

THE CRYSTAL AND MOLECULAR STRUCTURE OF PHENYL HYDRAZINE ($C_6H_5.NH.NH_2$)

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PHENYL hydrazine ($C_6H_5.NH.NH_2$) (Fig. 1) is a liquid at room temperature solidifying at $19^\circ C$. The unit cell dimensions and space group of this substance were determined by S. Ramaseshan at the Polytechnic Institute of Brooklyn, U.S.A., from Weissenberg and precession X-ray diffraction photographs of single crystals grown by the Fankuchen (1949) method. In this laboratory the crystal was again grown *in situ* on a Weissenberg goniometer by the method developed by Singh and Ramaseshan (1964) and the three-dimensional data were collected using $Cu K_\alpha$ radiation.

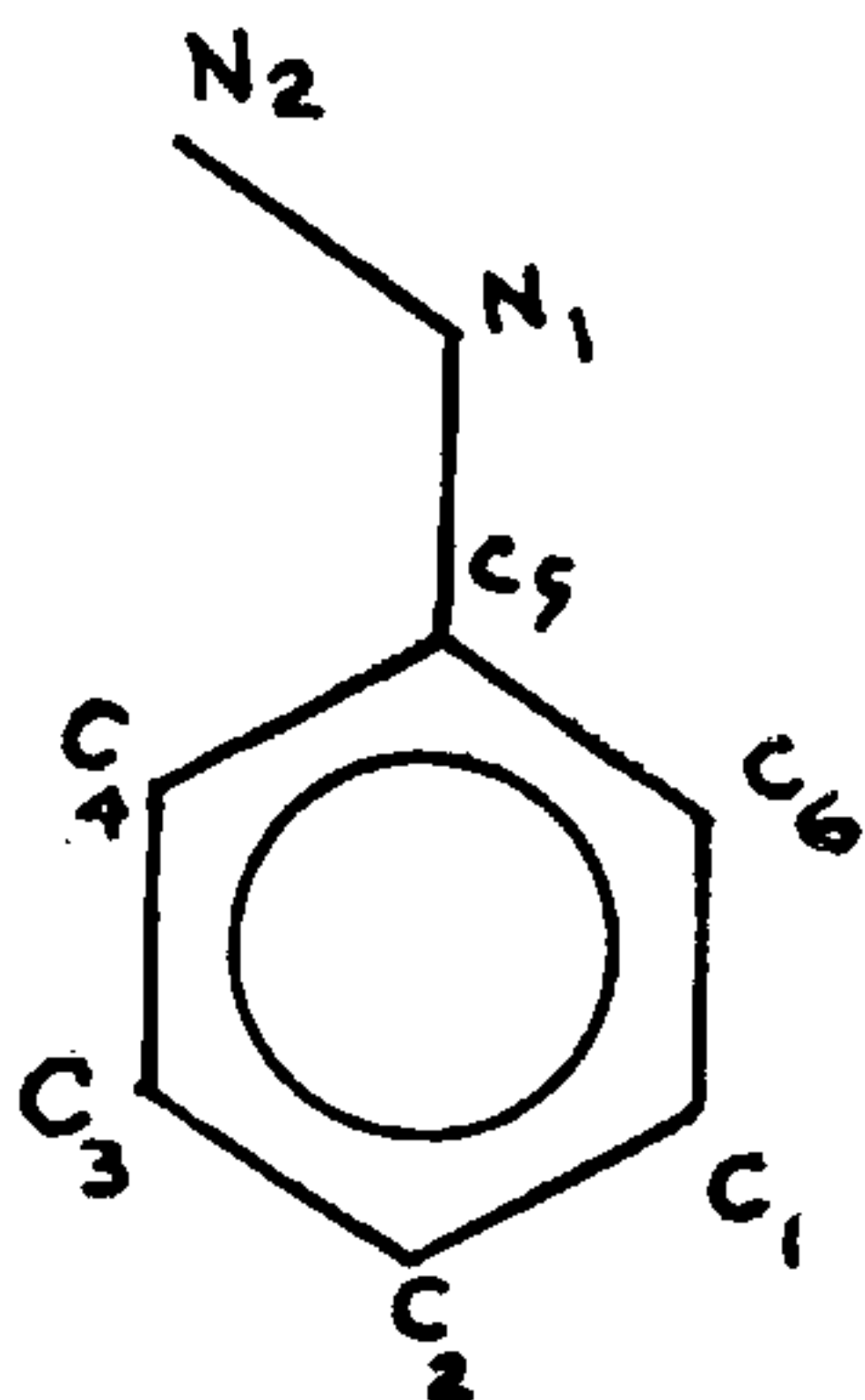


FIG. 1

The X-ray diffraction photographs gave the following data:

Space group $P 2_1/c$.

unit cell dimensions:

$$a = 9.59 \pm 0.02 \text{ \AA}$$

$$b = 5.91 \pm 0.02 \text{ \AA}$$

$$c = 12.32 \pm 0.02 \text{ \AA}$$

$$\beta = 118^\circ \pm 0.5^\circ$$

density (liquid) = $1.0978 \text{ gm. cm.}^{-3}$

density (crystal) calculated = $1.1340 \text{ gm. cm.}^{-3}$

number of molecules per unit cell = 4.

The structure analysis was taken up with the $h0l$, $h1l$, $h2l$ and $h3l$ Weissenberg equiinclination data. The structure was solved first in the (010) projection. A (010) Patterson projection gave only the projection co-ordinates of the

ring centre without any other information about the orientation of the molecule. The Patterson map was modified to remove the origin peak and the ring-to-ring vector peak, and \hat{f} sharpened and this enabled the orientation of the molecule to be fixed. The structure in projection was refined by trial and error and difference synthesis till the R factor fell to 0.25.

Lacking data in another projection, the approximate values of the third co-ordinates (the y co-ordinates) were obtained by packing considerations, assuming the standard intermolecular contact distances. These were adjusted to fit the $h1l$ data and later the hkl data ($k = 0, 1, 2$ and 3) till the overall R factor came to 0.24. The co-ordinates and temperature factors were then refined by four cycles of least squares refinement on the CDC 3600 computer at the Tata Institute of Fundamental Research, Bombay. The final R factor is 0.16. The atomic co-ordinates with the individual isotropic temperature factors are given in Table I, the interatomic bond lengths and the bond angles in Table II and the intermolecular contacts in Table III.

TABLE I

Fractional atomic co-ordinates and isotropic temperature factors

	x	y	z	(\AA^2)
C_1	0.136	0.795	0.321	3.72
C_2	0.122	0.719	0.428	4.47
C_3	0.192	0.517	0.484	3.35
C_4	0.284	0.374	0.443	2.93
C_5	0.299	0.451	0.335	2.84
C_6	0.227	0.657	0.278	2.90
N_1	0.376	0.314	0.285	3.21
N_2	0.481	0.127	0.355	3.34

TABLE II

Interatomic distances and angles

Lengths	Values in \AA	Angles	Values in degrees
C_1-C_2	1.45 ± 0.04	$C_1-C_2-C_3$	120 ± 1
C_2-C_3	1.38 ± 0.03	$C_2-C_3-C_4$	121 ± 2
C_3-C_4	1.45 ± 0.04	$C_3-C_4-C_5$	119 ± 1
C_4-C_5	1.45 ± 0.04	$C_4-C_5-C_6$	118 ± 2
C_5-C_6	1.40 ± 0.03	$C_5-C_6-C_1$	120 ± 1
C_6-C_1	1.46 ± 0.04	$C_4-C_5-N_1$	121 ± 1
C_5-N_1	1.42 ± 0.03	$C_6-C_5-N_1$	120 ± 1
N_1-N_2	1.46 ± 0.04	$C_6-N_1-N_2$	121 ± 2

TABLE III
Shortest intermolecular contacts (Å)

I	molecule at	x	y	z
II	"	\bar{x}	$1/2+y$	\bar{z}
III	"	$1-x$	$1/2+y$	\bar{z}
IV	"	$1-x$	$1-y$	$1-z$
N ₁ (I) - N ₂ (III)			3.17	
C ₃ (I) - N ₂ (IV)			3.56	
N ₂ (I) - C ₄ (IV)			3.67	
C ₁ (I) - C ₆ (II)			3.70	
C ₁ (I) - C ₂ (II)			3.71	
N ₂ (I) - N ₂ (III)			3.79	

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1. Kaufmann, H. S. and Fankuchen, I., *Rev. Sc. Instrum.*, 1949, **20**, 733.
2. Singh, A. K. and Ramaseshan, S., *Proc. Ind. Acad. Sci.*, 1964, **60 A**, 20.

GAMMA-GAMMA ANGULAR CORRELATION IN W^{182} *

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THE level structure of tungsten W^{182} has been investigated by a number of workers.^{1,2} The spin of various levels and multipolarity of various gamma-rays have been determined, both by internal conversion method³⁻⁵ and by gamma-gamma angular correlation measurements.⁶⁻⁸ Due to the complexity of the level structure of W^{182} , shown in Fig. 1, it is found that some of

ray to be pure E_2 and 222 Kev gamma-ray to be pure E_1 in character, as determined by Murray *et al.*³ from the internal conversion measurements. Whereas more recently Korkman and Backlin⁵ have reported, from internal conversion measurements, that 1231 Kev gamma-ray is an admixture of E_2 and M_1 , dipole component being of the order of $29 \pm 8\%$. In view of this discrepancy, it was thought worthwhile to study the angular correlation of 68-1222, 264-68 and 222-1231 Kev cascades in order to establish the spin 1289, 1331 and 1554 Kev levels and thereby determine the multipolarity of 1231 Kev gamma-ray from angular correlation measurements.

Measurements and Results.—The measurements were carried out with a conventional fast-slow coincidence arrangement with an effective resolving time of about 70 n-secs and two $3'' \times 3''$ NaI(Tl) crystals. The source was used in liquid form in order to minimise any electric quadrupole interaction. The coincidence data was taken with a 256 channel analyser to precisely assess the Compton contribution in the low energy region because of the high energy gamma-ray cascades. The gate was set at about 30 Kev higher than the composite photopeak of 1222-1231 Kev gamma-rays, with a window of 70 KeV, in order to avoid any interference because of 1122 Kev gamma-ray. In this way 68-1222 and 222-1231 Kev cascades could be run simultaneously. 264-68 Kev cascade was run separately with single channel analysers. The results of these measurements are as follows:

68-1222 Kev Cascade.—In this correlation, at each angle, the Compton component of 100, 151

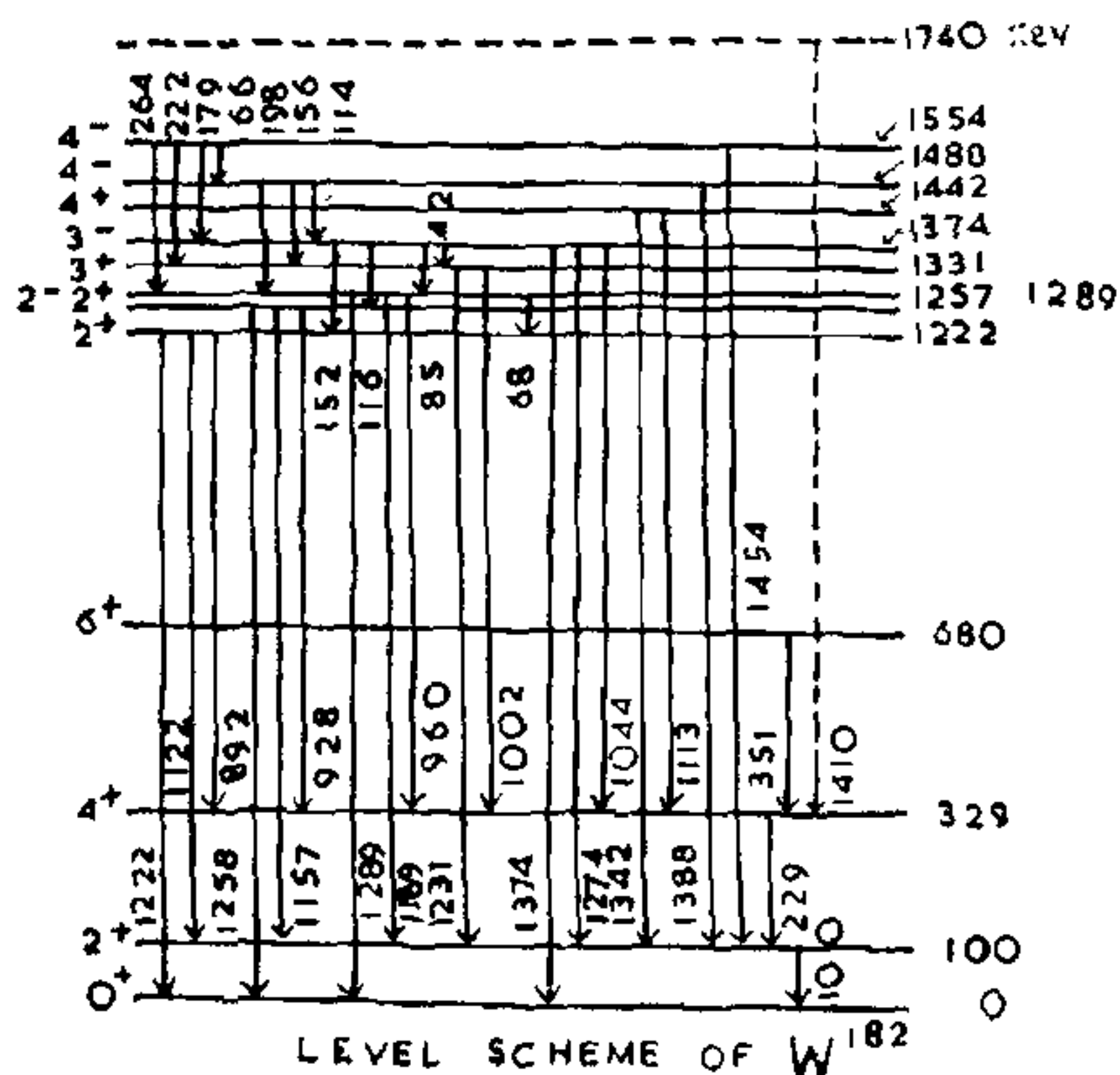


FIG. 1

the results obtained from these measurements are inconsistent. In particular Hickman and Wieldenbeck⁶ has assigned a spin 4 to 1554 Kev level on the basis of the angular correlation measurements of 222-1231 Kev cascade. In doing so, they have assumed 1231 Kev gamma-

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