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Crystal Structures of Two Triazoles

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I. 3,5-diphenyl triazole, $C_{14}H_{11}N_3$, Mr = 221.26, monoclinic, *Cc*, $a = 26.982(6)$ Å, $b = 5.6806(14)$ Å, $c = 7.439(2)$ Å, $\beta = 105.54(2)°$, $V = 1098.6(5)$ Å^3, $Z = 4$, $D_m = 1.312\,\text{gm}\cdot\text{cm}^{-3}$, $D_c = 1.338\,\text{gm}\cdot\text{cm}^{-3}$, $\mu = 0.083\,\text{cm}^{-1}$, $F_{000} = 464$, (Mo$K\alpha$) = 0.71069 Å, final *R*1 and *wR*2 are 0.0335 and 0.0985 respectively. Hydrogen bonds of the type CH···N are observed in the crystal structure of the compound. The molecules are packed in layers when viewed along *b* and *c*, whereas down *a* it shows a herringbone type of arrangement.

II. 4-phenyl imidazole, $C_9H_8N_2$, Mr = 144.18, monoclinic, *Pa*, $a = 7.0291(13)$ Å, $b = 12.5142(8)$ Å, $c = 9.4476(12)$ Å, $\beta = 100.639(12)°$, $V = 816.76(18)$ Å^3, $Z = 4$, $D_m = 1.161\,\text{gm}\cdot\text{cm}^{-3}$, $D_c = 1.172\,\text{gm}\cdot\text{cm}^{-3}$, $\mu = 0.072\,\text{cm}^{-1}$, $F_{000} = 304$, (Mo$K\alpha$) = 0.71069 Å, final *R*1 and *wR*2 are 0.0548 and 0.1526 respectively. Hydrogen bonds of the type CH···N and NH···N are observed in this structure. The hydrogen bonded molecules are stacked in layers when viewed along *b*.

Keywords: Crystal structure; Nitrenium ion; triazole

INTRODUCTION

Nitrenium ions (ions pairs of nitrenium ions) which are isoelectronic with carbenes are known to be the reactive intermediates in many reactions [1–3]. Nitrenium ions are considered to be the ultimate carcinogens in the carcinogenesis initiated by aromatic amines [4–7]. This is suggested from the structures of the *in vitro* and *in vivo* formed adducts with the guanine

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residues of the DNA. Therefore the isolation and structural characterization of stable nitrenium ions are found to be very interesting in order to discuss the important reactions.

I. CRYSTAL STRUCTURE OF 3,5-DIPHENYL TRIAZOLE

Experimental

A colourless rectangular crystal with approximate dimensions of $0.35 \times 0.3 \times 0.25$ mm was mounted on a Rigaku AFC7S diffractometer equipped with a graphite monochromated Mo$K\alpha$ X-ray source ($\lambda = 0.71069$ Å). The data were collected at a temperature of 293° K using the $\omega - 2\theta$ scan technique. The unit cell parameters were obtained by using the method of short vectors followed by least squares refinement of 13 reflections. All reflections could be indexed with respect to a *C*-centered monoclinic cell. A total of 1057 intensities were collected in the range of $0 \leq 2\theta \leq 50°$ of which 965 were unique ($R_{int} = 0.00$). The structure was solved using SHELXS97 [8]. The structure was refined by full matrix least-squares using SHELX97 [9] using 965 unique reflections. The hydrogen atoms were generated at chemically acceptable positions and refined with isotropic thermal parameters assigned to them. 188 parameters were refined using 965 observed reflections with $I > 2\sigma(I)$ to $R1 = 0.0335$ and $wR2 = 0.0985$. In the final difference map $(\Delta/\sigma)_{max} = 0.132$, $(\delta\rho)_{min} = -0.183$, $(\delta\rho)_{max} = 0.034$ eÅ^{-3} and goodness of fit = 0.980.

Results and Discussions

The positional parameters and equivalent temperature factors for non-hydrogen atoms are given in Table I. Anisotropic thermal parameters (U_{ij}) are listed in Table II. Table III gives the bond distances and angles. Figure 1 represents the ORTEP [10] of the molecule at 50% probability. Figure 2 shows packings of molecules in the unit cell down *b* axis. Intramolecular hydrogen bonds [11] of the type CH···N are observed in the structure (shown as dashed lines in Fig. 1) It is observed that the phenyl ring defined by atoms C1 to C6 (Plane 1) is planar with atom C1 having a maximum deviation of 0.006 Å. But, in the plane defined by the atoms C12 to C17 (Plane 2), the maximum deviation observed is 0.01 Å for the atom C12.

TABLE I Atomic coordinates and U_{eq} (Å^2) for I

Atom	*x*	*y*	*z*	U_{eq}
C1	0.39247(13)	0.0308(6)	0.4884(6)	0.0370(8)
C2	0.34164(13)	0.0819(8)	0.3942(6)	0.0420(9)
C3	0.32884(15)	0.2941(10)	0.2983(5)	0.0501(10)
C4	0.36784(15)	0.4566(7)	0.2962(5)	0.0461(11)
C5	0.41905(11)	0.4071(8)	0.3924(5)	0.406(9)
C6	0.43075(15)	0.1960(7)	0.4877(5)	0.0367(9)
C7	0.48389(12)	0.1405(6)	0.5871(4)	0.0313(7)
N8	0.49964(12)	−0.0840(6)	0.6141(6)	0.0486(9)
N9	0.54842(11)	−0.0850(6)	0.7051(5)	0.0483(9)
N10	0.56436(13)	0.1441(6)	0.7378(4)	0.0406(8)
C11	0.52261(19)	0.2882(3)	0.6593(7)	0.0370(4)
C12	0.61651(13)	0.1987(7)	0.8364(4)	0.0331(7)
C13	0.62901(14)	0.4096(8)	0.9330(5)	0.0416(9)
C14	0.67903(16)	0.4558(8)	1.0272(6)	0.0472(11)
C15	0.71704(16)	0.2974(7)	1.0250(5)	0.0453(10)
C16	0.70580(16)	0.0915(9)	1.9276(6)	0.0490(10)
C17	0.65526(13)	0.0414(7)	0.8316(6)	0.0417(9)

TABLE II Anisotropic displacement parameters (Å^2) for I

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0368(19)	0.0303(17)	0.0405(19)	0.0027(14)	0.0047(15)	−0.0036(14)
C2	0.0265(16)	0.0458(19)	0.049(2)	−0.0008(17)	0.0018(16)	−0.0042(13)
C3	0.029(2)	0.072(3)	0.043(2)	0.003(2)	−0.0012(17)	0.0038(17)
C4	0.048(2)	0.043(2)	0.041(2)	−0.0028(17)	0.003(2)	0.0132(17)
C5	0.0299(18)	0.0439(19)	0.044(2)	−0.0005(18)	0.0033(16)	0.0006(15)
C6	0.040(2)	0.0310(18)	0.038(2)	−0.0041(18)	0.0075(16)	0.0019(16)
C7	0.0292(16)	0.0310(17)	0.0312(14)	−0.0028(14)	0.0035(12)	0.0058(13)
N8	0.0348(15)	0.0277(14)	0.076(2)	0.0020(16)	0.0019(15)	−0.0032(11)
N9	0.0401(16)	0.0342(16)	0.066(2)	0.0017(16)	0.0070(15)	−0.0018(13)
N10	0.0380(17)	0.0287(15)	0.0526(17)	−0.0016(14)	0.0079(14)	0.0045(13)
C11	0.0336(9)	0.0293(9)	0.0442(8)	0.005(2)	0.0039(6)	0.0042(19)
C12	0.0260(15)	0.0377(19)	0.0321(18)	0.0015(18)	0.0018(13)	0.0005(15)
C13	0.051(2)	0.0293(16)	0.044(2)	−0.0028(16)	0.0117(19)	0.0010(16)
C14	0.044(2)	0.047(2)	0.046(3)	−0.0086(18)	0.004(2)	−0.0069(17)
C15	0.042(2)	0.044(2)	0.044(2)	0.009(2)	0.003(2)	−0.0111(15)
C16	0.043(2)	0.056(2)	0.046(2)	0.0100(19)	0.0097(18)	0.0076(18)
C17	0.035(2)	0.044(2)	0.043(2)	0.0030(16)	0.0052(17)	−0.0017(15)

The five membered ring comprising the atoms C7, N8, N9, N10 and C11 (Plane 3) is also planar with a maximum deviation of 0.006 Å for C11. The dihedral angle between Planes 1 and 2 is 51.6(2)°, whereas both Planes 1 and 2 make an angle of 26.4(2)° with Plane 3 [12].

TABLE III Bond lengths (Å) and angles (°) for I

Atom 1	*Atom* 2	*Distance*	*Atom* 1	*Atom* 2	*Distance*
C1	C2	1.393(5)	C1	C6	1.397(5)
C2	C3	1.396(7)	C3	C4	1.403(6)
C4	C5	1.404(5)	C5	C6	1.386(6)
C6	C7	1.461(6)	C7	C11	1.336(5)
C7	N8	1.342(6)	N8	N9	1.309(2)
N9	N10	1.371(5)	N10	C11	1.389(5)
N10	C12	1.436(6)	C12	C17	1.383(5)
C12	C13	1.392(6)	C13	C14	1.369(5)
C14	C15	1.368(6)	C15	C16	1.366(7)
C16	C17	1.389(6)			
C1–H1···N8		2.866(5)	C17–H17···N9		2.874(4)

Atom 1	*Atom* 2	*Atom* 3	*Angle*	*Atom* 1	*Atom* 2	*Atom* 3	*Angle*
C2	C1	C6	119.2(3)	C1	C2	C3	120.8(3)
C2	C3	C4	119.3(4)	C3	C4	C5	120.1(4)
C6	C5	C4	119.5(3)	C5	C6	C1	121.1(4)
C5	C6	C7	120.3(3)	C1	C6	C7	118.7(3)
C11	C7	N8	110.8(3)	C11	C7	C6	128.6(3)
N8	C7	C6	120.6(4)	N9	N8	C7	108.4(4)
N8	N9	N10	108.2(4)	N9	N10	C11	107.7(3)
N9	N10	C12	120.9(3)	C11	N10	C12	131.4(3)
C7	C11	N10	104.9(4)	C17	C12	C13	119.3(4)
C17	C12	N10	119.4(3)	C13	C12	N10	121.3(3)
C14	C13	C12	119.9(4)	C15	C14	C13	120.5(4)
C16	C15	C14	120.6(4)	C15	C16	C17	119.7(4)
C12	C17	C16	120.0(4)				
C1	H1	N8	94(2)	C17	H17	N9	99(1)

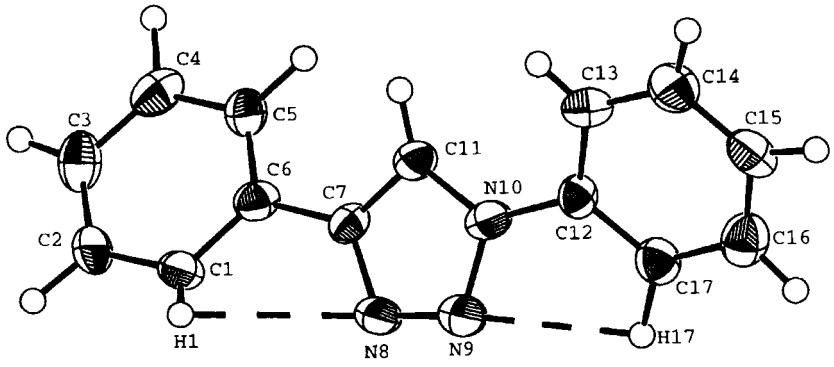

FIGURE 1 ORTEP of I at 50% probability.

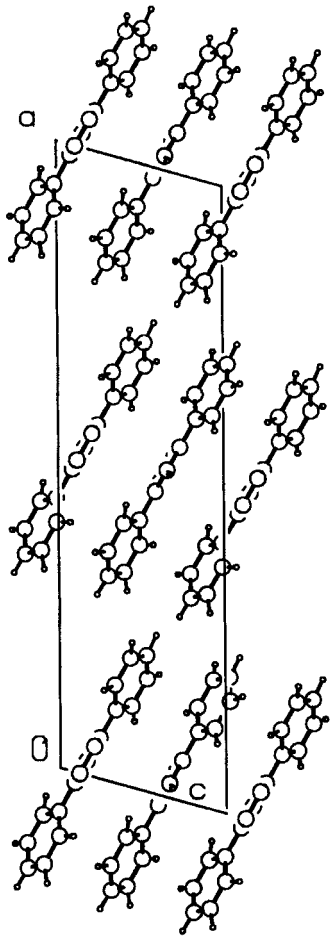

FIGURE 2 Packing of the molecules of I down *b*.

II. CRYSTAL STRUCTURE OF 4-PHENYL IMIDAZOLE

Experimental

A colourless prismatic crystal with approximate dimensions of $0.5 \times 0.4 \times 0.2$ mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC7S diffractometer equipped with a graphite monochromated Mo$K\alpha$ X-ray source ($\lambda = 0.71069$ Å). The data were collected at a temperature of 293° K using the $\omega - 2\theta$ scan technique. The unit cell parameters were obtained by using the method of short vectors followed by least squares refinement of 18 reflections. All reflections could

be indexed with respect to a monoclinic cell. A total of 1650 intensities were collected in the range of $0 \leq 2\theta \leq 50°$ of which 1526 were unique ($R_{int} = 0.00$). The structure was solved using SHELXS97 [8]. The structure was refined by full matrix least-squares using SHELXL97 [9] using 1526 unique reflections. The hydrogen atoms were generated at chemically acceptable positions and refined with isotropic thermal parameters assigned to them. 248 parameters were refined using 1526 observed reflections with $I > 2\sigma(I)$ to $R1 = 0.0548$ and $wR2 = 0.1526$. In the final difference map $(\Delta/\sigma)_{max} = 0.277$, $(\delta\rho)_{min} = -0.258$, $(\delta\rho)_{max} = 0.075\,e\text{Å}^{-3}$ and goodness of fit = 1.628.

Results and Discussions

The positional parameters and equivalent temperature factors for non-hydrogen atoms are given in Table IV. Anisotropic parameters (U_{ij}) are listed in Table V. Table VI gives the bond distances and angles. Figure 3 represents the ORTEP [10] of the molecule at 50% probability. Figure 4 shows packing of molecules in the unit cell down *a* axis. The bond distances and angles are in good agreement when compared with other carbenes and triazoles [13]. Intermolecular and intramolecular hydrogen bonds of the type

TABLE IV Atomic coordinates and U_{eq} (Å^2) for II

Atom	*x*	*y*	*z*	U_{eq}
C1	0.2136(9)	0.8387(5)	0.3347(6)	0.0768(15)
C2	0.2499(11)	0.9196(5)	0.2483(8)	0.095(2)
C3	0.1408(10)	0.9292(5)	0.1050(6)	0.0781(15)
C4	0.0009(9)	0.8585(4)	0.0605(5)	0.0709(14)
C5	−0.0368(8)	0.7732(4)	0.1501(5)	0.0631(12)
C6	0.0708(6)	0.7614(4)	0.2861(4)	0.0546(10)
C7	0.0348(6)	0.6721(3)	0.3793(4)	0.0530(10)
N8	−0.0576(6)	0.5818(3)	0.3235(4)	0.0632(10)
C9	−0.0614(8)	0.5209(5)	0.4357(6)	0.0652(13)
N10	0.0218(6)	0.5635(3)	0.5570(4)	0.0601(10)
C11	0.0852(8)	0.6621(4)	0.5235(5)	0.0607(11)
C1′	0.1393(8)	0.2791(4)	−0.3090(6)	0.0665(12)
C2′	0.1644(10)	0.1897(6)	−0.3881(7)	0.0817(17)
C3′	0.0980(12)	0.0930(6)	−0.3576(8)	0.094(2)
C4′	−0.0013(12)	0.0858(5)	−0.2407(8)	0.099(3)
C5′	−0.0323(9)	0.1759(5)	−0.1645(7)	0.0801(16)
C6′	0.0384(6)	0.2757(4)	−0.1966(5)	0.0603(12)
C7′	0.0084(6)	0.3721(4)	−0.1148(5)	0.0568(11)
N8′	0.0210(6)	0.4714(3)	−0.1696(4)	0.0566(9)
C9′	−0.0063(8)	0.5390(4)	−0.0675(5)	0.0644(12)
N10′	−0.0354(6)	0.4868(4)	0.0516(4)	0.0656(11)
C11′	−0.0282(8)	0.3788(5)	0.0215(5)	0.0716(14)

TABLE V Anisotropic displacement parameters (Å^2) for II

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.076(3)	0.083(3)	0.065(3)	0.012(3)	−0.004(3)	−0.012(3)
C2	0.092(4)	0.087(4)	0.100(5)	0.014(4)	0.006(4)	−0.026(3)
C3	0.092(4)	0.072(3)	0.071(3)	0.016(3)	0.017(3)	−0.002(3)
C4	0.084(3)	0.075(3)	0.055(3)	0.018(2)	0.015(2)	0.005(3)
C5	0.073(3)	0.063(3)	0.052(2)	0.007(2)	0.009(2)	0.004(2)
C6	0.056(2)	0.063(2)	0.047(2)	0.0062(17)	0.0141(8)	0.0031(18)
C7	0.052(2)	0.061(2)	0.046(2)	0.0006(17)	0.0093(18)	0.0048(18)
N8	0.072(2)	0.077(2)	0.0428(19)	0.0070(18)	0.0154(17)	−0.014(2)
C9	0.071(3)	0.073(2)	0.054(2)	0.006(2)	0.018(2)	−0.013(2)
N10	0.074(2)	0.070(2)	0.0397(19)	0.0099(172)	0.0188(17)	0.0083(18)
C11	0.071(3)	0.071(3)	0.041(2)	0.002(2)	0.012(2)	0.010(2)
C1′	0.065(3)	0.066(3)	0.069(3)	0.002(2)	0.014(2)	−0.004(2)
C2′	0.0083(4)	0.083(4)	0.077(4)	−0.007(3)	0.008(3)	0.010(3)
C3′	0.092(4)	0.082(4)	0.094(5)	−0.005(3)	−0.018(4)	0.009(3)
C4′	0.108(5)	0.066(3)	0.107(5)	0.016(3)	−0.019(5)	−0.015(3)
C5′	0.087(4)	0.079(4)	0.070(3)	0.016(3)	0.002(3)	−0.016(3)
C6′	0.050(2)	0.075(3)	0.052(2)	0.021(2)	−0.0008(18)	−0.002(2)
C7′	0.048(2)	0.075(3)	0.047(2)	0.014(2)	0.0079(17)	−0.0019(19)
N8′	0.060(2)	0.073(2)	0.0374(17)	0.0070(17)	0.0104(15)	−0.0005(18)
C9′	0.062(2)	0.091(3)	0.042(2)	0.006(2)	0.0127(19)	0.004(2)
N10′	0.062(2)	0.091(3)	0.044(2)	0.000(2)	0.0110(17)	0.007(2)
C11′	0.061(3)	0.102(4)	0.053(3)	0.024(3)	0.013(2)	−0.002(3)

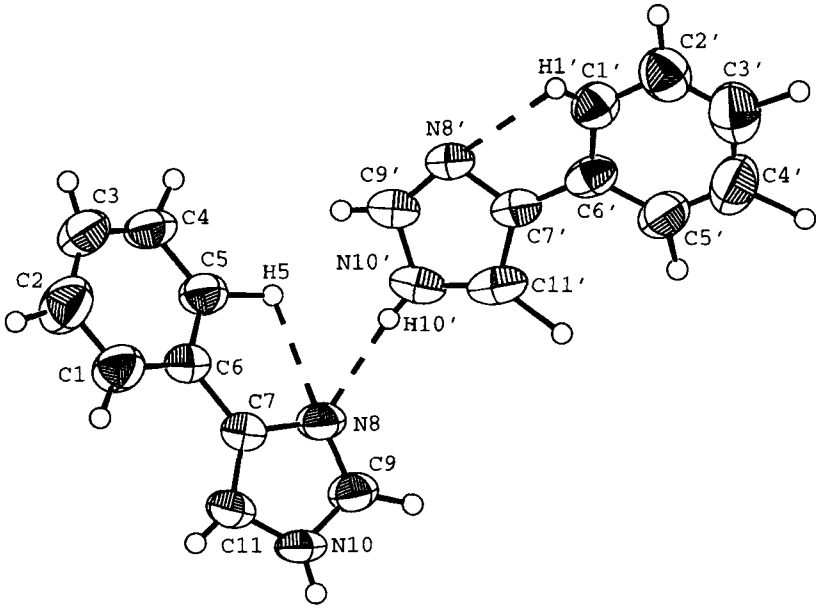

FIGURE 3 ORTEP of II at 50% probability.

TABLE VI Bond lengths (Å) and angles (°) for II

Atom 1	*Atom* 2	*Distance*	*Atom* 1	*Atom* 2	*Distance*
C1	C2	1.354(8)	C1	C6	1.409(7)
C2	C3	1.432(9)	C3	C4	1.332(9)
C4	C5	1.418(7)	C5	C6	1.373(6)
C6	C7	1.473(6)	C7	C11	1.348(6)
C7	N8	1.361(6)	N8	C9	1.310(7)
C9	N10	1.302(7)	N10	C11	1.369(7)
C1′	C2′	1.374(9)	C1′	C6′	1.383(7)
C2′	C3′	1.347(11)	C3′	C4′	1.414(12)
C4′	C5′	1.376(10)	C5′	C6′	1.398(7)
C6′	C7′	1.468(8)	C7′	N8′	1.355(6)
C7′	C11′	1.362(7)	N8′	C9′	1.323(6)
C9′	N10′	1.348(6)	N10′	C11′	1.385(9)
C5–H5···N8		2.928(6)	N10–H10···N8′		2.829(5)
C1′–H1′···N8		2.936(7)	N10′–H10′···N8		2.861(6)

Atom 1	*Atom* 2	*Atom* 3	*Angle*	*Atom* 1	*Atom* 2	*Atom* 3	
C2	C1	C6	121.3(5)	C1	C2	C3	120.3(6)
C4	C3	C2	118.6(5)	C3	C4	C5	121.3(5)
C6	C5	C4	120.6(5)	C5	C6	C1	117.9(4)
C5	C6	C7	121.1(4)	C1	C6	C7	121.1(4)
C11	C7	N8	109.4(4)	C11	C7	C6	129.3(4)
N8	C7	C6	121.3(4)	C9	N8	C7	104.4(4)
N10	C9	N8	113.6(5)	C9	N10	C11	106.2(4)
C7	C11	N10	106.4(5)	C2′	C1′	C6′	121.9(6)
C3′	C2′	C1′	122.1(7)	C2′	C3′	C4′	117.6(7)
C5′	C4′	C3′	120.3(6)	C4′	C5′	C6′	121.5(6)
C1′	C6′	C5′	116.5(6)	C1′	C6′	C7′	121.6(5)
C5′	C6′	C7′	121.9(5)	N8′	C7′	C11′	110.0(5)
N8′	C7′	C6′	121.7(4)	C11′	C7′	C6′	128.2(5)
C9′	N8′	C7′	106.2(4)	N8′	C9′	N10′	111.3(5)
C9′	N10′	C11′	106.6(4)	C7′	C11′	N10′	105.9(5)
C5′	H5	N8	99(2)	N10	H10	N8′	160(5)
C1′	H1′	N8′	87(4)	N10′	H10′	N8	137(7)

CH···N and NH···N are observed in the structure (shown as dashed lines in Fig. 3). All the geometry calculations were done using PARST97 [11]. The bond lengths and angles of these hydrogen bonds are comparable with those found in 3,5,6-trichloropyridine-2-ol-piperazine complex [14], 3,5,6-trichloropyridine-2-ol-piperdine complex [15] (both intermolecular) and 10-(N-Morpholinoacetyl)-2-trifluoromethyl phenoxazine [16] (intramolecular). Planes 1 and 2 comprising atoms C1 to C6 and C1′ to C6′, respectively, are very nearly planar with maximum deviations of 0.017 and 0.014Å respectively. Similarly, planes 3 and 4 of atoms C7 to C11 and C7′ to C11′, respectively, are highly planar with maximum deviations of 0.004 and

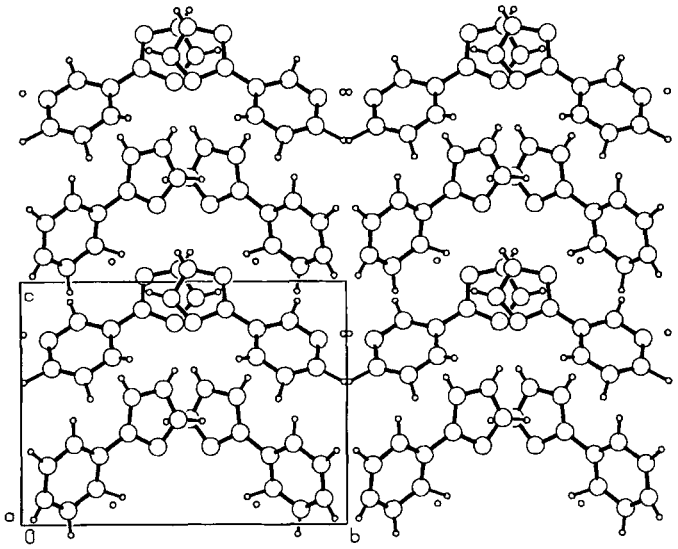

FIGURE 4 Packing of the molecules of II down *a*.

0.005 Å respectively. In both the molecules the phenyl ring and the five membered ring make an angle of 22° with each other [12].

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