

2,4,6-Trichloro-1,3,5-trimethylborazine

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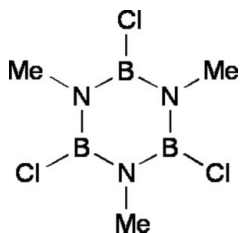
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{N}-\text{B}) = 0.004$ Å; R factor = 0.061; wR factor = 0.173; data-to-parameter ratio = 24.7.

The title compound, $(\text{CH}_3\text{NBCl})_3$ or $\text{C}_3\text{H}_9\text{B}_3\text{Cl}_3\text{N}_3$, is the first crystallographically characterized trialkyltrichloroborazine derivative. It crystallizes with two independent molecules in the asymmetric unit. The B_3N_3 rings are essentially planar, with B–N distances ranging from 1.428 (5) to 1.449 (4) Å, and with B–N–B and N–B–N angles in the ranges 118.4 (3)–119.4 (3) and 120.7 (3)–121.8 (3)°, respectively. The two independent molecules are staggered parallel along $[x, 0, 0]$ and $[x, \frac{1}{2}, \frac{1}{2}]$, most probably due to the formation of weak intermolecular C–H...Cl hydrogen bonds.

Related literature

The title compound was first obtained by Burg & Kuljian (1950) and Nöth & Sprague (1970) *via* different reaction routes. For the synthesis of the borazine precursor compound dichloroborylmethyltrichlorosilylamine used in the present study, see: Kroschel (2001); Kroschel & Jansen (2002); Weinmann *et al.* (2007). For structure determinations of the related compounds $(\text{HNBCl})_3$, $(\text{Ph}_3\text{NBCl})_3$, $[\text{CH}_3\text{NB}(\text{NMe}_2)]_3$ and $(\text{ClNBCl})_3$, see: Coursen & Hoard (1952); Schwarz *et al.* (1977); Rodriguez & Borek (2006); Haasnoot *et al.* (1972), respectively.



Experimental

Crystal data

$\text{C}_3\text{H}_9\text{B}_3\text{Cl}_3\text{N}_3$
 $M_r = 225.91$
 Triclinic, $P\bar{1}$
 $a = 7.720$ (4) Å
 $b = 9.167$ (5) Å

$c = 15.054$ (8) Å
 $\alpha = 90.446$ (12)°
 $\beta = 91.727$ (12)°
 $\gamma = 114.380$ (10)°
 $V = 969.7$ (9) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.89$ mm⁻¹

$T = 100$ (2) K
 $0.50 \times 0.20 \times 0.05$ mm

Data collection

Bruker SMART APEX CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2007)
 $T_{\text{min}} = 0.665$, $T_{\text{max}} = 0.957$

9812 measured reflections
 5513 independent reflections
 3775 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.060$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.173$
 $S = 1.02$
 5513 reflections

223 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.89$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.59$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C13–H13A...Cl12 ⁱ	0.98	3.04	3.846 (4)	140
C21–H21C...Cl13 ⁱⁱ	0.98	2.97	3.938 (4)	170
C22–H22C...Cl13 ⁱⁱⁱ	0.98	2.99	3.778 (4)	138
C21–H21A...Cl23 ^{iv}	0.98	3.01	3.942 (4)	160
C22–H22B...Cl21 ^{iv}	0.98	3.03	3.783 (4)	134
C23–H23C...Cl22 ^{iv}	0.98	3.09	4.018 (5)	158

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+1, -y+1, -z+2$; (iii) $x-1, y-1, z$; (iv) $-x+1, -y, -z+2$.

Data collection: SMART (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ATOMS (Dowty, 2005); software used to prepare material for publication: enCIFer (Allen *et al.*, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2148).

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supplementary materials

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2,4,6-Trichloro-1,3,5-trimethylborazine

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Comment

We recently reported on the first approach towards the continuous synthesis of molecular precursors of high-temperature Si/C/B/N ceramics. $\text{Cl}_3\text{SiN}(\text{Me})\text{BCl}_2$ (DMTA, dichloroborylmethyltrichlorosilylamine) was obtained in a straightforward reaction from silicon tetrachloride, methylamine, and boron trichloride (Kroschel, 2001; Kroschel & Jansen, 2002; Weinmann *et al.*, 2007). DMTA is thermally unstable. At elevated temperature it decomposes by SiCl_4 elimination to yield *B,B',B''*-trichloro-*N,N',N''*-trimethylborazine, $(\text{CH}_3\text{NBCl})_3$ (Scheme 2).

The title compound was first obtained by Burg & Kuljian (1950) by chance from equal gas volumes of $\text{MeN}(\text{SiH}_3)_2$ and BCl_3 . Initially, $\text{Me}(\text{SiH}_3)\text{NBCl}_2$ formed at 195 K but on warming to room temperature it lost ClSiH_3 and quantitatively converted into $(\text{CH}_3\text{NBCl})_3$. Similarly, $(\text{CH}_3\text{NBCl})_3$ was obtained by Nöth & Sprague (1970) from $\text{MeN}(\text{SiMe}_3)_2$ and BCl_3 . The N atom in DMTA is clearly less basic than those in $\text{Me}(\text{SiH}_3)\text{NBCl}_2$ or $\text{Me}(\text{SiMe}_3)\text{NBCl}_2$. Therefore, decomposition (*i.e.* SiCl_4 elimination) proceeds slower and requires higher temperatures. On the other hand the retarded degradation results in the formation of single crystals of $(\text{CH}_3\text{NBCl})_3$ which accumulate as colorless needles.

Figure 1 shows the structures of the two independent molecules which are nearly identical. B, N, C, and Cl atoms are in a strictly planar arrangement with B and N atoms spanning an almost perfect hexagon. In average, the B—N distances measure 1.437 and 1.435 Å; the maximum deviations from these values are 0.012 and 0.008 Å, respectively. The B—N bonds are thus only slightly longer than those found in $(\text{HNBCl})_3$ (1.413 Å; Coursen & Hoard, 1952) and $(\text{Ph}_3\text{NBCl})_3$ (1.428 Å; Schwarz *et al.*, 1977). In contrast, there is neither evidence for distortion such as in $(\text{CH}_3\text{NB}(\text{NMe}_2))_3$ (Rodríguez & Borek, 2006) nor for the existence of "long" and "short" B—N bonds as reported for $(\text{CINBCl})_3$ (1.398 *versus* 1.451 Å; Haasnoot *et al.*, 1972), indicating a perfect π -delocalization of the N electron lone pairs. This is reflected by the B—N—B and N—B—N bond angles which approach 120°. However, the former (average 118.8 and 118.9°) are slightly smaller than the latter (121.1°). B—Cl and N—C bond lengths are similar to those reported in the literature for other *B*-chloro and *N*-methyl borazine derivatives. From Figure 2 it is evident that molecules 1 and 2 are staggered parallel along $[a, 0, 0]$ and $[a, 1/2, 1/2]$, respectively. $\text{H}\cdots\text{Cl}$ separations of *ca* 3 Å ($\text{H}\cdots\text{Cl}$ distances are thus within the sum of the Van-der-Vaals radii of hydrogen and chlorine) indicate that weak $\text{H}\cdots\text{Cl}$ hydrogen bridges enforce the special arrangement (see Table).

Experimental

DMTA was synthesized by a continuous gas phase procedure starting from SiCl_4 and MeNH_2 . As-obtained Cl_3SiNHMe was directly reacted with BCl_3 . Solid amine hydrochloride byproducts were removed by filtration through inductively heated ceramic filters. Details of the experimental setup are found elsewhere (Weinmann *et al.*, 2007). Raw DMTA was purified by fractional distillation. Subsequent heating to 323–343 K over 4–6 weeks resulted in partial decomposition by SiCl_4 elimination and formation of crystalline $(\text{CH}_3\text{NBCl})_3$.

Refinement

H atoms were placed geometrically and were refined with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ of the attached carbon atom.

Figures

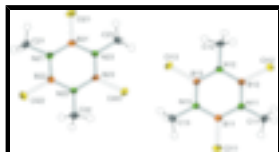


Fig. 1. Structure of the two independent $(\text{CH}_3\text{NBCl})_3$ molecules, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

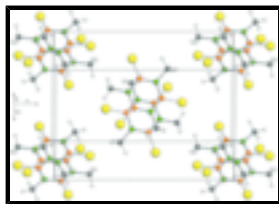


Fig. 2. Packing diagram of $(\text{MeNBCl})_3$.



Fig. 3. The formation of the title compound.

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Crystal data

$\text{C}_3\text{H}_9\text{B}_3\text{Cl}_3\text{N}_3$

$M_r = 225.91$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.720$ (4) Å

$b = 9.167$ (5) Å

$c = 15.054$ (8) Å

$\alpha = 90.446$ (12)°

$\beta = 91.727$ (12)°

$\gamma = 114.380$ (10)°

$V = 969.7$ (9) Å³

$Z = 4$

$F_{000} = 456$

$D_x = 1.547$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 4305 reflections

$\theta = 2.4\text{--}30.9^\circ$

$\mu = 0.89$ mm⁻¹

$T = 100$ (2) K

Plate, colourless

$0.50 \times 0.20 \times 0.05$ mm

Data collection

Bruker SMART APEX CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 100$ (2) K

ω scans

5513 independent reflections

3775 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.060$

$\theta_{\text{max}} = 30.0^\circ$

$\theta_{\text{min}} = 1.4^\circ$

Absorption correction: multi-scan
(SADABS; Bruker, 2007) $h = -10 \rightarrow 10$
 $T_{\min} = 0.665$, $T_{\max} = 0.957$ $k = -12 \rightarrow 12$
 9812 measured reflections $l = -20 \rightarrow 21$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.061$	H-atom parameters constrained
$wR(F^2) = 0.173$	$w = 1/[\sigma^2(F_o^2) + (0.0958P)^2 + 0.0723P]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
5513 reflections	$(\Delta/\sigma)_{\max} = 0.001$
223 parameters	$\Delta\rho_{\max} = 0.89 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.58 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger. H atoms were placed geometrically applying restraints of isotropic displacement parameters 1.5 times of the attached carbon atom.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl11	0.73512 (13)	0.15475 (10)	0.43707 (5)	0.0267 (2)
Cl12	0.82179 (13)	0.78156 (10)	0.37427 (5)	0.0277 (2)
Cl13	0.68452 (13)	0.53493 (10)	0.71585 (5)	0.0266 (2)
N11	0.7664 (4)	0.4683 (3)	0.41615 (16)	0.0196 (5)
N12	0.7499 (4)	0.6409 (3)	0.54139 (16)	0.0202 (5)
N13	0.7120 (4)	0.3594 (3)	0.56975 (16)	0.0194 (5)
B11	0.7372 (5)	0.3398 (4)	0.4775 (2)	0.0189 (6)
B12	0.7754 (5)	0.6179 (4)	0.4491 (2)	0.0195 (6)
B13	0.7169 (5)	0.5096 (4)	0.6006 (2)	0.0197 (6)
C11	0.7896 (6)	0.4449 (4)	0.32036 (19)	0.0260 (7)
H11A	0.9217	0.4625	0.3106	0.039*
H11B	0.7581	0.5213	0.2857	0.039*
H11C	0.7044	0.3354	0.3015	0.039*

supplementary materials

C12	0.7621 (6)	0.7972 (4)	0.5763 (2)	0.0269 (7)
H12A	0.7591	0.8645	0.5265	0.040*
H12B	0.8813	0.8511	0.6114	0.040*
H12C	0.6541	0.7789	0.6140	0.040*
C13	0.6809 (5)	0.2273 (4)	0.6338 (2)	0.0256 (7)
H13A	0.5563	0.1959	0.6601	0.038*
H13B	0.7808	0.2648	0.6809	0.038*
H13C	0.6852	0.1350	0.6023	0.038*
Cl21	0.56506 (13)	0.33268 (10)	1.08219 (5)	0.0285 (2)
Cl22	0.00324 (13)	-0.28065 (10)	1.13894 (5)	0.02597 (19)
Cl23	0.17428 (14)	-0.06954 (11)	0.79348 (5)	0.0287 (2)
N21	0.2749 (4)	0.0253 (3)	1.09944 (15)	0.0195 (5)
N22	0.1009 (4)	-0.1550 (3)	0.97015 (16)	0.0199 (5)
N23	0.3520 (4)	0.1197 (3)	0.94460 (16)	0.0208 (5)
B21	0.3843 (5)	0.1462 (4)	1.0388 (2)	0.0196 (6)
B22	0.1358 (5)	-0.1254 (4)	1.0643 (2)	0.0183 (6)
B23	0.2113 (6)	-0.0326 (4)	0.9112 (2)	0.0211 (7)
C21	0.3086 (5)	0.0565 (4)	1.19702 (19)	0.0255 (7)
H21A	0.4276	0.0493	1.2155	0.038*
H21B	0.2028	-0.0232	1.2284	0.038*
H21C	0.3177	0.1640	1.2114	0.038*
C22	-0.0423 (5)	-0.3115 (4)	0.9340 (2)	0.0264 (7)
H22A	-0.1314	-0.3666	0.9802	0.040*
H22B	0.0226	-0.3778	0.9149	0.040*
H22C	-0.1123	-0.2930	0.8831	0.040*
C23	0.4641 (5)	0.2474 (4)	0.8818 (2)	0.0272 (7)
H23A	0.5060	0.3526	0.9113	0.041*
H23B	0.3841	0.2427	0.8291	0.041*
H23C	0.5754	0.2303	0.8639	0.041*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl11	0.0341 (5)	0.0167 (4)	0.0302 (4)	0.0115 (3)	0.0014 (3)	-0.0038 (3)
Cl12	0.0372 (5)	0.0175 (4)	0.0258 (4)	0.0086 (3)	0.0025 (3)	0.0054 (3)
Cl13	0.0358 (5)	0.0223 (4)	0.0191 (3)	0.0093 (4)	0.0019 (3)	-0.0017 (3)
N11	0.0208 (14)	0.0157 (13)	0.0197 (11)	0.0049 (11)	0.0006 (9)	-0.0004 (9)
N12	0.0253 (15)	0.0128 (12)	0.0214 (11)	0.0068 (11)	-0.0011 (9)	-0.0033 (9)
N13	0.0200 (14)	0.0157 (13)	0.0209 (11)	0.0058 (11)	-0.0008 (9)	0.0011 (9)
B11	0.0184 (17)	0.0146 (16)	0.0228 (15)	0.0060 (13)	-0.0003 (11)	-0.0018 (12)
B12	0.0191 (17)	0.0178 (16)	0.0201 (14)	0.0061 (14)	0.0002 (11)	0.0018 (12)
B13	0.0204 (18)	0.0160 (16)	0.0208 (14)	0.0059 (14)	-0.0018 (11)	-0.0012 (11)
C11	0.033 (2)	0.0240 (17)	0.0197 (13)	0.0105 (15)	0.0014 (12)	-0.0019 (11)
C12	0.031 (2)	0.0136 (16)	0.0336 (16)	0.0066 (14)	0.0007 (13)	-0.0059 (12)
C13	0.0322 (19)	0.0210 (17)	0.0236 (14)	0.0110 (15)	0.0011 (12)	0.0055 (12)
Cl21	0.0276 (5)	0.0161 (4)	0.0356 (4)	0.0031 (3)	-0.0027 (3)	-0.0027 (3)
Cl22	0.0296 (5)	0.0193 (4)	0.0248 (3)	0.0056 (3)	0.0044 (3)	0.0049 (3)
Cl23	0.0404 (5)	0.0282 (4)	0.0186 (3)	0.0153 (4)	0.0003 (3)	-0.0011 (3)

N21	0.0236 (15)	0.0160 (13)	0.0185 (11)	0.0080 (11)	-0.0014 (9)	-0.0013 (9)
N22	0.0229 (15)	0.0140 (13)	0.0212 (11)	0.0063 (11)	-0.0031 (9)	-0.0025 (9)
N23	0.0239 (15)	0.0158 (13)	0.0227 (12)	0.0081 (11)	0.0040 (10)	0.0024 (9)
B21	0.0184 (18)	0.0125 (16)	0.0269 (16)	0.0058 (14)	-0.0009 (12)	-0.0013 (12)
B22	0.0216 (18)	0.0112 (15)	0.0237 (15)	0.0084 (14)	0.0018 (12)	0.0009 (11)
B23	0.0254 (19)	0.0194 (17)	0.0195 (14)	0.0103 (15)	0.0009 (12)	-0.0010 (12)
C21	0.035 (2)	0.0230 (17)	0.0175 (13)	0.0106 (15)	-0.0006 (12)	-0.0017 (11)
C22	0.031 (2)	0.0170 (16)	0.0275 (15)	0.0065 (15)	-0.0030 (13)	-0.0045 (12)
C23	0.032 (2)	0.0180 (16)	0.0299 (16)	0.0085 (15)	0.0066 (13)	0.0049 (12)

Geometric parameters (Å, °)

Cl11—B11	1.791 (4)	Cl21—B21	1.804 (4)
Cl12—B12	1.804 (3)	Cl22—B22	1.796 (3)
Cl13—B13	1.787 (3)	Cl23—B23	1.793 (4)
N11—B12	1.428 (5)	N21—B21	1.437 (4)
N11—B11	1.449 (4)	N21—B22	1.440 (5)
N11—C11	1.483 (4)	N21—C21	1.489 (4)
N12—B12	1.436 (4)	N22—B23	1.430 (4)
N12—B13	1.445 (4)	N22—B22	1.437 (4)
N12—C12	1.487 (4)	N22—C22	1.489 (4)
N13—B11	1.429 (4)	N23—B21	1.434 (4)
N13—B13	1.435 (4)	N23—B23	1.444 (5)
N13—C13	1.498 (4)	N23—C23	1.498 (4)
C11—H11A	0.9800	C21—H21A	0.9800
C11—H11B	0.9800	C21—H21B	0.9800
C11—H11C	0.9800	C21—H21C	0.9800
C12—H12A	0.9800	C22—H22A	0.9800
C12—H12B	0.9800	C22—H22B	0.9800
C12—H12C	0.9800	C22—H22C	0.9800
C13—H13A	0.9800	C23—H23A	0.9800
C13—H13B	0.9800	C23—H23B	0.9800
C13—H13C	0.9800	C23—H23C	0.9800
B12—N11—B11	119.4 (3)	B21—N21—B22	119.0 (3)
B12—N11—C11	120.5 (2)	B21—N21—C21	119.8 (3)
B11—N11—C11	120.1 (3)	B22—N21—C21	121.1 (3)
B12—N12—B13	118.4 (3)	B23—N22—B22	118.6 (3)
B12—N12—C12	121.2 (3)	B23—N22—C22	120.2 (3)
B13—N12—C12	120.4 (3)	B22—N22—C22	121.1 (3)
B11—N13—B13	118.5 (3)	B21—N23—B23	119.0 (3)
B11—N13—C13	121.5 (3)	B21—N23—C23	120.5 (3)
B13—N13—C13	120.0 (3)	B23—N23—C23	120.5 (3)
N13—B11—N11	120.9 (3)	N23—B21—N21	120.7 (3)
N13—B11—Cl11	119.6 (2)	N23—B21—Cl21	119.9 (2)
N11—B11—Cl11	119.5 (2)	N21—B21—Cl21	119.4 (2)
N11—B12—N12	120.9 (3)	N22—B22—N21	121.2 (3)
N11—B12—Cl12	119.8 (2)	N22—B22—Cl22	119.0 (3)
N12—B12—Cl12	119.2 (3)	N21—B22—Cl22	119.7 (2)
N13—B13—N12	121.8 (3)	N22—B23—N23	121.3 (3)

supplementary materials

N13—B13—C113	118.8 (2)	N22—B23—C123	119.2 (3)
N12—B13—C113	119.3 (2)	N23—B23—C123	119.5 (2)
N11—C11—H11A	109.5	N21—C21—H21A	109.5
N11—C11—H11B	109.5	N21—C21—H21B	109.5
H11A—C11—H11B	109.5	H21A—C21—H21B	109.5
N11—C11—H11C	109.5	N21—C21—H21C	109.5
H11A—C11—H11C	109.5	H21A—C21—H21C	109.5
H11B—C11—H11C	109.5	H21B—C21—H21C	109.5
N12—C12—H12A	109.5	N22—C22—H22A	109.5
N12—C12—H12B	109.5	N22—C22—H22B	109.5
H12A—C12—H12B	109.5	H22A—C22—H22B	109.5
N12—C12—H12C	109.5	N22—C22—H22C	109.5
H12A—C12—H12C	109.5	H22A—C22—H22C	109.5
H12B—C12—H12C	109.5	H22B—C22—H22C	109.5
N13—C13—H13A	109.5	N23—C23—H23A	109.5
N13—C13—H13B	109.5	N23—C23—H23B	109.5
H13A—C13—H13B	109.5	H23A—C23—H23B	109.5
N13—C13—H13C	109.5	N23—C23—H23C	109.5
H13A—C13—H13C	109.5	H23A—C23—H23C	109.5
H13B—C13—H13C	109.5	H23B—C23—H23C	109.5

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C13—H13A \cdots C112 ⁱ	0.98	3.04	3.846 (4)	140
C21—H21C \cdots C113 ⁱⁱ	0.98	2.97	3.938 (4)	170
C22—H22C \cdots C113 ⁱⁱⁱ	0.98	2.99	3.778 (4)	138
C21—H21A \cdots C123 ^{iv}	0.98	3.01	3.942 (4)	160
C22—H22B \cdots C121 ^{iv}	0.98	3.03	3.783 (4)	134
C23—H23C \cdots C122 ^{iv}	0.98	3.09	4.018 (5)	158

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+1, -y+1, -z+2$; (iii) $x-1, y-1, z$; (iv) $-x+1, -y, -z+2$.

Fig. 1

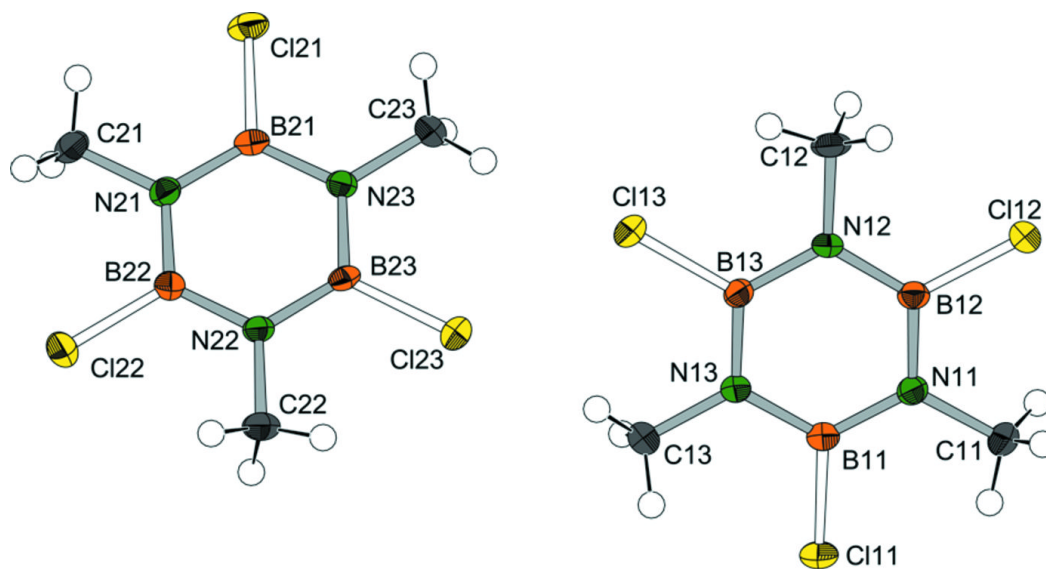


Fig. 2

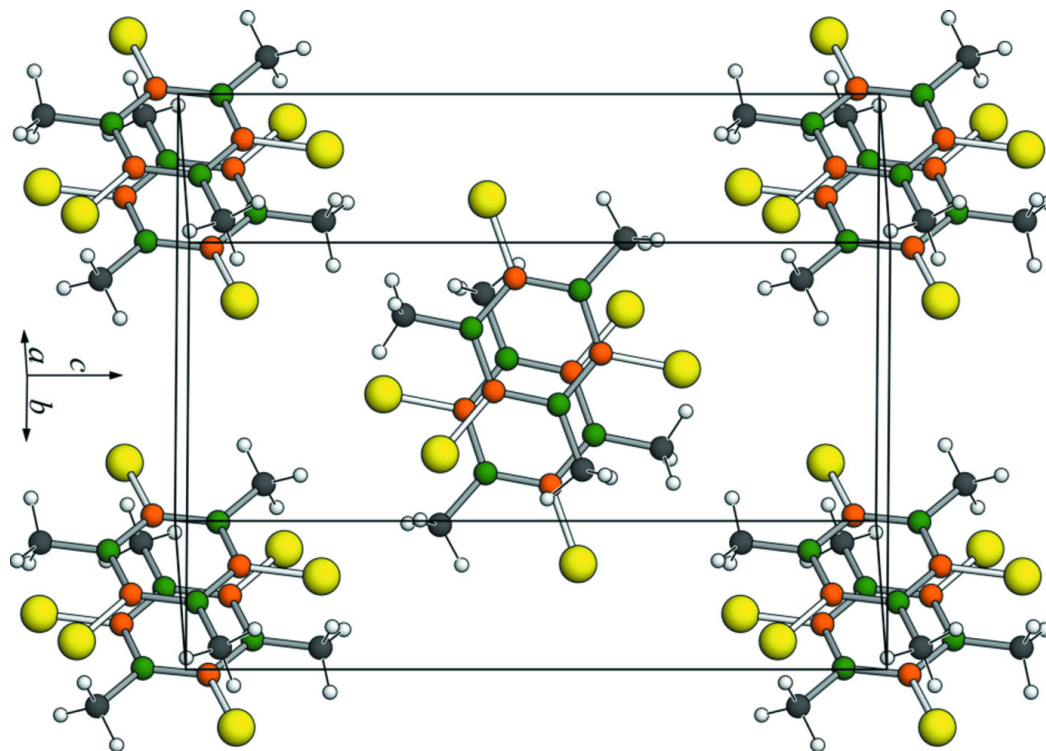


Fig. 3

