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The Crystal Structure of (+)-*cis*-9-(3-Dimethylaminopropyl)-10-methyl-2-(trifluoromethyl)-9,10-dihydroanthracene Hydrochloride Monohydrate, SKF d-28175, Acetone Solvate

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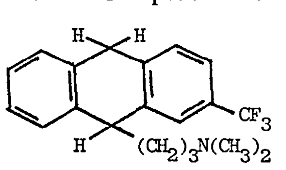
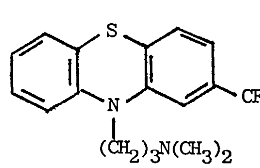
The crystal structure of (+)-*cis*-9-(3-dimethylaminopropyl)-10-methyl-2-(trifluoromethyl)-9,10-dihydroanthracene hydrochloride monohydrate acetone solvate, $C_{21}H_{24}NF_3.HCl.H_2O.C_3H_6O$, has been determined by the direct method. The refinement was carried out by the block-diagonal least-squares method with anisotropic temperature factors based on three-dimensional data to give a final R value of 0·062 for 2175 reflections. The crystal is monoclinic, and the space group is $P2_1$,with $Z=4$. The unit-cell parameters are $a=12·833$ (4), $b=28·595$ (7), $c=7·232$ (2) Å, and $\beta=110·03$ (2)°. There are two crystallographically independent molecules in one asymmetric unit of the crystal, and the two molecules do not have the same conformation. The best planes of the benzene rings make dihedral angles of 155·7° and 155·6° in the two molecules. Both the substituents at C(9) and C(10) are in the 'boat-axial' conformation with respect to the central dihydroanthracene ring. All the bond lengths and bond angles are within the normal range of magnitude. The packing of molecules in the crystal is determined by the hydrogen bonding and van der Waals interactions. Each chloride ion is associated with three hydrogen bonds; one links to a quaternary ammonium ion, and the other two link to two different water molecules.

Introduction

9-(3-Dimethylaminopropyl)-2-(trifluoromethyl)-9,10-dihydroanthracene (I) is a dihydroanthracene analog of triflupromazine (II), a phenothiazine derivative. Triflupromazine is a potent antipsychotic agent (Domino, 1967; Zirkle & Kaiser, 1970), and the replacement of the S and N atoms with various other atoms results in no appreciable loss of antipsychotic activity (Zirkle & Kaiser, 1970). The substitution of a methyl group at the 10-position of (I) produces 9-(3-dimethylaminopropyl)-10-methyl-2-(trifluoromethyl)-9,10-dihydroanthracene (III), which may exist as *cis* and *trans* isomers (Fig. 1). The *trans* isomer of (III) has very little antipsychotic activity (Zirkle & Kaiser, 1974). However, the *cis* isomer of (III) shows neuroleptic properties in animals, although it is generally less potent than (I) (Fowler, Tedeschi, Zirkle & Macko, 1971). In this work, the crystal structure of the *cis* isomer of (III) has been studied to determine the effect of the 10-methyl substituent on the conformation of the molecule. For comparison, the crystal structure of (I) will be studied to determine if the decrease in neuroleptic activity is due to the steric effect of the methyl group which inhibits the interaction of the tricyclic molecule with the receptor sites.



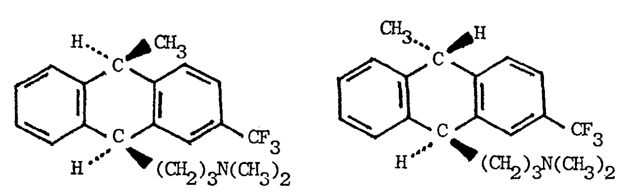
(I)



(II)

Experimental

Samples of SKF d-28175 [the hydrochloride monohydrate of the *cis* isomer of (III)] were obtained through the courtesy of Dr C. L. Zirkle of Smith, Kline and French Laboratories. Transparent, prismatic crystals of this material were grown from an acetone solution under refrigeration in the form of an acetone solvate. The unit-cell parameters were obtained from the least-squares fitting of carefully centered reflections. The space group is $P2_1$ or $P2_1/m$ from the systematic extinction: $0k0$ absent when $k=2n+1$. $P2_1$ is correct because only the (+) isomer of the molecule is present in the crystal. $P2_1/m$ is not possible from the packing of the molecules and the unit-cell dimensions. The crystal data are summarized in Table 1. The intensity data were collected on a Datex-Syntex automatic diffractometer. A $\theta/2\theta$ time variable scanning mode with Zr-filtered Mo $K\alpha$ radiation was used to measure 3343 independent reflections with 2θ values below 45°, of which 2175 reflections were considered as observed. A reflection was considered as observed if its intensity



(III) *cis* (III) *trans*

Fig. 1. The *cis* and *trans* isomers of 9-(3-dimethylaminopropyl)-10-methyl-2-(trifluoromethyl)-9,10-dihydroanthracene (III).

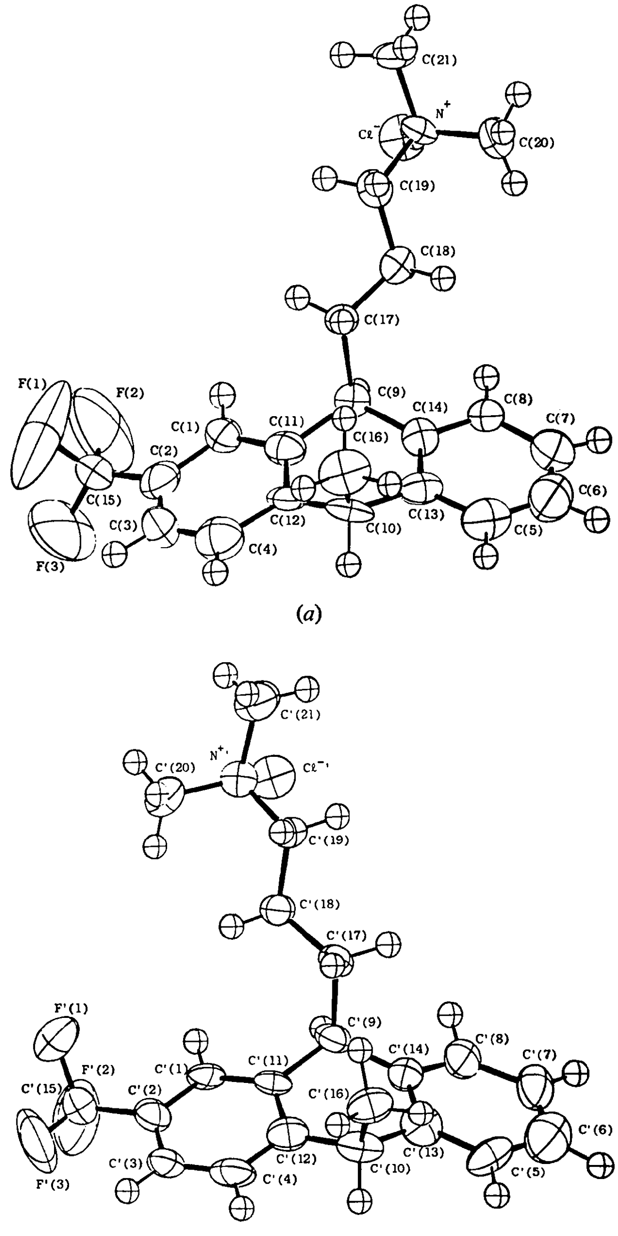


Fig. 2. The configuration of the two crystallographically independent molecules, (*a*) main molecule *A* and (*b*) main molecule *B*, of SKF d-28175 in *ORTEP* drawing (Johnson, 1965).

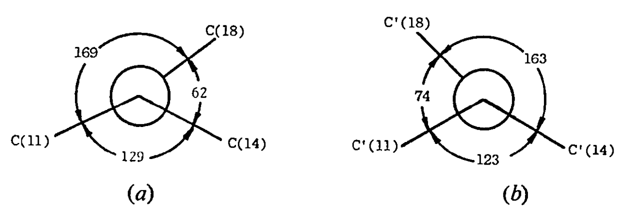


Fig. 3. Comparison of the torsion angles between (*a*) C(9)–C(17) and (*b*) C′(9)–C′(17) bonds in molecules *A* and *B*, respectively.

was greater than $2\sigma(I)$, where $\sigma(I)$ was determined from counting statistics. The intensity data were reduced to structure factors by the application of Lorentz and polarization factors; no absorption corrections were applied.

Table 1. *Crystal data*

$C_{21}H_{24}NF_3.HCl.H_2O.C_3H_6O$	F.W. 459·98
Space group: $P2_1$	
$a=12·833$ (4) Å	$Z=4$
$b=28·595$ (7)	$V=2493·3$ Å^3
$c=7·232$ (2)	μ(Mo $K\alpha$)$=2·04$ cm^{-1}
$\beta=110·03$ (2)°	λ(Mo $K\alpha$)$=0·7107$ Å
$D_m=1·21$ g cm^{-3}	(by flotation in carbon tetrachloride and toluene mixture)
$D_x=1·23$ g cm^{-3}	with one molecule of acetone of crystallization per molecule of SKF d-28175 (verified from structure determination)
Crystal size:	0·15 × 0·21 × 0·09 mm

Structure determination and refinement

The presence of four molecular units of SKF d-28175 in a unit cell with space group $P2_1$ requires two crystallographically independent molecules per asymmetric unit. The structure was determined by the application of direct methods using the weighted multisolution tangent refinement technique (Germain, Main & Woolfson, 1971). The distribution of normalized structure factors is listed in Table 2.

Table 2. *Distribution of normalized structure factors*

		Theoretical	
	Experimental	Centric	Acentric
$\|E\|>1·0$ (%)	31·7	31·7	36·8
$\|E\|>2·0$ (%)	4·1	4·6	1·8
$\|E\|>3·0$ (%)	0·4	0·3	0·01
$\langle\|E\|\rangle$	0·829	0·798	0·886
$\langle\|E\|^2\rangle$	1·004	1·000	1·000
$\langle\|E^2-1\|\rangle$	0·927	0·968	0·736

The E map calculated from 369 reflections with $|E|\geq 1·5$ showed the positions of two Cl atoms and eight C atoms. The remaining non-hydrogen atoms were located by successive Fourier syntheses. It is apparent from the Fourier syntheses that there is one acetone molecule of crystallization with each molecule of SKF d-28175. The refinement was carried out by the block-diagonal least-squares method with anisotropic temperature factors. The unobserved reflections were given zero weight in the refinement. The positions of the H atoms were calculated with reasonable bond lengths and bond angles with respect to the atoms to which they are bonded. The H atoms were included in the structure factor calculations but their parameters were not refined; their thermal parameters were assigned the same values as those of the atoms to which they are bonded. Cruickshank's (1965) weighting scheme was used, and the weight of the reflections was calculated according to the formula $1/w=6·5-$

Table 3. *Fractional atomic coordinates* ($\times 10^4$) *and thermal parameters* ($Å^2 \times 10^3$) *with standard deviations in parentheses*

The expression for the temperature factor exponent consistent with U values is
$$[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{12}hka^*b^*+2U_{13}hla^*c^*+2U_{23}klb^*c^*)]\,.$$

Main molecule *A*	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl	2988 (2)	0 (0)	118 (3)	92 (1)	86 (1)	47 (1)	1 (1)	31 (0)	1 (1)
F(1)	8723 (7)	1721 (6)	7304 (17)	140 (7)	355 (17)	284 (12)	150 (10)	90 (8)	117 (13)
F(2)	7896 (7)	1717 (5)	4587 (17)	148 (6)	268 (12)	324 (11)	−94 (8)	165 (7)	−142 (10)
F(3)	8542 (8)	2316 (3)	5887 (23)	161 (7)	141 (6)	517 (20)	−45 (6)	228 (11)	−22 (9)
N	2723 (4)	−35 (2)	4175 (8)	57 (3)	30 (2)	58 (3)	−17 (2)	26 (2)	−5 (2)
C(1)	6064 (6)	1809 (3)	5583 (11)	55 (4)	44 (4)	68 (4)	−2 (3)	24 (3)	7 (3)
C(2)	7037 (6)	2057 (3)	6585 (14)	63 (5)	34 (4)	120 (7)	12 (3)	39 (4)	19 (4)
C(3)	7069 (7)	2356 (3)	8004 (16)	60 (4)	57 (5)	133 (8)	−10 (4)	15 (5)	1 (5)
C(4)	6184 (8)	2448 (3)	8635 (13)	99 (6)	66 (5)	69 (5)	−1 (4)	26 (4)	−6 (4)
C(5)	2137 (7)	2438 (3)	6567 (13)	103 (6)	58 (5)	87 (6)	9 (4)	54 (5)	0 (4)
C(6)	1120 (7)	2336 (3)	5084 (15)	71 (5)	88 (6)	114 (7)	9 (5)	43 (5)	25 (5)
C(7)	1055 (7)	2039 (3)	3682 (15)	80 (5)	80 (5)	91 (6)	−8 (4)	19 (5)	13 (5)
C(8)	2005 (6)	1793 (2)	3648 (11)	57 (4)	41 (4)	64 (4)	1 (3)	21 (3)	1 (3)
C(9)	4058 (5)	1644 (2)	5067 (8)	55 (4)	48 (4)	26 (3)	−7 (3)	23 (3)	−8 (2)
C(10)	4199 (7)	2326 (2)	8214 (12)	86 (5)	27 (3)	78 (5)	−23 (3)	37 (4)	−19 (3)
C(11)	5124 (6)	1915 (2)	6068 (10)	64 (4)	43 (4)	48 (4)	−4 (3)	9 (3)	−1 (3)
C(12)	5142 (6)	2225 (2)	7508 (10)	58 (4)	19 (3)	60 (4)	−10 (3)	6 (3)	0 (3)
C(13)	3101 (7)	2223 (3)	6601 (11)	77 (5)	49 (4)	66 (4)	−3 (3)	51 (4)	3 (3)
C(14)	3044 (6)	1897 (3)	5119 (10)	57 (4)	58 (4)	45 (3)	−4 (3)	21 (3)	2 (3)
C(15)	8015 (7)	1971 (3)	5967 (21)	50 (4)	62 (5)	194 (12)	−8 (4)	31 (6)	−10 (6)
C(16)	4275 (8)	2083 (3)	10128 (12)	118 (7)	75 (5)	52 (4)	4 (5)	38 (4)	−2 (4)
C(17)	4137 (5)	1135 (2)	5835 (9)	46 (3)	37 (3)	36 (3)	−6 (2)	17 (3)	0 (3)
C(18)	3230 (5)	809 (3)	4718 (9)	45 (4)	59 (4)	42 (3)	12 (3)	19 (3)	9 (3)
C(19)	3482 (6)	313 (2)	5437 (9)	56 (4)	56 (4)	33 (3)	−4 (3)	21 (3)	3 (3)
C(20)	1549 (7)	46 (3)	3891 (16)	56 (4)	62 (5)	128 (7)	−5 (4)	34 (5)	4 (5)
C(21)	3043 (6)	−521 (2)	4951 (11)	77 (5)	36 (4)	63 (4)	−2 (3)	19 (4)	10 (3)
Main molecule *B*									
Cl′	3168 (2)	5570 (1)	−4725 (3)	85 (1)	73 (1)	50 (1)	1 (1)	32 (0)	2 (1)
F′(1)	−369 (5)	3936 (2)	−2383 (10)	88 (3)	80 (3)	170 (5)	27 (2)	2 (3)	3 (3)
F′(2)	−117 (5)	3409 (3)	−4079 (9)	99 (4)	250 (8)	101 (4)	41 (5)	−21 (3)	−71 (5)
F′(3)	−776 (5)	3255 (3)	−1929 (16)	71 (3)	189 (7)	281 (9)	−57 (4)	−3 (4)	102 (7)
N′	2653 (4)	5579 (2)	−830 (7)	60 (3)	47 (3)	37 (2)	−3 (2)	19 (2)	−9 (2)
C′(1)	1967 (5)	3668 (2)	−1070 (9)	60 (4)	26 (3)	42 (3)	−1 (3)	14 (3)	13 (2)
C′(2)	1112 (6)	3425 (3)	−893 (10)	66 (4)	55 (4)	55 (4)	−10 (3)	23 (3)	−12 (3)
C′(3)	1286 (6)	3101 (2)	615 (11)	65 (4)	36 (4)	76 (5)	−11 (3)	33 (4)	−1 (3)
C′(4)	2339 (7)	3035 (2)	1913 (12)	91 (5)	31 (4)	87 (5)	−6 (3)	51 (4)	7 (3)
C′(5)	6337 (6)	3140 (3)	3259 (9)	82 (5)	59 (4)	23 (3)	29 (4)	3 (3)	3 (3)
C′(6)	7203 (8)	3259 (4)	2707 (14)	82 (6)	101 (7)	80 (5)	20 (5)	7 (4)	−33 (5)
C′(7)	7024 (6)	3587 (3)	1153 (11)	53 (4)	83 (6)	68 (5)	4 (4)	16 (3)	−19 (4)
C′(8)	5960 (6)	3762 (3)	283 (11)	56 (4)	57 (4)	66 (4)	−4 (3)	−2 (3)	−30 (4)
C′(9)	3993 (6)	3903 (2)	−30 (10)	58 (4)	28 (3)	51 (4)	−11 (3)	8 (3)	−12 (3)
C′(10)	4373 (6)	3204 (2)	3196 (8)	90 (5)	42 (3)	17 (3)	−5 (3)	7 (3)	−3 (2)
C′(11)	3036 (5)	3621 (2)	230 (9)	60 (4)	15 (2)	48 (3)	−4 (2)	16 (3)	−12 (2)
C′(12)	3238 (6)	3289 (2)	1734 (9)	78 (4)	40 (4)	37 (3)	6 (3)	14 (3)	−3 (3)
C′(13)	5305 (6)	3345 (3)	2489 (9)	69 (4)	72 (5)	26 (3)	0 (4)	3 (3)	−12 (3)
C′(14)	5088 (5)	3669 (2)	939 (8)	55 (3)	34 (3)	33 (3)	3 (3)	13 (2)	−6 (3)
C′(15)	−39 (6)	3494 (3)	−2277 (14)	47 (4)	71 (5)	116 (7)	−13 (4)	2 (4)	−27 (5)
C′(16)	4516 (7)	3467 (3)	5163 (10)	92 (5)	58 (4)	36 (3)	17 (4)	15 (3)	1 (3)
C′(17)	4069 (5)	4393 (2)	872 (9)	55 (4)	43 (3)	40 (3)	−11 (3)	13 (3)	5 (3)
C′(18)	3171 (5)	4729 (2)	−344 (9)	56 (4)	23 (3)	45 (3)	2 (3)	17 (3)	2 (3)
C′(19)	3396 (5)	5213 (2)	478 (9)	52 (4)	32 (3)	46 (3)	−1 (3)	11 (3)	0 (3)
C′(20)	1465 (7)	5498 (3)	−1196 (13)	70 (5)	70 (5)	82 (5)	16 (4)	11 (4)	−14 (4)
C′(21)	2966 (7)	6052 (3)	−62 (13)	87 (6)	49 (4)	88 (6)	10 (4)	23 (5)	0 (4)
Acetone molecule *A*									
O(*A*)	622 (5)	947 (3)	287 (11)	103 (5)	156 (7)	122 (5)	−41 (4)	60 (4)	13 (5)
C(*A*)1	787 (7)	978 (3)	−1257 (14)	83 (5)	94 (6)	78 (5)	−6 (5)	41 (4)	−3 (5)
C(*A*)2	1718 (11)	1251 (5)	−1428 (17)	140 (10)	170 (11)	101 (8)	18 (8)	78 (7)	39 (8)
C(*A*)3	87 (10)	763 (7)	−2993 (23)	104 (9)	229 (20)	173 (12)	−3 (10)	36 (8)	−116 (14)
Acetone molecule *B*									
O′(*A*)	774 (5)	4602 (2)	5405 (8)	91 (3)	100 (4)	73 (3)	−9 (3)	33 (3)	−6 (3)
C′(*A*)1	765 (7)	4441 (3)	3854 (12)	88 (5)	88 (6)	59 (5)	8 (5)	17 (4)	−26 (5)
C′(*A*)2	1814 (9)	4322 (5)	3657 (16)	88 (7)	178 (11)	94 (7)	7 (7)	40 (6)	−47 (8)
C′(*A*)3	−204 (11)	4349 (9)	2373 (27)	108 (9)	384 (27)	202 (17)	41 (14)	−27 (10)	−189 (19)
Water molecules									
O(*W*)	4652 (6)	884 (2)	1138 (10)	121 (4)	62 (3)	102 (4)	−2 (3)	49 (3)	26 (3)
O′(*W*)	4730 (5)	4698 (2)	−3579 (10)	88 (4)	69 (4)	122 (4)	−8 (3)	49 (3)	−17 (3)

Table 3 (*cont.*)

Hydrogen atomic coordinates ($\times 10^3$)

Main molecule *A*	*x*	*y*	*z*	Main molecule *B*	*x*	*y*	*z*
H(N)	279	0	308	H′(N)	271	553	−233
H(1)	603	156	454	H′(1)	184	390	−215
H(3)	779	249	868	H′(3)	65	292	71
H(4)	630	265	979	H′(4)	248	281	302
H(5)	219	267	760	H′(5)	642	289	428
H(6)	44	249	515	H′(6)	795	309	342
H(7)	31	200	267	H′(7)	763	366	70
H(8)	195	156	261	H′(8)	579	403	−65
H(9)	396	159	368	H′(9)	383	396	−154
H(10)	428	266	841	H′(10)	433	285	339
H(16)1	420	175	980	H′(16)1	440	382	490
H(16)2	357	218	1041	H′(16)2	531	341	608
H(16)3	490	217	1120	H′(16)3	403	335	582
H(17)1	413	115	703	H′(17)1	402	436	210
H(17)2	479	97	579	H′(17)2	488	448	121
H(18)1	313	82	331	H′(18)1	245	463	−36
H(18)2	252	91	489	H′(18)2	322	473	−175
H(19)1	424	23	549	H′(19)1	328	522	176
H(19)2	344	29	676	H′(19)2	416	530	68
H(20)1	115	−23	300	H′(20)1	133	550	7
H(20)2	131	33	330	H′(20)2	127	516	−176
H(20)3	144	1	517	H′(20)3	102	571	−212
H(21)1	383	−54	526	H′(21)1	248	623	−115
H(21)2	264	−75	405	H′(21)2	375	611	13
H(21)3	286	−57	616	H′(21)3	282	610	115
Acetone molecule *A*				Acetone molecule *B*			
H(*A*)1	153	136	−277	H′(*A*)1	193	457	265
H(*A*)2	236	108	−102	H′(*A*)2	178	403	299
H(*A*)3	180	153	−60	H′(*A*)3	242	434	477
H(*A*)4	−63	73	−295	H′(*A*)4	−45	402	233
H(*A*)5	38	44	−301	H′(*A*)5	−85	453	246
H(*A*)6	10	92	−406	H′(*A*)6	−17	442	104
Water molecules							
H(*W*)1	417	57	63	H′(*W*)1	422	509	−395
H(*W*)2	526	75	238	H′(*W*)2	541	489	−252

$0{\cdot}785|F_o|+0{\cdot}026|F_o|^2$. The quantity $\sum w\{||F_o|-|F_c||\}^2$ was minimized. The magnitude, $\{\sum(F_o-F_c)^2/(m-n)\}^{1/2}$, where m is the number of reflections and n the number of parameters refined, was 0·89. The final R index, $(\sum||F_o|-|F_c||/\sum|F_o|)$, was 0·062 for observed reflections. There was no residual electron density above 0·3 e Å^{-3}. The atomic scattering factors used for Cl^-, F, O, N, and C atoms were those from *International Tables for X-ray Crystallography* (1962). For H, the values given by Stewart, Davidson & Simpson (1965) were used. The final positional and thermal parameters are given in Table 3.*

The computer programs used in this analysis were a block-diagonal least-squares program (Shiono, 1971), the Zalkin Fourier synthesis program modified by Dr R. Shiono of the University of Pittsburgh, *MULTAN* (Germain, Main & Woolfson, 1971) and a number of structure interpretation programs (Shiono, 1971; Chu, 1973). All calculations were carried out on a CDC CYBER 72 computer in the Bradfield Computing Laboratory at Southern Methodist University.

* A table of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31301 (18 pp., 1 microfiche). Copies may be obtained from The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Description of the structure

The configuration of the two crystallographically independent molecules of SKF d-28175, *A* and *B*, and the identification of the atoms are shown in Fig. 2. The two molecules do not have the same conformation, and the 9-(3-dimethylaminopropyl) group propagates toward the two different benzene rings for the two molecules. This is illustrated by the torsion angles about the C(9)–C(17) bond in both molecules (Fig. 3). The 3-dimethylaminopropyl and trifluoromethyl groups are in a *trans* configuration about C(9)–C(17) in molecule *A*, while in molecule *B*, the two groups are in a *cis* configuration about C′(9)–C′(17) (Fig. 2). A similar relation was also observed between the two crystallographically independent molecules of triflupromazine (II) (Phelps & Cordes, 1974). An attempt to determine the absolute configuration of the molecule was not successful. The refinement of the atomic parameters reported in Table 3 and the refinement of the atomic parameters with opposite y values gave the same R value. The calculated values of $\{I(hkl)-I(h\bar{k}l)\}/I(hkl)$ for ten Bijvoet pairs of strong reflections with Cu $K\alpha$ radiation are in the range of $5{\cdot}7\times10^{-4}$ to $1{\cdot}5\times10^{-2}$. This is due apparently to the presence of the pseudo-center of symmetry relation between the two crystallographically independent molecules. (The two molecules, with the exception of the trifluoromethyl group, are approximately related by the symmetry elements of the space group $P2_1/c$.) This also explains the facts that the distribution of the normalized structure factors are close to the centrosymmetric structure, and that there are few observed $h0l$ reflections with l odd.

The bond lengths and bond angles with their standard deviations are shown in Fig. 4. The mean value of the six C–F bond lengths is 1·26 (2) Å, which is significantly shorter than the bond length for the C–F single bond. This can be attributed to the large thermal motion of the F atoms (Fig. 2), which, together with the short C–F bond length, was also observed in triflupromazine (Phelps & Cordes, 1974) and in other compounds with the trifluoromethyl group (Cotton & Norman, 1972; Johnston, Rohrbaugh & Horrocks, 1971).

Both the 9-(3-dimethylaminopropyl) and 10-methyl groups in SKF d-28175 are in the 'boat-axial' conformation. The C(17)–C(9)···C(10) and C(16)–C(10)··· C(9) angles are 111° and 112°, respectively, for molecule *A*, and those for molecule *B* are 108° and 109°, respectively. The 'boat-axial' conformation was also observed for both 9-methyl and 10-ethyl groups in the crystal structure of *cis*-9-methyl-10-ethyl-9,10-dihydroanthracene (Bordner, Stanford & Zieger, 1973). Crystal structure studies have also shown that the 9-isopropyl group is in a 'boat-axial' and the 10-methyl group is in a 'boat-equatorial' conformation in *trans*-9-isopropyl-10-methyl-9,10-dihydroanthracene (Stanford, 1973), and that the 9-t-butyl group is in a 'boat-axial' conformation in 9-t-butyl-9,10-dihydroanthracene (Brennan,

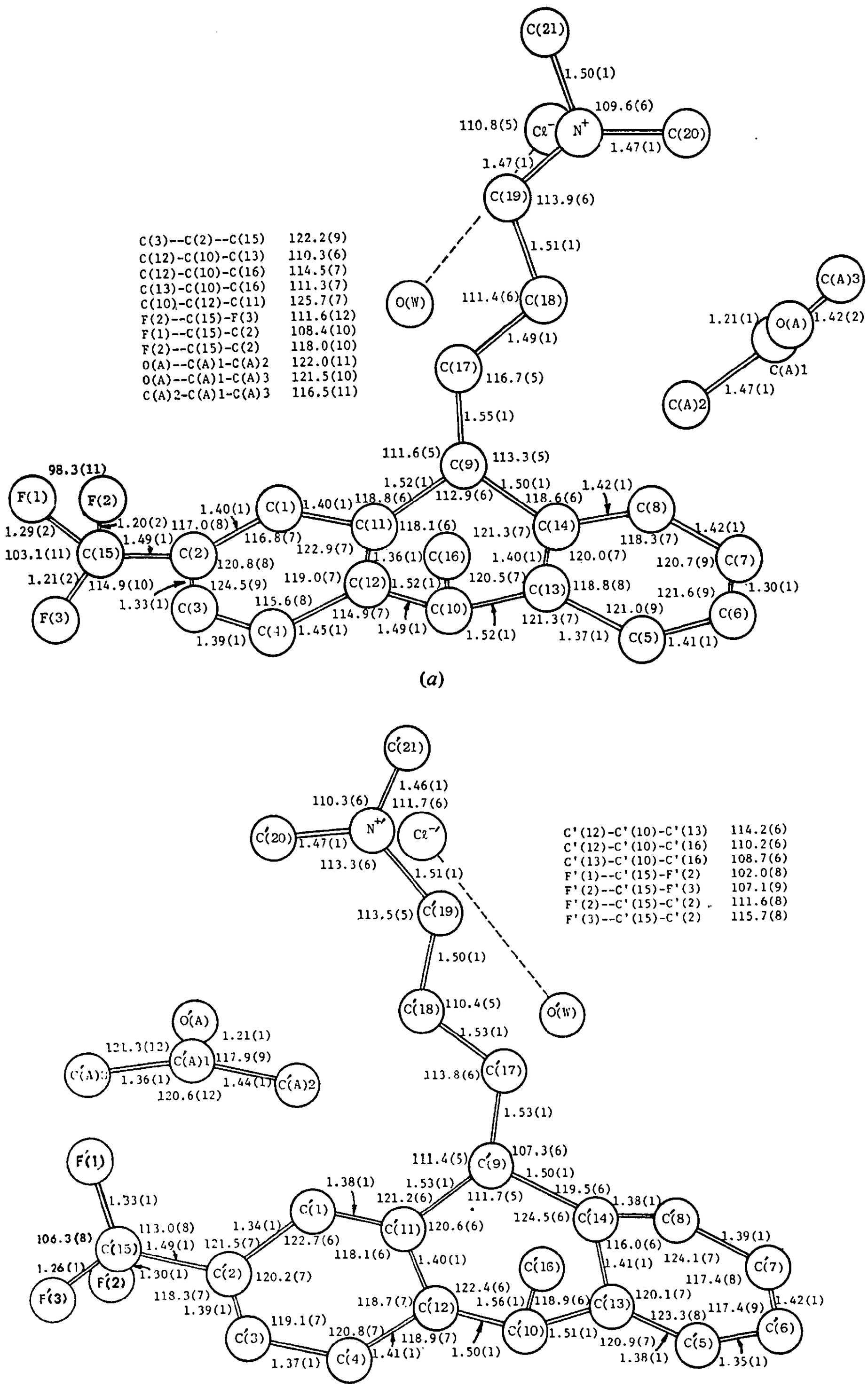


Fig. 4. The bond lengths (Å) and bond angles (°), with their e.s.d.'s in parentheses, of the two crystallographically independent molecules *A*(*a*) and *B*(*b*).

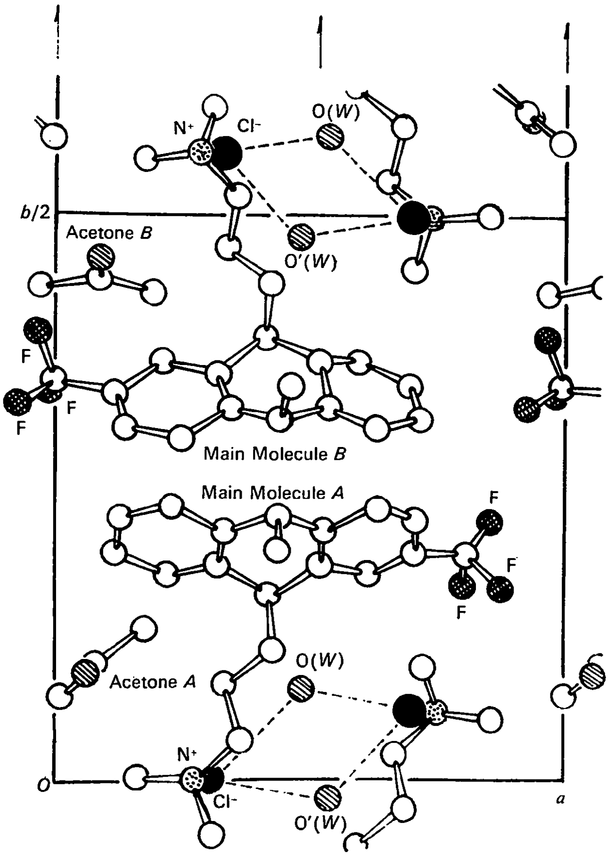


Fig. 5. The molecular packing diagram of the SKF d-28175 molecules, excluding H atoms, in the unit cell projected down **c**. The dashed lines are hydrogen bonds.

Putkey & Sundaralingam, 1971). These results have confirmed the conformation studies of the *meso*-substituted 9,10-dihydroanthracenes carried out by n.m.r. nuclear Overhauser enhancement technique (Brinkman, Gordon, Hawey, Rabideau, Stothers & Ternay, 1970).

The dihedral angles between the least-squares planes of the two benzene rings in SKF d-28175 are 155·7° and 155·6° for molecules *A* and *B*, respectively, compared with 134·4° and 141·0° for the two crystallographically independent molecules in triflupromazine (II). A comparison of the dihedral angles in dihydroanthracene derivatives is given in Table 4.

There are three hydrogen bonds within one molecular unit of SKF d-28175. One is formed between the quaternary ammonium ion and chloride ion, and the other two link the chloride ion with the two water molecules. The packing of the molecules in the crystal is illustrated in Fig. 5. Each chloride ion is associated with three hydrogen bonds, and each water molecule has two hydrogen bonds. The hydrogen-bond distances and angles are shown in Table 5. The non-bonded distances less than 3·5 Å are also listed in Table 5. There is no close contact between the acetone and the main molecule.

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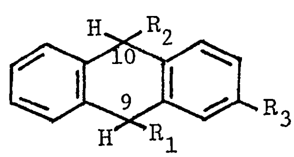
Table 4. *Comparison of dihedral angles in dihydroanthracene derivatives*



Compound	R_1	R_2	R_3	Average intra-ring angle at C(9) and C(10)(°)	Dihedral angle (°)	References
9-t-Butyl-9,10-dihydroanthracene*	$C(CH_3)_3$	H	H		147	Brennan, Putkey & Sundaralingam (1971)
cis-9-Methyl-10-ethyl-9,10-dihydroanthracene	CH_3	CH_2CH_3	H	112	152	Bordner & Stanford (1973)
trans-9-Isopropyl-10-methyl-9,10-dihydroanthracene	$CH(CH_3)_2$	CH_3	H	108	129	Stanford (1973)
(+)-*cis*-9-(3-Dimethylaminopropyl)-10-methyl-2-(trifluoromethyl)-9,10-dihydroanthracene	$(CH_2)_3N(CH_3)_2$	CH_3	CF_3	112	155	This work

* The average intraring angle at C(9) and C(10) for 9-t-butyl-9,10-dihydroanthracene was not reported and the atomic parameters also were not given in the published paper.

Table 5. *Hydrogen-bond distances and angles*

i	*j*	*k*	*D*(*ij*)	*D*(*jk*)	∠*ijk*
N	Cl	O(*W*)	3·07 Å	3·23 Å	95·1°
N	Cl	O′(*W*)^{a}	3·07	3·25	67·8
O(*W*)	Cl	O′(*W*)^{a}	3·23	3·25	71·2
N′	Cl′	O′(*W*)	3·11	3·13	94·7
N′	Cl′	O(*W*)^{b}	3·11	3·23	70·3
O′(*W*)	Cl′	O(*W*)^{b}	3·13	3·23	73·1
Cl	O(*W*)	Cl′^{a}	3·23	3·22	106·9
Cl′	O′(*W*)	Cl^{b}	3·13	3·25	108·5

Intermolecular non-bonded distances less than 3·5 Å

i	*j*	*D*(*ij*)	*i*	*j*	*D*(*ij*)
F(1)	O(*A*)^{c}	3·45 Å	O(*A*)	C′(20)^{e}	3·23 Å
F(1)	C(*A*)1^{c}	3·28	F′(1)	O′(*A*)^{d}	3·15
F(1)	C(*A*)3^{c}	3·30	O′(*W*)	C(19)^{b}	3·50
F(3)	F′(3)^{c}	3·09	O′(*W*)	C(21)^{b}	3·39
O(*W*)	C(16)^{d}	3·50	O′(*A*)	C(20)^{f}	3·43
O(*W*)	C′(21)^{b}	3·44	O′(*A*)	C′(20)^{g}	3·45
O(*A*)	C(8)	3·46			

Symmetry code

Superscript							
None	x,	y,	z	(*d*)	x,	y,	$-1+z$
(*a*)	$1-x$,	$-\frac{1}{2}+y$,	$-z$	(*e*)	$-x$,	$\frac{1}{2}+y$,	$-z$
(*b*)	$1-x$,	$\frac{1}{2}+y$,	$-z$	(*f*)	$-x$,	$\frac{1}{2}+y$,	$1-z$
(*c*)	$1+x$,	y,	$1+z$	(*g*)	x,	y,	$1+z$

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The Crystal and Molecular Structure of Tris(biguanidato)chromium(III) Monohydrate

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Crystals of the title compound are monoclinic ($P2_1/c$) with unit-cell dimensions: $a=9{\cdot}260$, $b=10{\cdot}616$, $c=15{\cdot}928$ Å, $\beta=106{\cdot}0°$, $Z=4$. The structure has been determined from diffractometer data by direct methods and refined by block-diagonal least-squares calculations to $R=3{\cdot}6$% for 2451 independent reflexions. The main feature in this compound is the deprotonation of the organic ligand, which causes an increase of the π conjugation along the C–N–C system reducing the bond angle on nitrogen to the theoretical value of 120°. Coordination around metal is octahedral and involves six nitrogen atoms of the biguanide molecules, which act as bidentate ligands. The water molecule is involved in four hydrogen bonds with nitrogen atoms of the biguanide molecules.

Introduction

Biguanide (BG) and its substituted derivatives ethylenebisbiguanide (EBG) or 2-aminoethylbiguanide (AEBG) are interesting ligands which form highly coloured complexes with many transition metals, the following X-ray determinations of which have already been reported: $Ni(BG)_2Cl_2$ (Creitz, Gsell & Wampler, 1969), $Cr(BG)_3$-*d*-10-camphorsulphonic acid.$3H_2O$ (Brubaker & Webb, 1969), $Co(BG)_3Cl_3.H_2O$ (Snow, 1974), $Cu(EBG)Cl_2.H_2O$ (Mathew & Kunchur, 1970), $Ni(EBG)Cl_2.H_2O$ (Coghi, Mangia, Nardelli & Pelizzi,