IUCrData

ISSN 2414-3146

4-{(1*E*,3*E*)-4-[4-(Dimethylamino)phenyl]buta-1,3-dien-1-yl}-1-methylpyridin-1-ium iodide

S. John Sundaram,[a] P. Sagayaraj,[a] A. Antony Raj,[a] E. R. A. Dravida Thendral[b] and G. Usha[b]*

[a]Department of Physics, Loyola College, Chennai-34, Tamilnadu, India, and [b]PG and Research Department of Physics, Queen Mary's College, Chennai-4, Tamilnadu, India. *Correspondence e-mail: guqmc@yahoo.com

Received 7 September 2016
Accepted 15 September 2016

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

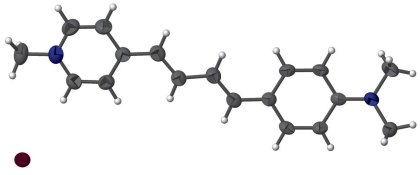
Keywords: crystal structure; pyridinium; iodide; C–H···I hydrogen bonds; C–H···π interactions.

CCDC reference: 1487591

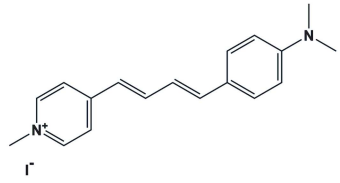
Structural data: full structural data are available from iucrdata.iucr.org

The title molecular salt, $C_{18}H_{21}N_2^+\cdot I^-$, consists of a pyridinium cation and an I^- anion. The cation exists in an *E,E* conformation with respect to the two C=C double bonds, and is roughly planar with the pyridinium ring being inclined to the benzene ring by 10.8 (2) °. In the crystal, the ions are linked by a C—H···I hydrogen bond, and the cations are linked by C—H···π interactions, forming zigzag chains propagating along [010].

3D view



Chemical scheme



Structure description

Pyridinium derivatives have long been observed to exhibit antiseptic properties (Browning *et al.*, 1923). Pyridinium chromophore compounds are particularly important because of their activity against methicillin-resistant *Staphylococcus aureus* (MRSA),-which is a drug-resistant bacterium (Wainwright & Kristiansen, 2003; Chanawanno *et al.*, 2010). Pyridinium halide salts possess promising antimicrobial properties due to the reactive functional groups covalently bonded to the long hydrophobic chain (Fisicaro *et al.*, 1990; Chanawanno *et al.*, 2010). Anions in pyridinium derivatives have been proven to control their antimicrobial activity and different anion kinds can exhibit different anti-microbial activities (Pernak *et al.*, 2001). The crystal structures of some closely related pyridinium iodide salts have been reported, *viz*. (*E*)-2-[4-(dimethylamino)styryl]-1-methylpyridinium triiodide (Fun *et al.*, 2011), 2-[(*E*)-4-(diethylamino)styryl]-1-methyl-pyridinium iodide (Kaewmanee *et al.*, 2010), (*E*)-1-methyl-2-styrylpyridinium iodide (Fun *et al.*, 2009) and 2-[(*E*)-2-(4-chlorophenyl)ethenyl]-1-methylpyridinium iodide monohydrate (Chanawanno *et al.* 2008).

The asymmetric unit of the title molecular salt, Fig. 1, comprises a pyridinium cation and an I^- anion. The bond lengths and angles for the cation are comparable with those of the closely related structures mentioned above. The cation exists in an *E,E* conformation

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

Cg is the centroid of the C10–C15 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···I1	0.93	2.99	3.855 (5)	154
C12—H12···*Cg*^{i}	0.93	2.95	3.577 (5)	126

Symmetry code: (i) $-x-1, y-\frac{1}{2}, -z+\frac{3}{2}$.

with respect to the two C═C double bonds, C6═C7 and C8═C9. It is roughly planar with the pyridinium ring (N1/C1–C5) being inclined to the benzene ring (C10–C15) by 10.8 (2)°.

In the crystal, the ions are linked by a C—H···I hydrogen bond, and the cations are linked by C—H···π interactions, forming zigzag chains propagating along the *b*-axis direction (Table 1 and Fig. 2).

Synthesis and crystallization

The title molecular salt was synthesized by the Knoevenagel condensation of 1,4-dimethyl pyridinium iodide (2.35 g, 10 mmol) in methanol (30 ml) and 4-*N*,*N*-dimethylamino cinnamaldehyde (1.75 g, 10 mmol) in the presence of piperidine (0.2 ml). The total mixture was taken in a round-bottom flask (1000 ml capacity) of a Dean–Stark apparatus. The mixture was refluxed for 12 h and then cooled to room temperature. The product was filtered and recrystallized three times from methanol solution yielding black block-like crystals (m.p. 539 K).

Refinement

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

Acknowledgements

The authors thank DST–SERB, India for funding, the Central Instrumentation Facility, Queen Mary's College, Chennai-4 for computing facilities and V. Ramkumar, Department of Chemistry, IIT, Madras, for the X-ray data collection facility.

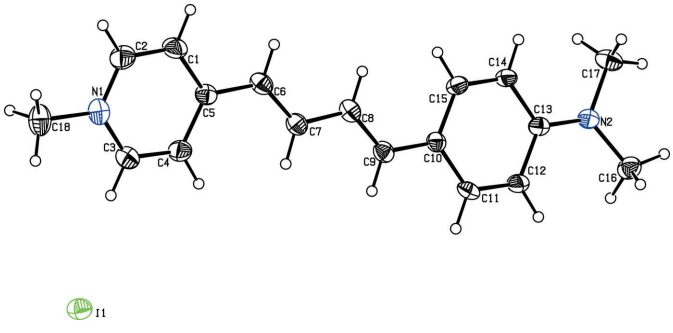


Figure 1
The molecular structure of the title molecular salt, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{21}N_2^+\cdot I^-$
M_r	392.27
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, *b*, *c* (Å)	6.6659 (4), 7.4965 (5), 34.7364 (17)
β (°)	94.7132 (19)
V (Å^3)	1729.94 (18)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	1.85
Crystal size (mm)	0.25 × 0.22 × 0.18
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.636, 0.732
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12906, 3028, 2887
R_{int}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.041, 0.080, 1.33
No. of reflections	3022
No. of parameters	193
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.35, −1.04

Computer programs: *APEX2* (Bruker, 2004), *SAINT* (Bruker, 2004), *SHELXS97* (Sheldrick 2008), *SHELXL-2014/7* (Sheldrick, 2015), *SHELXTL* (Sheldrick 2008) and *Mercury* (Macrae *et al.*, 2008), *SHELXTL* (Sheldrick 2008).

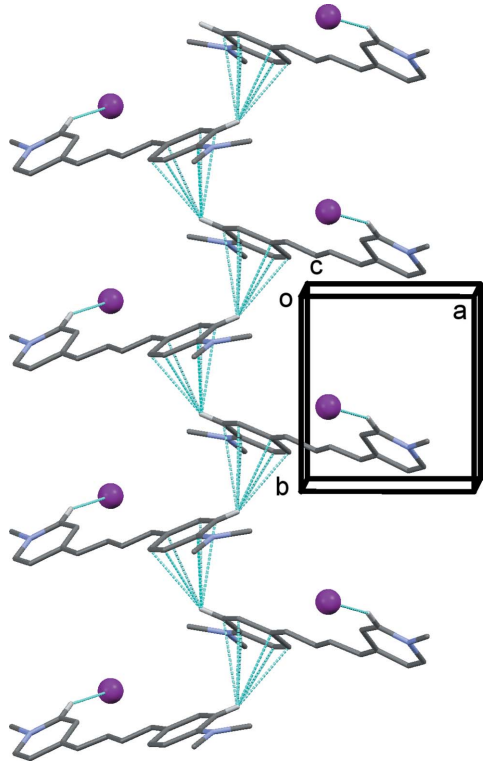


Figure 2
A partial view along the *c* axis of the crystal packing of the title molecular salt. The C—H···I hydrogen bond and the C—H···π interactions are shown as dashed lines (see Table 1), and for clarity only H atoms H3 and H12 have been included.

References

Browning, C. H., Cohen, J. B., Ellingworth, S. & Gulbransen, R. (1923). *BMJ*, **2**, 326.

Bruker (2004). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chanawanno, K., Chantrapromma, S., Anantapong, T., Kanjana-Opas, A. & Fun, H. K. (2010). *Eur. J. Med. Chem.* **45**, 4199–4208.

Chanawanno, K., Chantrapromma, S. & Fun, H.-K. (2008). *Acta Cryst.* E**64**, o1882–o1883.

Fisicaro, E., Pelizzetti, E., Barbieri, M., Savarino, P. & Viscardi, G. (1990). *Thermochim. Acta*, **168**, 143–159.

Fun, H.-K., Chanawanno, K. & Chantrapromma, S. (2009). *Acta Cryst.* E**65**, o1934–o1935.

Fun, H.-K., Chanawanno, K. & Chantrapromma, S. (2011). *Acta Cryst.* E**67**, o2151.

Kaewmanee, N., Chanawanno, K., Chantrapromma, S. & Fun, H.-K. (2010). *Acta Cryst.* E**66**, o2639–o2640.

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.

Pernak, J., Kalewska, J., Ksycińska, H. & Cybulski, J. (2001). *Eur. J. Med. Chem.* **36**, 899–907.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Wainwright, M. & Kristiansen, J. E. (2003). *Int. J. Antimicrob. Agents*, **22**, 479–486.

full crystallographic data

IUCrData (2016). **1**, x161463 [doi:10.1107/S2414314616014632]

4-{(1*E*,3*E*)-4-[4-(Dimethylamino)phenyl]buta-1,3-dien-1-yl}-1-methylpyridin-1-ium iodide

S. John Sundaram, P. Sagayaraj, A. Antony Raj, E. R. A. Dravida Thendral and G. Usha

4-{(1*E*,3*E*)-4-[4-(Dimethylamino)phenyl]buta-1,3-dien-1-yl}-1-methylpyridin-1-ium iodide

Crystal data

$C_{18}H_{21}N_2^+\cdot I^-$
M_r = 392.27
Monoclinic, $P2_1/c$
a = 6.6659 (4) Å
b = 7.4965 (5) Å
c = 34.7364 (17) Å
β = 94.7132 (19)°
V = 1729.94 (18) Å^3
Z = 4
$F(000)$ = 784
D_x = 1.506 Mg m^{-3}
Melting point: 539 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7371 reflections
θ = 2.8–26.8°
μ = 1.85 mm^{-1}
T = 296 K
Block, black
0.25 × 0.22 × 0.18 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.636, T_{max} = 0.732
12906 measured reflections
3028 independent reflections
2887 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 25.0°, θ_{min} = 1.2°
h = −7→7
k = −7→8
l = −41→38

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.080
S = 1.33
3022 reflections
193 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0122P)^2 + 3.3223P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −1.04 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}
C1	0.6000 (7)	0.9316 (6)	0.58510 (14)	0.0499 (12)
H1	0.6626	1.0027	0.6045	0.060*
C2	0.6891 (7)	0.9085 (7)	0.55189 (15)	0.0531 (12)
H2	0.8120	0.9635	0.5490	0.064*
C3	0.4274 (7)	0.7278 (6)	0.52763 (13)	0.0458 (11)
H3	0.3689	0.6573	0.5077	0.055*
C4	0.3317 (7)	0.7466 (6)	0.56030 (13)	0.0445 (11)
H4	0.2091	0.6897	0.5624	0.053*
C5	0.4162 (6)	0.8511 (5)	0.59099 (12)	0.0374 (10)
C6	0.3244 (7)	0.8776 (6)	0.62645 (13)	0.0435 (11)
H6	0.3955	0.9468	0.6451	0.052*
C7	0.1463 (7)	0.8128 (6)	0.63545 (13)	0.0402 (10)
H7	0.0702	0.7484	0.6166	0.048*
C8	0.0656 (7)	0.8357 (6)	0.67190 (13)	0.0427 (11)
H8	0.1385	0.9038	0.6906	0.051*
C9	−0.1093 (6)	0.7647 (6)	0.68079 (12)	0.0392 (10)
H9	−0.1829	0.7036	0.6611	0.047*
C10	−0.1968 (6)	0.7721 (5)	0.71744 (12)	0.0346 (9)
C11	−0.3882 (7)	0.6997 (6)	0.72128 (12)	0.0395 (10)
H11	−0.4579	0.6484	0.6998	0.047*
C12	−0.4762 (6)	0.7016 (6)	0.75548 (12)	0.0394 (10)
H12	−0.6042	0.6535	0.7565	0.047*
C13	−0.3763 (6)	0.7755 (5)	0.78926 (12)	0.0327 (9)
C14	−0.1835 (6)	0.8477 (6)	0.78520 (13)	0.0393 (10)
H14	−0.1121	0.8973	0.8067	0.047*
C15	−0.0986 (6)	0.8470 (6)	0.75079 (13)	0.0404 (10)
H15	0.0280	0.8977	0.7494	0.048*
C16	−0.6777 (6)	0.7474 (7)	0.82429 (13)	0.0485 (12)
H16A	−0.7505	0.8254	0.8062	0.073*
H16B	−0.7169	0.7704	0.8498	0.073*
H16C	−0.7076	0.6257	0.8174	0.073*
C17	−0.3576 (8)	0.8521 (7)	0.85801 (13)	0.0516 (13)
H17A	−0.2294	0.7944	0.8627	0.077*
H17B	−0.4360	0.8327	0.8796	0.077*
H17C	−0.3378	0.9778	0.8546	0.077*
C18	0.7111 (8)	0.7743 (8)	0.48821 (15)	0.0601 (14)
H18A	0.8084	0.6812	0.4934	0.090*
H18B	0.7783	0.8812	0.4811	0.090*
H18C	0.6160	0.7384	0.4674	0.090*
I1	0.14120 (5)	0.59463 (4)	0.43097 (2)	0.04734 (12)
N1	0.6043 (6)	0.8081 (5)	0.52308 (11)	0.0443 (9)
N2	−0.4628 (5)	0.7789 (5)	0.82341 (10)	0.0389 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.053 (3)	0.042 (3)	0.054 (3)	−0.015 (2)	0.005 (2)	−0.009 (2)
C2	0.050 (3)	0.045 (3)	0.066 (3)	−0.014 (2)	0.012 (2)	0.002 (3)
C3	0.053 (3)	0.045 (3)	0.040 (2)	−0.003 (2)	0.002 (2)	−0.002 (2)
C4	0.043 (3)	0.040 (3)	0.051 (3)	−0.008 (2)	0.004 (2)	−0.001 (2)
C5	0.042 (2)	0.028 (2)	0.042 (2)	−0.0023 (18)	0.0025 (19)	0.0016 (18)
C6	0.054 (3)	0.034 (3)	0.042 (2)	−0.005 (2)	0.004 (2)	−0.004 (2)
C7	0.047 (3)	0.033 (2)	0.041 (2)	0.000 (2)	0.004 (2)	−0.002 (2)
C8	0.050 (3)	0.037 (2)	0.041 (2)	0.001 (2)	0.005 (2)	−0.004 (2)
C9	0.044 (3)	0.032 (2)	0.040 (2)	0.0036 (19)	0.0011 (19)	−0.0025 (19)
C10	0.036 (2)	0.028 (2)	0.039 (2)	0.0037 (18)	0.0005 (18)	−0.0025 (18)
C11	0.047 (3)	0.032 (2)	0.038 (2)	−0.006 (2)	−0.0032 (19)	−0.0099 (19)
C12	0.036 (2)	0.038 (2)	0.043 (2)	−0.009 (2)	0.0010 (19)	−0.004 (2)
C13	0.033 (2)	0.025 (2)	0.038 (2)	0.0037 (17)	−0.0066 (17)	0.0030 (18)
C14	0.032 (2)	0.043 (3)	0.041 (2)	−0.0004 (19)	−0.0071 (18)	−0.007 (2)
C15	0.030 (2)	0.044 (3)	0.046 (3)	−0.0022 (18)	−0.0033 (19)	−0.003 (2)
C16	0.041 (3)	0.063 (3)	0.041 (3)	−0.006 (2)	0.005 (2)	0.003 (2)
C17	0.061 (3)	0.054 (3)	0.038 (3)	−0.010 (2)	−0.007 (2)	−0.002 (2)
C18	0.070 (4)	0.058 (3)	0.055 (3)	0.007 (3)	0.028 (3)	0.010 (3)
I1	0.04592 (18)	0.0529 (2)	0.04281 (18)	−0.01053 (15)	0.00116 (12)	0.00207 (16)
N1	0.054 (2)	0.036 (2)	0.044 (2)	0.0072 (18)	0.0102 (18)	0.0074 (18)
N2	0.0374 (19)	0.044 (2)	0.0345 (19)	−0.0011 (17)	−0.0022 (15)	−0.0032 (17)

Geometric parameters (Å, º)

C1—C2	1.351 (7)	C11—C12	1.367 (6)
C1—C5	1.396 (6)	C11—H11	0.9300
C1—H1	0.9300	C12—C13	1.414 (6)
C2—N1	1.339 (6)	C12—H12	0.9300
C2—H2	0.9300	C13—N2	1.361 (5)
C3—N1	1.345 (6)	C13—C14	1.412 (6)
C3—C4	1.354 (6)	C14—C15	1.364 (6)
C3—H3	0.9300	C14—H14	0.9300
C4—C5	1.403 (6)	C15—H15	0.9300
C4—H4	0.9300	C16—N2	1.455 (5)
C5—C6	1.433 (6)	C16—H16A	0.9600
C6—C7	1.344 (6)	C16—H16B	0.9600
C6—H6	0.9300	C16—H16C	0.9600
C7—C8	1.426 (6)	C17—N2	1.449 (5)
C7—H7	0.9300	C17—H17A	0.9600
C8—C9	1.341 (6)	C17—H17B	0.9600
C8—H8	0.9300	C17—H17C	0.9600
C9—C10	1.444 (6)	C18—N1	1.476 (6)
C9—H9	0.9300	C18—H18A	0.9600
C10—C15	1.401 (6)	C18—H18B	0.9600
C10—C11	1.403 (6)	C18—H18C	0.9600

C2—C1—C5	121.5 (4)	C13—C12—H12	119.4
C2—C1—H1	119.3	N2—C13—C14	122.3 (4)
C5—C1—H1	119.3	N2—C13—C12	121.8 (4)
N1—C2—C1	121.5 (4)	C14—C13—C12	115.9 (4)
N1—C2—H2	119.3	C15—C14—C13	122.2 (4)
C1—C2—H2	119.3	C15—C14—H14	118.9
N1—C3—C4	122.0 (4)	C13—C14—H14	118.9
N1—C3—H3	119.0	C14—C15—C10	121.8 (4)
C4—C3—H3	119.0	C14—C15—H15	119.1
C3—C4—C5	120.5 (4)	C10—C15—H15	119.1
C3—C4—H4	119.7	N2—C16—H16A	109.5
C5—C4—H4	119.7	N2—C16—H16B	109.5
C1—C5—C4	115.6 (4)	H16A—C16—H16B	109.5
C1—C5—C6	120.5 (4)	N2—C16—H16C	109.5
C4—C5—C6	123.9 (4)	H16A—C16—H16C	109.5
C7—C6—C5	126.7 (4)	H16B—C16—H16C	109.5
C7—C6—H6	116.7	N2—C17—H17A	109.5
C5—C6—H6	116.7	N2—C17—H17B	109.5
C6—C7—C8	124.6 (4)	H17A—C17—H17B	109.5
C6—C7—H7	117.7	N2—C17—H17C	109.5
C8—C7—H7	117.7	H17A—C17—H17C	109.5
C9—C8—C7	123.7 (4)	H17B—C17—H17C	109.5
C9—C8—H8	118.1	N1—C18—H18A	109.5
C7—C8—H8	118.1	N1—C18—H18B	109.5
C8—C9—C10	127.6 (4)	H18A—C18—H18B	109.5
C8—C9—H9	116.2	N1—C18—H18C	109.5
C10—C9—H9	116.2	H18A—C18—H18C	109.5
C15—C10—C11	116.3 (4)	H18B—C18—H18C	109.5
C15—C10—C9	123.3 (4)	C2—N1—C3	118.9 (4)
C11—C10—C9	120.3 (4)	C2—N1—C18	120.5 (4)
C12—C11—C10	122.5 (4)	C3—N1—C18	120.4 (4)
C12—C11—H11	118.8	C13—N2—C17	121.3 (4)
C10—C11—H11	118.8	C13—N2—C16	120.2 (3)
C11—C12—C13	121.3 (4)	C17—N2—C16	117.0 (4)
C11—C12—H12	119.4		

C5—C1—C2—N1	0.4 (8)	C11—C12—C13—N2	179.7 (4)
N1—C3—C4—C5	−0.4 (7)	C11—C12—C13—C14	0.7 (6)
C2—C1—C5—C4	−0.2 (7)	N2—C13—C14—C15	−178.8 (4)
C2—C1—C5—C6	−180.0 (5)	C12—C13—C14—C15	0.2 (6)
C3—C4—C5—C1	0.2 (7)	C13—C14—C15—C10	−0.9 (7)
C3—C4—C5—C6	179.9 (4)	C11—C10—C15—C14	0.7 (6)
C1—C5—C6—C7	178.0 (5)	C9—C10—C15—C14	−178.3 (4)
C4—C5—C6—C7	−1.8 (7)	C1—C2—N1—C3	−0.5 (7)
C5—C6—C7—C8	176.8 (4)	C1—C2—N1—C18	−175.6 (5)
C6—C7—C8—C9	−177.6 (5)	C4—C3—N1—C2	0.5 (7)
C7—C8—C9—C10	176.1 (4)	C4—C3—N1—C18	175.6 (5)

C8—C9—C10—C15	−5.0 (7)	C14—C13—N2—C17	−2.1 (6)
C8—C9—C10—C11	176.1 (4)	C12—C13—N2—C17	178.9 (4)
C15—C10—C11—C12	0.2 (6)	C14—C13—N2—C16	163.7 (4)
C9—C10—C11—C12	179.2 (4)	C12—C13—N2—C16	−15.2 (6)
C10—C11—C12—C13	−0.9 (7)		

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C10–C15 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···I1	0.93	2.99	3.855 (5)	154
C12—H12···*Cg*^{i}	0.93	2.95	3.577 (5)	126

Symmetry code: (i) −*x*−1, *y*−1/2, −*z*+3/2.