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Determining the crystal structure of twinned 2-methylpyrazine

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Although systematic absences and symmetry relations among reflections pointed to space group $I4_122$ (one molecule in the asymmetric unit), a direct methods solution could only be obtained in $I\bar{4}$ (two molecules in the asymmetric unit). Refinement in $I\bar{4}$ was unsatisfactory until merohedral twinning was taken into account. The resulting molecular dimensions are in excellent agreement with analogous values in the literature. The molecular arrangement is described.

1. Introduction

Pyrazine and phenazine, and their derivatives, are interesting candidates for the investigation of hydrogen-bridged C—H···N systems. Some of these features are shown in the crystal packing of pyrazine (With *et al.*, 1976) and of the molecular compound of phenazine and 3,5-dinitrobenzoic acid (Pedireddi *et al.*, 1996). As H atoms attached to Csp^3 atoms tend to form much weaker hydrogen bridges than those attached to Csp^2 atoms, it is expected that C—H···N hydrogen bridging will be a less important factor in the packing arrangements of methylated pyrazines and phenazines. Therefore, we have determined the crystal structures of 2-methyl-, 2,3-dimethyl-, 2,5-dimethyl- and 2,6-dimethylpyrazines (m.p.s 244, 283, 287 and 311 K, respectively), and 2,3,5-trimethylpyrazine; 2,6-dimethylpyrazine has also been reported by Kaiser-Morris *et al.* (1998). The structure determinations, apart from that of 2-methylpyrazine, were routine and will be described elsewhere (Boese & Gehrke, 2000), together with a discussion of the packing arrangements (including those of tetramethylpyrazine, Braam *et al.*, 1981, and 5-methylpyrimidine, Furberg *et al.*, 1979). The crystal structure of 2-methylpyrazine, where the crystals were twinned, will be described here, emphasizing the methodology used.

2. Experimental

The diffraction measurements were made in Essen and the structure solution carried out in Essen and Haifa. 2-Methylpyrazine was crystallized using the '*in situ*' method at 230 K (Boese & Nussbaumer, 1994; Thalladi *et al.*, 1998). Diffraction patterns measured at 168 and 123 K with a Nicolet R3 four-circle diffractometer showed the compound to be tetragonal, with cell dimensions as given in Table 1; it is important to note that the sample was melted between these two sets of measurements which were thus made on *different* crystals. The following description refers specifically to the 123 K measurements, but the 168 K measurements gave similar results and are also briefly discussed. The condition governing general reflections was $h + k + l = 2n$, showing the crystals to

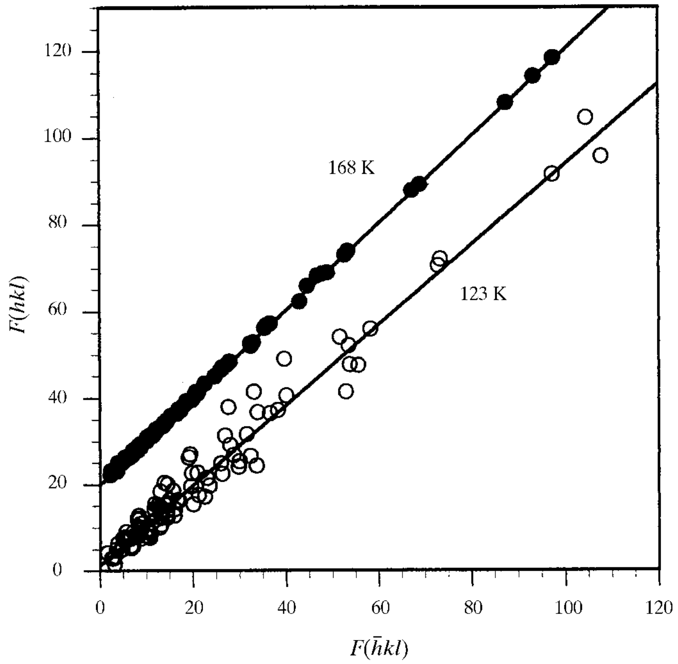

Figure 1
Plot of paired values of $F(hkl)$ against $F(\bar{h}kl)$ to check the Laue group; 104 pairs (chosen at random) have been included, out of approximately 400 available. For clarity, the 168 K values have been offset along the ordinate by 20 units. At 123 K the equation of the straight line is $F(hkl) = 0.927F(\bar{h}kl) + 1.189$, and the correlation factor is $R^2 = 0.9726$. The value of $R_{\rm int}$ is 0.0952. At 168 K the equation of the straight line is $F(hkl) = 1.005F(\bar{h}kl) - 0.061$ and the correlation factor is $R^2 = 0.9997$. The value of $R_{\rm int}$ is 0.0085. We are grateful to a referee for suggesting that the correlations at both temperatures should be compared.

Table 1
Cell dimensions of 2-methylpyrazine at 168 and 123 K, $Z = 16$; there was no change in crystal structure over the temperature range studied.

T (K)	a (Å)	c (Å)	V (Å^3)
168	13.7988 (16)	10.8761 (22)	2070.9 (8)
123	13.7248 (19)	10.8585 (22)	2045.6 (8)

Table 2
Values of $F(00l)$, showing the apparent absence of $00l$ reflections for $l = 4n$.

F(calc) from final parameters for 123 K structure analysis (space group $I\bar{4}$, $Z = 16$, twinned crystal). The final column shows that reflections with $l = 4n + 2$ have intensities significantly different from zero and thus that the apparent fourfold screw axis along [001] is not exact.

$00l$	F(obs) at 168 K	F(obs) at 123 K	F(calc) at 123 K	$(F(\rm obs)^2)/\{u(F(\rm obs)^2)\}$ at 123 K
002	2.50	2.67	3.01	12
004	97.91	104.4	100.4	18166
006	2.59	4.28	3.04	23
008	29.60	31.1	33.2	1612
00,10	4.27	4.71	5.48	18.5

be body-centered. Values of the $00l$ structure factors are given in Table 2; the condition $00l = 4n$ is obeyed to a good *approximation*. At this stage the possible space groups appeared to be $I4_1$ or $I4_122$, which belong to Laue groups $4/m$ and $4/mmm$, respectively (see Table 4.4.3 of *International Tables for X-ray Crystallography*, 1965). The space groups can be distinguished through the condition $F(hkl) = F(\bar{h}kl)$ which holds for $I4_122$, but not for $I4_1$ (see pp. 419 and 431 of *International Tables for X-ray Crystallography*, 1965). We have plotted $F(hkl)$ against $F(\bar{h}kl)$ in Fig. 1, obtaining linear relationships at both temperatures (we return to this plot later). Thus, the space group *appeared* to be confirmed as $I4_122$, with one molecule in the asymmetric unit. Attempts were then made to solve the structure in $I4_122$ by direct methods (*SHELXS*86; Sheldrick, 1990), but completely without success. Other tetragonal space groups were then tried and an apparently satisfactory direct methods solution was obtained in $I\bar{4}$ with two molecules in the asymmetric unit. This was refined by standard methods (*SHELXL*97; Sheldrick, 1997), but with unacceptable results ($wR_2 = 0.272$, $R_1 = 0.104$ for all 1338 data, 139 parameters; $S = 0.962$; some atomic displacement parameters, a.d.p.s, and H atoms unsatisfactory). Analogous coordinates in the two independent

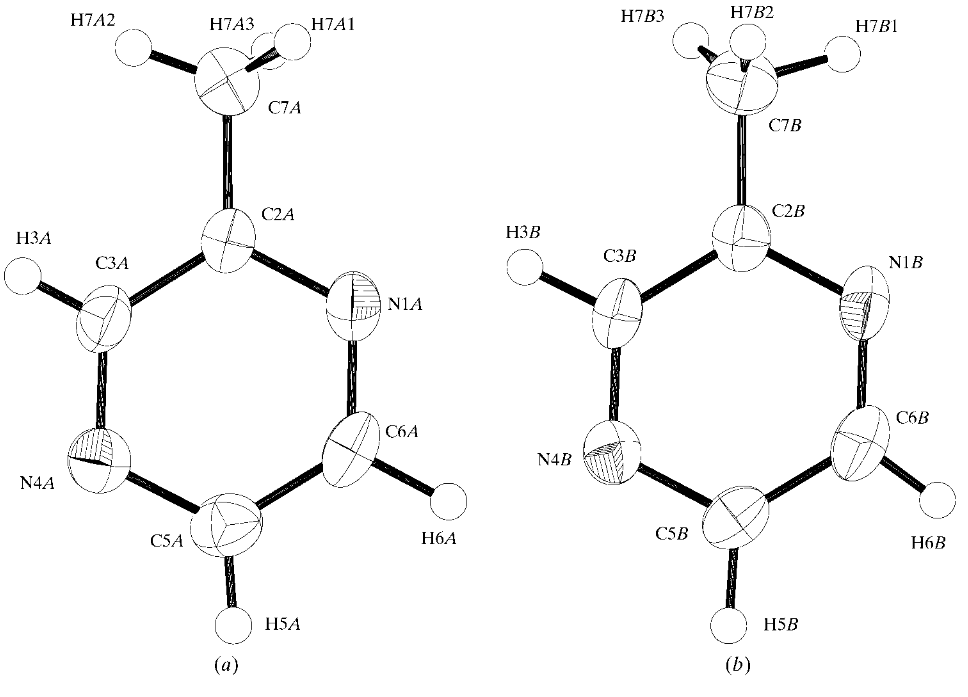

Figure 2
*ORTEP*II (Johnson, 1976) diagrams of the two crystallographically independent molecules, showing numbering of the atoms and atomic displacement (50% probability) ellipsoids.

Table 3
Experimental details.

	168 K	123 K
Crystal data		
Chemical formula	$C_5H_6N_2$	$C_5H_6N_2$
Chemical formula weight	94.12	94.12
Cell setting	Tetragonal	Tetragonal
Space group	$I\bar{4}$	$I\bar{4}$
a (Å)	13.799 (2)	13.725 (2)
b (Å)	13.799 (2)	13.725 (2)
c (Å)	10.876 (2)	10.859 (2)
V (Å^3)	2071.0 (6)	2045.6 (6)
Z	16	16
D_x (Mg m^{-3})	1.208	1.222
Radiation type	Mo $K\alpha$	Mo $K\alpha$
Wavelength (Å)	0.71073	0.71073
No. of reflections for cell parameters	50	50
θ range (°)	3–45	3–45
μ (mm^{-1})	0.079	0.079
Temperature (K)	168 (2)	123 (2)
Crystal form	Cylindrical	Cylindrical
Crystal radius (mm)	0.15	0.15
Crystal color	Colorless	Colorless
Data collection		
Diffractometer	Nicolet *R3m/V* four circle	Nicolet *R3m/V* four circle
Data collection method	Wyckoff scan mode	Wyckoff scan mode
Absorption correction	None	None
No. of measured reflections	2877	1474
No. of independent reflections	1357	1338
No. of observed reflections	1322	1301
Criterion for observed reflections	$I > 2\sigma(I)$	$I > 2\sigma(I)$
$R_{\rm int}$	0.0225	0.0241
$\theta_{\rm max}$ (°)	22.55	22.50
Range of h, k, l	$-11 \rightarrow h \rightarrow 14$ $-11 \rightarrow k \rightarrow 14$ $-10 \rightarrow l \rightarrow 11$	$-14 \rightarrow h \rightarrow 10$ $-10 \rightarrow k \rightarrow 14$ $0 \rightarrow l \rightarrow 11$
No. of standard reflections	2	2
Frequency of standard reflections	Every 100 reflections	Every 100 reflections
Intensity decay (%)	2.7	0.7
Refinement		
Refinement on	F^2	F^2
$R[F^2>2\sigma(F^2)]$	0.0213	0.0257
$wR(F^2)$	0.0501	0.0679
S	1.057	1.082
No. of reflections used in refinement	1357	1338
No. of parameters used	177	176
H-atom treatment	Mixed	Mixed
Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (0.0324P)^2 + 0.0103P]$, where $P = (F_o^2 + 2F_c^2)/3$	$w = 1/[\sigma^2(F_o^2) + (0.0428P)^2 + 0.2414P]$, where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$	0.009	0.004
$\Delta\rho_{\rm max}$ (e Å^{-3})	0.105	0.086
$\Delta\rho_{\rm min}$ (e Å^{-3})	−0.110	−0.124
Extinction method	*SHELXL*97 (Sheldrick, 1997)	None
Extinction coefficient	0.0118 (11)	–
Source of atomic scattering factors	*International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)	*International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Computer programs		
Data collection	*P3/PC* (Siemens, 1989*a*)	*P3/PC* (Siemens, 1989*a*)
Cell refinement	*P3/PC* (Siemens, 1989*a*)	*P3/PC* (Siemens, 1989*a*)
Data reduction	*XDISK* (Siemens, 1989*b*)	*XDISK* (Siemens, 1989*b*)
Structure solution	*SHELXS*97 (Sheldrick, 1990)	*SHELXS*97 (Sheldrick, 1990)
Structure refinement	*SHELXL*97 (Sheldrick, 1997)	*SHELXL*97 (Sheldrick, 1997)

molecules were approximately related as x, y, z and $\frac{1}{2} - x, y, -\frac{1}{4} + z$; this is not a relation between coordinates in space group $I4_122$. Application of various disorder models did not produce any improvement.

At this stage, Marsh (1998) suggested to us that the source of our problems might lie in twinning and this was reinforced by the serendipitous appearance of 'Refinement of twinned structures with *SHELXL*97' by Herbst-Irmer & Sheldrick (1998). We also draw attention to a somewhat different approach to merohedral twinning (Kahlenberg, 1999), which appeared after this paper had been accepted. Inspection showed that many features of our results matched those listed by these authors as characteristic indicators of twinning. Following Herbst-Irmer & Sheldrick we note that the twinning in 2-methylpyrazine is by merohedry, where the twin law is a symmetry operator of the crystal system but not of the point group of the crystal. This means that the reciprocal lattices of the different twin domains superimpose and the twinning is not directly detectable from the diffraction pattern. The twin law is $\mathbf{R} = (010, 100, 00\bar{1})$, *i.e.* a non-crystallographic twofold axis along [110]. Insertion of the appropriate instructions in *SHELXL*97 led to $wR_2 = 0.0676$, $R_1 = 0.0271$ for all 1338 data measured at 123 K, 139 parameters; $S = 1.082$; twin component fraction 0.315 (2) (experimental details and the refinement process are described in Table 3; note that the absolute structure cannot be reliably determined for this combination of X-ray wavelength and unit-cell contents; Flack, 1983). Similar refinement of the 168 K measurements gave the twin component fraction as 0.511 (2); as noted above, the measurements were made on two different crystals.

The 123 K atomic parameters are given in Table 4 (a.d.p.s, hydrogen parameters and all 168 K

Table 4
Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2).

$$U_{\rm eq} = (1/3)\Sigma_i \Sigma_j U^{ij} a^i a^j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N1*A*	0.01857 (15)	0.13349 (14)	0.15884 (18)	0.0377 (6)
C2*A*	0.04218 (18)	0.13973 (16)	0.2772 (2)	0.0310 (6)
C3*A*	−0.0289 (2)	0.12985 (19)	0.3668 (2)	0.0381 (6)
N4*A*	−0.12254 (16)	0.11445 (18)	0.34271 (19)	0.0437 (6)
C5*A*	−0.1452 (2)	0.1090 (2)	0.2227 (3)	0.0427 (7)
C6*A*	−0.0756 (2)	0.11701 (19)	0.1330 (2)	0.0416 (7)
C7*A*	0.1464 (2)	0.1572 (3)	0.3096 (3)	0.0452 (8)
N1*B*	−0.52689 (17)	0.12727 (17)	−0.09554 (18)	0.0414 (6)
C2*B*	−0.55250 (18)	0.13104 (18)	0.0227 (2)	0.0349 (6)
C3*B*	−0.48228 (19)	0.12616 (19)	0.1148 (2)	0.0369 (6)
N4*B*	−0.38712 (16)	0.11870 (17)	0.09235 (19)	0.0391 (6)
C5*B*	−0.3620 (2)	0.11554 (19)	−0.0264 (3)	0.0386 (6)
C6*B*	−0.4314 (2)	0.11949 (19)	−0.1188 (3)	0.0432 (7)
C7*B*	−0.6584 (2)	0.1387 (2)	0.0524 (3)	0.0438 (7)

Table 5
Dimensions (Å, °) of the two independent 2-methylpyrazine molecules (*A* and *B*) at 123 K.

The C—H bond lengths vary between 0.93 and 1.04 Å. The mean measured values for seven 2-methylpyrazine molecules/fragments and semi-empirical calculated (MNDO-PM3; Stewart, 1989) values are also given.

Bond/Angle	2-Methylpyrazine		Mean measured values (1, 2)	PM3 calculations
	Molecule *A*	Molecule *B*		
N1—C2	1.328 (3)	1.331 (3)	1.330 (1)	1.357
C2—C3	1.384 (4)	1.390 (4)	1.384 (3)	1.407
C3—N4	1.329 (4)	1.332 (4)	1.329 (4)	1.350
N4—C5	1.342 (3)	1.335 (4)	1.330 (6)	1.353
C5—C6	1.368 (4)	1.385 (4)	1.379 (3)	1.406
C6—N1	1.342 (4)	1.339 (4)	1.344 (1)	1.362
C2—C7(methyl)	1.492 (4)	1.492 (4)	1.495 (4)	1.488
C6—N1—C2	116.6 (2)	116.3 (2)	116.9 (2)	119.33
N1—C2—C3	120.1 (2)	120.5 (2)	120.7 (1)	120.00
C2—C3—N4	124.0 (2)	123.5 (2)	122.9 (3)	120.65
C3—N4—C5	115.1 (2)	115.6 (2)	116.0 (2)	119.52
N4—C5—C6	121.6 (3)	121.4 (2)	122.1 (3)	120.31
C5—C6—N1	122.5 (2)	122.7 (3)	121.4 (4)	120.19
C7—C2—N1	118.2 (2)	117.9 (2)	118.0 (2)	118.45
C7—C2—C3	121.7 (2)	121.5 (2)	121.2 (2)	121.54

Notes to Table 5: (i) The data set for the averaging comprises: present values for molecules *A* and *B*: WIKDUF, *N*-(4,6-dimethylpyrid-2-yl)-5-methylpyrazine-2-carboxamide (Rodier *et al.*, 1994); YEHPIK 6,6′-dimethyl-2,2′-pyrazine, two independent sets of values (Regnouf de Vains *et al.*, 1994); ZIXNID *S*-ethyl-4-(2-(6-methyl-2-pyrazinyl)ethenyl)thiocinnamate (Saigo *et al.*, 1995); ZIXNOJ ethyl-4-(2-(6-methyl-2-pyrazinyl)ethenyl)cinnamate (Saigo *et al.*, 1995) [ZIXNUP (Saigo *et al.*, 1995) was not included because R = 0.10]. (ii) The bracketed values are the s.u.s of the means; the sample s.u.s are about three times as large. (iii) Pyrazine at 184 K has molecular symmetry 2/*m* and bond lengths N1—C2 1.334 (1), C2—C3 1.387 (1) Å, angles C6—N1—C2 116.2 (1), N1—C2—C3 121.9 (1)° (With *et al.*, 1976). (iv) In comparing these dimensions (some at 300 K, others at 123 K) we assume that molecular dimensions do not change with temperature.

values have been deposited);[1] the relations given above between coordinates in the two independent molecules still appear, but even less precisely. *ORTEP*II (Johnson, 1976) diagrams showing the displacement ellipsoids of the two independent molecules are shown in Fig. 2. The coordinates obtained for the refinement that did not take twinning into account are similar to those in Table 4 (as would be expected), but with significant differences. The non-zero F(calc) values for the 00l reflections (Table 2) confirm the existence of a pseudo-4_1 axis along [001]. With the benefit of hindsight, we note that the condition $F(hkl) = F(\bar{h}kl)$ is better adhered to for the 168 K measurements than for the 123 K measurements (see caption to Fig. 1), in accordance with the twin ratio of 0.51:0.49 at 168 K and 0.32:0.68 at 123 K. Indeed, the $R_{\rm int}$ value of 0.09 for the special group of reflections at 123 K should have aroused suspicion when compared with the overall $R_{\rm int}$ values of 0.02 at both temperatures.

3. Molecular structure

Dimensions for the two independent 2-methylpyrazine molecules (*A* and *B*) are given in Table 5; the agreement is excellent. The only difference between the two independent molecules is in the disposition of the H atoms of the methyl groups (see Fig. 2); the analogous torsion angles H7*A*1—C7*A*—C2*A*—N1*A* and H7*B*2—C7*B*—C2*B*—N1*B* are −55 (2) and −87 (3)°, respectively. The largest deviations from the best planes of *A* and *B* are 0.009 and 0.007 Å, respectively. C—N—C angles of 116° are also found in pyrazine and phenazine (Wozniak *et al.*, 1991) and 2,6-dimethylpyrazine (Kaiser-Morris *et al.*, 1998).

A Cambridge Structural Database (CSD; Allen *et al.*, 1991) search for 2-methylpyrazine fragments gave five hits and four sets of values for bond lengths and angles. All six sets of values are in excellent agreement and mean dimensions for 2-methylpyrazine have been added to Table 4. Semi-empirical calculations at the 'modified neglect of diatomic overlap, parametric method 3' (MNDO-PM3) level (Stewart, 1989) have been made for 6,6′-dimethyl-2,2′-pyrazine (YEHPIK; Regnouf de Vains *et al.*, 1994). The dimensions of the 2-methylpyrazine fragment (Table 5) are only approximately reproduced; for example, the calculated value of the C—N—C angle is 3.5° larger than the measured value, indicating that the hybridization of the N atoms has not been properly taken into account.

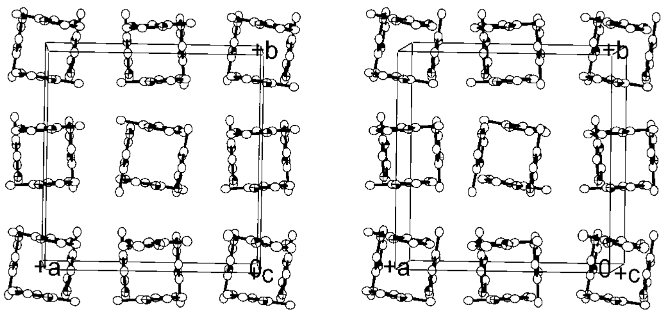

Figure 3
Stereoview *down* [001] of the crystal structure of 2-methylpyrazine at 123 K. The *A* reference molecule (coordinates in Table 4) is at the upper-right corner of the unit cell and *B* reference molecule about −*a*/2.

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[1] Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF0007). Services for accessing these data are described at the back of the journal.

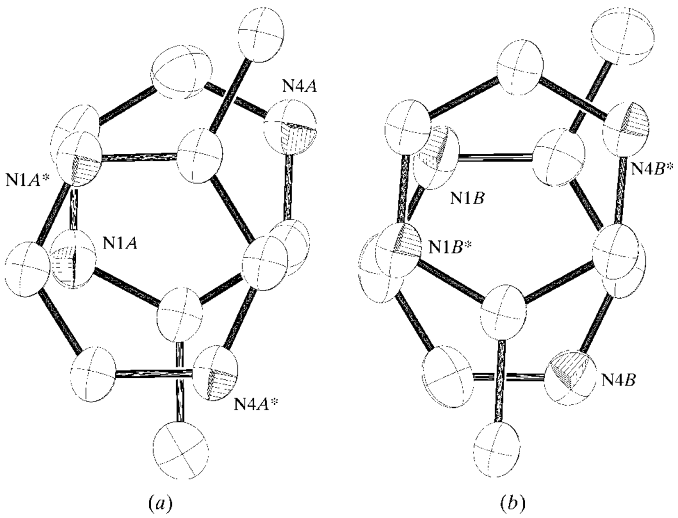

Figure 4
Comparison of (*a*) the *A*/*A** (interplanar distance 3.439 Å) and (*b*) *B*/*B** (interplanar distance 3.456 Å) overlap diagrams.

4. Crystal structure

Arrangement of the molecules in the unit cell is not particularly complicated, but nevertheless somewhat difficult to depict. A stereoview down [001] is shown in Fig. 3; the *A* and *B* molecules are segregated into crystallographically independent columns running respectively along 0, 0, *z* (Wyckoff position *e*) and $\frac{1}{2}$, 0, *z* (Wyckoff position *f*). The column of *A* molecules is rotated 13.5° anti-clockwise from [100] and that of *B* molecules rotated 4.5° clockwise from [100]. The *A* and *B* molecular planes are almost perpendicular to (001). The structures of the two types of column are similar, differing only in numerical detail. Consider the *A* 'reference molecule' (coordinates in Table 4) – the fourfold inversion axis (and translations) produces the arrangement along 0, 0, *z* shown in Fig. 3; an analogous arrangement of *B* molecules, shifted along [001] by *c*/4, is formed along $\frac{1}{2}$, 0, *z*. The interplanar distance between two parallel *A*, *A** molecules is 3.439 Å and between two *B*, *B** molecules 3.456 Å. The *A*/*A** and *B*/*B** overlap diagrams (Figs. 4*a* and *b*) are very similar. Details of the intermolecular distances will be discussed elsewhere (Boese & Gehrke, 2000).

The space group $I\bar{4}$ with $Z = 16$ is rare; we have found only two other examples in the October, 1998, version of the CSD. These are *N*-isopropyl-3-methyl-4,4-diphenyl-3-butenamide (JAHSAM; Declerq *et al.*, 1988) and *cis*-bis(η^5-cyclopentadienyl)-2,3-di-*tert*-butyl-2,3-diphenyl-1,4-dithia-2,3-disila-5-titanacyclo-pentane (YUJWAB10; Choi *et al.*, 1996). JAHSAM does not appear to show any unusual features; coordinates are not available for YUJWAB10.

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