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5-(4-Methoxyphenyl)-3,3-dimethyl-2,3,4,5-tetrahydro-1*H*-benzo[*b*]-carbazole-1,6,11-trione chloroform solvate

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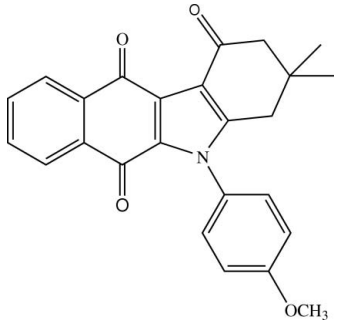
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.125; data-to-parameter ratio = 21.9.

In the molecular structure of the title compound, $C_{25}H_{21}NO_4\cdot CHCl_3$, the indolequinone unit is essentially planar and the cyclohexen-1-one ring is in an envelope conformation. The dihedral angle between the indole and methoxyphenyl groups is 81.78 (8)°. The molecules are linked *via* weak intermolecular C—H···O interactions to form dimers and these dimers are stacked approximately down the *b* axis. The crystal structure is stabilized by weak intermolecular C—H···O interactions and C—H···π interactions.

Related literature

For values of bond lengths, see Allen *et al.* (1987). For ring conformations, see Cremer & Pople (1975). For related structures, see, for example, Fun *et al.* (2007). For naturally occurring quinones and their activities, see, for example, Patai & Rappoport (1988); Thomson (1997); Bolton *et al.* (2000); Hu *et al.* (2006); Fun *et al.* (2007).



Experimental

Crystal data

$C_{25}H_{21}NO_4\cdot CHCl_3$
M_r = 518.80
Triclinic, $P\overline{1}$
a = 8.9167 (5) Å
b = 10.1639 (5) Å
c = 14.3550 (8) Å
α = 71.547 (3)°
β = 72.281 (3)°
γ = 85.367 (3)°
V = 1175.36 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.43 mm^{-1}
T = 100.0 (1) K
0.58 × 0.34 × 0.24 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.791, T_{max} = 0.903
16460 measured reflections
6780 independent reflections
5521 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.125
S = 1.05
6780 reflections
310 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10A···O3^{i}	0.93	2.55	3.238 (2)	131
C18—H18A···O3^{ii}	0.93	2.41	3.2333 (19)	148
C16—H16A···$Cg1^{ii}$	0.97	2.60	3.5242 (18)	160
C19—H19A···$Cg1^{iii}$	0.93	2.81	3.6035 (17)	143
C26—H26A···$Cg2^{iv}$	0.98	2.61	3.565 (2)	164

Symmetry codes: (i) $-x-1, -y+2, -z+2$; (ii) $-x, -y+2, -z+2$; (iii) $x+1, y, z$; (iv) $-x+1, -y+2, -z+1$. Note: $Cg1$ and $Cg2$ are the centroids of the C6–C11 and C17–C22 benzene rings, respectively.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2133).

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supplementary materials

Acta Cryst. (2007). E**63**, o3236-o3237 [**doi:10.1107/S1600536807028334**]

5-(4-Methoxyphenyl)-3,3-dimethyl-2,3,4,5-tetrahydro-1*H*-benzo[*b*]carbazole-1,6,11-trione chloroform solvate

H.-K. Fun, S. Chantrapromma, Y. Liu and J.-H. Xu

Comment

Naturally occurring quinones constitute an important class of natural products (Patai & Rappoport, 1988; Thomson, 1997) that have a wide range of biological activities (Bolton *et al.*, 2000). A basic structural unit in these quinine natural products is the indolequinone moiety. Synthesis of the benzannulated indolequinones, benzo[*b*]carbazole-6,11-dione, attracts much current attention (Hu *et al.*, 2006). As an extension of our research on the direct one-pot syntheses of benzo[*b*]carbazole-6,11-dione derivatives, the title compound was synthesized by a *C*,*N*-dialkylation reaction between 2,3-dichloro-1,4-naphthoquinone and 3-(4-methoxyphenylamino)-5,5- dimethylcyclohex-2-enone. An *x*-ray crystallographic analysis was undertaken to elucidate its three-dimensional structure.

In the asymmetric unit of the title compound in Fig. 1, the indolequinone moiety [N1/O2–O3/C3–C14] is essentially planar with atom C12 having the maximum deviation of 0.094 (1) Å and the cyclohexene-1-one ring [O4/C1–C3/C14–C16 ring adopts an envelope conformation (Cremer & Pople, 1975) with atom C1 displaced from the C2–C3/C14–C16 mean plane by −0.3155 (17) Å and with the puckering parameters Q = 0.454 (2) Å, θ = 132.1 (4)° and φ = 169.3 (3)°. The methoxy group is slightly deviated from the plane of C17–C22 benzene ring as indicated by the torsion angle C19/C20/O1/C23 = 4.5 (2)°. The methoxyphenyl is almost perpendicularly attached at atom N1, the dihedral angle between the indole and methoxyphenyl rings is 81.78 (8)°. All bond lengths and angles are in normal ranges (Allen *et al.*, 1987).

The crystal packing (Fig. 2) shows that the molecules are linked *via* weak C10—H10A···O3 [symmetry code; $-1-x, 2-y, 2-z$] and C18—H18A···O3 [symmetry code; $-x, 2-y, 2-z$] weak interactions to form dimers and these dimers are arranged into chains running approximately along the *b* axis (Fig. 2). The crystal is stabilized by weak C—H···O intermolecular interactions and C—H···π interactions (Table 1); Cg_1 and Cg_2 are the centroids of C6–C11 and C17–C22 benzene rings, respectively.

Experimental

A mixture of 2,3-dichloro-1,4-naphthoquinone (1.1 mmol), 3-(4-methoxyphenylamino)-5,5-dimethylcyclohex-2-enone (1 mmol) and Na_2CO_3 (2.5 mmol) in dimethylformamide (15 ml) was stirred at 353 K for 6 h. After evaporation of the solvent, the title compound was isolated using silica-gel column chromatography with petroleum ether-ethyl acetate (4:1) as eluents (yield 83%). Single crystals of the title compound in yellow block shape were obtained by slow evaporation of a petroleum ether/ethyl acetate (3:1 *v*/*v*) solution of the title compound; m. p. 531–533 K.

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the

remaining H atoms. A rotating group model was used for the methyl groups. The highest residual peak is located 0.75 Å from Cl3 and the deepest hole is located 0.69 Å from Cl3.

Figures

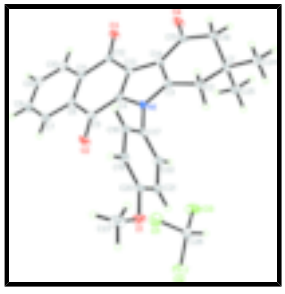

Fig. 1. The asymmetric unit showing 50% probability displacement ellipsoids and the atomic numbering scheme.

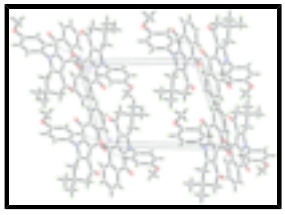

Fig. 2. The crystal packing of viewed along the *a* axis. C—H···O weak interactions are drawn as dashed lines. $CHCl_3$ molecules were omitted for clarity.

5-(4-Methoxyphenyl)-3,3-dimethyl-2,3,4,5-tetrahydro- 1*H*-benzo[*b*]carbazole-1,6,11-trione chloroform solvate

Crystal data

$C_{25}H_{21}NO_4 \cdot CHCl_3$	$Z = 2$
$M_r = 518.80$	$F_{000} = 536$
Triclinic, $P\overline{1}$	$D_x = 1.466$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.9167$ (5) Å	Cell parameters from 6780 reflections
$b = 10.1639$ (5) Å	$\theta = 2.2$–30.0°
$c = 14.3550$ (8) Å	$\mu = 0.43$ mm^{-1}
$\alpha = 71.547$ (3)°	$T = 100.0$ (1) K
$\beta = 72.281$ (3)°	Block, yellow
$\gamma = 85.367$ (3)°	0.58 × 0.34 × 0.24 mm
$V = 1175.36$ (11) Å^3	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	6780 independent reflections
Radiation source: fine-focus sealed tube	5521 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.038$
Detector resolution: 8.33 pixels mm^{-1}	$\theta_{max} = 30.0^\circ$
$T = 100.0(1)$ K	$\theta_{min} = 2.2^\circ$
ω scans	$h = -12 \rightarrow 12$
Absorption correction: multi-scan	$k = -14 \rightarrow 14$

(SADABS; Bruker, 2005)
T_{min} = 0.791, T_{max} = 0.903
16460 measured reflections

l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.125
S = 1.05
6780 reflections
310 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
$w = 1/[\sigma^2(F_o^2) + (0.0639P)^2 + 0.385P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}
Extinction correction: none

Special details

Experimental. The data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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}
Cl1	0.23402 (7)	0.79707 (5)	0.48120 (3)	0.03451 (13)
Cl2	0.24023 (5)	0.79013 (5)	0.27970 (4)	0.02991 (12)
Cl3	0.04991 (6)	0.99133 (5)	0.36617 (4)	0.03617 (13)
C26	0.2247 (2)	0.89613 (18)	0.35806 (13)	0.0234 (3)
H26A	0.3139	0.9620	0.3264	0.028*
O3	−0.27979 (13)	1.05545 (11)	0.98946 (9)	0.0188 (2)
O4	−0.11827 (14)	1.32503 (12)	0.86984 (10)	0.0232 (3)
N1	0.21722 (15)	0.97595 (12)	0.79625 (9)	0.0133 (2)
C1	0.27863 (18)	1.36341 (15)	0.69063 (11)	0.0158 (3)
C2	0.34101 (18)	1.21556 (15)	0.72643 (11)	0.0161 (3)
H2A	0.4042	1.1894	0.6671	0.019*
H2B	0.4076	1.2138	0.7691	0.019*
C3	0.20809 (17)	1.11473 (14)	0.78568 (11)	0.0135 (3)
C4	0.07375 (17)	0.91363 (14)	0.85738 (10)	0.0132 (3)

C5	0.03251 (18)	0.76871 (15)	0.87948 (11)	0.0155 (3)
C6	−0.13426 (18)	0.72965 (15)	0.94308 (11)	0.0148 (3)
C7	−0.18473 (19)	0.59248 (15)	0.96924 (12)	0.0172 (3)
H7A	−0.1148	0.5272	0.9483	0.021*
C8	−0.3395 (2)	0.55401 (16)	1.02656 (12)	0.0195 (3)
H8A	−0.3727	0.4624	1.0450	0.023*
C9	−0.44544 (19)	0.65209 (17)	1.05668 (12)	0.0194 (3)
H9A	−0.5497	0.6263	1.0934	0.023*
C10	−0.39547 (18)	0.78851 (16)	1.03195 (11)	0.0173 (3)
H10A	−0.4661	0.8534	1.0529	0.021*
C11	−0.23914 (17)	0.82815 (15)	0.97570 (11)	0.0139 (3)
C12	−0.18920 (17)	0.97554 (15)	0.95264 (11)	0.0138 (3)
C13	−0.02768 (17)	1.01407 (14)	0.88585 (11)	0.0129 (3)
C14	0.05774 (17)	1.14358 (14)	0.83969 (11)	0.0132 (3)
C15	0.01523 (18)	1.28831 (15)	0.83443 (11)	0.0151 (3)
C16	0.15193 (19)	1.39154 (15)	0.78256 (12)	0.0167 (3)
H16A	0.2021	1.3928	0.8335	0.020*
H16B	0.1103	1.4832	0.7590	0.020*
C17	0.35632 (17)	0.90683 (14)	0.75367 (11)	0.0137 (3)
C18	0.45051 (18)	0.83945 (15)	0.81394 (11)	0.0164 (3)
H18A	0.4260	0.8409	0.8814	0.020*
C19	0.58302 (18)	0.76889 (16)	0.77371 (12)	0.0178 (3)
H19A	0.6470	0.7234	0.8141	0.021*
C20	0.61841 (18)	0.76729 (15)	0.67262 (12)	0.0166 (3)
C21	0.52359 (19)	0.83835 (16)	0.61160 (12)	0.0191 (3)
H21A	0.5487	0.8389	0.5437	0.023*
C22	0.39252 (18)	0.90782 (15)	0.65204 (11)	0.0167 (3)
H22A	0.3291	0.9548	0.6116	0.020*
C23	0.8358 (2)	0.6179 (2)	0.68665 (15)	0.0313 (4)
H23A	0.9141	0.5713	0.6464	0.047*
H23B	0.7698	0.5506	0.7453	0.047*
H23C	0.8865	0.6765	0.7094	0.047*
C24	0.4156 (2)	1.46772 (16)	0.65440 (13)	0.0219 (3)
H24A	0.4970	1.4486	0.5989	0.033*
H24B	0.4570	1.4595	0.7104	0.033*
H24C	0.3785	1.5601	0.6313	0.033*
C25	0.2105 (2)	1.37711 (16)	0.60257 (12)	0.0204 (3)
H25A	0.2907	1.3582	0.5465	0.031*
H25B	0.1733	1.4696	0.5801	0.031*
H25C	0.1246	1.3121	0.6258	0.031*
O1	0.74217 (14)	0.70017 (13)	0.62578 (9)	0.0232 (3)
O2	0.12306 (14)	0.68601 (12)	0.84612 (10)	0.0236 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0441 (3)	0.0347 (2)	0.0262 (2)	0.0059 (2)	−0.01755 (19)	−0.00567 (17)
Cl2	0.0242 (2)	0.0402 (2)	0.0363 (2)	0.00893 (18)	−0.01432 (17)	−0.02350 (19)

Cl3	0.0386 (3)	0.0423 (3)	0.0352 (2)	0.0203 (2)	−0.0178 (2)	−0.0204 (2)
C26	0.0266 (8)	0.0230 (7)	0.0231 (7)	0.0032 (7)	−0.0114 (6)	−0.0076 (6)
O3	0.0156 (5)	0.0176 (5)	0.0237 (5)	0.0040 (4)	−0.0034 (4)	−0.0099 (4)
O4	0.0182 (5)	0.0169 (5)	0.0336 (6)	0.0049 (4)	−0.0032 (5)	−0.0119 (4)
N1	0.0127 (5)	0.0117 (5)	0.0159 (5)	0.0022 (4)	−0.0041 (4)	−0.0051 (4)
C1	0.0175 (7)	0.0121 (6)	0.0175 (6)	0.0011 (5)	−0.0051 (5)	−0.0044 (5)
C2	0.0140 (6)	0.0140 (6)	0.0191 (6)	0.0015 (5)	−0.0038 (5)	−0.0046 (5)
C3	0.0146 (6)	0.0118 (6)	0.0157 (6)	0.0027 (5)	−0.0064 (5)	−0.0053 (5)
C4	0.0130 (6)	0.0127 (6)	0.0145 (6)	0.0023 (5)	−0.0042 (5)	−0.0054 (5)
C5	0.0160 (6)	0.0135 (6)	0.0183 (6)	0.0022 (5)	−0.0056 (5)	−0.0064 (5)
C6	0.0152 (6)	0.0141 (6)	0.0161 (6)	0.0004 (5)	−0.0055 (5)	−0.0053 (5)
C7	0.0205 (7)	0.0144 (6)	0.0194 (6)	0.0008 (5)	−0.0088 (6)	−0.0063 (5)
C8	0.0213 (7)	0.0179 (7)	0.0216 (7)	−0.0034 (6)	−0.0094 (6)	−0.0053 (5)
C9	0.0161 (7)	0.0238 (7)	0.0189 (7)	−0.0026 (6)	−0.0064 (6)	−0.0053 (6)
C10	0.0141 (7)	0.0213 (7)	0.0175 (6)	0.0017 (6)	−0.0059 (5)	−0.0067 (5)
C11	0.0129 (6)	0.0156 (6)	0.0139 (6)	0.0010 (5)	−0.0045 (5)	−0.0050 (5)
C12	0.0145 (6)	0.0151 (6)	0.0139 (6)	0.0034 (5)	−0.0061 (5)	−0.0061 (5)
C13	0.0128 (6)	0.0128 (6)	0.0146 (6)	0.0014 (5)	−0.0049 (5)	−0.0057 (5)
C14	0.0136 (6)	0.0117 (6)	0.0165 (6)	0.0028 (5)	−0.0056 (5)	−0.0069 (5)
C15	0.0186 (7)	0.0126 (6)	0.0167 (6)	0.0035 (5)	−0.0071 (5)	−0.0066 (5)
C16	0.0193 (7)	0.0117 (6)	0.0201 (6)	0.0014 (5)	−0.0059 (6)	−0.0065 (5)
C17	0.0133 (6)	0.0116 (6)	0.0168 (6)	0.0030 (5)	−0.0043 (5)	−0.0060 (5)
C18	0.0179 (7)	0.0166 (6)	0.0158 (6)	0.0050 (5)	−0.0056 (5)	−0.0073 (5)
C19	0.0159 (7)	0.0189 (7)	0.0199 (7)	0.0074 (6)	−0.0071 (6)	−0.0075 (5)
C20	0.0141 (6)	0.0150 (6)	0.0205 (7)	0.0032 (5)	−0.0029 (5)	−0.0080 (5)
C21	0.0207 (7)	0.0206 (7)	0.0161 (6)	0.0045 (6)	−0.0038 (6)	−0.0084 (5)
C22	0.0162 (7)	0.0182 (6)	0.0171 (6)	0.0045 (5)	−0.0066 (5)	−0.0067 (5)
C23	0.0262 (9)	0.0337 (9)	0.0335 (9)	0.0189 (8)	−0.0087 (7)	−0.0141 (7)
C24	0.0217 (8)	0.0165 (7)	0.0248 (7)	−0.0030 (6)	−0.0048 (6)	−0.0040 (6)
C25	0.0248 (8)	0.0192 (7)	0.0178 (6)	0.0027 (6)	−0.0079 (6)	−0.0055 (5)
O1	0.0198 (5)	0.0269 (6)	0.0232 (5)	0.0118 (5)	−0.0045 (5)	−0.0126 (5)
O2	0.0204 (6)	0.0153 (5)	0.0331 (6)	0.0024 (4)	−0.0008 (5)	−0.0120 (4)

Geometric parameters (Å, °)

Cl1—C26	1.7591 (18)	C10—H10A	0.9300
Cl2—C26	1.7588 (18)	C11—C12	1.502 (2)
Cl3—C26	1.7598 (18)	C12—C13	1.470 (2)
C26—H26A	0.9800	C13—C14	1.433 (2)
O3—C12	1.2237 (17)	C14—C15	1.4732 (19)
O4—C15	1.2226 (19)	C15—C16	1.517 (2)
N1—C3	1.3686 (17)	C16—H16A	0.9700
N1—C4	1.3797 (18)	C16—H16B	0.9700
N1—C17	1.4404 (17)	C17—C18	1.377 (2)
C1—C25	1.526 (2)	C17—C22	1.392 (2)
C1—C24	1.536 (2)	C18—C19	1.3977 (19)
C1—C16	1.542 (2)	C18—H18A	0.9300
C1—C2	1.543 (2)	C19—C20	1.393 (2)
C2—C3	1.486 (2)	C19—H19A	0.9300

C2—H2A	0.9700	C20—O1	1.3611 (17)
C2—H2B	0.9700	C20—C21	1.398 (2)
C3—C14	1.3900 (19)	C21—C22	1.384 (2)
C4—C13	1.3845 (18)	C21—H21A	0.9300
C4—C5	1.458 (2)	C22—H22A	0.9300
C5—O2	1.2238 (17)	C23—O1	1.424 (2)
C5—C6	1.497 (2)	C23—H23A	0.9600
C6—C7	1.398 (2)	C23—H23B	0.9600
C6—C11	1.4054 (19)	C23—H23C	0.9600
C7—C8	1.388 (2)	C24—H24A	0.9600
C7—H7A	0.9300	C24—H24B	0.9600
C8—C9	1.395 (2)	C24—H24C	0.9600
C8—H8A	0.9300	C25—H25A	0.9600
C9—C10	1.392 (2)	C25—H25B	0.9600
C9—H9A	0.9300	C25—H25C	0.9600
C10—C11	1.399 (2)		
Cl2—C26—Cl1	111.01 (9)	C4—C13—C12	120.10 (13)
Cl2—C26—Cl3	110.59 (10)	C14—C13—C12	132.78 (12)
Cl1—C26—Cl3	110.73 (9)	C3—C14—C13	106.38 (12)
Cl2—C26—H26A	108.1	C3—C14—C15	119.49 (13)
Cl1—C26—H26A	108.1	C13—C14—C15	134.00 (13)
Cl3—C26—H26A	108.1	O4—C15—C14	123.93 (14)
C3—N1—C4	108.67 (11)	O4—C15—C16	121.28 (13)
C3—N1—C17	125.14 (12)	C14—C15—C16	114.78 (12)
C4—N1—C17	126.15 (12)	C15—C16—C1	115.60 (12)
C25—C1—C24	109.86 (13)	C15—C16—H16A	108.4
C25—C1—C16	110.76 (13)	C1—C16—H16A	108.4
C24—C1—C16	108.88 (13)	C15—C16—H16B	108.4
C25—C1—C2	109.91 (13)	C1—C16—H16B	108.4
C24—C1—C2	108.67 (13)	H16A—C16—H16B	107.4
C16—C1—C2	108.73 (11)	C18—C17—C22	120.88 (13)
C3—C2—C1	110.49 (12)	C18—C17—N1	119.88 (13)
C3—C2—H2A	109.6	C22—C17—N1	119.24 (13)
C1—C2—H2A	109.6	C17—C18—C19	119.99 (14)
C3—C2—H2B	109.6	C17—C18—H18A	120.0
C1—C2—H2B	109.6	C19—C18—H18A	120.0
H2A—C2—H2B	108.1	C20—C19—C18	119.46 (14)
N1—C3—C14	109.19 (13)	C20—C19—H19A	120.3
N1—C3—C2	124.21 (12)	C18—C19—H19A	120.3
C14—C3—C2	126.59 (13)	O1—C20—C19	124.76 (14)
N1—C4—C13	108.65 (12)	O1—C20—C21	115.24 (13)
N1—C4—C5	125.15 (12)	C19—C20—C21	120.00 (13)
C13—C4—C5	125.95 (13)	C22—C21—C20	120.16 (14)
O2—C5—C4	123.65 (14)	C22—C21—H21A	119.9
O2—C5—C6	121.99 (14)	C20—C21—H21A	119.9
C4—C5—C6	114.31 (12)	C21—C22—C17	119.48 (14)
C7—C6—C11	120.22 (14)	C21—C22—H22A	120.3
C7—C6—C5	118.61 (13)	C17—C22—H22A	120.3
C11—C6—C5	121.17 (13)	O1—C23—H23A	109.5

C8—C7—C6	119.81 (14)	O1—C23—H23B	109.5
C8—C7—H7A	120.1	H23A—C23—H23B	109.5
C6—C7—H7A	120.1	O1—C23—H23C	109.5
C7—C8—C9	120.29 (15)	H23A—C23—H23C	109.5
C7—C8—H8A	119.9	H23B—C23—H23C	109.5
C9—C8—H8A	119.9	C1—C24—H24A	109.5
C10—C9—C8	120.18 (14)	C1—C24—H24B	109.5
C10—C9—H9A	119.9	H24A—C24—H24B	109.5
C8—C9—H9A	119.9	C1—C24—H24C	109.5
C9—C10—C11	120.11 (14)	H24A—C24—H24C	109.5
C9—C10—H10A	119.9	H24B—C24—H24C	109.5
C11—C10—H10A	119.9	C1—C25—H25A	109.5
C10—C11—C6	119.36 (14)	C1—C25—H25B	109.5
C10—C11—C12	118.41 (13)	H25A—C25—H25B	109.5
C6—C11—C12	122.24 (13)	C1—C25—H25C	109.5
O3—C12—C13	123.66 (13)	H25A—C25—H25C	109.5
O3—C12—C11	120.47 (13)	H25B—C25—H25C	109.5
C13—C12—C11	115.86 (12)	C20—O1—C23	117.51 (13)
C4—C13—C14	107.12 (12)		
C25—C1—C2—C3	−73.84 (16)	C5—C4—C13—C12	6.2 (2)
C24—C1—C2—C3	165.91 (13)	O3—C12—C13—C4	172.17 (15)
C16—C1—C2—C3	47.54 (17)	C11—C12—C13—C4	−7.0 (2)
C4—N1—C3—C14	−0.31 (17)	O3—C12—C13—C14	−7.1 (3)
C17—N1—C3—C14	−177.96 (14)	C11—C12—C13—C14	173.71 (16)
C4—N1—C3—C2	178.90 (14)	N1—C3—C14—C13	0.41 (17)
C17—N1—C3—C2	1.2 (2)	C2—C3—C14—C13	−178.77 (15)
C1—C2—C3—N1	155.67 (14)	N1—C3—C14—C15	−175.94 (13)
C1—C2—C3—C14	−25.3 (2)	C2—C3—C14—C15	4.9 (2)
C3—N1—C4—C13	0.07 (17)	C4—C13—C14—C3	−0.37 (17)
C17—N1—C4—C13	177.69 (14)	C12—C13—C14—C3	179.00 (16)
C3—N1—C4—C5	174.60 (14)	C4—C13—C14—C15	175.22 (16)
C17—N1—C4—C5	−7.8 (2)	C12—C13—C14—C15	−5.4 (3)
N1—C4—C5—O2	0.6 (3)	C3—C14—C15—O4	171.65 (15)
C13—C4—C5—O2	174.24 (16)	C13—C14—C15—O4	−3.5 (3)
N1—C4—C5—C6	−176.57 (14)	C3—C14—C15—C16	−9.4 (2)
C13—C4—C5—C6	−3.0 (2)	C13—C14—C15—C16	175.42 (16)
O2—C5—C6—C7	3.4 (2)	O4—C15—C16—C1	−144.74 (15)
C4—C5—C6—C7	−179.37 (14)	C14—C15—C16—C1	36.32 (19)
O2—C5—C6—C11	−176.26 (16)	C25—C1—C16—C15	64.85 (17)
C4—C5—C6—C11	1.0 (2)	C24—C1—C16—C15	−174.25 (13)
C11—C6—C7—C8	0.8 (2)	C2—C1—C16—C15	−56.02 (18)
C5—C6—C7—C8	−178.84 (14)	C3—N1—C17—C18	97.14 (18)
C6—C7—C8—C9	1.1 (2)	C4—N1—C17—C18	−80.1 (2)
C7—C8—C9—C10	−1.9 (3)	C3—N1—C17—C22	−83.6 (2)
C8—C9—C10—C11	0.8 (2)	C4—N1—C17—C22	99.11 (18)
C9—C10—C11—C6	1.1 (2)	C22—C17—C18—C19	−1.1 (2)
C9—C10—C11—C12	−178.68 (14)	N1—C17—C18—C19	178.14 (14)
C7—C6—C11—C10	−1.9 (2)	C17—C18—C19—C20	−0.1 (2)
C5—C6—C11—C10	177.76 (14)	C18—C19—C20—O1	−178.73 (15)

C7—C6—C11—C12	177.89 (14)	C18—C19—C20—C21	1.3 (2)
C5—C6—C11—C12	−2.5 (2)	O1—C20—C21—C22	178.64 (15)
C10—C11—C12—O3	5.9 (2)	C19—C20—C21—C22	−1.4 (2)
C6—C11—C12—O3	−173.84 (15)	C20—C21—C22—C17	0.3 (2)
C10—C11—C12—C13	−174.91 (14)	C18—C17—C22—C21	1.0 (2)
C6—C11—C12—C13	5.3 (2)	N1—C17—C22—C21	−178.23 (14)
N1—C4—C13—C14	0.19 (17)	C19—C20—O1—C23	4.5 (2)
C5—C4—C13—C14	−174.29 (14)	C21—C20—O1—C23	−175.56 (16)
N1—C4—C13—C12	−179.28 (13)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10A···O3^{i}	0.93	2.55	3.238 (2)	131
C18—H18A···O3^{ii}	0.93	2.41	3.2333 (19)	148
C16—H16A···Cg1^{ii}	0.97	2.60	3.5242 (18)	160
C19—H19A···Cg1^{iii}	0.93	2.81	3.6035 (17)	143
C26—H26A···Cg2^{iv}	0.98	2.61	3.565 (2)	164

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

Fig. 1

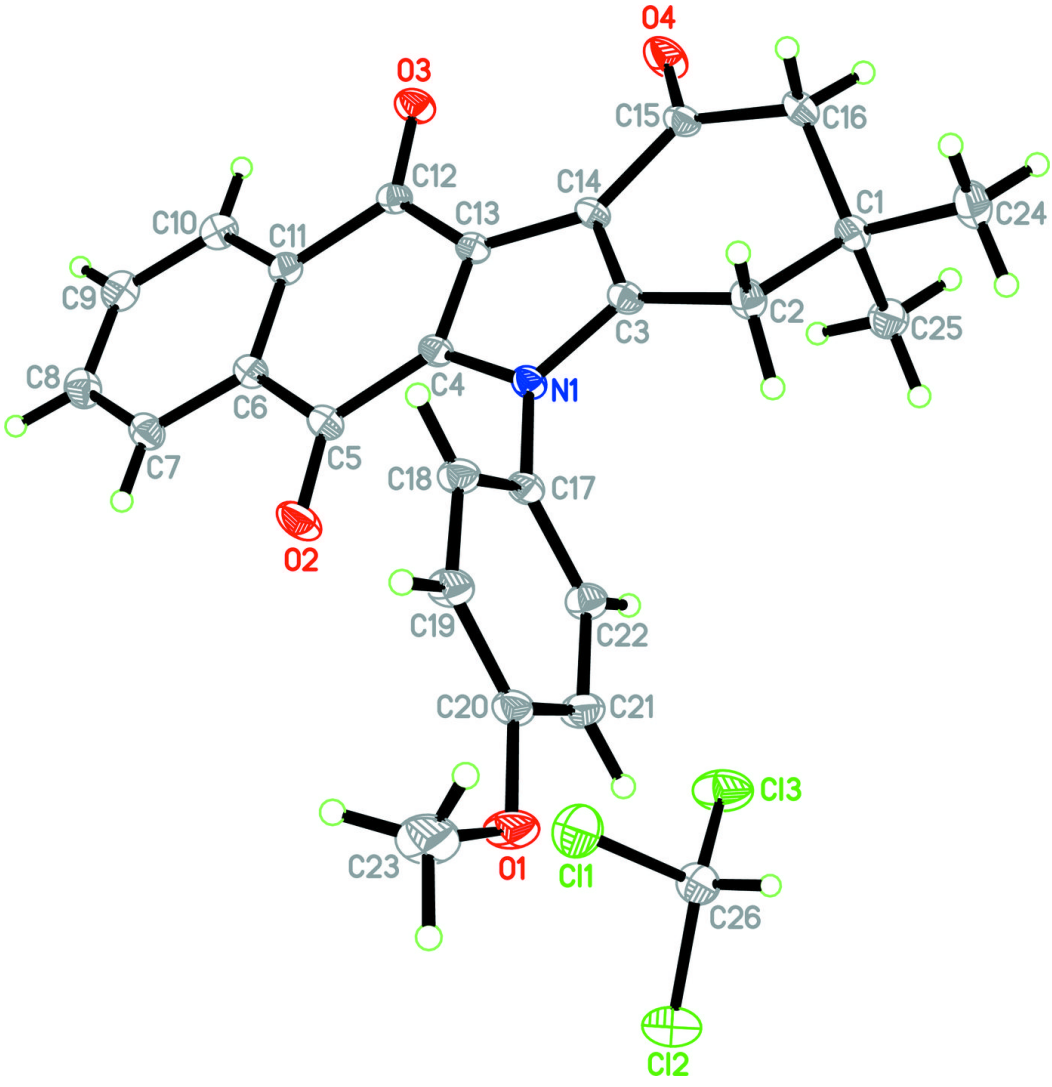
O4
O3
C15
C16
C12
C14
C1
C24
C10
C11
C13
C9
C3
C2
C25
C8
C4
N1
C6
C5
C7
C18
C17
O2
C19
C22
C20
C21
Cl3
C23
O1
Cl1
C26
Cl2

Fig. 2