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Crystal structures of three new *N*-halomethylated quaternary ammonium salts

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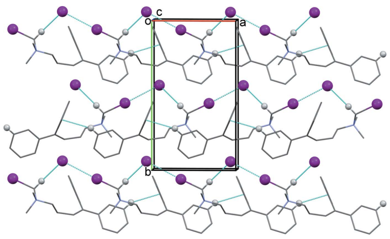
In the crystals of the title *N*-halomethylated quaternary ammonium salts, $C_{19}H_{23}IN^{+}\cdot I^{-}$, (I) [systematic name: *N*-(4,4-diphenylbut-3-en-1-yl)-*N*-iodomethyl-*N*,*N*-dimethylammonium iodide], $C_{20}H_{25}IN^{+}\cdot I^{-}$, (II) [systematic name: *N*-(5,5-diphenylpent-4-en-1-yl)-*N*-iodomethyl-*N*,*N*-dimethylammonium iodide], and $C_{21}H_{27}IN^{+}\cdot I^{-}$, (III) [systematic name: *N*-(6,6-diphenylhex-5-en-1-yl)-*N*-iodomethyl-*N*,*N*-dimethylammonium iodide], there are short $I\cdots I^{-}$ interactions of 3.564 (4), 3.506 (1) and 3.557 (1) Å for compounds (I), (II) and (III), respectively. Compound (I) crystallizes in the Sohncke group $P2_1$ as an 'enantiopure' compound and is therefore a potential material for NLO properties. In the crystal of compound (I), molecules are linked by $C—H\cdots I^{-}$ and $C—H\cdots\pi$ interactions which, together with the $I\cdots I^{-}$ interactions, lead to the formation of ribbons along [100]. In (II), there are only $C—H\cdots I^{-}$ interactions which, together with the $I\cdots I^{-}$ interactions, lead to the formation of helices along [010]. In (III), apart from the $I\cdots I^{-}$ interactions, there are no significant intermolecular interactions present.



1. Chemical context and background to halogen bonding and cation–π interactions

Quaternary ammonium salts have been widely studied as anti-cancer (Wang *et al.*, 2012; Song *et al.*, 2013), anti-fungal (Ng *et al.*, 2006), anti-HIV-1 (Shiraishi *et al.*, 2000), anti-bacterial (Calvani *et al.*, 1998), anti-malarial (Calas *et al.*, 1997; Calas *et al.*, 2000) and anti-leishmanial (Mavromoustakos *et al.*, 2001) pharmaceuticals. Our research group has been working in the past few years on the activity of quaternary *N*-halomethyl ammonium salts for likely pharmaceutical purposes, specifically against axenic *L. (V) panamensis* and *L. (L) amazonensis* parasites, human pathogenic species that cause cutaneous and mucocutaneous leishmaniasis. The experiments proved that these compounds are very promising anti-leishmanial molecules, and very significant changes in their activity were observed upon a slight modification of the carbon skeleton by only a single methylene unit (Ríos-Vásquez *et al.*, 2015). A preliminary effort at understanding a structure–activity relationship with three *N*-iodomethyl quaternary ammonium salts (I), (II) and (III) of the form $[ICH_2N(CH_3)_3(CH_2)_nCH{=}C(Ph)_2]^{+}\cdot I^{-}$ (with n = 2, 3 and 4, respectively) is currently being carried out. One possible approach to understand the different activities is to establish what kind of interactions are present in compounds (I)–(III), for example whether $C—I\cdots I^{-}$ (Desiraju *et al.*, 2013), $C—H\cdots I^{-}$ (Glidewell *et al.*, 1994), $C—H\cdots\pi$ (Nishio *et al.*, 1998) or cation–π (Dougherty, 1996), and if so, how these interactions may affect their structure and biological properties.

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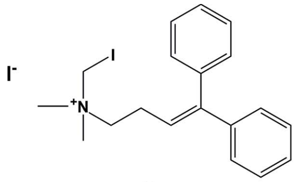
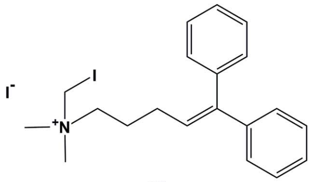
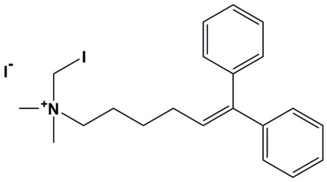
As defined by International Union for Pure and Applied Chemistry (IUPAC): *a halogen bond occurs when there is evidence of a net attractive interaction between an electrophilic region associated with a halogen atom in a molecular entity and a nucleophilic region in another, or the same, molecular entity* (Desiraju *et al.*, 2013). Halogen bonds are characterized by $X \cdots X$ distances that are clearly shorter than the van der Waals radii sum (Formigué, 2009; Awwadi *et al.*; 2006); otherwise this interaction is neglected. In a similar way, the existence of C—H$\cdots X$ hydrogen bonds (X = F, Cl, Br or I) in neutral organic molecules (Aakeröy & Seddon, 1993) and even in organic salts has been recognized. On the other hand, a special kind of hydrogen bond, defined as a weak interaction between a soft acid (*i.e.* an sp^3, sp^2 or sp C—H system) and a soft base (*i.e.* an aromatic, olefinic or acetilenic *p* system), with a significant role on diverse chemical and biological phenomena has recently been described (Nishio, 2012). In particular, this interaction exerts an observable influence on host–guest recognition and crystal packing in the solid state. A related attraction is the cation–π interaction, which is regarded as an electrostatic attraction between a positive charge and the quadrupole moment of an aromatic ring (Dougherty, 1996). A cation–π interaction between aromatic and ammonium ions is known to play an important role in many biological systems (Ma & Dougherty, 1997; Dougherty, 2013; Sussman *et al.*, 1991; Chen *et al.*, 2011). Part of our research interest is focused not only in understanding the reactive nature of alpha ammonium distonic radical cations which are generated from *N*-halomethylated quaternary ammonium salts (Ríos *et al.*, 1996; Ríos, Bartberger *et al.*, 1997), but also in trying to understand how these salts behave against *Leishmania* parasites (Ríos-Vásquez *et al.*, 2015). The recognition of the occurrence of some supramolecular interactions in these salts may lead to a better understanding of the likely novel biological binding sites, and therefore to new suggestions about biocatalytic mechanisms.



(I)



(II)



(III)

The title *N*-iodomethyl quaternary ammonium salts, (I)–(III), were synthesized following standard procedures used for other related compounds (Newcomb *et al.*, 1993; Horner *et al.*, 1995) and suitable crystals were obtained (Múnera-Orozco, 2014). This paper reports a comparative crystal structure and supramolecular interactions analysis for the aforementioned compounds.

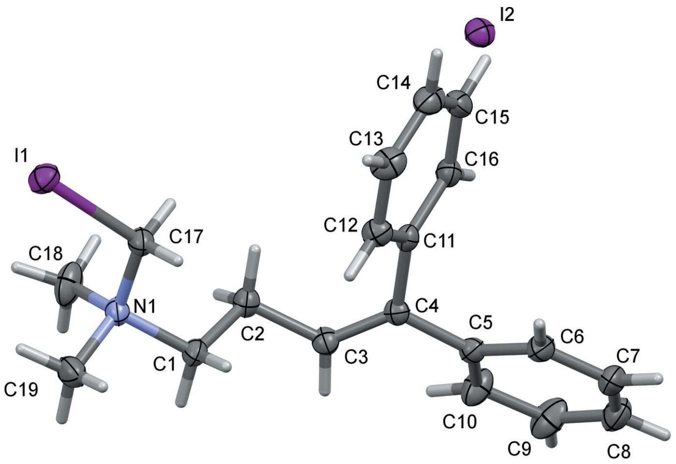


Figure 1
The molecular structure of compound (I), showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

2. Structural commentary

Compound (I), Fig. 1, crystallizes in the non-centrosymmetric monoclinic space group $P2_1$ and is therefore, a potential material for NLO properties. The asymmetric unit consists of an ammonium cation and an iodide anion. In the geminal-substituted diphenylethene unit, the phenyl rings (C5–C10 and C11–C16) are inclined to one another by 74.6 (2)°, and are twisted from the mean plane of the central C═C bond fragment (C2–C5/C11) by 33.2 (2) and 61.4 (2)°, respectively. Coplanarity of the olefin skeleton and the peripheral phenyl rings is prevented because of steric congestion between the associated phenyl rings. The conformation of the side chain reveals an *all-trans* extended conformation with the iodomethyl moiety on one side of the backbone chain, with bond lengths and angles in the expected ranges.

In compound (II), Fig. 2, the dihedral angles between the mean planes of the C═C double-bond fragment (C3–C6/C12) and the two phenyl rings (C6–C11 and C12–C17) are 31.1 (4)

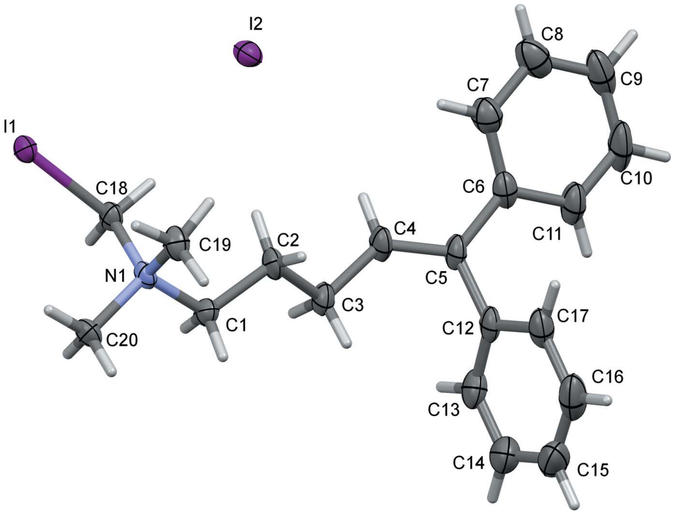


Figure 2
The molecular structure of compound (II), showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

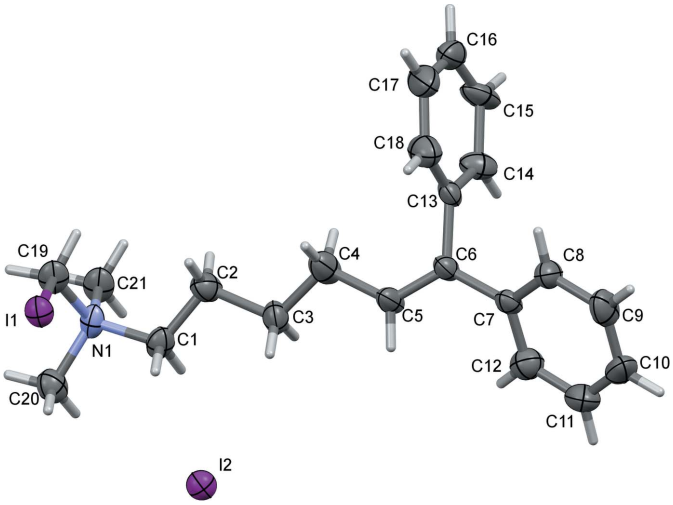


Figure 3
The molecular structure of compound (III), showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

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

Cg is the centroid of the C11–C16 ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C17—H17$B\cdots$I2^{i}	0.97	3.00	3.919 (5)	159
C7—H7$\cdots Cg^{ii}$	0.93	2.84	3.030 (5)	143

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

Table 2
Hydrogen-bond geometry (Å, °) for (II).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C2—H2$B\cdots$I2	0.97	3.06	4.001 (7)	165

and 58.6 (4)°, respectively, while the phenyl rings are inclined to one another by 76.2 (4)°. The *N*-iodomethyl-*N*,*N*-dimethyl-*N*-propylammonium moiety adopts a fully extended conformation with one methyl group and the iodomethyl unit on opposite sides of the backbone of the side chain (Fig. 2). This conformation seems to be partially supported by a C—H$\cdots$I$^-$ hydrogen bond (Table 2 and *Supramolecular features*).

In compound (III), Fig. 3, the phenyl rings are twisted out of the plane defined by the ethylene moiety (C4–C7/C13), making dihedral angles of 38.7 (4) and 78.7 (6)° for the *trans* (C7–C12) and *cis* (C13–C18) phenyl rings, respectively. The phenyl rings are inclined to one another by 78.5 (6)°. The alkylamino side chain is almost fully extended away from the geminal-substituted ethene group.

3. Supramolecular features

In the crystal of (I), ribbons are formed, by I1$\cdots$I2^{i} contacts [3.564 (4) Å; symmetry code: (i) $-x - 1, y - \frac{1}{2}, -z + 1$] and C—H$\cdots$I$^-$ hydrogen bonds, along the *a*-axis direction. The chains are reinforced by C—H$\cdots\pi$ interactions (Fig. 4 and Table 1).

In the crystal of (II), helical chains along the *b*-axis direction are formed by molecules linked *via* C—H$\cdots$I$^-$ (Table 2) and I1$\cdots$I2^{ii} interactions [3.506 (1) Å; symmetry code: (ii) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$]; as shown in Fig. 5. Here no C—H$\cdots\pi$ interactions are present in the crystal packing. The closest distance between the ammonium substituents and any of the phenyl rings is *ca* 7.18 Å. These features clearly rule out an intramolecular cation–π interaction for this molecule in the solid state. However, in studies of distonic radical cation (Ríos *et al.* 1996; Yates *et al.*, 1986), evidence is presented that the active conformation of the alkylamino side chain is oriented

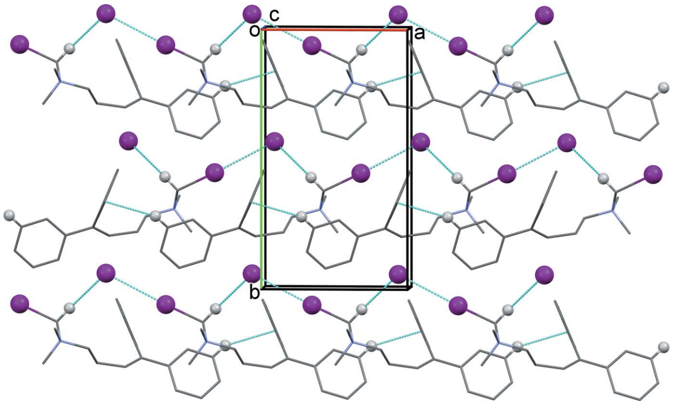


Figure 4
The crystal packing of compound (I), viewed along the *b* axis, showing the intermolecular contacts (dashed lines; see Table 1).

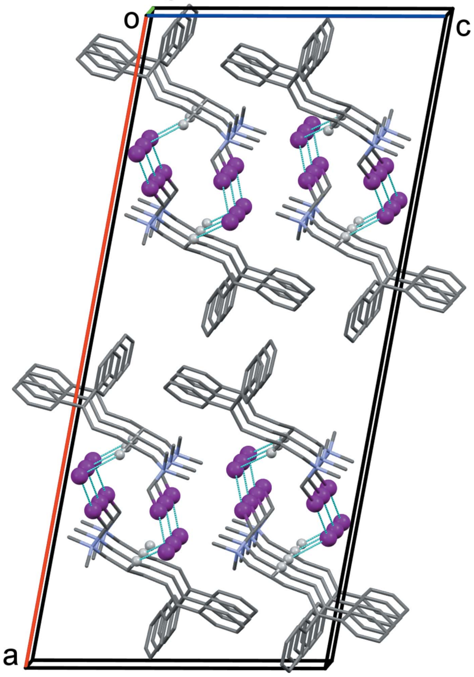


Figure 5
The crystal packing of compound (II), viewed along the *b* axis, showing the intermolecular contacts (dashed lines; see Table 2).

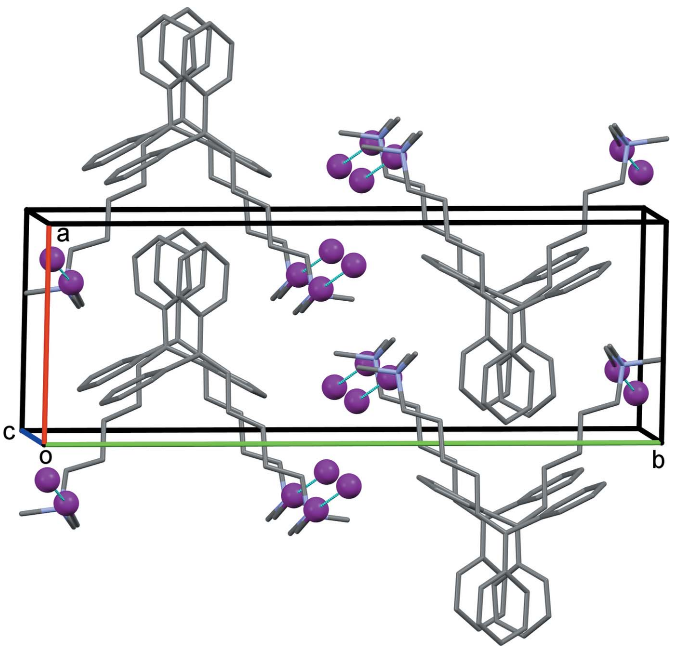


Figure 6
The crystal packing of compound (III), viewed along the *b* axis.

toward and above the plane of the C═C double bond of the geminal-substituted ethene group. These results confirm that there is considerable freedom of rotation about the bonds separating the basic amino function and the tricyclic system, and thus numerous interconvertible side-chain conformations, differing only slightly in potential energy, may exist.

In the crystal of (III), apart from the I1$\cdots$I2$^{\rm iii}$ contact of 3.557 (1) Å [symmetry code: (iii) $-x, -y+1, -z$], there are no other significant intermolecular contacts present (Fig. 6). The only possible conclusion regarding the crystal structure of (III) is that the steric requirements in this molecule outweigh the additional stabilization obtained by an intramolecular cation–π interaction.

4. Synthesis and crystallization

The general procedure for the preparation of the title quaternary ammonium salts is illustrated in Fig. 7 for compounds (I)–(III). The reactions were carried out following a standard literature method (Ríos *et al.*, 1996) starting from the appropriate amine [*N*,*N*-dimethyl-4,4-diphenylbut-3-en-1-amine 1(a), *N*,*N*-dimethyl-5,5-diphenylpent-4-en-1-amine 1(b) and *N*,*N*-dimethyl-6,6-diphenylhex-5-en-1-amine 1(c)]. Typically, CH_2I_2 (4 eq) and 1 eq of the starting tertiary amine [for example, compound 1(a) for the synthesis of (I); as shown in Fig. 7] were dissolved in acetonitrile. The reactions were allowed to run overnight at room temperature, and the precipitated salts were filtered off and washed several times with diethyl ether, and then recrystallized from a binary mixture water–isopropanol. The desired products were obtained as colourless crystals.

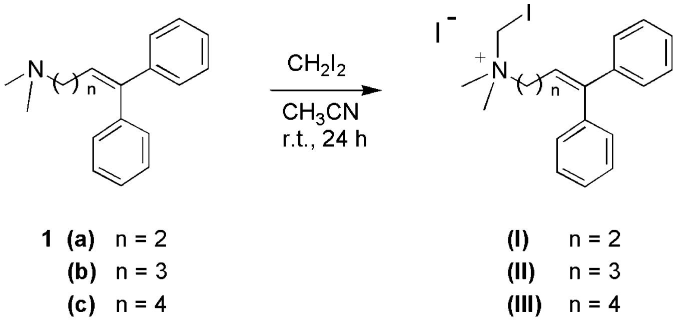


Figure 7
The general procedure for the preparation of the title quaternary ammonium salts.

Compound (I): The product was obtained as a white solid in 74% yield; m.p. 425–427 K. ^{1}H NMR (DMSO, 300 MHz, δ, p.p.m.): 2.49 (*m*, 2H), 3.12 (*s*, 6H), 3.50 (*m*, 2H), 5.05 (*s*, 2H), 6.07 (*t*, J = 7.4 Hz, 1H), 7.15–7.58 (*m*, 10H) p.p.m. ^{13}C NMR (DMSO, 75 MHz,, p.p.m.) 23.70, 31.49, 51.66, 63.58, 121.92, 127.19–129.51, 138.79, 141.62, 145.03 p.p.m. Elemental analysis calculated for $C_{19}H_{23}NI_2$: C, 43.95%; H, 4.46%; N, 2.70%; found, C, 43.48%; H, 4.35%; N, 2.68%. MS–ESI calculated for $C_{19}H_{23}NI$: 392.09, found: 391.95.

Compound (II): The product was obtained as a white solid in 77% yield; m.p. 430–437 K. ^{1}H NMR (DMSO, 300 MHz, δ, p.p.m.): 1.85 (*m*, 2H), 2.12 (*m*, 2H), 2.51 (*m*, 2H), 3.15 (*s*, 6H), 5.18 (*s*, 2H), 6.14 (*t*, J = 7.2 Hz, 1H), 7.11–7.51 (10H). ^{13}C NMR (DMSO, 75 MHz, p.p.m.): 22.30, 25.91, 39.01, 51.19, 63.84, 126.84–141.68. ESI–MS *m*/*z* calculated for $C_{20}H_{25}NI$: 406.10, found: 406.20.

Compound (III): The product was obtained as a white solid in 72% yield; m.p. 429–431 K. ^{1}H NMR (DMSO, 300 MHz, δ, p.p.m.): 1.45 (*m*, 2H), 1.68 (*m*, 2H), 2.12 (*m*, 2H), 2.51 (*m*, 2H), 3.10 (*s*, 6H), 5.14 (*s*, 2H), 6.14 (*t*, J = 7.3 Hz, 1H), 7.06–7.51 (m, 10H) p.p.m. ^{13}C NMR (DMSO, 75 MHz, p.p.m.): 25.07, 28.91, 31.91, 35.35, 54.29, 67.34, 129.89–132.50, 130.19, 142.45, 144.34, 145.02. Elemental analysis calculated for $C_{21}H_{27}NI_2$: C, 46.09%; H, 4.97%; N, 2.56%; found C, 45.91%; H, 4.93%; N, 2.58%. ESI–MS *m*/*z* calculated for $C_{21}H_{27}NI$: 420.12, found: 420.20.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. For all three compounds the C-bound H atoms were included in calculated positions and treated as riding atoms: C—H = 0.93–0.99 Å with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for methyl H atoms and 1.2$U_{\rm eq}$(C) for other H atoms. Refining the structure of compound (I) in the non-centrosymmetric space group gives a value of 0.02 (3) for the Flack parameter (Flack & Bernardinelli, 1999), confirming that the direction of the polar axis has been correctly determined. The studied crystal of compound (III) was a non-merohedral twin with a ratio of two major domains of 0.374 (2):0.626 (2). The two domains are rotated from each other by 180.0° about the reciprocal axis a^*, as determined by the *CELL NOW* program (Sheldrick, 2004). The final refinement was carried out using the twinned data set.

Table 3
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{19}H_{23}IN^{+}\cdot I^{-}$	$C_{20}H_{25}IN^{+}\cdot I^{-}$	$C_{21}H_{27}IN^{+}\cdot I^{-}$
M_r	519.18	533.21	547.23
Crystal system, space group	Monoclinic, *P*2$_1$	Monoclinic, *C*2/*c*	Monoclinic, *P*2$_1$/*c*
Temperature (K)	298	298	298
a, *b*, *c* (Å)	7.9254 (2), 13.6161 (3), 9.4632 (2)	37.778 (7), 6.6323 (12), 17.021 (3)	8.9423 (12), 24.058 (3), 10.3749 (13)
β (°)	103.320 (1)	100.567 (4)	103.656 (3)
V (Å^3)	993.73 (4)	4192.3 (13)	2168.9 (5)
Z	2	8	4
Radiation type	Mo *Kα*	Mo *Kα*	Mo *Kα*
μ (mm^{-1})	3.16	3.00	2.90
Crystal size (mm)	0.23 × 0.19 × 0.12	0.21 × 0.20 × 0.08	0.32 × 0.22 × 0.04
Data collection			
Diffractometer	Bruker SMART APEX CCD	Bruker SMART APEX CCD	Bruker SMART APEX CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)	Multi-scan (*SADABS*; Bruker, 2012)	Multi-scan (*TWINABS*; Bruker, 2012)
$T_{\min}$, $T_{\max}$	0.624, 0.745	0.349, 0.745	0.273, 0.429
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5791, 3085, 3013	16925, 3808, 3114	3961, 3961, 2941
$R_{\rm int}$	0.016	0.079	0.079
$(\sin \theta/\lambda)_{\max}$ (Å^{-1})	0.602	0.602	0.603
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.017, 0.038, 1.08	0.052, 0.145, 1.05	0.060, 0.138, 1.05
No. of reflections	3085	3808	3961
No. of parameters	202	210	220
No. of restraints	1	0	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å^{-3})	0.27, −0.46	1.90, −1.98	0.82, −0.80
Absolute structure	Refined as an inversion twin	–	–
Absolute structure parameter	0.02 (3)	–	–

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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supporting information

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Crystal structures of three new *N*-halomethylated quaternary ammonium salts

Carolina Múnera-Orozco, Rogelio Ocampo-Cardona, David L. Cedeño, Rubén A. Toscano and Luz Amalia Ríos-Vásquez

Computing details

For all compounds, data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

(I) *N*-(4,4-Diphenylbut-3-en-1-yl)-*N*-iodomethyl-*N*,*N*-dimethylammonium iodide

Crystal data

$C_{19}H_{23}IN^+\cdot I^-$
$M_r = 519.18$
Monoclinic, $P2_1$
$a = 7.9254$ (2) Å
$b = 13.6161$ (3) Å
$c = 9.4632$ (2) Å
$\beta = 103.320$ (1)°
$V = 993.73$ (4) Å^3
$Z = 2$
$F(000) = 500$

$D_x = 1.735$ Mg m^{-3}
Melting point = 425–427 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4987 reflections
$\theta = 2.2$–25.3°
$\mu = 3.16$ mm^{-1}
$T = 298$ K
Prism, colourless
0.23 × 0.19 × 0.12 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.333 pixels mm^{-1}
ω–scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2012)
$T_{min} = 0.624$, $T_{max} = 0.745$

5791 measured reflections
3085 independent reflections
3013 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 25.3°$, $\theta_{min} = 2.2°$
$h = -9\rightarrow9$
$k = -16\rightarrow16$
$l = -11\rightarrow11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.017$
$wR(F^2) = 0.038$
$S = 1.08$
3085 reflections
202 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
$w = 1/[\sigma^2(F_o^2) + (0.0138P)^2 + 0.0203P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.46$ e Å^{-3}

Absolute structure: Refined as an inversion twin
Absolute structure parameter: 0.02 (3)

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. Refined as a 2-component inversion twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
I1	−0.65685 (3)	0.06224 (2)	0.13054 (3)	0.04839 (9)
I2	0.06519 (4)	0.44189 (2)	0.91487 (3)	0.04368 (9)
N1	−0.3770 (4)	0.2023 (3)	0.0555 (4)	0.0376 (8)
C1	−0.1849 (5)	0.2322 (4)	0.0948 (5)	0.0402 (10)
H1A	−0.1144	0.1733	0.1019	0.048*
H1B	−0.1609	0.2719	0.0166	0.048*
C2	−0.1311 (5)	0.2891 (3)	0.2358 (5)	0.0409 (11)
H2A	−0.1888	0.3524	0.2249	0.049*
H2B	−0.1676	0.2536	0.3124	0.049*
C3	0.0613 (6)	0.3044 (3)	0.2779 (5)	0.0382 (10)
H3	0.1101	0.3408	0.2143	0.046*
C4	0.1686 (5)	0.2711 (3)	0.3970 (4)	0.0329 (9)
C5	0.3545 (5)	0.3021 (3)	0.4380 (4)	0.0349 (9)
C6	0.4815 (6)	0.2388 (4)	0.5116 (5)	0.0457 (11)
H6	0.4514	0.1752	0.5318	0.055*
C7	0.6528 (6)	0.2687 (4)	0.5557 (5)	0.0546 (13)
H7	0.7362	0.2248	0.6040	0.065*
C8	0.7001 (6)	0.3612 (5)	0.5292 (6)	0.0598 (15)
H8	0.8148	0.3812	0.5610	0.072*
C9	0.5768 (7)	0.4253 (4)	0.4549 (7)	0.0661 (16)
H9	0.6087	0.4887	0.4357	0.079*
C10	0.4056 (6)	0.3959 (4)	0.4085 (6)	0.0532 (13)
H10	0.3239	0.4395	0.3569	0.064*
C11	0.1128 (5)	0.2030 (3)	0.5013 (4)	0.0326 (9)
C12	0.0439 (6)	0.1106 (3)	0.4575 (5)	0.0422 (10)
H12	0.0314	0.0909	0.3616	0.051*
C13	−0.0060 (6)	0.0480 (3)	0.5561 (5)	0.0500 (12)
H13	−0.0522	−0.0134	0.5258	0.060*
C14	0.0124 (6)	0.0761 (4)	0.6978 (5)	0.0523 (13)
H14	−0.0198	0.0334	0.7637	0.063*
C15	0.0784 (6)	0.1674 (4)	0.7431 (5)	0.0467 (12)
H15	0.0883	0.1871	0.8388	0.056*
C16	0.1298 (5)	0.2295 (4)	0.6457 (5)	0.0403 (10)
H16	0.1768	0.2905	0.6774	0.048*
C17	−0.4036 (5)	0.1302 (3)	0.1685 (5)	0.0411 (10)

H17A	−0.3821	0.1637	0.2614	0.049*
H17B	−0.3174	0.0787	0.1761	0.049*
C18	−0.4928 (7)	0.2892 (4)	0.0510 (7)	0.0633 (15)
H18A	−0.6106	0.2703	0.0100	0.095*
H18B	−0.4590	0.3397	−0.0076	0.095*
H18C	−0.4832	0.3135	0.1478	0.095*
C19	−0.4089 (7)	0.1546 (5)	−0.0910 (5)	0.0596 (14)
H19A	−0.5290	0.1369	−0.1218	0.089*
H19B	−0.3385	0.0967	−0.0855	0.089*
H19C	−0.3796	0.1997	−0.1596	0.089*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.03965 (16)	0.05161 (19)	0.05521 (18)	−0.00709 (15)	0.01359 (13)	−0.00180 (15)
I2	0.04705 (17)	0.03979 (15)	0.04515 (16)	0.00372 (15)	0.01257 (12)	0.00767 (14)
N1	0.0317 (17)	0.043 (2)	0.0351 (19)	−0.0020 (17)	0.0010 (15)	0.0047 (16)
C1	0.029 (2)	0.051 (3)	0.039 (2)	−0.005 (2)	0.0042 (18)	0.006 (2)
C2	0.035 (2)	0.043 (3)	0.042 (3)	0.001 (2)	0.004 (2)	−0.001 (2)
C3	0.038 (2)	0.037 (2)	0.038 (2)	−0.004 (2)	0.0058 (19)	0.003 (2)
C4	0.034 (2)	0.031 (2)	0.033 (2)	−0.0022 (18)	0.0073 (18)	−0.0027 (18)
C5	0.033 (2)	0.037 (2)	0.035 (2)	−0.002 (2)	0.0087 (18)	−0.0047 (19)
C6	0.037 (3)	0.055 (3)	0.046 (3)	0.003 (2)	0.012 (2)	0.011 (2)
C7	0.033 (2)	0.083 (4)	0.049 (3)	0.007 (3)	0.010 (2)	0.009 (3)
C8	0.034 (3)	0.080 (4)	0.065 (4)	−0.009 (3)	0.011 (3)	−0.014 (3)
C9	0.049 (3)	0.048 (4)	0.103 (4)	−0.019 (3)	0.021 (3)	−0.017 (3)
C10	0.040 (3)	0.037 (2)	0.082 (4)	−0.003 (2)	0.012 (3)	0.003 (3)
C11	0.0266 (19)	0.035 (2)	0.035 (2)	0.0031 (18)	0.0054 (17)	0.0010 (18)
C12	0.043 (2)	0.040 (2)	0.045 (2)	−0.002 (2)	0.013 (2)	−0.004 (2)
C13	0.054 (3)	0.032 (3)	0.068 (3)	−0.001 (2)	0.022 (2)	0.004 (2)
C14	0.046 (3)	0.054 (3)	0.061 (3)	0.009 (3)	0.019 (2)	0.023 (3)
C15	0.043 (3)	0.065 (3)	0.031 (2)	0.007 (3)	0.008 (2)	0.007 (2)
C16	0.031 (2)	0.048 (3)	0.040 (2)	−0.004 (2)	0.0043 (19)	−0.004 (2)
C17	0.035 (2)	0.049 (3)	0.037 (2)	−0.005 (2)	0.0035 (18)	0.005 (2)
C18	0.041 (3)	0.049 (3)	0.091 (4)	0.009 (3)	−0.002 (3)	0.013 (3)
C19	0.055 (3)	0.084 (4)	0.036 (3)	−0.014 (3)	0.003 (2)	0.001 (3)

Geometric parameters (Å, º)

I1—C17	2.164 (4)	C8—H8	0.9300
I1—I2$^{\rm i}$	3.5640 (4)	C9—C10	1.386 (7)
N1—C18	1.492 (6)	C9—H9	0.9300
N1—C19	1.499 (6)	C10—H10	0.9300
N1—C17	1.502 (5)	C11—C16	1.390 (6)
N1—C1	1.536 (5)	C11—C12	1.396 (6)
C1—C2	1.517 (6)	C12—C13	1.387 (6)
C1—H1A	0.9700	C12—H12	0.9300
C1—H1B	0.9700	C13—C14	1.370 (7)

C2—C3	1.499 (6)	C13—H13	0.9300
C2—H2A	0.9700	C14—C15	1.379 (7)
C2—H2B	0.9700	C14—H14	0.9300
C3—C4	1.326 (6)	C15—C16	1.379 (6)
C3—H3	0.9300	C15—H15	0.9300
C4—C11	1.493 (6)	C16—H16	0.9300
C4—C5	1.495 (6)	C17—H17A	0.9700
C5—C6	1.385 (6)	C17—H17B	0.9700
C5—C10	1.388 (6)	C18—H18A	0.9600
C6—C7	1.387 (7)	C18—H18B	0.9600
C6—H6	0.9300	C18—H18C	0.9600
C7—C8	1.353 (8)	C19—H19A	0.9600
C7—H7	0.9300	C19—H19B	0.9600
C8—C9	1.376 (8)	C19—H19C	0.9600

C17—I1—I2^{i}	176.81 (12)	C9—C10—C5	120.8 (5)
C18—N1—C19	110.2 (4)	C9—C10—H10	119.6
C18—N1—C17	110.7 (4)	C5—C10—H10	119.6
C19—N1—C17	110.7 (4)	C16—C11—C12	118.0 (4)
C18—N1—C1	111.4 (4)	C16—C11—C4	120.8 (4)
C19—N1—C1	106.6 (3)	C12—C11—C4	121.2 (4)
C17—N1—C1	107.1 (3)	C13—C12—C11	120.5 (4)
C2—C1—N1	114.2 (3)	C13—C12—H12	119.8
C2—C1—H1A	108.7	C11—C12—H12	119.8
N1—C1—H1A	108.7	C14—C13—C12	120.3 (5)
C2—C1—H1B	108.7	C14—C13—H13	119.9
N1—C1—H1B	108.7	C12—C13—H13	119.9
H1A—C1—H1B	107.6	C13—C14—C15	120.2 (4)
C3—C2—C1	111.6 (4)	C13—C14—H14	119.9
C3—C2—H2A	109.3	C15—C14—H14	119.9
C1—C2—H2A	109.3	C14—C15—C16	119.7 (4)
C3—C2—H2B	109.3	C14—C15—H15	120.2
C1—C2—H2B	109.3	C16—C15—H15	120.2
H2A—C2—H2B	108.0	C15—C16—C11	121.4 (4)
C4—C3—C2	126.3 (4)	C15—C16—H16	119.3
C4—C3—H3	116.8	C11—C16—H16	119.3
C2—C3—H3	116.8	N1—C17—I1	116.0 (3)
C3—C4—C11	123.0 (4)	N1—C17—H17A	108.3
C3—C4—C5	121.6 (4)	I1—C17—H17A	108.3
C11—C4—C5	115.3 (3)	N1—C17—H17B	108.3
C6—C5—C10	117.5 (4)	I1—C17—H17B	108.3
C6—C5—C4	120.9 (4)	H17A—C17—H17B	107.4
C10—C5—C4	121.6 (4)	N1—C18—H18A	109.5
C5—C6—C7	121.1 (5)	N1—C18—H18B	109.5
C5—C6—H6	119.4	H18A—C18—H18B	109.5
C7—C6—H6	119.4	N1—C18—H18C	109.5
C8—C7—C6	120.7 (5)	H18A—C18—H18C	109.5
C8—C7—H7	119.6	H18B—C18—H18C	109.5

C6—C7—H7	119.6	N1—C19—H19A	109.5
C7—C8—C9	119.4 (5)	N1—C19—H19B	109.5
C7—C8—H8	120.3	H19A—C19—H19B	109.5
C9—C8—H8	120.3	N1—C19—H19C	109.5
C8—C9—C10	120.5 (5)	H19A—C19—H19C	109.5
C8—C9—H9	119.8	H19B—C19—H19C	109.5
C10—C9—H9	119.8		

C18—N1—C1—C2	55.2 (5)	C6—C5—C10—C9	−1.8 (8)
C19—N1—C1—C2	175.5 (4)	C4—C5—C10—C9	176.1 (5)
C17—N1—C1—C2	−65.9 (5)	C3—C4—C11—C16	−120.5 (5)
N1—C1—C2—C3	172.5 (4)	C5—C4—C11—C16	57.8 (5)
C1—C2—C3—C4	−117.9 (5)	C3—C4—C11—C12	60.2 (6)
C2—C3—C4—C11	6.6 (7)	C5—C4—C11—C12	−121.5 (4)
C2—C3—C4—C5	−171.6 (4)	C16—C11—C12—C13	0.2 (6)
C3—C4—C5—C6	−148.1 (5)	C4—C11—C12—C13	179.5 (4)
C11—C4—C5—C6	33.5 (5)	C11—C12—C13—C14	−0.3 (7)
C3—C4—C5—C10	34.0 (6)	C12—C13—C14—C15	0.9 (7)
C11—C4—C5—C10	−144.3 (4)	C13—C14—C15—C16	−1.5 (7)
C10—C5—C6—C7	0.9 (7)	C14—C15—C16—C11	1.5 (7)
C4—C5—C6—C7	−177.0 (4)	C12—C11—C16—C15	−0.8 (6)
C5—C6—C7—C8	0.7 (7)	C4—C11—C16—C15	179.9 (4)
C6—C7—C8—C9	−1.4 (8)	C18—N1—C17—I1	64.6 (4)
C7—C8—C9—C10	0.5 (9)	C19—N1—C17—I1	−57.9 (4)
C8—C9—C10—C5	1.1 (9)	C1—N1—C17—I1	−173.8 (3)

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

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C11–C16 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17*B*···I2^{ii}	0.97	3.00	3.919 (5)	159
C7—H7···*Cg*^{iii}	0.93	2.84	3.030 (5)	143

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

(II) *N*-(5,5-Diphenylpent-4-en-1-yl)-*N*-iodomethyl-*N*,*N*-dimethylammonium iodide

Crystal data

$C_{20}H_{25}IN^{+}\cdot I^{-}$
M_r = 533.21
Monoclinic, *C*2/*c*
a = 37.778 (7) Å
b = 6.6323 (12) Å
c = 17.021 (3) Å
β = 100.567 (4)°
V = 4192.3 (13) Å^3
Z = 8
F(000) = 2064

D_x = 1.690 Mg m^{-3}
Melting point = 430–431 K
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 4726 reflections
θ = 2.2–25.3°
μ = 3.00 mm^{-1}
T = 298 K
Platy-prism, colourless
0.21 × 0.20 × 0.08 mm

Data collection

Bruker SMART APEX CCD diffractometer	16925 measured reflections
Radiation source: fine-focus sealed tube	3808 independent reflections
Graphite monochromator	3114 reflections with $I > 2\sigma(I)$
ω–scans	$R_{\text{int}} = 0.079$
Absorption correction: multi-scan (*SADABS*; Bruker, 2012)	$\theta_{\text{max}} = 25.4°$, $\theta_{\text{min}} = 2.2°$
$T_{\text{min}} = 0.349$, $T_{\text{max}} = 0.745$	$h = -45 \rightarrow 45$
	$k = -7 \rightarrow 7$
	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.052$	H-atom parameters constrained
$wR(F^2) = 0.145$	$w = 1/[\sigma^2(F_o^2) + (0.0918P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\text{max}} = 0.001$
3808 reflections	$\Delta\rho_{\text{max}} = 1.90$ e Å^{-3}
210 parameters	$\Delta\rho_{\text{min}} = -1.98$ e Å^{-3}
0 restraints	
Primary atom site location: structure-invariant direct methods	

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.25244 (2)	−0.01229 (6)	0.13640 (2)	0.04488 (19)
I2	0.30512 (2)	0.07869 (7)	0.42246 (3)	0.04999 (19)
N1	0.31227 (12)	0.3200 (8)	0.1496 (2)	0.0383 (11)
C1	0.32956 (18)	0.4989 (9)	0.1981 (4)	0.0446 (15)
H1A	0.3463	0.5629	0.1690	0.053*
H1B	0.3109	0.5965	0.2029	0.053*
C2	0.34941 (19)	0.4459 (10)	0.2810 (4)	0.0503 (16)
H2A	0.3713	0.3736	0.2770	0.060*
H2B	0.3345	0.3583	0.3069	0.060*
C3	0.35877 (18)	0.6363 (11)	0.3312 (4)	0.0498 (15)
H3A	0.3769	0.7134	0.3107	0.060*
H3B	0.3375	0.7199	0.3284	0.060*
C4	0.37268 (18)	0.5764 (12)	0.4160 (4)	0.0536 (17)
H4	0.3577	0.4935	0.4394	0.064*
C5	0.40427 (16)	0.6276 (11)	0.4628 (3)	0.0476 (15)
C6	0.41456 (18)	0.5452 (12)	0.5446 (4)	0.0537 (18)
C7	0.4042 (2)	0.3523 (14)	0.5653 (4)	0.066 (2)
H7	0.3905	0.2719	0.5265	0.080*
C8	0.4140 (2)	0.2791 (17)	0.6421 (5)	0.083 (3)
H8	0.4068	0.1506	0.6544	0.099*

C9	0.4346 (2)	0.396 (2)	0.7012 (5)	0.095 (4)
H9	0.4411	0.3468	0.7530	0.114*
C10	0.4453 (2)	0.586 (2)	0.6822 (5)	0.098 (4)
H10	0.4592	0.6652	0.7213	0.117*
C11	0.43528 (19)	0.6603 (16)	0.6054 (4)	0.073 (2)
H11	0.4425	0.7893	0.5937	0.088*
C12	0.42943 (16)	0.7710 (12)	0.4341 (3)	0.0494 (16)
C13	0.4190 (2)	0.9644 (13)	0.4097 (5)	0.067 (2)
H13	0.3957	1.0067	0.4121	0.080*
C14	0.4417 (3)	1.0942 (16)	0.3824 (6)	0.087 (3)
H14	0.4339	1.2226	0.3655	0.104*
C15	0.4761 (3)	1.036 (2)	0.3796 (7)	0.097 (4)
H15	0.4914	1.1257	0.3604	0.117*
C16	0.4882 (2)	0.849 (2)	0.4045 (5)	0.091 (3)
H16	0.5118	0.8115	0.4026	0.109*
C17	0.46478 (18)	0.7137 (15)	0.4330 (4)	0.067 (2)
H17	0.4729	0.5865	0.4509	0.080*
C18	0.28176 (16)	0.2484 (10)	0.1869 (3)	0.0425 (14)
H18A	0.2648	0.3587	0.1853	0.051*
H18B	0.2911	0.2201	0.2428	0.051*
C19	0.33945 (17)	0.1549 (11)	0.1464 (4)	0.0509 (16)
H19A	0.3288	0.0516	0.1101	0.076*
H19B	0.3468	0.0982	0.1988	0.076*
H19C	0.3601	0.2098	0.1283	0.076*
C20	0.29821 (18)	0.3908 (10)	0.0658 (3)	0.0495 (16)
H20A	0.2870	0.2801	0.0343	0.074*
H20B	0.3178	0.4416	0.0427	0.074*
H20C	0.2808	0.4959	0.0668	0.074*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0501 (3)	0.0520 (3)	0.0289 (3)	−0.01023 (17)	−0.00240 (18)	0.00087 (16)
I2	0.0512 (3)	0.0512 (3)	0.0461 (3)	0.00415 (19)	0.0052 (2)	0.01397 (19)
N1	0.041 (3)	0.050 (3)	0.021 (2)	−0.003 (2)	−0.0006 (19)	−0.002 (2)
C1	0.052 (4)	0.047 (4)	0.033 (3)	−0.013 (3)	0.001 (3)	−0.003 (3)
C2	0.052 (4)	0.062 (4)	0.031 (3)	−0.008 (3)	−0.009 (3)	−0.005 (3)
C3	0.044 (3)	0.062 (4)	0.039 (3)	−0.010 (3)	−0.003 (3)	−0.011 (3)
C4	0.047 (4)	0.082 (5)	0.028 (3)	−0.012 (3)	−0.001 (3)	−0.011 (3)
C5	0.042 (3)	0.067 (4)	0.031 (3)	−0.009 (3)	0.000 (2)	−0.019 (3)
C6	0.035 (3)	0.089 (5)	0.036 (3)	0.001 (3)	0.004 (3)	−0.008 (3)
C7	0.064 (5)	0.088 (6)	0.046 (4)	0.000 (4)	0.008 (3)	−0.002 (4)
C8	0.066 (5)	0.126 (8)	0.058 (5)	0.013 (5)	0.018 (4)	0.021 (5)
C9	0.051 (5)	0.188 (12)	0.044 (5)	0.007 (6)	0.002 (4)	0.014 (6)
C10	0.059 (5)	0.190 (12)	0.039 (4)	−0.033 (7)	−0.004 (4)	−0.024 (6)
C11	0.057 (4)	0.118 (7)	0.042 (4)	−0.023 (5)	0.001 (3)	−0.021 (4)
C12	0.040 (3)	0.075 (5)	0.029 (3)	−0.008 (3)	−0.003 (2)	−0.016 (3)
C13	0.057 (5)	0.075 (5)	0.061 (5)	−0.012 (4)	−0.007 (4)	−0.020 (4)

C14	0.079 (6)	0.092 (7)	0.079 (6)	−0.027 (5)	−0.014 (5)	0.009 (5)
C15	0.073 (7)	0.136 (10)	0.076 (6)	−0.044 (6)	−0.004 (5)	0.025 (7)
C16	0.047 (4)	0.167 (11)	0.056 (5)	−0.019 (6)	0.004 (4)	−0.005 (6)
C17	0.043 (4)	0.111 (7)	0.042 (4)	−0.004 (4)	−0.002 (3)	−0.004 (4)
C18	0.044 (3)	0.058 (4)	0.025 (3)	−0.009 (3)	0.003 (2)	−0.004 (3)
C19	0.048 (4)	0.058 (4)	0.044 (3)	0.005 (3)	0.002 (3)	−0.011 (3)
C20	0.057 (4)	0.061 (4)	0.028 (3)	−0.004 (3)	0.000 (3)	0.006 (3)

Geometric parameters (Å, º)

I1—C18	2.146 (6)	C9—C10	1.381 (16)
I1—I2^{i}	3.5058 (7)	C9—H9	0.9300
N1—C18	1.492 (7)	C10—C11	1.382 (12)
N1—C20	1.504 (7)	C10—H10	0.9300
N1—C19	1.509 (8)	C11—H11	0.9300
N1—C1	1.522 (8)	C12—C13	1.383 (11)
C1—C2	1.513 (9)	C12—C17	1.392 (9)
C1—H1A	0.9700	C13—C14	1.357 (12)
C1—H1B	0.9700	C13—H13	0.9300
C2—C3	1.530 (9)	C14—C15	1.361 (14)
C2—H2A	0.9700	C14—H14	0.9300
C2—H2B	0.9700	C15—C16	1.363 (16)
C3—C4	1.496 (9)	C15—H15	0.9300
C3—H3A	0.9700	C16—C17	1.410 (13)
C3—H3B	0.9700	C16—H16	0.9300
C4—C5	1.351 (9)	C17—H17	0.9300
C4—H4	0.9300	C18—H18A	0.9700
C5—C6	1.480 (10)	C18—H18B	0.9700
C5—C12	1.489 (9)	C19—H19A	0.9600
C6—C7	1.402 (12)	C19—H19B	0.9600
C6—C11	1.404 (10)	C19—H19C	0.9600
C7—C8	1.379 (11)	C20—H20A	0.9600
C7—H7	0.9300	C20—H20B	0.9600
C8—C9	1.390 (14)	C20—H20C	0.9600
C8—H8	0.9300		
C18—I1—I2^{i}	170.16 (15)	C9—C10—C11	120.3 (9)
C18—N1—C20	109.7 (4)	C9—C10—H10	119.9
C18—N1—C19	111.6 (5)	C11—C10—H10	119.9
C20—N1—C19	108.5 (5)	C10—C11—C6	121.6 (9)
C18—N1—C1	107.8 (4)	C10—C11—H11	119.2
C20—N1—C1	108.2 (5)	C6—C11—H11	119.2
C19—N1—C1	111.0 (5)	C13—C12—C17	118.1 (7)
C2—C1—N1	114.4 (5)	C13—C12—C5	121.8 (6)
C2—C1—H1A	108.6	C17—C12—C5	120.1 (7)
N1—C1—H1A	108.6	C14—C13—C12	121.8 (8)
C2—C1—H1B	108.6	C14—C13—H13	119.1
N1—C1—H1B	108.6	C12—C13—H13	119.1

H1A—C1—H1B	107.6
C1—C2—C3	110.7 (6)
C1—C2—H2A	109.5
C3—C2—H2A	109.5
C1—C2—H2B	109.5
C3—C2—H2B	109.5
H2A—C2—H2B	108.1
C4—C3—C2	108.9 (6)
C4—C3—H3A	109.9
C2—C3—H3A	109.9
C4—C3—H3B	109.9
C2—C3—H3B	109.9
H3A—C3—H3B	108.3
C5—C4—C3	128.2 (7)
C5—C4—H4	115.9
C3—C4—H4	115.9
C4—C5—C6	120.9 (6)
C4—C5—C12	121.0 (6)
C6—C5—C12	118.1 (5)
C7—C6—C11	117.0 (7)
C7—C6—C5	122.5 (6)
C11—C6—C5	120.5 (7)
C8—C7—C6	121.4 (8)
C8—C7—H7	119.3
C6—C7—H7	119.3
C7—C8—C9	120.4 (10)
C7—C8—H8	119.8
C9—C8—H8	119.8
C10—C9—C8	119.3 (8)
C10—C9—H9	120.3
C8—C9—H9	120.3
C13—C14—C15	120.0 (10)
C13—C14—H14	120.0
C15—C14—H14	120.0
C14—C15—C16	121.0 (9)
C14—C15—H15	119.5
C16—C15—H15	119.5
C15—C16—C17	119.3 (9)
C15—C16—H16	120.3
C17—C16—H16	120.3
C12—C17—C16	119.7 (9)
C12—C17—H17	120.1
C16—C17—H17	120.1
N1—C18—I1	117.9 (4)
N1—C18—H18A	107.8
I1—C18—H18A	107.8
N1—C18—H18B	107.8
I1—C18—H18B	107.8
H18A—C18—H18B	107.2
N1—C19—H19A	109.5
N1—C19—H19B	109.5
H19A—C19—H19B	109.5
N1—C19—H19C	109.5
H19A—C19—H19C	109.5
H19B—C19—H19C	109.5
N1—C20—H20A	109.5
N1—C20—H20B	109.5
H20A—C20—H20B	109.5
N1—C20—H20C	109.5
H20A—C20—H20C	109.5
H20B—C20—H20C	109.5

C18—N1—C1—C2	69.2 (7)
C20—N1—C1—C2	−172.2 (5)
C19—N1—C1—C2	−53.3 (7)
N1—C1—C2—C3	−167.8 (5)
C1—C2—C3—C4	170.6 (6)
C2—C3—C4—C5	124.9 (8)
C3—C4—C5—C6	−176.7 (7)
C3—C4—C5—C12	4.3 (12)
C4—C5—C6—C7	32.1 (11)
C12—C5—C6—C7	−148.9 (7)
C4—C5—C6—C11	−147.5 (7)
C12—C5—C6—C11	31.5 (10)
C11—C6—C7—C8	0.0 (11)
C5—C6—C7—C8	−179.7 (7)
C6—C7—C8—C9	0.0 (12)
C7—C8—C9—C10	−0.2 (14)
C7—C6—C11—C10	0.4 (12)
C5—C6—C11—C10	−179.9 (8)
C4—C5—C12—C13	57.8 (9)
C6—C5—C12—C13	−121.2 (7)
C4—C5—C12—C17	−123.6 (8)
C6—C5—C12—C17	57.4 (8)
C17—C12—C13—C14	2.5 (11)
C5—C12—C13—C14	−179.0 (7)
C12—C13—C14—C15	−1.0 (14)
C13—C14—C15—C16	−0.4 (16)
C14—C15—C16—C17	0.4 (16)
C13—C12—C17—C16	−2.5 (10)
C5—C12—C17—C16	179.0 (6)
C15—C16—C17—C12	1.1 (13)
C20—N1—C18—I1	64.9 (6)
C19—N1—C18—I1	−55.4 (5)

C8—C9—C10—C11	0.6 (15)	C1—N1—C18—I1	−177.5 (4)
C9—C10—C11—C6	−0.7 (14)		

Symmetry code: (i) $-x+1/2, y-1/2, -z+1/2$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*B*···I2	0.97	3.06	4.001 (7)	165

(III) *N*-(6,6-Diphenylhex-5-en-1-yl)-*N*-iodomethyl-*N*,*N*-dimethylammonium iodide

Crystal data

$C_{21}H_{27}IN^+\cdot I^-$
$M_r = 547.23$
Monoclinic, $P2_1/c$
$a = 8.9423$ (12) Å
$b = 24.058$ (3) Å
$c = 10.3749$ (13) Å
$\beta = 103.656$ (3)°
$V = 2168.9$ (5) Å^3
$Z = 4$
$F(000) = 1064$
$D_x = 1.676$ Mg m^{-3}
Melting point = 429–431 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3046 reflections
$\theta = 2.3$–24.4°
$\mu = 2.90$ mm^{-1}
$T = 298$ K
Prism, colourless
0.32 × 0.22 × 0.04 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.333 pixels mm^{-1}
ω–scans
Absorption correction: multi-scan (*TWINABS*; Bruker, 2012)
$T_{min} = 0.273$, $T_{max} = 0.429$
3961 measured reflections
3961 independent reflections
2941 reflections with $I > 2\sigma(I)$
$R_{int} = 0.079$
$\theta_{max} = 25.4°$, $\theta_{min} = 1.7°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 28$
$l = 0 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.138$
$S = 1.05$
3961 reflections
220 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
$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 2.9321P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.82$ e Å^{-3}
$\Delta\rho_{min} = -0.80$ e Å^{-3}

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. Refined as a 2-component twin. The studied crystal was a nonmerohedral twin with a ratio of two major domains of 0.374 (2):0.626 (2). The two domains were rotated from each other by 180.0° about the recipocal axis (1 0 0), which was determined by the CELL NOW program (Sheldrick, 2004). The final refinement was carried out using twinned data set.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	y	z	U_{iso}*/U_{eq}
I1	−0.29957 (8)	0.44339 (3)	0.01740 (6)	0.0597 (2)
I2	0.17494 (8)	0.51476 (3)	0.26901 (6)	0.0598 (2)
N1	−0.3411 (8)	0.4415 (4)	0.3070 (7)	0.056 (2)
C1	−0.1733 (11)	0.4319 (4)	0.3631 (10)	0.063 (3)
H1A	−0.1159	0.4525	0.3103	0.076*
H1B	−0.1459	0.4469	0.4524	0.076*
C2	−0.1252 (12)	0.3731 (4)	0.3678 (12)	0.072 (3)
H2A	−0.1407	0.3584	0.2786	0.087*
H2B	−0.1860	0.3512	0.4153	0.087*
C3	0.0467 (11)	0.3699 (4)	0.4394 (11)	0.068 (3)
H3A	0.1029	0.3971	0.4001	0.082*
H3B	0.0578	0.3800	0.5317	0.082*
C4	0.1182 (12)	0.3134 (4)	0.4333 (15)	0.091 (4)
H4A	0.1013	0.3019	0.3413	0.109*
H4B	0.0681	0.2866	0.4789	0.109*
C5	0.2892 (10)	0.3135 (4)	0.4957 (12)	0.068 (3)
H5	0.3381	0.3478	0.5120	0.082*
C6	0.3743 (10)	0.2685 (3)	0.5287 (10)	0.055 (2)
C7	0.5451 (11)	0.2703 (3)	0.5813 (9)	0.052 (2)
C8	0.6398 (11)	0.2322 (4)	0.5435 (11)	0.066 (3)
H8	0.5966	0.2031	0.4881	0.080*
C9	0.8003 (12)	0.2359 (5)	0.5862 (14)	0.091 (4)
H9	0.8636	0.2109	0.5560	0.109*
C10	0.8623 (15)	0.2778 (5)	0.6747 (14)	0.097 (5)
H10	0.9683	0.2798	0.7079	0.116*
C11	0.7701 (16)	0.3158 (5)	0.7133 (14)	0.094 (4)
H11	0.8128	0.3446	0.7699	0.113*
C12	0.6122 (14)	0.3116 (5)	0.6681 (11)	0.079 (3)
H12	0.5495	0.3373	0.6969	0.094*
C13	0.3026 (11)	0.2115 (3)	0.5086 (10)	0.055 (2)
C14	0.2723 (13)	0.1847 (4)	0.6156 (12)	0.074 (3)
H14	0.2993	0.2012	0.6990	0.089*
C15	0.2004 (14)	0.1323 (4)	0.6000 (16)	0.088 (4)
H15	0.1840	0.1135	0.6738	0.105*
C16	0.1551 (14)	0.1091 (5)	0.4782 (18)	0.095 (4)
H16	0.1010	0.0757	0.4681	0.114*
C17	0.1888 (18)	0.1347 (6)	0.3678 (16)	0.116 (6)
H17	0.1614	0.1184	0.2842	0.139*
C18	0.2659 (17)	0.1861 (5)	0.3880 (12)	0.091 (4)
H18	0.2926	0.2034	0.3164	0.109*

C19	−0.4077 (11)	0.4192 (4)	0.1718 (10)	0.065 (3)
H19A	−0.5148	0.4304	0.1460	0.078*
H19B	−0.4058	0.3789	0.1771	0.078*
C20	−0.3648 (13)	0.5048 (4)	0.3059 (11)	0.074 (3)
H20A	−0.4723	0.5131	0.2740	0.111*
H20B	−0.3078	0.5218	0.2488	0.111*
H20C	−0.3294	0.5191	0.3943	0.111*
C21	−0.4312 (12)	0.4165 (5)	0.3995 (10)	0.071 (3)
H21A	−0.5382	0.4256	0.3684	0.107*
H21B	−0.3936	0.4313	0.4872	0.107*
H21C	−0.4189	0.3769	0.4015	0.107*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0559 (4)	0.0659 (4)	0.0605 (4)	0.0039 (3)	0.0202 (3)	0.0118 (3)
I2	0.0586 (4)	0.0618 (4)	0.0632 (4)	0.0015 (3)	0.0228 (3)	0.0043 (3)
N1	0.041 (4)	0.081 (6)	0.046 (4)	0.002 (4)	0.012 (4)	0.020 (4)
C1	0.045 (5)	0.078 (7)	0.069 (6)	−0.006 (5)	0.015 (5)	0.014 (5)
C2	0.058 (6)	0.056 (6)	0.106 (9)	0.003 (5)	0.024 (6)	−0.001 (6)
C3	0.057 (6)	0.069 (6)	0.075 (7)	0.012 (5)	0.007 (6)	0.017 (6)
C4	0.054 (7)	0.065 (7)	0.158 (12)	0.006 (5)	0.036 (8)	0.023 (8)
C5	0.040 (5)	0.043 (5)	0.123 (9)	0.002 (4)	0.022 (6)	0.012 (6)
C6	0.047 (5)	0.038 (5)	0.082 (7)	0.005 (4)	0.019 (5)	0.010 (5)
C7	0.052 (6)	0.038 (5)	0.069 (6)	−0.002 (4)	0.019 (5)	0.010 (4)
C8	0.051 (6)	0.055 (6)	0.090 (7)	0.004 (5)	0.009 (6)	−0.006 (5)
C9	0.051 (7)	0.083 (8)	0.140 (11)	0.017 (6)	0.026 (8)	0.022 (8)
C10	0.066 (8)	0.077 (8)	0.129 (11)	−0.028 (7)	−0.014 (8)	0.022 (8)
C11	0.100 (10)	0.054 (7)	0.116 (11)	−0.008 (7)	−0.001 (9)	−0.007 (7)
C12	0.075 (8)	0.074 (7)	0.087 (8)	−0.007 (6)	0.019 (7)	−0.002 (7)
C13	0.056 (6)	0.040 (5)	0.067 (6)	0.012 (4)	0.011 (5)	0.014 (5)
C14	0.079 (8)	0.053 (6)	0.101 (9)	−0.004 (6)	0.043 (7)	−0.012 (6)
C15	0.085 (9)	0.041 (6)	0.152 (13)	−0.009 (6)	0.058 (9)	0.011 (7)
C16	0.053 (7)	0.063 (7)	0.155 (14)	−0.001 (6)	−0.004 (9)	−0.005 (10)
C17	0.135 (14)	0.072 (9)	0.108 (11)	0.001 (9)	−0.038 (10)	−0.002 (8)
C18	0.116 (11)	0.069 (8)	0.075 (8)	−0.001 (8)	−0.002 (7)	0.001 (7)
C19	0.049 (6)	0.080 (7)	0.073 (7)	−0.005 (5)	0.029 (5)	0.024 (6)
C20	0.066 (7)	0.071 (7)	0.089 (8)	0.009 (6)	0.027 (6)	0.008 (6)
C21	0.056 (6)	0.091 (8)	0.072 (7)	−0.006 (6)	0.025 (6)	0.032 (6)

Geometric parameters (Å, º)

I1—C19	2.138 (9)	C9—C10	1.388 (17)
I1—I2^{i}	3.5565 (9)	C9—H9	0.9300
N1—C19	1.489 (12)	C10—C11	1.353 (18)
N1—C1	1.494 (12)	C10—H10	0.9300
N1—C21	1.515 (11)	C11—C12	1.383 (16)
N1—C20	1.537 (12)	C11—H11	0.9300

C1—C2	1.477 (13)	C12—H12	0.9300
C1—H1A	0.9700	C13—C18	1.360 (14)
C1—H1B	0.9700	C13—C14	1.365 (14)
C2—C3	1.543 (13)	C14—C15	1.407 (14)
C2—H2A	0.9700	C14—H14	0.9300
C2—H2B	0.9700	C15—C16	1.353 (18)
C3—C4	1.510 (14)	C15—H15	0.9300
C3—H3A	0.9700	C16—C17	1.39 (2)
C3—H3B	0.9700	C16—H16	0.9300
C4—C5	1.514 (14)	C17—C18	1.409 (17)
C4—H4A	0.9700	C17—H17	0.9300
C4—H4B	0.9700	C18—H18	0.9300
C5—C6	1.323 (12)	C19—H19A	0.9700
C5—H5	0.9300	C19—H19B	0.9700
C6—C7	1.497 (12)	C20—H20A	0.9600
C6—C13	1.507 (12)	C20—H20B	0.9600
C7—C8	1.366 (12)	C20—H20C	0.9600
C7—C12	1.379 (14)	C21—H21A	0.9600
C8—C9	1.401 (14)	C21—H21B	0.9600
C8—H8	0.9300	C21—H21C	0.9600

C19—I1—I2^{i}	171.6 (3)	C11—C10—C9	120.6 (11)
C19—N1—C1	116.9 (8)	C11—C10—H10	119.7
C19—N1—C21	107.4 (7)	C9—C10—H10	119.7
C1—N1—C21	109.1 (7)	C10—C11—C12	119.7 (12)
C19—N1—C20	109.1 (7)	C10—C11—H11	120.1
C1—N1—C20	106.3 (7)	C12—C11—H11	120.1
C21—N1—C20	107.8 (8)	C7—C12—C11	121.7 (12)
C2—C1—N1	114.8 (8)	C7—C12—H12	119.1
C2—C1—H1A	108.6	C11—C12—H12	119.1
N1—C1—H1A	108.6	C18—C13—C14	119.1 (9)
C2—C1—H1B	108.6	C18—C13—C6	122.5 (9)
N1—C1—H1B	108.6	C14—C13—C6	118.4 (9)
H1A—C1—H1B	107.5	C13—C14—C15	120.1 (12)
C1—C2—C3	108.2 (8)	C13—C14—H14	119.9
C1—C2—H2A	110.1	C15—C14—H14	119.9
C3—C2—H2A	110.1	C16—C15—C14	120.4 (12)
C1—C2—H2B	110.1	C16—C15—H15	119.8
C3—C2—H2B	110.1	C14—C15—H15	119.8
H2A—C2—H2B	108.4	C15—C16—C17	120.6 (11)
C4—C3—C2	114.0 (9)	C15—C16—H16	119.7
C4—C3—H3A	108.7	C17—C16—H16	119.7
C2—C3—H3A	108.7	C16—C17—C18	117.3 (13)
C4—C3—H3B	108.7	C16—C17—H17	121.3
C2—C3—H3B	108.7	C18—C17—H17	121.3
H3A—C3—H3B	107.6	C13—C18—C17	122.2 (13)
C3—C4—C5	112.1 (9)	C13—C18—H18	118.9
C3—C4—H4A	109.2	C17—C18—H18	118.9

C5—C4—H4A	109.2	N1—C19—I1	117.2 (6)
C3—C4—H4B	109.2	N1—C19—H19A	108.0
C5—C4—H4B	109.2	I1—C19—H19A	108.0
H4A—C4—H4B	107.9	N1—C19—H19B	108.0
C6—C5—C4	124.8 (9)	I1—C19—H19B	108.0
C6—C5—H5	117.6	H19A—C19—H19B	107.2
C4—C5—H5	117.6	N1—C20—H20A	109.5
C5—C6—C7	123.1 (8)	N1—C20—H20B	109.5
C5—C6—C13	120.7 (8)	H20A—C20—H20B	109.5
C7—C6—C13	116.2 (7)	N1—C20—H20C	109.5
C8—C7—C12	117.9 (10)	H20A—C20—H20C	109.5
C8—C7—C6	121.6 (9)	H20B—C20—H20C	109.5
C12—C7—C6	120.5 (9)	N1—C21—H21A	109.5
C7—C8—C9	121.6 (10)	N1—C21—H21B	109.5
C7—C8—H8	119.2	H21A—C21—H21B	109.5
C9—C8—H8	119.2	N1—C21—H21C	109.5
C10—C9—C8	118.4 (11)	H21A—C21—H21C	109.5
C10—C9—H9	120.8	H21B—C21—H21C	109.5
C8—C9—H9	120.8		

C19—N1—C1—C2	−55.7 (12)	C8—C7—C12—C11	1.8 (16)
C21—N1—C1—C2	66.3 (12)	C6—C7—C12—C11	−176.9 (10)
C20—N1—C1—C2	−177.7 (9)	C10—C11—C12—C7	−2 (2)
N1—C1—C2—C3	−174.9 (8)	C5—C6—C13—C18	78.5 (15)
C1—C2—C3—C4	−171.1 (10)	C7—C6—C13—C18	−98.8 (13)
C2—C3—C4—C5	175.7 (10)	C5—C6—C13—C14	−100.9 (13)
C3—C4—C5—C6	165.3 (12)	C7—C6—C13—C14	81.9 (12)
C4—C5—C6—C7	175.5 (11)	C18—C13—C14—C15	−1.6 (16)
C4—C5—C6—C13	−1.5 (18)	C6—C13—C14—C15	177.7 (9)
C5—C6—C7—C8	−140.0 (11)	C13—C14—C15—C16	−2.7 (18)
C13—C6—C7—C8	37.2 (13)	C14—C15—C16—C17	4.7 (19)
C5—C6—C7—C12	38.6 (15)	C15—C16—C17—C18	−2 (2)
C13—C6—C7—C12	−144.2 (10)	C14—C13—C18—C17	4.0 (19)
C12—C7—C8—C9	−2.7 (16)	C6—C13—C18—C17	−175.3 (11)
C6—C7—C8—C9	175.9 (10)	C16—C17—C18—C13	−2 (2)
C7—C8—C9—C10	3.5 (18)	C1—N1—C19—I1	−54.3 (10)
C8—C9—C10—C11	−3.4 (19)	C21—N1—C19—I1	−177.2 (7)
C9—C10—C11—C12	3 (2)	C20—N1—C19—I1	66.3 (9)

Symmetry code: (i) $-x$, $-y+1$, $-z$.