

radii. The shortest contact that the Zn makes with a methyl C atom (2.90 Å) involves C(4) and this situation would be made worse by a shortening of the Zn(1)–N(2) bond.

The short N...N contacts (2.85 and 2.96 Å) are those across the four- and five-membered rings respectively. Both are much greater than the sum (1.40 Å) of the tetrahedral covalent radii of two N atoms.

The intermolecular packing is shown in Fig. 2 which is a projection along **b**. Only two of the approach distances are <4.0 Å and both of these are >3.6 Å so that there does not appear to be any significant interaction between separate dimer molecules. The C–H bond lengths all appear to be normal compared with previously published lengths (Sequeira, Rajagopal & Chidambaram, 1972) and with the sum of the covalent radii for C and H (1.09 Å).

#### *The zinc–hydrogen bond*

The neutron diffraction study has provided a reliable position for the H atom attached to Zn with a bond length of 1.618 Å, not significantly different from the value (1.59 Å) obtained for a short-lived species by spectroscopic methods (Herzberg, 1950). The length of the Zn–H bond is close to the sum (1.62 Å) of the covalent radii (Sanderson, 1976) and much less than the sum of the ionic radii, 2.82 Å (Pauling, 1960), suggesting that the bond is predominantly covalent in character.

In contrast to the beryllium hydrides where Be...H...Be bridging predominates, there can be no question of H bridging between Zn atoms in the present compound since the shortest contact between Zn atoms not of the same dimer is >5 Å.

NAB, PTM, and CBS acknowledge the inspiration of their co-author and the interest shown in this work

by Professor G. E. Coates. PTM and CBS are grateful for SRC studentships.

#### References

- ALLEN, P. W. & SUTTON, L. E. (1950). *Acta Cryst.* **3**, 46–72.  
 BELL, N. A. & COATES, G. E. (1968). *J. Chem. Soc. A*, pp. 823–826.  
 BELL, N. A., MOSELEY, P. T., SHEARER, H. M. M. & SPENCER, C. B. (1980). *Chem. Commun.* pp. 359–360.  
 COATES, G. E. & HESLOP, J. A. (1968). *J. Chem. Soc. A*, pp. 514–518.  
 COATES, G. E. & MORGAN, G. L. (1970). *Adv. Organomet. Chem.* **9**, 195–257.  
 COATES, G. E. & RIDLEY, D. (1965). *J. Chem. Soc.* pp. 1870–1877.  
 COATES, G. E. & ROBERTS, P. D. (1968). *J. Chem. Soc. A*, pp. 2651–2655.  
 DE KONING, A. J., BOERSMA, J. & VAN DER KERK, G. J. M. (1977). *Tetrahedron Lett.* pp. 2547–2548.  
 DE KONING, A. J., BOERSMA, J. & VAN DER KERK, G. J. M. (1978). *J. Organomet. Chem.* **155**, C5–C7.  
 HERZBERG, G. (1950). *Molecular Spectra and Molecular Structure. Infrared Spectra of Diatomic Molecules*. New York: Van Nostrand.  
 PALENIK, G. J. (1964). *Acta Cryst.* **17**, 1573–1580.  
 PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed. Ithaca: Cornell Univ. Press.  
 SANDERSON, R. T. (1976). *Chemical Bonds and Bond Energy*, 2nd ed. London: Academic Press.  
 SEQUEIRA, A., RAJAGOPAL, H. & CHIDAMBARAM, R. (1972). *Acta Cryst.* **B28**, 2514–2519.  
 SPENCER, C. B. (1967). *The Crystal Structures of Three Associated Zinc Complexes*. Ph.D. Thesis, Univ. of Durham.  
 WILLIS, B. T. M. & PRYOR, A. W. (1975). *Thermal Vibrations in Crystallography*. Cambridge Univ. Press.

*Acta Cryst.* (1980). **B36**, 2954–2958

## The Structure of Tris(1-pyrrolidinecarbodithioato)antimony(III)

BY C. A. KAVOUNIS, S. C. KOKKOU AND P. J. RENTZEPERIS

*Applied Physics Laboratory, Aristotle University of Thessaloniki, Thessaloniki, Greece*

AND P. KARAGIANNIDIS

*Laboratory of Inorganic Chemistry, Aristotle University of Thessaloniki, Thessaloniki, Greece*

(Received 12 February 1980; accepted 27 June 1980)

#### Abstract

The crystal structure of [Sb(C<sub>3</sub>H<sub>8</sub>NS<sub>2</sub>)<sub>3</sub>] has been determined from three-dimensional intensities, measured on a computer-controlled Philips PW 1100

single-crystal diffractometer (1904 observed reflexions). The cell constants, obtained from direct  $\theta$ -value measurements on the diffractometer, are:  $a = 16.442$  (1),  $b = 11.883$  (1),  $c = 23.677$  (2) Å,  $\beta = 102.95$  (1)°,  $Z = 8$ ; the space group is  $C2/c$ . The

0567-7408/80/122954-05\$01.00

© 1980 International Union of Crystallography

structure was solved by direct phase determination with *MULTAN*. The positional and thermal atomic parameters were refined by full-matrix least-squares calculations to a final  $R = 6.9\%$ . The molecule consists of three substantially planar, asymmetrically bidentate, ligands surrounding the central Sb atom in approximately  $C_s$  symmetry. There is a considerable gap in the metal coordination due to a stereochemically active lone pair and the proximity of the rotation image of the molecule. However, two weak intermolecular interactions [Sb—Sb' and Sb—S'(1)] are present, resulting in the formation of a distorted icosahedron by the coordination polyhedra of the two rotation-equivalent  $SbS_6$  cores.

### Introduction

A large number of dithiocarbamate structures have been investigated during the past two decades, because of the special theoretical and practical interest presented by these compounds, from either a chemical or a biological point of view. Notwithstanding the variety of the known structure types, many new compounds show special and sometimes unexpected features. The present structure of tris(1-pyrrolidinecarbodithioato)antimony(III) (TPCA hereafter) shows such features and is the first of a series of compounds, prepared at the Laboratory of Inorganic Chemistry of the University of Thessaloniki (Manoussakis & Karagiannidis, 1970).

### Experimental

Good, yellow needles of TPCA were obtained from a chloroform and petroleum ether solution of the compound. A very small needle was selected and mounted on our computer-controlled Philips PW 1100 four-circle single-crystal diffractometer. To obtain accurate cell parameters, the values of the  $\theta$  angles of 102 strong reflexions were measured very carefully on the diffractometer and then processed with the least-squares program *PARAM* [part of the XRAY system of crystallographic programs (Stewart, Kruger, Ammon, Dickinson & Hall, 1972)]. The final values are given in the *Abstract*. The density of the crystal, measured by flotation in  $CCl_4$ , is  $1.62 \text{ Mg m}^{-3}$ ; the calculated value is  $1.65 \text{ Mg m}^{-3}$ .

Systematic absences were consistent with the two monoclinic space groups  $C2/c$  and  $Cc$ . Since, however, intensity statistics indicated the presence of a center of symmetry, the first was assumed and confirmed later by the successful refinement.

Three-dimensional data were collected with a scintillation counter on the PW 1100 diffractometer operating in the  $\theta/2\theta$  scan mode. Mo  $K\alpha$  radiation, monochromatized with a graphite monochromator ( $\lambda$

$= 0.71069 \text{ \AA}$ ), was used. Intensities were measured for 2626 unique reflexions in the range  $\theta = 2\text{--}22^\circ$  (max.  $h, k, l$ : 16, 12,  $\pm 24$ ). Of these, 1904 with  $I > 2\sigma(I)$  were considered as observed and included in the further steps of the structure analysis. Data reduction was carried out using the program *DATRED* (Main, 1970). Since  $\mu = 1.71 \text{ mm}^{-1}$ , no absorption correction was made. For further calculations the programs of the *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1974) and XRAY systems were used.

### Structure determination and refinement

The structure was essentially solved by direct phase determination using the *MULTAN* system. The phases of 200 reflexions with  $E \geq 1.70$  were determined and on the resulting  $E$  map it was possible to locate 15 of the 25 non-hydrogen atoms in the asymmetric unit. The remaining atoms were located by means of a Fourier synthesis.

The structure was refined by full-matrix least-squares calculations. The atomic scattering factors for all the atoms and anomalous-dispersion corrections for Sb and S were taken from *International Tables for X-ray Crystallography* (1974). A single scale factor was used for the whole set of reflexion data. The weighting scheme which proved most suitable was No. 3 in the XRAY system:  $w = xy = (\sin \theta/B)(C/F_o)$ , where  $x = 1$  if  $\sin \theta > B$ , otherwise  $x = \sin \theta/B$ ; and  $y = 1$  if  $C > F_o$ ,

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic temperature factors for the non-hydrogen atoms in TPCA

	$x$	$y$	$z$	$B (\text{\AA}^2)$
Sb	329 (1)	34 (1)	3260 (1)	5.2
S(1)	372 (3)	9565 (3)	4288 (2)	5.7
S(2)	1022 (3)	7841 (4)	3597 (2)	7.1
S(3)	654 (3)	2160 (3)	3610 (2)	5.9
S(4)	2051 (3)	548 (4)	3651 (2)	6.4
S(5)	8848 (3)	1017 (3)	3161 (2)	5.8
S(6)	8930 (3)	8583 (3)	2898 (2)	5.6
N(1)	954 (9)	7598 (11)	4704 (6)	6.7
N(2)	2228 (8)	2646 (11)	4010 (5)	6.9
N(3)	7518 (8)	9789 (9)	2700 (5)	5.5
C(1)	789 (10)	8253 (12)	4233 (7)	5.6
C(2)	822 (11)	7951 (15)	5275 (6)	6.8
C(3)	1184 (19)	6925 (17)	5661 (10)	10.9
C(4)	1222 (23)	5991 (20)	5295 (10)	12.9
C(5)	1281 (16)	6431 (16)	4695 (9)	9.3
C(6)	1712 (8)	1836 (11)	3773 (6)	4.7
C(7)	1987 (11)	3807 (13)	4158 (8)	6.5
C(8)	2803 (13)	4383 (18)	4402 (9)	8.4
C(9)	3415 (13)	3436 (17)	4620 (8)	7.8
C(10)	3156 (10)	2481 (15)	4212 (7)	6.6
C(11)	8343 (10)	9769 (11)	2900 (6)	5.1
C(12)	7037 (10)	8782 (13)	2448 (7)	6.1
C(13)	6184 (13)	9286 (19)	2213 (11)	9.1
C(14)	6137 (13)	361 (17)	2452 (14)	10.6
C(15)	7003 (10)	805 (13)	2682 (8)	6.6

Table 2. Atomic coordinates ( $\times 10^3$ ) and isotropic temperature factors for the hydrogen atoms in TPCA

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (Å <sup>2</sup> )
H1(C2)	128	755	562	6.8
H2(C2)	20	765	532	6.8
H1(C3)	173	741	589	10.9
H2(C3)	64	750	562	10.9
H1(C4)	64	549	526	12.9
H2(C4)	174	545	549	12.9
H1(C5)	187	683	465	9.3
H2(C5)	77	690	443	9.3
H1(C7)	161	378	449	6.5
H2(C7)	162	425	379	6.5
H1(C8)	276	496	475	8.4
H2(C8)	300	485	406	8.4
H1(C9)	357	323	508	7.8
H2(C9)	389	400	454	7.8
H1(C10)	330	167	442	6.6
H2(C10)	347	252	385	6.6
H1(C12)	729	807	226	6.1
H2(C12)	753	924	273	6.1
H1(C13)	571	872	232	9.1
H2(C13)	607	932	174	9.1
H1(C14)	580	33	281	10.6
H2(C14)	579	94	213	10.6
H1(C15)	727	151	250	6.6
H2(C15)	749	34	297	6.6

Table 3. Interatomic distances (Å) in TPCA

Sb—S(1)	2.484 (4)	S(3)—C(6)	1.74 (1)
Sb—S(2)	2.884 (5)	S(4)—C(6)	1.68 (1)
Sb—S(3)	2.675 (4)	C(6)—N(2)	1.32 (2)
Sb—S(4)	2.842 (9)	N(2)—C(7)	1.50 (2)
Sb—S(5)	2.663 (5)	C(7)—C(8)	1.50 (3)
Sb—S(6)	2.848 (7)	C(8)—C(9)	1.52 (3)
		C(9)—C(10)	1.49 (3)
		C(10)—N(2)	1.51 (2)
S(1)—C(1)	1.72 (1)		
S(2)—C(1)	1.71 (2)		
C(1)—N(1)	1.34 (2)	S(5)—C(1)	1.74 (1)
N(1)—C(2)	1.48 (2)	S(6)—C(11)	1.71 (1)
C(2)—C(3)	1.56 (3)	C(11)—N(3)	1.33 (2)
C(3)—C(4)	1.42 (3)	N(3)—C(12)	1.48 (2)
C(4)—C(5)	1.43 (3)	C(12)—C(13)	1.51 (3)
C(5)—N(1)	1.49 (2)	C(13)—C(14)	1.41 (3)
		C(14)—C(15)	1.50 (3)
		C(15)—N(3)	1.47 (2)

otherwise  $y = C/F_o$ . In this case  $B = 0.25$  and  $C = 150$ , the latter corresponding to  $F_o$  on the absolute scale. After several cycles of isotropic and anisotropic refinement, the  $R$  factor reduced to 0.075.

A difference Fourier synthesis revealed most of the H atoms of the pyrrolidine rings at positions very close to the calculated ones. They were then included in the subsequent refinement cycles with individual isotropic temperature coefficients equal to those of the C atoms to which they are bonded. Attempts to refine their parameters proved unsuccessful. The  $R$  factor reduced very little, to 0.074. At this stage it was observed that 24 strong, low-angle reflexions ( $F_o \geq \frac{1}{4}F_o \text{ max.}, \sin \theta/\lambda$

Table 4. Bond angles (°) in TPCA

S(1)—Sb—S(2)	66.7 (0.1)		
S(1)—Sb—S(3)	87.0 (0.1)		
S(1)—Sb—S(4)	85.5 (0.1)	Sb—S(3)—C(6)	88.9 (0.5)
S(1)—Sb—S(5)	90.5 (0.1)	Sb—S(4)—C(6)	84.7 (0.5)
S(1)—Sb—S(6)	90.2 (0.1)	S(3)—C(6)—S(4)	121.2 (0.8)
S(2)—Sb—S(3)	136.8 (0.1)	S(3)—C(6)—N(2)	116.6 (1.1)
S(2)—Sb—S(4)	78.5 (0.1)	S(4)—C(6)—N(2)	122.2 (1.1)
S(2)—Sb—S(5)	135.9 (0.1)	C(6)—N(2)—C(7)	126.3 (1.3)
S(2)—Sb—S(6)	77.2 (0.1)	C(6)—N(2)—C(10)	123.2 (1.3)
S(3)—Sb—S(4)	65.2 (0.1)	C(7)—N(2)—C(10)	110.4 (1.2)
S(3)—Sb—S(5)	74.2 (0.1)	N(2)—C(7)—C(8)	104.4 (1.4)
S(3)—Sb—S(6)	139.2 (0.1)	C(7)—C(8)—C(9)	105.1 (1.6)
S(4)—Sb—S(5)	139.4 (0.1)	C(8)—C(9)—C(10)	105.8 (1.5)
S(4)—Sb—S(6)	155.1 (0.1)	C(9)—C(10)—N(2)	103.2 (1.4)
S(5)—Sb—S(6)	65.1 (0.1)		
Sb—S(1)—C(1)	93.0 (0.6)	Sb—S(5)—C(11)	90.6 (0.5)
Sb—S(2)—C(1)	80.3 (0.5)	Sb—S(6)—C(11)	85.3 (0.5)
S(1)—C(1)—S(2)	120.0 (0.9)	S(5)—C(11)—S(6)	118.6 (0.9)
S(1)—C(1)—N(1)	118.3 (1.3)	S(5)—C(11)—N(3)	118.4 (1.0)
S(2)—C(1)—N(1)	121.6 (1.2)	S(6)—C(11)—N(3)	123.0 (1.0)
C(1)—N(1)—C(2)	123.6 (1.3)	C(11)—N(3)—C(12)	122.4 (1.1)
C(1)—N(1)—C(5)	122.4 (1.5)	C(11)—N(3)—C(15)	124.0 (1.1)
C(2)—N(1)—C(5)	113.9 (1.4)	C(12)—N(3)—C(15)	113.5 (1.2)
N(1)—C(2)—C(3)	101.5 (1.5)	N(3)—C(12)—C(13)	101.3 (1.3)
C(2)—C(3)—C(4)	108.5 (1.8)	C(12)—C(13)—C(14)	110.0 (1.7)
C(3)—C(4)—C(5)	108.6 (2.0)	C(13)—C(14)—C(15)	109.4 (1.8)
C(4)—C(5)—N(1)	101.8 (1.8)	C(14)—C(15)—N(3)	102.4 (1.3)

$< 0.2 \text{ \AA}^{-1}$ ) appeared affected by secondary extinction. A plot of  $I_c/I_o$  versus  $I_c$  for these reflexions yielded a straight line with a slope  $2g = 0.56 \times 10^{-6}$ . The intensities of these reflexions were then corrected by means of the equation  $I_o(\text{corrected}) \approx I_o(1 + 2gI_c)$  (Stout & Jensen, 1968). The  $R$  factor converged to 0.069. The final positional and isotropic thermal parameters for the non-hydrogen atoms are given in Table 1 and those for the H atoms in Table 2. Interatomic distances and bond angles are given in Tables 3 and 4.\*

### Description of the structure and discussion

Fig. 1 shows a clinographic projection of the TPCA molecule. As in the case of tris(diethyldithiocarbamato)antimony(III) and -bismuth(III) (Raston & White, 1976) and tris(diethyldithiocarbamato)-arsenic(III) (Colapietro, Domenicano, Scaramuzza & Vaciago, 1968; Raston & White, 1975) the metal atom is coordinated by three asymmetrically bidentate ligands and, due to the existence of a stereochemically active lone pair, a large gap in the coordination sphere of the Sb atom is observed. In the present case, however, the angular distortion of the  $\text{SbS}_6$  core is more

\* Lists of structure factors, anisotropic thermal parameters of the non-hydrogen atoms and various least-squares planes of the TPCA molecule have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35520 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

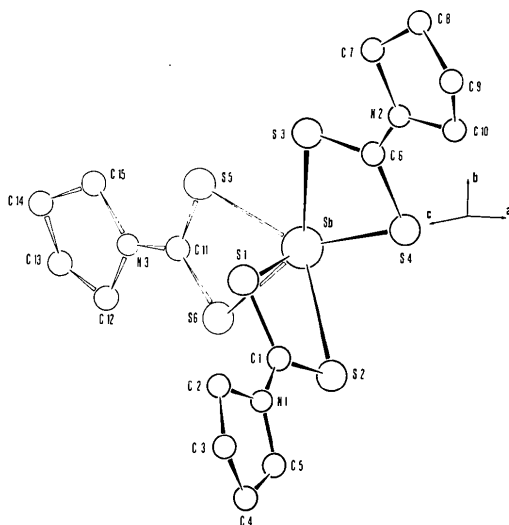
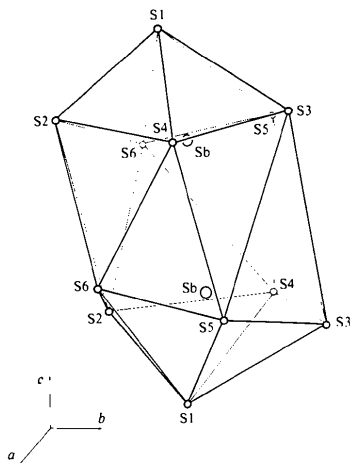


Fig. 1. Clinographic projection of the TPCA molecule.

Fig. 2. Clinographic projection of the two rotation-equivalent  $SbS_6$  cores forming a distorted icosahedron.

pronounced, because of the close proximity of a second rotation equivalent molecule (*cf.* Figs. 2 and 3). The two molecules have their lone pairs facing each other and form, as it were, a sort of a double molecule, which may be considered as the true repeating unit in the crystal.

In this way, however, the usual  $D_3$  symmetry of the  $MS_6$  core in the metal dithiocarbamates is degraded not to an approximate  $C_3$  symmetry, as in the case of the thiocarbamates mentioned above, but very closely to  $C_s$ . The two groups  $Sb-S(3)-S(4)$  and  $Sb-S(5)-S(6)$  are almost coplanar and perpendicular to the third group  $Sb-S(1)-S(2)$ , which constitutes the mirror plane of the core. The rotation-equivalent atoms  $Sb'$  and  $S'(6)$  of the accompanying molecule approach the Sb atom quite closely. In fact, the distances  $Sb-Sb' = 3.517(9)$  and  $Sb-S'(6) = 3.671(9)$  Å are less than

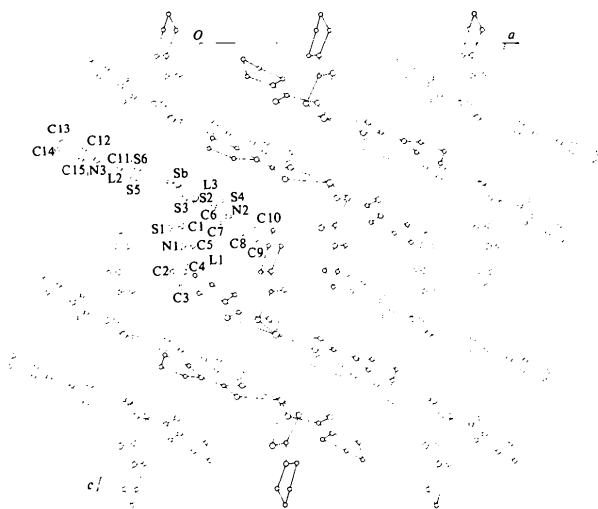


Fig. 3. Packing of the molecules in the unit cell.

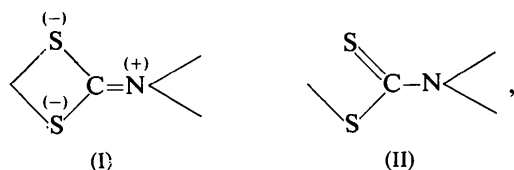
the sums, 4.00 and 3.80 Å, of the corresponding van der Waals radii (Bondi, 1964), so that a weak intermolecular interaction takes place. An interesting consequence is that the coordination polyhedra of the two rotation-equivalent  $SbS_6$  cores form a distorted icosahedron (Fig. 2).

As expected from other dithiocarbamate structures with the same metal coordination (Colapietro *et al.*, 1968; Raston & White, 1975, 1976; Raston, White & Willis, 1975) three of the  $Sb-S$  bonds, nearly at right angles to one another, are practically covalent and the corresponding distances are shorter than the rest (*cf.* Table 3). In particular the distance  $Sb-S(1)$  is much shorter than all the others, a fact which may be attributed to the lone pair, which is expected to be in the plane of the group  $Sb-S(1)-S(2)-C(1)$  and opposite  $S(1)$ .

All three  $S_2CN$  ligand fragments are planar to a very good approximation and the Sb atom does not deviate from these planes by more than 0.324 Å.\* The angles between the three planes are 84.0, 83.2 and 4.9°; that is, the one plane is almost perpendicular to the other two, which are almost coplanar. The general features of these fragments are similar to those found in other dithiocarbamates: The three  $S-C$  bonds not adjacent to the predominantly covalent  $Sb-S$  bonds have some double-bond character and are shorter than the other three (*cf.* Table 3). The mean values 1.70 and 1.73 Å for the  $S-C$  bonds and 1.33 Å for the  $C-N$  bonds compare well with values found in the other dithiocarbamates mentioned above. The  $C-N$  distances imply that the  $C-N$  bonds have a strong double-bond character.

\* See previous footnote.

The contributions of the resonance structures usually assumed for the dithiocarbamate ligands,



calculated according to the valence-bond theory (Pauling, 1960), are 65% for structure (I) and 35% for structure (II). Once more it is confirmed that the form  $\cdots\overset{(-)}{S}_2\overset{(+)}{C}=\overset{(+)}{N}R_2$ , makes a major contribution to the structure of dithiocarbamates (cf. Chatt, Duncanson & Venanzi, 1956; Bonamico, Dessy, Mariani, Vaciago & Zambonelli, 1965; Bonamico, Dessy, Mugnoli, Vaciago & Zambonelli, 1965; Peyronel & Pignedoli, 1967).

The three pyrrolidine rings of the molecule are puckered. As in the case of other compounds (cf. Pitzer & Donath, 1959; Eliel, Allinger, Angyal & Morrison, 1965) the maximum puckering occurs at the carbon atoms C(3), C(4); C(8), C(9); C(13), C(14) respectively, away from the N heteroatom.\* Thus, the conformation of the rings is closer to the half-chair than to the envelope form. The temperature factors of the above C atoms are, naturally enough, relatively large (Table 1).

In Fig. 3 is given a projection of the structure showing the molecular packing of TPCA in the unit cell. The roughly coplanar systems formed by ligands L2 and L3 are approximately parallel to (104).

Thanks are due to the University of Thessaloniki for the use of its computing facilities.

\* See first footnote.

*Acta Cryst.* (1980). B36, 2958–2963

### *fac*-Trichloro(diethylenetriamine- $N^1, N^2, N^3$ )platinum(IV) Chloride Monohydrate

BY J. F. BRITTEN AND C. J. L. LOCK

*Institute for Materials Research, McMaster University, Hamilton, Ontario, Canada L8S 4M1*

(Received 2 May 1980; accepted 1 July 1980)

#### Abstract

$[Pt(C_4H_{13}N_3)Cl_3]Cl \cdot H_2O$ ,  $C_4H_{13}Cl_3N_3Pt^+ \cdot Cl^- \cdot H_2O$ ,  $M_r = 458.08$ , orthorhombic, *Pbca*,  $a = 16.606$  (8),  $b = 12.490$  (4),  $c = 11.293$  (2) Å,  $V = 2342$  (1) Å<sup>3</sup>,  $Z = 8$ ,

0567-7408/80/122958-06\$01.00

#### References

- BONAMICO, M., DESSY, G., MARIANI, C., VACIAGO, A. & ZAMBONELLI, L. (1965). *Acta Cryst.* **19**, 619–626.  
 BONAMICO, M., DESSY, G., MUGNOLI, A., VACIAGO, A. & ZAMBONELLI, L. (1965). *Acta Cryst.* **19**, 886–895.  
 BONDI, A. (1964). *J. Phys. Chem.* **68**, 441–451.  
 CHATT, J., DUNCANSON, L. A. & VENANZI, L. M. (1956). *Nature (London)*, **177**, 1042–1043.  
 COLAPIETRO, M., DOMENICANO, A., SCARAMUZZA, L. & VACIAGO, A. (1968). *Chem. Commun.* pp. 302–303.  
 ELIEL, E. L., ALLINGER, N. L., ANGYAL, S. J. & MORRISON, G. A. (1965). *Conformational Analysis*, pp. 200–206. New York: John Wiley.  
*International Tables for X-ray Crystallography* (1974). Vol. IV, pp. 71–151. Birmingham: Kynoch Press.  
 MAIN, P. (1970). Private communication. Univ. of York, England.  
 MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J. P. (1974). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain-la-Neuve, Belgium.  
 MANOUSSAKIS, G. & KARAGIANNIDIS, P. (1970). *Inorg. Nucl. Chem. Lett.* **6**, 71.  
 PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed. Ithaca: Cornell Univ. Press.  
 PEYRONEL, G. & PIGNEDOLI, A. (1967). *Acta Cryst.* **23**, 398–408.  
 PITZER, K. S. & DONATH, W. E. (1959). *J. Am. Chem. Soc.* **81**, 3213–3218.  
 RASTON, C. L. & WHITE, A. H. (1975). *J. Chem. Soc. Dalton Trans.* pp. 2425–2429.  
 RASTON, C. L. & WHITE, A. H. (1976). *J. Chem. Soc. Dalton Trans.* pp. 791–794.  
 RASTON, C. L., WHITE, A. H. & WILLIS, A. C. (1975). *J. Chem. Soc. Dalton Trans.* pp. 2429–2432.  
 STEWART, J. M., KRUGER, G. J., AMMON, H. L., DICKINSON, C. & HALL, S. R. (1972). The XRAY system – version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland.  
 STOUT, G. H. & JENSEN, L. H. (1968). *X-ray Structure Determination*, pp. 411–412. New York: Macmillan.

$D_{\text{calc}} = 2.60$ ,  $D_{\text{obs}} = 2.60$  (2) Mg m<sup>-3</sup>. The structure was determined by Patterson and electron density difference methods and refined by full-matrix least squares to  $R = 0.058$ ,  $R_w = 0.059$  based on 2207 independent reflections. The structure comprises dis-

© 1980 International Union of Crystallography