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## The Crystal Structure of 3-(*N*-Phenyl)aminopyrrolidine-2,5-dione

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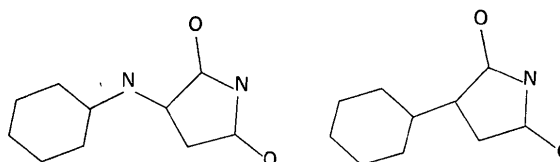
(Received 30 January 1973; accepted 2 February 1973)

The structure of 3-(*N*-phenyl)aminopyrrolidine-2,5-dione,  $C_{10}H_{10}N_2O_2$ , has been determined from X-ray intensity data collected on a Stoe-Güttinger automatic off-line Weissenberg goniometer. The space group is *Pbca*, with  $a = 24.756$ ,  $b = 7.453$ ,  $c = 9.943$  Å and  $Z = 8$ . The structure was solved by direct methods. The positional and vibrational parameters were refined by the method of least squares with anisotropic thermal parameters for the non-hydrogen atoms only. The final  $R$  was 0.049. The standard deviations of the bond lengths and angles were less than 0.003 Å and 0.2° respectively. Two molecules related by a centre of symmetry form a dimer through N-H...O bonds.

### Introduction

The synthesis of many succinimide compounds has recently been studied by Seres (1970). This work is part of a systematic programme to elucidate the crystal structure of these compounds. We present here the crystal and molecular structure of 3-(*N*-phenyl)aminopyrrolidine-2,5-dione [Fig. 1(a)] (hereafter PHA-PD).

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(a) PHA-PD. (b) PH-PD(O).

Experimental

PHA-PD crystallized from ethanol as prismatic needles. Lattice parameters were determined from Weissenberg photographs and refined on a diffractometer.

Crystal data

C<sub>10</sub>H<sub>10</sub>N<sub>2</sub>O<sub>2</sub>, *M* = 190.2. Orthorhombic.  
*a* = 24.756 (8), *b* = 7.453 (6), *c* = 9.943 (10) Å.  
*V* = 1835 Å<sup>3</sup>, *Z* = 8,  
*D<sub>c</sub>* = 1.378, *D<sub>x</sub>* = 1.374 g cm<sup>-3</sup>,  
*F*(000) = 800,  
 $\mu$  for Cu *K*α ( $\lambda$  = 1.54178 Å) = 6.1 cm<sup>-1</sup>,  
 Space group *Pbca* (No. 61).

The intensities were measured on a Stoe-Güttinger automatic Weissenberg goniometer using Ni-filtered Cu *K*α radiation to a maximum counter-angle of 140°. Data reduction including estimation of  $\sigma(F_o)$  was accomplished with a program written by Paulus (1966). All intensity measurements (*hk*0 to *hk*9) were carried out with a well shaped crystal of dimensions 0.3 × 0.22 mm (needle axis = *c*). No absorption correction was applied ( $\mu R < 0.2$ ). 1662 reflexions were collected, of which 228 were unobserved [*F<sub>o</sub>* < 2.0σ(*F<sub>o</sub>*)].

Table 2. Observed and calculated structure factors

<i>h</i>	<i>k</i>	<i>l</i>	<i>F<sub>o</sub></i>	<i>F<sub>c</sub></i>	<i>F<sub>o</sub></i>	<i>F<sub>c</sub></i>	<i>F<sub>o</sub></i>	<i>F<sub>c</sub></i>	<i>F<sub>o</sub></i>	<i>F<sub>c</sub></i>	<i>F<sub>o</sub></i>	<i>F<sub>c</sub></i>	<i>F<sub>o</sub></i>	<i>F<sub>c</sub></i>
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Table 1. Final fractional atomic coordinates and anisotropic (isotropic for the hydrogen atoms) vibration parameters for PHA-PD

The *b<sub>ij</sub>* are defined by  $T = \exp[-10^{-5}(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + 2b_{12}hk + 2b_{13}hl + 2b_{23}kl)]$ .  
 The e.s.d.'s are in parentheses and refer to the least significant digits.

	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>b<sub>11</sub></i>	<i>b<sub>22</sub></i>	<i>b<sub>33</sub></i>	<i>b<sub>12</sub></i>	<i>b<sub>13</sub></i>	<i>b<sub>23</sub></i>
C(1)	0.18859 (7)	0.12075 (28)	0.49043 (17)	148 (3)	1990 (46)	1103 (20)	-49 (10)	33 (6)	-16 (24)
C(2)	0.23171 (7)	0.14384 (30)	0.57674 (21)	140 (3)	2059 (52)	1480 (25)	-5 (11)	38 (7)	-265 (28)
C(3)	0.22978 (8)	0.08266 (33)	0.70658 (19)	155 (4)	2409 (56)	1337 (26)	100 (11)	-47 (7)	-308 (28)
C(4)	0.18333 (8)	-0.00370 (29)	0.75025 (20)	192 (4)	2191 (50)	1113 (21)	143 (12)	-9 (7)	0 (25)
C(5)	0.13979 (8)	-0.02700 (28)	0.66648 (19)	153 (3)	1837 (43)	1186 (22)	18 (10)	35 (7)	37 (24)
C(6)	0.14122 (8)	0.03670 (26)	0.53469 (17)	144 (3)	1637 (41)	1106 (22)	-19 (9)	8 (6)	-51 (22)
C(7)	0.09002 (7)	0.11087 (28)	0.32955 (16)	142 (3)	1803 (43)	1104 (20)	-94 (10)	4 (6)	106 (23)
C(8)	0.03049 (8)	0.12041 (27)	0.29353 (17)	150 (3)	1772 (44)	1139 (21)	-77 (10)	11 (7)	85 (23)
C(9)	0.11640 (7)	0.02964 (29)	0.20352 (19)	135 (3)	2012 (46)	1362 (23)	5 (10)	-4 (7)	25 (25)
C(10)	0.07209 (7)	0.02429 (27)	0.10038 (17)	141 (3)	1953 (47)	1161 (21)	-37 (10)	10 (7)	27 (24)
N(1)	0.09699 (7)	0.00968 (25)	0.45222 (14)	158 (3)	2753 (50)	1214 (20)	-157 (10)	-25 (6)	434 (23)
N(2)	0.02481 (6)	0.08272 (25)	0.16014 (13)	135 (3)	2430 (44)	1071 (17)	0 (9)	-8 (5)	-74 (20)
O(1)	-0.00633 (5)	0.15591 (22)	0.36955 (12)	156 (3)	2986 (45)	1365 (16)	-35 (9)	83 (5)	-162 (20)
O(2)	0.07538 (6)	-0.02220 (22)	-0.01584 (12)	166 (3)	3037 (44)	1206 (15)	-11 (8)	32 (5)	-213 (20)

Table 1 (cont.)

	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>B</i>
H(C1)	0.189 (2)	0.168 (6)	0.402 (4)	2.5 (9)
H(C2)	0.263 (2)	0.212 (7)	0.539 (4)	4.5 (12)
H(C3)	0.258 (2)	0.099 (8)	0.770 (5)	5.1 (13)
H(C4)	0.180 (2)	-0.043 (7)	0.834 (4)	3.9 (11)
H(C5)	0.107 (2)	-0.080 (6)	0.692 (3)	2.5 (9)
H(C7)	0.104 (2)	0.234 (8)	0.336 (4)	4.9 (11)
H(C91)	0.149 (2)	0.099 (7)	0.173 (4)	3.4 (10)
H(C92)	0.131 (2)	-0.078 (6)	0.215 (4)	3.1 (10)
H(N1)	0.072 (2)	0.042 (6)	0.496 (4)	3.4 (11)
H(N2)	-0.008 (2)	0.076 (7)	0.102 (4)	3.8 (10)

Structure determination and refinement

The signs of 110 reflexions with *E* greater than 1.70 were determined by Long's (1965) program. The starting set of reflexions was

<i>h</i>	<i>k</i>	<i>l</i>	<i>E</i>	<i>h</i>	<i>k</i>	<i>l</i>	<i>E</i>
2	2	1	2.18	5	1	9	3.87
9	0	8	3.23	15	7	4	2.21
12	3	6	2.22	9	2	7	2.95
				16	5	7	3.09

The first three were used to fix the origin. Both iteration procedures NBACK=0 and 1 (with a consistency index of 0.89) gave the correct solution (all signs were determined correctly as was seen later). The corresponding *E* map could be unambiguously

Table 3. Bond lengths and angles with their standard deviations

Distances			
C(1)—C(2)	1.380 (3) Å	C(1)—H(C1)	0.95 (4) Å
C(2)—C(3)	1.371 (3)	C(2)—H(C2)	1.00 (5)
C(3)—C(4)	1.386 (3)	C(3)—H(C3)	0.95 (5)
C(4)—C(5)	1.373 (3)	C(4)—H(C4)	0.89 (4)
C(5)—C(6)	1.394 (3)	C(5)—H(C5)	0.95 (4)
C(6)—C(1)	1.400 (3)		
C(6)—N(1)	1.383 (3)	N(1)—H(N1)	0.78 (5)
N(1)—C(7)	1.444 (3)		
C(7)—C(8)	1.518 (3)	C(7)—H(C7)	0.99 (6)
C(7)—C(9)	1.537 (3)	C(9)—H(C91)	1.00 (4)
C(9)—C(10)	1.502 (3)	C(9)—H(C92)	0.88 (5)
C(8)—N(2)	1.363 (3)		
C(10)—N(2)	1.383 (2)	N(2)—H(N2)	1.01 (4)
C(8)—O(1)	1.213 (2)		
C(10)—O(2)	1.209 (2)		
Angles			
C(6)—C(1)—C(2)	120.53 (17)°		
C(1)—C(2)—C(3)	121.11 (18)		
C(2)—C(3)—C(4)	118.60 (18)		
C(3)—C(4)—C(5)	121.31 (19)		
C(4)—C(5)—C(6)	120.47 (19)		
C(5)—C(6)—C(1)	117.96 (17)		
C(1)—C(6)—N(1)	122.81 (16)		
C(5)—C(6)—N(1)	119.19 (18)		
C(6)—N(1)—C(7)	121.29 (17)		
N(1)—C(7)—C(8)	109.85 (15)		
N(1)—C(7)—C(9)	115.59 (18)		
C(8)—C(7)—C(9)	103.80 (14)		
C(7)—C(8)—N(2)	108.66 (15)		
C(8)—N(2)—C(10)	113.31 (15)		
C(9)—C(10)—N(2)	108.44 (15)		
C(7)—C(9)—C(10)	104.88 (15)		
C(9)—C(10)—O(2)	127.65 (17)		
N(2)—C(10)—O(2)	123.91 (16)		
C(7)—C(8)—O(1)	126.33 (16)		
N(2)—C(8)—O(1)	125.00 (18)		
H(C1)—C(1)—C(6)	118.4 (23)°		
H(C1)—C(1)—C(2)	120.9 (23)		
H(C2)—C(2)—C(1)	115.5 (25)		
H(C2)—C(2)—C(3)	123.3 (25)		
H(C3)—C(3)—C(2)	123.4 (30)		
H(C3)—C(3)—C(4)	117.9 (30)		
H(C4)—C(4)—C(3)	121.9 (29)		
H(C4)—C(4)—C(5)	116.7 (29)		
H(C5)—C(5)—C(4)	124.9 (21)		
H(C5)—C(5)—C(6)	114.6 (21)		
H(N1)—N(1)—C(6)	104.2 (32)		
H(N1)—N(1)—C(7)	102.3 (32)		
H(C7)—C(7)—N(1)	113.0 (25)		
H(C7)—C(7)—C(8)	108.6 (27)		
H(C7)—C(7)—C(9)	105.4 (25)		
H(C91)—C(9)—C(10)	113.0 (23)		
H(C91)—C(9)—C(7)	112.6 (25)		
H(C91)—C(9)—H(C92)	100.7 (38)		
H(C92)—C(9)—C(7)	115.0 (25)		
H(C92)—C(9)—C(10)	111.0 (27)		
H(N2)—N(2)—C(8)	130.9 (23)		
H(N2)—N(2)—C(10)	115.3 (23)		

interpreted, resulting in the location of all heavy atoms.

The model derived from the *E* map ( $R=0.27$ ) was then refined by the method of full-matrix least squares (Busing, Martin & Levy, 1962) using the weighting scheme  $w=1/[\sigma(F_o)]^2$ . After eight cycles of isotropic refinement ( $R=0.13$ ) using reflexions of  $F_o \geq 2.0\sigma(F_o)$ , it turned out that 10 were affected by extinction. These were omitted from further refinement.

The positions of the hydrogen atoms were generated by the *BONDLA* program of the *X-RAY-63* system (1963) and were refined isotropically.

A further six cycles of refinement were performed with anisotropic vibrational parameters for the heavy atoms and isotropic ones for hydrogen atoms until the shifts of the positional and vibrational parameters were less than  $0.3\sigma$  and  $0.1\sigma$ , respectively. The final *R* is 0.049 and  $R_w = (\sum w\Delta^2 / \sum w|F_o|^2)^{1/2} = 0.054$ .

The positions of the hydrogen atoms were checked on a difference-map, with good results (Fig. 2).

All atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962). The final positional and vibrational parameters are given in Table 1. The final structure factor calculation (Table 2) with inclusion of the reflexions affected by extinction and 228 unobserved reflexions gave an *R* value of 0.055. Reflexions affected by extinction were corrected in the form  $I_{\text{obs, corr}} = I_o + 0.184 \times 10^{-6}(I_o - I_1)$  when  $I_o$  was greater than  $I_1 = 5 \cdot 10^6$ .

## Discussion

The bond lengths and angles with their estimated standard deviations are given in Table 3 and are shown in Fig. 3.

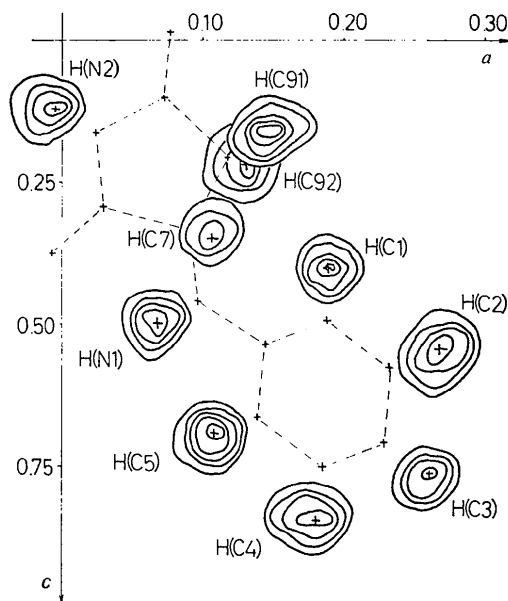


Fig. 2. Difference map showing hydrogen atoms. Contours are at intervals of  $0.1 e \text{ \AA}^{-3}$  starting at  $0.1 e \text{ \AA}^{-3}$ .

The PHA-PD molecule differs from the previously described PHA-PD(*o*) [Fig. 1(*b*)] molecule (Argay & Kálmán, 1973) only in that the succinimide ring is not linked to the phenyl group directly, but through a nitrogen atom [Fig. 1(*a*)].

The phenyl ring was found to be planar within experimental error (Table 4). The mean C-C bond length of 1.384 (3) Å is in good agreement with the values found in other structures with a C<sub>6</sub>H<sub>5</sub>-NH-group (e.g. 1.386 (3) Å, Brown, 1966; 1.384 (3) Å, Jarchow, 1969). The mean C(sp<sup>2</sup>)-H bond distance is rather short (0.95 Å). The r.m.s. deviation of the C-C bond length in PHA-PD (0.005 Å) is considerably smaller than in PH-PD(*o*) (0.011 Å), owing to the use of about twice as many reflexions for approximately the same number of parameters as in PH-PD(*o*). This supports our remark (Argay & Kálmán, 1973) on the rather distorted phenyl ring in PH-PD(*o*).

The angle C(9)-C(7)-N(1) is significantly greater than the angle C(8)-C(7)-N(1). This phenomenon and the N(1)···O(1)=2.90 Å and O(1)···H(N1)=2.47 Å distances suggest that a hydrogen bond of type N-H···O is formed within the molecule. The deviations of atoms from the best planes (plane II/*a* excluding the oxygen atoms, plane II/*b* including them) of the succinimide ring indicates that the latter is not quite planar and especially that the O(1) atom lies rather out of it. This is presumably due to the effect of the intramolecular hydrogen bond. It is noteworthy

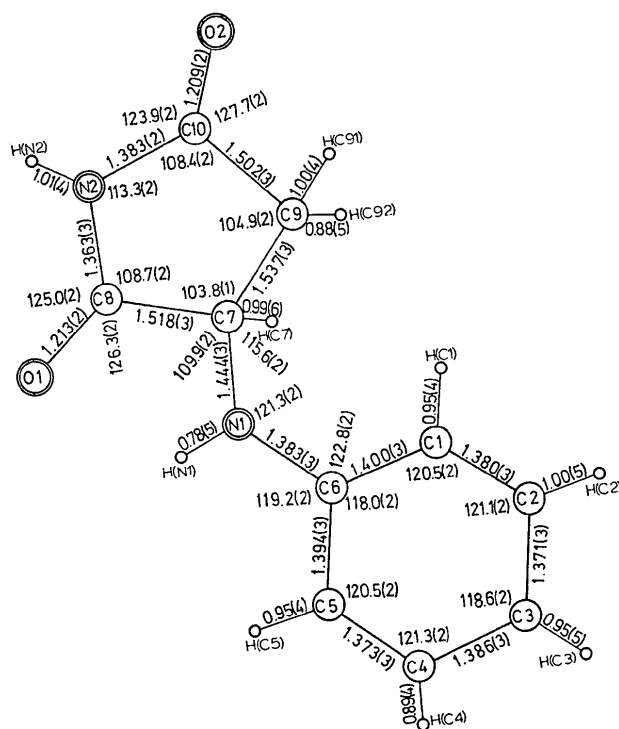


Fig. 3. Bond distances and angles with their e.s.d.'s for the title compound.

Table 4. *Least-squares planes*

The equation of the plane through the C atoms of the phenyl ring:

$$\text{I} \quad 9.2057x + 6.5497y + 2.9734z + 0.52144 = 0$$

The equations of the planes through the atoms of the succinimide-ring:

$$\text{IIa} \quad 4.6402x + 7.0109y - 2.8122z - 0.21424 = 0$$

$$\text{IIb} \quad 3.8052x + 7.0970y - 2.6234z - 0.18760 = 0.$$

	Distances (Å) from		
	plane I	plane IIa	plane IIb
C(1)	-0.008*	-0.128	
C(2)	0.003*	-0.247	
C(3)	0.003*	0.558	
C(4)	-0.003*	1.499	
C(5)	-0.003*	1.629	
C(6)	0.009*	0.805	0.792
N(1)	-0.006	0.968	0.936
C(7)	0.356	-0.054*	-0.077*
C(8)	0.859	0.054*	-0.013*
C(9)	-0.794	0.039*	0.068*
C(10)	-0.728	-0.008*	0.004*
N(2)	0.268	-0.030*	-0.074*
O(1)	1.657	0.189	0.075*
O(2)	-1.408	-0.024	0.017*
H(C1)	0.031	-0.712	
H(C2)	0.053	-0.977	
H(C3)	0.040	0.485	
H(C4)	0.027	2.024	
H(C5)	0.033	2.226	
H(C7)	1.050	-0.967	-0.990
H(C91)	-0.728	-0.684	-0.627
H(C92)	-1.596	0.757	0.806
H(N1)	0.559	0.980	0.916
H(N2)	0.355	0.009	-0.050
Angle to plane I		47.7°	45.5°
Angle to plane IIa			45.4°
Angle to plane IIb	75.5°	45.0°	

\* Denotes atoms defining the plane.

that in PH-PD(*o*) and in succinimide (Mason, 1961), where only intermolecular hydrogen bonds are formed, the succinimide group is almost planar. Similarly to PH-PD(*o*) and succinimide (Mason, 1961) two PHA-PD molecules form a dimer through N[2] (*x*, *y*, *z*)···O[2] (*x̄*, *ȳ*, *z̄*) and its symmetry equivalent O[2] (*x*, *y*, *z*)···N[2] (*x̄*, *ȳ*, *z̄*) linked by hydrogen bonds of 2.90 Å.

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*Acta Cryst.* (1973). B29, 1190

## The Crystal and Molecular Structure of 2-Phenyl-4-(4-bromophenyl)-5,6-dimethyl-5,6-dihydro-4*H*-1,3-oxazine, C<sub>18</sub>H<sub>18</sub>NOBr

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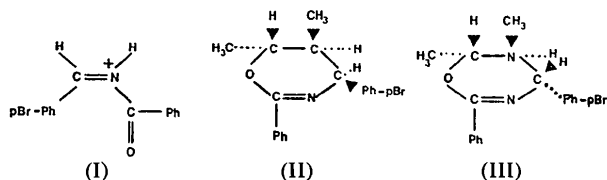
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C<sub>18</sub>H<sub>18</sub>NOBr is monoclinic, space group  $P2_1/c$ , with  $a=11.985$  (6),  $b=18.576$  (11),  $c=7.260$  (6) Å,  $\beta=94.6$  (1)° and  $Z=4$ . The three-dimensional intensity data were collected on a single-crystal automated diffractometer (1651 observed structure factors). The structure was solved by Patterson and Fourier syntheses and refined by the least-squares method. The final  $R$  value, with isotropic hydrogen atoms, is 0.062. The heterocyclic ring is in a *trans-syn* configuration, with the two methyl groups *equatorial* and *trans*, while the bromophenyl substituent is *axial* and *syn* with respect to the adjacent methyl group. A survey of the bond lengths suggests a weak delocalization of the C(4)–N double bond to the other bonds to which C(4) contributes. The conformation of the oxazine ring is the 'half-boat' type, with five atoms nearly planar and one, C(2), out of plane. Molecules are packed with van der Waals contacts.

### Introduction

In the reaction between benzonitrile and *p*-bromobenzaldehyde in acetic acid solution, catalyzed by H<sub>2</sub>SO<sub>4</sub> (Schmidt, 1970; Giordano, Ribaldone & Borsotti, 1971) the formation of an amidomethyl ion (I) can be hypothesized. Its reaction with *trans*-butene gives two oxazine diastereomers (II) and (III), by means of a stereospecific 1,4-cycloaddition on the olefin.



The two products are in the ratio 4:1 and can be identified as *trans-syn* and *trans-anti* configurations.\*

An X-ray structural determination of the oxazine obtained should give confirmation of n.m.r. configurational and conformational studies of these compounds (Giordano & Abis, 1973) and of corresponding thia-

zines (Abis & Giordano, 1973), and seemed useful in a class of heterocyclic compounds where no crystallographic literature was found.

### Experimental

Colourless prismatic crystals were provided by Dr C. Giordano of the Organic Chemistry Research Centre, Montecatini Edison Co., Novara. For crystallographic measurements, a crystal was cut of dimensions approximately 0.15 × 0.3 × 0.4 mm. The unit-cell dimensions were determined from rotation and Weissenberg photographs and refined by a least-squares analysis of 12 reflexions on a four circle automated Picker diffractometer, using Mo  $K\alpha$  radiation ( $\lambda=0.71609$  Å). Weissenberg photographs showed the space group extinctions  $k=2n+1$  for  $0k0$  and  $l=2n+1$  for  $h0l$ ; the space group was therefore  $P2_1/c$ .

The crystal data are: C<sub>18</sub>H<sub>18</sub>NOBr; M.W. 344.254; space group  $P2_1/c$ ;  $a=11.985$  (6),  $b=18.765$  (11),  $c=7.260$  (6) Å,  $\beta=94.6$  (1)°,  $Z=4$ ,  $V=1611.2$  Å<sup>3</sup>,  $\rho_c=1.42$ ,  $\rho_o=1.40$  g cm<sup>-3</sup>,  $\mu=33.4$  cm<sup>-1</sup>.

Intensities were collected on the Picker diffractometer with Zr-filtered Mo  $K\alpha$  radiation. The mode was  $\theta$ - $2\theta$  scan, the scan speed was 2°/min over angles of 2°. Stationary-crystal and counter background counts of 20 sec were taken at each end of the scan. The measurements were made up to  $2\theta=55^\circ$ . The reflexions 560,

\* The same nomenclature used for cyclohexane was adopted, where *cis-trans* refers to the configuration of substituents on 5 and 6 [i.e. C(2) and C(3)], while *syn-anti* refers to substituents on 4 and 5 [i.e. C(1) and C(2)].