

In the sulphonate group electron densities are found in all S–O bonds and negative regions occur between the bonds. Negative regions are also observed at all oxygen sites, in agreement with earlier studies (*cf.* Almlöf, Kvik & Thomas, 1973). Electron densities corresponding to the lone pairs are observed around all oxygen atoms, but the densities are in most cases smeared so that no lone-pair directions can be deduced from the maps.

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Conformation of Non-Aromatic Ring Compounds. LXXXVI. The Crystal and Molecular Structure of 4-Phenyl-2,4,6-triazatricyclo[5,2,2,0^{2,6}]undecane-3,5-dione at -170°C

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The camphane-like compound 4-phenyl-2,4,6-triazatricyclo[5,2,2,0^{2,6}]undecane-3,5-dione crystallizes in the monoclinic system with $a = 5.8399$ (6), $b = 15.6402$ (16), $c = 6.7462$ (7) Å, $\beta = 100.74$ (5) $^{\circ}$ at -170°C , space group $P2_1$ and $Z = 2$. 1756 significant reflexions were collected on a three-circle diffractometer at -170°C with Mo $K\alpha$ radiation. The structure was solved by direct methods. Block-diagonal refinement including all atoms reduced the R index to 3.54%. The bicyclo[2,2,2]octane ring is slightly distorted. In this ring system the hybridization of the two vicinal atoms N(2) and N(6) is pyramidal, while that of the third nitrogen atom, N(4), is planar. The 1,2,4-triazacyclopentane-2,4-dione ring is slightly puckered: its largest torsion angle is -8.5° and its conformation an envelope with N(4) as flap. The phenyl ring has an asymmetric orientation with respect to the five-membered ring.

Introduction

This study is a continuation of earlier investigations (Altona & Sundaralingam, 1970; Altona & Sundaralingam, 1972; Offereins, Altona & Romers, 1973) into the twist of norbornane- and camphane-like structures containing nitrogen atoms in pyramidal hybridization. The title compound (hereinafter PTT) contains a bicyclo[2,2,2]octane nucleus in which two vicinal secondary carbon atoms are substituted by nitrogen atoms. Its chemical structure and the numbering of atoms are indicated in Fig. 1.

Evidence of a twisting motion about the threefold symmetry axis of bicyclo[2,2,2]octane has been reported by Ermer & Dunitz (1969) for the solid-state structure of bicyclo[2,2,2]octane-1,4-dicarboxylic acid (BOD) and by Yokozeki, Kuchitsu & Morino (1970) for the gas-phase structure of bicyclo[2,2,2]octane. This motion does not lead to a deviation from D_{3h} symmetry for the average structures observed; however, the lower D_3 symmetry has been reported by Cameron, Ferguson & Morris (1968) for the solid-state molecular structure of 1-*p*-bromobenzenesulphonyloxymethylbicyclo[2,2,2]octane (BBO). Apart from the two aza atoms,

N(2) and N(6), PTT contains a third nitrogen atom N(4). The latter, which in pyramidal hybridization might induce asymmetric distortion, was found to occur in planar hybridization.

Experimental

The title compound has not been described before. It was synthesized (Hameeteman, de Man & Altona, 1972) from a known precursor, the 8,9-unsaturated compound (Gillis & Hagarty, 1967), via a hydrogenation procedure as described by Askani (1965). The compound was recrystallized from methanol in the form of transparent irregular blocks. A specimen with dimensions $0.26 \times 0.28 \times 0.51$ mm was selected and mounted along its longest edge on a goniometer head. This direction turned out to be the longest axis (the monoclinic b axis) of the unit cell. The lattice constants (Table 1) at 20°C and at -170°C were measured manually with a three-circle diffractometer. The observed extinctions $0k0$ for k odd indicate the space groups $P2_1$ or $P2_1/m$.

Table 1. *Crystal data of PTT*

4-Phenyl-2,4,6-triazatricyclo[5,2,2,0^{2,6}]undecane-3,5-dione
 $\text{C}_{14}\text{O}_2\text{N}_3\text{H}_{15}$, melting point 211°C , density $d_{20}^{20} = 1.356$ g cm⁻³
 $d_x^{20} = 1.362$ g cm⁻³, space group $P2_1$, $Z = 2$ molecules per unit cell, $\mu(\text{Mo } K\alpha) = 1.05$ cm⁻¹, $F(000) = 272$

	20°C	-170°C
a	5.9770 (6) Å	5.8399 (6) Å
b	15.7468 (16)	15.6402 (16)
c	6.7302 (7)	6.7462 (7)
β	100.71 (5)°	100.74 (5)°

The reflexion intensities at -170°C were recorded during eight consecutive days with a three-circle diffractometer using graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å). The cooling system adopted has been described by van Bolhuis (1971). The θ - 2θ scan was employed and the scan width varied from 0.9° at $\theta_{\min} = 4^\circ$ to 1.4° at $\theta_{\max} = 30^\circ$. After each series of 19 reflexions one of three reference reflexions ($\bar{1}21$, $1\bar{1}1$ or $\bar{1}71$) was recorded to monitor instrumental fluctuations and crystal stability.

1817 symmetry-independent reflexions were scanned. Of these, 1756 having counts larger than twice the background count were accounted as observed during the refinement. The non-observed reflexions were also taken into consideration for the calculation of normalized structure factors (see below). The intensities were corrected for the decrease in scattering power of the crystal by means of a fifth-order polynomial function of the exposure time. In view of the crystal size and the relatively small linear absorption coefficient the transmission was estimated to vary between 97 and 99%. Hence the correction for absorption was omitted. Finally the reflexions were reduced to structure factors, each being assigned an estimated standard deviation σ_F . A preliminary scale and overall isotropic

temperature factor ($B = 0.13$ Å²) were obtained by means of a Wilson plot.

Solution of the structure

The chemical structure (Fig. 1) *a priori* does not exclude a mirror plane bisecting the lines C(8)–C(9), C(10)–C(11), N(2)–N(6) and coinciding with atoms N(4), C(12) and C(15). However, such a molecule with symmetry C_s cannot be accommodated in the unit cell with symmetry $P2_1/m$, since its largest dimension (~ 11.8 Å) would be perpendicular to $[010]$ and would exceed the longest repeat unit in the plane (010) . The non-centrosymmetric space group $P2_1$ must therefore be accepted as correct.

The phases of the structure factors were determined by the multisolution method (Germain, Main & Woolfson, 1971), with a program designed by Motherwell & Isaacs (1971). The structure factors were converted into their normalized form E . To fulfil the statistical demands for $\langle |E| \rangle$ and $\langle |E^2 - 1| \rangle$ listed in Table 2, the scale and isotropic temperature factors were modified using the information of *all* scanned reciprocal-lattice points.

Table 2. *Direct-methods data of PTT*

	Theory	Exp.	
$\langle E^2 - 1 \rangle$	0.736	0.806	acentric distribution
$\langle E \rangle$	0.886	0.864	
Number of E values	> 1.5 is 211		

h	k	l	E	φ (radians)		
1, 10, 5			3.17	0	} origin-defining phases	
2, 0, 1			3.00	0		
4, 1, 4			2.51	$\pi/4$		
2, 12, 4			2.88	$\pi/4, 3\pi/4, 5\pi/4, 7\pi/4$		
Solution	R_{Karle}	α'	t	$\langle \varphi \rangle$	$\langle \varphi \rangle$	Triplets
1	29	133	69	-0.034π	0.364π	5725
2	31	131	68	-0.032π	0.454π	5630
3	17	170	81	-0.098π	0.494π	6157
4	16	173	81	-0.012π	0.464π	6274

See Kennard *et al.* (1972) and Drew *et al.* (1969) for the meaning of R_{Karle} , α_{Karle} , and t ; $\alpha' = \alpha_{\text{Karle}} \sigma^{3/2} (2\sigma_3)^{-1}$.

The ideal situation for the fixation of the origin of a unit cell with space group $P2_1$ is the assignment of three zero phases to a general reflexion hkl and two

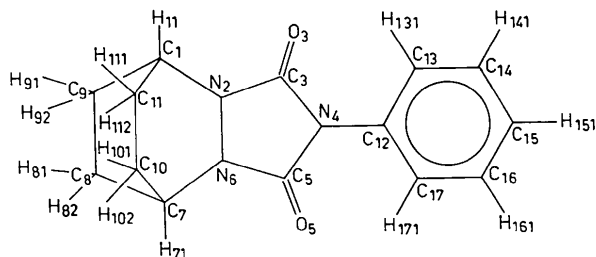


Fig. 1. Structural diagram and numbering of the molecule PTT.

the values 0.0029, 0.0031, 0.0033 and 0.037 Å for C–O, C–N, C–C and C–H bond lengths respectively. The corresponding standard deviations in valency and torsion angles involving the heavy atoms are 0.2 and 0.3°, respectively.

Discussion of the structure

The bond lengths and valency angles are depicted in Fig. 2(a) and (b). The difference between the smallest

Table 5. Fractional coordinates (10^3 units) and isotropic B values (10 \AA^2 units) of hydrogen atoms

Estimated standard deviations in the least significant digit are given in parentheses.

	x	y	z	B
H(11)	-33 (5)	63 (2)	74 (4)	19 (5)
H(71)	-319 (4)	290 (2)	-295 (4)	9 (4)
H(111)	-478 (5)	246 (2)	-21 (5)	22 (5)
H(112)	-595 (5)	195 (2)	-230 (5)	20 (5)
H(101)	-453 (5)	75 (2)	-51 (5)	24 (5)
H(102)	-361 (4)	122 (2)	150 (4)	16 (4)
H(91)	-174 (6)	44 (2)	-293 (5)	26 (5)
H(92)	78 (4)	87 (2)	-265 (4)	16 (4)
H(81)	-337 (5)	155 (2)	-467 (4)	13 (4)
H(82)	-95 (5)	202 (2)	-455 (5)	25 (5)
H(131)	543 (6)	379 (2)	183 (5)	28 (6)
H(141)	746 (5)	474 (2)	416 (4)	21 (5)
H(151)	619 (4)	505 (2)	718 (4)	16 (4)
H(161)	274 (5)	440 (2)	788 (5)	22 (5)
H(171)	71 (4)	343 (1)	561 (3)	5 (3)

Table 6. Thermal parameters U_{ij} (10^3 \AA^2 units) of non-hydrogen atoms

Estimated standard deviations in the least significant digit are given in parentheses. The temperature factor is defined by

$$\exp[-2\pi^2 \sum_{i,j} h_i h_j a_i^* a_j^* U_{ij}], \quad i, j = 1, 2, 3.$$

	U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{23}$	$2U_{13}$
C(1)	16 (1)	7 (1)	20 (1)	-2 (1)	-2 (1)	0 (1)
N(2)	16 (1)	4 (1)	18 (1)	1 (1)	2 (1)	-1 (1)
C(3)	13 (1)	6 (1)	18 (1)	0 (1)	-4 (1)	5 (1)
N(4)	16 (1)	5 (1)	14 (1)	-1 (1)	-1 (1)	0 (1)
C(5)	15 (1)	8 (1)	14 (1)	-3 (1)	0 (1)	0 (1)
N(6)	18 (1)	5 (1)	15 (1)	-2 (1)	1 (1)	-1 (1)
C(7)	14 (1)	10 (1)	14 (1)	-2 (1)	0 (1)	0 (1)
C(8)	15 (1)	14 (1)	26 (1)	5 (1)	2 (1)	18 (1)
C(9)	17 (1)	15 (1)	20 (1)	-6 (1)	2 (1)	9 (1)
C(10)	17 (1)	13 (1)	23 (1)	1 (1)	-15 (1)	5 (1)
C(11)	20 (1)	16 (1)	15 (1)	-6 (1)	-7 (1)	7 (1)
O(3)	19 (1)	9 (1)	20 (1)	4 (1)	3 (1)	-4 (1)
O(5)	25 (1)	6 (1)	22 (1)	-2 (1)	6 (1)	-6 (1)
C(12)	14 (1)	7 (1)	15 (1)	-1 (1)	0 (1)	1 (1)
C(13)	16 (1)	12 (1)	19 (1)	0 (1)	1 (1)	8 (1)
C(14)	15 (1)	11 (1)	27 (1)	-4 (1)	6 (1)	0 (1)
C(15)	22 (1)	7 (1)	23 (1)	0 (1)	0 (1)	-9 (1)
C(16)	23 (1)	12 (1)	15 (1)	3 (1)	-3 (1)	2 (1)
C(17)	15 (1)	10 (1)	17 (1)	-1 (1)	0 (1)	6 (1)

Table 7. Intramolecular C–H bond lengths (Å) of PTT

C(1)–H(11)	1.00	C(9)–H(92)	1.03	C(13)–H(131)	1.00
C(7)–H(71)	0.93	C(10)–H(101)	0.96	C(14)–H(141)	0.94
C(8)–H(81)	0.94	C(10)–H(102)	1.02	C(15)–H(151)	0.90
C(8)–H(82)	1.04	C(11)–H(111)	0.94	C(16)–H(161)	0.94
C(9)–H(91)	0.92	C(11)–H(112)	1.04	C(17)–H(171)	0.98

and largest $C(sp^3)$ – $C(sp^3)$ distance is 0.025(5) Å. Such variation is quite common and needs no comment. The C–H bond lengths, varying between 0.90 and 1.04 Å, are listed in Table 7. The sums of valency angles involving the nitrogen atoms N(2), N(6) and N(4) amount to 353.7, 350.3 and 359.9° respectively. The first two sums, significantly deviating from 360° by amounts of 6.3 and 9.7°, point to a pyramidal configuration of N(2) and N(6), while N(4) clearly shows a planar hybridization of its bonds.

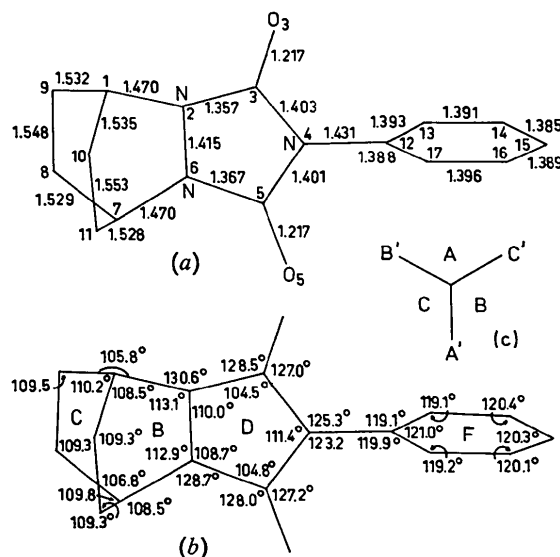


Fig. 2. (a) Bond lengths (Å), (b) valency angles, (c) indication of rings and least-squares planes.

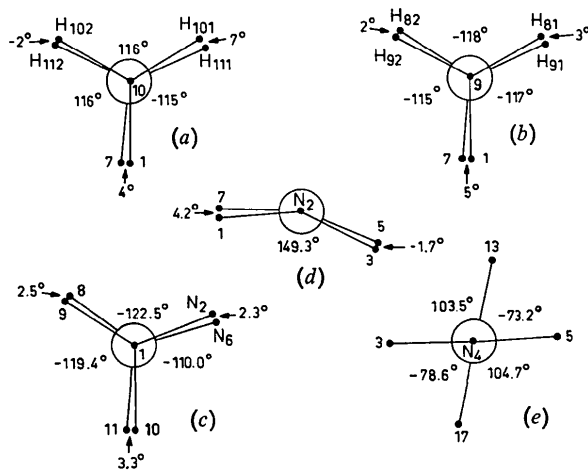


Fig. 3. Newman projections along (a) C(10)–C(11), (b) C(9)–C(8), (c) C(1)–C(7), (d) N(2)–N(6) and (e) N(4)–C(12).

Although the average *twist* about the axis C(1)···C(7) [2.7° , see Fig. 3(c)] is about the same as that reported by Cameron *et al.* (1968) for BOO (3°) and is also in reasonable agreement with an average *twist* motion of about 7° for BOD and unsubstituted bicyclo[2,2,2]octane, the bicyclo[2,2,2]-like moiety of PTT has no D_3 or C_3 symmetry. Neither is there found an indication for a *twisting* about the C(1)···C(7) axis, the largest principal axes of thermal ellipsoids of atoms N(2), N(6), C(8), C(9), C(10) and C(11) deviating by 10, 15, 69, 50, 48 and 62° respectively from the direction C(1)···C(7). We have also inspected the thermal motion of the whole molecule in terms of rigid-body movement (Schomaker & Trueblood, 1968). The agreement between calculated and observed matrix elements U_{ij} is, however, rather poor. Low-temperature diffraction data are not very well suited to inspection of thermal vibrations and we realize that the outcome of our thermal analysis is inconclusive with respect to a *twist* as well as rigid-body motion.

Except torsion angle H(102)–C(10)–C(11)–H(112) [see Fig. 3(a), (b) and (c)] which has an opposite sign, the distortions about C(10)–C(11), C(9)–C(8) and C(1)···C(7) are consistent. In view of the relatively large uncertainty in the positions of the hydrogen atoms the deviation of the exceptional dihedral angle is not disturbing.

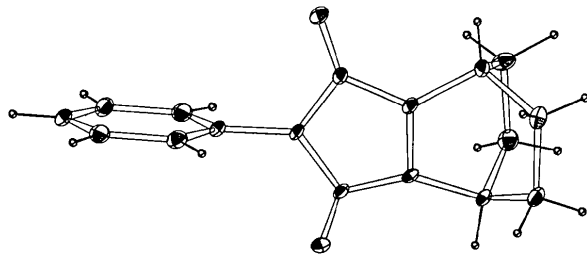


Fig. 4. ORTEP projection of PTT. The thermal ellipsoids of the non-hydrogen atoms are scaled to enclose 75% probability.

Table 8. Endocyclic torsion angles and additional dihedral angles between least-squares planes $A'(1,10,11,7)$, $B'(1,N_2,N_6,7)$, $C'(1,9,8,7)$, D' (five-membered ring) and F' (benzene ring)

Ring A		Ring C	
C(9)–C(1)–N(2)–N(6)	57.6°	C(9)–C(1)–C(10)–C(11)	–61.9°
C(1)–N(2)–N(6)–C(7)	4.2	C(1)–C(10)–C(11)–C(7)	4.2
N(2)–N(6)–C(7)–C(8)	–62.0	C(10)–C(11)–C(7)–C(8)	57.6
N(6)–C(7)–C(8)–C(9)	54.0	C(11)–C(7)–C(8)–C(9)	–63.5
C(7)–C(8)–C(9)–C(1)	5.5	C(7)–C(8)–C(9)–C(1)	5.5
C(8)–C(9)–C(1)–N(2)	–61.0	C(8)–C(9)–C(1)–C(10)	56.1
Ring B		Ring D	
C(10)–C(1)–N(2)–N(6)	–60.6	N(6)–N(2)–C(3)–N(4)	–3.4
C(1)–N(2)–N(6)–C(7)	4.2	N(2)–C(3)–N(4)–C(5)	7.4
N(2)–N(6)–C(7)–C(11)	56.3	C(3)–N(4)–C(5)–N(6)	–8.5
N(6)–C(7)–C(11)–C(10)	–58.9	N(4)–C(5)–N(6)–N(2)	6.1
C(7)–C(11)–C(10)–C(1)	4.2	C(5)–N(6)–N(2)–C(3)	–1.8
C(11)–C(10)–C(1)–N(2)	53.6		
Dihedral angles			
A'/B'	120.6°	C'/D'	27.8°
A'/C'	121.1	D'/F'	75.3
B'/C'	118.3		

Fig. 3(d) again demonstrates the pyramidal hybridization of N(2) and N(6). Finally Fig. 3(e) shows that the benzene ring is neither perpendicular nor parallel to the average plane of ring D . The overall molecular shape which lacks any symmetry is depicted in Fig. 4.

The endocyclic torsion angles of rings A , B , C and D together with a number of dihedral angles between the least-squares planes A' , B' , D' and F' are listed in Table 8. The conformations of rings A , B and C are, of course, slightly distorted tub-boats, while ring D is a nearly ideal envelope with N(4) as flap.

Packing

A part of the packing is illustrated (Fig. 5) in a projection along [100]. The molecules are inclined towards [010] by an angle of approximately 45° , resulting in a herringbone type of packing.

There are 66 contacts of the types $H\cdots H$, $O\cdots H$, $N\cdots H$ and $C\cdots H$ smaller than 3.0 \AA , and molecule (I) at position x, y, z is surrounded by 14 neighbouring molecules. The shortest contact (2.36 \AA , Table 9) is of the type $H\cdots H$; the shortest $O\cdots H$ interaction involving the screw-axis operation between molecules (I) and (VIII) amounts to 2.58 \AA . The tight packing and the large coordination number are in agreement with the fairly high density (1.36 g cm^{-3}).

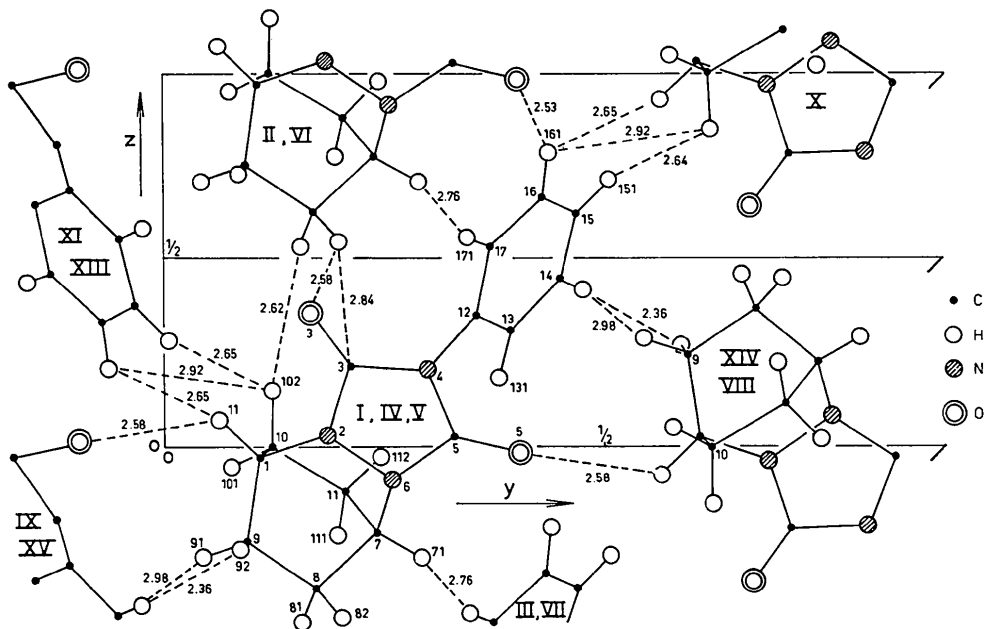
All calculations were performed on a 360/65 computer of the University of Leiden. The authors are indebted to Dr G.C. Verschoor for experimental assistance and to Mrs E.W.M. Rutten for computational aid.

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Table 9. Intermolecular contacts (\AA) between molecule (I) (x, y, z) and neighbouring molecules at positions indicated

Molecule	Position	Interaction	Number	Average	Minimum
(II)	$x, y, 1+z$	$\text{H}\cdots\text{H}$	4	2.69	2.40
(III)	$x, y, -1+z$	$\text{O}\cdots\text{H}$	2	2.56	2.53
		$\text{C}\cdots\text{H}$	1	3.00	3.00
		$\text{N}\cdots\text{H}$	1	2.95	2.95
(IV)	$1+x, y, z$	$\text{H}\cdots\text{H}$	4	2.68	2.52
(V)	$-1+x, y, z$	$\text{O}\cdots\text{H}$	2	2.86	2.73
		$\text{C}\cdots\text{H}$	1	2.99	2.99
(VI)	$1+x, y, 1+z$	$\text{O}\cdots\text{H}$	3	2.74	2.52
(VII)	$-1+x, y, -1+z$	$\text{C}\cdots\text{H}$	3	2.90	2.85
(VIII)	$-x, \frac{1}{2}+y, -z$	$\text{O}\cdots\text{H}$	1	2.58	2.58
(IX)	$-x, -\frac{1}{2}+y, -z$	$\text{C}\cdots\text{H}$	2	2.96	2.95
(X)	$-x, \frac{1}{2}+y, 1-z$	$\text{H}\cdots\text{H}$	5	2.77	2.64
(XI)	$-x, -\frac{1}{2}+y, 1-z$				
(XII)	$1-x, \frac{1}{2}+y, 1-z$	$\text{O}\cdots\text{H}$	1	2.73	2.73
(XIII)	$1-x, -\frac{1}{2}+y, 1-z$	$\text{H}\cdots\text{H}$	1	2.72	2.72
(XIV)	$1-x, \frac{1}{2}+y, z$	$\text{H}\cdots\text{H}$	2	2.67	2.36
(XV)	$1-x, -\frac{1}{2}+y, -z$				

Fig. 5. The packing of PTT molecules in a projection along $[100]$. The Roman numerals are explained in Table 8.

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