.0043 (10)	0.0026 (9)
C6	0.0447 (15)	0.0527 (15)	0.0359 (14)	0.0026 (11)	0.0031 (11)	0.0021 (11)

C7	0.0474 (15)	0.0617 (17)	0.0481 (15)	−0.0019 (13)	0.0001 (13)	−0.0083 (13)
C8	0.0451 (16)	0.0598 (17)	0.0673 (19)	−0.0092 (12)	0.0121 (14)	−0.0085 (14)
C9	0.0510 (16)	0.0495 (15)	0.0571 (17)	0.0015 (12)	0.0206 (13)	0.0049 (12)
C10	0.0449 (15)	0.0560 (15)	0.0354 (14)	0.0067 (11)	0.0086 (11)	0.0070 (11)
C11	0.0339 (13)	0.0443 (13)	0.0323 (13)	0.0082 (10)	0.0042 (10)	−0.0006 (10)
C12	0.0349 (13)	0.0409 (13)	0.0345 (13)	0.0064 (10)	0.0037 (10)	0.0028 (10)
C13	0.0397 (13)	0.0413 (13)	0.0392 (13)	0.0061 (10)	0.0089 (11)	0.0042 (10)
C14	0.0374 (13)	0.0524 (14)	0.0300 (13)	0.0098 (11)	0.0013 (10)	0.0017 (10)
C15	0.0464 (14)	0.0441 (14)	0.0367 (13)	−0.0013 (11)	0.0077 (11)	−0.0020 (10)
C16	0.0391 (13)	0.0441 (13)	0.0328 (12)	−0.0002 (10)	0.0056 (10)	−0.0015 (10)
C17	0.0389 (13)	0.0429 (13)	0.0377 (13)	−0.0014 (10)	0.0054 (11)	−0.0041 (10)
C18	0.0336 (12)	0.0447 (14)	0.0367 (13)	−0.0015 (10)	0.0101 (10)	0.0025 (10)
C19	0.0396 (13)	0.0516 (15)	0.0398 (14)	0.0003 (11)	0.0150 (11)	−0.0102 (11)
C20	0.0432 (14)	0.0403 (13)	0.0463 (14)	−0.0028 (10)	0.0115 (12)	−0.0037 (11)
C21	0.0448 (14)	0.0452 (14)	0.0390 (14)	−0.0010 (11)	0.0132 (11)	0.0028 (10)
C22	0.107 (2)	0.0527 (17)	0.068 (2)	−0.0253 (16)	0.0358 (18)	−0.0068 (14)

Geometric parameters (Å, °)

O1—C10	1.218 (2)	C7—C8	1.381 (3)
O2—N1	1.218 (2)	C7—H7	0.9300
O3—N1	1.240 (2)	C8—C9	1.371 (3)
O4—C19	1.340 (3)	C8—H8	0.9300
O4—H4X	0.86 (3)	C9—C13	1.391 (3)
O5—C20	1.350 (2)	C9—H9	0.9300
O5—C22	1.416 (3)	C10—C14	1.471 (3)
N1—C18	1.446 (3)	C10—C13	1.478 (3)
C1—C2	1.362 (3)	C11—C14	1.398 (3)
C1—C14	1.395 (3)	C12—C13	1.405 (3)
C1—H1	0.9300	C15—C16	1.474 (3)
C2—C3	1.380 (3)	C15—H15	0.9300
C2—H2	0.9300	C16—C17	1.378 (3)
C3—C4	1.373 (3)	C16—C21	1.395 (3)
C3—H3	0.9300	C17—C18	1.389 (3)
C4—C11	1.390 (3)	C17—H17	0.9300
C4—H4	0.9300	C18—C19	1.383 (3)
C5—C15	1.347 (3)	C19—C20	1.407 (3)
C5—C12	1.473 (3)	C20—C21	1.367 (3)
C5—C11	1.490 (3)	C21—H21	0.9300
C6—C7	1.382 (3)	C22—H22A	0.9599
C6—C12	1.392 (3)	C22—H22B	0.9599
C6—H6	0.9300	C22—H22C	0.9599
C19—O4—H4X	102 (2)	C14—C11—C5	119.54 (19)
C20—O5—C22	117.26 (18)	C6—C12—C13	117.6 (2)
O2—N1—O3	121.83 (19)	C6—C12—C5	123.17 (19)
O2—N1—C18	119.34 (19)	C13—C12—C5	119.20 (19)
O3—N1—C18	118.83 (19)	C9—C13—C12	120.0 (2)
C2—C1—C14	121.1 (2)	C9—C13—C10	119.3 (2)
C2—C1—H1	119.4	C12—C13—C10	120.6 (2)

C14—C1—H1	119.4	C1—C14—C11	119.6 (2)
C1—C2—C3	119.4 (2)	C1—C14—C10	119.8 (2)
C1—C2—H2	120.3	C11—C14—C10	120.53 (19)
C3—C2—H2	120.3	C5—C15—C16	133.5 (2)
C4—C3—C2	120.6 (2)	C5—C15—H15	113.2
C4—C3—H3	119.7	C16—C15—H15	113.2
C2—C3—H3	119.7	C17—C16—C21	117.88 (19)
C3—C4—C11	120.9 (2)	C17—C16—C15	116.9 (2)
C3—C4—H4	119.5	C21—C16—C15	125.22 (19)
C11—C4—H4	119.5	C16—C17—C18	120.3 (2)
C15—C5—C12	127.0 (2)	C16—C17—H17	119.8
C15—C5—C11	117.8 (2)	C18—C17—H17	119.8
C12—C5—C11	115.14 (18)	C19—C18—C17	121.83 (19)
C7—C6—C12	121.3 (2)	C19—C18—N1	120.36 (19)
C7—C6—H6	119.4	C17—C18—N1	117.8 (2)
C12—C6—H6	119.4	O4—C19—C18	125.4 (2)
C8—C7—C6	120.6 (2)	O4—C19—C20	117.0 (2)
C8—C7—H7	119.7	C18—C19—C20	117.54 (19)
C6—C7—H7	119.7	O5—C20—C21	125.6 (2)
C9—C8—C7	119.0 (2)	O5—C20—C19	114.26 (19)
C9—C8—H8	120.5	C21—C20—C19	120.2 (2)
C7—C8—H8	120.5	C20—C21—C16	122.0 (2)
C8—C9—C13	121.3 (2)	C20—C21—H21	119.0
C8—C9—H9	119.4	C16—C21—H21	119.0
C13—C9—H9	119.4	O5—C22—H22A	109.5
O1—C10—C14	122.0 (2)	O5—C22—H22B	109.5
O1—C10—C13	121.6 (2)	H22A—C22—H22B	109.5
C14—C10—C13	116.18 (19)	O5—C22—H22C	109.5
C4—C11—C14	118.3 (2)	H22A—C22—H22C	109.5
C4—C11—C5	122.13 (19)	H22B—C22—H22C	109.5
C14—C1—C2—C3	1.0 (4)	C4—C11—C14—C10	−177.2 (2)
C1—C2—C3—C4	0.0 (4)	C5—C11—C14—C10	1.9 (3)
C2—C3—C4—C11	−0.5 (4)	O1—C10—C14—C1	16.0 (3)
C12—C6—C7—C8	−0.6 (4)	C13—C10—C14—C1	−158.6 (2)
C6—C7—C8—C9	−1.9 (4)	O1—C10—C14—C11	−165.8 (2)
C7—C8—C9—C13	0.7 (4)	C13—C10—C14—C11	19.6 (3)
C3—C4—C11—C14	0.0 (3)	C12—C5—C15—C16	0.7 (4)
C3—C4—C11—C5	−179.1 (2)	C11—C5—C15—C16	−176.8 (2)
C15—C5—C11—C4	−29.3 (3)	C5—C15—C16—C17	−150.8 (2)
C12—C5—C11—C4	152.9 (2)	C5—C15—C16—C21	30.5 (4)
C15—C5—C11—C14	151.6 (2)	C21—C16—C17—C18	−5.0 (3)
C12—C5—C11—C14	−26.2 (3)	C15—C16—C17—C18	176.3 (2)
C7—C6—C12—C13	4.1 (3)	C16—C17—C18—C19	1.4 (3)
C7—C6—C12—C5	−177.0 (2)	C16—C17—C18—N1	−176.7 (2)
C15—C5—C12—C6	32.7 (3)	O2—N1—C18—C19	174.1 (2)
C11—C5—C12—C6	−149.8 (2)	O3—N1—C18—C19	−6.6 (3)
C15—C5—C12—C13	−148.3 (2)	O2—N1—C18—C17	−7.8 (3)
C11—C5—C12—C13	29.2 (3)	O3—N1—C18—C17	171.6 (2)
C8—C9—C13—C12	2.9 (3)	C17—C18—C19—O4	−177.7 (2)

C8—C9—C13—C10	−173.2 (2)	N1—C18—C19—O4	0.4 (4)
C6—C12—C13—C9	−5.2 (3)	C17—C18—C19—C20	2.4 (3)
C5—C12—C13—C9	175.8 (2)	N1—C18—C19—C20	−179.5 (2)
C6—C12—C13—C10	170.92 (19)	C22—O5—C20—C21	−18.0 (4)
C5—C12—C13—C10	−8.1 (3)	C22—O5—C20—C19	162.3 (2)
O1—C10—C13—C9	−15.0 (3)	O4—C19—C20—O5	−2.7 (3)
C14—C10—C13—C9	159.6 (2)	C18—C19—C20—O5	177.2 (2)
O1—C10—C13—C12	168.9 (2)	O4—C19—C20—C21	177.6 (2)
C14—C10—C13—C12	−16.5 (3)	C18—C19—C20—C21	−2.5 (3)
C2—C1—C14—C11	−1.5 (3)	O5—C20—C21—C16	179.1 (2)
C2—C1—C14—C10	176.7 (2)	C19—C20—C21—C16	−1.1 (4)
C4—C11—C14—C1	1.0 (3)	C17—C16—C21—C20	4.9 (3)
C5—C11—C14—C1	−179.9 (2)	C15—C16—C21—C20	−176.5 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4X···O3	0.86 (3)	1.78 (3)	2.571 (3)	152 (3)
C22—H22C···O5^{i}	0.96	2.56	3.518 (3)	173

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

Fig. 1

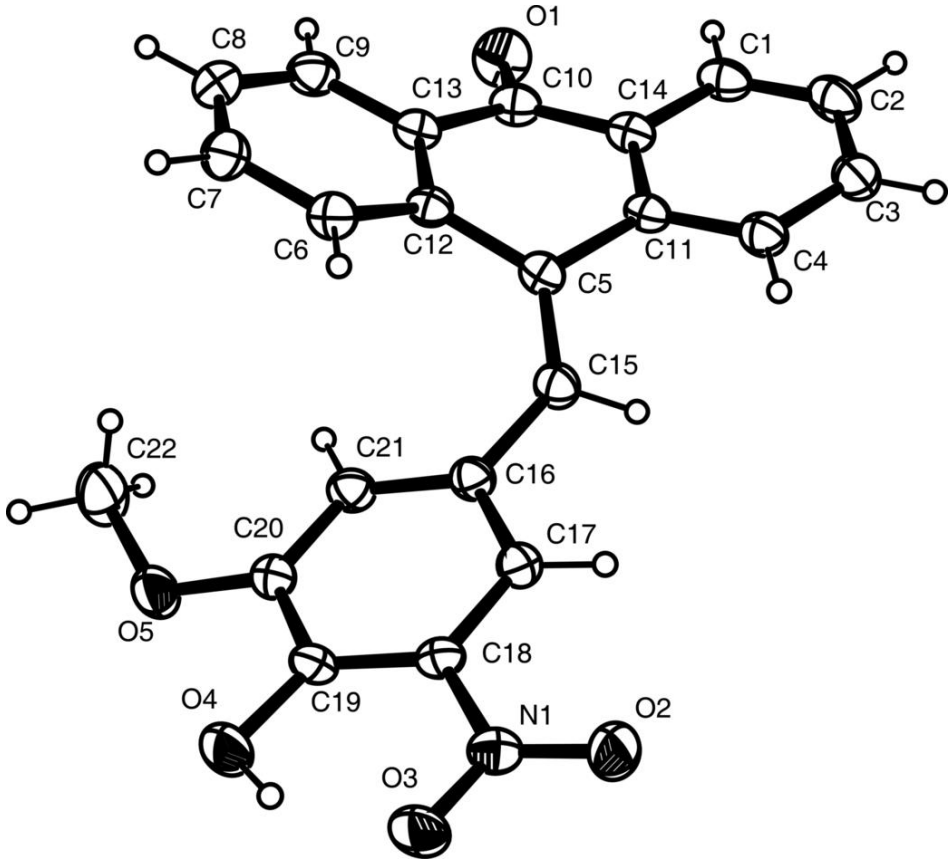
O1
C8
C9
C13
C10
C14
C1
C2
C7
C3
C6
C12
C11
C4
C5
C15
C22
C21
C16
C20
C17
O5
C18
O4
C19
N1
O2
O3

Fig. 2