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2-(4-Methoxyphenyl)phenanthro-[9,10-*d*]imidazole methanol solvate

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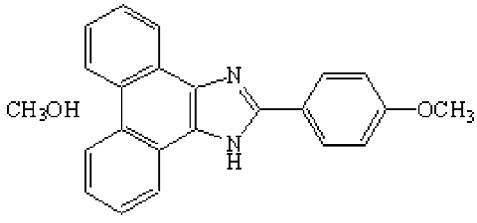
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.055; wR factor = 0.131; data-to-parameter ratio = 14.1.

The title compound, $C_{22}H_{16}N_2O\cdot CH_4O$, is a product of the condensation reaction between phenanthrenequinone and 4-methoxybenzadehyde. There are two imidazole molecules and two methanol molecules in the asymmetric unit. The phenanthryl and imidazole rings are almost parallel in both molecules, with interplanar angles of 6.65 (1) and 5.40 (3)°. The dihedral angles between the imidazole and the attached benzene rings are 5.40 (3) and 6.65 (1)° in the two molecules. Intermolecular O—H···N and N—H···O hydrogen bonds stabilize the crystal packing.

Related literature

For an example of fluorescence properties, see: Krebs & Spanggaard (2002). For a related structure, see: Krebs *et al.* (2001).



Experimental

Crystal data

$C_{22}H_{16}N_2O\cdot CH_4O$
M_r = 356.41
Monoclinic, $C2/c$
a = 17.755 (3) Å
b = 17.681 (3) Å
c = 25.131 (4) Å
β = 107.890 (3)°
V = 7508 (2) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
0.47 × 0.34 × 0.16 mm

Data collection

Bruker SMART CCD area detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.963, T_{max} = 0.987
19569 measured reflections
6972 independent reflections
3852 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.131
S = 0.95
6972 reflections
493 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4A···N4^{i}	0.82	1.94	2.755 (2)	173
O3—H3A···N2^{ii}	0.82	1.95	2.768 (2)	175
N3—H3···O3	0.86	1.99	2.840 (3)	168
N1—H1···O4	0.86	1.98	2.825 (2)	166

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2001); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge Dr Jianping Ma of Shandong Normal University for his help in the crystallographic analysis.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SQ2004).

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supporting information

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2-(4-Methoxyphenyl)phenanthro[9,10-*d*]imidazole methanol solvate

Fanpeng Kong, Yunliang Gao, Jibang Ling and Qingjian Liu

S1. Comment

The 1*H*-phenanthro[9,10 - d]imidazole is a promising building block in the field of molecular materials. It has many desirable properties such as good heat stability, ease of introduction into molecules used as chromophores with high extinction coefficient, readily tunable absorption wavelength, and fluorescent properties. For these reasons, the molecule is used as a large planar synthetic building block in supramolecular chemistry (Krebs & Spanggaard, 2002). As part of our studies of phenanthro[9,10 - d]imidazole derivatives, we report here the structure of the title compound (I), a 1:1 solvate with MeOH.

The bond lengths and angles in (I) agree well with those reported for the related compounds (Krebs *et al.*, 2001). There are two molecules in the asymmetric unit. The phenanthryl and imidazole rings in each molecule are almost parallel, with the interplanar angles being 6.65 (1)° and 5.40 (3)°.

There are intermolecular O4—H4A···N4, O3—H3A···N2, N3—H3···O3, and N1—H1···O4 close contacts (Table 2) in the crystal for (I). These contacts and the cross-linking interactions stabilize the crystal packing.

S2. Experimental

A mixture of phenanthrenequinone (4.161 g, 20 mmol), 4-methoxybenzaldehyde (2.723 g, 20 mmol), and ammonium acetate (7.708 g, 100 mmol) in acetic acid (50 ml) was refluxed for 1 h. Upon cooling to room temperature, the precipitate obtained on addition of water was purified by flash column chromatography on silica gel. Single crystals suitable for X-ray diffraction were obtained by recrystallization from methanol solution.

S3. Refinement

All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms attached to anisotropically refined atoms were placed in geometrically idealized positions and included as riding atoms with aromatic C—H = 0.93 Å and U_{iso}(H) = 1.2*U_{eq}(C); methyl C—H = 0.97 Å and U_{iso}(H) = 1.5*U_{eq}(C); O—H = 0.82 Å and U_{iso}(H) = 1.2*U_{eq}(O); N—H = 0.86 Å and U_{iso}(H) = 1.2*U_{eq}(N).

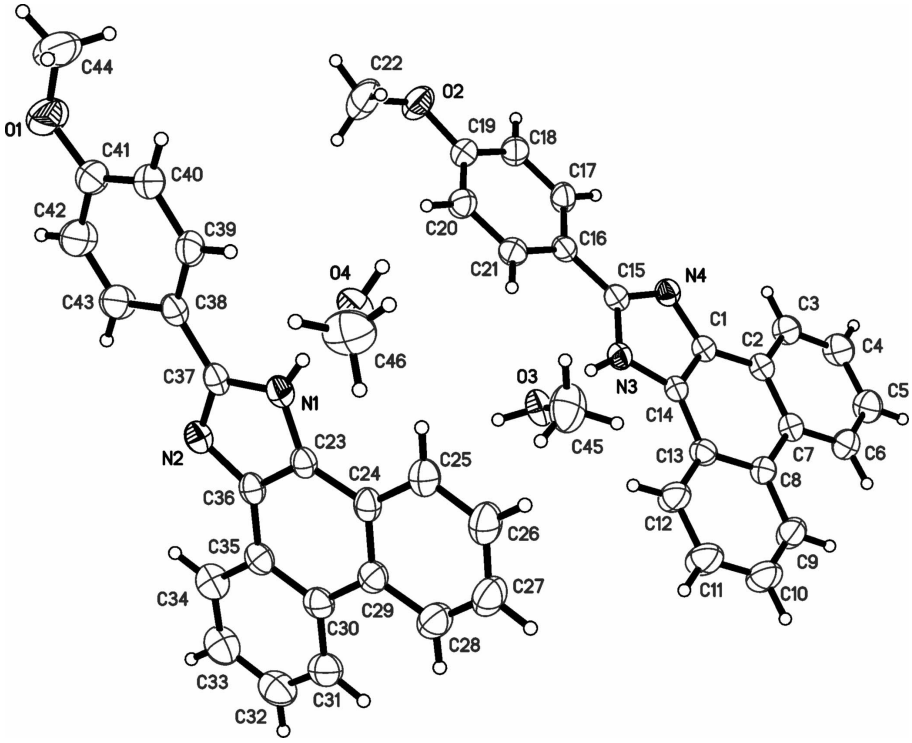

Figure 1

The molecular structure of (I) with the atom-numbering scheme and ellipsoids drawn at the 30% probability level. Both independent imidazole and methanol solvate molecules are shown.

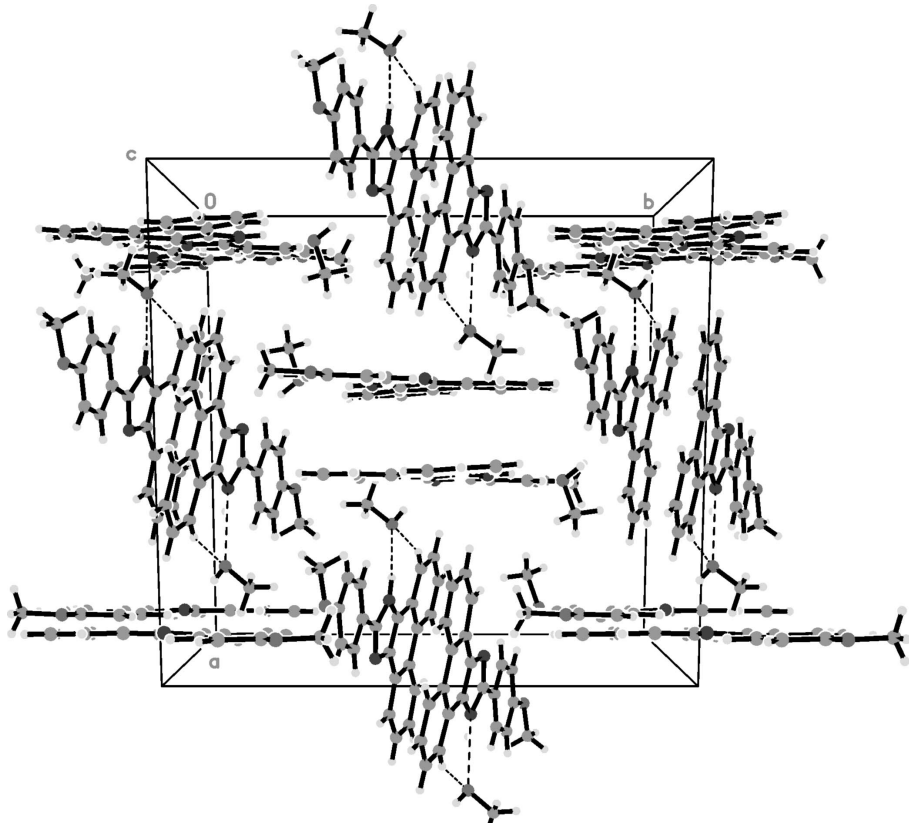

Figure 2

The packing diagram for (I) viewed along the *c* axis. Hydrogen bonds are shown as dashed lines.

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Crystal data

$C_{22}H_{16}N_2O\cdot CH_4O$
$M_r = 356.41$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 17.755\ (3)$ Å
$b = 17.681\ (3)$ Å
$c = 25.131\ (4)$ Å
$\beta = 107.890\ (3)°$
$V = 7508\ (2)$ Å^3
$Z = 16$
$F(000) = 3008$
$D_x = 1.261$ Mg m^{-3}
Melting point: 527 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2357 reflections
$\theta = 2.3–20.6°$
$\mu = 0.08$ mm^{-1}
$T = 298$ K
Plan, colourless
0.47 × 0.34 × 0.16 mm

Data collection

Bruker SMART CCD area detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.963$, $T_{max} = 0.987$
19569 measured reflections
6972 independent reflections
3852 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 25.5°$, $\theta_{min} = 1.7°$
$h = -20\rightarrow21$
$k = -18\rightarrow21$
$l = -30\rightarrow30$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.131$
$S = 0.95$
6972 reflections
493 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.13$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}

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^2against 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}
C1	0.00685 (14)	0.41421 (11)	0.03594 (9)	0.0457 (6)
C2	−0.05442 (14)	0.42635 (12)	0.06132 (10)	0.0488 (6)
C3	−0.13452 (14)	0.41336 (13)	0.03316 (11)	0.0612 (7)
H3B	−0.1494	0.3949	−0.0033	0.073*
C4	−0.19126 (16)	0.42739 (15)	0.05849 (12)	0.0730 (8)
H4	−0.2443	0.4185	0.0394	0.088*
C5	−0.16933 (17)	0.45483 (15)	0.11244 (13)	0.0717 (8)
H5	−0.2078	0.4641	0.1297	0.086*
C6	−0.09215 (16)	0.46835 (13)	0.14050 (11)	0.0637 (7)
H6	−0.0789	0.4868	0.1769	0.076*
C7	−0.03134 (15)	0.45537 (12)	0.11639 (10)	0.0518 (6)
C8	0.05131 (15)	0.47244 (12)	0.14516 (10)	0.0521 (6)
C9	0.07707 (17)	0.50153 (15)	0.19970 (11)	0.0696 (8)
H9	0.0399	0.5095	0.2184	0.084*
C10	0.15405 (19)	0.51847 (16)	0.22634 (12)	0.0838 (9)
H10	0.1687	0.5369	0.2628	0.101*
C11	0.21063 (18)	0.50848 (17)	0.19957 (12)	0.0917 (10)
H11	0.2633	0.5203	0.2177	0.110*
C12	0.18846 (16)	0.48101 (15)	0.14599 (11)	0.0729 (8)
H12	0.2265	0.4751	0.1278	0.087*
C13	0.11032 (15)	0.46176 (12)	0.11817 (10)	0.0523 (6)
C14	0.08366 (13)	0.43179 (11)	0.06299 (9)	0.0451 (6)
C15	0.07191 (14)	0.38688 (12)	−0.02125 (10)	0.0475 (6)
C16	0.09225 (14)	0.36201 (12)	−0.07017 (10)	0.0481 (6)
C17	0.03205 (15)	0.34411 (13)	−0.11865 (10)	0.0591 (7)

H17	−0.0203	0.3476	−0.1189	0.071*
C18	0.04807 (16)	0.32133 (13)	−0.16623 (10)	0.0641 (7)
H18	0.0067	0.3094	−0.1981	0.077*
C19	0.12526 (17)	0.31611 (13)	−0.16687 (10)	0.0589 (7)
C20	0.18619 (15)	0.33390 (13)	−0.11954 (10)	0.0597 (7)
H20	0.2384	0.3305	−0.1197	0.072*
C21	0.16935 (15)	0.35687 (12)	−0.07173 (10)	0.0568 (7)
H21	0.2108	0.3691	−0.0400	0.068*
C22	0.21299 (17)	0.28221 (18)	−0.21888 (12)	0.1021 (11)
H22A	0.2405	0.3297	−0.2122	0.153*
H22B	0.2110	0.2637	−0.2552	0.153*
H22C	0.2404	0.2464	−0.1909	0.153*
C23	0.59455 (13)	0.43889 (12)	0.05693 (9)	0.0468 (6)
C24	0.58717 (13)	0.43623 (13)	0.11163 (10)	0.0525 (6)
C25	0.57882 (15)	0.36890 (15)	0.13888 (11)	0.0660 (7)
H25	0.5761	0.3229	0.1204	0.079*
C26	0.57471 (17)	0.37032 (18)	0.19234 (12)	0.0803 (9)
H26	0.5697	0.3255	0.2103	0.096*
C27	0.57810 (17)	0.43864 (19)	0.21961 (12)	0.0822 (9)
H27	0.5763	0.4395	0.2562	0.099*
C28	0.58407 (16)	0.50466 (17)	0.19342 (11)	0.0754 (8)
H28	0.5845	0.5501	0.2122	0.090*
C29	0.58955 (14)	0.50622 (14)	0.13907 (10)	0.0554 (6)
C30	0.59432 (13)	0.57775 (14)	0.11076 (11)	0.0563 (6)
C31	0.59150 (15)	0.64903 (16)	0.13541 (12)	0.0706 (8)
H31	0.5885	0.6515	0.1717	0.085*
C32	0.59311 (17)	0.71454 (16)	0.10672 (15)	0.0826 (9)
H32	0.5905	0.7608	0.1236	0.099*
C33	0.59845 (17)	0.71294 (16)	0.05339 (14)	0.0807 (9)
H33	0.5998	0.7579	0.0345	0.097*
C34	0.60174 (15)	0.64507 (14)	0.02809 (12)	0.0681 (7)
H34	0.6051	0.6442	−0.0081	0.082*
C35	0.60016 (13)	0.57697 (13)	0.05601 (10)	0.0531 (6)
C36	0.60142 (13)	0.50504 (12)	0.03021 (10)	0.0470 (6)
C37	0.60503 (13)	0.41509 (13)	−0.02666 (9)	0.0472 (6)
C38	0.60895 (13)	0.37375 (13)	−0.07583 (9)	0.0504 (6)
C39	0.61133 (15)	0.29564 (14)	−0.07878 (10)	0.0606 (7)
H39	0.6117	0.2673	−0.0475	0.073*
C40	0.61311 (15)	0.25848 (14)	−0.12690 (11)	0.0659 (7)
H40	0.6144	0.2059	−0.1278	0.079*
C41	0.61303 (17)	0.29935 (17)	−0.17316 (11)	0.0722 (8)
C42	0.61049 (19)	0.37649 (17)	−0.17058 (11)	0.0900 (10)
H42	0.6103	0.4046	−0.2019	0.108*
C43	0.60826 (17)	0.41370 (15)	−0.12323 (11)	0.0749 (8)
H43	0.6063	0.4663	−0.1230	0.090*
C44	0.6210 (3)	0.1907 (2)	−0.22710 (14)	0.1336 (16)
H44A	0.6685	0.1726	−0.2001	0.200*
H44B	0.6219	0.1779	−0.2640	0.200*

H44C	0.5759	0.1677	−0.2203	0.200*
C45	0.3353 (2)	0.36715 (17)	0.10140 (14)	0.1058 (12)
H45A	0.3887	0.3845	0.1178	0.159*
H45B	0.3358	0.3191	0.0837	0.159*
H45C	0.3103	0.3618	0.1301	0.159*
C46	0.67782 (19)	0.20684 (18)	0.09661 (14)	0.1066 (11)
H46A	0.6914	0.2432	0.1264	0.160*
H46B	0.7200	0.2035	0.0803	0.160*
H46C	0.6698	0.1583	0.1111	0.160*
N1	0.59680 (11)	0.38208 (10)	0.02006 (7)	0.0503 (5)
H1	0.5936	0.3344	0.0256	0.060*
N2	0.60849 (11)	0.48982 (10)	−0.02171 (8)	0.0515 (5)
N3	0.12500 (11)	0.41427 (10)	0.02640 (7)	0.0496 (5)
H3	0.1751	0.4196	0.0324	0.060*
N4	−0.00083 (11)	0.38598 (10)	−0.01671 (7)	0.0498 (5)
O1	0.61638 (14)	0.26985 (12)	−0.22247 (8)	0.1066 (8)
O2	0.13454 (11)	0.29238 (10)	−0.21638 (7)	0.0811 (6)
O3	0.29293 (9)	0.42005 (10)	0.06127 (7)	0.0671 (5)
H3A	0.3239	0.4444	0.0497	0.101*
O4	0.60764 (11)	0.22969 (9)	0.05543 (8)	0.0670 (5)
H4A	0.5773	0.1936	0.0467	0.100*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0442 (16)	0.0433 (13)	0.0494 (15)	−0.0028 (11)	0.0143 (13)	0.0034 (11)
C2	0.0448 (16)	0.0468 (14)	0.0543 (16)	−0.0003 (11)	0.0144 (13)	0.0054 (11)
C3	0.0467 (17)	0.0692 (17)	0.0664 (18)	0.0000 (13)	0.0154 (15)	−0.0022 (13)
C4	0.0459 (18)	0.087 (2)	0.084 (2)	−0.0004 (15)	0.0162 (17)	−0.0036 (16)
C5	0.051 (2)	0.084 (2)	0.087 (2)	0.0026 (15)	0.0304 (18)	−0.0023 (16)
C6	0.063 (2)	0.0697 (18)	0.0632 (18)	0.0026 (14)	0.0262 (17)	−0.0007 (13)
C7	0.0510 (17)	0.0503 (14)	0.0555 (16)	−0.0016 (12)	0.0183 (14)	0.0050 (11)
C8	0.0555 (18)	0.0530 (15)	0.0488 (15)	−0.0019 (12)	0.0173 (14)	0.0027 (11)
C9	0.063 (2)	0.092 (2)	0.0567 (18)	−0.0030 (16)	0.0230 (16)	−0.0077 (14)
C10	0.069 (2)	0.118 (3)	0.0590 (19)	−0.0111 (19)	0.0118 (18)	−0.0222 (16)
C11	0.060 (2)	0.138 (3)	0.073 (2)	−0.0167 (18)	0.0150 (18)	−0.0346 (19)
C12	0.055 (2)	0.102 (2)	0.0629 (19)	−0.0105 (16)	0.0195 (16)	−0.0223 (15)
C13	0.0489 (17)	0.0533 (15)	0.0523 (16)	−0.0058 (12)	0.0123 (14)	−0.0017 (11)
C14	0.0455 (16)	0.0445 (14)	0.0473 (15)	−0.0063 (11)	0.0170 (13)	0.0016 (10)
C15	0.0456 (16)	0.0460 (14)	0.0501 (15)	−0.0043 (11)	0.0138 (13)	0.0033 (11)
C16	0.0473 (16)	0.0485 (14)	0.0473 (15)	−0.0077 (11)	0.0128 (13)	0.0022 (11)
C17	0.0495 (17)	0.0760 (18)	0.0534 (16)	−0.0096 (13)	0.0184 (14)	0.0026 (13)
C18	0.0570 (19)	0.0855 (19)	0.0462 (16)	−0.0162 (15)	0.0105 (14)	−0.0049 (13)
C19	0.066 (2)	0.0627 (16)	0.0512 (17)	−0.0064 (14)	0.0231 (16)	−0.0037 (12)
C20	0.0525 (18)	0.0690 (17)	0.0592 (17)	−0.0027 (13)	0.0197 (15)	−0.0057 (13)
C21	0.0506 (17)	0.0628 (16)	0.0551 (16)	−0.0064 (12)	0.0131 (14)	−0.0037 (12)
C22	0.081 (3)	0.154 (3)	0.082 (2)	0.006 (2)	0.041 (2)	−0.020 (2)
C23	0.0355 (14)	0.0550 (15)	0.0495 (15)	−0.0035 (11)	0.0124 (12)	0.0043 (12)

C24	0.0398 (15)	0.0668 (17)	0.0517 (15)	−0.0008 (12)	0.0153 (13)	0.0077 (13)
C25	0.069 (2)	0.0697 (18)	0.0633 (18)	−0.0016 (14)	0.0257 (16)	0.0096 (13)
C26	0.089 (2)	0.093 (2)	0.065 (2)	−0.0011 (17)	0.0319 (19)	0.0168 (17)
C27	0.086 (2)	0.109 (3)	0.0541 (19)	−0.0026 (19)	0.0253 (18)	0.0034 (18)
C28	0.071 (2)	0.096 (2)	0.0592 (19)	−0.0017 (16)	0.0200 (16)	−0.0068 (16)
C29	0.0391 (15)	0.0707 (18)	0.0556 (16)	−0.0012 (12)	0.0134 (13)	−0.0030 (13)
C30	0.0380 (15)	0.0638 (17)	0.0649 (18)	−0.0021 (12)	0.0126 (13)	−0.0049 (13)
C31	0.0551 (19)	0.076 (2)	0.081 (2)	0.0004 (15)	0.0211 (17)	−0.0118 (16)
C32	0.071 (2)	0.063 (2)	0.115 (3)	0.0017 (16)	0.029 (2)	−0.0114 (18)
C33	0.075 (2)	0.060 (2)	0.110 (3)	0.0066 (15)	0.032 (2)	0.0086 (17)
C34	0.0615 (19)	0.0601 (18)	0.082 (2)	0.0070 (14)	0.0211 (16)	0.0093 (15)
C35	0.0343 (14)	0.0578 (16)	0.0646 (17)	0.0017 (12)	0.0114 (13)	0.0033 (13)
C36	0.0315 (14)	0.0555 (16)	0.0537 (16)	0.0005 (11)	0.0124 (12)	0.0065 (12)
C37	0.0374 (14)	0.0547 (16)	0.0485 (15)	−0.0067 (11)	0.0117 (12)	0.0088 (12)
C38	0.0416 (15)	0.0579 (16)	0.0497 (16)	−0.0085 (12)	0.0108 (13)	0.0066 (12)
C39	0.0669 (19)	0.0655 (18)	0.0549 (17)	−0.0066 (14)	0.0267 (15)	0.0082 (13)
C40	0.075 (2)	0.0658 (17)	0.0630 (19)	−0.0090 (14)	0.0296 (16)	−0.0002 (14)
C41	0.086 (2)	0.082 (2)	0.0515 (18)	−0.0163 (17)	0.0256 (17)	−0.0037 (15)
C42	0.138 (3)	0.083 (2)	0.0513 (19)	−0.022 (2)	0.033 (2)	0.0096 (15)
C43	0.102 (2)	0.0657 (18)	0.0552 (18)	−0.0145 (16)	0.0213 (17)	0.0077 (14)
C44	0.224 (5)	0.101 (3)	0.099 (3)	−0.033 (3)	0.082 (3)	−0.034 (2)
C45	0.126 (3)	0.095 (2)	0.109 (3)	0.024 (2)	0.054 (3)	0.039 (2)
C46	0.070 (2)	0.126 (3)	0.103 (3)	0.009 (2)	−0.003 (2)	−0.017 (2)
N1	0.0485 (13)	0.0509 (12)	0.0520 (13)	−0.0066 (10)	0.0159 (11)	0.0070 (10)
N2	0.0432 (13)	0.0544 (13)	0.0557 (13)	−0.0025 (9)	0.0132 (11)	0.0076 (9)
N3	0.0436 (12)	0.0558 (12)	0.0510 (12)	−0.0090 (9)	0.0169 (11)	−0.0015 (9)
N4	0.0463 (13)	0.0532 (12)	0.0492 (13)	−0.0027 (9)	0.0135 (11)	0.0031 (9)
O1	0.167 (2)	0.1000 (17)	0.0643 (14)	−0.0238 (15)	0.0526 (15)	−0.0116 (11)
O2	0.0743 (15)	0.1156 (16)	0.0573 (12)	−0.0037 (11)	0.0261 (11)	−0.0177 (10)
O3	0.0538 (12)	0.0723 (12)	0.0782 (13)	−0.0097 (10)	0.0250 (11)	0.0134 (9)
O4	0.0602 (13)	0.0616 (11)	0.0740 (13)	−0.0136 (9)	0.0130 (11)	0.0048 (9)

Geometric parameters (Å, º)

C1—C14	1.360 (3)	C25—H25	0.9300
C1—N4	1.381 (3)	C26—C27	1.381 (4)
C1—C2	1.436 (3)	C26—H26	0.9300
C2—C3	1.399 (3)	C27—C28	1.360 (3)
C2—C7	1.414 (3)	C27—H27	0.9300
C3—C4	1.370 (3)	C28—C29	1.399 (3)
C3—H3B	0.9300	C28—H28	0.9300
C4—C5	1.379 (3)	C29—C30	1.466 (3)
C4—H4	0.9300	C30—C35	1.411 (3)
C5—C6	1.356 (3)	C30—C31	1.412 (3)
C5—H5	0.9300	C31—C32	1.369 (3)
C6—C7	1.410 (3)	C31—H31	0.9300
C6—H6	0.9300	C32—C33	1.373 (4)
C7—C8	1.454 (3)	C32—H32	0.9300

C8—C9	1.403 (3)	C33—C34	1.368 (3)
C8—C13	1.425 (3)	C33—H33	0.9300
C9—C10	1.358 (4)	C34—C35	1.398 (3)
C9—H9	0.9300	C34—H34	0.9300
C10—C11	1.382 (3)	C35—C36	1.431 (3)
C10—H10	0.9300	C36—N2	1.375 (3)
C11—C12	1.371 (3)	C37—N2	1.327 (3)
C11—H11	0.9300	C37—N1	1.358 (2)
C12—C13	1.391 (3)	C37—C38	1.456 (3)
C12—H12	0.9300	C38—C43	1.382 (3)
C13—C14	1.423 (3)	C38—C39	1.384 (3)
C14—N3	1.377 (2)	C39—C40	1.385 (3)
C15—N4	1.331 (3)	C39—H39	0.9300
C15—N3	1.365 (3)	C40—C41	1.368 (3)
C15—C16	1.452 (3)	C40—H40	0.9300
C16—C21	1.384 (3)	C41—O1	1.363 (3)
C16—C17	1.388 (3)	C41—C42	1.367 (3)
C17—C18	1.372 (3)	C42—C43	1.371 (3)
C17—H17	0.9300	C42—H42	0.9300
C18—C19	1.379 (3)	C43—H43	0.9300
C18—H18	0.9300	C44—O1	1.408 (3)
C19—O2	1.370 (3)	C44—H44A	0.9600
C19—C20	1.376 (3)	C44—H44B	0.9600
C20—C21	1.385 (3)	C44—H44C	0.9600
C20—H20	0.9300	C45—O3	1.411 (3)
C21—H21	0.9300	C45—H45A	0.9600
C22—O2	1.425 (3)	C45—H45B	0.9600
C22—H22A	0.9600	C45—H45C	0.9600
C22—H22B	0.9600	C46—O4	1.412 (3)
C22—H22C	0.9600	C46—H46A	0.9600
C23—C36	1.373 (3)	C46—H46B	0.9600
C23—N1	1.375 (3)	C46—H46C	0.9600
C23—C24	1.421 (3)	N1—H1	0.8600
C24—C25	1.404 (3)	N3—H3	0.8600
C24—C29	1.411 (3)	O3—H3A	0.8200
C25—C26	1.368 (3)	O4—H4A	0.8200

C14—C1—N4	110.51 (19)	C28—C27—C26	120.6 (3)
C14—C1—C2	121.6 (2)	C28—C27—H27	119.7
N4—C1—C2	127.9 (2)	C26—C27—H27	119.7
C3—C2—C7	119.9 (2)	C27—C28—C29	121.9 (3)
C3—C2—C1	122.8 (2)	C27—C28—H28	119.0
C7—C2—C1	117.2 (2)	C29—C28—H28	119.0
C4—C3—C2	120.9 (2)	C28—C29—C24	117.3 (2)
C4—C3—H3B	119.5	C28—C29—C30	121.5 (2)
C2—C3—H3B	119.5	C24—C29—C30	121.1 (2)
C3—C4—C5	119.7 (3)	C35—C30—C31	117.4 (2)
C3—C4—H4	120.2	C35—C30—C29	119.8 (2)

C5—C4—H4	120.2	C31—C30—C29	122.8 (2)
C6—C5—C4	120.6 (3)	C32—C31—C30	121.0 (3)
C6—C5—H5	119.7	C32—C31—H31	119.5
C4—C5—H5	119.7	C30—C31—H31	119.5
C5—C6—C7	122.2 (3)	C31—C32—C33	121.0 (3)
C5—C6—H6	118.9	C31—C32—H32	119.5
C7—C6—H6	118.9	C33—C32—H32	119.5
C6—C7—C2	116.7 (2)	C34—C33—C32	119.8 (3)
C6—C7—C8	122.8 (2)	C34—C33—H33	120.1
C2—C7—C8	120.4 (2)	C32—C33—H33	120.1
C9—C8—C13	116.6 (2)	C33—C34—C35	120.8 (3)
C9—C8—C7	122.6 (2)	C33—C34—H34	119.6
C13—C8—C7	120.9 (2)	C35—C34—H34	119.6
C10—C9—C8	122.7 (2)	C34—C35—C30	120.0 (2)
C10—C9—H9	118.7	C34—C35—C36	122.2 (2)
C8—C9—H9	118.7	C30—C35—C36	117.8 (2)
C9—C10—C11	120.3 (3)	C23—C36—N2	110.2 (2)
C9—C10—H10	119.9	C23—C36—C35	121.3 (2)
C11—C10—H10	119.9	N2—C36—C35	128.5 (2)
C12—C11—C10	119.4 (3)	N2—C37—N1	111.16 (19)
C12—C11—H11	120.3	N2—C37—C38	124.52 (19)
C10—C11—H11	120.3	N1—C37—C38	124.3 (2)
C11—C12—C13	121.5 (2)	C43—C38—C39	117.1 (2)
C11—C12—H12	119.3	C43—C38—C37	119.0 (2)
C13—C12—H12	119.3	C39—C38—C37	123.8 (2)
C12—C13—C14	124.3 (2)	C38—C39—C40	121.9 (2)
C12—C13—C8	119.6 (2)	C38—C39—H39	119.0
C14—C13—C8	116.1 (2)	C40—C39—H39	119.0
C1—C14—N3	105.9 (2)	C41—C40—C39	119.8 (2)
C1—C14—C13	123.7 (2)	C41—C40—H40	120.1
N3—C14—C13	130.5 (2)	C39—C40—H40	120.1
N4—C15—N3	110.84 (19)	O1—C41—C42	115.9 (2)
N4—C15—C16	124.7 (2)	O1—C41—C40	125.6 (3)
N3—C15—C16	124.5 (2)	C42—C41—C40	118.6 (2)
C21—C16—C17	117.5 (2)	C41—C42—C43	122.0 (2)
C21—C16—C15	123.3 (2)	C41—C42—H42	119.0
C17—C16—C15	119.2 (2)	C43—C42—H42	119.0
C18—C17—C16	121.5 (2)	C42—C43—C38	120.5 (2)
C18—C17—H17	119.3	C42—C43—H43	119.7
C16—C17—H17	119.3	C38—C43—H43	119.7
C17—C18—C19	120.2 (2)	O1—C44—H44A	109.5
C17—C18—H18	119.9	O1—C44—H44B	109.5
C19—C18—H18	119.9	H44A—C44—H44B	109.5
O2—C19—C20	125.0 (2)	O1—C44—H44C	109.5
O2—C19—C18	115.4 (2)	H44A—C44—H44C	109.5
C20—C19—C18	119.7 (2)	H44B—C44—H44C	109.5
C19—C20—C21	119.7 (2)	O3—C45—H45A	109.5
C19—C20—H20	120.2	O3—C45—H45B	109.5

C21—C20—H20	120.2	H45A—C45—H45B	109.5
C16—C21—C20	121.5 (2)	O3—C45—H45C	109.5
C16—C21—H21	119.2	H45A—C45—H45C	109.5
C20—C21—H21	119.2	H45B—C45—H45C	109.5
O2—C22—H22A	109.5	O4—C46—H46A	109.5
O2—C22—H22B	109.5	O4—C46—H46B	109.5
H22A—C22—H22B	109.5	H46A—C46—H46B	109.5
O2—C22—H22C	109.5	O4—C46—H46C	109.5
H22A—C22—H22C	109.5	H46A—C46—H46C	109.5
H22B—C22—H22C	109.5	H46B—C46—H46C	109.5
C36—C23—N1	105.50 (19)	C37—N1—C23	107.56 (18)
C36—C23—C24	123.4 (2)	C37—N1—H1	126.2
N1—C23—C24	131.1 (2)	C23—N1—H1	126.2
C25—C24—C29	119.8 (2)	C37—N2—C36	105.59 (17)
C25—C24—C23	123.7 (2)	C15—N3—C14	107.44 (18)
C29—C24—C23	116.5 (2)	C15—N3—H3	126.3
C26—C25—C24	120.6 (3)	C14—N3—H3	126.3
C26—C25—H25	119.7	C15—N4—C1	105.36 (19)
C24—C25—H25	119.7	C41—O1—C44	118.4 (2)
C25—C26—C27	119.8 (3)	C19—O2—C22	118.1 (2)
C25—C26—H26	120.1	C45—O3—H3A	109.5
C27—C26—H26	120.1	C46—O4—H4A	109.5

C14—C1—C2—C3	175.8 (2)	C27—C28—C29—C30	178.7 (2)
N4—C1—C2—C3	−3.1 (3)	C25—C24—C29—C28	0.6 (4)
C14—C1—C2—C7	−1.8 (3)	C23—C24—C29—C28	−178.7 (2)
N4—C1—C2—C7	179.4 (2)	C25—C24—C29—C30	−177.0 (2)
C7—C2—C3—C4	−0.9 (3)	C23—C24—C29—C30	3.8 (3)
C1—C2—C3—C4	−178.4 (2)	C28—C29—C30—C35	179.7 (2)
C2—C3—C4—C5	0.1 (4)	C24—C29—C30—C35	−2.8 (3)
C3—C4—C5—C6	0.3 (4)	C28—C29—C30—C31	−1.8 (4)
C4—C5—C6—C7	0.1 (4)	C24—C29—C30—C31	175.7 (2)
C5—C6—C7—C2	−0.8 (3)	C35—C30—C31—C32	1.0 (4)
C5—C6—C7—C8	177.5 (2)	C29—C30—C31—C32	−177.5 (2)
C3—C2—C7—C6	1.2 (3)	C30—C31—C32—C33	−0.8 (4)
C1—C2—C7—C6	178.88 (19)	C31—C32—C33—C34	0.4 (4)
C3—C2—C7—C8	−177.1 (2)	C32—C33—C34—C35	−0.3 (4)
C1—C2—C7—C8	0.5 (3)	C33—C34—C35—C30	0.6 (4)
C6—C7—C8—C9	1.5 (3)	C33—C34—C35—C36	178.3 (2)
C2—C7—C8—C9	179.8 (2)	C31—C30—C35—C34	−0.9 (3)
C6—C7—C8—C13	−177.0 (2)	C29—C30—C35—C34	177.6 (2)
C2—C7—C8—C13	1.3 (3)	C31—C30—C35—C36	−178.8 (2)
C13—C8—C9—C10	−0.5 (4)	C29—C30—C35—C36	−0.2 (3)
C7—C8—C9—C10	−179.1 (3)	N1—C23—C36—N2	−0.5 (2)
C8—C9—C10—C11	1.1 (5)	C24—C23—C36—N2	179.3 (2)
C9—C10—C11—C12	−0.3 (5)	N1—C23—C36—C35	179.0 (2)
C10—C11—C12—C13	−1.0 (5)	C24—C23—C36—C35	−1.1 (3)
C11—C12—C13—C14	−178.9 (3)	C34—C35—C36—C23	−175.6 (2)

C11—C12—C13—C8	1.6 (4)	C30—C35—C36—C23	2.1 (3)
C9—C8—C13—C12	−0.8 (3)	C34—C35—C36—N2	3.8 (4)
C7—C8—C13—C12	177.8 (2)	C30—C35—C36—N2	−178.4 (2)
C9—C8—C13—C14	179.6 (2)	N2—C37—C38—C43	−6.9 (3)
C7—C8—C13—C14	−1.8 (3)	N1—C37—C38—C43	172.7 (2)
N4—C1—C14—N3	0.1 (2)	N2—C37—C38—C39	174.9 (2)
C2—C1—C14—N3	−178.94 (18)	N1—C37—C38—C39	−5.6 (4)
N4—C1—C14—C13	−179.68 (19)	C43—C38—C39—C40	0.1 (4)
C2—C1—C14—C13	1.3 (3)	C37—C38—C39—C40	178.4 (2)
C12—C13—C14—C1	−179.0 (2)	C38—C39—C40—C41	0.4 (4)
C8—C13—C14—C1	0.5 (3)	C39—C40—C41—O1	178.5 (3)
C12—C13—C14—N3	1.3 (4)	C39—C40—C41—C42	−0.6 (4)
C8—C13—C14—N3	−179.2 (2)	O1—C41—C42—C43	−179.0 (3)
N4—C15—C16—C21	−173.7 (2)	C40—C41—C42—C43	0.2 (5)
N3—C15—C16—C21	6.2 (3)	C41—C42—C43—C38	0.4 (5)
N4—C15—C16—C17	8.1 (3)	C39—C38—C43—C42	−0.5 (4)
N3—C15—C16—C17	−172.0 (2)	C37—C38—C43—C42	−178.9 (3)
C21—C16—C17—C18	0.7 (3)	N2—C37—N1—C23	0.2 (2)
C15—C16—C17—C18	179.1 (2)	C38—C37—N1—C23	−179.4 (2)
C16—C17—C18—C19	−0.4 (4)	C36—C23—N1—C37	0.2 (2)
C17—C18—C19—O2	179.6 (2)	C24—C23—N1—C37	−179.7 (2)
C17—C18—C19—C20	0.0 (4)	N1—C37—N2—C36	−0.6 (2)
O2—C19—C20—C21	−179.6 (2)	C38—C37—N2—C36	179.0 (2)
C18—C19—C20—C21	0.0 (4)	C23—C36—N2—C37	0.7 (2)
C17—C16—C21—C20	−0.8 (3)	C35—C36—N2—C37	−178.8 (2)
C15—C16—C21—C20	−179.1 (2)	N4—C15—N3—C14	−0.1 (2)
C19—C20—C21—C16	0.4 (3)	C16—C15—N3—C14	179.96 (19)
C36—C23—C24—C25	178.9 (2)	C1—C14—N3—C15	0.0 (2)
N1—C23—C24—C25	−1.3 (4)	C13—C14—N3—C15	179.8 (2)
C36—C23—C24—C29	−1.9 (3)	N3—C15—N4—C1	0.2 (2)
N1—C23—C24—C29	178.0 (2)	C16—C15—N4—C1	−179.91 (19)
C29—C24—C25—C26	−1.5 (4)	C14—C1—N4—C15	−0.2 (2)
C23—C24—C25—C26	177.7 (2)	C2—C1—N4—C15	178.8 (2)
C24—C25—C26—C27	0.6 (4)	C42—C41—O1—C44	178.5 (3)
C25—C26—C27—C28	1.2 (5)	C40—C41—O1—C44	−0.6 (5)
C26—C27—C28—C29	−2.1 (4)	C20—C19—O2—C22	2.7 (4)
C27—C28—C29—C24	1.2 (4)	C18—C19—O2—C22	−176.9 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*A*···N4^{i}	0.82	1.94	2.755 (2)	173
O3—H3*A*···N2^{ii}	0.82	1.95	2.768 (2)	175
N3—H3···O3	0.86	1.99	2.840 (3)	168
N1—H1···O4	0.86	1.98	2.825 (2)	166

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