The Crystal Structure of Nicotinic Acid

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The crystal structure of nicotinic acid has been determined by two- and three-dimensional X-ray methods. The bond lengths, which have a probable error of 0·014 Å, are: pyridine ring C–C = 1·38 Å, C–N = 1·34 Å, carboxyl group C_2–C_6 = 1·48 Å, C_6–O_1 = 1·34 Å, C_6–O_2 = 1·18 Å. The molecules are linked in chains parallel to the b axis by hydrogen bonds of length 2·66 Å between O_1 and the nitrogen atom of the next molecule.

Introduction

Nicotinic acid is 3-pyridine carboxylic acid. Its optical properties, unit cell and space group have been described in a previous communication (Wright & King, 1950). This paper is concerned with the determination, by three-dimensional X-ray methods, of the bond lengths in the molecule and of the nature of the intermolecular linkages.

Experimental

The cell dimensions reported in the previous paper are

$$a=7·175\pm0·002, \quad b=11·682\pm0·002, \quad c=7·220\pm0·002 \text{ Å},$$
$$\beta=113° 23'\pm3'.$$

Space group $P2_1/c$.

Multiple-film equi-inclination Weissenberg photographs were taken about the $a(h = 0 \to 3)$, $b(k = 0 \to 5)$, $c(l = 0 \to 3)$ and [101] ($l = \bar{h} \to 3-h$) axes using copper radiation. All but 12 of the 1256 reflexions within the limiting sphere for copper $K\alpha$ radiation could be recorded in this way, but 305 reflexions were too weak to be observed. Intensities were estimated visually by comparison with standard spots. A correction for variation in spot shape was made by the method given by Broomhead (1948) to allow for the

non-uniform cross-section of the crystals. As the largest dimension of any of the crystals used was less than 0·5 mm. no correction was made for absorption. Lorentz and polarization corrections were made by the method of Goldschmidt & Pitt (1948). It was reported in the preliminary paper that all crystals of nicotinic acid are twinned across (100). The observed $hk0$ intensities, which are the sums of the contributions from each twin, were corrected by means of a twinning ratio obtained from the b-axis photographs.

A few of the strongest reflexions were found during the course of the analysis to have calculated structure factors much greater than the observed values. This may be due to extinction for which no allowance was made, or to the fact that these strong reflexions could only be correlated with very weak spots on the final films of the multiple-film series. For the final correlation of these strong reflexions a larger number of film-to-film ratios was used than was needed for the other reflexions so that the initial errors may have been much increased.

Determination of the structure

The c-axis projection

The optical properties showed that the crystals are negatively birefringent and indicated that the pyridine
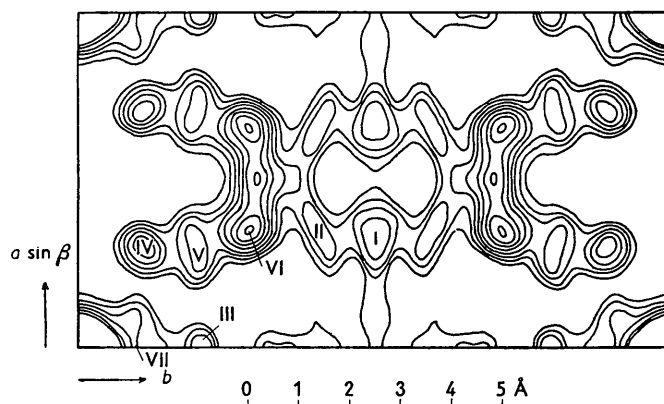

Fig. 1. The c-axis Patterson projection.

ring makes an angle of about 76° with the c axis. The c-axis projection was therefore expected to give the best picture of the structure in spite of the overlap inherent in the space group $P2_1/c$.

A c-axis Patterson projection (Fig. 1) was computed. It has been shown (Patterson, 1949) that a Patterson synthesis may be used for locating centrosymmetrical groups such as benzene rings. In nicotinic acid, therefore, two pyridine rings which occur in a centrosymmetric relation should give rise to a heavy peak in the Patterson projection at a point corresponding to the distance between their centres. If it is assumed that the coordinates of the centre of the pyridine ring of one molecule are (x, y) in the c-axis projection, there should be a single weight peak in the Patterson projection at $(2x, 2y)$ with related peaks of double weight at $(2x, \frac{1}{2})$ and at $(0, \frac{1}{2}-2y)$.

The only peak at $y = \frac{1}{2}$ is I in Fig. 1 and is situated at $(\frac{1}{3}, \frac{1}{2})$. II, IV, V and VI have approximately the same x coordinate as I, but VI is so heavy that only a portion of it could be attributed to vectors between centres of pyridine rings. The third related peaks of the triplets are III corresponding to VI and a possible
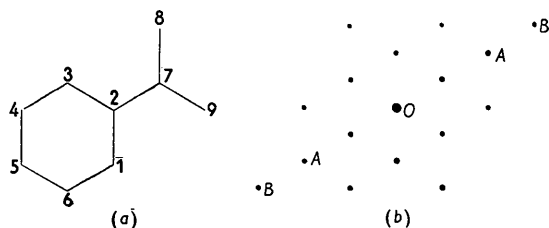

Fig. 2. (a) Diagrammatic representation of the nicotinic acid molecule. (b) Vector map of molecule in Fig. 2(a).

peak at VII, which would be obscured by the origin peak, corresponding to II. There are no peaks of suit-

able height corresponding to IV and V which cannot, therefore, be due to inter-ring vectors.

Fig. 2(a) is a simplified drawing of the molecule in which all the atoms (numbered 1–9) have been given the same weight, all bond lengths are equal and all bond angles are 120°. Fig. 2(b) is the corresponding vector diagram in which only those points at which two or more vectors coincide are marked. Peak A is made up of vectors 3–8, 2–5 and 1–9 in Fig. 2(a).
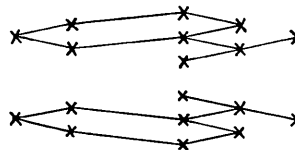
Fig. 4. The two possible molecular orientations compatible with atomic positions from the b-axis projection.

These three vectors will coincide only if the carboxyl group is coplanar with the ring. Peak B is due to vectors 4–8, 5–7 and 6–9. As peaks V and VI occur in the Patterson projection in positions corresponding to A and B, the molecule is probably planar. Peaks III and IV in the Patterson projection correspond to vectors of the type 2–4. As peak III is smaller than peak I, and yet must consist partly of vectors of the type 2–4, it is unlikely to be one of the intermolecular peaks so that peaks I, II and VII are the triplet due to vectors between ring centres.

The centre of the ring was therefore placed at $(\frac{1}{6}, \frac{1}{6})$, corresponding to peak II on the vector map $(\frac{1}{3}, \frac{2}{5})$, the carboxyl group was made coplanar with the ring, and the molecular orientation was fixed by rotating the molecule until A and B in Fig. 2(b) corresponded with peaks V and VI in the Patterson projection. Of the four different orientations of the

Fig. 3. The c-axis Fourier projection. Contours at intervals of 1 e.Å^{-2} starting at 2 e.Å^{-2} (broken line).
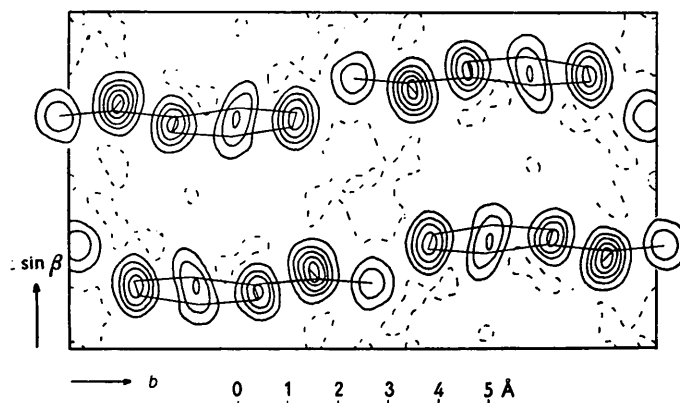
Fig. 5. The a-axis Fourier projection. Contours at intervals of 2 e.Å^{-2}; 2 e.Å^{-2} line broken.

molecule which satisfy these conditions, three were eliminated on packing considerations.

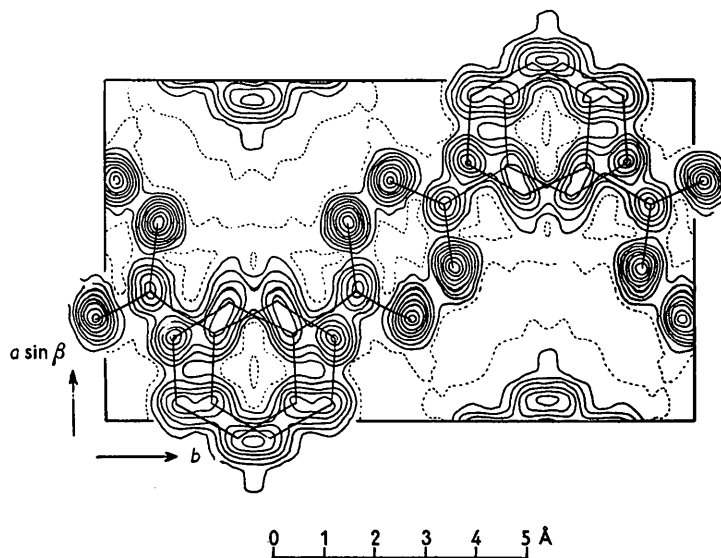The fourth orientation was used as a trial structure for the calculation of $hk0$ structure factors using the unitary atomic scattering factors given by Robertson (1935).

The x and y coordinates were refined by a series of nine Fourier projections. The structure factor disagreement $R = \Sigma||F_o|-|F_c||\div\Sigma F_o$ was 28·5% at this stage. This high value indicates that refinement was incomplete. It was not possible to reduce the disagreement further by Fourier methods because of the overlap in projection. Fig. 3 shows a c-axis Fourier projection based on the final atomic coordinates obtained from the three-dimensional analysis.

The a- and b-axis projections

As the orientation of the molecular plane was known from the optical data, it was decided to use the b-axis projection to determine the z coordinates. A projection of the molecule was drawn in the unit cell so that hydrogen bonds of length 2·8 Å could be formed between carboxyl groups related by a centre of symmetry. One of the four possible positions gave much better agreement than the others between observed and calculated structure factors. This was refined by two Fourier projections, followed by two applications of the method of steepest descents, as modified by Qurashi (1949), which reduced R_{h0l} to 31%. Further attempts at refinement did not improve the agreement.

The $0kl$ structure factors were calculated using y coordinates from the c-axis projection and z coordinates from the b-axis projection. The high disagreement of 62% was due to the incorrect bonding of the atoms in the b-axis projection. This is shown in Fig. 4 in which the nine atoms are bonded in two different ways to give two different orientations of the molecule.

In one case the carboxyl group is attached to the side of the ring with the higher z coordinates in order to permit hydrogen-bond formation between carboxyl

groups related by the centre of symmetry at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$. In the other case the carboxyl group is attached to the side of the ring with lower z coordinates so that hydrogen bonds can be formed between this group and the nitrogen atom of the molecule related to the first by the operation of the screw axis $(\frac{1}{2}, y, \frac{1}{4})$. As all the atoms have approximately the same scattering power, it is impossible to distinguish between the two orientations in the b-axis projection. In the course of the a-axis and three-dimensional refinements the molecule moved from the position postulated for the first case to that compatible with the hydrogen bonds between oxygen and nitrogen.

Two Fourier refinements of the a-axis projection reduced R_{0kl} to 40%. It was not possible to proceed further owing to lack of resolution. Figs. 5 and 6 show
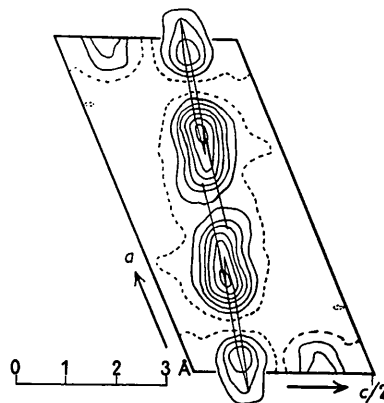
Fig. 6. The b-axis Fourier projection. Contours at intervals of 2 e.Å^{-2}; 2 e.Å^{-2} line broken.

a- and b-axis Fourier projections based on the final atomic coordinates.

Three-dimensional refinement

The final atomic coordinates from the a- and c-axis projections, which are shown in column 0 of Table 1, were used to calculate hkl structure factors. As the

Table 1. *Atomic parameters obtained from three-dimensional Fourier syntheses*

Atom		0	1	2	3	4	5*
C_1	x	0·356	—	0·3500	0·3466	0·3462	0·3459
	y	0·208	—	0·2064	0·2068	0·2085	0·2091
	z	0·210	0·215	0·2200	0·2161	0·2184	0·2171
C_2	x	0·253	—	0·2564	0·2570	0·2558	0·2585
	y	0·319	—	0·3168	0·3161	0·3152	0·3163
	z	0·187	0·183	0·1879	0·1900	0·1900	0·1906
C_3	x	0·049	—	0·0519	0·0526	0·0531	0·0519
	y	0·325	—	0·3227	0·3226	0·3221	0·3228
	z	0·171	0·153	0·1471	0·1454	0·1454	0·1454
C_4	x	0·932	—	0·9409	0·9423	0·9423	0·9441
	y	0·219	—	0·2187	0·2218	0·2223	0·2232
	z	0·165	0·139	0·1353	0·1320	0·1306	0·1312
C_5	x	0·042	—	0·0452	0·0455	0·0475	0·0446
	y	0·114	—	0·1177	0·1192	0·1196	0·1192
	z	0·158	0·157	0·1590	0·1597	0·1617	0·1607
C_6	x	0·385	—	0·3807	0·3776	0·3758	0·3722
	y	0·423	—	0·4240	0·4257	0·4261	0·4246
	z	0·234	0·226	0·2137	0·2113	0·2107	0·2096
N	x	0·239	—	0·2415	0·2435	0·2436	0·2444
	y	0·109	—	0·1123	0·1127	0·1126·	0·1116
	z	0·178	0·190	0·1997	0·2029	0·2051	0·2048
O_1	x	0·563	—	0·5622	0·5659	0·5653	0·5677
	y	0·409	—	0·4097	0·4101	0·4102	0·4100
	z	0·256	0·252	0·2442	0·2418	0·2404	0·2416
O_2	x	0·296	—	0·2980	0·3002	0·2993	0·2991
	y	0·524	—	0·5174	0·5155	0·5160	0·5167
	z	0·205	0·203	0·1948	0·1945	0·1939	0·1956

* Corrected for series-termination errors.

errors in the x and y coordinates were considered to be small compared with the errors in z, owing to lack of resolution in the a- and b-axis projections, it was decided that the z coordinates should first be obtained more accurately by means of line syntheses parallel to the c axis as nearly as possible through the atomic centres.

The amount of computation was reduced by grouping the atoms in pairs having approximately the same y coordinate (C_5 and N 7/60, C_1 and C_4 13/60, C_2 and C_3 19/60, C_6 and O_1 25/60 and O_2 31/60). The syntheses were computed at intervals of 1/60 of the cell sides, using Robertson's strip-and-stencil method (Robertson, 1948). The new z coordinates are shown in column 1 of Table 1. It can be seen from these values that the atoms may conveniently be divided into three groups such that only three plane sections parallel to (001) are necessary for the centre of each atom to be within $c/60$ of one or other of these sections. Using structure factors with recalculated signs, plane sections were computed at $z = 9/60$ for C_3, C_4 and C_5, at $z = 12/60$ for C_1, C_2, C_6, N and O_2 and at $z = 15/60$ for O_1, together with line sections as before. Atomic coordinates were calculated from the peak positions in the line sections and plane sections, using the method

of Parry & Pitt (1949), and are given in column 2 of Table 1.

Two more sets of plane sections at $z = 17/120$, 24/120 and 29/120, and line sections as before, were computed at intervals of 1/120 of the cell sides. The coordinates obtained are given in columns 3 and 4 of Table 1. The last Fourier synthesis did not cause any changes of sign in the calculated structure factors. Values of the structure factors to the nearest 0·2 were multiplied by 5 for these syntheses in order to minimise rounding-off errors. All computed sections were within 0·15 Å of atomic centres.

It was observed during the three-dimensional structure-factor calculations that $\Sigma F_c / \Sigma F_o$ for each c-axis layer line increased with l. This indicated that thermal vibration is greater in the direction perpendicular to the molecular plane than in other directions. A temperature factor of the form $\exp\left[-(B\sin^2\theta + Cl^2)\right]$ was determined from the ·structure factors calculated after the fourth Fourier synthesis by the method of least squares. The values obtained were: $B = -0·404$, $C = 0·0124$.

The atomic coordinates from the fourth Fourier synthesis were corrected for series termination errors by the F_c-synthesis method (Booth, 1946). Final

atomic coordinates are given in column 5 of Table 1. Fig. 7 is a composite electron-density map of the molecule prepared from the last set of plane sections. Table 5 shows the observed structure factors and those
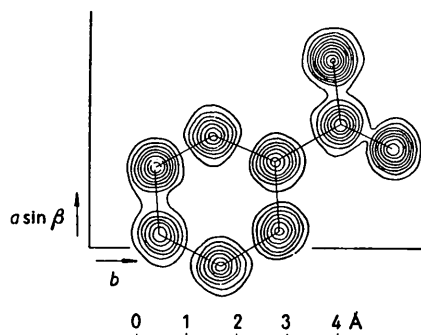

Fig. 7. Electron density map of nicotinic acid. Contours at intervals of 1 e.Å^{-3}.

calculated using the values of the atomic parameters given in column 5 of Table 1.

The structure-factor disagreements for the final coordinates are $R_{hkl} = 21 \cdot 9\%$, $R_{hk0} = 20 \cdot 8\%$, $R_{h0l} = 18 \cdot 7\%$ and $R_{0kl} = 22 \cdot 6\%$, including those terms too weak to be observed which were assigned half the minimum observable value of $|F_o|$.

Accuracy of the determination

The standard deviation $\sigma(x)$ etc. of atomic co-ordinates were estimated from the differences ΔF between the magnitudes of observed and calculated values of the structure factors (Cruickshank, 1949). It can be shown that

$$\sigma\left(\frac{\partial\varrho}{\partial x_j}\right) = \frac{2\pi}{a_j V}\left\{\sum_h\sum_k\sum_l h_j^2\Delta F^2\right\}^{\frac{1}{2}}.$$

In a monoclinic cell

$$\sigma(x) = \frac{\{\sigma^2(\partial\varrho/\partial x) - \sigma^2(\partial\varrho/\partial z)\cos^2\beta\}^{\frac{1}{2}}}{(\partial^2\varrho/\partial x^2)\sin^2\beta},$$

$$\sigma(y) = \frac{\sigma(\partial\varrho/\partial y)}{\partial^2\varrho/\partial y^2},$$

$$\sigma(z) = \frac{\{\sigma^2(\partial\varrho/\partial z) - \sigma^2(\partial\varrho/\partial x)\cos^2\beta\}^{\frac{1}{2}}}{(\partial^2\varrho/\partial z^2)\sin^2\beta}.$$

The values of $\partial^2\varrho/\partial x^2$ etc. were obtained from the values of electron density in the neighbourhood of the final Fourier peaks. Mean values for all atoms are

$$\partial^2\varrho/\partial x^2 = 71, \quad \partial^2\varrho/\partial y^2 = 86, \quad \partial^2\varrho/\partial z^2 = 65 \text{ e.Å}^{-5}.$$

The standard deviations of atomic position are $\sigma(x) = 0 \cdot 0158$, $\sigma(y) = 0 \cdot 0117$ and $\sigma(z) = 0 \cdot 0168$ Å. The standard deviations in the bond lengths were calculated by compounding the above values in the bond directions and multiplying by $\sqrt{2}$. They are $0 \cdot 022$ Å for C_2–C_3, C_5–N and C_6–O_1, and $0 \cdot 018$ Å for

all other bonds. The corresponding probable errors in bond lengths are $0 \cdot 015$ Å and $0 \cdot 012$ Å. The probable error in the bond angles is $1 \cdot 1°$.

Description of the structure

Bond lengths and other interatomic distances are given in Table 2 and bond angles in Table 3. The molecule

Table 2. *Interatomic distances*

Intramolecular bond lengths		Intermolecular distances	
N–C_1	1·336 Å	O_1–N′	2·664 Å
C_1–C_2	1·379	O_2–N′	3·256
C_2–C_3	1·388	O_2–C_1'	3·265
C_3–C_4	1·378	O_1–C_1'	3·540
C_4–C_5	1·385	$O_1''{-}_zO_2$	3·742
C_5–N	1·343	$O_1$$-_x$$C_4$	3·795
C_2–C_6	1·482	$O_1''{-}O_2$	3·883
C_6–O_1	1·338		
C_6–O_2	1·184		

Table 3. *Bond angles*

C_5–N–C_1	117·5°	C_3–C_2–C_6	118·2°
N–C_1–C_2	124·0	C_2–C_6–O_1	114·1
C_1–C_2–C_3	117·8	C_2–C_6–O_2	124·0
C_2–C_3–C_4	119·1	O_1–C_6–O_2	121·9
C_3–C_4–C_5	119·2	C_6–O_1–N′	110·4
C_4–C_5–N	122·4	O_1–N′–C_1'	120·9
C_1–C_2–C_6	124·0	O_1–N′–C_5'	121·6

M (Fig. 8) is related to M' by the screw axis $(\frac{1}{2}, y, \frac{1}{4})$, to M'' by the symmetry centre $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$, to M''' by the glide plane $(x, \frac{1}{4}, z)$ and to $_xM$, $_zM$ by unit translations in the x and z directions.

The pyridine ring is planar and its equation, determined by the method of least squares, is

$$-1 \cdot 3181x + 0 \cdot 5165y + 6 \cdot 1912z = 1.$$

The plane makes angles of 12°, 3° and 78° with the a, b and c axes respectively. The displacements of the atoms from this plane are given in Table 4.

Table 4. *Displacements of the atoms from the ring plane*

Atom	Displacement (Å)
C_1	−0·004
C_2	0·003
C_3	−0·002
C_4	0·001
C_5	−0·003
N	0·004
C_6	0·030
O_1	−0·046
O_2	0·095

Discussion

The molecule

The bond lengths in the ring do not differ significantly from the values of 1·39 Å for C–C and 1·37±0·03 Å for C–N found in an electron-diffraction study of pyridine by Schomaker & Pauling (1939). Fig. 9 com-

Table 5. *Values of observed and calculated structure factors* ($\times 10$)

hkl	$|F_o|$	F_c
000	—	2560
020	40	−67
040	123	−132
060	81	−67
080	18	−23
0,10,0	200	198
0,12,0	20	13
0,14,0	30	−22
100	13	−8
110	249	−416
120	258	−331
130	45	−43
140	29	−17
150	182	149
160	138	131
170	44	23
180	74	68
190	132	129
1,10,0	63	−46
1,11,0	13	−13
1,12,0	59	−55
1,13,0	46	33
1,14,0	52	−46
1,15,0	53	74
200	< 6	−46
210	66	65
220	120	−127
230	213	−201
240	108	−92
250	97	112
260	148	−125
270	15	3
280	55	−18
290	62	−53
2,10,0	40	−39
2,11,0	36	−26
2,12,0	22	16
2,13,0	96	−91
2,14,0	30	19
300	293	393
310	124	123
320	< 6	−38
330	51	3
340	91	87
350	< 9	7
360	64	47
370	110	−111
380	< 9	−12
390	64	−65
3,10,0	40	48
3,11,0	20	19
3,12,0	22	−19
3,13,0	23	15
3,14,0	23	26
400	107	105
410	115	−126
420	35	−22
430	89	−105
440	59	−64
450	99	98
460	79	88
470	45	32
480	62	62
490	97	103
4,10,0	32	30
4,11,0	13	15

hkl	$|F_o|$	F_c
4,12,0	20	−18
4,13,0	18	−13
500	< 9	−13
510	< 9	−15
520	< 9	−4
530	37	−45
540	87	−109
550	123	163
560	95	−101
570	45	−35
580	31	−27
590	37	−42
5,10,0	< 9	−4
5,11,0	31	−34
5,12,0	13	13
600	89	−108
610	60	−74
620	< 9	12
630	96	−122
640	70	82
650	< 9	0
660	31	29
670	< 9	−3
680	< 9	3
690	20	18
6,10,0	37	−34
700	29	−29
710	31	24
720	59	81
730	27	27
740	9	4
750	40	36
760	13	19
770	< 6	−7
780	18	23
800	30	−41
810	< 6	11
820	< 6	−3
830	9	−9
840	20	−34
850	22	43
$\bar{8}$11	20	−15
$\bar{8}$21	< 13	−14
$\bar{8}$31	31	25
$\bar{8}$41	23	11
$\bar{8}$51	64	−74
$\bar{8}$61	36	35
$\bar{7}$11	25	−18
$\bar{7}$21	59	47
$\bar{7}$31	31	−27
$\bar{7}$41	15	−12
$\bar{7}$51	41	−28
$\bar{7}$61	< 18	−17
$\bar{7}$71	30	−30
$\bar{7}$81	79	−74
$\bar{7}$91	58	−58
$\bar{6}$11	< 20	7
$\bar{6}$21	74	−50
$\bar{6}$31	25	17
$\bar{6}$41	56	56
$\bar{6}$51	72	61
$\bar{6}$61	110	−112
$\bar{6}$71	92	80
$\bar{6}$81	23	1

hkl	$|F_o|$	F_c
$\bar{6}$91	35	26
$\bar{6}$,10,1	74	59
$\bar{6}$,11,1	< 9	−3
$\bar{5}$11	119	131
$\bar{5}$21	70	51
$\bar{5}$31	82	92
$\bar{5}$41	150	175
$\bar{5}$51	191	−259
$\bar{5}$61	57	−33
$\bar{5}$71	78	57
$\bar{5}$81	27	−11
$\bar{5}$91	74	55
$\bar{5}$,10,1	39	24
$\bar{5}$,11,1	30	25
$\bar{5}$,12,1	27	19
$\bar{4}$11	154	−149
$\bar{4}$21	115	−110
$\bar{4}$31	41	−27
$\bar{4}$41	119	−134
$\bar{4}$51	107	−122
$\bar{4}$61	72	63
$\bar{4}$71	29	19
$\bar{4}$81	53	59
$\bar{4}$91	< 13	19
$\bar{4}$,10,1	29	−6
4,11,1	99	−88
4,12,1	31	18
4,13,1	37	−40
$\bar{3}$11	78	55
$\bar{3}$21	86	−86
$\bar{3}$31	25	6
$\bar{3}$41	40	46
$\bar{3}$51	38	11
$\bar{3}$61	103	−97
$\bar{3}$71	31	6
$\bar{3}$81	29	48
$\bar{3}$91	61	47
$\bar{3}$,10,1	100	−124
$\bar{3}$,11,1	35	17
$\bar{3}$,12,1	29	−16
$\bar{3}$,13,1	25	−16
$\bar{3}$,14,1	40	−38
$\bar{2}$11	94	139
$\bar{2}$21	129	131
$\bar{2}$31	214	225
$\bar{2}$41	292	248
$\bar{2}$51	25	−42
$\bar{2}$61	108	−99
$\bar{2}$71	138	106
$\bar{2}$81	42	−9
$\bar{2}$91	< 13	11
$\bar{2}$,10,1	25	−3
$\bar{2}$,11,1	33	−23
$\bar{2}$,12,1	73	58
$\bar{2}$,13,1	18	7
$\bar{2}$,14,1	47	30
$\bar{1}$11	< 6	−14
$\bar{1}$21	237	−265
$\bar{1}$31	145	−153
$\bar{1}$41	178	−172
$\bar{1}$51	294	−305
$\bar{1}$61	80	65
$\bar{1}$71	86	−64
$\bar{1}$81	160	155
$\bar{1}$91	167	183
$\bar{1}$,10,1	74	45

Table 5 (cont.)

hkl	|Fo|	Fc	hkl	|Fo|	Fc	hkl	|Fo|	Fc
1̄,11,1	97	−85	471	25	−21	6̄82	32	−20
1̄,12,1	76	42	481	< 20	17	6̄92	< 18	11
1̄,13,1	48	−30	491	29	24	6̄,10,2	44	32
1̄,14,1	38	−23	4,10,1	64	−57	6̄,11,2	18	− 6
			4,11,1	66	54			
011	42	−56	4,12,1	< 13	−14	5̄02	64	64
021	51	57				5̄12	< 13	4
031	63	−63	511	67	−70	5̄22	< 13	14
041	85	61	521	57	42	5̄32	48	−49
051	94	−91	531	55	−51	5̄42	32	−32
061	18	23	541	30	−11	5̄52	67	77
071	51	−32	551	166	241	5̄62	142	170
081	25	15	561	85	104	5̄72	18	−15
091	104	108	571	< 20	− 5	5̄82	40	31
0,10,1	275	−299	581	27	− 6	5̄92	66	−68
0,11,1	83	−76	591	56	−39	5̄,10,2	25	19
0,12,1	23	− 7	5,10,1	31	− 9	5̄,11,2	44	−44
0,13,1	18	− 6	5,11,1	< 11	− 8	5̄,12,2	< 6	−12
0,14,1	51	30						
0,15,1	< 4	− 2	611	< 20	−15	4̄02	91	107
			621	45	−25	4̄12	185	−200
111	128	168	631	41	−41	4̄22	65	65
121	212	−237	641	29	24	4̄32	76	−55
131	168	171	651	22	−17	4̄42	< 11	− 9
141	296	−299	661	35	−20	4̄52	92	109
151	202	187	671	72	−80	4̄62	22	21
161	162	144	681	< 15	6	4̄72	74	67
171	37	15	691	67	−71	4̄82	56	−60
181	141	143				4̄92	80	88
191	58	−33	711	20	−10	4̄,10,2	< 13	13
1,10,1	51	−56	721	39	34	4̄,11,2	22	−17
1,11,1	20	9	731	20	23	4̄,12,2	27	27
1,12,1	20	8	741	43	−38	4̄,13,2	18	−12
1,13,1	65	54	751	< 13	− 2			
1,14,1	35	−24	761	15	− 3	3̄02	241	−322
			771	32	34	3̄12	59	−75
211	< 9	−25				3̄22	25	20
221	154	127	811	13	20	3̄32	114	110
231	146	−158				3̄42	163	−174
241	101	66	9̄02	39	39	3̄52	27	−22
251	119	118	9̄12	< 9	−14	3̄62	46	−21
261	39	45	9̄22	18	−17	3̄72	74	−57
271	110	−104				3̄82	29	−15
281	32	−11	8̄02	< 18	28	3̄92	22	−16
291	65	67	8̄12	43	43	3̄,10,2	55	−60
2,10,1	33	23	8̄22	43	44	3̄,11,2	22	17
2,11,1	22	−20	8̄32	41	−34	3̄,12,2	31	24
2,12,1	67	62	8̄42	< 13	6	3̄,13,2	66	60
2,13,1	18	7	8̄52	20	19	3̄,14,2	27	−32
2,14,1	69	64	8̄62	33	36			
			8̄72	20	−12	2̄02	32	66
311	105	−98				2̄12	61	−58
321	< 13	−15	7̄02	32	− 9	2̄22	27	− 4
331	63	−74	7̄12	83	87	2̄32	127	−103
341	< 13	13	7̄22	39	−30	2̄42	76	−74
351	56	−47	7̄32	41	42	2̄52	23	4
361	31	− 4	7̄42	< 15	−10	2̄62	154	144
371	42	−47	7̄52	35	27	2̄72	22	23
381	< 20	19	7̄62	< 18	26	2̄82	56	27
391	< 20	28	7̄72	< 15	3	2̄92	97	−88
3,10,1	77	−92	7̄82	< 15	− 4	2̄,10,2	32	22
3,11,1	95	−81	7̄92	20	− 8	2̄,11,2	45	−34
3,12,1	42	−30				2̄,12,2	27	−22
3,13,1	25	7	6̄02	82	107	2̄,13,2	83	−72
			6̄12	< 20	− 3	2̄,14,2	73	−65
411	129	151	6̄22	27	12			
421	70	−63	6̄32	66	−75	1̄02	445	634
431	37	37	6̄42	126	−188	1̄12	371	−499
441	155	−228	6̄52	56	−52	1̄22	148	161
451	31	−21	6̄62	43	−44	1̄32	51	60
461	108	128	6̄72	< 20	7	1̄42	53	−37

Table 5 (cont.)

hkl	$\|F_o\|$	F_c	hkl	$\|F_o\|$	F_c	hkl	$\|F_o\|$	F_c
$\bar{1}52$	126	124	3,12,2	<15	4	$\bar{6}43$	<13	−8
$\bar{1}62$	49	−37	3,13,2	20	−8	$\bar{6}53$	139	−146
$\bar{1}72$	44	26				$\bar{6}63$	112	119
$\bar{1}82$	69	−62	402	94	−127	$\bar{6}73$	<13	0
$\bar{1}92$	65	53	412	29	27	$\bar{6}83$	29	−19
$\bar{1}$,10,2	125	110	422	27	−43	$\bar{6}93$	30	22
$\bar{1}$,11,2	64	−49	432	39	32	$\bar{6}$,10,3	41	−30
$\bar{1}$,12,2	55	44	442	74	75	$\bar{6}$,11,3	9	−9
$\bar{1}$,13,2	29	26	452	36	41			
$\bar{1}$,14,2	9	5	462	108	−114	$\bar{5}13$	122	−139
			472	45	−32	$\bar{5}23$	80	−74
002	515	−822	482	56	−39	$\bar{5}33$	20	−28
012	304	−498	492	75	−69	$\bar{5}43$	173	−225
022	101	−87	4,10,2	48	−43	$\bar{5}53$	59	52
032	23	−4	4,11,2	33	−29	$\bar{5}63$	111	121
042	99	104	4,12,2	<9	−1	$\bar{5}73$	32	−24
052	73	76				$\bar{5}83$	29	29
062	78	74	502	63	−51	$\bar{5}93$	37	−40
072	64	47	512	<20	−3	$\bar{5}$,10,3	32	−28
082	38	22	522	20	−2	$\bar{5}$,11,3	37	−39
092	101	94	532	45	−43	$\bar{5}$,12,3	13	−18
0,10,2	144	−136	542	97	138			
0,11,2	29	−22	552	82	−83	$\bar{4}13$	64	56
0,12,2	22	−20	562	22	10	$\bar{4}23$	74	64
0,13,2	<13	12	572	29	9	$\bar{4}33$	55	55
0,14,2	<9	7	582	29	17	$\bar{4}43$	39	29
			592	45	27	$\bar{4}53$	65	59
102	56	−108	5,10,2	35	−16	$\bar{4}63$	30	−18
112	15	16				$\bar{4}73$	27	30
122	36	39	602	33	27	$\bar{4}83$	35	−37
132	20	41	612	78	92	$\bar{4}93$	<13	−29
142	59	36	622	<18	16	$\bar{4}$,10,3	79	−71
152	41	−13	632	58	68	$\bar{4}$,11,3	83	75
162	158	−159	642	13	10	$\bar{4}$,12,3	18	−18
172	42	−26	652	15	11	$\bar{4}$,13,3	<9	−1
182	49	−40	662	33	−17			
192	109	−108	672	15	−8	$\bar{3}13$	20	9
1,10,2	31	−6	682	22	21	$\bar{3}23$	71	62
1,11,2	20	−20				$\bar{3}33$	97	122
1,12,2	42	34	702	<13	−8	$\bar{3}43$	25	−5
1,13,2	58	−47	712	25	26	$\bar{3}53$	67	−49
1,14,2	65	56	722	37	−53	$\bar{3}63$	72	79
			732	35	−38	$\bar{3}73$	77	83
202	145	186	742	22	2	$\bar{3}83$	<13	−4
212	97	−107	752	9	7	$\bar{3}93$	57	−60
222	<15	5				$\bar{3}$,10,3	51	40
232	96	97	$\bar{9}13$	20	−15	$\bar{3}$,11,3	<13	19
242	136	169	$\bar{9}23$	<13	0	$\bar{3}$,12,3	30	25
252	60	−56	$\bar{9}33$	<9	3	$\bar{3}$,13,3	13	−6
262	59	45						
272	47	−32	$\bar{8}13$	<18	−3	$\bar{2}13$	72	−94
282	30	11	$\bar{8}23$	41	28	$\bar{2}23$	103	−129
292	<20	−2	$\bar{8}33$	31	−28	$\bar{2}33$	86	−96
2,10,2	48	39	$\bar{8}43$	42	−31	$\bar{2}43$	180	−204
2,11,2	27	5	$\bar{8}53$	30	22	$\bar{2}53$	128	−163
2,12,2	37	−27	$\bar{8}63$	<15	−1	$\bar{2}63$	126	121
2,13,2	69	51	$\bar{8}73$	37	−37	$\bar{2}73$	62	−51
						$\bar{2}83$	86	90
302	91	−97	$\bar{7}13$	41	34	$\bar{2}93$	22	−3
312	156	−184	$\bar{7}23$	69	−54	$\bar{2}$,10,3	32	−29
322	27	−20	$\bar{7}33$	36	31	$\bar{2}$,11,3	23	23
332	58	−37	$\bar{7}43$	<15	0	$\bar{2}$,12,3	<13	1
342	15	16	$\bar{7}53$	<22	3	$\bar{2}$,13,3	22	−22
352	60	55	$\bar{7}63$	<22	1	$\bar{2}$,14,3	<6	−2
362	42	−36	$\bar{7}73$	42	50			
372	66	74	$\bar{7}83$	23	22	$\bar{1}13$	126	−119
382	44	37	$\bar{7}93$	70	69	$\bar{1}23$	86	85
392	91	85				$\bar{1}33$	76	79
3,10,2	<18	−4	$\bar{6}13$	23	22	$\bar{1}43$	42	27
3,11,2	29	−11	$\bar{6}23$	59	45	$\bar{1}53$	125	147
			$\bar{6}33$	60	56	$\bar{1}63$	<13	−1

Table 5 (cont.)

hkl	$|F_o|$	F_c	hkl	$|F_o|$	F_c	hkl	$|F_o|$	F_c
$\bar{1}73$	66	62	473	< 23	−15	$\bar{4}04$	185	−220
$\bar{1}83$	20	−17	483	< 20	13	$\bar{4}14$	91	95
$\bar{1}93$	126	−140	493	35	−37	$\bar{4}24$	< 13	−11
$\bar{1},10,3$	182	−189	4,10,3	38	43	$\bar{4}34$	< 13	− 4
$\bar{1},11,3$	67	48				$\bar{4}44$	18	−24
$\bar{1},12,3$	9	−11	513	33	23	$\bar{4}54$	< 13	− 5
$\bar{1},13,3$	< 9	3	523	27	−13	$\bar{4}64$	25	−27
$\bar{1},14,3$	27	14	533	< 18	−21	$\bar{4}74$	62	−70
			543	62	65	$\bar{4}84$	20	16
013	62	87	553	73	−93	$\bar{4}94$	39	−35
023	64	−66	563	78	−100	$\bar{4},10,4$	47	−43
033	79	95	573	49	−58	$\bar{4},11,4$	40	29
043	102	−98	583	< 15	− 6	$\bar{4},12,4$	< 9	− 4
053	127	137	593	< 13	0			
063	33	7				$\bar{3}04$	52	55
073	23	24	613	37	6	$\bar{3}14$	49	43
083	78	75	623	< 20	14	$\bar{3}24$	35	40
093	135	−137	633	< 15	17	$\bar{3}34$	117	−131
0,10,3	85	71	643	18	−16	$\bar{3}44$	59	59
0,11,3	122	106	653	< 15	25	$\bar{3}54$	59	52
0,12,3	42	35	663	< 13	−15	$\bar{3}64$	43	40
0,13,3	37	29				$\bar{3}74$	< 13	8
0,14,3	37	−32	$\bar{9}04$	< 13	19	$\bar{3}84$	< 13	4
			$\bar{9}14$	23	5	$\bar{3}94$	23	−20
113	81	−97	$\bar{9}24$	< 13	− 1	$\bar{3},10,4$	46	38
123	105	107	$\bar{9}34$	< 9	14	$\bar{3},11,4$	13	−20
133	109	−121				$\bar{3},12,4$	9	− 5
143	154	178	$\bar{8}04$	< 18	21	$\bar{3},13,4$	75	−77
153	40	29	$\bar{8}14$	< 18	− 7			
163	102	−96	$\bar{8}24$	65	−62	$\bar{2}04$	62	−41
173	32	−17	$\bar{8}34$	< 15	13	$\bar{2}14$	76	−68
183	30	−21	$\bar{8}44$	23	4	$\bar{2}24$	92	116
193	< 18	−17	$\bar{8}54$	30	22	$\bar{2}34$	36	39
1,10,3	29	17				$\bar{2}44$	64	65
1,11,3	< 23	16	$\bar{7}04$	67	63	$\bar{2}54$	80	76
1,12,3	51	14	$\bar{7}14$	52	−55	$\bar{2}64$	53	−46
1,13,3	35	−30	$\bar{7}24$	< 13	−12	$\bar{2}74$	35	−31
			$\bar{7}34$	66	−69	$\bar{2}84$	56	−43
213	40	−38	$\bar{7}44$	40	−38	$\bar{2}94$	70	60
223	72	−79	$\bar{7}54$	< 13	5	$\bar{2},10,4$	< 13	6
233	< 18	7	$\bar{7}64$	38	−34	$\bar{2},11,4$	< 13	2
243	< 15	13	$\bar{7}74$	< 9	− 4	$\bar{2},12,4$	49	38
253	55	−45	$\bar{7}84$	15	−16	$\bar{2},13,4$	22	30
263	55	−63	$\bar{7}94$	< 9	0			
273	30	7				$\bar{1}04$	445	−563
283	48	54	$\bar{6}04$	46	−38	$\bar{1}14$	78	63
293	45	−50	$\bar{6}14$	23	−22	$\bar{1}24$	56	24
2,10,3	83	−75	$\bar{6}24$	36	−25	$\bar{1}34$	37	−35
2,11,3	< 20	4	$\bar{6}34$	48	−37	$\bar{1}44$	59	38
2,12,3	23	−27	$\bar{6}44$	107	126	$\bar{1}54$	31	−29
2,13,3	20	8	$\bar{6}54$	83	89	$\bar{1}64$	23	16
			$\bar{6}64$	76	85	$\bar{1}74$	< 13	−10
313	67	86	$\bar{6}74$	18	−25	$\bar{1}84$	38	20
323	< 20	−13	$\bar{6}84$	9	9	$\bar{1}94$	32	32
333	< 20	33	$\bar{6}94$	13	−15	$\bar{1},10,4$	134	−106
343	56	−51	$\bar{6},10,4$	15	−15	$\bar{1},11,4$	48	38
353	66	80	$\bar{6},11,4$	13	−17	$\bar{1},12,4$	< 9	−10
363	< 20	1				$\bar{1},13,4$	20	−15
373	< 22	− 7	$\bar{5}04$	48	−29			
383	< 20	10	$\bar{5}14$	45	−43	004	56	49
393	36	−33	$\bar{5}24$	27	21	014	182	218
3,10,3	< 18	− 3	$\bar{5}34$	20	−10	024	102	110
3,11,3	62	73	$\bar{5}44$	51	51	034	73	60
3,12,3	35	28	$\bar{5}54$	58	54	044	41	−39
			$\bar{5}64$	81	−88	054	65	−58
413	65	−82	$\bar{5}74$	< 13	− 9	064	80	−76
423	< 25	27	$\bar{5}84$	40	−42	074	38	−49
433	51	−55	$\bar{5}94$	56	67	084	22	−17
443	91	113	$\bar{5},10,4$	9	− 9	094	96	−91
453	108	158	$\bar{5},11,4$	9	23	0,10,4	52	42
463	56	−56	$\bar{5},12,4$	22	27	0,11,4	18	12

Table 5 (cont.)

hkl	$\lvert F_o \rvert$	F_c	hkl	$\lvert F_o \rvert$	F_c	hkl	$\lvert F_o \rvert$	F_c
0,12,4	15	24	$\bar{7}15$	31	−29	$\bar{1}15$	82	90
0,13,4	< 13	−14	$\bar{7}25$	45	45	$\bar{1}25$	< 13	5
			$\bar{7}35$	< 18	− 2	$\bar{1}35$	27	− 9
104	< 18	18	$\bar{7}45$	27	−15	$\bar{1}45$	29	− 8
114	38	−23	$\bar{7}55$	< 13	0	$\bar{1}55$	20	−16
124	49	65	$\bar{7}65$	60	53	$\bar{1}65$	18	−11
134	38	28	$\bar{7}75$	40	−44	$\bar{1}75$	< 13	− 8
144	< 15	26	$\bar{7}85$	< 9	− 2	$\bar{1}85$	63	−24
154	59	−57	$\bar{7}95$	23	−32	$\bar{1}95$	< 13	− 3
164	57	80				$\bar{1}$,10,5	181	153
174	< 25	16	$\bar{6}15$	33	−45	$\bar{1}$,11,5	47	44
184	< 25	16	$\bar{6}25$	< 13	−18	$\bar{1}$,12,5	< 9	1
194	51	47	$\bar{6}35$	50	−55			
1,10,4	< 22	16	$\bar{6}45$	75	77	015	36	−58
1,11,4	< 20	13	$\bar{6}55$	112	115	025	39	46
1,12,4	< 13	−16	$\bar{6}65$	< 13	−16	035	38	−61
			$\bar{6}75$	51	−51	045	88	91
204	115	−152	$\bar{6}85$	22	18	055	54	−55
214	22	−15	$\bar{6}95$	30	−31	065	46	−35
224	44	36	$\bar{6}$,10,5	< 6	2	075	29	12
234	< 20	5				085	48	−54
244	57	−65	$\bar{5}15$	51	67	095	58	31
254	< 20	1	$\bar{5}25$	76	78	0,10,5	40	33
264	< 23	−24	$\bar{5}35$	22	− 1	0,11,5	37	−34
274	29	43	$\bar{5}45$	88	89	0,12,5	15	−13
284	< 23	5	$\bar{5}55$	63	59			
294	< 22	34	$\bar{5}65$	59	−58	115	22	20
2,10,4	< 15	−23	$\bar{5}75$	< 13	1	125	32	−47
2,11,4	< 13	2	$\bar{5}85$	39	−47	135	44	46
2,12,4	18	22	$\bar{5}95$	< 13	4	145	39	−40
			$\bar{5}$,10,5	< 9	1	155	68	−65
304	48	−66	$\bar{5}$,11,5	35	41	165	< 22	5
314	66	79				175	22	32
324	< 20	4	$\bar{4}15$	< 13	0	185	< 20	0
334	46	66	$\bar{4}25$	< 13	5	195	18	−23
344	23	15	$\bar{4}35$	< 13	−13	1,10,5	< 15	− 7
354	47	−51	$\bar{4}45$	32	−28	1,11,5	< 13	12
364	20	−20	$\bar{4}55$	23	2			
374	< 18	−30	$\bar{4}65$	31	44	215	25	36
384	< 18	−29	$\bar{4}75$	< 13	−13	225	< 20	29
394	36	−66	$\bar{4}85$	< 13	1	235	< 20	25
3,10,4	20	−28	$\bar{4}95$	< 13	− 7	245	30	− 1
			$\bar{4}$,10,5	74	80	255	33	20
404	< 22	50	$\bar{4}$,11,5	13	−18	265	< 20	2
414	47	1	$\bar{4}$,12,5	< 6	0	275	< 18	27
424	< 25	28				285	22	−36
434	< 20	12	$\bar{3}15$	23	−37	295	< 15	− 7
444	< 15	5	$\bar{3}25$	< 13	−11	2,10,5	48	51
454	74	−94	$\bar{3}35$	69	−89			
			$\bar{3}45$	68	−62	315	22	−42
504	50	48	$\bar{3}55$	48	20	325	15	3
514	33	35	$\bar{3}65$	22	14	335	< 15	−19
524	< 20	− 6	$\bar{3}75$	50	−64	345	80	72
534	46	73	$\bar{3}85$	< 13	− 9	355	< 18	−14
544	42	−62	$\bar{3}95$	< 13	3	365	29	−41
554	< 15	10	$\bar{3}$,10,5	< 13	7	375	15	23
			$\bar{3}$,11,5	< 9	5	385	< 13	4
604	22	20	$\bar{3}$,12,5	15	−21	395	< 9	3
614	23	−36						
624	18	−43	$\bar{2}15$	20	−12	415	23	36
			$\bar{2}25$	87	88	425	< 20	−16
$\bar{9}15$	< 9	− 7	$\bar{2}35$	< 13	30	435	38	22
$\bar{9}25$	< 13	3	$\bar{2}45$	61	52	445	< 13	6
			$\bar{2}55$	107	125	455	80	−136
$\bar{8}15$	< 9	18	$\bar{2}65$	42	−43			
$\bar{8}25$	40	−34	$\bar{2}75$	29	38	515	13	− 4
$\bar{8}35$	15	23	$\bar{2}85$	63	−73	525	< 13	− 9
$\bar{8}45$	18	9	$\bar{2}95$	88	−65			
$\bar{8}55$	< 9	11	$\bar{2}$,10,5	< 13	0	$\bar{8}06$	9	−12
$\bar{8}65$	< 9	2	$\bar{2}$,11,5	30	19	$\bar{8}16$	38	−32
			$\bar{2}$,12,5	22	−26	$\bar{8}26$	27	21

Table 5 (cont.)

hkl	$|F_o|$	F_c
$\bar{8}36$	< 9	− 4
$\bar{8}46$	18	4
$\bar{8}56$	18	−19
$\bar{7}06$	58	−51
$\bar{7}16$	< 13	0
$\bar{7}26$	< 13	− 2
$\bar{7}36$	41	38
$\bar{7}46$	73	85
$\bar{7}56$	< 9	5
$\bar{7}66$	9	30
$\bar{7}76$	6	−10
$\bar{7}86$	< 6	16
$\bar{6}06$	< 13	3
$\bar{6}16$	< 13	9
$\bar{6}26$	< 13	10
$\bar{6}36$	56	50
$\bar{6}46$	23	−16
$\bar{6}56$	45	−36
$\bar{6}66$	81	−87
$\bar{6}76$	< 13	− 2
$\bar{6}86$	< 9	− 3
$\bar{6}96$	18	25
$\bar{5}06$	< 13	−34
$\bar{5}16$	55	48
$\bar{5}26$	25	−30
$\bar{5}36$	< 13	6
$\bar{5}46$	< 13	− 6
$\bar{5}56$	48	−45
$\bar{5}66$	< 13	1
$\bar{5}76$	< 13	− 8
$\bar{5}86$	40	35
$\bar{5}96$	27	−35
$\bar{5},10,6$	6	−11
$\bar{4}06$	99	117
$\bar{4}16$	22	20
$\bar{4}26$	38	− 8
$\bar{4}36$	< 20	−29
$\bar{4}46$	40	44
$\bar{4}56$	< 13	5
$\bar{4}66$	< 13	− 1
$\bar{4}76$	15	21
$\bar{4}86$	< 13	7
$\bar{4}96$	< 9	3
$\bar{4},10,6$	37	38
$\bar{4},11,6$	15	−14
$\bar{3}06$	< 13	− 9
$\bar{3}16$	< 13	17
$\bar{3}26$	< 13	− 7
$\bar{3}36$	49	56
$\bar{3}46$	37	26
$\bar{3}56$	22	− 8
$\bar{3}66$	35	−33
$\bar{3}76$	20	−15
$\bar{3}86$	< 13	0
$\bar{3}96$	39	29
$\bar{3},10,6$	25	−19
$\bar{3},11,6$	9	19
$\bar{2}06$	58	−76
$\bar{2}16$	112	94
$\bar{2}26$	22	−45
$\bar{2}36$	22	−31
$\bar{2}46$	< 15	−20
$\bar{2}56$	46	−43
$\bar{2}66$	< 13	−10
$\bar{2}76$	< 13	6

hkl	$|F_o|$	F_c
$\bar{2}86$	53	34
$\bar{2}96$	20	−11
$\bar{2},10,6$	25	−29
$\bar{2},11,6$	38	27
$\bar{1}06$	185	203
$\bar{1}16$	110	97
$\bar{1}26$	33	− 2
$\bar{1}36$	20	11
$\bar{1}46$	49	−29
$\bar{1}56$	< 13	− 7
$\bar{1}66$	30	−20
$\bar{1}76$	< 13	−12
$\bar{1}86$	< 13	3
$\bar{1}96$	59	−55
$\bar{1},10,6$	71	60
$\bar{1},11,6$	< 9	− 9
006	47	41
016	36	−29
026	22	−15
036	46	−43
046	30	1
056	< 15	7
066	33	48
076	< 15	29
086	< 13	13
096	35	54
0,10,6	15	− 1
106	25	−32
116	36	37
126	< 22	−13
136	23	−18
146	25	−44
156	25	29
166	< 20	−20
176	< 18	1
186	< 15	− 4
196	< 13	−10
206	33	33
216	43	47
226	< 15	− 7
236	< 18	− 2
246	27	− 3
256	< 15	−10
266	< 15	15
276	< 13	−25
286	15	−10
306	29	65
316	< 13	−16
326	< 13	22
336	22	−32
346	35	−36
356	< 9	− 3
366	29	32
406	13	− 3
416	9	0
426	< 9	− 8
$\bar{8}17$	15	−21
$\bar{8}27$	27	12
$\bar{8}37$	< 6	−13
$\bar{7}17$	18	12
$\bar{7}27$	13	−22
$\bar{7}37$	36	−28
$\bar{7}47$	22	4

hkl	$|F_o|$	F_c
$\bar{7}57$	36	27
$\bar{7}67$	52	−54
$\bar{6}17$	23	53
$\bar{6}27$	< 13	18
$\bar{6}37$	< 13	11
$\bar{6}47$	82	78
$\bar{6}57$	37	−24
$\bar{6}67$	49	−39
$\bar{6}77$	33	31
$\bar{6}87$	13	−14
$\bar{5}17$	< 13	− 7
$\bar{5}27$	35	−37
$\bar{5}37$	< 13	−11
$\bar{5}47$	18	−13
$\bar{5}57$	40	−30
$\bar{5}67$	< 13	15
$\bar{5}77$	< 9	−11
$\bar{5}87$	18	30
$\bar{5}97$	13	11
$\bar{4}17$	< 13	− 8
$\bar{4}27$	< 13	− 7
$\bar{4}37$	23	−27
$\bar{4}47$	20	15
$\bar{4}57$	30	7
$\bar{4}67$	23	−33
$\bar{4}77$	< 9	−20
$\bar{4}87$	9	− 2
$\bar{4}97$	22	19
$\bar{3}17$	23	21
$\bar{3}27$	< 13	10
$\bar{3}37$	< 13	19
$\bar{3}47$	51	42
$\bar{3}57$	< 15	24
$\bar{3}67$	27	−30
$\bar{3}77$	< 13	25
$\bar{3}87$	< 9	−16
$\bar{3}97$	< 9	9
$\bar{2}17$	40	42
$\bar{2}27$	33	−27
$\bar{2}37$	13	−18
$\bar{2}47$	< 13	2
$\bar{2}57$	42	−50
$\bar{2}67$	< 13	7
$\bar{2}77$	18	−26
$\bar{2}87$	< 9	12
$\bar{2}97$	64	60
$\bar{1}17$	27	−40
$\bar{1}27$	18	−10
$\bar{1}37$	< 13	16
$\bar{1}47$	25	16
$\bar{1}57$	< 13	−16
$\bar{1}67$	< 13	7
$\bar{1}77$	< 9	− 2
$\bar{1}87$	< 9	− 4
$\bar{1}97$	56	39
017	15	25
027	< 15	−22
037	20	28
047	43	−43
057	< 13	− 9
067	38	29
077	< 13	− 7
087	< 9	− 1

Table 5 (cont.)

| hkl	$|F_o|$	F_c	hkl	$|F_o|$	F_c	hkl	$|F_o|$	F_c
117	< 15	6	$\bar{5}58$	< 9	7	$\bar{1}28$	< 13	−16
127	< 15	22	$\bar{5}68$	< 6	9	$\bar{1}38$	15	−17
137	< 15	− 3	$\bar{5}78$	< 4	16	$\bar{1}48$	18	16
147	22	− 1				$\bar{1}58$	< 9	9
157	23	30	$\bar{4}08$	< 18	−23	$\bar{1}68$	18	17
167	< 13	17	$\bar{4}18$	31	−13			
177	< 9	− 5	$\bar{4}28$	< 22	− .3	008	< 13	−11
			$\bar{4}38$	29	28	018	< 13	7
217	< 13	−21	$\bar{4}48$	25	−15	028	< 13	−17
227	< 9	−12	$\bar{4}58$	22	−10	038	< 9	10
237	< 9	−12				048	15	− 2
247	18	7	$\bar{3}08$	20	13	058	13	13
257	15	−25	$\bar{3}18$	15	0			
			$\bar{3}28$	< 13	−18	108	23	32
$\bar{7}08$	22	18	$\bar{3}38$	< 13	−18	118	< 9	− 8
$\bar{7}18$	15	10	$\bar{3}48$	27	−26	128	13	−10
$\bar{7}28$	22	13	$\bar{3}58$	< 9	−16			
$\bar{7}38$	13	10	$\bar{3}68$	< 9	5	$\bar{5}19$	15	− 7
			$\bar{3}78$	23	18	$\bar{5}29$	< 13	6
$\bar{6}08$	6	− 3				$\bar{5}39$	< 6	5
$\bar{6}18$	< 9	3	$\bar{2}08$	71	88			
$\bar{6}28$	< 9	− 7	$\bar{2}18$	32	−23	$\bar{4}19$	< 13	8
$\bar{6}38$	27	−18	$\bar{2}28$	30	− 6	$\bar{4}29$	< 13	− 3
$\bar{6}48$	29	−14	$\bar{2}38$	< 13	13	$\bar{4}39$	15	20
$\bar{6}58$	9	−14	$\bar{2}48$	18	3			
			$\bar{2}58$	< 9	12	$\bar{3}19$	< 6	0
$\bar{5}08$	41	49	$\bar{2}68$	< 13	8	$\bar{3}29$	15	−14
$\bar{5}18$	33	−22	$\bar{2}78$	< 6	4	$\bar{3}39$	13	− 1
$\bar{5}28$	< 9	9						
$\bar{5}38$	22	6	$\bar{1}08$	< 13	−18	$\bar{2}19$	20	−29
$\bar{5}48$	20	3	$\bar{1}18$	67	−56	$\bar{2}29$	< 6	− 3
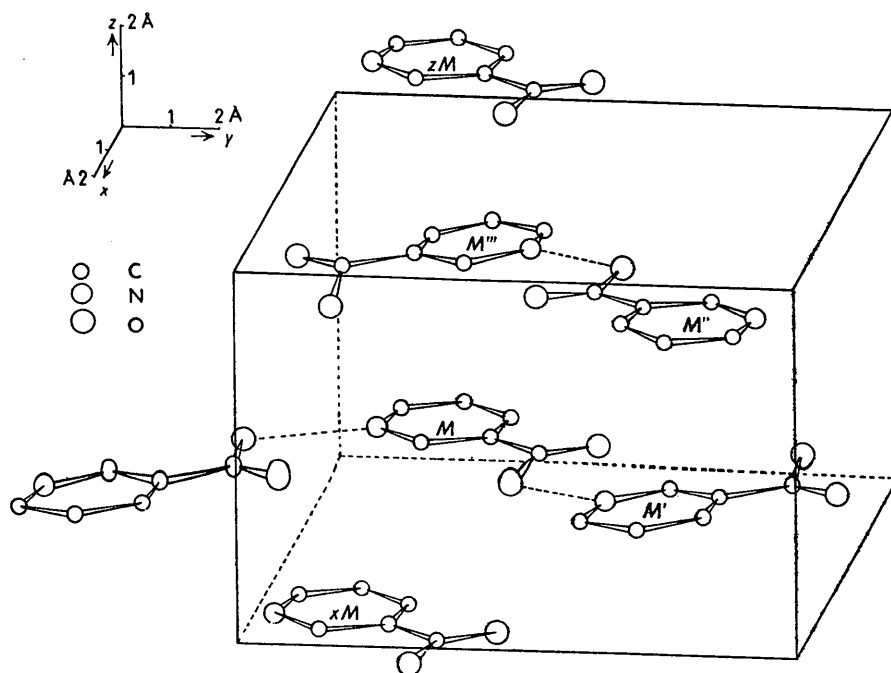
Fig. 8. The structure, showing one unit cell and associated molecules.

pares observed dimensions of the molecule with calculated bond lengths in the ring (given in parentheses). These values were obtained by converting the bond orders calculated for pyridine by Longuet-Higgins &
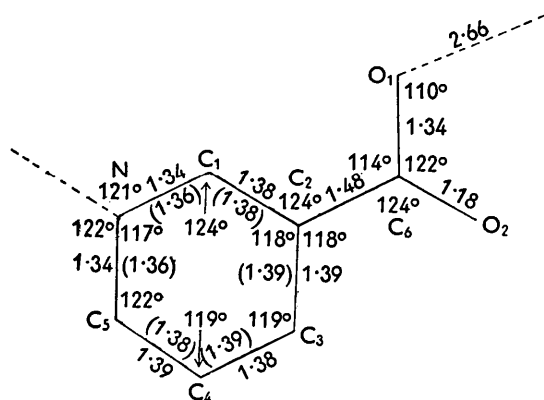

Fig. 9. Bond lengths and bond angles in nicotinic acid. Calculated bond lengths in parentheses.

Coulson (1947) to bond lengths using curves given by Coulson (1951) for C–C bonds and by Cox & Jeffrey (1951) for C–N bonds.

The bond C_2–C_6 joining the ring to the carboxyl group is significantly shorter than the C–C single bond in diamond. The fact that the C–O_2 bond is much shorter than C–O_1 indicates that the hydrogen atom is attached to O_1 and not to O_2.

The molecular environment

There is only one active hydrogen atom in the molecule. This links O_1 of molecule M (Fig. 8) to the nitrogen atom of the molecule M' by a bond of length 2·66 Å. No other intermolecular contact is closer than 3·2 Å. As the hydrogen bond angles are 110° at O_1 and 121° at N' there is no distortion from the tetrahedral configuration at O_1 or from the trigonal configuration at N'.

The molecules are linked by the hydrogen bonds in zigzag chains parallel to the *b* axis, the chains being held together only by van der Waals forces.

Twinning

The most probable explanation of the twinning is that it takes place in such a way that the molecule $_xM'''$ (not shown in Fig. 8) is reflected across (100) and translated in the *y* direction so as to form hydrogen bonds between its carboxyl group and that of the molecule M. These O–H . . . O bonds are normally stronger than O–H . . . N bonds but in this case do not permit such close packing of the molecules.

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