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2-[(*E*)-2-(4-Ethoxyphenyl)ethenyl]-1-methylpyridinium iodide monohydrate

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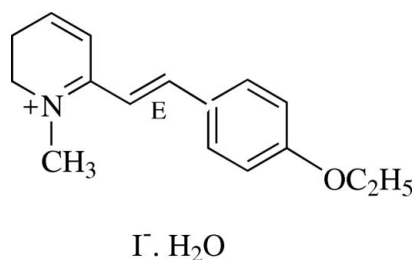
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 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.040; wR factor = 0.105; data-to-parameter ratio = 25.2.

In the title compound, $\text{C}_{16}\text{H}_{18}\text{NO}^+\cdot\text{I}^-\cdot\text{H}_2\text{O}$, the cation is essentially planar, with a dihedral angle of $3.13(16)^\circ$ between the pyridinium and benzene rings. The molecule adopts an *E* configuration with respect to the alkene double bond. In the crystal structure, the cations are packed in an anti-parallel manner through π - π interactions between adjacent pyridinium and benzene rings along the *a* axis, with centroid-to-centroid distances of $3.615(2)$ and $3.630(2)$ Å. Water molecules bind the iodide ions through $\text{O}-\text{H}\cdots\text{I}$ hydrogen bonds into layers. These layers link with the cations through weak $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{I}$ interactions.

Related literature

For values of bond lengths, see Allen *et al.* (1987). For related structures, see, for example: Chantrapromma *et al.* (2005, 2006); Chantrapromma, Jindawong & Fun (2007); Chantrapromma, Jindawong, Fun & Patil (2007); Chantrapromma, Jindawong, Fun, Patil & Karalai (2007); Jindawong *et al.* (2005); Zhang *et al.* (2000). For background to nonlinear optics, see, for example: Oudar & Chemla (1977); Williams (1984).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{18}\text{NO}^+\cdot\text{I}^-\cdot\text{H}_2\text{O}$
 $M_r = 385.23$
 Triclinic, $P\bar{1}$
 $a = 6.9261(4)$ Å
 $b = 10.1857(6)$ Å
 $c = 11.6303(6)$ Å
 $\alpha = 100.829(2)^\circ$
 $\beta = 97.399(2)^\circ$
 $\gamma = 92.892(2)^\circ$
 $V = 796.81(8)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 2.01$ mm⁻¹
 $T = 100.0(1)$ K
 $0.36 \times 0.15 \times 0.12$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
 $T_{\min} = 0.528$, $T_{\max} = 0.797$
 18790 measured reflections
 4635 independent reflections
 4333 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.105$
 $S = 1.14$
 4635 reflections
 184 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 2.16$ e Å⁻³
 $\Delta\rho_{\min} = -0.83$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> -H \cdots <i>A</i>	<i>D</i> -H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> -H \cdots <i>A</i>
O1W-H1W1 \cdots I1	0.85	2.88	3.681(3)	158
O1W-H2W1 \cdots I1 ⁱ	0.85	2.88	3.690(3)	159
C16-H16B \cdots O1W ⁱⁱ	0.96	2.49	3.346(5)	148

 Symmetry codes: (i) $x + 1, y, z$; (ii) $x - 1, y, z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); 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: SJ2440).

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supporting information

Acta Cryst. (2008). E64, o145–o146 [https://doi.org/10.1107/S1600536807061375]

2-[(*E*)-2-(4-Ethoxyphenyl)ethenyl]-1-methylpyridinium iodide monohydrate**Chotika Laksana, Pumsak Ruanwas, Suchada Chantrapromma and Hoong-Kun Fun****S1. Comment**

The design of nonlinear optical (NLO) materials is of great interest due to the various applications of NLO materials. At molecular level, such compounds are likely to exhibit large values of molecular hyperpolarizability (β) and they have to have polarizable electrons (conjugated π system) spread over a large distance (Oudar & Chemla, 1977). We have been previously synthesized pyridinium and quinolinium derivatives to study their non-linear optical properties (Chantrapromma *et al.*, 2005, 2006; Chantrapromma, Jindawong & Fun, 2007; Chantrapromma, Jindawong, Fun & Patil, 2007; Chantrapromma, Jindawong, Fun, Patil & Karalai, 2007; Jindawong *et al.*, 2005). The single-crystal *x*-ray structural study of the title compound was undertaken in order to establish the structure and conformation of the various groups. However, the title compound crystallized in the centrosymmetric $P\bar{1}$ triclinic space group and therefore does not exhibit non-linear optical properties (Williams, 1984).

The asymmetric unit of the title compound consists of the pyridinium cation, iodide anion and one water molecule (Fig. 1). The water molecule forms an O1W—H1W1 \cdots I1 hydrogen bond to the iodide ion (Table 1). The cation is essentially planar and exist in *E* configuration with respect to the C6=C7 double bond [1.342 (5) Å]. The dihedral angle between the pyridinium and benzene rings is 3.13 (16)°. The ethenyl unit is also planar with respect to the two aromatic rings with the torsion angles C4—C5—C6—C7 = -1.1 (5)° and C6—C7—C8—C13 = 3.8 (6)°. The ethoxy substituent deviates only slightly from the benzene ring plane, with a C14—O1—C11—C10 torsion angle of 5.3 (5)°. Bond lengths and angles are in normal ranges (Allen *et al.*, 1987) and the bond lengths and angles of the cation are comparable with those for closely related structures (Chantrapromma, Jindawong & Fun, 2007; Chantrapromma, Jindawong, Fun & Patil, 2007; Zhang *et al.*, 2000).

In the crystal structure, the cations are packed in an anti-parallel fashion through $\pi\cdots\pi$ interactions along the *a* axis with $Cg_1\cdots Cg_2$ distances 3.615 (2) Å (symmetry code -*x*, 1 - *y*, 1 - *z*) and 3.630 (2) Å (symmetry code 1 - *x*, 1 - *y*, 1 - *z*) where Cg_1 is the centroid of the C1—C5/N1 pyridinium ring Cg_2 is the centroid of the C8—C13 benzene ring. Water molecules bind to iodide ions by O—H \cdots I hydrogen bonds forming layers. These layers are linked with the cations through weak C—H \cdots O and C—H \cdots I interactions, Table 1.

S2. Experimental

The title compound was synthesized by mixing a solution (1:1:1 molar ratio) of 1,2-dimethylpyridinium iodide (2.00 g, 8.51 mmol), 4-ethoxybenzaldehyde (1.28 g, 8.51 mmol) and piperidine (0.72 g, 8.51 mmol) in hot methanol (45 ml) and refluxing for 3 hrs under a nitrogen atmosphere. The solid which formed was filtered, washed with cold ethanol and dried. Yellow single crystals of the title compound suitable for *x*-ray structure determination were recrystallized from methanol by slow evaporation of the solvent at room temperature over several days (Mp. 481–483 K).

S3. Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with O—H = 0.85 ° and C—H distances in the range 0.93–0.97 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 electron density peak is located at 0.85 Å from I1 and the deepest hole is located at 0.63 Å from I1.

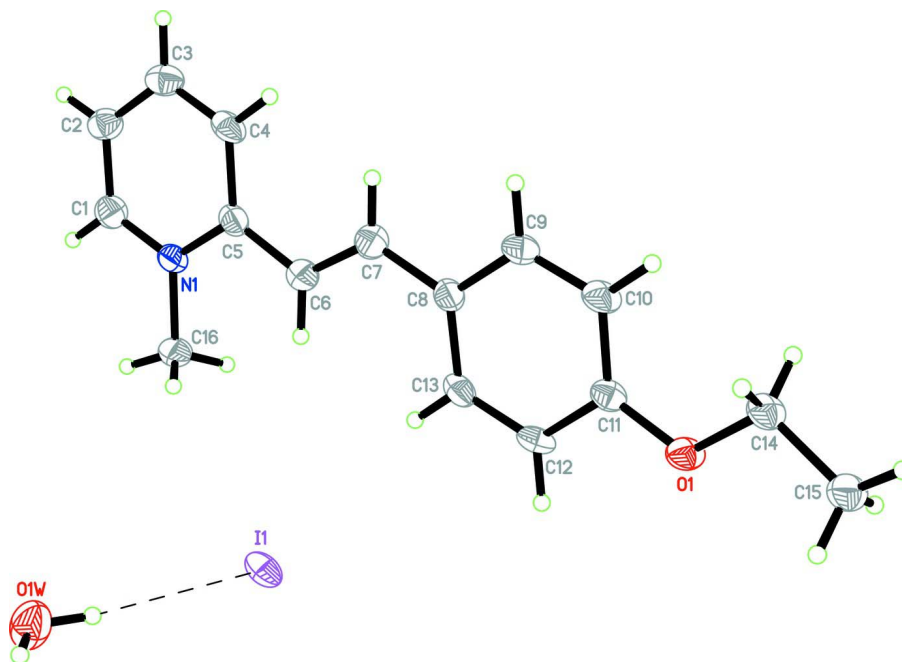


Figure 1

The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme. The O—H···I hydrogen bond is drawn as a dashed line.

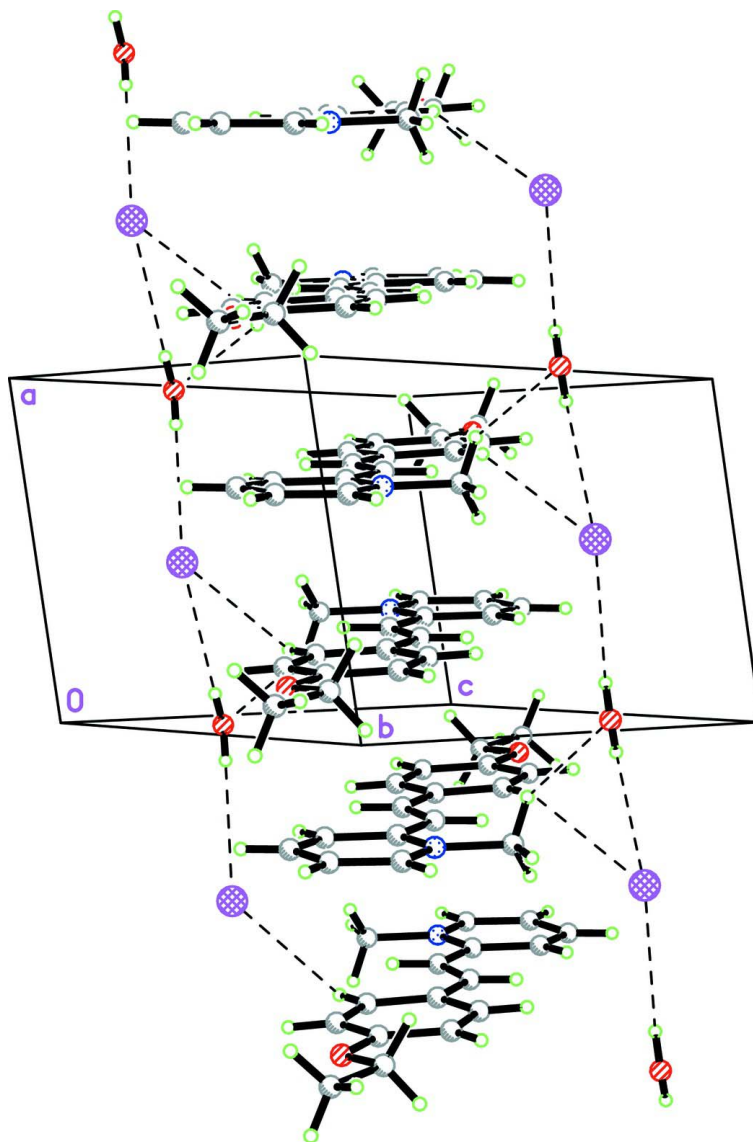


Figure 2

The crystal packing of (I). The O—H···I hydrogen bond and weak C—H···O and C—H···I interactions are drawn as dashed lines.

2-[(*E*)-2-(4-Ethoxyphenyl)ethenyl]-1-methylpyridinium iodide monohydrate

Crystal data

$C_{16}H_{18}NO^+ \cdot I^- \cdot H_2O$

$M_r = 385.23$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 6.9261(4) \text{ \AA}$

$b = 10.1857(6) \text{ \AA}$

$c = 11.6303(6) \text{ \AA}$

$\alpha = 100.829(2)^\circ$

$\beta = 97.399(2)^\circ$

$\gamma = 92.892(2)^\circ$

$V = 796.81(8) \text{ \AA}^3$

$Z = 2$

$F(000) = 384$

$D_x = 1.606 \text{ Mg m}^{-3}$

Melting point = 481–483 K

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4635 reflections

$\theta = 1.8\text{--}30.0^\circ$

$\mu = 2.01 \text{ mm}^{-1}$
 $T = 100 \text{ K}$

Block, yellow
 $0.36 \times 0.15 \times 0.12 \text{ mm}$

Data collection

Bruker APEXII CCD area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Detector resolution: 8.33 pixels mm^{-1}
 ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
 $T_{\min} = 0.528$, $T_{\max} = 0.797$

18790 measured reflections
 4635 independent reflections
 4333 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$
 $\theta_{\max} = 30.0^\circ$, $\theta_{\min} = 1.8^\circ$
 $h = -9 \rightarrow 9$
 $k = -14 \rightarrow 14$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.105$
 $S = 1.14$
 4635 reflections
 184 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
 $w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 1.6362P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 2.16 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.83 \text{ e } \text{\AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	0.46635 (3)	0.14640 (2)	0.24655 (2)	0.02735 (8)
O1	0.1292 (4)	0.6828 (2)	0.0835 (2)	0.0265 (5)
N1	0.3219 (4)	0.2513 (3)	0.6774 (2)	0.0246 (5)
C1	0.3453 (5)	0.2172 (4)	0.7851 (3)	0.0283 (7)
H1A	0.3605	0.1281	0.7900	0.034*
C2	0.3471 (5)	0.3101 (4)	0.8871 (3)	0.0287 (7)
H2A	0.3621	0.2850	0.9604	0.034*
C3	0.3261 (5)	0.4426 (4)	0.8781 (3)	0.0293 (7)
H3A	0.3244	0.5075	0.9458	0.035*
C4	0.3077 (5)	0.4781 (3)	0.7687 (3)	0.0270 (6)
H4A	0.2993	0.5677	0.7632	0.032*
C5	0.3015 (5)	0.3798 (3)	0.6650 (3)	0.0238 (6)

C6	0.2736 (5)	0.4093 (3)	0.5481 (3)	0.0258 (6)
H6A	0.2734	0.3395	0.4837	0.031*
C7	0.2478 (5)	0.5331 (3)	0.5277 (3)	0.0261 (6)
H7A	0.2481	0.6010	0.5935	0.031*
C8	0.2192 (5)	0.5716 (3)	0.4114 (3)	0.0240 (6)
C9	0.2019 (5)	0.7071 (3)	0.4050 (3)	0.0268 (6)
H9A	0.2116	0.7703	0.4749	0.032*
C10	0.1712 (5)	0.7494 (3)	0.2986 (3)	0.0269 (6)
H10A	0.1592	0.8396	0.2969	0.032*
C11	0.1585 (5)	0.6551 (3)	0.1938 (3)	0.0222 (6)
C12	0.1731 (5)	0.5193 (3)	0.1975 (3)	0.0243 (6)
H12A	0.1616	0.4560	0.1275	0.029*
C13	0.2042 (5)	0.4792 (3)	0.3044 (3)	0.0246 (6)
H13A	0.2156	0.3889	0.3057	0.029*
C14	0.1273 (5)	0.8225 (3)	0.0753 (3)	0.0269 (6)
H14A	0.2506	0.8705	0.1130	0.032*
H14B	0.0233	0.8631	0.1145	0.032*
C15	0.0951 (6)	0.8291 (4)	-0.0538 (3)	0.0322 (7)
H15A	0.1122	0.9207	-0.0624	0.048*
H15B	-0.0351	0.7933	-0.0875	0.048*
H15C	0.1875	0.7775	-0.0939	0.048*
C16	0.3131 (6)	0.1419 (3)	0.5723 (3)	0.0306 (7)
H16A	0.4084	0.1630	0.5241	0.046*
H16B	0.1852	0.1326	0.5273	0.046*
H16D	0.3401	0.0594	0.5975	0.046*
O1W	0.9790 (4)	0.0827 (4)	0.3301 (3)	0.0424 (7)
H1W1	0.8765	0.1147	0.3016	0.064*
H2W1	1.0745	0.1130	0.3010	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
II	0.02420 (12)	0.01775 (11)	0.03903 (14)	0.00238 (7)	0.00703 (8)	0.00088 (8)
O1	0.0389 (13)	0.0160 (10)	0.0252 (11)	-0.0005 (9)	0.0091 (9)	0.0027 (8)
N1	0.0246 (13)	0.0208 (12)	0.0261 (13)	0.0056 (10)	0.0014 (10)	-0.0005 (10)
C1	0.0253 (15)	0.0303 (17)	0.0294 (16)	0.0034 (13)	0.0053 (12)	0.0052 (13)
C2	0.0282 (16)	0.0319 (17)	0.0259 (15)	0.0016 (13)	0.0069 (12)	0.0037 (13)
C3	0.0275 (16)	0.0299 (17)	0.0289 (16)	-0.0004 (13)	0.0080 (13)	-0.0005 (13)
C4	0.0228 (15)	0.0164 (13)	0.0381 (17)	0.0012 (11)	0.0019 (12)	-0.0019 (12)
C5	0.0202 (13)	0.0215 (14)	0.0286 (15)	0.0041 (11)	0.0014 (11)	0.0025 (12)
C6	0.0291 (16)	0.0233 (15)	0.0249 (14)	0.0042 (12)	0.0036 (12)	0.0042 (12)
C7	0.0277 (15)	0.0236 (15)	0.0266 (15)	-0.0001 (12)	0.0021 (12)	0.0054 (12)
C8	0.0223 (14)	0.0207 (14)	0.0268 (15)	0.0037 (11)	-0.0014 (11)	0.0015 (11)
C9	0.0326 (17)	0.0175 (14)	0.0267 (15)	0.0008 (12)	0.0012 (12)	-0.0027 (11)
C10	0.0360 (17)	0.0137 (13)	0.0294 (15)	-0.0009 (12)	0.0062 (13)	0.0002 (11)
C11	0.0234 (14)	0.0175 (13)	0.0261 (14)	0.0002 (11)	0.0075 (11)	0.0028 (11)
C12	0.0280 (15)	0.0161 (13)	0.0274 (14)	0.0022 (11)	0.0092 (12)	-0.0030 (11)
C13	0.0245 (15)	0.0172 (13)	0.0311 (15)	0.0053 (11)	0.0030 (12)	0.0021 (11)

C14	0.0357 (17)	0.0156 (13)	0.0297 (15)	0.0001 (12)	0.0073 (13)	0.0037 (11)
C15	0.048 (2)	0.0227 (16)	0.0302 (16)	0.0060 (14)	0.0148 (15)	0.0080 (13)
C16	0.042 (2)	0.0209 (15)	0.0265 (15)	0.0068 (14)	0.0019 (14)	-0.0004 (12)
O1W	0.0321 (14)	0.060 (2)	0.0382 (15)	0.0036 (13)	0.0045 (11)	0.0178 (14)

Geometric parameters (Å, °)

O1—C11	1.357 (4)	C9—C10	1.381 (5)
O1—C14	1.445 (4)	C9—H9A	0.9300
N1—C1	1.353 (4)	C10—C11	1.393 (4)
N1—C5	1.357 (4)	C10—H10A	0.9300
N1—C16	1.483 (4)	C11—C12	1.400 (4)
C1—C2	1.370 (5)	C12—C13	1.374 (5)
C1—H1A	0.9300	C12—H12A	0.9300
C2—C3	1.387 (5)	C13—H13A	0.9300
C2—H2A	0.9300	C14—C15	1.504 (5)
C3—C4	1.380 (5)	C14—H14A	0.9700
C3—H3A	0.9300	C14—H14B	0.9700
C4—C5	1.410 (5)	C15—H15A	0.9600
C4—H4A	0.9300	C15—H15B	0.9600
C5—C6	1.438 (5)	C15—H15C	0.9600
C6—C7	1.342 (5)	C16—H16A	0.9600
C6—H6A	0.9300	C16—H16B	0.9600
C7—C8	1.470 (5)	C16—H16D	0.9600
C7—H7A	0.9300	O1W—H1W1	0.8501
C8—C13	1.401 (5)	O1W—H2W1	0.8500
C8—C9	1.406 (4)		
C11—O1—C14	116.9 (3)	C9—C10—C11	119.0 (3)
C1—N1—C5	121.6 (3)	C9—C10—H10A	120.5
C1—N1—C16	117.5 (3)	C11—C10—H10A	120.5
C5—N1—C16	120.8 (3)	O1—C11—C10	125.1 (3)
N1—C1—C2	121.9 (3)	O1—C11—C12	114.9 (3)
N1—C1—H1A	119.1	C10—C11—C12	120.0 (3)
C2—C1—H1A	119.1	C13—C12—C11	120.1 (3)
C1—C2—C3	118.2 (3)	C13—C12—H12A	119.9
C1—C2—H2A	120.9	C11—C12—H12A	119.9
C3—C2—H2A	120.9	C12—C13—C8	121.4 (3)
C4—C3—C2	119.9 (3)	C12—C13—H13A	119.3
C4—C3—H3A	120.0	C8—C13—H13A	119.3
C2—C3—H3A	120.0	O1—C14—C15	107.6 (3)
C3—C4—C5	120.5 (3)	O1—C14—H14A	110.2
C3—C4—H4A	119.7	C15—C14—H14A	110.2
C5—C4—H4A	119.7	O1—C14—H14B	110.2
N1—C5—C4	117.7 (3)	C15—C14—H14B	110.2
N1—C5—C6	119.0 (3)	H14A—C14—H14B	108.5
C4—C5—C6	123.3 (3)	C14—C15—H15A	109.5
C7—C6—C5	122.9 (3)	C14—C15—H15B	109.5

C7—C6—H6A	118.5	H15A—C15—H15B	109.5
C5—C6—H6A	118.5	C14—C15—H15C	109.5
C6—C7—C8	126.3 (3)	H15A—C15—H15C	109.5
C6—C7—H7A	116.9	H15B—C15—H15C	109.5
C8—C7—H7A	116.9	N1—C16—H16A	109.5
C13—C8—C9	117.3 (3)	N1—C16—H16B	109.5
C13—C8—C7	123.3 (3)	H16A—C16—H16B	109.5
C9—C8—C7	119.4 (3)	N1—C16—H16D	109.5
C10—C9—C8	122.3 (3)	H16A—C16—H16D	109.5
C10—C9—H9A	118.9	H16B—C16—H16D	109.5
C8—C9—H9A	118.9	H1W1—O1W—H2W1	107.7
C5—N1—C1—C2	0.8 (5)	C6—C7—C8—C9	-177.4 (4)
C16—N1—C1—C2	-177.3 (3)	C13—C8—C9—C10	0.1 (5)
N1—C1—C2—C3	-0.6 (5)	C7—C8—C9—C10	-178.8 (3)
C1—C2—C3—C4	-1.2 (5)	C8—C9—C10—C11	-0.6 (5)
C2—C3—C4—C5	2.8 (5)	C14—O1—C11—C10	5.3 (5)
C1—N1—C5—C4	0.8 (5)	C14—O1—C11—C12	-175.7 (3)
C16—N1—C5—C4	178.8 (3)	C9—C10—C11—O1	-179.8 (3)
C1—N1—C5—C6	-178.9 (3)	C9—C10—C11—C12	1.2 (5)
C16—N1—C5—C6	-0.8 (5)	O1—C11—C12—C13	179.5 (3)
C3—C4—C5—N1	-2.5 (5)	C10—C11—C12—C13	-1.5 (5)
C3—C4—C5—C6	177.1 (3)	C11—C12—C13—C8	1.0 (5)
N1—C5—C6—C7	178.5 (3)	C9—C8—C13—C12	-0.3 (5)
C4—C5—C6—C7	-1.1 (5)	C7—C8—C13—C12	178.5 (3)
C5—C6—C7—C8	179.8 (3)	C11—O1—C14—C15	179.4 (3)
C6—C7—C8—C13	3.8 (6)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1 <i>W</i> —H1 <i>W</i> 1...I1	0.85	2.88	3.681 (3)	158
O1 <i>W</i> —H2 <i>W</i> 1...I1 ⁱ	0.85	2.88	3.690 (3)	159
C16—H16 <i>B</i> ...O1 <i>W</i> ⁱⁱ	0.96	2.49	3.346 (5)	148

Symmetry codes: (i) $x+1, y, z$; (ii) $x-1, y, z$.