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## 2,6-Diisopropylanilinium chloride

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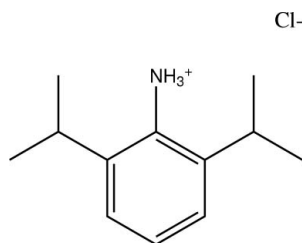
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Key indicators: single-crystal X-ray study;  $T = 173$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.037;  $wR$  factor = 0.112; data-to-parameter ratio = 16.3.

The title compound,  $\text{C}_{12}\text{H}_{20}\text{N}^+\cdot\text{Cl}^-$ , crystallizes with the chloride anions situated on twofold axes, while the cation is on a general position. All conventional hydrogen-bond donors and acceptors are utilized, forming a hydrogen-bonded ladder motif along the  $c$  axis. Investigation of the torsion angles between aromatic systems and isopropyl groups reveals unusual geometrical features. One isopropyl group exhibits an expected eclipsed conformation with respect to the aromatic ring. The other isopropyl group shows a slight twist with respect to the aromatic ring. The short  $\text{Cl}\cdots\text{HC}$  (methine) contact (2.88 Å) observed in the asymmetric unit is the probable reason for the twist feature around the isopropyl area.

## Related literature

For the structure of the tetrahydrofuran solvate of the title salt, see: Bond & Doyle (2003).



## Experimental

## Crystal data

$\text{C}_{12}\text{H}_{20}\text{N}^+\cdot\text{Cl}^-$   
 $M_r = 213.74$   
 Orthorhombic,  $Pbcn$   
 $a = 13.0390$  (3) Å  
 $b = 21.0436$  (4) Å  
 $c = 8.9968$  (2) Å

$V = 2468.61$  (9) Å<sup>3</sup>  
 $Z = 8$   
 Cu  $K\alpha$  radiation  
 $\mu = 2.43$  mm<sup>-1</sup>  
 $T = 173$  (2) K  
 $0.36 \times 0.23 \times 0.21$  mm

## Data collection

Bruker SMART APEXII CCD diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 2002)  
 $T_{\min} = 0.451$ ,  $T_{\max} = 0.597$

23541 measured reflections  
 2343 independent reflections  
 2248 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.028$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$   
 $wR(F^2) = 0.112$   
 $S = 1.00$   
 2343 reflections  
 144 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.35$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.21$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N13}-\text{H13X}\cdots\text{Cl1X}^1$	0.90 (2)	2.54 (2)	3.3777 (12)	154.7 (15)
$\text{N13}-\text{H13Y}\cdots\text{Cl1Y}$	0.93 (2)	2.16 (2)	3.0753 (12)	167.2 (16)
$\text{N13}-\text{H13Z}\cdots\text{Cl1X}$	0.921 (19)	2.352 (19)	3.2493 (12)	164.8 (14)

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

Data collection: SMART (Bruker, 2006); cell refinement: SAINT (Bruker, 2006); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

The authors acknowledge Jon Bordner for his crystallographic mentoring and support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2172).

## References

- Bond, A. D. & Doyle, E. L. (2003). *Chem. Commun.* pp. 2324–2325.  
 Bruker (2006). SMART for WNT/2000 and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Sheldrick, G. M. (2002). SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

## supporting information

*Acta Cryst.* (2008). E64, o1480 [doi:10.1107/S160053680801790X]

## 2,6-Diisopropylanilinium chloride

Ivan Samardjiev and Brian Samas

### S1. Comment

2,6-DIPA chloride is frequently used as a starting material for pharmaceutical synthesis. Hydrochloric acid is a desirable acid for salt formation and during our efforts to purify DIPA by salt formation and crystallization, we formed the title 1:1 salt (Fig. 1). Chlorine anions have an interesting 50:50 occupancy positions, sitting on 2-fold axis with  $x,y,z$  coordinates as follows: C11X (0,  $y$ , 1/4) and C11Y (0,  $y$ , 3/4).

The structure has all conventional hydrogen-bond donors used. In the crystal structure, four of the protonated  $\text{NH}_3$  groups face the counter-ions sitting on above mentioned special positions, forming two-dimensional sheets parallel to the (010) planes (Fig. 2). Hydrogen-bond network confirms the following interactions between 50% populated  $\text{Cl}^-$  anions and polar ends of  $\text{DIPAH}^+$  ( $\text{NH}_3^+$  groups): C11X participates a hydrogen-bonding interaction with H13X (2.54 Å separation), H13Z (2.35 Å), and H13Y (2.16 Å) are hydrogen-bonded to C11X and C11Y, respectively. There is an additional short contact within van der Waals radii between C11X and H7 (2.88 Å) yet this interaction does not occur with the C11Y occupancy.

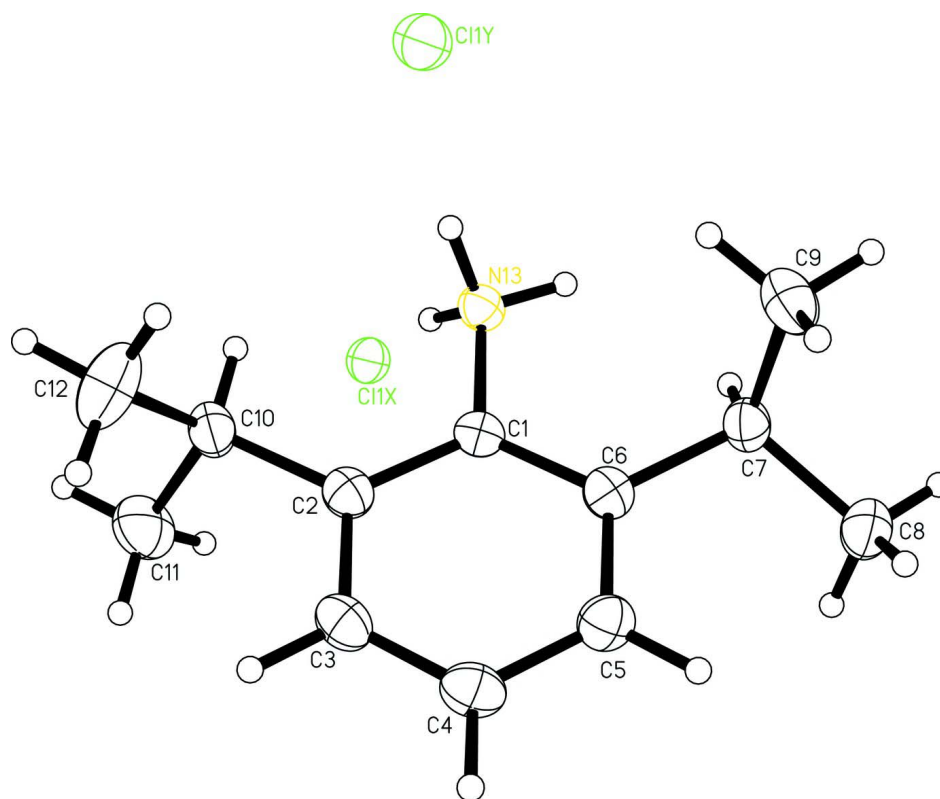
The torsion angles from the aromatic group to the isopropyl groups are interesting. The expectation is that the isopropyl groups would eclipse the aromatic ring plane so that the C7—H7 and C10—H10 bonds lie in the same plane as atoms N13, C1, C6 and C7. One of these groups exhibits such a conformation (C1—C2—C10—H10 =  $-0.21^\circ$ ), while the other shows a slight twist (C1—C6—C7—H7 =  $-34.62^\circ$ ). The above mentioned C11X $\cdots$ H7 short contact is the probable reason for the obvious twist event around C1—C6—C7—H7 area. Similar events are observed in a related DIPA chloride salt structure, where only one of isopropyl hydrogen experiences van der Waals contacts with a  $\text{Cl}^-$  anion (Bond & Doyle, 2003).

### S2. Experimental

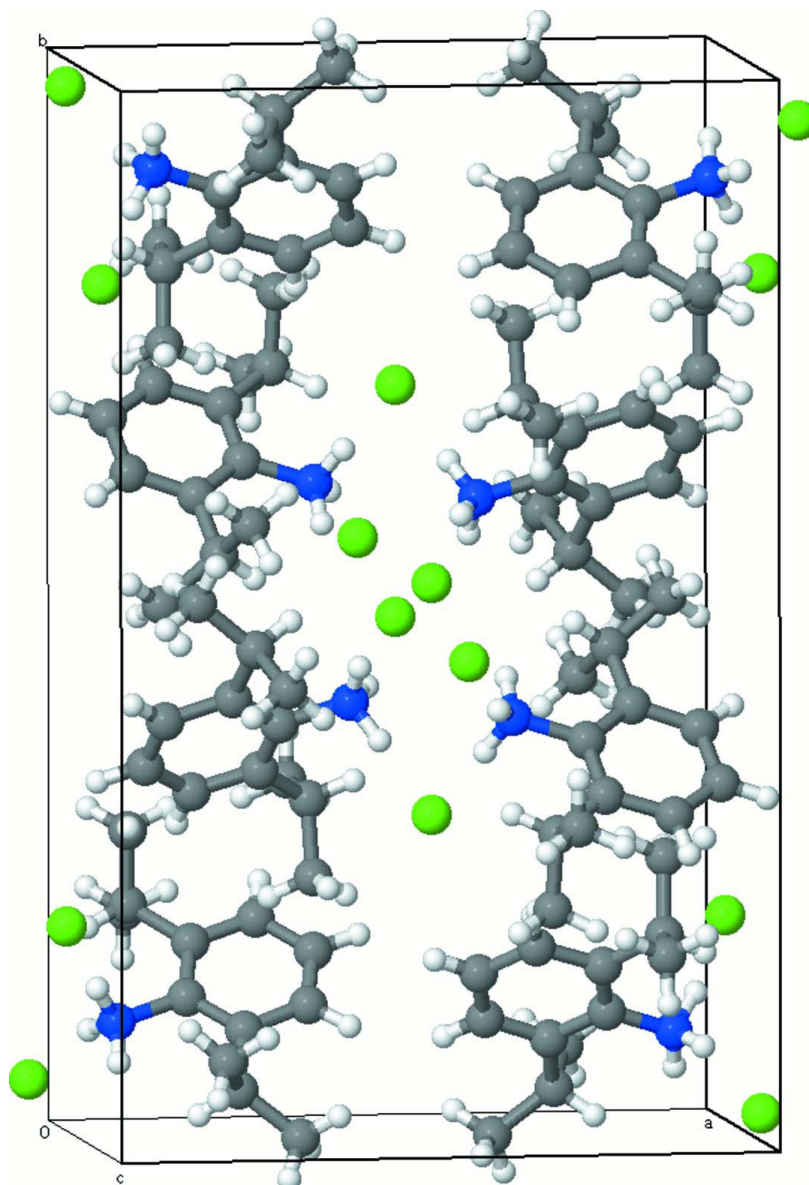
A stock solution of DIPA was made in 2-propanol (85 mg, 2 ml). To a crystallizer vessel, 0.43 ml of stock solution was added with 1 equivalent of concentrated hydrochloric acid. For salt formation participation we gradually added 6 ml of methyl *t*-butyl ether, then the sample was purged with dry nitrogen for evaporation until dryness, allowed to evaporate over 24 h mark. A crystal of the title salt was removed from the crystallizer vessel and mounted on a MiTeGen loop with Paratone-N oil.

### S3. Refinement

H atoms bonded to C atoms were placed in idealized positions and refined using a riding model with C—H = 0.93 Å for  $C_{sp^2}$ —H, 0.96 Å for  $\text{CH}_3$ , and 0.98 Å for CH.  $U_{\text{iso}}(\text{H})$  values were fixed to 0.08 Å<sup>2</sup>. H atoms bound to N3 were located in a difference maps and their positions and isotropic displacement parameters were refined freely.

**Figure 1**

View of the constituents of (I), showing the atom-labeling scheme and displacement ellipsoids drawn at the 30% probability level. H atoms are represented by circles of arbitrary size.



**Figure 2**

The crystal structure viewed along the [001] direction, showing four Cl<sup>-</sup> anions spaced between four DIPA<sup>+</sup> cations.

### 2,6-Diisopropylanilinium chloride

#### Crystal data

C<sub>12</sub>H<sub>20</sub>N<sup>+</sup>·Cl<sup>-</sup>

*M<sub>r</sub>* = 213.74

Orthorhombic, *Pbcn*

Hall symbol: -P 2n 2ab

*a* = 13.0390 (3) Å

*b* = 21.0436 (4) Å

*c* = 8.9968 (2) Å

*V* = 2468.61 (9) Å<sup>3</sup>

*Z* = 8

*F*(000) = 928

*D<sub>x</sub>* = 1.150 Mg m<sup>-3</sup>

Cu *Kα* radiation, λ = 1.54178 Å

Cell parameters from 9259 reflections

θ = 4.0–70.8°

μ = 2.43 mm<sup>-1</sup>

*T* = 173 K

Prism, colourless

0.36 × 0.23 × 0.21 mm

Data collection

Bruker SMART APEXII CCD  
diffractometer  
Radiation source: Rotating Anode  
Montel Multilayer Optics monochromator  
 $\varphi$  and  $\omega$  scans  
Absorption correction: multi-scan  
(SADABS; Sheldrick, 2002)  
 $T_{\min} = 0.451$ ,  $T_{\max} = 0.597$

23541 measured reflections  
2343 independent reflections  
2248 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.028$   
 $\theta_{\max} = 71.7^\circ$ ,  $\theta_{\min} = 4.0^\circ$   
 $h = -15 \rightarrow 15$   
 $k = -25 \rightarrow 20$   
 $l = -10 \rightarrow 9$

Refinement

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.037$   
 $wR(F^2) = 0.112$   
 $S = 1.00$   
2343 reflections  
144 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 atoms treated by a mixture of independent  
and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0758P)^2 + 1.0624P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.029$   
 $\Delta\rho_{\max} = 0.35 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.79245 (10)	0.88741 (6)	0.98360 (14)	0.0207 (3)
C2	0.77502 (10)	0.84000 (6)	1.08919 (15)	0.0238 (3)
C3	0.67345 (11)	0.82064 (6)	1.11109 (16)	0.0276 (3)
H3	0.6592	0.7891	1.1806	0.080*
C4	0.59411 (11)	0.84748 (7)	1.03134 (17)	0.0296 (3)
H4	0.5271	0.8341	1.0476	0.080*
C5	0.61414 (10)	0.89426 (7)	0.92708 (16)	0.0276 (3)
H5	0.5601	0.9119	0.8738	0.080*
C6	0.71389 (11)	0.91548 (6)	0.90040 (15)	0.0229 (3)
C7	0.73500 (11)	0.96575 (6)	0.78298 (16)	0.0250 (3)
H7	0.7931	0.9913	0.8179	0.080*
C8	0.64526 (13)	1.01091 (8)	0.75817 (17)	0.0356 (4)
H8A	0.5916	0.9891	0.7059	0.080*
H8B	0.6678	1.0467	0.7005	0.080*
H8C	0.6199	1.0254	0.8524	0.080*
C9	0.76786 (13)	0.93367 (8)	0.63763 (16)	0.0357 (4)
H9A	0.8242	0.9054	0.6568	0.080*
H9B	0.7887	0.9655	0.5674	0.080*
H9C	0.7113	0.9100	0.5977	0.080*
C10	0.85921 (11)	0.80846 (6)	1.17924 (16)	0.0276 (3)
H10	0.9249	0.8271	1.1493	0.080*
C11	0.84443 (14)	0.82083 (9)	1.34518 (18)	0.0418 (4)
H11A	0.8456	0.8658	1.3635	0.080*
H11B	0.8987	0.8008	1.3999	0.080*

H11C	0.7797	0.8037	1.3764	0.080*
C12	0.86270 (15)	0.73722 (8)	1.1448 (2)	0.0491 (5)
H12A	0.7992	0.7179	1.1742	0.080*
H12B	0.9182	0.7180	1.1985	0.080*
H12C	0.8729	0.7311	1.0401	0.080*
N13	0.89957 (8)	0.90795 (6)	0.95727 (13)	0.0218 (3)
H13X	0.9045 (14)	0.9424 (9)	0.899 (2)	0.034 (5)*
H13Y	0.9357 (15)	0.8761 (9)	0.908 (2)	0.036 (4)*
H13Z	0.9318 (14)	0.9187 (8)	1.045 (2)	0.030 (4)*
C11X	1.0000	0.97357 (2)	1.2500	0.02472 (17)
C11Y	1.0000	0.80980 (2)	0.7500	0.03006 (18)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0213 (6)	0.0208 (6)	0.0200 (6)	-0.0023 (4)	0.0011 (5)	-0.0029 (5)
C2	0.0270 (7)	0.0224 (6)	0.0221 (7)	-0.0008 (5)	0.0012 (5)	-0.0014 (5)
C3	0.0306 (7)	0.0243 (6)	0.0279 (7)	-0.0061 (5)	0.0033 (6)	0.0016 (5)
C4	0.0244 (7)	0.0315 (7)	0.0329 (8)	-0.0085 (5)	0.0015 (6)	-0.0022 (6)
C5	0.0238 (7)	0.0307 (7)	0.0282 (7)	-0.0025 (5)	-0.0039 (5)	-0.0015 (6)
C6	0.0255 (7)	0.0229 (6)	0.0204 (7)	-0.0018 (5)	-0.0013 (5)	-0.0023 (5)
C7	0.0247 (7)	0.0267 (7)	0.0237 (6)	-0.0012 (5)	-0.0028 (5)	0.0032 (5)
C8	0.0318 (8)	0.0357 (8)	0.0392 (9)	0.0041 (7)	-0.0018 (6)	0.0106 (6)
C9	0.0462 (9)	0.0374 (8)	0.0235 (7)	-0.0010 (7)	0.0013 (6)	0.0029 (6)
C10	0.0296 (7)	0.0275 (7)	0.0258 (7)	-0.0001 (5)	-0.0005 (6)	0.0059 (5)
C11	0.0436 (9)	0.0560 (10)	0.0259 (8)	-0.0009 (7)	-0.0025 (7)	0.0044 (7)
C12	0.0539 (11)	0.0319 (8)	0.0614 (12)	0.0133 (7)	-0.0163 (9)	-0.0029 (8)
N13	0.0217 (6)	0.0230 (6)	0.0208 (6)	-0.0015 (4)	0.0001 (4)	0.0008 (5)
C11X	0.0248 (3)	0.0249 (3)	0.0244 (3)	0.000	-0.00197 (15)	0.000
C11Y	0.0339 (3)	0.0247 (3)	0.0315 (3)	0.000	0.00491 (17)	0.000

*Geometric parameters (Å, °)*

C1—C2	1.3962 (18)	C8—H8C	0.9600
C1—C6	1.3994 (19)	C9—H9A	0.9600
C1—N13	1.4812 (16)	C9—H9B	0.9600
C2—C3	1.3996 (19)	C9—H9C	0.9600
C2—C10	1.5171 (19)	C10—C11	1.528 (2)
C3—C4	1.380 (2)	C10—C12	1.532 (2)
C3—H3	0.9300	C10—H10	0.9800
C4—C5	1.385 (2)	C11—H11A	0.9600
C4—H4	0.9300	C11—H11B	0.9600
C5—C6	1.3959 (19)	C11—H11C	0.9600
C5—H5	0.9300	C12—H12A	0.9600
C6—C7	1.5202 (19)	C12—H12B	0.9600
C7—C8	1.524 (2)	C12—H12C	0.9600
C7—C9	1.533 (2)	N13—H13X	0.90 (2)
C7—H7	0.9800	N13—H13Y	0.93 (2)

C8—H8A	0.9600	N13—H13Z	0.921 (19)
C8—H8B	0.9600		
C2—C1—C6	123.12 (12)	C7—C9—H9A	109.5
C2—C1—N13	118.08 (11)	C7—C9—H9B	109.5
C6—C1—N13	118.79 (11)	H9A—C9—H9B	109.5
C1—C2—C3	117.24 (12)	C7—C9—H9C	109.5
C1—C2—C10	123.93 (12)	H9A—C9—H9C	109.5
C3—C2—C10	118.83 (12)	H9B—C9—H9C	109.5
C4—C3—C2	121.14 (13)	C2—C10—C11	110.86 (12)
C4—C3—H3	119.4	C2—C10—C12	109.97 (12)
C2—C3—H3	119.4	C11—C10—C12	111.61 (14)
C3—C4—C5	120.12 (13)	C2—C10—H10	108.1
C3—C4—H4	119.9	C11—C10—H10	108.1
C5—C4—H4	119.9	C12—C10—H10	108.1
C4—C5—C6	121.31 (13)	C10—C11—H11A	109.5
C4—C5—H5	119.3	C10—C11—H11B	109.5
C6—C5—H5	119.3	H11A—C11—H11B	109.5
C5—C6—C1	117.07 (12)	C10—C11—H11C	109.5
C5—C6—C7	120.72 (12)	H11A—C11—H11C	109.5
C1—C6—C7	122.20 (12)	H11B—C11—H11C	109.5
C6—C7—C8	113.37 (12)	C10—C12—H12A	109.5
C6—C7—C9	109.69 (11)	C10—C12—H12B	109.5
C8—C7—C9	111.36 (12)	H12A—C12—H12B	109.5
C6—C7—H7	107.4	C10—C12—H12C	109.5
C8—C7—H7	107.4	H12A—C12—H12C	109.5
C9—C7—H7	107.4	H12B—C12—H12C	109.5
C7—C8—H8A	109.5	C1—N13—H13X	113.4 (11)
C7—C8—H8B	109.5	C1—N13—H13Y	110.0 (12)
H8A—C8—H8B	109.5	H13X—N13—H13Y	105.1 (16)
C7—C8—H8C	109.5	C1—N13—H13Z	111.5 (11)
H8A—C8—H8C	109.5	H13X—N13—H13Z	105.7 (15)
H8B—C8—H8C	109.5	H13Y—N13—H13Z	110.9 (16)
C6—C1—C2—C3	-0.23 (19)	N13—C1—C6—C5	179.19 (11)
N13—C1—C2—C3	-179.18 (11)	C2—C1—C6—C7	-178.28 (12)
C6—C1—C2—C10	179.28 (12)	N13—C1—C6—C7	0.66 (18)
N13—C1—C2—C10	0.33 (19)	C5—C6—C7—C8	28.51 (18)
C1—C2—C3—C4	0.0 (2)	C1—C6—C7—C8	-153.01 (13)
C10—C2—C3—C4	-179.55 (13)	C5—C6—C7—C9	-96.67 (15)
C2—C3—C4—C5	0.2 (2)	C1—C6—C7—C9	81.80 (16)
C3—C4—C5—C6	-0.2 (2)	C1—C2—C10—C11	118.08 (15)
C4—C5—C6—C1	0.0 (2)	C3—C2—C10—C11	-62.42 (16)
C4—C5—C6—C7	178.53 (13)	C1—C2—C10—C12	-118.01 (16)
C2—C1—C6—C5	0.25 (19)	C3—C2—C10—C12	61.49 (18)

*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>
N13—H13X···C11X <sup>i</sup>	0.90 (2)	2.54 (2)	3.3777 (12)	154.7 (15)
N13—H13Y···C11Y	0.93 (2)	2.16 (2)	3.0753 (12)	167.2 (16)
N13—H13Z···C11X	0.921 (19)	2.352 (19)	3.2493 (12)	164.8 (14)

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