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1,3-Dichloro-5-(dimethylamino)borazine

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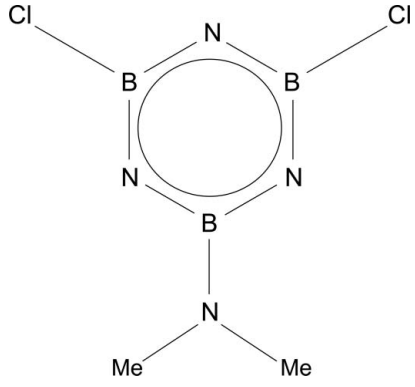
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(N–C) = 0.003 Å; R factor = 0.045; wR factor = 0.118; data-to-parameter ratio = 18.1.

In the crystal structure of the title compound, $C_2H_9B_3Cl_2N_4$, the asymmetric unit consists of two independent molecules. Both molecules show a planar conformation and are essentially identical in shape and size, differing mainly in their packing behavior.

Related literature

The planar nature of the title compound, with the Cl atoms, the borazine ring, and the amine group all lying in the same plane, is a common conformation. Both *B*-trichloroborazine (Coursen & Hoard, 1952) and *B*-tris(dimethylamino)borazine (Hess & Reiser, 1971) display planar molecular structures.

For related literature, see: Beachley & Durkin (1974).



Experimental

Crystal data

$C_2H_9B_3Cl_2N_4$
M_r = 192.46
Triclinic, $P\overline{1}$
a = 7.851 (3) Å
b = 8.215 (3) Å
c = 14.931 (5) Å
α = 74.986 (7)°
β = 77.000 (6)°
γ = 88.773 (6)°
V = 905.5 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.66 mm^{-1}
T = 173 (2) K
0.21 × 0.18 × 0.13 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)
T_{min} = 0.874, T_{max} = 0.921
7264 measured reflections
3675 independent reflections
2965 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.118
S = 1.05
3675 reflections
203 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 2001); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *XSHELL* (Bruker, 2000); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2128).

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supplementary materials

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1,3-Dichloro-5-(dimethylamino)borazine

M. A. Rodriguez and T. T. Borek

Comment

(dimethylamino)dichloroborazine (I) is a solid white material with a melting temperature of 92–92.5°C as first reported by Beachley and Durkin (1974). Figure 1 shows the molecular structure of (I) as a displacement ellipsoid plot. The structure contains two independent molecules, labeled mol-A and mol-B for clarification purposes. The two independent molecules in the asymmetric unit are essentially the same in appearance and connectivity. All bond lengths for the dimetylamine ligands, B—N bonds, and B—Cl bonds are consistent with expected values.

The main difference between the two molecules is revealed upon viewing the packing arrangement. The entire structure can be considered as an interweaving of two alternating layers of (I). Figure 2 and 3 give a perspective on these two layers as viewed down the *c* axis of the cell. Figure 2 shows the A-layer (generated from mol-A molecules) in relation to the unit cell. We see the A-layer (which propagates parallel to the a-b plane of the unit cell) replicating by translation in the *a* axis, but demonstrating inversion along the *b* axis. In figure 3, the B-layer (generated from mol-B molecules) shows inversion along the *a* axis and translation along the *b* axis. The inversion of (I) within a layer aids packing by increasing separation of the Cl atoms from each other within their respective layer. It is also worth noting that there is a significant tilt of the molecules as the layers alternate. In comparing figure 2 and figure 3, we see that the tilt of the planar molecules mol-A and mol-B (relative to the b-c plane of the unit cell) show that mol-A and mol-B are rotated by almost 90 degrees to each other. This packing arrangement appears beneficial as it accommodates the coordination of Cl atoms of the A-layer with the amine groups of mol-B residing in the B-layer. Likewise, the Cl atoms of mol-A coordinate to mol-B amine groups in similar fashion.

Experimental

Compound (I) was obtained using the published synthetic procedure of Beachley and Durkin (1974), which reacts one equivalent of *B*-trichloroborazine with 2 equivalents of anhydrous dimethylamine in anhydrous diethyl ether at -78°C. After the reaction warmed to room temperature, the crude mixture was filtered to remove precipitated dimethylammonium chloride, and the solvent was removed using vacuum techniques. This product was then recrystallized from anhydrous hexane, and vacuum sublimed. The resulting white solid had a melting point of 100 to 102°C (lit 92°C) and product purity was determined by nuclear magnetic resonance (1*H*, 11B, 13 C).

Figures

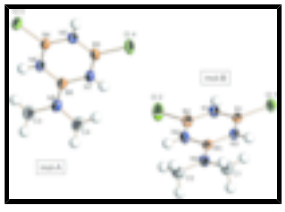

Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

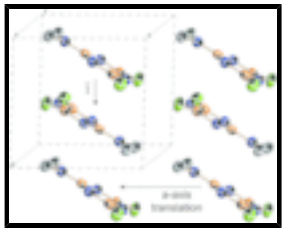

Fig. 2. A-layer packing of (I) as observed down *c* axis of unit cell. See text for details.

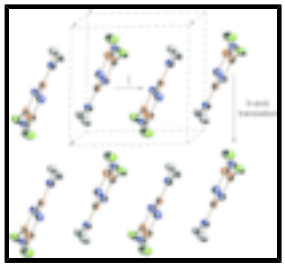

Fig. 3. B-layer packing of (I) as observed down *c* axis of unit cell. See text for details.

1,3-Dichloro-5-(dimethylamino)borazine

Crystal data

$C_2H_9B_3Cl_2N_4$	$Z = 4$
$M_r = 192.46$	$F_{000} = 392$
Triclinic, $P\overline{1}$	$D_x = 1.412$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 373 K
$a = 7.851$ (3) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 8.215$ (3) Å	Cell parameters from 100 reflections
$c = 14.931$ (5) Å	$\theta = 1.5$–26.5°
$\alpha = 74.986$ (7)°	$\mu = 0.66$ mm^{-1}
$\beta = 77.000$ (6)°	$T = 173$ (2) K
$\gamma = 88.773$ (6)°	Block, colorless
$V = 905.5$ (6) Å^3	0.21 × 0.18 × 0.13 mm

Data collection

Bruker SMART CCD area-detector diffractometer	3675 independent reflections
Radiation source: fine-focus sealed tube	2965 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.020$
$T = 173(2)$ K	$\theta_{max} = 26.5^\circ$
φ and ω scans	$\theta_{min} = 1.5^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1999)	$h = -9 \rightarrow 9$
$T_{min} = 0.874$, $T_{max} = 0.921$	$k = -10 \rightarrow 10$
7264 measured reflections	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	H-atom parameters constrained

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.118$

$S = 1.05$

3675 reflections

203 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$w = 1/[\sigma^2(F_o^2) + (0.0599P)^2 + 0.3134P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.57$ e Å^{-3}

$\Delta\rho_{min} = -0.28$ e Å^{-3}

Extinction correction: none

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.

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
B1	0.6261 (3)	0.7074 (3)	1.02589 (19)	0.0304 (6)
B2	0.6959 (4)	0.7428 (4)	0.85369 (19)	0.0362 (6)
B3	0.7750 (3)	0.4773 (3)	0.96019 (19)	0.0310 (6)
B4	0.3073 (4)	0.6356 (4)	0.42926 (18)	0.0332 (6)
B5	0.3334 (4)	0.6488 (4)	0.58748 (19)	0.0358 (6)
B6	0.5722 (4)	0.7796 (3)	0.44540 (19)	0.0329 (6)
C1	0.8494 (4)	0.2067 (3)	1.06702 (18)	0.0443 (6)
H1A	0.7824	0.2563	1.1165	0.066*
H1B	0.9707	0.1945	1.0737	0.066*
H1C	0.7973	0.0956	1.0737	0.066*
C2	0.9277 (4)	0.2425 (3)	0.89643 (19)	0.0422 (6)
H2A	0.9224	0.3209	0.8353	0.063*
H2B	0.8654	0.1362	0.9034	0.063*
H2C	1.0501	0.2207	0.8985	0.063*
C3	0.8137 (4)	0.9095 (4)	0.30292 (18)	0.0497 (7)
H3A	0.7354	0.8706	0.2694	0.074*
H3B	0.9260	0.8553	0.2916	0.074*
H3C	0.8325	1.0322	0.2793	0.074*
C4	0.8445 (4)	0.9189 (4)	0.4593 (2)	0.0479 (7)
H4A	0.7826	0.8931	0.5264	0.072*

H4B	0.8705	1.0406	0.4351	0.072*
H4C	0.9540	0.8588	0.4534	0.072*
N1	0.6252 (3)	0.8066 (2)	0.93346 (13)	0.0340 (5)
H1	0.5810	0.9073	0.9253	0.041*
N2	0.7702 (3)	0.5847 (2)	0.86706 (13)	0.0357 (5)
H2	0.8162	0.5487	0.8166	0.043*
N3	0.6985 (2)	0.5480 (2)	1.03919 (13)	0.0307 (4)
H3	0.6975	0.4885	1.0975	0.037*
N4	0.8461 (3)	0.3160 (2)	0.97372 (14)	0.0350 (5)
N5	0.2374 (3)	0.5985 (3)	0.52846 (13)	0.0354 (5)
H5	0.1352	0.5446	0.5535	0.043*
N6	0.4668 (3)	0.7245 (2)	0.38913 (13)	0.0335 (4)
H6	0.5058	0.7485	0.3270	0.040*
N7	0.4947 (3)	0.7372 (3)	0.54697 (13)	0.0356 (5)
H7	0.5515	0.7684	0.5847	0.043*
N8	0.7352 (3)	0.8657 (3)	0.40443 (14)	0.0374 (5)
Cl1	0.53274 (8)	0.78685 (8)	1.12721 (4)	0.03994 (18)
Cl2	0.68821 (12)	0.86972 (9)	0.73701 (5)	0.0595 (2)
Cl3	0.18580 (9)	0.56757 (10)	0.35536 (4)	0.0503 (2)
Cl4	0.24297 (10)	0.59670 (10)	0.71343 (4)	0.0538 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
B1	0.0278 (13)	0.0353 (14)	0.0321 (13)	−0.0005 (11)	−0.0074 (10)	−0.0153 (11)
B2	0.0414 (16)	0.0363 (15)	0.0301 (13)	0.0019 (12)	−0.0098 (12)	−0.0061 (11)
B3	0.0269 (13)	0.0335 (14)	0.0350 (14)	0.0005 (11)	−0.0083 (11)	−0.0118 (11)
B4	0.0352 (15)	0.0391 (15)	0.0294 (13)	0.0059 (12)	−0.0121 (11)	−0.0125 (11)
B5	0.0411 (16)	0.0391 (15)	0.0277 (13)	0.0096 (12)	−0.0067 (11)	−0.0114 (11)
B6	0.0386 (15)	0.0317 (14)	0.0305 (13)	0.0059 (12)	−0.0105 (11)	−0.0097 (11)
C1	0.0553 (17)	0.0354 (13)	0.0441 (15)	0.0110 (12)	−0.0180 (13)	−0.0087 (11)
C2	0.0438 (15)	0.0386 (14)	0.0484 (15)	0.0122 (11)	−0.0109 (12)	−0.0194 (12)
C3	0.0505 (17)	0.0588 (18)	0.0378 (14)	−0.0100 (14)	−0.0069 (12)	−0.0110 (13)
C4	0.0501 (16)	0.0532 (17)	0.0461 (15)	−0.0069 (13)	−0.0172 (13)	−0.0167 (13)
N1	0.0422 (12)	0.0286 (10)	0.0333 (10)	0.0092 (9)	−0.0110 (9)	−0.0102 (8)
N2	0.0425 (12)	0.0375 (11)	0.0284 (10)	0.0067 (9)	−0.0061 (9)	−0.0130 (9)
N3	0.0371 (11)	0.0301 (10)	0.0268 (9)	0.0047 (8)	−0.0101 (8)	−0.0083 (8)
N4	0.0401 (11)	0.0338 (11)	0.0336 (10)	0.0072 (9)	−0.0101 (9)	−0.0120 (9)
N5	0.0330 (11)	0.0449 (12)	0.0291 (10)	0.0015 (9)	−0.0072 (8)	−0.0106 (9)
N6	0.0374 (11)	0.0396 (11)	0.0246 (9)	0.0010 (9)	−0.0099 (8)	−0.0078 (8)
N7	0.0412 (12)	0.0423 (12)	0.0290 (10)	0.0025 (9)	−0.0122 (9)	−0.0159 (9)
N8	0.0425 (12)	0.0413 (12)	0.0314 (10)	−0.0017 (9)	−0.0115 (9)	−0.0116 (9)
Cl1	0.0510 (4)	0.0410 (3)	0.0329 (3)	0.0130 (3)	−0.0117 (3)	−0.0176 (3)
Cl2	0.0951 (6)	0.0502 (4)	0.0319 (3)	0.0177 (4)	−0.0185 (4)	−0.0060 (3)
Cl3	0.0417 (4)	0.0803 (5)	0.0339 (3)	−0.0100 (3)	−0.0122 (3)	−0.0196 (3)
Cl4	0.0597 (5)	0.0742 (5)	0.0267 (3)	−0.0045 (4)	−0.0039 (3)	−0.0162 (3)

Geometric parameters (Å, °)

B4—Cl3	1.804 (3)	C1—N4	1.454 (3)
B5—Cl4	1.794 (3)	C2—N4	1.455 (3)
B4—N5	1.412 (3)	C1—H1A	0.9800
B4—N6	1.391 (4)	C1—H1B	0.9800
B5—N5	1.420 (3)	C1—H1C	0.9800
B5—N7	1.401 (4)	C2—H2A	0.9800
B6—N6	1.457 (3)	C2—H2B	0.9800
B6—N7	1.453 (3)	C2—H2C	0.9800
B6—N8	1.405 (4)	C3—H3A	0.9800
C3—N8	1.454 (3)	C3—H3B	0.9800
C4—N8	1.455 (3)	C3—H3C	0.9800
B1—Cl1	1.801 (3)	C4—H4A	0.9800
B2—Cl2	1.797 (3)	C4—H4B	0.9800
B1—N1	1.411 (3)	C4—H4C	0.9800
B1—N3	1.400 (3)	N1—H1	0.8800
B2—N1	1.419 (3)	N2—H2	0.8800
B2—N2	1.398 (3)	N3—H3	0.8800
B3—N2	1.449 (3)	N5—H5	0.8800
B3—N3	1.453 (3)	N6—H6	0.8800
B3—N4	1.411 (3)	N7—H7	0.8800
N3—B1—N1	120.5 (2)	N8—C3—H3C	109.5
N3—B1—Cl1	120.00 (19)	H3A—C3—H3C	109.5
N1—B1—Cl1	119.53 (19)	H3B—C3—H3C	109.5
N2—B2—N1	119.8 (2)	N8—C4—H4A	109.5
N2—B2—Cl2	121.5 (2)	N8—C4—H4B	109.5
N1—B2—Cl2	118.6 (2)	H4A—C4—H4B	109.5
N4—B3—N2	123.0 (2)	N8—C4—H4C	109.5
N4—B3—N3	122.4 (2)	H4A—C4—H4C	109.5
N2—B3—N3	114.7 (2)	H4B—C4—H4C	109.5
N6—B4—N5	120.4 (2)	B1—N1—B2	119.6 (2)
N6—B4—Cl3	120.54 (19)	B1—N1—H1	120.2
N5—B4—Cl3	119.03 (19)	B2—N1—H1	120.2
N7—B5—N5	120.0 (2)	B2—N2—B3	123.0 (2)
N7—B5—Cl4	121.6 (2)	B2—N2—H2	118.5
N5—B5—Cl4	118.5 (2)	B3—N2—H2	118.5
N8—B6—N7	123.2 (2)	B1—N3—B3	122.4 (2)
N8—B6—N6	122.5 (2)	B1—N3—H3	118.8
N7—B6—N6	114.3 (2)	B3—N3—H3	118.8
N4—C1—H1A	109.5	B3—N4—C1	123.3 (2)
N4—C1—H1B	109.5	B3—N4—C2	124.0 (2)
H1A—C1—H1B	109.5	C1—N4—C2	112.7 (2)
N4—C1—H1C	109.5	B4—N5—B5	119.5 (2)
H1A—C1—H1C	109.5	B4—N5—H5	120.3
H1B—C1—H1C	109.5	B5—N5—H5	120.3
N4—C2—H2A	109.5	B4—N6—B6	123.0 (2)
N4—C2—H2B	109.5	B4—N6—H6	118.5

H2A—C2—H2B	109.5	B6—N6—H6	118.5
N4—C2—H2C	109.5	B5—N7—B6	122.9 (2)
H2A—C2—H2C	109.5	B5—N7—H7	118.6
H2B—C2—H2C	109.5	B6—N7—H7	118.6
N8—C3—H3A	109.5	B6—N8—C3	123.7 (2)
N8—C3—H3B	109.5	B6—N8—C4	123.5 (2)
H3A—C3—H3B	109.5	C3—N8—C4	112.7 (2)

Fig. 1

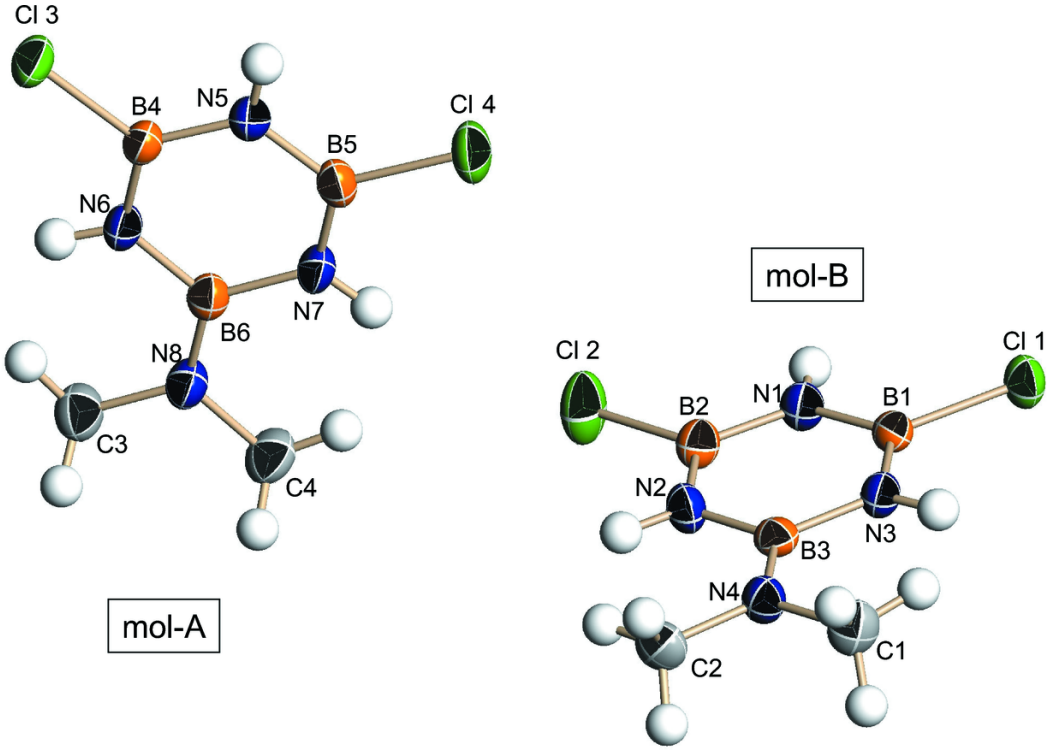
Cl 3
B4
N5
Cl 4
B5
N6
N7
B6
N8
C3
C4
mol-A
mol-B
Cl 2
Cl 1
B2
N1
B1
N2
N3
B3
N4
C2
C1

Fig. 2

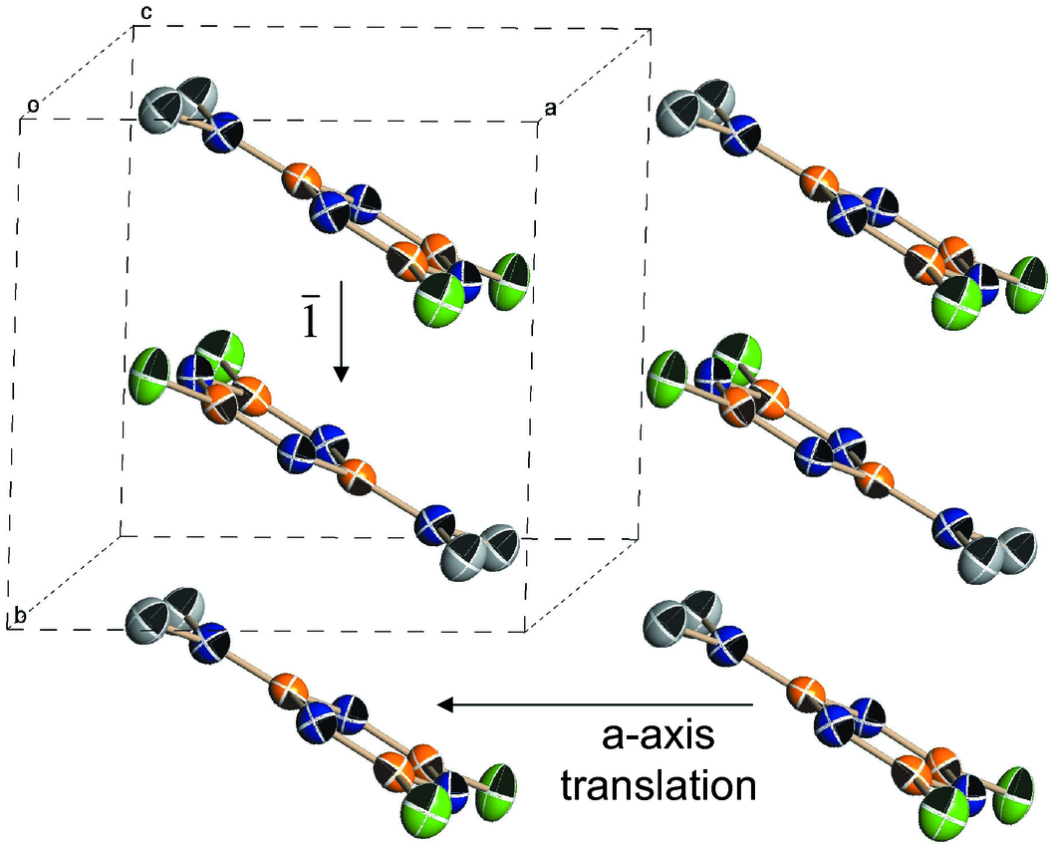

Fig. 3