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The crystal structure of 2,2′-pyridil, a refinement. By T. ASHIDA, *Institute for Protein Research, Osaka University, Osaka, Japan* and S. HIROKAWA, *Department of Chemistry, Defense Academy, Yokosuka, Japan*

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The crystal structure of 2,2′-pyridil has been further refined to an R index of 0·067. Some structural parameters are markedly improved.

The crystal structure of 2,2′-pyridil (1,2-di-2-pyridylethanedione) (Hirokawa & Ashida, 1961) has been further refined. The cell dimensions are $a=6{\cdot}41$, $b=13{\cdot}03$, $c=12{\cdot}79$ Å and $\beta=99{\cdot}5°$, the space group being $P2_1/n$. The block-diagonal matrix least-squares method was applied to all atoms using 1301 observed reflexions. The initial parameters were those listed in the previous report, and the hydrogen atoms were first located geometrically. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962); the weighting scheme was: $w=0{\cdot}2$ for $|F|\leq 1{\cdot}5$, $w=1{\cdot}0$ for $1{\cdot}5<|F|\leq 10{\cdot}0$ and $w=(10{\cdot}0/|F|)^2$ for $|F|>10{\cdot}0$; the NEAC 2200-500 of the Computing Centre of Osaka University was used, the program being *HBLS* IV (Ashida, 1967).

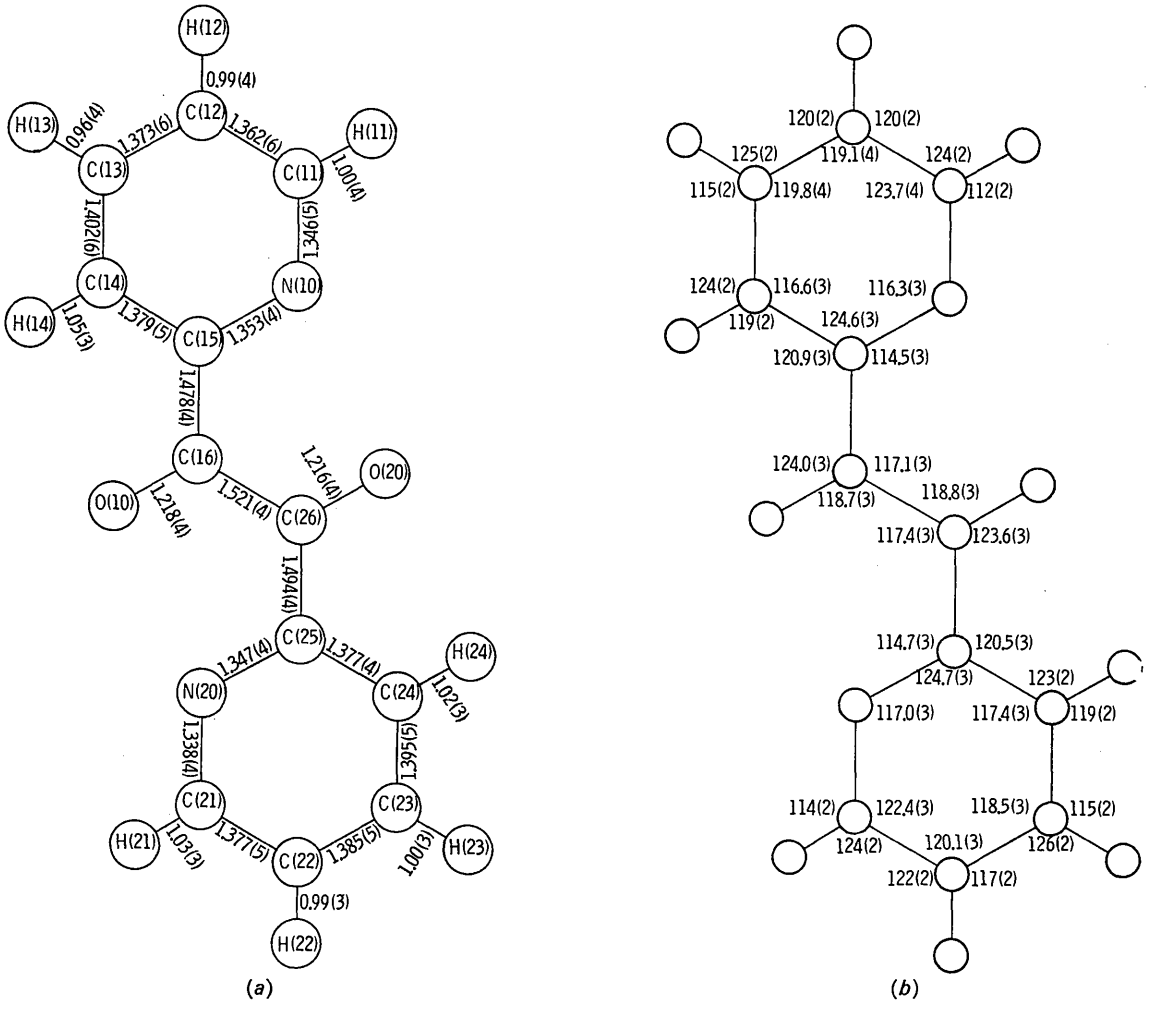


Fig. 1. Bond distances (*a*) and angles (*b*). The standard deviations are in parenthesis.

The R index decreased from 0·16 to 0·067. At this stage the index was calculated to be 0·10 if the hydrogen atoms were not included. The final parameters are listed in Table 1, the mean standard deviation of the positional parameters of the non-hydrogen atoms being 0·003 Å. The observed and calculated structure factors are listed in Table 2.

The bond distances and angles are shown in Fig. 1. The lengths of the bonds C(12)–C(13) and C(25)–N(20) are markedly improved from 1·343 and 1·321 to 1·371 and 1·347 Å respectively. The angle C(25)–N(20)–C(21) is now 117·1°, which appears to be more reasonable than 120·5°. Thus the shapes and sizes of the pyridil groups are much improved, and the conformations of these two groups and that of 1-phenyl-2-(2-pyridyl)ethanedione (Ashida, Hirokawa & Okaya, 1966) resemble one another more closely.

The equations of the best planes for the pyridyl groups

Table 1. *Atomic coordinates and thermal parameters*

(*a*) Final atomic coordinates and their standard deviations in units of the last decimal position

	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$
N(10)	0·5925	5	0·1329	2	0·2491	2
C(11)	0·5191	7	0·0652	3	0·1724	3
C(12)	0·3125	7	0·0591	3	0·1258	3
C(13)	0·1688	7	0·1257	4	0·1572	3
C(14)	0·2356	6	0·1974	3	0·2374	3
C(15)	0·4477	5	0·1967	2	0·2801	2
C(16)	0·5339	5	0·2681	2	0·3663	2
O(10)	0·4285	4	0·3320	2	0·4037	2
N(20)	0·7051	4	0·1270	2	0·5176	2
C(21)	0·7708	6	0·0566	3	0·5919	3
C(22)	0·9800	6	0·0467	3	0·6375	3
C(23)	1·1310	5	0·1114	3	0·6073	3
C(24)	1·0658	5	0·1851	2	0·5297	2
C(25)	0·8535	5	0·1891	2	0·4885	2
C(26)	0·7716	5	0·2654	2	0·4045	2
O(20)	0·8826	4	0·3282	2	0·3697	2

(*b*) Thermal parameters and their standard deviations ($\times 10^{-4}$)
The thermal parameters are of the form: $\exp\{-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+\beta_{12}hk+\beta_{13}hl+\beta_{23}kl)\}$.

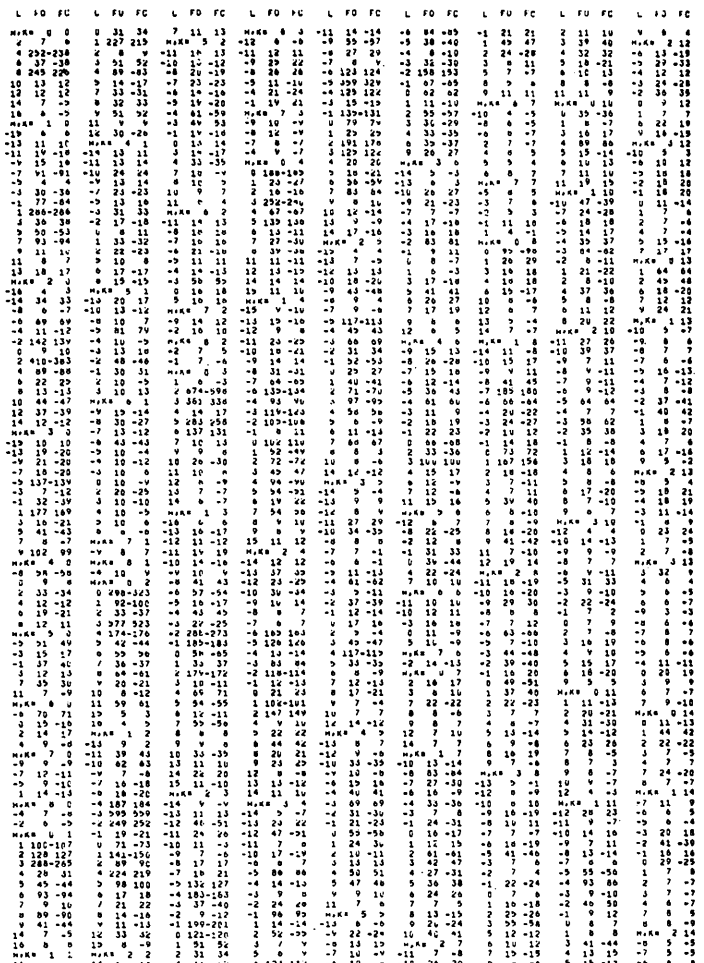
	β_{11}	σ	β_{22}	σ	β_{33}	σ	β_{12}	σ	β_{13}	σ	β_{23}	σ
N(10)	523	11	62	2	92	2	27	7	61	8	2	3
C(11)	681	16	70	2	105	3	−21	11	84	11	−4	4
C(12)	741	18	92	3	96	3	−183	12	42	12	0	5
C(13)	558	15	137	4	93	3	−180	13	5	10	47	5
C(14)	464	12	101	3	85	2	−29	10	58	9	36	4
C(15)	407	10	57	2	79	2	3	7	55	8	32	3
C(16)	423	11	56	2	83	2	40	8	88	8	21	3
O(10)	497	9	92	2	114	2	109	7	63	7	−26	3
N(20)	419	9	65	2	87	2	1	7	100	7	9	3
C(21)	508	13	72	2	97	3	16	9	122	9	32	4
C(22)	563	14	81	2	83	2	63	10	78	9	29	4
C(23)	410	11	100	3	85	2	75	10	16	8	−6	4
C(24)	372	10	77	2	86	2	−9	8	73	8	−21	4
C(25)	384	10	54	2	72	2	10	7	85	7	−9	3
C(26)	402	10	55	2	86	2	−4	7	102	8	1	3
O(20)	483	8	81	2	128	2	−37	6	99	7	52	3

(*c*) Final hydrogen atom parameters

	x	y	z	B
H(11)	0·636	0·026	0·149	5·3
H(12)	0·268	0·009	0·069	7·6
H(13)	0·020	0·127	0·128	5·7
H(14)	0·135	0·250	0·267	5·0
H(21)	0·650	0·015	0·615	4·4
H(22)	1·029	−0·006	0·692	4·9
H(23)	1·286	0·111	0·636	4·1
H(24)	1·177	0·229	0·503	3·7

$\langle\sigma(x)\rangle \simeq \langle\sigma(y)\rangle \simeq \langle\sigma(z)\rangle = 0{\cdot}03$ Å
$\langle\sigma(B)\rangle = 1{\cdot}0$ Å^2

Table 2. *Observed and calculated structure factors* (×5)



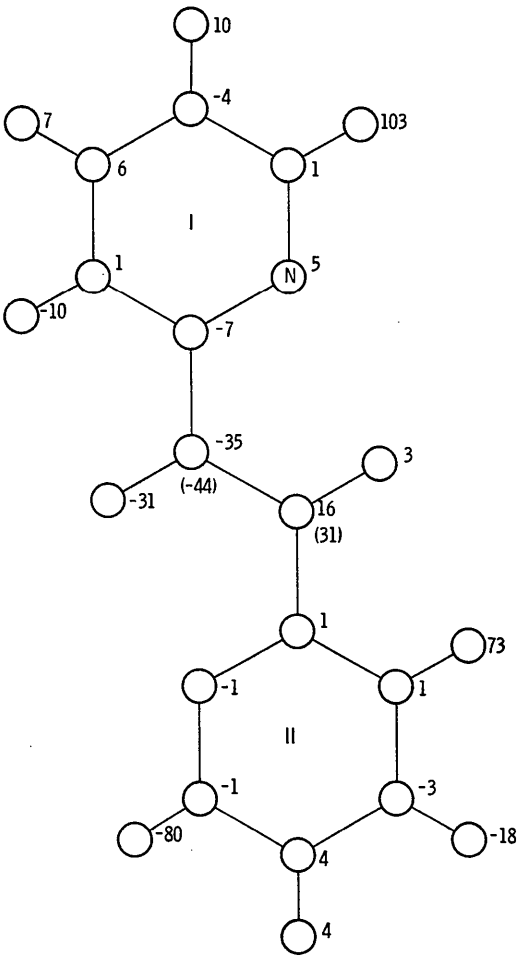


Fig. 2. The displacements of atoms from the best planes in Å (multiplied by 10^3). The shift of C(16) from plane II is −0·044 Å and that of C(26) from plane I is 0·031 Å.

are:

$$X+2\cdot2598Y-2\cdot3631Z+\ 0\cdot2566=0$$

for plane I of N(10), C(11), C(12), C(13), C(14) and C(15), and

$$X-2\cdot4685Y-2\cdot6987Z+18\cdot2762=0$$

for plane II of N(20), C(21), C(22), C(23), C(24) and C(25), where $X=ax+cz\cos\beta$, $Y=by$ and $Z=cz\sin\beta$. The displacements of atoms from the planes are shown in Fig. 2. The dihedral angle between the planes is 82·0°.

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