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## Structure of [2.2.2.2]Paracyclophane,\* $C_{32}H_{32}$

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**Abstract.**  $M_r = 416.6$ , monoclinic,  $P2_1/c$ ,  $a = 12.026$  (3),  $b = 9.731$  (3),  $c = 20.978$  (6) Å,  $\beta = 92.35$  (5)°,  $V = 2455$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.13$  Mg m<sup>-3</sup>, Ni-filtered Cu  $K\alpha$  radiation,  $\lambda = 1.5418$  Å,  $\mu = 0.482$  mm<sup>-1</sup>,  $F(000) = 896$ ,  $T = 293$  K,  $R = 0.061$  for 2763 observed reflexions. The observed *gauche* conformation of the 4-PCP molecule is in agreement with previous NMR studies in solution. The four benzene rings have a non-planar arrangement analogous to that observed in the homologous compound, 4-PCP-TCNE.

**Introduction.** This work is part of a series of structural studies on paracyclophanes and their complexes with metal ions or electron-acceptor molecules such as tetracyanoethylene, TCNE (Pierre, Baret, Chautemps & Armand, 1981; Cohen-Addad, Baret, Chautemps & Pierre, 1983; Cohen-Addad, Lebars, Renault & Baret, 1984). We have previously studied the complex [2.2.2.2]paracyclophane-TCNE. For comparison, the structure determination of [2.2.2.2]paracyclophane (4-PCP) was undertaken.

**Experimental.** Compound prepared by P. Baret and P. Chautemps (Laboratoire d'Etudes Dynamiques et Structurales de la Sélectivité, Grenoble). Colourless  $0.35 \times 0.40 \times 0.45$  mm crystal obtained by slow evaporation at 277 K from a solution in chloroform.

\* *Chemical Abstracts* name: pentacyclo[20.2.2.2<sup>4,7</sup>.2<sup>10,13</sup>.2<sup>16,19</sup>]. dodeca-4,6,10,12,16,18,22,24,25,27,29,31-dodecaene.

Measurements with a Siemens diffractometer,† Ni-filtered Cu  $K\alpha$  radiation, five-points method (Troughton, 1969),  $\omega$ -scan, no absorption correction. Lattice parameters refined with 15 reflexions. 2763 reflexions with  $I > 3\sigma(I)$  (4205 measured),  $\sin\theta/\lambda < 0.58$  Å<sup>-1</sup> ( $hkl, \bar{h}k\bar{l}$ ). Standard reflexions  $\bar{4}08, \bar{4}25, \bar{2}38$  (3% variation). Multisolution direct method (Germain, Main & Woolfson, 1971), least-squares refinement minimizing  $\sum w|F_o - F_c|^2$  (Busing & Levy, 1962). Positional and anisotropic parameters refined for C atoms, H-atom positional parameters calculated (not refined), isotropic temperature factors for H estimated (not refined). Weighting scheme approximately linear, obtained empirically by plotting  $(F_o - F_c)^2$  as a function of  $F_o$  (Rollett, 1965). Final  $R = 0.061$ ,  $wR = 0.084$  for 2763 reflexions.  $(\Delta/\sigma)_{\max} = 0.8$ , final  $\Delta\rho < 0.25$  e Å<sup>-3</sup>. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

**Discussion.** The atomic parameters are listed in Table 1; interatomic distances, valence and torsion angles in Table 2.‡

† Measurements performed at Laue-Langevin Institute, Grenoble, France.

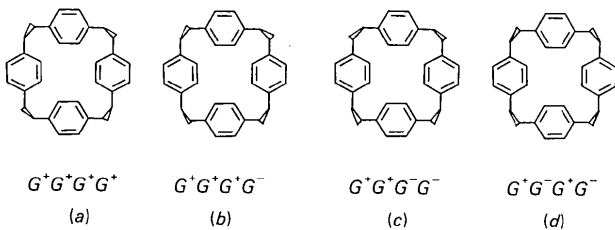
‡ Lists of structure factors, anisotropic thermal parameters, calculated H-atom parameters and mean-plane equations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42031 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. [2.2.2.2]Paracyclophane: atomic coordinates ( $\times 10^4$ ) with standard deviations in parentheses

$B_{eq} = 8\pi^2(U_1U_2U_3)^{1/3}$ . Standard deviations in  $B_{eq}$  are 0.2–0.3 Å<sup>2</sup>.

	x	y	z	$B_{eq}$ (Å <sup>2</sup> )
C(1)	3029 (3)	1904 (3)	3154 (1)	5.1
C(2)	2861 (3)	1198 (3)	3715 (1)	4.9
C(3)	3579 (3)	1362 (3)	4249 (1)	5.0
C(4)	4475 (3)	2250 (4)	4242 (2)	5.5
C(5)	4638 (3)	2972 (4)	3676 (2)	6.2
C(6)	3928 (3)	2801 (4)	3149 (2)	6.0
C(7)	5283 (3)	2399 (5)	4803 (2)	7.0
C(8)	4815 (4)	3126 (5)	5385 (2)	7.6
C(9)	3951 (3)	2301 (4)	5715 (2)	5.7
C(10)	2831 (4)	2637 (4)	5665 (2)	5.8
C(11)	2026 (3)	1834 (4)	5929 (2)	5.4
C(12)	2313 (3)	638 (3)	6263 (1)	4.6
C(13)	3424 (3)	313 (4)	6325 (1)	5.4
C(14)	4233 (3)	1132 (4)	6062 (2)	6.2
C(15)	1448 (3)	-244 (4)	6553 (1)	5.9
C(16)	528 (3)	-737 (4)	6086 (2)	6.6
C(17)	920 (3)	-1739 (4)	5594 (2)	5.3
C(18)	1453 (3)	-1317 (4)	5046 (1)	5.3
C(19)	1825 (3)	-2250 (4)	4609 (1)	5.5
C(20)	1674 (3)	-3651 (3)	4687 (1)	5.0
C(21)	1161 (3)	-4071 (4)	5233 (2)	6.5
C(22)	794 (4)	-3138 (5)	5672 (2)	6.9
C(23)	2065 (3)	-4693 (4)	4213 (2)	6.0
C(24)	1487 (3)	-4609 (4)	3540 (2)	6.0
C(25)	1765 (3)	-3336 (3)	3167 (1)	4.7
C(26)	2853 (3)	-3012 (4)	3027 (2)	5.7
C(27)	3104 (3)	-1830 (4)	2703 (2)	5.7
C(28)	2273 (3)	-901 (4)	2502 (1)	5.1
C(29)	1185 (3)	-1251 (4)	2628 (1)	5.4
C(30)	941 (3)	-2429 (4)	2962 (1)	5.1
C(31)	2539 (4)	431 (5)	2175 (2)	6.6
C(32)	2269 (3)	1727 (4)	2571 (2)	6.2

NMR studies of the 4-PCP molecule were previously performed in solution (Tabushi, Yamada & Kuroda, 1975; Olsson, Tanner, Thulin, Wennerström & Liljefors, 1981). The possible *gauche* conformations can be defined by the following scheme:



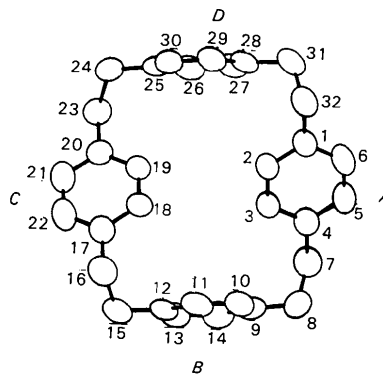
It was found that only (a) and (d) are compatible with low-temperature NMR spectra.

Fig. 1 and the angle values of Table 2 show that the (a) conformation is observed in the solid state. On the other hand, a (d)-type conformation was observed in 4-PCP-TCNE (Cohen-Addad *et al.*, 1984). These results are, thus, in good agreement with NMR studies. Both conformations are probably energetically equivalent and may be governed by steric interactions within the crystal.

Each of the four aromatic rings is planar (maximum deviation 0.014 Å) and their barycentres are in a plane *P* (maximum deviation 0.028 Å). The orientation of the four rings is analogous to that found in 4-PCP-TCNE. The dihedral angles between two

Table 2. [2.2.2.2]Paracyclophane: interatomic distances (Å), valence and torsion angles (°)

C(1)–C(2)	1.384 (4)	C(17)–C(22)	1.381 (5)
C(1)–C(6)	1.390 (5)	C(18)–C(19)	1.378 (5)
C(2)–C(3)	1.394 (4)	C(19)–C(20)	1.385 (5)
C(3)–C(4)	1.382 (4)	C(20)–C(21)	1.385 (5)
C(4)–C(5)	1.401 (5)	C(20)–C(23)	1.510 (5)
C(4)–C(7)	1.502 (5)	C(21)–C(22)	1.378 (6)
C(5)–C(6)	1.378 (5)	C(23)–C(24)	1.549 (5)
C(7)–C(8)	1.538 (5)	C(24)–C(25)	1.510 (5)
C(8)–C(9)	1.509 (6)	C(25)–C(26)	1.389 (5)
C(9)–C(10)	1.386 (5)	C(25)–C(30)	1.383 (5)
C(9)–C(14)	1.386 (5)	C(26)–C(27)	1.376 (5)
C(10)–C(11)	1.378 (5)	C(27)–C(28)	1.400 (5)
C(11)–C(12)	1.394 (5)	C(28)–C(29)	1.388 (5)
C(12)–C(13)	1.375 (5)	C(28)–C(31)	1.507 (5)
C(12)–C(15)	1.497 (5)	C(29)–C(30)	1.382 (5)
C(13)–C(14)	1.388 (5)	C(31)–C(32)	1.553 (6)
C(15)–C(16)	1.527 (5)	C(32)–C(1)	1.507 (5)
C(16)–C(17)	1.510 (5)		
C(17)–C(18)	1.399 (4)		
C(2)–C(1)–C(6)	117.3 (3)	C(22)–C(17)–C(18)	116.3 (3)
C(1)–C(2)–C(3)	121.2 (3)	C(22)–C(17)–C(16)	121.0 (3)
C(4)–C(3)–C(2)	121.5 (3)	C(18)–C(17)–C(16)	122.6 (3)
C(3)–C(4)–C(5)	117.2 (3)	C(19)–C(18)–C(17)	121.3 (3)
C(3)–C(4)–C(7)	122.2 (3)	C(18)–C(19)–C(20)	121.5 (3)
C(5)–C(4)–C(7)	120.5 (3)	C(21)–C(20)–C(19)	116.9 (3)
C(6)–C(5)–C(4)	121.0 (3)	C(21)–C(20)–C(23)	120.5 (3)
C(5)–C(6)–C(1)	121.8 (3)	C(19)–C(20)–C(23)	122.5 (3)
C(4)–C(7)–C(8)	114.9 (3)	C(22)–C(21)–C(20)	121.6 (3)
C(9)–C(8)–C(7)	113.4 (3)	C(21)–C(22)–C(17)	122.0 (3)
C(10)–C(9)–C(14)	116.7 (4)	C(20)–C(23)–C(24)	115.2 (3)
C(10)–C(9)–C(8)	121.9 (4)	C(25)–C(24)–C(23)	114.6 (3)
C(14)–C(9)–C(8)	121.4 (4)	C(30)–C(25)–C(26)	117.4 (3)
C(11)–C(10)–C(9)	122.2 (3)	C(30)–C(25)–C(24)	120.9 (3)
C(10)–C(11)–C(12)	120.3 (3)	C(26)–C(25)–C(24)	121.7 (3)
C(13)–C(12)–C(11)	117.3 (3)	C(27)–C(26)–C(25)	121.4 (3)
C(13)–C(12)–C(15)	121.3 (3)	C(26)–C(27)–C(28)	121.4 (3)
C(11)–C(12)–C(15)	121.4 (3)	C(29)–C(28)–C(27)	116.8 (3)
C(12)–C(13)–C(14)	121.3 (3)	C(29)–C(28)–C(31)	121.1 (3)
C(9)–C(14)–C(13)	121.3 (3)	C(27)–C(28)–C(31)	122.1 (3)
C(12)–C(15)–C(16)	114.7 (3)	C(30)–C(29)–C(28)	121.5 (3)
C(17)–C(16)–C(15)	113.4 (3)	C(29)–C(30)–C(25)	121.5 (3)
		C(28)–C(31)–C(32)	113.4 (3)
		C(1)–C(32)–C(31)	113.3 (3)
C(4)–C(7)–C(8)–C(9)	69.0 (5)	C(20)–C(23)–C(24)–C(25)	68.3 (4)
C(5)–C(4)–C(7)–C(8)	112.4 (4)	C(21)–C(20)–C(23)–C(24)	117.1 (4)
C(3)–C(4)–C(7)–C(8)	-70.7 (5)	C(19)–C(20)–C(23)–C(24)	-64.2 (4)
C(14)–C(9)–C(8)–C(7)	70.7 (5)	C(30)–C(25)–C(24)–C(23)	-120.1 (3)
C(10)–C(9)–C(8)–C(7)	-106.7 (5)	C(26)–C(25)–C(24)–C(23)	58.4 (4)
C(12)–C(15)–C(16)–C(17)	67.5 (4)	C(28)–C(31)–C(32)–C(1)	69.3 (4)
C(13)–C(12)–C(15)–C(16)	-125.3 (3)	C(29)–C(28)–C(31)–C(32)	65.2 (4)
C(11)–C(12)–C(15)–C(16)	55.8 (4)	C(27)–C(28)–C(31)–C(32)	-113.7 (4)
C(18)–C(17)–C(16)–C(15)	-80.6 (4)	C(2)–C(1)–C(32)–C(31)	-81.5 (4)
C(22)–C(17)–C(16)–C(15)	97.8 (4)	C(6)–C(1)–C(32)–C(31)	99.1 (4)

Fig. 1. Conformation of [2.2.2.2]paracyclophane: projection of the molecule on the plane *P* defined by the phenyl-ring barycentres.

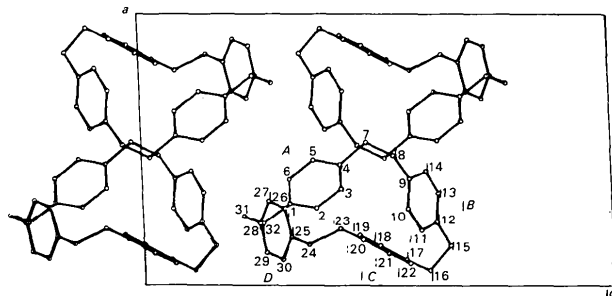


Fig. 2. Intermolecular arrangement in [2.2.2.2]paracyclophane: projection on the *ac* plane.

adjacent rings are:  $A,B = 50.6$ ,  $B,C = 64.0$ ,  $C,D = 62.3$ ,  $A,D = 53.9^\circ$  with standard deviations of  $0.8^\circ$ .

The *A* and *C* rings make angles of respectively  $-33$  and  $34^\circ$  with plane *P*, while *B* and *D* make angles of  $83$  and  $85^\circ$  with that plane. This arrangement is less planar than found in the related molecule [2.2.2.2]paracyclophanetetraene (Ljungström, Lindquist & Wennerström, 1978).

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## Structures du $N^1$ -(Méthoxy-6 méthyl-2 pyrimidinyl-4)sulfanilamide (Sulfamétomidine), $C_{12}H_{14}N_4O_3S$ , et d'un de ses Solvates, $C_{12}H_{14}N_4O_3S \cdot H_2O$

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**Abstract.** Sulfamethomidine, form I:  $M_r = 294.33$ , triclinic,  $P\bar{1}$ ,  $a = 14.180$  (5),  $b = 11.643$  (4),  $c = 8.619$  (2) Å,  $\alpha = 91.08$  (2),  $\beta = 101.39$  (2),  $\gamma = 90.62$  (3)°,  $V = 1395$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.393$ ,  $D_x = 1.40$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $F(000) = 616$ ,  $\mu = 0.234$  mm<sup>-1</sup>, room temperature, final  $R = 0.0514$  for 3221 observed reflections. Sulfamethomidine monohydrate, form SII:  $M_r = 312.35$ , orthorhombic,  $P2_12_12_1$ ,  $a = 7.525$  (2),  $b = 16.924$  (4),  $c = 11.785$  (3) Å,  $V = 1501.0$  (7) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.365$ ,  $D_x = 1.38$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,

$F(000) = 656$ ,  $\mu = 0.225$  mm<sup>-1</sup>, room temperature, final  $R = 0.0405$  for 1108 observed reflections. The asymmetric unit of form I contains two independent molecules. The molecular structures of I and SII show significant differences in bond lengths and bond angles because of the transfer of the proton on the amide nitrogen to the pyrimidinyl nitrogen in SII [value for SII first, average value for molecules *A* and *B* of I second: S(8)–N(11) 1.582 (4), 1.644 (3); C(12)–N(11) 1.339 (6), 1.400 (6); C(12)–N(17) 1.375 (6), 1.342 (5) Å. S(8)–N(11)–C(12) 122.1 (3), 125.9 (3);

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