IUCrData

ISSN 2414-3146

(*Z*)-2-(4-{[(*E*)-2-Hydroxybenzylidene]amino}phenyl)-3-[4-(1,4,7,10-tetraoxa-13-azacyclopentadecan-13-yl)phenyl]acrylonitrile

Xin Zhang[a,b] and Zhi-Chao Wu[a,b]*

[a]Department of Chemistry, Anhui University, Hefei 230601, People's Republic of China, and [b]Key Laboratory of Functional Inorganic Materials, Chemistry, Hefei 230601, People's Republic of China. *Correspondence e-mail: 820008032@qq.com

Received 7 September 2016
Accepted 10 October 2016

Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

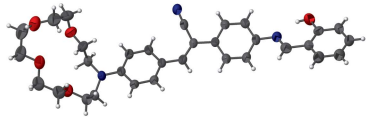
Keywords: crystal structure; azacrown ether; acrylonitrile; fluorescence probe; hydrogen bonding.

CCDC reference: 1413056

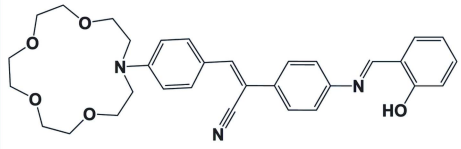
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{32}H_{35}N_3O_5$, is an important fluorescence probe. It is composed of a 1,4,7,10-tetraoxa-13λ^2-azacyclopentadecane unit linked to a twisted (*Z*)-2-(4-{[(*E*)-2-hydroxybenzylidene]amino}phenyl)-3-phenylacrylonitrile chain. In the molecule, an O—H···N hydrogen bond forms an *S*(6) ring motif. There is also in intramolecular C—H···N hydrogen bond in the azacrown ether moiety, which also forms an *S*(6) ring motif. In the crystal, molecules are linked *via* pairs of C—H···O hydrogen bonds, forming inversion dimers.

3D view



Chemical scheme



Structure description

The cavity size of a crown ether provides selectivity for a great many cations, such as K^+ (Wang *et al.*, 2012), Na^+ (Kim *et al.*, 2010; Gunnlaugsson *et al.*, 2001; 2002), or Ba^{2+} (Weissenstein & Würthner, 2015). Herein, we report on the synthesis and crystal structure of the title azacrown ether derivative.

The molecular structure of the title compound is illustrated in Fig. 1. It is composed of a 1,4,7,10-tetraoxa-13λ^2-azacyclopentadecane unit linked to a twisted (*Z*)-2-(4-{[(*E*)-2-hydroxybenzylidene]amino)yl)-3-phenylacrylonitrile chain. There is an intramolecular O—H···N hydrogen bond forming an *S*(6) ring motif, and an intramolecular C—H···O hydrogen bond also forming an *S*(6) ring motif (Table 1 and Fig. 1). The central benzene ring, C8–C13 is inclined to the two neighbouring benzene rings, C1–C6 and C17–C22, by 32.0 (3) and 16.8 (3)°, respectively. The latter two rings are inclined to one another by 16.6 (3)°.

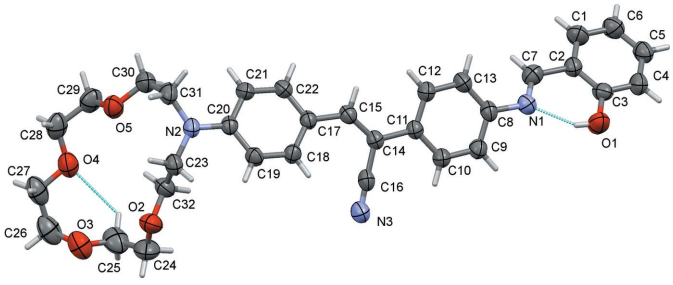


Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

In the crystal, molecules are linked *via* pairs of C—H···O hydrogen bonds, forming inversion dimers (Table 1 and Fig. 2).

Synthesis and crystallization

We first prepared the title compound by a solid-state grinding reaction. The present sample was prepared by mixing (*Z*)-3-(4-(1,4,7,10-tetraoxa-13-azacyclopentadecan-13-yl)phenyl)-2-(4-aminophenyl)acrylonitrile (0.1 g, 0.24 mmol) and 2-hydroxybenzaldehyde (0.06 g, 0.49 mmol) in an ethanol solution, which was then refluxed at 353 K for 2 h. After the completion of the reaction, the solvent was removed *via* vacuum filter and dried to give a red powder. Red block-like crystals were obtained by slow evaporation of a solution in ethanol .

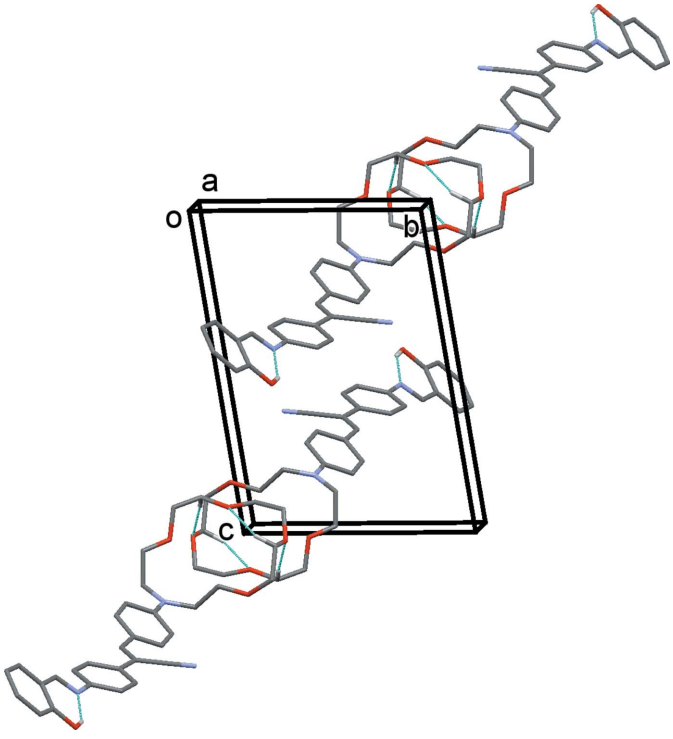


Figure 2
A view along the *a* axis of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1), and H atoms not involved in these interactions have been omitted for clarity.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N1	0.82	1.87	2.600 (6)	148
C25—H25*A*···O4	0.97	2.50	3.129 (9)	122
C28—H28*A*···O3^{i}	0.97	2.53	3.493 (8)	170

Symmetry code: (i) $-x+3, -y+2, -z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{32}H_{35}N_3O_5$
M_r	541.69
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, *b*, *c* (Å)	9.938 (2), 10.085 (2), 14.230 (4)
α, β, γ (°)	80.194 (3), 86.588 (3), 81.534 (3)
V (Å^3)	1389.2 (6)
Z	2
Radiation type	Mo *Kα*
μ (mm^{-1})	0.09
Crystal size (mm)	0.22 × 0.18 × 0.17
Data collection	
Diffractometer	Bruker SMART CCD area-detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2002)
T_{min}, T_{max}	0.392, 0.439
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9986, 4829, 2894
R_{int}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.052, 0.155, 1.04
No. of reflections	4829
No. of parameters	362
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.39

Computer programs: *SMART* and *SAINT* (Bruker, 2002), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008) and *PLATON* (Spek, 2009).

Refinement

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

Acknowledgements

This work was supported by The National Natural Science Foundation of China (Project Nos. 21271003, 21271004, 51432001 and 51472002),

References

Bruker (2002). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Gunnlaugsson, T., Nieuwenhuyzen, M., Richard, L. & Thoss, V. (2001). *Tetrahedron Lett.* **42**, 4725–4728.
Gunnlaugsson, T., Nieuwenhuyzen, M., Richard, L. & Thoss, V. (2002). *J. Chem. Soc. Perkin Trans. 2*, pp. 141–151.
Kim, M. K., Lim, C. S., Hong, J. T., Han, J., Jang, H., Kim, H. & Cho, B. (2010). *Angew. Chem.* **122**, 374–377.

Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Wang, X., Hu, J., Liu, T., Zhang, G. & Liu, S. (2012). *J. Mater. Chem.* **22**, 8622–8628.
Weissenstein, A. & Würthner, F. (2015). *Chem. Commun.* **51**, 3415–3418.

full crystallographic data

IUCrData (2016). **1**, x161598 [https://doi.org/10.1107/S2414314616015984]

(*Z*)-2-(4-{[(*E*)-2-Hydroxybenzylidene]amino}phenyl)-3-[4-(1,4,7,10-tetraoxa-13-azacyclopentadecan-13-yl)phenyl]acrylonitrile

Xin Zhang and Zhi-Chao Wu

(*Z*)-2-(4-{[(*E*)-2-Hydroxybenzylidene]amino}phenyl)-3-[4-(1,4,7,10-tetraoxa-13-azacyclopentadecan-13-yl)phenyl]acrylonitrile

Crystal data

$C_{32}H_{35}N_3O_5$
M_r = 541.69
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 9.938 (2) Å
b = 10.085 (2) Å
c = 14.230 (4) Å
α = 80.194 (3)°
β = 86.588 (3)°
γ = 81.534 (3)°
V = 1389.2 (6) Å^3
Z = 2
$F(000)$ = 576
D_x = 1.295 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1871 reflections
θ = 2.3–22.1°
μ = 0.09 mm^{-1}
T = 296 K
Block, red
0.22 × 0.18 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2002)
T_{min} = 0.392, T_{max} = 0.439
9986 measured reflections
4829 independent reflections
2894 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 25.0°, θ_{min} = 2.3°
h = −11→11
k = −11→10
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.155
S = 1.04
4829 reflections
362 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0758P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.012
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.1268 (4)	0.2355 (5)	0.5334 (3)	0.0742 (17)
O2	1.1875 (5)	1.0122 (4)	0.1648 (3)	0.0741 (16)
O3	1.2731 (5)	1.2092 (4)	0.0017 (3)	0.0830 (17)
O4	1.3478 (4)	0.9838 (5)	−0.1010 (3)	0.0720 (17)
O5	1.3624 (4)	0.7060 (4)	−0.0019 (3)	0.0664 (17)
N1	0.0979 (4)	0.2588 (4)	0.4326 (3)	0.0524 (17)
N2	1.2016 (4)	0.6432 (4)	0.1855 (3)	0.0509 (17)
N3	0.5432 (5)	0.7601 (5)	0.3652 (4)	0.0675 (19)
C1	−0.0975 (6)	0.0350 (6)	0.3417 (4)	0.066 (2)
C2	−0.0666 (5)	0.1183 (5)	0.4012 (4)	0.0507 (17)
C3	−0.1545 (6)	0.1532 (5)	0.4742 (4)	0.0524 (19)
C4	−0.2770 (6)	0.1000 (6)	0.4876 (5)	0.064 (2)
C5	−0.3080 (6)	0.0156 (6)	0.4276 (5)	0.063 (2)
C6	−0.2205 (6)	−0.0168 (6)	0.3551 (5)	0.067 (3)
C7	0.0698 (6)	0.1770 (6)	0.3829 (4)	0.059 (2)
C8	0.2230 (5)	0.3134 (5)	0.4106 (4)	0.0490 (17)
C9	0.2272 (5)	0.4415 (5)	0.4316 (4)	0.0505 (17)
C10	0.3425 (5)	0.5032 (5)	0.4111 (4)	0.0473 (17)
C11	0.4591 (5)	0.4382 (5)	0.3706 (3)	0.0426 (17)
C12	0.4554 (5)	0.3075 (5)	0.3526 (4)	0.0535 (19)
C13	0.3393 (6)	0.2459 (6)	0.3719 (4)	0.058 (2)
C14	0.5825 (5)	0.5073 (5)	0.3466 (3)	0.0424 (17)
C15	0.7019 (5)	0.4500 (5)	0.3115 (4)	0.0469 (17)
C16	0.5637 (5)	0.6479 (6)	0.3578 (4)	0.0508 (19)
C17	0.8284 (5)	0.5046 (5)	0.2822 (3)	0.0436 (17)
C18	0.8683 (5)	0.6176 (5)	0.3111 (4)	0.0468 (17)
C19	0.9887 (5)	0.6643 (5)	0.2787 (4)	0.0473 (17)
C20	1.0785 (5)	0.5994 (5)	0.2154 (4)	0.0441 (17)
C21	1.0372 (5)	0.4867 (5)	0.1855 (4)	0.0537 (19)
C22	0.9174 (5)	0.4412 (5)	0.2196 (4)	0.0523 (19)
C23	1.2333 (5)	0.7728 (5)	0.2042 (4)	0.055 (2)
C24	1.1088 (8)	1.1308 (7)	0.1164 (6)	0.095 (3)
C25	1.1428 (7)	1.1679 (7)	0.0138 (6)	0.088 (3)
C26	1.3235 (8)	1.2203 (8)	−0.0946 (5)	0.089 (3)
C27	1.4176 (8)	1.0987 (7)	−0.1148 (5)	0.083 (3)
C28	1.4290 (7)	0.8691 (7)	−0.1303 (4)	0.074 (3)

C29	1.3603 (7)	0.7466 (7)	−0.1017 (4)	0.072 (3)
C30	1.3043 (6)	0.5853 (6)	0.0309 (5)	0.067 (3)
C31	1.3047 (5)	0.5591 (6)	0.1374 (4)	0.062 (2)
C32	1.1600 (6)	0.8918 (5)	0.1376 (4)	0.061 (2)
H1	−0.03660	0.01310	0.29270	0.0790*
H1A	−0.05330	0.26180	0.51740	0.1110*
H4	−0.33780	0.12120	0.53690	0.0770*
H5	−0.39000	−0.01970	0.43680	0.0760*
H6	−0.24280	−0.07320	0.31490	0.0810*
H7	0.13060	0.15120	0.33490	0.0700*
H9	0.15120	0.48660	0.45990	0.0610*
H10	0.34250	0.59020	0.42470	0.0570*
H12	0.53270	0.26060	0.32700	0.0640*
H13	0.33910	0.15870	0.35890	0.0700*
H15	0.70380	0.35960	0.30480	0.0560*
H18	0.81190	0.66280	0.35350	0.0570*
H19	1.01110	0.74080	0.29930	0.0570*
H21	1.09190	0.44220	0.14190	0.0650*
H22	0.89490	0.36420	0.19980	0.0630*
H23A	1.33070	0.77410	0.19620	0.0660*
H23B	1.20690	0.78210	0.26970	0.0660*
H24A	1.11920	1.20670	0.14780	0.1130*
H24B	1.01370	1.11800	0.12340	0.1130*
H25A	1.14130	1.09040	−0.01800	0.1050*
H25B	1.07570	1.24120	−0.01470	0.1050*
H26A	1.37080	1.29930	−0.10940	0.1070*
H26B	1.24710	1.23500	−0.13630	0.1070*
H27A	1.45310	1.11490	−0.18010	0.1000*
H27B	1.49370	1.08200	−0.07280	0.1000*
H28A	1.51690	0.85450	−0.10130	0.0890*
H28B	1.44380	0.88530	−0.19910	0.0890*
H29A	1.26690	0.76630	−0.12180	0.0860*
H29B	1.40630	0.67330	−0.13290	0.0860*
H30A	1.35700	0.50950	0.00560	0.0810*
H30B	1.21190	0.59570	0.00970	0.0810*
H31A	1.29350	0.46470	0.15920	0.0730*
H31B	1.39330	0.57160	0.15680	0.0730*
H32A	1.19180	0.88810	0.07210	0.0740*
H32B	1.06280	0.88840	0.14190	0.0740*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.066 (3)	0.084 (3)	0.084 (3)	−0.023 (2)	0.014 (2)	−0.041 (3)
O2	0.095 (3)	0.050 (2)	0.079 (3)	−0.015 (2)	0.011 (2)	−0.016 (2)
O3	0.092 (3)	0.084 (3)	0.075 (3)	−0.033 (3)	−0.014 (3)	0.003 (2)
O4	0.075 (3)	0.080 (3)	0.064 (3)	−0.029 (3)	0.005 (2)	−0.007 (2)
O5	0.077 (3)	0.072 (3)	0.055 (3)	−0.027 (2)	0.010 (2)	−0.014 (2)

N1	0.050 (3)	0.051 (3)	0.059 (3)	−0.013 (2)	−0.001 (2)	−0.012 (2)
N2	0.041 (3)	0.047 (3)	0.064 (3)	−0.011 (2)	0.009 (2)	−0.007 (2)
N3	0.058 (3)	0.056 (3)	0.091 (4)	−0.019 (3)	0.013 (3)	−0.015 (3)
C1	0.069 (4)	0.067 (4)	0.071 (4)	−0.022 (3)	0.003 (3)	−0.028 (3)
C2	0.046 (3)	0.048 (3)	0.058 (3)	−0.010 (3)	0.003 (3)	−0.007 (3)
C3	0.051 (3)	0.044 (3)	0.065 (4)	−0.006 (3)	−0.005 (3)	−0.016 (3)
C4	0.047 (3)	0.063 (4)	0.084 (4)	−0.015 (3)	0.010 (3)	−0.016 (3)
C5	0.046 (3)	0.057 (4)	0.090 (5)	−0.015 (3)	−0.002 (3)	−0.014 (3)
C6	0.062 (4)	0.066 (4)	0.082 (5)	−0.019 (3)	−0.003 (4)	−0.025 (3)
C7	0.059 (4)	0.062 (4)	0.055 (4)	−0.006 (3)	0.002 (3)	−0.012 (3)
C8	0.041 (3)	0.051 (3)	0.054 (3)	−0.013 (3)	0.001 (3)	−0.001 (3)
C9	0.043 (3)	0.051 (3)	0.056 (3)	−0.007 (3)	0.003 (3)	−0.006 (3)
C10	0.044 (3)	0.046 (3)	0.052 (3)	−0.009 (3)	0.003 (3)	−0.008 (2)
C11	0.043 (3)	0.044 (3)	0.041 (3)	−0.011 (2)	0.001 (2)	−0.004 (2)
C12	0.045 (3)	0.051 (3)	0.065 (4)	−0.011 (3)	0.009 (3)	−0.011 (3)
C13	0.055 (4)	0.048 (3)	0.074 (4)	−0.018 (3)	0.009 (3)	−0.013 (3)
C14	0.043 (3)	0.043 (3)	0.042 (3)	−0.012 (2)	−0.001 (2)	−0.004 (2)
C15	0.047 (3)	0.042 (3)	0.052 (3)	−0.012 (3)	−0.001 (3)	−0.004 (2)
C16	0.042 (3)	0.056 (4)	0.055 (3)	−0.016 (3)	0.005 (2)	−0.005 (3)
C17	0.039 (3)	0.042 (3)	0.049 (3)	−0.009 (2)	0.001 (2)	−0.003 (2)
C18	0.044 (3)	0.055 (3)	0.044 (3)	−0.013 (3)	0.004 (2)	−0.012 (3)
C19	0.047 (3)	0.050 (3)	0.049 (3)	−0.016 (3)	−0.001 (3)	−0.012 (2)
C20	0.039 (3)	0.044 (3)	0.047 (3)	−0.007 (2)	0.001 (2)	−0.001 (2)
C21	0.052 (3)	0.042 (3)	0.069 (4)	−0.010 (3)	0.012 (3)	−0.016 (3)
C22	0.051 (3)	0.041 (3)	0.067 (4)	−0.013 (3)	0.008 (3)	−0.013 (3)
C23	0.047 (3)	0.056 (4)	0.064 (4)	−0.017 (3)	0.004 (3)	−0.007 (3)
C24	0.100 (6)	0.054 (4)	0.124 (7)	0.001 (4)	0.021 (5)	−0.014 (4)
C25	0.080 (5)	0.069 (5)	0.104 (6)	−0.003 (4)	−0.015 (4)	0.010 (4)
C26	0.112 (6)	0.083 (5)	0.069 (5)	−0.037 (5)	−0.020 (4)	0.018 (4)
C27	0.104 (6)	0.089 (5)	0.059 (4)	−0.045 (5)	−0.004 (4)	0.009 (4)
C28	0.080 (4)	0.097 (5)	0.046 (4)	−0.023 (4)	0.008 (3)	−0.009 (3)
C29	0.074 (4)	0.091 (5)	0.055 (4)	−0.016 (4)	0.005 (3)	−0.025 (3)
C30	0.061 (4)	0.065 (4)	0.081 (5)	−0.018 (3)	0.014 (3)	−0.024 (3)
C31	0.045 (3)	0.055 (4)	0.080 (4)	−0.008 (3)	0.010 (3)	−0.002 (3)
C32	0.075 (4)	0.048 (3)	0.063 (4)	−0.018 (3)	0.007 (3)	−0.010 (3)

Geometric parameters (Å, º)

O1—C3	1.344 (7)	C23—C32	1.519 (8)
O2—C24	1.423 (9)	C24—C25	1.476 (12)
O2—C32	1.403 (7)	C26—C27	1.487 (11)
O3—C25	1.410 (9)	C28—C29	1.484 (10)
O3—C26	1.423 (8)	C30—C31	1.494 (9)
O4—C27	1.416 (9)	C1—H1	0.9300
O4—C28	1.419 (8)	C4—H4	0.9300
O5—C29	1.410 (7)	C5—H5	0.9300
O5—C30	1.419 (7)	C6—H6	0.9300
N1—C7	1.245 (7)	C7—H7	0.9300

N1—C8	1.430 (6)	C9—H9	0.9300
O1—H1A	0.8200	C10—H10	0.9300
N2—C20	1.381 (7)	C12—H12	0.9300
N2—C31	1.448 (7)	C13—H13	0.9300
N2—C23	1.459 (7)	C15—H15	0.9300
N3—C16	1.142 (8)	C18—H18	0.9300
C1—C6	1.389 (9)	C19—H19	0.9300
C1—C2	1.366 (8)	C21—H21	0.9300
C2—C3	1.376 (8)	C22—H22	0.9300
C2—C7	1.548 (8)	C23—H23A	0.9700
C3—C4	1.392 (8)	C23—H23B	0.9700
C4—C5	1.380 (9)	C24—H24A	0.9700
C5—C6	1.361 (9)	C24—H24B	0.9700
C8—C13	1.387 (8)	C25—H25A	0.9700
C8—C9	1.382 (7)	C25—H25B	0.9700
C9—C10	1.376 (7)	C26—H26A	0.9700
C10—C11	1.389 (7)	C26—H26B	0.9700
C11—C12	1.391 (7)	C27—H27A	0.9700
C11—C14	1.492 (7)	C27—H27B	0.9700
C12—C13	1.381 (8)	C28—H28A	0.9700
C14—C16	1.438 (8)	C28—H28B	0.9700
C14—C15	1.348 (7)	C29—H29A	0.9700
C15—C17	1.456 (7)	C29—H29B	0.9700
C17—C18	1.392 (7)	C30—H30A	0.9700
C17—C22	1.385 (7)	C30—H30B	0.9700
C18—C19	1.376 (7)	C31—H31A	0.9700
C19—C20	1.403 (7)	C31—H31B	0.9700
C20—C21	1.402 (7)	C32—H32A	0.9700
C21—C22	1.371 (7)	C32—H32B	0.9700

C24—O2—C32	113.2 (5)	C9—C10—H10	119.00
C25—O3—C26	112.6 (6)	C11—C10—H10	119.00
C27—O4—C28	111.9 (5)	C11—C12—H12	119.00
C29—O5—C30	113.7 (5)	C13—C12—H12	119.00
C7—N1—C8	117.6 (5)	C8—C13—H13	120.00
C3—O1—H1A	110.00	C12—C13—H13	120.00
C20—N2—C31	120.6 (4)	C14—C15—H15	114.00
C23—N2—C31	118.2 (4)	C17—C15—H15	114.00
C20—N2—C23	121.2 (4)	C17—C18—H18	119.00
C2—C1—C6	119.5 (5)	C19—C18—H18	119.00
C1—C2—C7	118.2 (5)	C18—C19—H19	119.00
C3—C2—C7	120.0 (5)	C20—C19—H19	119.00
C1—C2—C3	121.8 (5)	C20—C21—H21	119.00
O1—C3—C2	122.9 (5)	C22—C21—H21	120.00
C2—C3—C4	118.4 (5)	C17—C22—H22	119.00
O1—C3—C4	118.7 (5)	C21—C22—H22	118.00
C3—C4—C5	119.8 (6)	N2—C23—H23A	109.00
C4—C5—C6	121.1 (6)	N2—C23—H23B	109.00

C1—C6—C5	119.5 (6)	C32—C23—H23A	109.00
N1—C7—C2	119.9 (5)	C32—C23—H23B	109.00
N1—C8—C9	116.8 (4)	H23A—C23—H23B	108.00
C9—C8—C13	118.6 (5)	O2—C24—H24A	108.00
N1—C8—C13	124.6 (5)	O2—C24—H24B	108.00
C8—C9—C10	120.8 (5)	C25—C24—H24A	108.00
C9—C10—C11	121.4 (5)	C25—C24—H24B	108.00
C10—C11—C14	120.9 (4)	H24A—C24—H24B	107.00
C12—C11—C14	121.7 (4)	O3—C25—H25A	110.00
C10—C11—C12	117.4 (5)	O3—C25—H25B	110.00
C11—C12—C13	121.4 (5)	C24—C25—H25A	110.00
C8—C13—C12	120.4 (5)	C24—C25—H25B	110.00
C11—C14—C16	114.6 (4)	H25A—C25—H25B	108.00
C15—C14—C16	120.3 (5)	O3—C26—H26A	109.00
C11—C14—C15	125.0 (5)	O3—C26—H26B	109.00
C14—C15—C17	131.4 (5)	C27—C26—H26A	109.00
N3—C16—C14	177.0 (6)	C27—C26—H26B	109.00
C15—C17—C18	125.5 (5)	H26A—C26—H26B	108.00
C15—C17—C22	118.4 (5)	O4—C27—H27A	110.00
C18—C17—C22	116.2 (5)	O4—C27—H27B	110.00
C17—C18—C19	121.9 (5)	C26—C27—H27A	110.00
C18—C19—C20	121.6 (5)	C26—C27—H27B	110.00
N2—C20—C21	122.0 (5)	H27A—C27—H27B	108.00
C19—C20—C21	116.3 (5)	O4—C28—H28A	110.00
N2—C20—C19	121.7 (5)	O4—C28—H28B	110.00
C20—C21—C22	121.0 (5)	C29—C28—H28A	110.00
C17—C22—C21	123.0 (5)	C29—C28—H28B	110.00
N2—C23—C32	111.6 (4)	H28A—C28—H28B	108.00
O2—C24—C25	115.9 (6)	O5—C29—H29A	110.00
O3—C25—C24	109.8 (6)	O5—C29—H29B	110.00
O3—C26—C27	113.6 (6)	C28—C29—H29A	110.00
O4—C27—C26	110.0 (6)	C28—C29—H29B	110.00
O4—C28—C29	110.1 (5)	H29A—C29—H29B	108.00
O5—C29—C28	110.2 (5)	O5—C30—H30A	110.00
O5—C30—C31	108.2 (5)	O5—C30—H30B	110.00
N2—C31—C30	115.9 (5)	C31—C30—H30A	110.00
O2—C32—C23	108.1 (5)	C31—C30—H30B	110.00
C2—C1—H1	120.00	H30A—C30—H30B	108.00
C6—C1—H1	120.00	N2—C31—H31A	108.00
C3—C4—H4	120.00	N2—C31—H31B	108.00
C5—C4—H4	120.00	C30—C31—H31A	108.00
C4—C5—H5	120.00	C30—C31—H31B	108.00
C6—C5—H5	119.00	H31A—C31—H31B	107.00
C1—C6—H6	120.00	O2—C32—H32A	110.00
C5—C6—H6	120.00	O2—C32—H32B	110.00
N1—C7—H7	120.00	C23—C32—H32A	110.00
C2—C7—H7	120.00	C23—C32—H32B	110.00
C8—C9—H9	120.00	H32A—C32—H32B	108.00

C10—C9—H9	120.00		
C24—O2—C32—C23	−171.5 (5)	N1—C8—C13—C12	179.7 (5)
C32—O2—C24—C25	−69.7 (8)	C9—C8—C13—C12	−1.7 (8)
C26—O3—C25—C24	168.2 (6)	C13—C8—C9—C10	2.6 (8)
C25—O3—C26—C27	−97.6 (7)	C8—C9—C10—C11	−1.3 (8)
C28—O4—C27—C26	173.0 (5)	C9—C10—C11—C14	178.6 (5)
C27—O4—C28—C29	171.7 (5)	C9—C10—C11—C12	−0.8 (8)
C30—O5—C29—C28	−177.1 (5)	C12—C11—C14—C15	−4.0 (7)
C29—O5—C30—C31	−176.3 (5)	C14—C11—C12—C13	−177.7 (5)
C7—N1—C8—C13	−30.1 (8)	C10—C11—C12—C13	1.6 (8)
C7—N1—C8—C9	151.2 (5)	C10—C11—C14—C16	−7.3 (6)
C8—N1—C7—C2	−177.9 (5)	C10—C11—C14—C15	176.7 (5)
C31—N2—C23—C32	105.9 (5)	C12—C11—C14—C16	172.1 (5)
C20—N2—C23—C32	−76.2 (6)	C11—C12—C13—C8	−0.4 (8)
C31—N2—C20—C21	−12.4 (8)	C16—C14—C15—C17	2.0 (9)
C23—N2—C20—C19	−11.3 (8)	C11—C14—C15—C17	177.9 (5)
C20—N2—C31—C30	93.6 (6)	C14—C15—C17—C18	21.2 (9)
C31—N2—C20—C19	166.6 (5)	C14—C15—C17—C22	−158.1 (6)
C23—N2—C20—C21	169.7 (5)	C18—C17—C22—C21	−1.3 (8)
C23—N2—C31—C30	−88.4 (6)	C15—C17—C22—C21	178.1 (5)
C2—C1—C6—C5	−0.4 (9)	C15—C17—C18—C19	−178.8 (5)
C6—C1—C2—C7	−178.6 (5)	C22—C17—C18—C19	0.5 (8)
C6—C1—C2—C3	−0.3 (9)	C17—C18—C19—C20	−0.7 (8)
C7—C2—C3—O1	−1.4 (8)	C18—C19—C20—C21	1.4 (8)
C3—C2—C7—N1	−2.1 (8)	C18—C19—C20—N2	−177.6 (5)
C7—C2—C3—C4	179.1 (5)	C19—C20—C21—C22	−2.1 (8)
C1—C2—C3—O1	−179.7 (5)	N2—C20—C21—C22	176.9 (5)
C1—C2—C7—N1	176.3 (5)	C20—C21—C22—C17	2.2 (8)
C1—C2—C3—C4	0.8 (8)	N2—C23—C32—O2	175.6 (4)
C2—C3—C4—C5	−0.7 (9)	O2—C24—C25—O3	−67.8 (8)
O1—C3—C4—C5	179.8 (6)	O3—C26—C27—O4	63.2 (8)
C3—C4—C5—C6	0.0 (10)	O4—C28—C29—O5	−71.0 (6)
C4—C5—C6—C1	0.5 (10)	O5—C30—C31—N2	76.3 (6)
N1—C8—C9—C10	−178.7 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N1	0.82	1.87	2.600 (6)	148
C25—H25*A*···O4	0.97	2.50	3.129 (9)	122
C28—H28*A*···O3^{i}	0.97	2.53	3.493 (8)	170

Symmetry code: (i) −*x*+3, −*y*+2, −*z*.