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4b,5,7,7a-Tetrahydro-4b,7a-epimino-methanoimino-6*H*-imidazo[4,5-*f*][1,10]-phenanthroline-6,13-dione monohydrate

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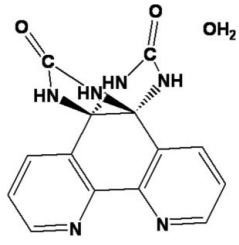
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.092; data-to-parameter ratio = 12.0.

The title compound, $C_{14}H_{10}N_6O_2\cdot H_2O$, has inherent hydrogen-bonding sites that allow for a very tight packing structure. There are two organic molecules and two water molecules in the asymmetric unit, which are connected by N—H···O hydrogen bonds. The O atoms come from both water and urea, giving rise to a complex three-dimensional molecular cluster. Features associated with the hydrogen bonding and solid-state emission and excitation spectra are discussed in the supplementary material.

Related literature

Synthesis: Deshpande *et al.* (2006); Elemans *et al.* (1998, 2002); Kurth *et al.* (2001).



Experimental

Crystal data

$C_{14}H_{10}N_6O_2\cdot H_2O$
M_r = 312.30
Monoclinic, $P2_1/c$
a = 18.5164 (18) Å
b = 12.3920 (12) Å
c = 12.6929 (13) Å
β = 108.528 (5)°
V = 2761.5 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 150 K
0.49 × 0.30 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.938, T_{max} = 0.973
68322 measured reflections
5422 independent reflections
4600 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.092
S = 1.03
5422 reflections
451 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N12—H12···O1	0.90 (2)	1.835 (2)	2.726 (2)	172.5 (1)
N3—H3···O2S	0.826 (19)	2.082 (2)	2.907 (2)	176.3 (1)
N10—H10···O2S	0.86	1.99	2.833 (2)	166

Data collection: *SMART* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2006); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* and *ORPEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2142).

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supplementary materials

Acta Cryst. (2007). E**63**, o3526 [**doi:10.1107/S1600536807034198**]

4b,5,7,7a-Tetrahydro-4b,7a-epiminomethanoimino-6*H*-imidazo[4,5-*f*][1,10]phenanthroline-6,13-dione monohydrate

D. P. Rillema, R. A. Kirgan, C. Moore and S. Koshy

Comment

The title compound (Scheme 1) has been snythesized before and included in many different metal systems as a ligand. Crystal structures of some of the complexes have been reported. (Deshpande *et al.*, 2006; Elemans *et al.*, 1998; Elemans *et al.*, 2002; Kurth *et al.*, 2001). To date the structure of the unbound ligand has not been solved. According to the crystal structure (Figure 1), each molecule forms three hydrogen bonds; one with another molecule and two with two different water molecules. Each water molecule forms four hydrogen bonds; two to urea NH groups, one to a urea ketone and one to a bipyridine nitrogen atom. The bipyridine rings are twisted out of plane which causes the urea groups to also twist. The hydrogen bonds of interest occur between H12—O1, H3—O2s, and H10—O2s, Table 1; these three bonds form a ring that gives stability to the molecule in the packing arrangement.

Photophysical Processes: The emission spectra and excitation spectra were determined in the solid state. The excitation spectra shows one major transition at 375 nm and is assigned to a $\pi \rightarrow \pi^*$ transition within the bipyridine moiety. The emission spectra and excitation spectra have good overlap. The emission spectra reveals very nice vibrational structure. The three major peaks are located at 405, 426, and 453 nm. The vibrational band spacing was found to be 1300 cm^{-1}.

Experimental

1,10-phenanthroline-5,6-dione was prepared as previously reported. Urea was purchased from Aldrich and used as received. Toluene and glacial acetic acid were purchased from Fisher Scientific Company and used as received. 200 proof ethanol was purchased from AAPER alcohol and used without further purification.

Physical Measurements: Fluorescence measurements were obtained with a Spex Fluorolog 2:1:2 spectrophotometer. The crystals were placed between two glass slides and then lined up within the excitation chamber using a mount. The slit widths were maintained at 1.4 mm and the light intensity collected with a Hamamatsu R928 photomultiplier tube with a 900 V bias. Data was gathered at intervals of 0.1 nanometers and integrated for 0.1 s. The data was processed using the Origin 6.1 suite of software and massaged to give smoother spectra without loss of spectral features.

The title molecule was prepared and purified as previously reported. Crystals were obtained by refluxing the title molecule in glacial acetic acid and then allowing the solution to cool. Crystals suitable for X-Ray analysis formed on the bottom of the flask. Elemental Anyalysis: Anal. for $C_{14}H_{10}N_6O_2 \cdot 2H_2O$: %C, 50.90; %H, 4.24; %N, 25.45; Found. %C, 50.90; %H, 3.60; %N, 24.77.

Refinement

All non-H atoms were refined using anisotropic displacement parameters. Hydrogen atoms on the solvate water molecule and urea N atoms were refined using isotropic displacement parameters. All other H atoms were included at idealized positions and not refined.

Figures

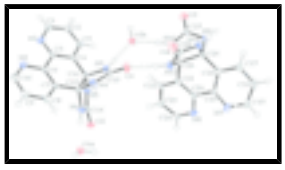

Fig. 1. View of (1) (50% probability displacement ellipsoids) H atoms have been added for clarity of hydrogen bonding (Farrugia, 1997).

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Crystal data

$C_{14}H_{10}N_6O_2·H_2O$	F_{000} = 1296
M_r = 312.30	D_x = 1.502 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P2ybc	Cell parameters from 9312 reflections
a = 18.5164 (18) Å	θ = 2.9–27.1º
b = 12.3920 (12) Å	μ = 0.11 mm^{-1}
c = 12.6929 (13) Å	T = 150 K
β = 108.528 (5)º	Block, lusterous grey
V = 2761.5 (5) Å^3	0.49 × 0.30 × 0.25 mm
Z = 8	

Data collection

Bruker SMART CCD area-detector diffractometer	5422 independent reflections
Radiation source: fine-focus sealed tube	4600 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.032
T = 296(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 3.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −22→22
T_{min} = 0.938, T_{max} = 0.973	k = −15→15
68322 measured reflections	l = −15→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.092$

$S = 1.03$

5422 reflections

451 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 1.7397P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.51$ e Å^{-3}

$\Delta\rho_{min} = -0.27$ e Å^{-3}

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 $F^2 > 2$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	0.57536 (9)	0.51053 (14)	0.23035 (13)	0.0250 (3)
H1	0.6246	0.5105	0.2261	0.030*
C2	0.52109 (9)	0.44802 (14)	0.15553 (14)	0.0269 (4)
H2	0.5337	0.4060	0.1032	0.032*
C3	0.44753 (9)	0.44943 (13)	0.16025 (13)	0.0235 (3)
H3A	0.4095	0.4093	0.1100	0.028*
C4	0.43105 (8)	0.51152 (11)	0.24095 (12)	0.0169 (3)
C5	0.48988 (8)	0.57012 (12)	0.31489 (12)	0.0165 (3)
C6	0.47687 (8)	0.63485 (11)	0.40588 (11)	0.0168 (3)
C7	0.40602 (8)	0.63639 (11)	0.42213 (11)	0.0163 (3)
C8	0.39791 (9)	0.69468 (13)	0.51159 (13)	0.0233 (3)
H8	0.3512	0.6975	0.5245	0.028*
C9	0.46028 (9)	0.74809 (13)	0.58058 (13)	0.0262 (4)
H9A	0.4568	0.7866	0.6416	0.031*
C10	0.52840 (9)	0.74329 (13)	0.55719 (13)	0.0244 (3)
H10A	0.5702	0.7800	0.6039	0.029*
C11	0.34995 (8)	0.51796 (11)	0.24373 (11)	0.0153 (3)
C12	0.33911 (8)	0.57274 (11)	0.34914 (11)	0.0156 (3)
C13	0.30689 (8)	0.39172 (12)	0.34652 (12)	0.0178 (3)
C14	0.25196 (8)	0.64364 (12)	0.18811 (12)	0.0183 (3)
C15	−0.10575 (9)	−0.01787 (15)	0.16880 (14)	0.0300 (4)

H15	−0.1562	−0.0171	0.1679	0.036*
C16	−0.06494 (9)	−0.11252 (14)	0.19842 (14)	0.0291 (4)
H16	−0.0884	−0.1752	0.2115	0.035*
C17	0.01127 (9)	−0.11196 (13)	0.20813 (13)	0.0241 (3)
H17	0.0410	−0.1732	0.2323	0.029*
C18	0.04323 (8)	−0.01847 (12)	0.18134 (12)	0.0182 (3)
C19	−0.00374 (8)	0.07056 (12)	0.14324 (12)	0.0194 (3)
C20	0.02424 (8)	0.16725 (12)	0.09892 (12)	0.0191 (3)
C21	0.10197 (8)	0.18120 (12)	0.11607 (12)	0.0176 (3)
C22	0.12512 (9)	0.27078 (12)	0.06906 (13)	0.0226 (3)
H22	0.1764	0.2821	0.0785	0.027*
C23	0.07080 (9)	0.34281 (13)	0.00801 (14)	0.0276 (4)
H23	0.0849	0.4038	−0.0235	0.033*
C24	−0.00518 (9)	0.32203 (13)	−0.00505 (14)	0.0274 (4)
H24	−0.0416	0.3704	−0.0464	0.033*
C25	0.12815 (8)	−0.01245 (11)	0.19996 (12)	0.0168 (3)
C26	0.15898 (8)	0.10310 (12)	0.18926 (12)	0.0169 (3)
C27	0.21494 (8)	−0.02458 (12)	0.10467 (12)	0.0197 (3)
C28	0.20351 (8)	0.05141 (12)	0.37338 (12)	0.0208 (3)
H3	0.3210 (10)	0.3610 (15)	0.2068 (15)	0.024 (5)*
H4	0.3084 (11)	0.4909 (16)	0.4657 (17)	0.034 (5)*
H5	0.2869 (11)	0.5538 (15)	0.0913 (17)	0.029 (5)*
H9	0.1498 (11)	−0.1433 (17)	0.1173 (16)	0.033 (5)*
H12	0.2169 (10)	0.1955 (16)	0.3208 (15)	0.030 (5)*
H1S	0.1442 (12)	0.7076 (18)	0.3928 (17)	0.042 (6)*
H2S	0.1900 (13)	0.7700 (18)	0.475 (2)	0.052 (7)*
H3S	0.3089 (13)	0.2631 (19)	0.538 (2)	0.052 (7)*
H4S	0.3637 (13)	0.3118 (18)	0.6218 (18)	0.047 (6)*
N1	0.56110 (7)	0.57086 (11)	0.30836 (10)	0.0212 (3)
N2	0.53740 (7)	0.68914 (10)	0.47175 (10)	0.0219 (3)
N3	0.31824 (7)	0.41111 (10)	0.24826 (11)	0.0188 (3)
N4	0.32376 (7)	0.48165 (10)	0.40966 (10)	0.0196 (3)
N5	0.30309 (7)	0.58453 (10)	0.15337 (10)	0.0185 (3)
N6	0.27320 (7)	0.64070 (11)	0.30005 (10)	0.0204 (3)
H6	0.2500	0.6755	0.3384	0.024*
N7	−0.07688 (7)	0.07247 (11)	0.14138 (11)	0.0254 (3)
N8	−0.02873 (7)	0.23627 (11)	0.03853 (11)	0.0256 (3)
N9	0.15350 (7)	−0.07390 (11)	0.12206 (11)	0.0203 (3)
N10	0.22032 (7)	0.07727 (10)	0.14546 (11)	0.0200 (3)
H10	0.2561	0.1215	0.1454	0.024*
N11	0.17069 (7)	−0.03684 (10)	0.31552 (10)	0.0206 (3)
H11	0.1744	−0.1005	0.3437	0.025*
N12	0.18480 (7)	0.13935 (10)	0.30379 (10)	0.0202 (3)
O1	0.28503 (6)	0.30545 (9)	0.37483 (9)	0.0255 (3)
O2	0.19669 (6)	0.69224 (9)	0.12627 (9)	0.0242 (2)
O3	0.25792 (6)	−0.06517 (9)	0.05968 (9)	0.0267 (3)
O4	0.24213 (7)	0.05573 (9)	0.47204 (9)	0.0297 (3)
O1S	0.17949 (6)	0.75357 (9)	0.40465 (10)	0.0208 (2)
O2S	0.32338 (6)	0.27096 (9)	0.60645 (10)	0.0197 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0189 (8)	0.0316 (9)	0.0270 (8)	0.0013 (7)	0.0110 (6)	−0.0040 (7)
C2	0.0283 (8)	0.0293 (9)	0.0277 (8)	−0.0016 (7)	0.0154 (7)	−0.0090 (7)
C3	0.0241 (8)	0.0244 (8)	0.0235 (8)	−0.0058 (6)	0.0095 (6)	−0.0075 (6)
C4	0.0193 (7)	0.0151 (7)	0.0170 (7)	−0.0002 (6)	0.0070 (6)	0.0016 (6)
C5	0.0168 (7)	0.0162 (7)	0.0163 (7)	0.0010 (6)	0.0048 (6)	0.0028 (6)
C6	0.0182 (7)	0.0147 (7)	0.0161 (7)	0.0008 (6)	0.0033 (6)	0.0018 (6)
C7	0.0181 (7)	0.0150 (7)	0.0149 (7)	0.0002 (6)	0.0040 (6)	0.0011 (6)
C8	0.0232 (8)	0.0251 (8)	0.0243 (8)	−0.0026 (6)	0.0113 (6)	−0.0050 (6)
C9	0.0293 (9)	0.0277 (9)	0.0216 (8)	−0.0017 (7)	0.0081 (7)	−0.0100 (7)
C10	0.0215 (8)	0.0238 (8)	0.0239 (8)	−0.0027 (6)	0.0016 (6)	−0.0076 (6)
C11	0.0169 (7)	0.0148 (7)	0.0137 (7)	−0.0023 (5)	0.0041 (5)	−0.0017 (5)
C12	0.0150 (7)	0.0170 (7)	0.0156 (7)	−0.0002 (6)	0.0059 (6)	0.0004 (6)
C13	0.0131 (7)	0.0207 (8)	0.0176 (7)	−0.0014 (6)	0.0020 (6)	0.0020 (6)
C14	0.0180 (7)	0.0163 (7)	0.0199 (7)	−0.0014 (6)	0.0049 (6)	0.0004 (6)
C15	0.0176 (8)	0.0381 (10)	0.0353 (9)	−0.0022 (7)	0.0098 (7)	0.0106 (8)
C16	0.0235 (8)	0.0294 (9)	0.0346 (9)	−0.0058 (7)	0.0094 (7)	0.0100 (7)
C17	0.0226 (8)	0.0224 (8)	0.0264 (8)	−0.0003 (6)	0.0065 (6)	0.0060 (7)
C18	0.0175 (7)	0.0208 (7)	0.0162 (7)	−0.0016 (6)	0.0053 (6)	0.0000 (6)
C19	0.0170 (7)	0.0233 (8)	0.0174 (7)	−0.0013 (6)	0.0050 (6)	0.0014 (6)
C20	0.0185 (7)	0.0196 (7)	0.0188 (7)	0.0008 (6)	0.0053 (6)	0.0001 (6)
C21	0.0193 (7)	0.0169 (7)	0.0168 (7)	0.0000 (6)	0.0059 (6)	−0.0011 (6)
C22	0.0195 (7)	0.0219 (8)	0.0270 (8)	−0.0012 (6)	0.0082 (6)	0.0025 (6)
C23	0.0282 (8)	0.0228 (8)	0.0330 (9)	0.0016 (7)	0.0116 (7)	0.0088 (7)
C24	0.0235 (8)	0.0232 (8)	0.0334 (9)	0.0048 (7)	0.0062 (7)	0.0105 (7)
C25	0.0181 (7)	0.0147 (7)	0.0168 (7)	0.0002 (6)	0.0044 (6)	0.0009 (6)
C26	0.0149 (7)	0.0166 (7)	0.0189 (7)	−0.0014 (6)	0.0052 (6)	−0.0009 (6)
C27	0.0191 (7)	0.0208 (8)	0.0188 (7)	0.0006 (6)	0.0055 (6)	0.0011 (6)
C28	0.0204 (7)	0.0212 (8)	0.0200 (8)	0.0008 (6)	0.0051 (6)	0.0003 (6)
N1	0.0167 (6)	0.0243 (7)	0.0227 (7)	0.0004 (5)	0.0063 (5)	−0.0017 (5)
N2	0.0185 (6)	0.0227 (7)	0.0223 (7)	−0.0014 (5)	0.0036 (5)	−0.0042 (5)
N3	0.0219 (7)	0.0158 (6)	0.0195 (6)	−0.0040 (5)	0.0078 (5)	−0.0043 (5)
N4	0.0241 (7)	0.0210 (7)	0.0160 (6)	−0.0051 (5)	0.0095 (5)	−0.0003 (5)
N5	0.0199 (6)	0.0215 (7)	0.0129 (6)	0.0017 (5)	0.0037 (5)	−0.0004 (5)
N6	0.0179 (6)	0.0258 (7)	0.0173 (6)	0.0067 (5)	0.0052 (5)	−0.0024 (5)
N7	0.0172 (6)	0.0297 (7)	0.0294 (7)	0.0008 (5)	0.0077 (6)	0.0072 (6)
N8	0.0198 (7)	0.0236 (7)	0.0317 (7)	0.0033 (5)	0.0059 (6)	0.0074 (6)
N9	0.0220 (7)	0.0166 (7)	0.0250 (7)	−0.0026 (5)	0.0113 (5)	−0.0040 (5)
N10	0.0155 (6)	0.0175 (6)	0.0289 (7)	−0.0023 (5)	0.0099 (5)	0.0000 (5)
N11	0.0224 (7)	0.0166 (6)	0.0194 (6)	−0.0005 (5)	0.0019 (5)	0.0039 (5)
N12	0.0230 (7)	0.0162 (6)	0.0182 (6)	−0.0031 (5)	0.0020 (5)	−0.0011 (5)
O1	0.0283 (6)	0.0230 (6)	0.0234 (6)	−0.0104 (5)	0.0056 (5)	0.0023 (5)
O2	0.0227 (6)	0.0239 (6)	0.0224 (6)	0.0061 (5)	0.0020 (5)	0.0036 (5)
O3	0.0278 (6)	0.0256 (6)	0.0324 (6)	0.0000 (5)	0.0178 (5)	−0.0029 (5)
O4	0.0353 (7)	0.0282 (6)	0.0179 (6)	−0.0016 (5)	−0.0022 (5)	0.0011 (5)

O1S	0.0223 (6)	0.0195 (6)	0.0216 (6)	−0.0005 (5)	0.0086 (5)	0.0013 (5)
O2S	0.0185 (6)	0.0214 (6)	0.0200 (6)	−0.0015 (4)	0.0072 (4)	0.0011 (4)

Geometric parameters (Å, °)

C1—N1	1.332 (2)	C17—H17	0.9300
C1—C2	1.380 (2)	C18—C19	1.393 (2)
C1—H1	0.9300	C18—C25	1.516 (2)
C2—C3	1.382 (2)	C19—N7	1.3471 (19)
C2—H2	0.9300	C19—C20	1.485 (2)
C3—C4	1.390 (2)	C20—N8	1.3428 (19)
C3—H3A	0.9300	C20—C21	1.396 (2)
C4—C5	1.395 (2)	C21—C22	1.391 (2)
C4—C11	1.515 (2)	C21—C26	1.513 (2)
C5—N1	1.3474 (19)	C22—C23	1.383 (2)
C5—C6	1.487 (2)	C22—H22	0.9300
C6—N2	1.3462 (19)	C23—C24	1.387 (2)
C6—C7	1.391 (2)	C23—H23	0.9300
C7—C8	1.394 (2)	C24—N8	1.334 (2)
C7—C12	1.5120 (19)	C24—H24	0.9300
C8—C9	1.377 (2)	C25—N9	1.4397 (19)
C8—H8	0.9300	C25—N11	1.4591 (18)
C9—C10	1.386 (2)	C25—C26	1.563 (2)
C9—H9A	0.9300	C26—N12	1.4498 (19)
C10—N2	1.330 (2)	C26—N10	1.4500 (19)
C10—H10A	0.9300	C27—O3	1.2253 (18)
C11—N5	1.4548 (18)	C27—N10	1.356 (2)
C11—N3	1.4569 (19)	C27—N9	1.370 (2)
C11—C12	1.5691 (19)	C28—O4	1.2302 (18)
C12—N4	1.4435 (19)	C28—N11	1.349 (2)
C12—N6	1.4502 (18)	C28—N12	1.376 (2)
C13—O1	1.2362 (18)	N3—H3	0.826 (19)
C13—N4	1.350 (2)	N4—H4	0.85 (2)
C13—N3	1.3506 (19)	N5—H5	0.84 (2)
C14—O2	1.2312 (18)	N6—H6	0.8600
C14—N6	1.3490 (19)	N9—H9	0.86 (2)
C14—N5	1.3756 (19)	N10—H10	0.8600
C15—N7	1.334 (2)	N11—H11	0.8600
C15—C16	1.381 (2)	N12—H12	0.90 (2)
C15—H15	0.9300	O1S—H1S	0.84 (2)
C16—C17	1.377 (2)	O1S—H2S	0.87 (2)
C16—H16	0.9300	O2S—H3S	0.83 (3)
C17—C18	1.391 (2)	O2S—H4S	0.87 (2)
N1—C1—C2	123.60 (14)	N8—C20—C21	122.67 (14)
N1—C1—H1	118.2	N8—C20—C19	116.78 (13)
C2—C1—H1	118.2	C21—C20—C19	120.48 (13)
C1—C2—C3	118.36 (14)	C22—C21—C20	118.35 (13)
C1—C2—H2	120.8	C22—C21—C26	121.59 (13)
C3—C2—H2	120.8	C20—C21—C26	119.99 (13)

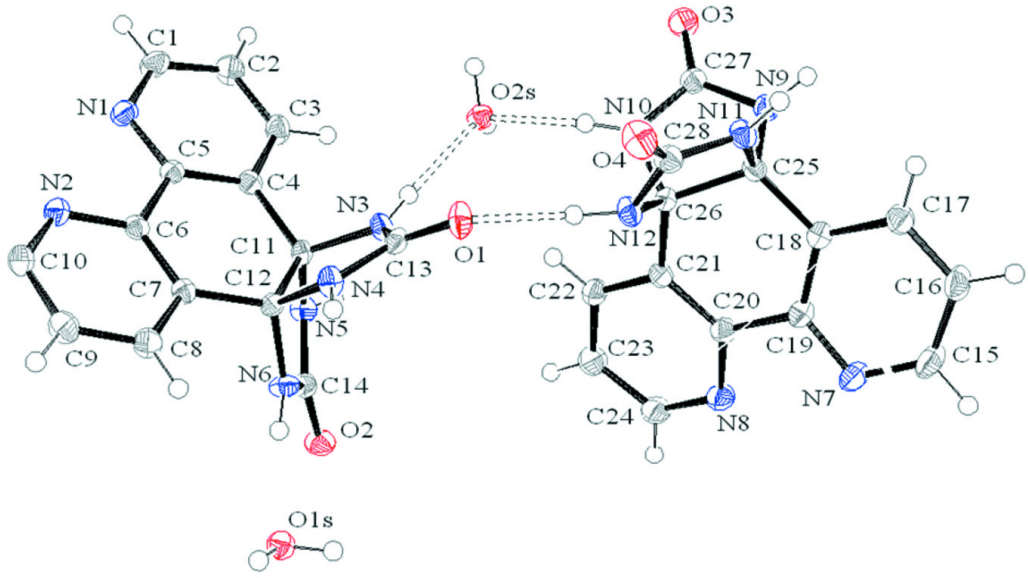
C2—C3—C4	119.27 (14)	C23—C22—C21	119.15 (14)
C2—C3—H3A	120.4	C23—C22—H22	120.4
C4—C3—H3A	120.4	C21—C22—H22	120.4
C3—C4—C5	118.46 (13)	C22—C23—C24	118.45 (15)
C3—C4—C11	119.83 (13)	C22—C23—H23	120.8
C5—C4—C11	121.66 (13)	C24—C23—H23	120.8
N1—C5—C4	122.18 (13)	N8—C24—C23	123.45 (15)
N1—C5—C6	116.57 (12)	N8—C24—H24	118.3
C4—C5—C6	121.25 (13)	C23—C24—H24	118.3
N2—C6—C7	122.63 (13)	N9—C25—N11	113.03 (12)
N2—C6—C5	116.33 (13)	N9—C25—C18	114.54 (12)
C7—C6—C5	121.03 (13)	N11—C25—C18	110.39 (12)
C6—C7—C8	118.60 (13)	N9—C25—C26	102.53 (11)
C6—C7—C12	121.99 (13)	N11—C25—C26	100.47 (11)
C8—C7—C12	119.35 (13)	C18—C25—C26	114.90 (12)
C9—C8—C7	118.79 (14)	N12—C26—N10	113.83 (12)
C9—C8—H8	120.6	N12—C26—C21	111.15 (12)
C7—C8—H8	120.6	N10—C26—C21	112.60 (12)
C8—C9—C10	118.66 (14)	N12—C26—C25	102.11 (11)
C8—C9—H9A	120.7	N10—C26—C25	100.56 (11)
C10—C9—H9A	120.7	C21—C26—C25	115.86 (11)
N2—C10—C9	123.69 (14)	O3—C27—N10	125.48 (14)
N2—C10—H10A	118.2	O3—C27—N9	125.99 (14)
C9—C10—H10A	118.2	N10—C27—N9	108.53 (13)
N5—C11—N3	113.56 (11)	O4—C28—N11	127.22 (14)
N5—C11—C4	111.02 (11)	O4—C28—N12	124.43 (14)
N3—C11—C4	111.58 (12)	N11—C28—N12	108.33 (12)
N5—C11—C12	102.77 (11)	C1—N1—C5	118.09 (13)
N3—C11—C12	101.37 (11)	C10—N2—C6	117.59 (13)
C4—C11—C12	116.03 (11)	C13—N3—C11	112.40 (12)
N4—C12—N6	113.68 (12)	C13—N3—H3	121.0 (12)
N4—C12—C7	110.09 (11)	C11—N3—H3	123.4 (12)
N6—C12—C7	111.97 (12)	C13—N4—C12	112.16 (12)
N4—C12—C11	102.45 (11)	C13—N4—H4	122.0 (13)
N6—C12—C11	101.72 (11)	C12—N4—H4	120.8 (13)
C7—C12—C11	116.50 (11)	C14—N5—C11	110.37 (12)
O1—C13—N4	125.22 (14)	C14—N5—H5	117.7 (13)
O1—C13—N3	125.71 (14)	C11—N5—H5	115.4 (13)
N4—C13—N3	109.07 (13)	C14—N6—C12	113.08 (12)
O2—C14—N6	126.14 (14)	C14—N6—H6	123.5
O2—C14—N5	124.99 (14)	C12—N6—H6	123.5
N6—C14—N5	108.86 (12)	C15—N7—C19	117.70 (14)
N7—C15—C16	123.63 (15)	C24—N8—C20	117.93 (13)
N7—C15—H15	118.2	C27—N9—C25	110.09 (12)
C16—C15—H15	118.2	C27—N9—H9	119.0 (13)
C17—C16—C15	118.47 (15)	C25—N9—H9	122.6 (13)
C17—C16—H16	120.8	C27—N10—C26	112.37 (12)
C15—C16—H16	120.8	C27—N10—H10	123.8
C16—C17—C18	119.06 (15)	C26—N10—H10	123.8

C16—C17—H17	120.5	C28—N11—C25	112.65 (12)
C18—C17—H17	120.5	C28—N11—H11	123.7
C17—C18—C19	118.50 (13)	C25—N11—H11	123.7
C17—C18—C25	120.24 (13)	C28—N12—C26	109.51 (12)
C19—C18—C25	121.16 (13)	C28—N12—H12	116.7 (12)
N7—C19—C18	122.23 (14)	C26—N12—H12	117.7 (12)
N7—C19—C20	116.68 (13)	H1S—O1S—H2S	104 (2)
C18—C19—C20	121.06 (13)	H3S—O2S—H4S	106 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N12—H12···O1	0.90 (2)	1.835 (2)	2.726 (2)	172.5 (1)
N3—H3···O2S	0.826 (19)	2.082 (2)	2.907 (2)	176.3 (1)
N10—H10···O2S	0.86	1.99	2.833 (2)	166

Fig. 1


C1
C2
N1
C3
C5
C4
N2
C6
C10
C11
C12
C7
C13
N3
O1
N4
N5
C9
C8
N6
C14
O2
O1s
O2s
O3
C27
N9
N10
N11
C28
O4
C25
C26
N12
C17
C18
C21
C22
C16
C20
C23
C19
C15
C24
N8
N7