Table 6. *Geometry* (Å) *of* 1,2-*dithia*-3,5-*diazoles*

	S–S	S–N	N–C	Reference
Ion				
$PhCN_2S_2^+$	1·990 (3)	1·590 (4)	1·340 (5)	*a*
$CCl_3CN_2S_2^+$	2·009 (6)	1·583 (7)	1·318 (10)	*b*
$ClCN_2S_2^+$	1·996 (2)	1·573 (3)	1·321 (6)	*c*
$CF_3CN_2S_2^+$	1·989 (3)	1·589 (4)	1·323 (7)	*d*
Dimer				
$[PhCN_2S_2]_2$	2·089	1·625	1·338	*e*
$[CF_3CN_2S_2]_2$	2·087 (1)	1·630 (2)	1·318 (2)	*d*
$[CF_3CN_2S_2]_2$	2·086 (1)	1·640 (2)	1·328 (4)	*d*
Monomer				
$CF_3CN_2S_2$(gas)	2·113 (6)	1·623 (3)	1·318 (6)	*d*

References: (*a*) This work; (*b*) Andreasen *et al.* (1977); (*c*) Höfs, Mews, Clegg, Noltemeyer, Schmidt & Sheldrick (1983); (*d*) Höfs *et al.* (1985); (*e*) Vegas *et al.* (1980).

is significantly shorter than that, 2·089 Å, in the corresponding phenyldithiadiazole dimer (Vegas, Pérez-Salazar, Banister & Hey, 1980), which is in agreement with the trend, Table 6, for the dimer to have an S–S bond intermediate between that of the 6π dithiadiazolium ion and the 7π dithiadiazole radical. The S–N bond of 1·590 (4) Å is also shorter than that of the dimer. There is a wide variation in the C–N distances, the longest being those of the phenyl derivatives.

References

ALANGE, G. G., BANISTER, A. J., BELL, N. & MILLEN, P. W. (1977). *Inorg. Nucl. Chem. Lett.* **13**, 143–144.

ANDREASEN, O., HAZELL, A. C. & HAZELL, R. G. (1977). *Acta Cryst.* B**33**, 1109–1112.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

GOLDSTEIN, H. (1969). *Classical Mechanics*, p. 107. Reading, MA: Addison–Wesley.

HÖFS, H.-U., BATS, J. W., GLEITER, R., HARTMANN, G., MEWS, R., ECKERT-MAKSIĆ, M., OBERHAMMER, H. & SHELDRICK, G. M. (1985). *Chem. Ber.* **118**, 3781–3804.

HÖFS, H.-U., MEWS, R., CLEGG, W., NOLTEMEYER, M., SCHMIDT, M. & SHELDRICK, G. M. (1983). *Chem. Ber.* **116**, 416–423.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed., pp. 511–518. Ithaca: Cornell Univ. Press.

PAWLEY, G. S. (1971). *Adv. Struct. Res. Diffr. Methods*, **4**, 1–64.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

VEGAS, A., PÉREZ-SALAZAR, A., BANISTER, A. J. & HEY, R. G. (1980). *J. Chem. Soc. Dalton Trans.* pp. 1812–1815.

Acta Cryst. (1988). C**44**, 1810–1813

Structural Studies on Molecular Complexes. II. Structure of Aminacrine–Sulfadimidine (1/1)

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Abstract. 1:1 molecular complex of 9-aminoacridine and N^1-(4,6-dimethyl-2-pyrimidinyl)sulfanilamide, $C_{13}H_{11}N_2^+.C_{12}H_{13}N_4O_2S^-$, M_r=472·57, monoclinic, $P2_1/c$, $a = 12{\cdot}131$ (1), $b = 12{\cdot}266$ (1), $c = 16{\cdot}287$ (2) Å, $\beta = 103{\cdot}79$ (1)°, $V = 2353{\cdot}6$ Å^3, $Z = 4$, $D_m = 1{\cdot}34$, $D_x = 1{\cdot}33$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda(\alpha_1) = 0{\cdot}7093$ Å, $\mu = 1{\cdot}64$ cm^{-1}, $F(000) = 992$, $T = 295$ K, $R = 0{\cdot}036$ for 1847 reflections. The structure contains an acridinium–sulfanilamidate ion pair with a strong N(acridinium)–H···N(pyrimidinyl) hydrogen bond. Some structural changes of the individual species in the molecular complex are discussed.

Introduction. Sulfonamides have been reported to form association complexes with a variety of small molecules (Shefter & Sackman, 1971). 9-Aminoacridine has been combined with sulfa drugs by salt linkages, and the resulting compounds were found to have synergistic antimicrobial activity (McIntosh, Robinson & Selbie, 1945; Dasgupta & Gupta, 1946), and some of them were used clinically for a long time. We have carried out X-ray crystallographic studies of a few such

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors* (Å²) *for non-H atoms with e.s.d.'s in parentheses*

	x	*y*	*z*	B_{eq}*
C(1)	0·2177 (4)	0·3532 (3)	0·3704 (3)	4·3 (2)
C(2)	0·2189 (4)	0·2926 (4)	0·3019 (3)	5·0 (3)
C(3)	0·3196 (5)	0·2404 (4)	0·2930 (3)	5·3 (3)
C(4)	0·4161 (4)	0·2491 (3)	0·3548 (3)	4·5 (3)
C(5)	0·6180 (4)	0·3676 (3)	0·6284 (3)	4·0 (2)
C(6)	0·6246 (4)	0·4226 (4)	0·7019 (3)	4·6 (3)
C(7)	0·5322 (4)	0·4823 (3)	0·7151 (3)	4·3 (3)
C(8)	0·4350 (4)	0·4867 (4)	0·6529 (3)	4·2 (3)
C(9)	0·3217 (3)	0·4298 (3)	0·5097 (2)	3·2 (2)
N(10)	0·5121 (3)	0·3146 (3)	0·4903 (2)	3·5 (1)
C(11)	0·4154 (4)	0·3100 (3)	0·4281 (2)	3·2 (2)
C(12)	0·4230 (3)	0·4290 (3)	0·5762 (2)	3·1 (2)
C(13)	0·3167 (3)	0·3643 (3)	0·4360 (2)	3·1 (2)
C(14)	0·5166 (3)	0·3703 (3)	0·5641 (2)	3·3 (2)
N(15)	0·2349 (3)	0·4896 (3)	0·5174 (2)	4·8 (2)
N(16)	0·9921 (3)	0·0665 (3)	0·0892 (2)	3·6 (2)
C(17)	0·9647 (3)	0·1225 (3)	0·1562 (2)	2·7 (2)
C(18)	0·9079 (3)	0·2219 (3)	0·1433 (2)	3·0 (2)
C(19)	0·8725 (3)	0·2726 (3)	0·2083 (2)	2·7 (2)
C(20)	0·8952 (3)	0·2259 (3)	0·2876 (2)	2·2 (2)
C(21)	0·9553 (3)	0·1287 (3)	0·3019 (2)	3·1 (2)
C(22)	0·9891 (3)	0·0778 (3)	0·2368 (2)	3·2 (2)
S(23)	0·85519 (9)	0·29395 (8)	0·37177 (6)	3·08 (5)
O(24)	0·9548 (2)	0·2878 (2)	0·4417 (1)	3·7 (1)
O(25)	0·8257 (2)	0·4040 (2)	0·3396 (1)	3·4 (1)
N(26)	0·7450 (2)	0·2502 (2)	0·3956 (2)	3·3 (2)
C(27)	0·7458 (3)	0·1544 (3)	0·4373 (2)	3·0 (2)
N(28)	0·6752 (2)	0·1516 (2)	0·4903 (2)	3·1 (2)
C(29)	0·6727 (3)	0·0595 (3)	0·5339 (2)	3·8 (2)
C(30)	0·7359 (4)	−0·0300 (3)	0·5236 (3)	4·4 (3)
C(31)	0·8020 (3)	−0·0229 (3)	0·4664 (3)	4·0 (2)
N(32)	0·8084 (2)	0·0687 (3)	0·4232 (2)	3·6 (2)
C(33)	0·5965 (7)	0·0586 (5)	0·5953 (5)	5·8 (4)
C(34)	0·8702 (7)	−0·1170 (5)	0·4466 (5)	6·6 (4)

* B_{eq} is the mean of the principal axes of the thermal ellipsoid.

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

9-Aminoacridine

C(1)–C(2)	1·344 (6)	C(8)–C(12)	1·413 (6)
C(2)–C(3)	1·417 (7)	C(12)–C(14)	1·397 (5)
C(3)–C(4)	1·354 (7)	C(5)–C(14)	1·413 (6)
C(4)–C(11)	1·411 (6)	N(10)–C(11)	1·356 (5)
C(11)–C(13)	1·403 (5)	N(10)–C(14)	1·372 (5)
C(1)–C(13)	1·411 (6)	C(9)–C(12)	1·432 (5)
C(5)–C(6)	1·359 (7)	C(9)–C(13)	1·433 (5)
C(6)–C(7)	1·398 (7)	C(9)–N(15)	1·313 (5)
C(7)–C(8)	1·359 (7)		

C(1)–C(2)–C(3)	121·0 (4)	C(1)–C(13)–C(9)	123·1 (4)
C(2)–C(3)–C(4)	120·2 (4)	C(9)–C(13)–C(11)	118·5 (4)
C(3)–C(4)–C(11)	119·5 (4)	C(13)–C(11)–N(10)	121·1 (3)
C(4)–C(11)–C(13)	120·5 (4)	C(11)–N(10)–C(14)	121·5 (3)
C(1)–C(13)–C(11)	118·4 (4)	N(10)–C(14)–C(12)	121·0 (4)
C(2)–C(1)–C(13)	120·4 (4)	C(9)–C(12)–C(14)	118·5 (3)
C(5)–C(6)–C(7)	120·9 (4)	C(12)–C(9)–C(13)	119·1 (4)
C(6)–C(7)–C(8)	119·4 (4)	C(5)–C(14)–N(10)	119·0 (4)
C(7)–C(8)–C(12)	121·8 (4)	N(10)–C(11)–C(4)	118·4 (4)
C(8)–C(12)–C(14)	117·8 (4)	C(8)–C(12)–C(9)	123·6 (4)
C(5)–C(14)–C(12)	120·0 (4)	C(12)–C(9)–N(15)	119·7 (4)
C(6)–C(5)–C(14)	120·0 (4)	C(13)–C(9)–N(15)	121·2 (4)

Sulfadimidine

N(16)–C(17)	1·395 (4)	S(23)–N(26)	1·572 (3)
C(17)–C(18)	1·391 (5)	N(26)–C(27)	1·355 (5)
C(18)–C(19)	1·381 (5)	C(27)–N(28)	1·354 (5)
C(19)–C(20)	1·379 (5)	N(28)–C(29)	1·338 (5)
C(20)–C(21)	1·388 (5)	C(29)–C(30)	1·372 (6)
C(21)–C(22)	1·375 (5)	C(30)–C(31)	1·368 (6)
C(17)–C(22)	1·388 (5)	C(31)–N(32)	1·338 (5)
C(20)–S(23)	1·769 (3)	C(27)–N(32)	1·348 (5)
S(23)–O(24)	1·451 (2)	C(29)–C(33)	1·515 (7)
S(23)–O(25)	1·462 (2)	C(31)–C(34)	1·499 (7)

C(20)–S(23)–O(24)	104·6 (2)	N(16)–C(17)–C(18)	121·1 (3)
C(20)–S(23)–O(25)	104·3 (1)	N(16)–C(17)–C(22)	120·5 (3)
C(20)–S(23)–N(26)	115·9 (2)	S(23)–N(26)–C(27)	121·0 (2)
O(24)–S(23)–O(25)	114·6 (1)	N(26)–C(27)–N(28)	114·3 (3)
O(24)–S(23)–N(26)	112·7 (1)	C(27)–N(28)–C(29)	117·3 (3)
O(25)–S(23)–N(26)	104·7 (2)	N(28)–C(29)–C(30)	121·7 (4)
C(17)–C(18)–C(19)	120·8 (3)	C(29)–C(20)–C(31)	117·9 (4)
C(18)–C(19)–C(20)	120·1 (3)	C(30)–C(31)–N(32)	121·9 (4)
C(19)–C(20)–C(21)	119·6 (3)	C(27)–N(32)–C(31)	117·4 (3)
C(20)–C(21)–C(22)	120·1 (3)	N(28)–C(27)–N(32)	123·8 (3)
C(17)–C(22)–C(21)	121·0 (3)	N(26)–C(27)–N(32)	121·9 (3)
C(18)–C(17)–C(22)	118·3 (3)	N(28)–C(29)–C(33)	116·8 (4)
C(19)–C(20)–S(23)	120·0 (3)	C(30)–C(29)–C(33)	121·5 (4)
C(21)–C(20)–S(23)	120·3 (3)	C(30)–C(31)–C(34)	122·7 (5)
		N(32)–C(31)–C(34)	115·4 (4)

molecular complexes to obtain some data on their molecular nature, on the interaction displayed by the molecular species of the complex, and on the role of hydrogen bonds in stabilizing such complexes. The structure of the 1:1 molecular complex of 9-aminoacridine with sulfamethoxypyridazine was reported earlier (Ghose, Chakrabarti & Dattagupta, 1987) and here we report the crystal and molecular structure of a 1:1 complex of 9-aminoacridine with sulfadimidine.

Experimental. Irregular yellow crystal, dimensions *ca* 0·25 mm; D_m by flotation; Picker diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; cell parameters from $\sin\theta$ values of 12 reflections and symmetry equivalents ($2\theta = 35$–$41°$); intensities for $2\theta \leq 45°$, $h = -13$ to 12, $k = 0$ to 13, $l = 0$ to 17; ω–2θ scan, ω-scan width $(0{\cdot}9 + 0{\cdot}35\tan\theta)°$, ω-scan speed 2° min^{-1}; three standard reflections, ±1% variation; Lp but no absorption corrections. Of 3103 reflections, 1847 (60%) had $I \geq 2{\cdot}5\sigma(I)$, most of the weaker reflections being at higher angles. Structure solved by *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); H atoms from a difference map; refinement by full-matrix least-squares method (Larson, Lee, Le Page & Gabe, 1984) with isotropic thermal parameters for H, anisotropic parameters for other atoms, one scale factor, and an extinction parameter; $w = 1/\sigma^2(F)$ from counting statistics gave reasonably uniform average values of $\sum w\Delta F^2$; max. Δ/σ in final cycle 0·11 for H, 0·04 for other atoms; $\Delta\rho$ within ±0·2 e Å^{-3} in final difference map; final $R = 0{\cdot}036$, $wR = 0{\cdot}019$, $S = 1{\cdot}9$ for 1847 reflections (with 308 scale, extinction and heavy-atom parameters, 96 H parameters), $R = 0{\cdot}092$ for all 3103 reflections; scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. The final atomic parameters are given in Table 1* and the bond lengths and angles in Table 2. The most significant feature of the structure (Fig. 1) is the transfer of an H ion from the sulfonamide N(26) atom to the N(10) atom of the acridine ring, so that the

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, and mean-plane calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51068 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

complex contains acridinium cations and sulfanilamidate anions. A similar situation has been found in the crystal structure of the 1:1 molecular complex of 9-aminoacridine with sulfamethoxypyridazine (Ghose *et al.*, 1987). Sulfanilamidate ions (Brennan, Shefter & Sackman, 1971) and acridinium ions (Kobayashi, 1974; Sakore, Reddy & Sobell, 1979) have also been observed in other molecular complexes. Sulfanilamide-type compounds are weak acids because the sulfonamide N atom of these molecules is sufficiently negative. Therefore, the H atom is bound less firmly to this N atom and it readily ionizes to liberate a proton in solution (Foernzler & Martin, 1967). Positively charged cationic acridines have been reported to have antibacterial activity (Wolfe, 1975). The essential structural differences found in the complex when compared with the values observed in the free sulfadimidine molecule (Tiwari, Haridas & Singh, 1984) and in the 9-aminoacridinium ion in the hydrochloride (Talacki, Carrell & Glusker, 1974) are listed in Table 3.

Sulfadimidine. The N(16)–C(17) bond in the sulfa drug is lengthened significantly from usual values, presumably as a result of the sp^3 character of N(16) caused by the acceptance of a hydrogen bond, and the consequent decrease in conjugation with the ring. A similar lengthening also occurs in the molecular complex of 9-aminoacridine with sulfamethoxypyridazine. For most sulfa drugs this bond has been found to be in the range 1·360–1·379 Å. A corresponding lengthening of the C–N bond has also been found in other molecular complexes of sulfa drugs (Shefter & Sackman, 1971).

The deprotonation of the sulfonamide N atom produces some minor changes in the dimensions of the sulfonamide grouping. The S–N and N–C bonds are amongst the shortest when compared with the values in related compounds (Shefter, Chmielewicz, Blount, Brennan, Sackman & Sackman, 1972; Chatterjee, Dattagupta & Saha, 1981, 1982) while the S–C and S–O bonds are amongst the longest of those observed in related compounds. The endocyclic angle at C(27) is 121·9 (3)°, and is within the normal range observed in sulfonamides with a pyrimidine ring.

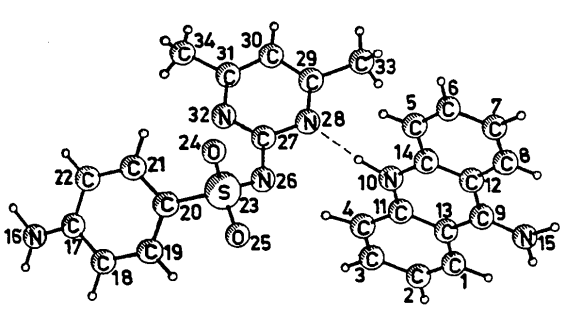

Fig. 1. View of the 9-aminoacridine–sulfadimidine complex (the broken line indicates a hydrogen bond).

Table 3. *Essential structural differences between the complex and the individual molecules*

Features	Present structure	Free molecule or ion
S–N bond	1·572 (3) Å	1·632 (7) Å[a]
N–C bond	1·355 (5) Å	1·41 (1) Å[a]
S–O bond	1·462 (2)–1·451 (2) Å	1·426 (8)–1·430 (6) Å[a]
C(17)–N(16) bond	1·395 (4) Å	1·36 (1) Å[a]
Pyrimidine ring N–C–N angle	121·9 (3)°	129·5 (9)°[a]
Dihedral angle between the two rings of the sulfa drug	33·3°	78·1°[a]
N(26)–S(23)–C(20)–C(19) torsion angle	102·6°	130·2°[a]
Maximum displacement of acridine ring	0·086 (5) Å	0·017 (4) Å[b]
Angles between outer rings of acridine molecules	4·9°	1°[b]

References: (*a*) Sulfadimidine (Tiwari, Haridas & Singh, 1984); (*b*) aminoacridine. HCl (Talacki, Carrell & Glusker, 1974).

The aromatic and the pyrimidine rings are planar within the limits of experimental error. The torsion angles N(26)–S(23)–C(20)–C(21) = −81·7 (3) and C(20)–S(23)–N(26)–C(27) = 73·7 (3)° which define the conformation of the sulfonamide group are within the ranges of 70–120 and 60–90°, respectively, observed in related molecules (Kálmán, Czugler & Argay, 1981). But the dihedral angle between the two rings, which has been found to be in the range 60–90° in sulfonamides, is 33·3° in the present complex, compared with 83·9° in the 9-aminoacridine–sulfamethoxypyridazine complex.

9-Aminoacridine. The dimensions of the 9-aminoacridinium ion are normal and compare well with those found in the hydrochloride (Talacki, Carrell & Glusker, 1974) and the 9-aminoacridine–sulfamethoxypyridazine complex (Ghose *et al.*, 1987). In both the complexes, the outer rings are found to be planar, while the central ring is buckled. The angle between the two outer rings of the acridine moiety in the present structure is 4·9 (2)° and compares well with the value of 5·1° found in the 9-aminoacridine–sulfamethoxypyridazine complex.

Intermolecular bonding. The strongest intermolecular link between the acridinium cation and sulfanilamide anion is through N(10) and the pyrimidine N(28) atom (Fig. 2 and Table 4). The ion pairs are linked to neighbouring pairs by hydrogen bonds involving all four amino H atoms. N(16) also acts as a hydrogen-bond acceptor and hence that amino group is pyramidal, in contrast to $N(15)H_2$ which is planar. N(26) and N(32) are not involved in hydrogen bonding. The crystal packing (Fig. 2) is dominated by the hydrogen-bond system, which results in pairs of acridinium ions sandwiched around centres of symmetry with an inter-ion spacing of about 3·5 Å.

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Table 4. *Hydrogen-bonding parameters*

A—H···*B*	*A*···*B* (Å)	*A*—H (Å)	H···*B* (Å)	∠*A*—H···*B* (°)
N(16)—H(16)···O(24) $x, \frac{1}{2}-y, -\frac{1}{2}+z$	2·942 (4)	0·96 (3)	2·01 (3)	163 (3)
N(16)—H(16')···O(25) $2-x, -\frac{1}{2}+y, \frac{1}{2}-z$	2·997 (5)	1·02 (4)	1·98 (4)	174 (3)
N(10)—H(10)···N(28) x, y, z	2·814 (4)	1·04 (4)	1·79 (4)	167 (3)
N(15)—H(15)···O(25) $1-x, 1-y, 1-z$	2·914 (4)	1·14 (4)	1·85 (4)	153 (3)
N(15)—H(15)···N(26) $1-x, 1-y, 1-z$	3·478 (4)	1·14 (4)	2·58 (4)	135 (3)
N(15)—H(15')···N(16) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$	3·034 (5)	0·88 (3)	2·20 (3)	159 (3)

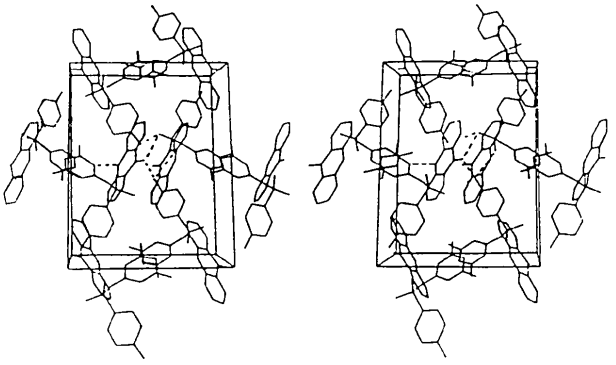

Fig. 2. Packing diagram; view along **a**, **b** horizontal, **c** vertical. One example of each type of hydrogen bond is shown.

References

BRENNAN, T. F., SHEFTER, E. & SACKMAN, P. (1971). *Chem. Pharm. Bull.* **19**, 1919–1924.

CHATTERJEE, C., DATTAGUPTA, J. K. & SAHA, N. N. (1981). *Acta Cryst.* B37, 1835–1838.

CHATTERJEE, C., DATTAGUPTA, J. K. & SAHA, N. N. (1982). *Acta Cryst.* B38, 1845–1847.

DASGUPTA, P. & GUPTA, P. (1946). *J. Indian Chem. Soc.* **23**, 241–243.

FOERNZLER, E. C. & MARTIN, A. N. (1967). *J. Pharm. Sci.* **56**, 608–615.

GHOSE, S., CHAKRABARTI, C. & DATTAGUPTA, J. K. (1987). *J. Crystallogr. Spectrosc. Res.* **17**, 197–206.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

KÁLMÁN, A., CZUGLER, M. & ARGAY, GY. (1981). *Acta Cryst.* B37, 868–877.

KOBAYASHI, H. (1974). *Bull. Chem. Soc. Jpn*, **47**, 1346–1352.

LARSON, A. C., LEE, F. L., LE PAGE, Y. & GABE, E. J. (1984). *The NRC VAX Crystal Structure System.* Chemistry Division, NRC, Ottawa, Canada.

MCINTOSH, J., ROBINSON, R. H. M. & SELBIE, F. R. (1945). *Lancet*, **249**, 97–99.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

SAKORE, T. D., REDDY, B. S. & SOBELL, H. M. (1979). *J. Mol. Biol.* **135**, 763–785.

SHEFTER, E., CHMIELEWICZ, Z. F., BLOUNT, J. F., BRENNAN, T. F., SACKMAN, B. F. & SACKMAN, P. (1972). *J. Pharm. Sci.* **61**, 872–877.

SHEFTER, E. & SACKMAN, P. (1971). *J. Pharm. Sci.* **60**, 282–286.

TALACKI, R., CARRELL, H. L. & GLUSKER, J. P. (1974). *Acta Cryst.* B30, 1044–1047.

TIWARI, R. K., HARIDAS, M. & SINGH, T. P. (1984). *Acta Cryst.* C40, 655–657.

WOLFE, A. D. (1975). *Antibiotics*, Vol. III, edited by J. W. CORCORAN & F. E. HAHN, p. 216. New York: Springer-Verlag.

Acta Cryst. (1988). C**44**, 1813–1815

Structure of the First Genuine Ansa Derivative, $N_3P_3Cl_4[HN(CH_2)_2O(CH_2)_2NH]$

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Abstract. 2,4,6,6-Tetrachloro-2,4-oxybis(ethyleneimino)cyclotriphosphazatriene, $C_4H_{10}Cl_4N_5OP_3$, $M_r = 378{\cdot}9$, orthorhombic, *Pnma*, $a = 14{\cdot}429\ (3)$, $b = 12{\cdot}258\ (5)$, $c = 8{\cdot}054\ (2)$ Å, $V = 1425\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}767\ (1)$, $D_m = 1{\cdot}75\ (3)$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}2$ mm^{-1}, $F(000) = 760$, $T = 292$ K, $R = 0{\cdot}039$ for 1265 unique observed reflections and 85 variable parameters. The structure exhibits a spatial

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