

**Discussion.** Owing to centrosymmetry, both  $\Delta$  and  $\Lambda$  configurations of the cations are present in the structure (Purcell & Kotz, 1977). Chelate ligands have  $\lambda\delta$  conformation with torsion angles around C(1)–C(2) and C(3)–C(4) 52.6 (6) and 56.1 (6)°. Inversion centres and glide planes present in the  $P2_1/c$  unit cell transform a  $\Delta\lambda\delta$  enantiomer of one cation into the  $\Lambda\delta\lambda$  enantiomer of the other. The same situation was found in the structures of *cis*-( $\pm$ )-[CoCl<sub>2</sub>(en)<sub>2</sub>]Cl·H<sub>2</sub>O (Matsumoto, Ooi & Kuroya, 1970) and *cis*-( $\pm$ )-[RhCl<sub>2</sub>(en)<sub>2</sub>]Cl·HgCl<sub>2</sub> (Podberez'skaja, Beljajev, Bokanin & Bajdina, 1981). By contrast the conformation of the ligands found in the racemic crystal of *cis*-( $\pm$ )-[RhCl<sub>2</sub>(en)<sub>2</sub>]NO<sub>3</sub> was  $\Delta\delta\delta$  and  $\Lambda\lambda\lambda$  (Bajdina, Podberez'skaja, Beljajev & Bokanin, 1979). It is known that the energy difference between different conformations of chelate ligands with the same molecular helicity is small and can easily be leveled off by crystal forces (Gollogly & Hawkins, 1970).

Cr–F bond lengths are comparable to the value 1.887 (6) Å found in *cis*-[CrF<sub>2</sub>(NH<sub>3</sub>)<sub>4</sub>]ClO<sub>4</sub> (Brenčič, Čeh & Leban, 1981). Cr–N(en) bonds in the range 2.05 to 2.10 Å were also found in the structures of *trans*-[CrF<sub>2</sub>(en)<sub>2</sub>]ClO<sub>4</sub> and *trans*-[CrF<sub>2</sub>(en)<sub>2</sub>]Cl (Brenčič & Leban, 1981, 1985).

Cations are connected by weak hydrogen bonds between N(2), N(3)(*x*, *y*, *z*) and F(1), F(2)(*x*–1, *y*, *z*) at 2.890 (5) and 2.853 (5) Å. Interactions between O atoms of the perchlorate anion and N atoms of the cation are from 3.03 (1) to 3.06 (1) Å, which is on the limit of the sum of the van der Waals radii (Huheey, 1972).

The average Cl–O bond length is 1.40 (1) Å. O–Cl–O angles are from 106.6 (4) to 113.6 (4)°.

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## Structure of Merohedrally Twinned Di- $\mu$ -fluoro-bis[tris(3,5-dimethylpyrazole-*N*<sup>2</sup>)-copper(II)] Bis(tetrafluoroborate)

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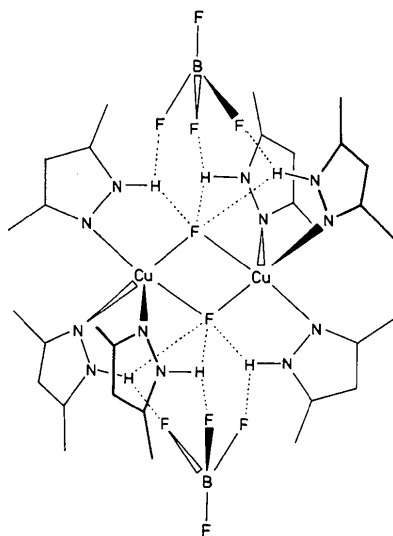
**Abstract.** [Cu<sub>2</sub>F<sub>2</sub>(C<sub>5</sub>H<sub>8</sub>N<sub>2</sub>)<sub>6</sub>](BF<sub>4</sub>)<sub>2</sub>, *M<sub>r</sub>* = 915.49, trigonal,  $R\bar{3}$ , *a* = 28.649 (7), *c* = 17.133 (4) Å, *V* = 12178 (5) Å<sup>3</sup>, *Z* = 12, *D<sub>x</sub>* = 1.498 g cm<sup>-3</sup>,  $\lambda(\text{Mo K}\alpha)$  = 0.71073 Å,  $\mu$  = 10.74 cm<sup>-1</sup>, 100 K, *F*(000) = 5640,

*R* = 0.0720 for 1669 observed reflections with *I* > 2.5 $\sigma$ (*I*). The crystal is twinned by merohedry ( $\alpha$  = 0.50). Domains are related by mirror planes of the  $R\bar{3}m$  space group. The molecules are dimeric, consisting of two CuF(dmpz)<sub>3</sub>(BF<sub>4</sub>) units, related by an inversion centre. The two Cu atoms are asymmetrically

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$\mu$ -fluoro-bridged. The coordination of Cu is square-based pyramidal. The dimers are placed on either  $\bar{1}$  or  $\bar{3}$  sites. Those on the  $\bar{3}$  sites are disordered. The dimeric cation and two anions form a globular neutral cluster, held together through electrostatic and hydrogen-bonding interactions. The globular clusters form a pseudo-cubic-close-packed structure.

**Introduction.** A number of dimeric, cluster and linear transition-metal compounds have been studied to investigate the magnetic exchange between paramagnetic transition-metal ions. The presence of simple bridging ligands in these compounds seems favourable. The ligand-exchange method developed by Reedijk & Ten Hoedt (1982), which involves exchange of non-bridging ligands with other ligands, resulted in a series of fluoro-bridged copper(II) dimers with pyrazole ligands. The crystal and molecular structures of three of these compounds have already been described (Rietmeijer, de Graaff & Reedijk, 1984; Ten Hoedt, Reedijk & Verschoor, 1981). We now report the structure of [CuF(dmpz)<sub>3</sub>]<sub>2</sub>(BF<sub>4</sub>)<sub>2</sub> (dmpz = 3,5-dimethylpyrazole). The structure determination of this compound was seriously hampered by a combination of pseudo-symmetry, disorder and twinning.



**Experimental.** Crystal selected was a small blue cube (0.19 × 0.19 × 0.19 mm), grown from ethanol. Weissenberg photographs (100 K) were indicative of a trigonal space group and disorder. Enraf–Nonius CAD-4F diffractometer, data collected at 100 K using graphite-monochromated Mo K $\alpha$  radiation. Lattice constants determined by least-squares refinement of the angular settings of 12 reflections ( $15 < \theta < 17^\circ$ ); data set (obverse hexagonal setting  $-h + k + l = 3n$ ),  $\omega/2\theta$  scan mode,  $\Delta\omega = 1 + 0.35 \tan\theta^\circ$ ,  $1 < \theta < 20^\circ$ , half a sphere,  $h -27 \rightarrow 27$ ;  $k -27 \rightarrow 27$ ;  $l 0 \rightarrow 16$ , total of 7880 reflections measured; 2532 unique reflections.

Standard reflections ( $\bar{6}63$ ,  $0\bar{6}3$ ,  $603$ ), measured every 5000 s, showed less than 1% decay during 115 hours of X-ray exposure time. Fluctuations in the intensity were less than 1%. Intensity data corrected for the small decay, Lorentz and polarization effects. The standard deviation  $\sigma(I)$  was calculated following McCandlish, Stout & Andrews (1975);  $\sigma^2(I) = \sigma_{cs}^2(I) + (PI)^2$  with  $P = 0.015$ . A  $260^\circ \psi$  scan for 006 suggested that no absorption correction was necessary. The diffraction pattern showed  $\bar{3}m$  Laue symmetry ( $R_{av} = 0.048$  for  $\bar{3}$  and 0.047 for  $\bar{3}m$ ). Attempts to solve the structure in  $R\bar{3}m$  failed. The Patterson map suggested pseudosymmetry, as indicated by the strong  $(\frac{1}{2}, 0, 0)$  vector. The structure was eventually solved in the low trigonal space group  $R\bar{3}$  using the  $R\bar{3}$  averaged data set. A satisfactory model with ordered dimers on  $\bar{1}$  sites and disordered dimers on  $\bar{3}$  sites was found using Patterson-interpretation and tangent-expansion techniques (SHELX84). Preliminary positions of the atoms of the disordered molecule were calculated making use of the pseudosymmetry. Refinement with hydrogens not included and isotropic temperature factors converged at  $R = 0.25$ . Subsequent refinement with all molecules disordered in  $R\bar{3}m$  gave no satisfactory results. Therefore the  $R\bar{3}m$  symmetry of the data set was interpreted as twinning by merohedry of the  $R\bar{3}$  structure with twin domains related by the  $R\bar{3}m$  mirror planes resulting in exactly superimposed reflection intensities for the reflections  $hkl$  and  $\bar{k}\bar{h}l$ . The data were deconvoluted into untwinned data on the basis of  $F_{calc}$  using different  $\alpha$  values. The most satisfactory result was obtained with  $\alpha = 0.5$ , resulting in a drop in the  $R$  value to 0.13. This process was repeated twice more during the final refinement. The model was refined on  $|F|$  by blocked full-matrix least-squares procedures. H atoms were introduced in calculated positions (C–H = 1.08 Å) and refined in the riding mode on their carrier atoms, except for those involved in hydrogen bonding that were refined with slack constraints (N–H = 0.98 Å,  $\sigma = 0.05$  Å). Anisotropic thermal parameters for the non-hydrogen atoms, and the Cu and F atoms of the disordered molecule. The thermal parameters of the hydrogen atoms were refined with separate overall parameters for the methyl, the ring and the N–H atoms. Owing to the poor quality of the crystal (twinning and disorder) problems arose with the refinement of the thermal parameters of a few carbon atoms. Final atomic parameters are listed in Table 1.\*

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and additional bond lengths and angles have been deposited together with a description of the twin correction procedure and a CPK model plot with the British Library Document Supply Centre as Supplementary Publication No. SUP 43684 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Convergence was reached at  $R = 0.0720$ ,  $wR = 0.0486$ ,  $w^{-1} = \sigma^2(F)$ , 381 parameters, 1669 observed reflections,  $\langle \Delta/\sigma \rangle 0.1$ , min. and max. residual density  $-0.46$  and  $0.56 \text{ e } \text{Å}^{-3}$ . The high  $R$  value is due to the poor crystal quality. Scattering factors of Cromer & Mann (1968). Anomalous-dispersion corrections from Cromer & Liberman (1970). Calculations carried out on the CYBER-855 of the University of Utrecht with programs of the *SHELX76* (Sheldrick 1976), *SHELX84* (Sheldrick, 1984) and *EUCLID* (Spek, 1982) packages.

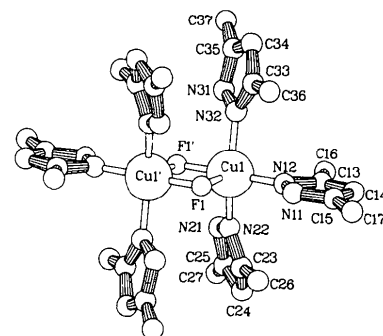


Fig. 1. *PLUTO* drawing (*EUCLID* version; Spek, 1982) of the cation  $[\text{Cu}_2\text{F}_2(\text{dmpz})_6]^{2+}$  (molecule 1) with adopted numbering scheme. Hydrogens omitted for clarity.

Table 1. Final positional parameters for the non-hydrogen atoms and equivalent isotropic temperature factors with *e.s.d.*'s in parentheses (molecule 2 is disordered)

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

Molecule 1	x	y	z	$U_{eq}(\text{Å}^2)$
Cu(1)	0.1092 (1)	0.3046 (1)	0.3478 (1)	0.0388 (5)
F(1)	0.1650 (2)	0.3317 (2)	0.2506 (3)	0.039 (2)
N(11)	0.0513 (4)	0.2732 (4)	0.1969 (5)	0.040 (5)
N(12)	0.0489 (4)	0.2745 (4)	0.2768 (6)	0.042 (4)
N(21)	0.0990 (4)	0.3778 (4)	0.4506 (5)	0.033 (4)
N(22)	0.1003 (3)	0.3663 (4)	0.3723 (5)	0.048 (4)
N(31)	0.1054 (4)	0.2231 (5)	0.4546 (6)	0.055 (6)
N(32)	0.1017 (3)	0.2333 (4)	0.3757 (5)	0.036 (4)
C(13)	-0.0037 (6)	0.2490 (5)	0.2926 (8)	0.046 (5)
C(14)	-0.0341 (5)	0.2335 (5)	0.2240 (9)	0.053 (6)
C(15)	0.0033 (5)	0.2497 (4)	0.1624 (6)	0.039 (5)
C(16)	-0.0229 (4)	0.2401 (5)	0.3747 (7)	0.074 (6)
C(17)	-0.0059 (4)	0.2462 (5)	0.0754 (6)	0.066 (5)
C(23)	0.0860 (5)	0.3997 (6)	0.3408 (7)	0.057 (5)
C(24)	0.0734 (4)	0.4285 (4)	0.3897 (8)	0.051 (6)
C(25)	0.0857 (5)	0.4168 (5)	0.4652 (8)	0.057 (6)
C(26)	0.0802 (4)	0.3975 (4)	0.2501 (6)	0.054 (5)
C(27)	0.0801 (5)	0.4356 (5)	0.5426 (6)	0.076 (5)
C(33)	0.0893 (5)	0.1887 (6)	0.3362 (7)	0.052 (6)
C(34)	0.0852 (5)	0.1496 (5)	0.3898 (8)	0.063 (6)
C(35)	0.0945 (5)	0.1724 (6)	0.4658 (7)	0.054 (6)
C(36)	0.0832 (4)	0.1810 (4)	0.2514 (6)	0.060 (5)
C(37)	0.0926 (5)	0.1515 (4)	0.5445 (6)	0.075 (6)
B(1)	0.3319 (6)	0.153 (1)	-0.0427 (9)	0.063 (8)
F(11)	0.3339 (2)	0.1601 (4)	0.0365 (3)	0.064 (3)
F(12)	0.3872 (2)	0.1828 (4)	-0.0715 (3)	0.059 (3)
F(13)	0.3069 (3)	0.1026 (6)	-0.0667 (3)	0.069 (3)
F(14)	0.3066 (3)	0.1813 (6)	-0.0730 (3)	0.067 (3)
Molecule 2				
Cu(2)	0.6093 (1)	0.3065 (2)	0.3503 (2)	0.025 (1)
F(2)	0.6667 (1)	0.3333 (2)	0.2484 (5)	0.045 (3)
N(41)	0.543 (2)	0.275 (2)	0.202 (2)	0.04 (1)
N(42)	0.552 (2)	0.282 (2)	0.276 (2)	0.03 (1)
N(51)	0.605 (1)	0.374 (1)	0.466 (1)	0.012 (9)
N(52)	0.599 (1)	0.368 (1)	0.385 (1)	0.025 (9)
N(61)	0.601 (1)	0.230 (1)	0.469 (1)	0.000 (7)
N(62)	0.593 (1)	0.232 (1)	0.382 (1)	0.02 (1)
C(43)	0.492 (1)	0.252 (1)	0.294 (2)	0.000 (9)
C(44)	0.456 (2)	0.234 (2)	0.235 (2)	0.05 (1)
C(45)	0.492 (2)	0.244 (2)	0.178 (2)	0.07 (2)
C(46)	0.481 (2)	0.250 (2)	0.381 (2)	0.05 (1)
C(47)	0.490 (2)	0.238 (2)	0.092 (2)	0.02 (1)
C(53)	0.588 (1)	0.402 (1)	0.375 (2)	0.000 (9)
C(54)	0.571 (2)	0.423 (1)	0.433 (2)	0.01 (1)
C(55)	0.590 (1)	0.408 (1)	0.496 (1)	0.00 (1)
C(56)	0.569 (2)	0.406 (2)	0.300 (3)	0.10 (2)
C(57)	0.594 (2)	0.419 (1)	0.582 (1)	0.007 (9)
C(63)	0.574 (1)	0.187 (1)	0.353 (1)	0.000 (8)
C(64)	0.561 (1)	0.149 (1)	0.412 (2)	0.00 (1)
C(65)	0.572 (2)	0.172 (2)	0.476 (2)	0.02 (1)
C(66)	0.572 (1)	0.178 (1)	0.266 (1)	0.04 (1)
C(67)	0.570 (2)	0.157 (3)	0.555 (3)	0.09 (2)
B(2)	$\frac{1}{2}$	$\frac{1}{2}$	0.386 (1)	0.016 (8)
F(21)	$\frac{1}{2}$	$\frac{1}{2}$	0.3038 (5)	0.075 (4)
F(22)	0.3838 (2)	0.6867 (3)	0.4141 (4)	0.076 (3)

Table 2. Selected data on the geometry for molecule 1

Bond distances (Å)					
Cu(1)—N(12)	1.93 (1)	C(13)—C(14)	1.40 (2)		
Cu(1)—N(22)	1.95 (1)	C(13)—C(16)	1.49 (2)		
Cu(1)—N(32)	2.00 (1)	C(14)—C(15)	1.41 (2)		
Cu(1)···Cu(1')	2.893 (4)	F(13)—B(1)	1.32 (3)		
Cu(1)—F(1)	2.166 (6)	F(21)—B(2)	1.41 (2)		
Cu(1')—F(1)	1.876 (6)	C(15)—C(17)	1.51 (1)		
N(11)—N(12)	1.37 (1)	C(23)—C(24)	1.35 (2)		
N(11)—C(15)	1.33 (2)	C(23)—C(26)	1.56 (2)		
N(12)—C(13)	1.33 (2)	C(24)—C(25)	1.42 (2)		
F(11)—B(1)	1.37 (2)	C(25)—C(27)	1.47 (2)		
F(14)—B(1)	1.43 (2)	C(33)—C(34)	1.41 (2)		
N(21)—N(22)	1.39 (1)	C(33)—C(36)	1.47 (2)		
N(21)—C(25)	1.37 (2)	C(34)—C(35)	1.42 (2)		
N(22)—C(23)	1.33 (2)	C(35)—C(37)	1.47 (2)		
N(31)—N(32)	1.40 (1)	F(12)—B(1)	1.46 (2)		
N(31)—C(35)	1.34 (2)	F(22)—B(2)	1.350 (9)		
N(32)—C(33)	1.33 (2)				
Bond angles (°)					
F(1)—Cu(1)—N(12)	90.6 (4)	N(12)—Cu(1)—N(32)	94.5 (4)		
F(1)—Cu(1)—N(22)	104.3 (3)	F(1')—Cu(1)—N(12)	179.1 (4)		
F(1)—Cu(1)—N(32)	103.5 (3)	N(22)—Cu(1)—N(32)	151.2 (4)		
F(1)—Cu(1)—F(1')	88.9 (3)	F(1')—Cu(1)—N(22)	86.7 (3)		
N(12)—Cu(1)—N(22)	92.7 (5)	F(1')—Cu(1)—N(32)	86.3 (3)		
Torsion angles (°)					
F(1)—Cu(1)—N(12)—N(11)	3 (1)	F(1')—Cu(1)—N(22)—N(21)	-50.8 (8)		
N(22)—Cu(1)—N(12)—N(11)	108 (1)	F(1)—Cu(1)—N(32)—N(31)	133.3 (9)		
N(32)—Cu(1)—N(12)—N(11)	-100 (1)	N(12)—Cu(1)—N(32)—N(31)	-135.0 (9)		
F(1)—Cu(1)—N(22)—N(21)	-138.8 (8)	N(22)—Cu(1)—N(32)—N(31)	-31 (1)		
N(12)—Cu(1)—N(22)—N(21)	129.9 (9)	F(1')—Cu(1)—N(32)—N(31)	45.3 (9)		
N(32)—Cu(1)—N(22)—N(21)	25 (1)				
Hydrogen bonds (Å, °)					
	N···F	N—H	H···F	N—H···F	F···H···F
N(11)—H(11)···F(1)	2.97 (1)	0.88 (9)	2.32 (9)	131 (7)	
N(11)—H(11)···F(14')	2.81 (1)	0.88 (9)	2.10 (9)	136 (8)	93 (3)
N(21)—H(21)···F(1 <sup>ii</sup> )	2.87 (1)	0.87 (9)	2.45 (9)	110 (6)	
N(21)—H(21)···F(13 <sup>iii</sup> )	2.83 (1)	0.87 (9)	2.09 (7)	142 (8)	102 (4)
N(31)—H(31)···F(1 <sup>ii</sup> )	2.86 (1)	0.91 (8)	2.49 (8)	105 (6)	
N(31)—H(31)···F(12 <sup>iii</sup> )	2.79 (1)	0.91 (8)	1.96 (8)	152 (8)	98 (3)
Symmetry code: (i) $x-y, x, -z$ ; (ii) $\frac{1}{2}-x, \frac{3}{2}-y, \frac{1}{2}-z$ ; (iii) $\frac{1}{2}-x+y, \frac{3}{2}-x, \frac{1}{2}+z$ ; (') $\frac{1}{2}-x, \frac{3}{2}-y, \frac{1}{2}-z$ .					
Relevant data on the geometry of four very similar fluoro-bridged copper(II) compounds (Å, °)					
	I	II	III	IV	
Cu···Cu <sup>ii</sup>	3.0141 (8)	2.9962 (9)	3.131 (3)	2.893 (4)	
Cu—F	2.183 (2)	2.195 (2)	2.258 (8)	2.166 (6)	
Cu—F <sup>ii</sup>	1.911 (2)	1.901 (2)	1.904 (2)	1.876 (6)	
Cu—N	2.013 (3)	1.984 (4)	1.973 (4)	1.93 (1)	
	2.009 (3)	1.997 (3)	1.977 (2)	1.95 (1)	
	1.965 (3)	1.964 (3)	1.978 (3)	2.00 (1)	
Cu—F—Cu <sup>ii</sup>	94.59 (7)	93.73 (8)	97.19 (8)	91.1 (2)	
(I) $[\text{Cu}_2\text{F}_2(\text{mpz})_6]^{2+}$ (Rietmeijer <i>et al.</i> , 1984); (II) $[\text{Cu}_2\text{F}_2(\text{mpz})_4(\text{dmpz})_2]^{2+}$ (Rietmeijer <i>et al.</i> , 1984); (III) $[\text{Cu}_2\text{F}_2(\text{mpz})_2(\text{dmpz})_4]^{2+}$ (Ten Hoedt <i>et al.</i> , 1981); (IV) title compound.					

**Discussion.** Fig. 1 shows the geometry of the [Cu<sub>2</sub>F<sub>2</sub>(dmpz)<sub>6</sub>]<sup>2+</sup> cation.

The cations are dimeric, consisting of two [CuF(dmpz)<sub>3</sub>]<sup>+</sup> units related by an inversion centre, and are joined into neutral globular clusters with two BF<sub>4</sub><sup>-</sup> anions. The units are connected by two asymmetric fluoro bridges with Cu—F bond lengths 2.166 (6) and 1.876 (6) Å in the ordered molecule. The coordination of the Cu atom may be viewed as square-based pyramidal with an F atom in apical position and a second F atom and three coordinating N atoms at the corners of the square base. The aromatic rings of the ligands are planar. Pyrazole ring [N(11)—C(15)] is almost coplanar with the Cu<sub>2</sub>F<sub>2</sub> plane [dihedral angle 2.0 (6)°]. The molecular geometry of the cation closely corresponds to the geometry of the fluoro-bridged copper(II) compounds described by Rietmeyer *et al.* (1984) and Ten Hoedt *et al.* (1981). Relevant data on these compounds are presented in Table 2.

Each N—H atom of the ligands is involved in an asymmetric bifurcated hydrogen bond to the bridging F and one of the F atoms of the BF<sub>4</sub><sup>-</sup> anion respectively (Table 2) (see Scheme in *Introduction*). The sums of the angles round the N—H atoms [360 (10)° for H(11), 354 (10)° for H(21) and 355 (10)° for H(31)] illustrate the planar geometry of the bifurcated hydrogen bonds. The boron atoms and the two bridging fluorine ions of a dimer lie on a straight line parallel to the *c* axis.

The crystal structure (Fig. 2) consists of an *R*-centred unit cell containing 12 dimeric cations and 24 BF<sub>4</sub><sup>-</sup> anions. Nine ordered cations are on  $\bar{1}$  sites, three disordered cations on  $\bar{3}$  sites, 18 BF<sub>4</sub><sup>-</sup> anions on general positions and six BF<sub>4</sub><sup>-</sup> anions on threefold axes. The relation between the ordered and disordered molecules explains the pseudosymmetry: except for a small rotation angle, 4.0 (6)°, they are translated by  $\frac{1}{2}a$ . The external pseudo-cube faces (021,  $\bar{2}01$ ,  $2\bar{2}1$ , angle 91°) correspond to close-packed planes of the globular molecules which form an approximate cubic close packing.

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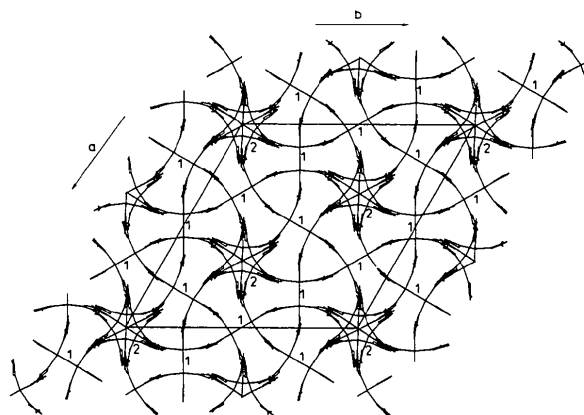


Fig. 2. A view of the unit-cell contents down the *c* axis showing all possible orientations of the disordered molecules superimposed. The ordered cations are marked with 1 and the disordered cations are marked with 2. From this illustration it is clear that the disordered molecules can occupy all three orientations without disrupting the packing of the molecules in the crystal. The BF<sub>4</sub><sup>-</sup> anions that project to positions coinciding with the centre of the molecule have been omitted for clarity.

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## The Na<sup>+</sup> Complex of the Ionophore A204A

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**Abstract.** Na[C<sub>49</sub>H<sub>83</sub>O<sub>17</sub>].C<sub>3</sub>H<sub>6</sub>O, *M<sub>r</sub>* = 1025, monoclinic, *C*2, *a* = 27.223 (7), *b* = 14.529 (3), *c* = 14.369 (3) Å, β = 91.18 (2)°, *V* = 5682 Å<sup>3</sup>, *Z* = 4,

*D<sub>x</sub>* = 1.19 g cm<sup>-3</sup>, Mo *Kα*, λ = 0.71069 Å, μ = 0.848 cm<sup>-1</sup>, *F*(000) = 2220, *T* = 295 K, *R* = 0.066 for 5340 observed data. A previously reported deter-

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