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7-{3-Ethoxy-4-[2-(2-methoxyethoxy)ethoxy]phenyl}-5,6,8,9-tetrahydrodibenzo[*c*,*h*]acridine

Sijing Zhang, Heguo Han, Yahan Xu, Wen Ma and Jieying Wu*

Department of Chemistry, Anhui University, Hefei, Anhui 230039, People's Republic of China. *Correspondence e-mail: jywu1957@163.com

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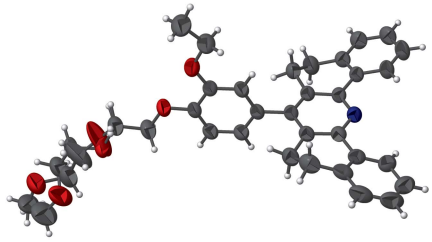
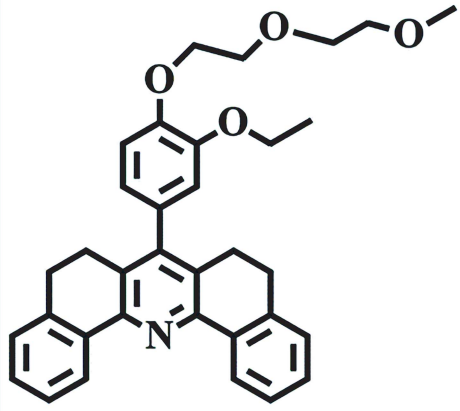
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{34}H_{35}NO_4$, the dihedral angle between the pyridine ring and attached benzene ring is 79.17 (8)°. The methoxyethoxy–ethoxy side chain is disordered over two orientations in a 0.732 (7):0.268 (7) ratio. In the crystal, very weak C—H···N and C—H···O interactions link the molecules.

3D view



Chemical scheme



Structure description

Ethyl vanillin derivatives have various biological properties (Sainsbury *et al.* 2013) but many of them show poor lipid solubility (Wu *et al.* 2018). In this study, an ether oxygen chain was introduced into the title compound in an attempt to improve its solublity.

In the crystal structure (Fig. 1), the dihedral angle between the pyridine ring and adjacent benzene ring is 79.17 (8)°. The methoxyethoxy-ethoxy side chain is disordered over two orientations in a 0.732 (7):0.268 (7) ratio. In the crystal, very weak C29—H29···O4 (2.69 Å) and C27—H27···N1 (2.67 Å) interactions (Table 1, Fig. 2) occur (Wang *et al.* 2019).

Synthesis and crystallization

Ammonium acetate (7.7 g, 100 mmol), ethyl vanillin (1.7 g, 10 mmol), 3,4-dihydronaphthalen-1(2*H*)-one (3.0 g, 20 mmol) and 25 ml acetic acid were added to a 100 ml flask and stirred for 15 min. After dissolution, the mixture was refluxed at 403 K for 6 h and filtered to obtain a yellow solid. Then, the yellow solid (2.4 g, 5.7 mmol), K_2CO_3 (0.69 g, 5 mmol) and 2-(2-methoxyethoxy)ethyl 4-methylbenzenesulfonate (1.67 g, 6 mmol) were dissolved in 30 ml of acetonitrile and the mixture was refluxed at 355 K for 12 h. After cooling to room temperature, the mixture was filtered to get a white solid (3.1 g). Colourless crystals of the title compound were recrystallized from ethanol solution.

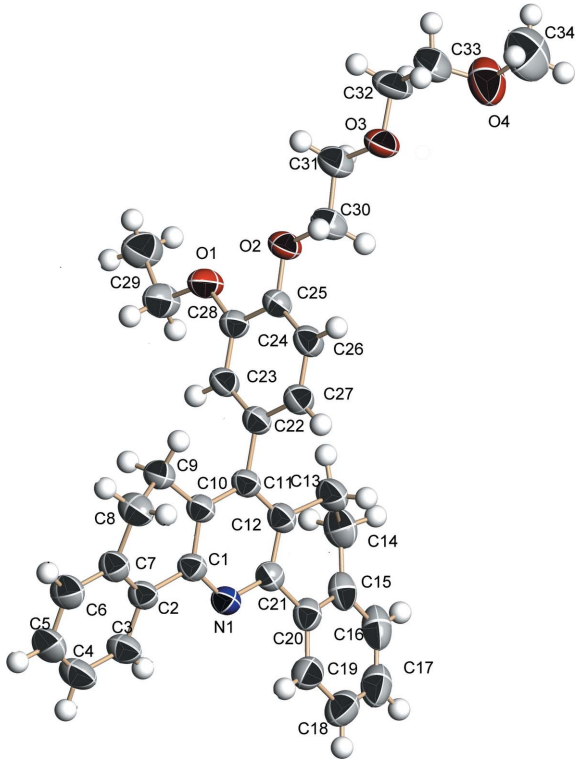


Figure 1
The molecular structure of the title compound showing 50% displacement ellipsoids. Only the major disorder component of the methoxyethoxy–ethoxy side chain is shown.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

This work was supported by the National Natural Science Foundation of China (award No. 51672002).

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

D—H···A	D—H	H···A	D···A	D—H···A
C27—H27···N1^{i}	0.93	2.67	3.537 (2)	156
C29—H29c···O4^{ii}	0.96	2.69	3.415 (6)	132

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{34}H_{35}NO_4$
M_r	521.63
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	9.1797 (13), 30.203 (4), 11.0131 (16)
β (°)	111.056 (2)
V (Å^3)	2849.6 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.12 × 0.11 × 0.10
Data collection	
Diffractometer	Bruker APEXII CCD area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.538, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	22024, 5934, 4199
R_{int}	0.044
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.646
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.051, 0.150, 1.03
No. of reflections	5934
No. of parameters	401
No. of restraints	54
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.21

Computer programs: *APEX2* and), *SAINT* (Bruker, 2009), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

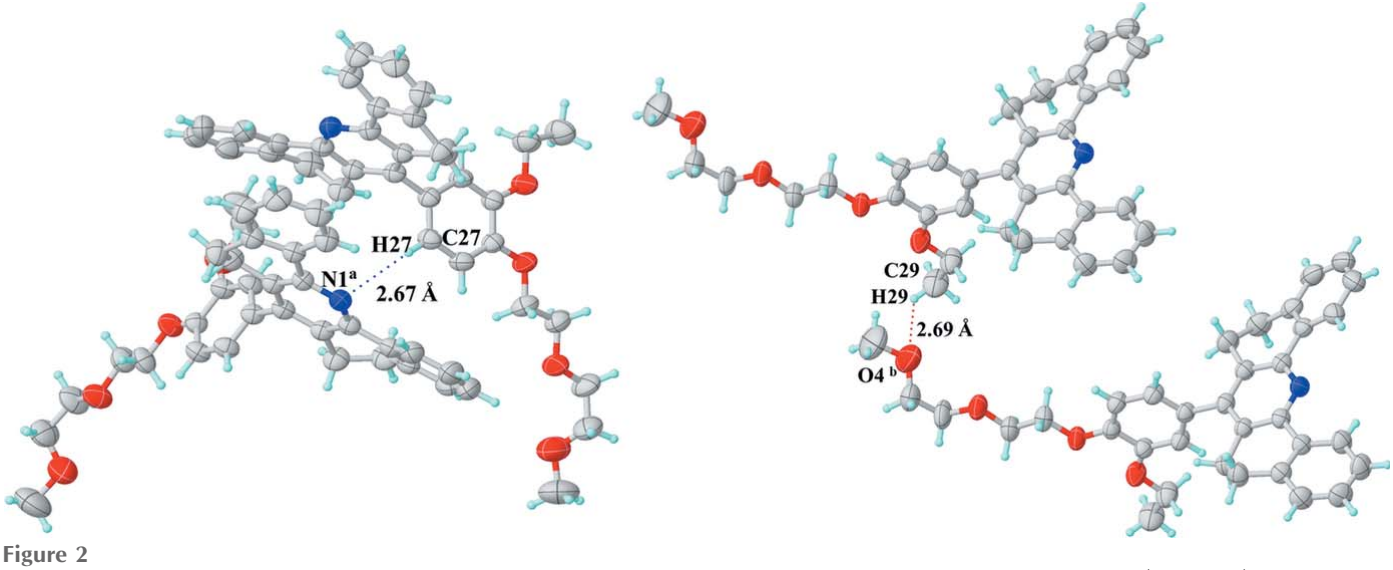


Figure 2
Weak C—H···N and C—H···O interactions in the title compound (blue and red lines, respectively). Symmetry codes: (*a*) $x, \frac{1}{2}-y, z-\frac{1}{2}$; (*b*) $x, y, z-1$.

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full crystallographic data

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7-{3-Ethoxy-4-[2-(2-methoxyethoxy)ethoxy]phenyl}-5,6,8,9-tetrahydrodibenzo[*c*,*h*]acridine

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Crystal data

$C_{34}H_{35}NO_4$
M_r = 521.63
Monoclinic, $P2_1/c$
a = 9.1797 (13) Å
b = 30.203 (4) Å
c = 11.0131 (16) Å
β = 111.056 (2)°
V = 2849.6 (7) Å^3
Z = 4
$F(000)$ = 1112
D_x = 1.216 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7039 reflections
θ = 2.4–23.8°
μ = 0.08 mm^{-1}
T = 296 K
Block, colourless
0.12 × 0.11 × 0.1 mm

Data collection

Bruker APEXII CCD area detector diffractometer
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.538, T_{max} = 0.746
22024 measured reflections
5934 independent reflections
4199 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 27.4°, θ_{min} = 1.4°
h = −11→11
k = −38→38
l = −14→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.150
S = 1.03
5934 reflections
401 parameters
54 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0721P)^2 + 0.4744P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.71434 (16)	0.39493 (4)	0.20577 (13)	0.0743 (4)	
O2	0.63910 (14)	0.42204 (4)	0.39754 (11)	0.0635 (3)	
O3	0.6825 (5)	0.47955 (14)	0.6935 (4)	0.0755 (11)	0.732 (7)
O3A	0.695 (2)	0.4900 (6)	0.6688 (17)	0.173 (8)	0.268 (7)
O4	0.7326 (5)	0.49775 (11)	0.9670 (3)	0.1180 (16)	0.732 (7)
O4A	0.8197 (10)	0.5302 (3)	0.9932 (7)	0.119 (4)	0.268 (7)
N1	0.10451 (15)	0.22075 (4)	−0.09581 (13)	0.0510 (3)	
C1	0.08312 (18)	0.26320 (5)	−0.13215 (15)	0.0485 (4)	
C2	−0.03102 (18)	0.27262 (5)	−0.26405 (15)	0.0509 (4)	
C3	−0.0832 (2)	0.23947 (6)	−0.35734 (17)	0.0633 (5)	
H3	−0.047216	0.210658	−0.336395	0.076*	
C4	−0.1871 (2)	0.24872 (7)	−0.48007 (19)	0.0749 (6)	
H4	−0.222315	0.226179	−0.541210	0.090*	
C5	−0.2390 (2)	0.29134 (7)	−0.5123 (2)	0.0754 (6)	
H5	−0.308762	0.297781	−0.595388	0.090*	
C6	−0.1874 (2)	0.32435 (7)	−0.42118 (19)	0.0692 (5)	
H6	−0.222341	0.353155	−0.443849	0.083*	
C7	−0.08478 (19)	0.31579 (6)	−0.29667 (16)	0.0563 (4)	
C8	−0.0320 (2)	0.35105 (6)	−0.19438 (19)	0.0708 (5)	
H8A	−0.099077	0.350812	−0.143513	0.085*	
H8B	−0.042310	0.379743	−0.236200	0.085*	
C9	0.1352 (2)	0.34464 (5)	−0.10467 (17)	0.0605 (4)	
H9A	0.204076	0.351483	−0.151368	0.073*	
H9B	0.158504	0.365084	−0.032218	0.073*	
C10	0.16608 (18)	0.29796 (5)	−0.05304 (15)	0.0491 (4)	
C11	0.27751 (18)	0.28769 (5)	0.06836 (15)	0.0504 (4)	
C12	0.30031 (19)	0.24333 (5)	0.10727 (15)	0.0518 (4)	
C13	0.4164 (2)	0.22877 (6)	0.23686 (18)	0.0672 (5)	
H13A	0.364811	0.226732	0.299653	0.081*	
H13B	0.498975	0.250599	0.268064	0.081*	
C14	0.4863 (2)	0.18407 (7)	0.2251 (2)	0.0758 (6)	
H14A	0.553373	0.187471	0.174858	0.091*	
H14B	0.550046	0.173689	0.311146	0.091*	
C15	0.3636 (2)	0.15035 (6)	0.16108 (17)	0.0638 (5)	
C16	0.3808 (3)	0.10565 (7)	0.1952 (2)	0.0820 (6)	
H16	0.470504	0.096199	0.261970	0.098*	
C17	0.2674 (4)	0.07543 (7)	0.1317 (2)	0.0920 (8)	
H17	0.281080	0.045700	0.155037	0.110*	
C18	0.1336 (3)	0.08896 (7)	0.0336 (2)	0.0856 (7)	
H18	0.056637	0.068414	−0.008815	0.103*	
C19	0.1130 (2)	0.13319 (6)	−0.00238 (19)	0.0674 (5)	
H19	0.022252	0.142257	−0.068636	0.081*	
C20	0.2281 (2)	0.16406 (5)	0.06058 (16)	0.0559 (4)	
C21	0.20972 (18)	0.21123 (5)	0.02191 (15)	0.0502 (4)	
C22	0.37005 (19)	0.32369 (5)	0.15619 (15)	0.0512 (4)	

C23	0.4972 (2)	0.34257 (6)	0.13558 (16)	0.0556 (4)	
H23	0.523515	0.333000	0.065869	0.067*	
C24	0.58527 (19)	0.37532 (5)	0.21675 (16)	0.0532 (4)	
C25	0.54418 (19)	0.39013 (5)	0.32045 (15)	0.0512 (4)	
C26	0.4170 (2)	0.37195 (6)	0.33955 (16)	0.0580 (4)	
H26	0.388129	0.382072	0.407437	0.070*	
C27	0.3310 (2)	0.33861 (6)	0.25851 (16)	0.0575 (4)	
H27	0.246238	0.326250	0.273544	0.069*	
C28	0.7627 (2)	0.38215 (8)	0.1035 (2)	0.0782 (6)	
H28A	0.685289	0.390937	0.020748	0.094*	
H28B	0.774643	0.350245	0.103402	0.094*	
C29	0.9141 (3)	0.40407 (9)	0.1224 (2)	0.0933 (7)	
H29A	0.943418	0.398295	0.048609	0.140*	
H29B	0.992816	0.392671	0.199593	0.140*	
H29C	0.903879	0.435423	0.131365	0.140*	
C30	0.6000 (2)	0.43698 (7)	0.50509 (18)	0.0688 (5)	
H30A	0.588985	0.411929	0.556253	0.083*	
H30B	0.501933	0.453036	0.474177	0.083*	
C31	0.7281 (3)	0.46647 (7)	0.58576 (19)	0.0746 (5)	
H31A	0.826874	0.450774	0.616634	0.090*	0.732 (7)
H31B	0.737913	0.492118	0.536238	0.090*	0.732 (7)
H31C	0.818457	0.448388	0.631757	0.090*	0.268 (7)
H31D	0.756965	0.486084	0.528369	0.090*	0.268 (7)
C32	0.7863 (7)	0.50939 (18)	0.7776 (6)	0.0878 (14)	0.732 (7)
H32A	0.803993	0.533688	0.726962	0.105*	0.732 (7)
H32B	0.885443	0.494623	0.820207	0.105*	0.732 (7)
C32A	0.790 (4)	0.4894 (10)	0.783 (2)	0.176 (11)	0.268 (7)
H32C	0.879303	0.507911	0.790106	0.211*	0.268 (7)
H32D	0.826563	0.459474	0.807728	0.211*	0.268 (7)
C33	0.7328 (8)	0.52691 (19)	0.8746 (6)	0.0969 (16)	0.732 (7)
H33A	0.798537	0.551803	0.916278	0.116*	0.732 (7)
H33B	0.627543	0.538043	0.832187	0.116*	0.732 (7)
C33A	0.697 (3)	0.5091 (6)	0.8795 (19)	0.123 (6)	0.268 (7)
H33C	0.617954	0.530617	0.833003	0.148*	0.268 (7)
H33D	0.646248	0.485121	0.908089	0.148*	0.268 (7)
C34	0.7022 (11)	0.5171 (3)	1.0730 (7)	0.156 (3)	0.732 (7)
H34A	0.611157	0.535544	1.040400	0.233*	0.732 (7)
H34B	0.790132	0.534744	1.123588	0.233*	0.732 (7)
H34C	0.684890	0.494192	1.126604	0.233*	0.732 (7)
C34A	0.752 (3)	0.5412 (6)	1.0832 (17)	0.138 (7)	0.268 (7)
H34D	0.781137	0.519691	1.152001	0.207*	0.268 (7)
H34E	0.640465	0.541513	1.041264	0.207*	0.268 (7)
H34F	0.787465	0.569941	1.118587	0.207*	0.268 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0755 (9)	0.0878 (9)	0.0700 (8)	−0.0311 (7)	0.0387 (7)	−0.0266 (7)

O2	0.0689 (8)	0.0666 (7)	0.0561 (7)	−0.0144 (6)	0.0239 (6)	−0.0193 (6)
O3	0.0841 (19)	0.0845 (17)	0.061 (2)	−0.0294 (16)	0.0303 (17)	−0.0267 (13)
O3A	0.212 (15)	0.171 (13)	0.089 (7)	0.098 (10)	−0.004 (7)	−0.065 (8)
O4	0.177 (4)	0.103 (2)	0.085 (2)	0.020 (2)	0.061 (2)	−0.0026 (17)
O4A	0.139 (7)	0.143 (8)	0.080 (5)	−0.042 (5)	0.045 (4)	−0.031 (4)
N1	0.0477 (7)	0.0540 (8)	0.0490 (8)	−0.0020 (6)	0.0145 (6)	−0.0028 (6)
C1	0.0430 (8)	0.0529 (9)	0.0479 (9)	−0.0019 (7)	0.0144 (7)	−0.0030 (7)
C2	0.0416 (8)	0.0586 (9)	0.0484 (9)	−0.0029 (7)	0.0114 (7)	−0.0020 (7)
C3	0.0562 (10)	0.0633 (10)	0.0580 (11)	−0.0026 (8)	0.0057 (9)	−0.0061 (8)
C4	0.0663 (12)	0.0814 (13)	0.0590 (12)	−0.0065 (10)	0.0006 (10)	−0.0115 (10)
C5	0.0631 (12)	0.0880 (14)	0.0553 (11)	−0.0015 (10)	−0.0025 (9)	0.0048 (10)
C6	0.0603 (11)	0.0702 (11)	0.0639 (12)	0.0052 (9)	0.0065 (9)	0.0074 (9)
C7	0.0489 (9)	0.0625 (10)	0.0533 (10)	0.0015 (7)	0.0133 (8)	0.0006 (8)
C8	0.0764 (13)	0.0598 (10)	0.0646 (12)	0.0092 (9)	0.0112 (10)	−0.0032 (8)
C9	0.0667 (11)	0.0550 (9)	0.0542 (10)	−0.0060 (8)	0.0149 (9)	−0.0042 (7)
C10	0.0461 (9)	0.0546 (9)	0.0458 (9)	−0.0034 (7)	0.0155 (7)	−0.0036 (7)
C11	0.0442 (9)	0.0603 (9)	0.0459 (9)	−0.0062 (7)	0.0152 (7)	−0.0059 (7)
C12	0.0464 (9)	0.0617 (10)	0.0453 (9)	−0.0011 (7)	0.0140 (7)	−0.0010 (7)
C13	0.0639 (11)	0.0733 (11)	0.0524 (10)	0.0006 (9)	0.0065 (9)	0.0021 (8)
C14	0.0665 (12)	0.0902 (14)	0.0617 (12)	0.0174 (10)	0.0121 (10)	0.0117 (10)
C15	0.0763 (12)	0.0697 (11)	0.0507 (10)	0.0180 (9)	0.0293 (9)	0.0071 (8)
C16	0.1162 (18)	0.0777 (14)	0.0589 (12)	0.0320 (13)	0.0396 (12)	0.0154 (10)
C17	0.155 (2)	0.0610 (12)	0.0754 (14)	0.0155 (14)	0.0597 (16)	0.0114 (11)
C18	0.123 (2)	0.0607 (11)	0.0880 (16)	−0.0111 (12)	0.0561 (15)	−0.0023 (11)
C19	0.0811 (13)	0.0600 (10)	0.0682 (12)	−0.0038 (9)	0.0355 (10)	−0.0017 (9)
C20	0.0655 (11)	0.0565 (9)	0.0527 (9)	0.0047 (8)	0.0295 (9)	0.0014 (7)
C21	0.0461 (9)	0.0572 (9)	0.0476 (9)	0.0000 (7)	0.0171 (7)	−0.0010 (7)
C22	0.0479 (9)	0.0588 (9)	0.0428 (8)	−0.0044 (7)	0.0113 (7)	−0.0033 (7)
C23	0.0557 (10)	0.0670 (10)	0.0460 (9)	−0.0088 (8)	0.0204 (8)	−0.0108 (7)
C24	0.0502 (9)	0.0605 (9)	0.0490 (9)	−0.0092 (7)	0.0180 (7)	−0.0056 (7)
C25	0.0518 (9)	0.0542 (9)	0.0434 (8)	−0.0032 (7)	0.0120 (7)	−0.0057 (7)
C26	0.0572 (10)	0.0709 (10)	0.0487 (9)	−0.0030 (8)	0.0225 (8)	−0.0087 (8)
C27	0.0509 (10)	0.0715 (11)	0.0512 (10)	−0.0092 (8)	0.0199 (8)	−0.0059 (8)
C28	0.0731 (13)	0.0934 (14)	0.0782 (13)	−0.0149 (11)	0.0395 (11)	−0.0201 (11)
C29	0.0768 (14)	0.1173 (18)	0.0985 (17)	−0.0265 (13)	0.0469 (13)	−0.0220 (14)
C30	0.0715 (12)	0.0710 (11)	0.0622 (11)	−0.0002 (9)	0.0219 (10)	−0.0207 (9)
C31	0.0886 (14)	0.0694 (11)	0.0621 (12)	−0.0079 (10)	0.0225 (11)	−0.0175 (9)
C32	0.097 (3)	0.093 (3)	0.072 (2)	−0.032 (2)	0.029 (2)	−0.040 (2)
C32A	0.224 (19)	0.22 (3)	0.062 (7)	0.017 (19)	0.030 (9)	−0.023 (13)
C33	0.132 (4)	0.088 (4)	0.069 (3)	−0.017 (3)	0.034 (2)	−0.024 (2)
C33A	0.184 (15)	0.096 (12)	0.068 (8)	−0.023 (10)	0.019 (8)	−0.010 (8)
C34	0.215 (8)	0.166 (6)	0.122 (4)	0.000 (6)	0.105 (5)	−0.032 (5)
C34A	0.23 (2)	0.118 (12)	0.100 (9)	−0.051 (12)	0.103 (11)	−0.037 (9)

Geometric parameters (Å, º)

O1—C24	1.368 (2)	C16—C17	1.373 (3)
O1—C28	1.405 (2)	C17—H17	0.9300

O2—C25	1.3701 (19)	C17—C18	1.375 (4)
O2—C30	1.429 (2)	C18—H18	0.9300
O3—C31	1.448 (4)	C18—C19	1.387 (3)
O3—C32	1.394 (6)	C19—H19	0.9300
O3A—C31	1.278 (14)	C19—C20	1.393 (3)
O3A—C32A	1.24 (3)	C20—C21	1.479 (2)
O4—C33	1.346 (8)	C22—C23	1.389 (2)
O4—C34	1.420 (6)	C22—C27	1.375 (2)
O4A—C33A	1.50 (2)	C23—H23	0.9300
O4A—C34A	1.386 (18)	C23—C24	1.383 (2)
N1—C1	1.336 (2)	C24—C25	1.399 (2)
N1—C21	1.340 (2)	C25—C26	1.373 (2)
C1—C2	1.482 (2)	C26—H26	0.9300
C1—C10	1.402 (2)	C26—C27	1.388 (2)
C2—C3	1.391 (2)	C27—H27	0.9300
C2—C7	1.395 (2)	C28—H28A	0.9700
C3—H3	0.9300	C28—H28B	0.9700
C3—C4	1.374 (3)	C28—C29	1.485 (3)
C4—H4	0.9300	C29—H29A	0.9600
C4—C5	1.374 (3)	C29—H29B	0.9600
C5—H5	0.9300	C29—H29C	0.9600
C5—C6	1.372 (3)	C30—H30A	0.9700
C6—H6	0.9300	C30—H30B	0.9700
C6—C7	1.381 (2)	C30—C31	1.488 (3)
C7—C8	1.499 (2)	C31—H31A	0.9700
C8—H8A	0.9700	C31—H31B	0.9700
C8—H8B	0.9700	C31—H31C	0.9700
C8—C9	1.510 (3)	C31—H31D	0.9700
C9—H9A	0.9700	C32—H32A	0.9700
C9—H9B	0.9700	C32—H32B	0.9700
C9—C10	1.508 (2)	C32—C33	1.428 (9)
C10—C11	1.396 (2)	C32A—H32C	0.9700
C11—C12	1.399 (2)	C32A—H32D	0.9700
C11—C22	1.500 (2)	C32A—C33A	1.70 (3)
C12—C13	1.508 (2)	C33—H33A	0.9700
C12—C21	1.398 (2)	C33—H33B	0.9700
C13—H13A	0.9700	C33A—H33C	0.9700
C13—H13B	0.9700	C33A—H33D	0.9700
C13—C14	1.520 (3)	C34—H34A	0.9600
C14—H14A	0.9700	C34—H34B	0.9600
C14—H14B	0.9700	C34—H34C	0.9600
C14—C15	1.494 (3)	C34A—H34D	0.9600
C15—C16	1.395 (3)	C34A—H34E	0.9600
C15—C20	1.398 (3)	C34A—H34F	0.9600
C16—H16	0.9300		

C24—O1—C28	119.02 (14)	C27—C22—C11	120.80 (15)
C25—O2—C30	116.26 (14)	C27—C22—C23	118.75 (15)

C32—O3—C31	113.0 (3)	C22—C23—H23	119.4
C32A—O3A—C31	116.7 (19)	C24—C23—C22	121.25 (15)
C33—O4—C34	113.9 (4)	C24—C23—H23	119.4
C34A—O4A—C33A	107.6 (14)	O1—C24—C23	125.20 (15)
C1—N1—C21	118.22 (13)	O1—C24—C25	115.46 (14)
N1—C1—C2	117.00 (14)	C23—C24—C25	119.33 (15)
N1—C1—C10	122.95 (14)	O2—C25—C24	115.65 (14)
C10—C1—C2	120.02 (14)	O2—C25—C26	124.95 (15)
C3—C2—C1	121.36 (15)	C26—C25—C24	119.39 (14)
C3—C2—C7	119.13 (15)	C25—C26—H26	119.7
C7—C2—C1	119.49 (14)	C25—C26—C27	120.65 (15)
C2—C3—H3	119.6	C27—C26—H26	119.7
C4—C3—C2	120.89 (17)	C22—C27—C26	120.61 (15)
C4—C3—H3	119.6	C22—C27—H27	119.7
C3—C4—H4	120.1	C26—C27—H27	119.7
C5—C4—C3	119.86 (18)	O1—C28—H28A	110.0
C5—C4—H4	120.1	O1—C28—H28B	110.0
C4—C5—H5	120.2	O1—C28—C29	108.33 (17)
C6—C5—C4	119.70 (18)	H28A—C28—H28B	108.4
C6—C5—H5	120.2	C29—C28—H28A	110.0
C5—C6—H6	119.2	C29—C28—H28B	110.0
C5—C6—C7	121.54 (18)	C28—C29—H29A	109.5
C7—C6—H6	119.2	C28—C29—H29B	109.5
C2—C7—C8	118.81 (15)	C28—C29—H29C	109.5
C6—C7—C2	118.87 (16)	H29A—C29—H29B	109.5
C6—C7—C8	122.31 (16)	H29A—C29—H29C	109.5
C7—C8—H8A	109.2	H29B—C29—H29C	109.5
C7—C8—H8B	109.2	O2—C30—H30A	110.2
C7—C8—C9	112.27 (15)	O2—C30—H30B	110.2
H8A—C8—H8B	107.9	O2—C30—C31	107.68 (16)
C9—C8—H8A	109.2	H30A—C30—H30B	108.5
C9—C8—H8B	109.1	C31—C30—H30A	110.2
C8—C9—H9A	109.2	C31—C30—H30B	110.2
C8—C9—H9B	109.2	O3—C31—C30	104.7 (2)
H9A—C9—H9B	107.9	O3—C31—H31A	110.8
C10—C9—C8	112.07 (14)	O3—C31—H31B	110.8
C10—C9—H9A	109.2	O3A—C31—C30	114.7 (9)
C10—C9—H9B	109.2	O3A—C31—H31C	108.6
C1—C10—C9	118.64 (14)	O3A—C31—H31D	108.6
C11—C10—C1	118.45 (15)	C30—C31—H31A	110.8
C11—C10—C9	122.87 (14)	C30—C31—H31B	110.8
C10—C11—C12	118.93 (14)	C30—C31—H31C	108.6
C10—C11—C22	120.48 (14)	C30—C31—H31D	108.6
C12—C11—C22	120.59 (14)	H31A—C31—H31B	108.9
C11—C12—C13	123.11 (15)	H31C—C31—H31D	107.6
C21—C12—C11	118.08 (14)	O3—C32—H32A	108.9
C21—C12—C13	118.80 (15)	O3—C32—H32B	108.9
C12—C13—H13A	109.5	O3—C32—C33	113.6 (5)

C12—C13—H13B	109.5	H32A—C32—H32B	107.7
C12—C13—C14	110.77 (15)	C33—C32—H32A	108.9
H13A—C13—H13B	108.1	C33—C32—H32B	108.9
C14—C13—H13A	109.5	O3A—C32A—H32C	110.3
C14—C13—H13B	109.5	O3A—C32A—H32D	110.3
C13—C14—H14A	109.2	O3A—C32A—C33A	107 (2)
C13—C14—H14B	109.2	H32C—C32A—H32D	108.5
H14A—C14—H14B	107.9	C33A—C32A—H32C	110.3
C15—C14—C13	112.08 (16)	C33A—C32A—H32D	110.3
C15—C14—H14A	109.2	O4—C33—C32	114.7 (5)
C15—C14—H14B	109.2	O4—C33—H33A	108.6
C16—C15—C14	122.63 (19)	O4—C33—H33B	108.6
C16—C15—C20	118.9 (2)	C32—C33—H33A	108.6
C20—C15—C14	118.49 (16)	C32—C33—H33B	108.6
C15—C16—H16	119.5	H33A—C33—H33B	107.6
C17—C16—C15	121.0 (2)	O4A—C33A—C32A	106.0 (18)
C17—C16—H16	119.5	O4A—C33A—H33C	110.5
C16—C17—H17	119.9	O4A—C33A—H33D	110.5
C16—C17—C18	120.2 (2)	C32A—C33A—H33C	110.5
C18—C17—H17	119.9	C32A—C33A—H33D	110.5
C17—C18—H18	119.9	H33C—C33A—H33D	108.7
C17—C18—C19	120.2 (2)	O4—C34—H34A	109.5
C19—C18—H18	119.9	O4—C34—H34B	109.5
C18—C19—H19	119.9	O4—C34—H34C	109.5
C18—C19—C20	120.1 (2)	H34A—C34—H34B	109.5
C20—C19—H19	119.9	H34A—C34—H34C	109.5
C15—C20—C21	119.37 (16)	H34B—C34—H34C	109.5
C19—C20—C15	119.72 (17)	O4A—C34A—H34D	109.5
C19—C20—C21	120.91 (16)	O4A—C34A—H34E	109.5
N1—C21—C12	123.34 (15)	O4A—C34A—H34F	109.5
N1—C21—C20	116.95 (14)	H34D—C34A—H34E	109.5
C12—C21—C20	119.71 (15)	H34D—C34A—H34F	109.5
C23—C22—C11	120.45 (14)	H34E—C34A—H34F	109.5

O1—C24—C25—O2	−0.4 (2)	C12—C11—C22—C27	−78.8 (2)
O1—C24—C25—C26	−179.39 (15)	C12—C13—C14—C15	−51.7 (2)
O2—C25—C26—C27	−177.74 (16)	C13—C12—C21—N1	−179.38 (16)
O2—C30—C31—O3	−178.9 (3)	C13—C12—C21—C20	0.6 (2)
O2—C30—C31—O3A	165.3 (10)	C13—C14—C15—C16	−145.65 (18)
O3—C32—C33—O4	70.8 (8)	C13—C14—C15—C20	36.2 (2)
O3A—C32A—C33A—O4A	−151 (3)	C14—C15—C16—C17	−178.1 (2)
N1—C1—C2—C3	−16.0 (2)	C14—C15—C20—C19	178.86 (17)
N1—C1—C2—C7	165.48 (15)	C14—C15—C20—C21	−0.7 (2)
N1—C1—C10—C9	178.78 (15)	C15—C16—C17—C18	−0.6 (3)
N1—C1—C10—C11	0.9 (2)	C15—C20—C21—N1	160.90 (15)
C1—N1—C21—C12	−1.4 (2)	C15—C20—C21—C12	−19.1 (2)
C1—N1—C21—C20	178.62 (14)	C16—C15—C20—C19	0.6 (3)
C1—C2—C3—C4	−178.93 (18)	C16—C15—C20—C21	−178.91 (16)

C1—C2—C7—C6	178.00 (16)	C16—C17—C18—C19	0.5 (3)
C1—C2—C7—C8	−3.4 (2)	C17—C18—C19—C20	0.2 (3)
C1—C10—C11—C12	−1.0 (2)	C18—C19—C20—C15	−0.7 (3)
C1—C10—C11—C22	179.60 (14)	C18—C19—C20—C21	178.80 (17)
C2—C1—C10—C9	0.6 (2)	C19—C20—C21—N1	−18.6 (2)
C2—C1—C10—C11	−177.26 (14)	C19—C20—C21—C12	161.38 (16)
C2—C3—C4—C5	0.9 (3)	C20—C15—C16—C17	0.0 (3)
C2—C7—C8—C9	36.3 (2)	C21—N1—C1—C2	178.51 (14)
C3—C2—C7—C6	−0.5 (3)	C21—N1—C1—C10	0.3 (2)
C3—C2—C7—C8	178.09 (17)	C21—C12—C13—C14	34.3 (2)
C3—C4—C5—C6	−0.5 (3)	C22—C11—C12—C13	0.0 (2)
C4—C5—C6—C7	−0.5 (3)	C22—C11—C12—C21	179.41 (14)
C5—C6—C7—C2	1.0 (3)	C22—C23—C24—O1	178.26 (16)
C5—C6—C7—C8	−177.6 (2)	C22—C23—C24—C25	−0.9 (3)
C6—C7—C8—C9	−145.14 (18)	C23—C22—C27—C26	0.1 (3)
C7—C2—C3—C4	−0.4 (3)	C23—C24—C25—O2	178.85 (15)
C7—C8—C9—C10	−49.5 (2)	C23—C24—C25—C26	−0.1 (3)
C8—C9—C10—C1	32.2 (2)	C24—O1—C28—C29	−173.35 (18)
C8—C9—C10—C11	−150.05 (16)	C24—C25—C26—C27	1.1 (3)
C9—C10—C11—C12	−178.80 (15)	C25—O2—C30—C31	172.29 (15)
C9—C10—C11—C22	1.8 (2)	C25—C26—C27—C22	−1.1 (3)
C10—C1—C2—C3	162.25 (16)	C27—C22—C23—C24	0.9 (3)
C10—C1—C2—C7	−16.2 (2)	C28—O1—C24—C23	1.3 (3)
C10—C11—C12—C13	−179.33 (16)	C28—O1—C24—C25	−179.51 (17)
C10—C11—C12—C21	0.0 (2)	C30—O2—C25—C24	−179.04 (15)
C10—C11—C22—C23	−79.5 (2)	C30—O2—C25—C26	−0.1 (2)
C10—C11—C22—C27	100.53 (19)	C31—O3—C32—C33	172.4 (5)
C11—C12—C13—C14	−146.32 (18)	C31—O3A—C32A—C33A	−162.8 (15)
C11—C12—C21—N1	1.2 (2)	C32—O3—C31—C30	−177.1 (4)
C11—C12—C21—C20	−178.78 (14)	C32A—O3A—C31—C30	130 (2)
C11—C22—C23—C24	−179.01 (15)	C34—O4—C33—C32	170.9 (6)
C11—C22—C27—C26	−179.98 (16)	C34A—O4A—C33A—C32A	−171.9 (15)
C12—C11—C22—C23	101.14 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C27—H27···N1^{i}	0.93	2.67	3.537 (2)	156
C29—H29c···O4^{ii}	0.96	2.69	3.415 (6)	132

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