Sakore, T. D., Tavale, S. S. & Pant, L. M. (1967). *Acta Cryst.* **22**, 720.
Schomaker, V., Waser, J., Marsh, R. E. & Bergman, G. (1959). *Acta Cryst.* **12**, 600.
Silverman, J. & Yannoni, N. F. (1967*a*). *J. Phys. Chem.* **71**, 1381.
Silverman, J. & Yannoni, N. F. (1967*b*). *J. Chem. Soc.* (B) p. 194.
Silverman, J., Krukonis, A. P. & Yannoni, N. F. (1967). *Acta Cryst.* **23**, 1057.
Trotter, J. & Williston, C. S. (1966). *Acta Cryst.* **21**, 285.
Trueblood, K. N., Goldish, E. & Donohue, J. (1961). *Acta Cryst.* **14**, 1009.
Williams, R. M. & Wallwork, S. C. (1966). *Acta Cryst.* **21**, 406.

Acta Cryst. (1968). B**24**, 1488

The Crystal Structures of DL-α-Amino-n-butyric Acid

By Tadayoshi Ichikawa and Yoichi Iitaka

Faculty of Pharmaceutical Sciences, University of Tokyo, Hongo, Tokyo, Japan

(*Received* 8 *January* 1968)

The structures of the *A* and *B* crystalline modifications of DL-α-amino-n-butyric acid have been determined. *A* is monoclinic, space group $P2_1/a$, with $a = 9{\cdot}85$, $b = 4{\cdot}77$, $c = 11{\cdot}94$ Å, $\beta = 101{\cdot}0°$. *B* is tetragonal, space group $P4_2/n$, with $a = 13{\cdot}40$, $c = 5{\cdot}82$ Å. Full-matrix least-squares refinement with anisotropic temperature factors yielded final *R* indices 0·13 and 0·12 for *A* and *B* respectively. In *A* the γ-C atom is distributed mainly among three positions corresponding to *trans*, *gauche* I and *gauche* II with respect to the nitrogen atom. In *B* γ-C is located only at the *trans* position to the nitrogen atom. Within the *A* crystals the molecules form a double layer parallel to the 001 plane through N–H···O hydrogen bonds so that single layers are formed by two of the three kinds of hydrogen bond and they are joined together by the third kind to form a double layer. In *B*, one of the three kinds of hydrogen bond links the molecules head to tail along the *c* axis. The second kind links the molecules to form a column and the columns are joined together through lateral hydrogen bonds of the third kind.

Introduction

In the course of a study on the infrared absorption spectra of amino acids, Tsuboi, Iitaka, Suzuki & Mizushima (1959) found two crystalline modifications of DL-α-amino-n-butyric acid ($CH_3CH_2CH(NH_2)COOH$). These two modifications were named *A* and *B*. On the basis of infrared absorption studies they suggested that the *A* and *B* modifications are composed of the molecules of different conformational isomers. To confirm this suggestion and to determine the accurate molecular conformations of the isomers, an X-ray analysis of the crystal structures of both *A* and *B* modifications has been carried out. A preliminary report of the present work has been published (Ichikawa, Iitaka & Tsuboi, 1968).

Experimental

The crystals of the *A* modification (monoclinic) were grown from warm saturated aqueous solutions by slow cooling. They are colorless plates elongated along the *b* axis with cleavages on (001) and bounded by {001} dominating and smaller {100} faces. Crystals of the *B* modification (tetragonal) were obtained by slow recrystallization from aqueous solutions with excess of ethanol. These crystals are fine needles often fibrous but sometimes square pyramids, elongated along the *c* axis and usually mixed with the tabular *A* form crystals. The lattice dimensions were measured on precession photographs taken with Cu *K* radiation. X-ray photographs of the crystal of the *A* modification generally showed weak diffuse scattering extending in the **c*** direction along the row lines in reciprocal space. Relative intensities of these diffuse lines to the Bragg reflexions were found to vary to some extent from specimen to specimen.

Crystal data

DL-α-Amino-n-butyric acid,
$C_4H_9NO_2$ ($CH_3CH_2CH(NH_2)COOH$)

Crystal system	*A* Monoclinic	*B* Tetragonal
a	9·85 ± 0·01 Å	13·40 ± 0·01 Å
b	4·77 ± 0·005	
c	11·94 ± 0·01	5·82 ± 0·005
β	101·0° ± 0·2°	
U	551·3 Å^3	1045·3 Å^3
Z	4	8
Volume/molecule	137·83 Å^3	130·66 Å^3
Systematic absences	*h*0*l* when $h \neq 2n$ 0*k*0 when $k \neq 2n$	*hk*0 when $h+k \neq 2n$ 00*l* when $l \neq 2n$
Space group	$P2_1/a$	$P4_2/n$

Three-dimensional intensity data were collected from equi-inclination Weissenberg photographs, taken with Cu $K\alpha$ radiation on multiple film packs. Intensities were measured visually with a calibrated intensity scale and corrected for Lorentz, polarization and spot-shape (Phillips, 1954) factors. Because of the low linear absorption coefficient ($\mu_{\text{Cu }K\alpha} \simeq 9{\cdot}8$ cm^{-1}) and the small cross-sections of the crystals, no absorption correction was applied. These intensity data were scaled by correlating various layers and the mean temperature factors were calculated by Wilson's (1942) method. Throughout the present study the atomic scattering factors given by Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) were used for carbon, oxygen and nitrogen atoms.

Intensity data

	A	*B*
a axis	0 to 3rd layer	
b axis	0 to 3	0 to 3
c axis		0 to 4
Total number of independent structure factors	809	630
Mean temperature factor by Wilson's method	3·2	2·0

Determination and refinement of the structure

Modification A

At the beginning, attempts were made to identify the intramolecular O(1)–O(2) vectors near the origin in the Patterson map and then to seek several intermolecular O–O vectors which may be interpreted in terms of the intramolecular O(1)–O(2) vectors. The x and z coordinates of these atoms were determined from the Harker section at $v=\frac{1}{2}$ (Fig. 1). These values were consistent with those obtained from the Patterson peaks due to the two molecules related by a centre of symmetry, which in turn gave the y coordinates of these oxygen atoms. Phases of the structure factors were then calculated from the positions of O(1) and O(2) and a three-dimensional electron density map was calculated. The structure of the molecule except the terminal C_γ atom [C(4)] was found out in this map. A subsequent Fourier synthesis phased by the six atoms shown in Fig. 2 ($R=0{\cdot}44$) revealed an unexpected feature of the C_γ atom. Although the electron density associated with C_γ was very low, this map indicated that this atom is distributed chiefly among three positions, one *trans* and two *gauche* positions to the amino nitrogen atom. A difference Fourier synthesis calculated at the same time showed clearly the distribution of the electron density around these sites.

Refinement of the structure was at first carried out by a block-matrix isotropic least-squares method with the six atoms. The R index decreased to 0·22. The site occupancy factors of the C_γ atom at the three maximum positions were estimated from the subsequent difference map (Fig. 3), in which all atoms except C_γ and hydrogen atoms are subtracted. These values were put in the full-matrix least-squares calculation [program by Busing, Martin & Levy (1962)]. In repeated cycles of the refinement with anisotropic temperature factors, the site occupancy factors and the temperature factors of the three C_γ atoms were fixed alternately in each cycle. After several cycles of calculation R converged to 0·13. Hydrogen atoms were not included throughout the refinement.

The final atomic parameters are listed in Table 1 together with their estimated standard deviations. Comparisons of the observed and calculated structure factors are given in Table 3.

Modification B

Many trial interpretations of the three-dimensional as well as the basal projections of the Patterson maps, by assuming several possible orientations of molecules,

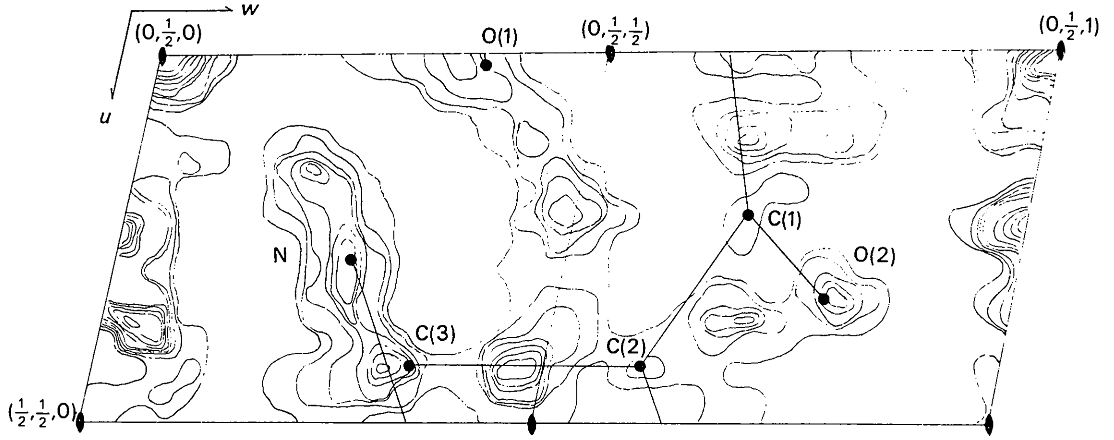

Fig. 1. The Harker section at $v=\frac{1}{2}$ of the A modification. The image of the molecule drawn by the final atomic coordinates is superposed on the map.

yielded no definite answer as to the location of the molecule within the crystal. This was due mainly to severe overlapping of the peaks even in a sharpened Patterson map calculated for the structure with atoms at rest. In the course of these trials, it was noticed that the Harker section at $w=\frac{1}{2}$ (Fig. 4) gave a prominent peak at $u=0{\cdot}202$, $v=0{\cdot}122$. The c-axis projection of the electron density function was, therefore, calculated by assuming that two superposed atoms were present at $x=-0{\cdot}210$, $y=-0{\cdot}088$. A plausible molecular model based on this Fourier synthesis gave the x and y coordinates of the two carboxyl oxygen atoms [O(1) and O(2)] and a nitrogen atom. The orientation of the molecule thus derived was consistent with the fact that the c-axis lattice constant ($c=5{\cdot}82$ Å) is nearly the same as that of L-alanine ($c=5{\cdot}78$ Å) in which the molecules are arranged head to tail along the c axis. The z parameters of these atoms were determined by model building taking account of hydrogen bondings between the carboxyl oxygen and the amino nitrogen atoms, and of the Patterson map previously calculated.

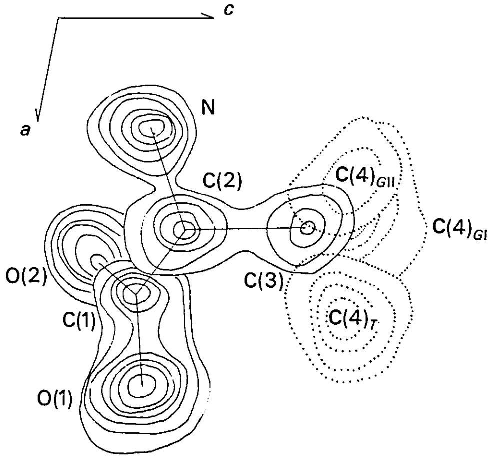

Fig. 2. Composite electron density map of the molecule viewed along the b axis of modification A. Contours are drawn at 1,2,3,...e.Å^{-3}. Dotted lines are drawn at 0·4, 0·8, 1·2,...e.Å^{-3}.

Table 1. *Atomic parameters and their standard deviations* (*A modification*)

	x	y	z	Occupancy factor, m
	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	$\sigma(m)$
O(1)	0·5103	0·4519	0·1749	1·0
	(0·0005)	(0·0013)	(0·0005)	fixed
O(2)	0·3337	0·1869	0·0934	1·0
	(0·0005)	(0·0011)	(0·0005)	fixed
N	0·1421	0·5535	0·1292	1·0
	(0·0005)	(0·0014)	(0·0005)	fixed
C(1)	0·3845	0·3957	0·1490	1·0
	(0·0007)	(0·0017)	(0·0006)	fixed
C(2)	0·2874	0·5913	0·1950	1·0
	(0·0007)	(0·0018)	(0·0006)	fixed
C(3)	0·2846	0·5225	0·3226	1·0
	(0·0010)	(0·0047)	(0·0008)	fixed
C(4)$_T$	0·411	0·571	0·400	0·44
	(0·002)	(0·010)	(0·002)	(0·02)
C(4)$_{GI}$	0·275	0·332	0·369	0·40
	(0·004)	(0·009)	(0·002)	(0·03)
C(4)$_{GII}$	0·195	0·658	0·369	0·26
	(0·006)	(0·017)	(0·006)	(0·02)

Thermal parameters

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
	$\sigma(\beta_{11})$	$\sigma(\beta_{22})$	$\sigma(\beta_{33})$	$\sigma(\beta_{12})$	$\sigma(\beta_{13})$	$\sigma(\beta_{23})$
O(1)	0·0052	0·0433	0·0118	0·0000	0·0019	−0·0015
	(0·0005)	(0·0035)	(0·0006)	(0·0011)	(0·0004)	(0·0012)
O(2)	0·0087	0·0260	0·0085	0·0014	0·0022	−0·0028
	(0·0006)	(0·0029)	(0·0005)	(0·0011)	(0·0004)	(0·0010)
N	0·0051	0·0397	0·0065	0·0019	0·0009	0·0010
	(0·0005)	(0·0040)	(0·0005)	(0·0012)	(0·0004)	(0·0011)
C(1)	0·0087	0·0263	0·0060	0·0022	0·0021	0·0020
	(0·0008)	(0·0042)	(0·0005)	(0·0015)	(0·0005)	(0·0013)
C(2)	0·0070	0·0406	0·0069	0·0007	0·0009	−0·0029
	(0·0007)	(0·0049)	(0·0006)	(0·0015)	(0·0005)	(0·0014)
C(3)	0·0126	0·2427	0·0046	0·0190	0·0007	−0·0053
	(0·0012)	(0·0191)	(0·0007)	(0·0042)	(0·0007)	(0·0032)
C(4)$_T$	0·008	0·250	0·007	0·005	−0·002	−0·017
	(0·002)	(0·035)	(0·002)	(0·008)	(0·002)	(0·007)
C(4)$_{GI}$	0·035	0·119	0·004	0·000	0·004	0·009
	(0·006)	(0·028)	(0·002)	(0·010)	(0·002)	(0·005)
C(4)$_{GII}$	0·018	0·154	0·019	0·015	−0·001	−0·039
	(0·005)	(0·055)	(0·006)	(0·013)	(0·004)	(0·016)

A three-dimensional Fourier synthesis phased by these three atoms ($R=0{\cdot}50$) revealed the remaining four atoms [C(1), C(2), C(3) and C(4)]. To confirm the fact that the γ-C atom occupies only the *trans* position to the nitrogen atom, a difference Fourier synthesis subtracting all the atoms except the C_γ and hydrogen atoms was calculated. The R value at this stage was 0·42.

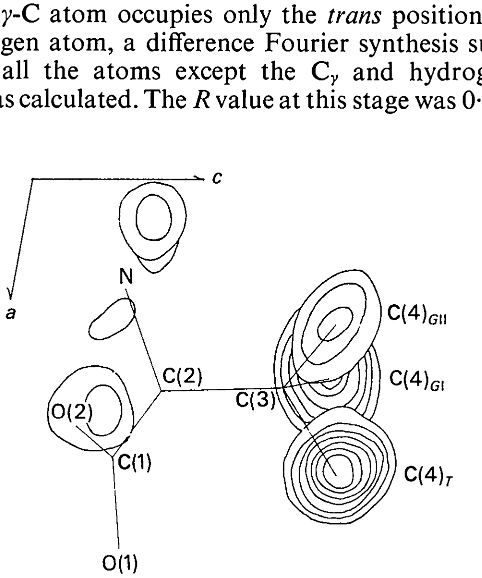

Fig. 3. Composite drawing of the difference electron density map of the *A* modification viewed along the *b* axis, subtracting all the atoms except C_γ and hydrogen. Contours are drawn at 0·2, 0·3, 0·4, . . . e.Å^{-3}.

Refinement of the structure was straightforward. The block-matrix isotropic least-squares calculation (program by Okaya and Ashida) yielded an R value of 0·14 after three cycles, and two more cycles of the full-matrix anisotropic least-squares calculation with the program by Busing, Martin & Levy (1962) reduced R to 0·12. A composite projection of the final Fourier synthesis is shown in Fig. 5. The final atomic parameters and their standard deviations are listed in Table 2, and a list of the observed and calculated structure factors is given in Table 4.

Description and discussion of the structure

Modification A

The molecular dimensions determined for the *A* crystals are shown in Fig. 6 and listed in Tables 5 and 6. The standard deviations of these values were calculated from those listed in Table 1.

The three positions of C(4) (γ-carbon atom) assigned in the present determination are neither very accurate nor quite definite. The difference electron-density section (synthesized without C(4) and all hydrogen atom contributions) cut through the three maximum peaks of C(4) (Fig. 7) shows that the electron density of C(4) is dispersed over a circular belt, indicating a rotation of C(4) about the C(2)–C(3) bond. However, the dis-

Table 2. *Positional parameters of atoms and estimated standard deviations* (*B modification*)

	x $\sigma(x)$	y $\sigma(y)$	z $\sigma(z)$
O(1)	−0·2101	−0·0825	−0·3604
	(0·0004)	(0·0004)	(0·0009)
O(2)	−0·0556	−0·0553	−0·2355
	(0·0004)	(0·0004)	(0·0009)
N	−0·1100	−0·0843	0·2108
	(0·0004)	(0·0004)	(0·0010)
C(1)	−0·1465	−0·0649	−0·2009
	(0·0005)	(0·0005)	(0·0012)
C(2)	−0·1870	−0·0519	0·0392
	(0·0005)	(0·0005)	(0·0012)
C(3)	−0·2128	0·0596	0·0915
	(0·0006)	(0·0006)	(0·0014)
C(4)	−0·2965	0·1002	−0·0622
	(0·0006)	(0·0006)	(0·0015)

Thermal parameters

	β_{11} $\sigma(\beta_{11})$	β_{22} $\sigma(\beta_{22})$	β_{33} $\sigma(\beta_{33})$	β_{12} $\sigma(\beta_{12})$	β_{13} $\sigma(\beta_{13})$	β_{23} $\sigma(\beta_{23})$
O(1)	0·0042	0·0071	0·0181	−0·0000	0·0010	−0·0020
	(0·0003)	(0·0004)	(0·0018)	(0·0003)	(0·0006)	(0·0007)
O(2)	0·0040	0·0066	0·0191	−0·0004	0·0023	−0·0013
	(0·0003)	(0·0004)	(0·0017)	(0·0003)	(0·0006)	(0·0007)
N	0·0041	0·0046	0·0154	−0·0004	−0·0002	0·0011
	(0·0004)	(0·0004)	(0·0019)	(0·0003)	(0·0007)	(0·0007)
C(1)	0·0032	0·0035	0·0162	−0·0002	0·0027	0·0006
	(0·0004)	(0·0004)	(0·0024)	(0·0003)	(0·0008)	(0·0008)
C(2)	0·0043	0·0043	0·0092	−0·0005	0·0003	0·0008
	(0·0004)	(0·0005)	(0·0022)	(0·0004)	(0·0008)	(0·0008)
C(3)	0·0055	0·0044	0·0278	0·0008	−0·0023	−0·0026
	(0·0005)	(0·0005)	(0·0031)	(0·0004)	(0·0011)	(0·0010)
C(4)	0·0065	0·0047	0·0314	0·0009	−0·0018	0·0006
	(0·0006)	(0·0005)	(0·0035)	(0·0005)	(0·0012)	(0·0011)

Table 3. *Observed and calculated structure factors* (*modification A*)

H	K	L	F(OBS)	F(CAL)
2	0	0	32.37	-30.58
4	0	0	5.41	-2.34
6	0	0	22.55	21.60
8	0	0	10.72	9.30
10	0	0	4.59	4.48
1	1	0	22.46	29.33
2	1	0	29.06	30.68
3	1	0	23.00	23.46
4	1	0	13.19	-13.75
5	1	0	18.65	16.81
6	1	0	11.82	-10.78
7	1	0	8.54	-8.60
8	1	0	7.76	7.29
9	1	0	14.46	15.48
10	1	0	2.72	-2.48
11	1	0	3.15	4.03
0	2	0	22.50	28.04
1	2	0	22.66	-23.86
2	2	0	11.40	12.65
3	2	0	8.34	7.19
4	2	0	9.07	8.38
5	2	0	4.67	5.49
6	2	0	2.64	1.84
7	2	0	10.25	-11.49
8	2	0	7.85	9.18
10	2	0	2.95	3.33
11	2	0	1.71	0.79
1	3	0	7.99	7.11
2	3	0	5.56	4.88
3	3	0	2.40	1.10
4	3	0	3.33	4.11
5	3	0	9.00	10.04
6	3	0	10.32	-12.61
7	3	0	1.94	2.08
8	3	0	2.18	1.22
9	3	0	2.41	-1.82
0	4	0	4.53	-4.07
2	4	0	4.21	3.93
1	5	0	4.86	-4.86
3	5	0	7.74	6.70
-12	0	1	1.06	1.02
-10	0	1	2.00	-0.93
-8	0	1	20.47	21.05
-6	0	1	12.45	13.25
-4	0	1	5.86	4.04
-2	0	1	23.91	-23.28
2	0	1	32.29	31.10
4	0	1	34.55	-34.98
6	0	1	32.20	27.32
8	0	1	8.18	-7.71
-12	1	1	1.84	-1.95
-10	1	1	1.95	2.68
-9	1	1	8.73	7.57
-8	1	1	8.43	-8.38
-7	1	1	8.45	8.64
-6	1	1	1.69	1.02
-5	1	1	10.78	10.07
-4	1	1	11.86	12.36
-3	1	1	30.47	28.88
-2	1	1	31.34	-33.81
-1	1	1	41.72	-44.84
0	1	1	9.90	-12.94
1	1	1	35.66	45.09
2	1	1	15.23	15.80
3	1	1	17.68	16.18
4	1	1	4.60	-4.97
6	1	1	11.99	-11.57
7	1	1	9.00	-9.16
9	1	1	4.58	3.49
10	1	1	2.65	-1.88
11	1	1	3.80	3.38
-11	2	1	2.90	-2.09
-8	2	1	12.70	15.71
-7	2	1	3.67	-4.06
-6	2	1	2.86	-3.43
-5	2	1	1.69	1.91
-4	2	1	1.88	2.62
-3	2	1	16.16	-15.07
-2	2	1	18.41	18.06
1	2	1	24.50	-26.09
2	2	1	2.10	0.22
3	2	1	3.98	-3.70
4	2	1	4.98	3.64
5	2	1	10.48	11.58
6	2	1	4.91	4.29
7	2	1	14.84	-16.30
8	2	1	4.13	-3.92
9	2	1	4.68	-4.97
11	2	1	1.91	1.82
-11	3	1	2.52	5.38
-10	3	1	4.53	-4.10
-9	3	1	3.68	-2.63
-8	3	1	5.21	-4.36
-6	3	1	7.84	6.25
-5	3	1	11.17	12.01
-4	3	1	4.86	-5.73
-2	3	1	18.98	-18.97
-1	3	1	1.71	-0.66
1	3	1	9.03	8.55
2	3	1	12.17	-10.68
3	3	1	4.20	-1.79
4	3	1	1.58	-1.16
6	3	1	6.05	-5.89
7	3	1	2.96	2.77
8	3	1	5.28	-4.65
9	3	1	2.61	-2.18
10	3	1	2.50	-1.87
-2	4	1	6.32	6.71
-1	4	1	12.66	-12.49
1	4	1	8.58	-6.61
2	4	1	4.22	-3.89
-2	5	1	5.78	-4.36
-1	5	1	4.20	2.70
0	5	1	3.33	1.48
1	5	1	8.70	-9.09
2	5	1	4.68	-4.00
3	5	1	5.00	3.79
-12	0	2	0.77	0.33
-10	0	2	2.73	-0.96
-8	0	2	1.65	-2.32
-4	0	2	7.91	-8.58
-2	0	2	5.60	-3.54
0	0	2	43.32	-39.77
2	0	2	3.63	8.80
4	0	2	38.54	-31.07
6	0	2	2.07	-1.82
8	0	2	7.66	-7.45
10	0	2	7.63	-8.92
-12	1	2	1.99	-2.69
-10	1	2	1.38	-1.41
-9	1	2	10.30	-9.84
-8	1	2	2.02	-0.39
-7	1	2	9.25	9.00
-6	1	2	13.79	-14.99
-5	1	2	2.21	-1.88
-4	1	2	19.94	19.64
-3	1	2	10.79	-10.49
-2	1	2	15.49	-13.24
-1	1	2	24.56	-26.21
0	1	2	22.01	-26.03
1	1	2	15.92	-14.58
2	1	2	4.40	2.39
3	1	2	15.04	12.25
4	1	2	2.84	0.36
5	1	2	26.49	-27.21
6	1	2	1.81	-3.10
7	1	2	6.57	-6.79
8	1	2	2.86	-2.16
10	1	2	1.65	-1.54
-11	2	2	2.26	-1.79
-10	2	2	1.80	-0.68
-9	2	2	4.12	-4.75
-8	2	2	5.93	6.11
-7	2	2	10.41	-10.27
-6	2	2	6.69	-7.19
-5	2	2	3.47	4.90
-4	2	2	14.81	-14.34
-2	2	2	20.29	19.12
-1	2	2	17.11	-16.49
0	2	2	17.81	-19.61
1	2	2	11.57	-11.28
2	2	2	17.37	-18.31
3	2	2	12.82	-13.64
4	2	2	3.15	2.21
5	2	2	12.36	13.96
6	2	2	3.71	-3.48
7	2	2	6.74	-6.19
8	2	2	7.36	-8.61
9	2	2	4.56	-4.87
10	2	2	2.49	-2.67
11	2	2	2.14	2.61
-11	3	2	2.42	3.35
-10	3	2	8.19	-6.99
-9	3	2	3.02	-2.47
-8	3	2	1.91	-1.85
-7	3	2	5.82	-4.32
-6	3	2	4.17	-3.72
-5	3	2	6.84	7.43
-4	3	2	2.83	-2.73
-3	3	2	4.48	-6.19
-2	3	2	13.47	-12.00
-1	3	2	10.91	-10.75
1	3	2	7.78	9.06
2	3	2	14.08	-13.30
3	3	2	8.76	-7.13
4	3	2	5.31	-5.68
5	3	2	8.38	-9.05
6	3	2	7.11	7.36
7	3	2	4.60	2.70
8	3	2	5.41	-4.38
9	3	2	1.92	-1.30
10	3	2	2.33	-2.62
-3	4	2	9.42	7.99
-2	4	2	3.65	3.63
-1	4	2	15.13	-16.10
1	4	2	7.81	-6.97
2	4	2	7.34	-8.00
-2	5	2	5.73	-3.93
-1	5	2	7.58	7.28
0	5	2	3.29	2.78
1	5	2	6.29	-6.67
2	5	2	4.59	-3.88
-12	0	3	2.84	-3.38
-10	0	3	2.02	2.54
-8	0	3	17.15	-17.35
-6	0	3	14.97	-15.77
-4	0	3	7.40	7.27
-2	0	3	40.58	-40.95
0	0	3	52.02	-48.56
2	0	3	15.89	-11.54
4	0	3	9.85	7.63
6	0	3	14.96	-15.26
8	0	3	6.52	-8.06
10	0	3	5.83	-6.38
-11	1	3	12.67	-4.93
-9	1	3	14.17	-12.76
-8	1	3	5.17	5.30
-6	1	3	17.64	-17.84
-5	1	3	4.20	-5.29
-4	1	3	7.32	6.22
-3	1	3	41.79	-38.62
-2	1	3	25.06	27.38
-1	1	3	23.76	-19.90
0	1	3	21.84	-22.62
1	1	3	13.06	-15.65
2	1	3	4.63	3.25
3	1	3	6.29	-7.25
4	1	3	2.06	2.28
5	1	3	10.65	-10.59
6	1	3	2.78	2.42
7	1	3	7.30	-6.06
9	1	3	4.25	-3.71
11	1	3	3.13	-4.06
-11	2	3	4.78	3.77
-10	2	3	2.54	-2.60
-9	2	3	6.96	-6.94
-8	2	3	2.87	-2.55
-7	2	3	4.24	-3.78
-6	2	3	8.37	-9.14
-5	2	3	2.14	0.46
-4	2	3	15.05	-15.64
-3	2	3	12.10	10.30
-2	2	3	5.56	4.98
-1	2	3	6.75	-7.37
0	2	3	18.65	-21.52
1	2	3	6.12	8.59
2	2	3	22.13	-23.09
3	2	3	5.54	-6.55
4	2	3	8.88	8.11
5	2	3	3.05	3.45
6	2	3	5.40	-5.00
7	2	3	6.36	6.23
8	2	3	7.01	-8.91
9	2	3	2.37	-1.18
-10	3	3	4.85	-3.33
-8	3	3	3.10	1.62
-7	3	3	6.86	-6.43
-6	3	3	4.35	-4.80
-4	3	3	4.79	-3.18
-3	3	3	9.29	-9.20
-2	3	3	7.77	8.50
-1	3	3	12.01	-11.29
0	3	3	5.20	4.83
1	3	3	4.77	6.08
2	3	3	6.15	-5.91
3	3	3	5.00	-5.82
4	3	3	4.45	-4.63
5	3	3	9.75	-10.56
6	3	3	11.11	13.34
7	3	3	3.56	2.15
10	3	3	1.68	-1.99
-3	4	3	13.13	11.32
-2	4	3	2.99	-2.00
-1	4	3	8.26	-7.26
0	4	3	2.91	3.84
2	4	3	3.68	-4.65
3	4	3	8.80	8.16
-1	5	3	4.70	5.75
3	5	3	5.40	-4.50
-12	0	4	2.15	-2.34
-10	0	4	3.93	2.99
-8	0	4	16.62	-18.36
-6	0	4	17.31	-18.16
-4	0	4	28.32	29.20
-2	0	4	4.13	3.76
0	0	4	75.78	-66.78
2	0	4	13.01	12.40
4	0	4	13.13	9.36
6	0	4	4.22	-3.92
8	0	4	2.69	3.38
-12	1	4	1.38	-1.38
-11	1	4	11.55	-4.96
-10	1	4	2.75	2.54
-9	1	4	10.68	-9.93
-8	1	4	9.21	8.37
-6	1	4	6.99	-6.83
-5	1	4	5.64	-6.64
-4	1	4	9.21	-9.66
-3	1	4	16.54	-13.54
-2	1	4	31.39	38.51
-1	1	4	9.60	8.33
0	1	4	12.87	-9.82
1	1	4	10.16	-11.09
2	1	4	4.89	-5.05
3	1	4	18.99	-17.35
4	1	4	10.79	12.60
5	1	4	7.16	7.94
7	1	4	7.97	7.30
9	1	4	2.44	-1.67
10	1	4	1.42	1.15
11	1	4	1.94	-2.61
-11	2	4	6.51	6.40
-10	2	4	2.41	-3.07
-9	2	4	2.59	-2.29
-8	2	4	5.20	-6.46
-7	2	4	1.49	2.40
-6	2	4	4.79	-3.61
-5	2	4	7.39	7.91
-4	2	4	3.02	-2.58
-2	2	4	1.38	-0.45
-1	2	4	8.47	7.56
0	2	4	6.99	-5.99
1	2	4	15.45	17.21
2	2	4	11.86	-11.42
4	2	4	6.29	7.29
5	2	4	4.16	-5.41
6	2	4	1.85	1.72
7	2	4	12.47	12.43
8	2	4	3.79	-3.49
10	2	4	2.50	2.63
-11	3	4	2.17	-3.24
-10	3	4	4.83	3.60
-8	3	4	2.18	3.70
-7	3	4	1.94	-3.11
-5	3	4	3.83	-3.46
-4	3	4	3.87	-2.32
-2	3	4	17.92	17.38
-1	3	4	8.12	-8.42
0	3	4	7.31	6.28
1	3	4	5.88	6.18
5	3	4	4.90	-6.49
6	3	4	8.03	8.34
7	3	4	2.14	2.99
8	3	4	2.78	2.96
9	3	4	2.37	2.27
-3	4	4	9.42	7.09
-2	4	4	3.68	-4.10
0	4	4	4.14	2.16
2	4	4	2.99	2.66
3	4	4	9.00	7.74
-3	5	4	5.50	-3.11
-2	5	4	6.27	3.02
-10	0	5	9.92	9.82
-8	0	5	7.03	-8.55
-6	0	5	7.22	-6.95
-4	0	5	23.20	24.04
-2	0	5	18.97	17.88
0	0	5	4.87	-4.06
2	0	5	21.70	16.46
4	0	5	17.60	15.81
6	0	5	6.80	-7.83
8	0	5	8.58	9.57
10	0	5	3.60	5.86
-12	1	5	1.67	-0.98
-10	1	5	2.06	1.27
-9	1	5	4.10	3.54
-8	1	5	10.32	8.24
-6	1	5	2.23	-2.61
-5	1	5	2.37	-2.07
-4	1	5	9.49	-8.13
-3	1	5	1.30	-1.90
-2	1	5	20.08	22.57
-1	1	5	45.85	42.89
0	1	5	1.87	1.98
2	1	5	9.41	-9.96
3	1	5	6.78	-5.48
4	1	5	8.88	9.20
5	1	5	11.63	11.68
6	1	5	2.87	-3.09
7	1	5	9.33	8.35
10	1	5	2.31	1.65
-11	2	5	3.94	3.72
-10	2	5	1.14	0.63
-9	2	5	2.54	1.37
-8	2	5	4.13	-5.11
-7	2	5	3.21	4.10
-6	2	5	6.89	8.55
-5	2	5	8.23	8.13
-4	2	5	2.48	2.04
-3	2	5	9.26	-8.28
-2	2	5	2.44	1.01
-1	2	5	8.88	8.98
0	2	5	10.91	11.02
1	2	5	14.35	15.93
2	2	5	4.39	4.29
3	2	5	1.83	-1.81
4	2	5	3.95	4.09
5	2	5	8.16	-10.05
6	2	5	2.65	2.54
7	2	5	7.35	7.48
8	2	5	2.41	2.34
9	2	5	3.25	3.32
10	2	5	2.29	3.26
-10	3	5	7.51	7.02
-9	3	5	3.90	3.93
-8	3	5	1.85	1.46
-7	3	5	2.23	0.79
-5	3	5	2.96	-3.43
-3	3	5	7.09	8.07
-2	3	5	8.45	7.70
0	3	5	5.41	4.92
1	3	5	2.40	2.46
2	3	5	2.68	1.96
3	3	5	8.07	6.97
4	3	5	1.34	1.80
5	3	5	1.37	-1.21
6	3	5	2.06	-2.23
7	3	5	1.25	1.97
8	3	5	3.06	2.01
9	3	5	1.04	1.03
-1	4	5	10.80	10.49
2	4	5	6.61	7.08
-3	5	5	5.52	-4.04
-2	5	5	4.86	2.12
-12	0	6	1.41	1.31
-10	0	6	5.27	5.31
-8	0	6	10.34	10.83
-6	0	6	3.09	-1.32
-4	0	6	18.52	21.09
0	0	6	23.41	19.87
2	0	6	11.17	7.86
4	0	6	6.67	9.13
8	0	6	5.09	5.43
10	0	6	4.32	4.86
-9	1	6	13.10	13.29
-8	1	6	3.68	2.92
-6	1	6	4.66	4.32
-5	1	6	5.08	5.39
-4	1	6	12.74	-13.23
-3	1	6	10.57	9.27
-2	1	6	3.76	3.53
-1	1	6	19.30	18.91
0	1	6	5.74	5.10
1	1	6	10.23	12.07
2	1	6	8.42	-7.06
3	1	6	10.91	-9.30
4	1	6	6.21	5.40
5	1	6	13.88	15.91
6	1	6	5.06	-4.71
7	1	6	3.33	2.56
8	1	6	2.96	-3.54
10	1	6	2.27	2.55
-11	2	6	2.10	-1.39
-10	2	6	1.41	0.92
-9	2	6	3.65	3.27
-7	2	6	2.17	1.70
-6	2	6	10.40	10.98
-5	2	6	2.82	1.94
-4	2	6	7.99	7.88
-3	2	6	12.26	-11.34
-2	2	6	7.97	-6.61
-1	2	6	1.98	-1.40
0	2	6	16.87	16.95
1	2	6	7.93	8.39
2	2	6	4.81	7.17
5	2	6	6.47	-7.56
6	2	6	3.05	3.25
7	2	6	2.29	-1.85
8	2	6	3.26	3.29
9	2	6	2.30	2.21
-10	3	6	4.17	4.31
-9	3	6	4.72	4.50
-8	3	6	3.60	-2.25
-7	3	6	1.56	1.82
-4	3	6	2.61	1.78
-3	3	6	7.63	9.06
-2	3	6	7.97	-8.08
-1	3	6	3.05	4.55
1	3	6	4.81	-6.18
2	3	6	2.64	1.68
3	3	6	8.20	8.47
4	3	6	1.37	2.01
5	3	6	2.07	2.98
6	3	6	7.97	-8.30
7	3	6	1.62	-1.54
-3	4	6	14.88	-8.51
-1	4	6	6.34	7.32
2	4	6	3.51	3.88
3	4	6	5.32	-5.15
-12	0	7	1.38	2.53
-10	0	7	4.71	-5.54
-8	0	7	15.72	17.36
-6	0	7	3.51	-2.17
-4	0	7	5.98	-6.52
-2	0	7	12.26	10.92
0	0	7	4.21	2.38
2	0	7	11.80	11.12
4	0	7	14.92	-16.34
-11	1	7	8.05	3.14
-10	1	7	4.74	-4.05
-9	1	7	6.85	6.10
-8	1	7	1.88	-1.96
-7	1	7	2.37	1.61
-6	1	7	6.81	7.17
-5	1	7	5.04	-5.37
-4	1	7	6.05	-5.42
-3	1	7	15.43	15.02
-2	1	7	12.52	-12.18
-1	1	7	6.94	-5.38
0	1	7	6.28	6.07
1	1	7	9.15	8.16
2	1	7	6.88	-7.06
3	1	7	4.93	-4.59
4	1	7	2.15	2.08
5	1	7	3.15	-1.77
8	1	7	4.70	-4.26
9	1	7	2.57	-2.25
-11	2	7	5.18	-5.73
-10	2	7	1.67	-1.18
-9	2	7	3.15	3.17
-8	2	7	1.98	2.41
-7	2	7	3.07	-4.00
-6	2	7	4.91	5.51
-5	2	7	2.07	1.62
-4	2	7	2.64	2.53
-3	2	7	6.00	-5.28
-2	2	7	9.73	-9.05
-1	2	7	9.67	-10.32
0	2	7	8.61	10.25
2	2	7	6.29	6.41
3	2	7	2.10	-2.31
4	2	7	10.30	-11.12
6	2	7	2.67	2.10
7	2	7	5.91	-4.07
8	2	7	1.38	1.36
-10	3	7	1.31	-1.60
-9	3	7	2.08	2.47
-8	3	7	2.63	-3.90
-6	3	7	2.23	-1.38
-5	3	7	3.71	-3.48
-4	3	7	5.79	6.04
-3	3	7	8.49	8.06
-2	3	7	13.35	-14.86
0	3	7	5.80	-5.51
1	3	7	5.89	-7.94
2	3	7	2.96	4.89
3	3	7	2.90	3.55
5	3	7	3.19	3.64
6	3	7	4.18	-4.93
7	3	7	3.37	-3.72
8	3	7	1.81	-2.57
-3	4	7	8.09	-6.57
3	4	7	6.96	-7.13
-2	5	7	3.10	-2.30
0	5	7	3.91	-3.80
-10	0	8	6.92	-7.79
-8	0	8	6.40	7.64
-6	0	8	6.36	7.19
-4	0	8	27.56	-30.32
-2	0	8	9.14	4.62
0	0	8	4.89	-3.11
2	0	8	2.95	-1.05
4	0	8	3.49	-4.14
6	0	8	4.47	-5.04
-11	1	8	9.02	3.37
-10	1	8	5.74	-5.45
-9	1	8	4.32	-3.96
-8	1	8	3.42	-2.63
-6	1	8	3.33	3.14
-5	1	8	2.79	-2.80
-4	1	8	1.91	2.91
-3	1	8	3.50	-2.89
-2	1	8	11.28	-10.93
-1	1	8	12.47	-12.81
0	1	8	4.40	3.75
1	1	8	4.93	-5.84
3	1	8	11.74	11.29
4	1	8	4.03	-4.97
5	1	8	12.70	-11.30
6	1	8	4.56	3.57
7	1	8	3.18	-3.12
8	1	8	3.52	-1.91
-11	2	8	2.64	-4.14
-10	2	8	1.31	-1.06
-8	2	8	1.61	-1.26
-7	2	8	2.84	-3.33
-6	2	8	3.34	3.70
-5	2	8	5.52	-5.69
-4	2	8	6.67	-7.80
-3	2	8	11.17	10.99
-2	2	8	5.58	-4.27
-1	2	8	10.72	-10.98
0	2	8	2.49	-2.52
1	2	8	3.45	-3.78
2	2	8	2.91	2.65
3	2	8	2.53	2.31
4	2	8	6.44	-7.32
5	2	8	2.10	2.16
6	2	8	1.54	-1.18
7	2	8	2.23	-1.82
8	2	8	1.27	1.08
-9	3	8	0.93	-0.76
-8	3	8	2.27	-2.50
-6	3	8	2.18	-1.55
-5	3	8	2.95	-2.99
-4	3	8	4.41	5.06
-3	3	8	1.58	1.16
-2	3	8	6.32	-6.75
-1	3	8	1.76	1.31
0	3	8	6.74	-6.66
1	3	8	6.74	-7.59
2	3	8	4.41	5.90
4	3	8	2.49	-2.34
5	3	8	1.71	2.32
7	3	8	2.02	-3.18
-10	0	9	4.82	-5.28
-8	0	9	2.33	-2.51
-6	0	9	5.73	5.72
-4	0	9	13.23	-15.65
2	0	9	17.22	-17.60
4	0	9	6.84	7.19
8	0	9	1.64	-1.18
-10	1	9	2.21	-2.02
-9	1	9	5.73	-6.04
-8	1	9	4.81	-3.63
-7	1	9	7.43	-7.03
-6	1	9	5.60	5.93
-5	1	9	8.88	9.04
-3	1	9	10.10	-8.79
-1	1	9	9.22	-10.06
1	1	9	4.94	-5.99
2	1	9	1.87	3.25
3	1	9	6.32	5.63
4	1	9	2.79	-2.72
5	1	9	4.58	-3.84
6	1	9	4.33	3.59
7	1	9	4.06	-4.26
-9	2	9	1.57	-0.93
-8	2	9	3.40	-2.98
-6	2	9	2.15	-2.61
-5	2	9	4.67	-5.40
-4	2	9	1.71	-1.92
-3	2	9	13.56	12.45
-2	2	9	3.33	-3.61
-1	2	9	1.68	-1.20
0	2	9	2.88	-1.65
1	2	9	8.19	-9.50
2	2	9	4.58	-4.55
3	2	9	8.04	8.09
5	2	9	2.07	2.26
6	2	9	1.49	-1.21
-9	3	9	1.34	-2.06
-8	3	9	1.31	-0.67
-7	3	9	1.52	-0.79
-6	3	9	1.94	1.47
-3	3	9	3.99	-3.92
-2	3	9	5.10	4.31
-1	3	9	2.50	2.77
0	3	9	3.93	-3.55
1	3	9	2.72	-3.39
2	3	9	2.64	2.29
3	3	9	2.76	-2.48
5	3	9	1.49	1.81
6	3	9	1.90	1.80
-10	0	10	1.39	-1.53
-8	0	10	2.46	-2.15
-6	0	10	1.71	-2.31
-4	0	10	2.08	1.08
0	0	10	3.07	4.04
2	0	10	8.51	-7.89
4	0	10	6.08	4.27
6	0	10	3.47	4.51
-10	1	10	2.02	2.59
-9	1	10	2.50	-2.73
-8	1	10	4.49	-3.91
-7	1	10	5.35	-4.42
-6	1	10	3.01	3.33
-5	1	10	5.60	5.71
-4	1	10	1.08	1.44
-1	1	10	5.47	-6.68
0	1	10	4.16	4.39
1	1	10	5.25	4.02
2	1	10	2.07	-2.29
4	1	10	1.60	-1.41
5	1	10	1.65	0.42
6	1	10	2.31	2.14
7	1	10	1.85	-2.08
-10	2	10	1.14	1.23
-7	2	10	1.01	-1.51
-6	2	10	7.80	-6.66
-4	2	10	5.48	4.34
-3	2	10	4.62	-4.68
-1	2	10	6.62	7.10
0	2	10	1.53	-1.27
1	2	10	6.38	-7.35
3	2	10	3.51	2.70
4	2	10	1.92	2.21
5	2	10	1.81	2.85
-7	3	10	2.33	1.97
-6	3	10	1.16	0.86
-4	3	10	2.11	-1.79
-3	3	10	3.80	-2.98
-2	3	10	7.84	7.17
0	3	10	2.79	2.19
1	3	10	3.56	3.87
2	3	10	2.99	-2.96
3	3	10	3.89	-2.99
-10	0	11	1.26	2.11
-8	0	11	1.87	-1.94
-2	0	11	3.42	2.98
0	0	11	2.58	-1.02
2	0	11	1.45	-1.17
4	0	11	2.80	3.02
6	0	11	5.24	5.67
-7	1	11	1.49	2.13
-6	1	11	2.10	-2.59
-4	1	11	2.84	2.28
-3	1	11	4.52	4.15
0	1	11	2.79	3.25
1	1	11	2.36	2.31
4	1	11	2.60	-2.39
5	1	11	2.08	1.38
-9	2	11	2.22	1.96
-8	2	11	1.41	0.85
-6	2	11	3.51	-2.95
-4	2	11	3.94	3.50
-2	2	11	6.86	6.22
-1	2	11	4.82	4.56
0	2	11	5.32	-5.34
2	2	11	1.84	1.90
3	2	11	1.72	-1.25
4	2	11	2.79	2.02
-7	3	11	2.10	3.33
-5	3	11	1.27	1.60
-4	3	11	3.82	-3.84
-3	3	11	4.50	-3.65
-2	3	11	3.71	3.85
-1	3	11	1.99	1.98
0	3	11	3.71	4.54
1	3	11	3.17	3.36
2	3	11	2.63	-4.58
-8	0	12	2.19	-2.91
-4	0	12	4.24	5.64
0	0	12	1.64	0.90
4	0	12	1.79	-0.90
-9	1	12	1.84	2.72
-8	1	12	1.61	1.27
-7	1	12	2.64	1.81
-6	1	12	3.32	-2.52
-5	1	12	3.59	-2.19
-4	1	12	1.37	0.32
-3	1	12	1.61	1.90
-1	1	12	6.85	6.94
0	1	12	1.12	-0.94
2	1	12	1.27	0.89
3	1	12	3.02	-3.29
4	1	12	1.03	-0.50
-7	2	12	1.72	1.99
-6	2	12	1.76	-1.57
-4	2	12	2.83	2.91
-3	2	12	5.97	-5.31
-2	2	12	3.17	2.97
-1	2	12	1.37	1.50
0	2	12	1.77	-0.95
1	2	12	2.65	2.68
3	2	12	2.68	-2.67
4	2	12	1.75	2.17
-6	3	12	2.10	-2.56
-5	3	12	2.60	2.81
-4	3	12	1.71	-1.91
-3	3	12	3.89	-3.06
0	3	12	1.41	2.10
1	3	12	2.38	2.40
2	3	12	2.06	-2.56
-8	0	13	1.14	-0.43
-4	0	13	1.46	1.73
-2	0	13	4.03	-3.49
0	0	13	1.64	1.12
4	0	13	2.38	-3.82
-8	1	13	1.34	1.03
-7	1	13	2.63	2.52
-6	1	13	2.95	-2.55
-5	1	13	5.47	-5.69
-4	1	13	2.44	1.86
-1	1	13	2.79	2.94
0	1	13	1.20	-0.84
2	1	13	2.11	1.17
3	1	13	2.17	-2.63
-7	2	13	2.56	2.62
-3	2	13	5.05	-4.39
0	2	13	1.69	-0.84
1	2	13	3.53	4.33
2	2	13	1.29	1.23
-2	3	13	0.99	-1.43
-1	3	13	1.42	-1.85
-6	0	14	1.75	2.57
-4	0	14	1.72	-2.66
-2	0	14	1.68	-2.21
2	0	14	2.18	3.67
-5	1	14	3.41	-4.03
-4	1	14	0.99	0.93
-1	1	14	1.85	1.58
0	1	14	3.33	-3.58
1	1	14	2.54	-2.90
-4	2	14	2.59	-3.20
-3	2	14	1.23	-1.23
-2	0	15	3.99	-2.89
-3	1	15	1.71	-3.42
-2	1	15	1.31	1.64

Table 4. *Observed and calculated structure factors* (*modification B*)

H	K	L	F(OBS)	F(CAL)
2	0	0	50.18	47.71
4	0	0	33.52	-26.86
6	0	0	73.92	-69.57
8	0	0	56.70	-60.23
12	0	0	11.46	-11.73
14	0	0	9.50	10.29
1	1	0	38.37	-39.00
3	1	0	137.19	-131.13
7	1	0	5.44	4.90
9	1	0	24.61	26.81
11	1	0	4.83	4.33
0	2	0	50.83	47.71
2	2	0	60.92	-52.57
4	2	0	94.22	93.60
6	2	0	26.30	23.04
8	2	0	6.90	-11.52
10	2	0	17.92	-15.55
12	2	0	18.81	-15.81
14	2	0	12.74	9.08
1	3	0	4.22	4.59
3	3	0	84.78	-76.60
5	3	0	23.48	-20.50
7	3	0	42.88	-37.67
9	3	0	12.72	-13.55
13	3	0	7.41	9.94
15	3	0	10.80	11.81
0	4	0	33.56	-26.86
2	4	0	56.43	-61.42
8	4	0	8.79	10.36
10	4	0	5.52	-6.76
1	5	0	66.29	-59.92
3	5	0	20.31	-16.64
7	5	0	6.70	-3.05
11	5	0	10.73	10.61
0	6	0	73.58	-69.57
2	6	0	25.04	22.31
4	6	0	8.08	7.42
8	6	0	13.19	-9.27
12	6	0	12.80	14.46
1	7	0	12.84	-15.28
3	7	0	24.92	26.19
5	7	0	20.11	-20.80
7	7	0	23.86	-24.91
9	7	0	15.24	-15.53
11	7	0	6.35	8.29
13	7	0	10.55	9.26
15	7	0	5.64	5.56
0	8	0	55.32	-60.23
4	8	0	8.79	-7.80
6	8	0	17.29	17.07
8	8	0	12.93	13.19
10	8	0	13.09	13.14
14	8	0	7.26	-7.59
1	9	0	8.87	-9.51
3	9	0	19.03	21.23
5	9	0	7.10	-7.30
7	9	0	8.95	7.53
9	9	0	5.72	4.91
13	9	0	3.61	-3.60
2	10	0	14.12	16.07
4	10	0	10.31	-10.37
6	10	0	19.12	-19.69
10	10	0	12.18	12.37
12	10	0	4.85	4.82
1	11	0	13.68	14.67
3	11	0	10.57	9.68
5	11	0	11.46	-11.15
7	11	0	4.91	4.57
9	11	0	5.00	6.40
0	12	0	11.46	-11.73
8	12	0	15.30	14.86
12	12	0	3.10	-3.96
1	13	0	10.23	11.10
7	13	0	7.02	6.54
0	14	0	10.06	10.29
4	14	0	10.49	-8.37
6	14	0	5.19	-5.47
8	14	0	5.82	5.80
5	15	0	5.11	5.87
7	15	0	5.64	8.04
6	16	0	2.82	5.24
1	0	1	8.12	-9.23
2	0	1	56.94	-55.47
3	0	1	34.27	33.39
4	0	1	4.24	-0.63
5	0	1	18.49	16.13
6	0	1	23.25	22.12
7	0	1	8.64	-6.93
8	0	1	4.34	3.18
10	0	1	10.45	10.56
15	0	1	7.97	-7.52
0	1	1	7.93	9.23
1	1	1	21.45	24.05
2	1	1	5.42	-11.09
3	1	1	36.46	-38.70
4	1	1	47.38	-46.61
6	1	1	44.99	-47.21
7	1	1	13.47	14.62
8	1	1	16.05	-16.60
9	1	1	7.81	-6.52
10	1	1	12.15	11.19
12	1	1	18.55	22.29
13	1	1	8.50	-8.15
0	2	1	56.37	55.47
1	2	1	28.94	-42.86
2	2	1	25.06	-27.08
3	2	1	17.78	-16.60
4	2	1	11.89	-10.82
5	2	1	46.18	-40.19
7	2	1	12.03	15.16
8	2	1	13.51	-14.31
9	2	1	16.92	19.21
11	2	1	23.40	25.36
12	2	1	7.65	-8.67
15	2	1	10.82	-11.75
0	3	1	30.50	-33.39
1	3	1	36.53	36.47
2	3	1	16.96	17.33
3	3	1	26.58	-27.28
5	3	1	23.60	26.19
7	3	1	7.12	-6.75
8	3	1	10.67	-4.05
9	3	1	6.94	-3.32
10	3	1	10.39	-4.38
11	3	1	6.43	13.15
0	4	1	3.81	0.63
1	4	1	11.71	-12.67
2	4	1	5.22	-8.57
3	4	1	4.28	-3.66
4	4	1	4.61	-1.52
5	4	1	18.12	-19.92
7	4	1	18.24	15.04
8	4	1	10.11	-9.53
9	4	1	5.38	6.42
10	4	1	10.17	10.62
11	4	1	17.57	19.65
0	5	1	18.10	-16.13
1	5	1	6.86	-9.79
2	5	1	25.61	-20.40
3	5	1	11.32	-19.95
4	5	1	33.34	-36.00
5	5	1	25.81	28.22
6	5	1	10.82	9.46
7	5	1	17.76	-14.80
8	5	1	21.57	23.40
10	5	1	14.85	16.29
14	5	1	12.01	-12.05
15	5	1	10.86	9.51
0	6	1	22.10	-22.12
1	6	1	20.29	-21.66
3	6	1	6.92	-9.45
4	6	1	9.54	9.63
5	6	1	15.81	16.95
6	6	1	8.79	7.30
7	6	1	16.59	19.24
8	6	1	6.07	-3.95
0	7	1	8.30	6.93
1	7	1	14.79	-13.79
2	7	1	26.50	26.40
4	7	1	13.74	13.75
9	7	1	15.20	16.32
15	7	1	2.76	-3.39
0	8	1	4.69	-3.18
1	8	1	21.47	-21.05
3	8	1	16.25	-14.80
5	8	1	11.40	11.44
6	8	1	13.82	-14.52
7	8	1	10.84	10.21
9	8	1	11.93	12.93
13	8	1	4.50	-4.23
1	9	1	15.91	-13.91
2	9	1	10.63	11.40
3	9	1	21.98	27.42
4	9	1	20.82	23.82
5	9	1	8.26	-8.83
6	9	1	8.93	9.32
10	9	1	10.00	-10.31
12	9	1	9.13	-8.50
13	9	1	6.70	5.92
0	10	1	10.51	-10.56
2	10	1	5.48	6.28
4	10	1	8.44	9.77
7	10	1	9.60	-6.50
8	10	1	9.90	8.40
13	10	1	2.94	3.96
1	11	1	4.93	-4.64
2	11	1	5.72	6.24
4	11	1	16.09	19.34
5	11	1	14.18	-15.47
6	11	1	9.11	8.12
7	11	1	9.82	9.17
12	11	1	5.86	-5.01
1	12	1	14.77	14.86
3	12	1	5.01	5.11
4	12	1	4.97	-4.77
5	12	1	8.04	-10.46
6	12	1	7.87	-7.91
7	12	1	12.89	-12.34
8	12	1	7.20	6.39
9	12	1	5.28	-4.96
1	13	1	5.68	5.99
2	13	1	8.93	-10.10
3	13	1	4.83	4.49
1	14	1	11.26	10.28
2	14	1	6.43	4.83
3	14	1	10.94	10.19
5	14	1	4.16	4.92
10	14	1	2.37	-2.40
0	15	1	8.16	7.52
2	15	1	4.00	-4.08
3	15	1	8.68	-7.93
4	15	1	9.35	-9.35
7	15	1	3.90	0.55
4	16	1	4.91	-1.37
0	0	2	48.19	-51.90
1	0	2	45.41	42.61
2	0	2	49.25	-50.13
4	0	2	20.82	22.58
5	0	2	7.35	5.50
6	0	2	31.90	27.68
7	0	2	16.74	-17.49
8	0	2	5.58	3.92
9	0	2	18.69	-19.90
10	0	2	13.56	14.39
11	0	2	13.98	14.03
13	0	2	4.28	6.04
14	0	2	4.34	-5.95
0	1	2	42.15	42.61
1	1	2	32.77	34.39
2	1	2	27.70	26.28
3	1	2	41.52	44.98
4	1	2	25.39	-26.60
5	1	2	14.69	15.38
6	1	2	10.33	11.14
7	1	2	19.22	-22.45
8	1	2	29.34	-31.33
9	1	2	14.06	-13.24
10	1	2	9.33	8.70
11	1	2	8.18	8.18
0	2	2	49.31	-50.13
1	2	2	12.54	-10.69
2	2	2	36.69	-42.06
3	2	2	31.09	-34.08
4	2	2	5.01	5.41
5	2	2	13.53	13.49
6	2	2	3.75	3.94
7	2	2	9.72	10.16
8	2	2	12.44	11.87
10	2	2	28.92	27.41
11	2	2	5.01	5.11
13	2	2	9.44	-7.70
16	2	2	5.15	-4.39
1	3	2	28.35	31.01
2	3	2	28.47	-26.73
3	3	2	44.11	40.68
5	3	2	40.08	43.84
6	3	2	9.01	9.60
7	3	2	4.20	3.86
10	3	2	15.60	17.10
11	3	2	6.47	-7.15
13	3	2	16.54	-15.24
0	4	2	19.95	22.58
1	4	2	28.77	28.75
2	4	2	13.09	15.14
3	4	2	7.18	2.51
4	4	2	13.25	-12.76
5	4	2	10.04	-12.06
6	4	2	21.25	-24.16
7	4	2	17.71	-16.39
10	4	2	7.61	8.04
0	5	2	7.18	5.50
1	5	2	7.51	10.40
2	5	2	9.76	-11.23
3	5	2	4.65	5.31
4	5	2	11.59	11.12
5	5	2	13.31	14.37
6	5	2	17.90	-17.05
7	5	2	13.01	12.48
9	5	2	23.58	21.88
13	5	2	12.13	-10.97
0	6	2	30.68	27.68
1	6	2	40.71	-39.60
2	6	2	29.73	33.80
5	6	2	9.84	10.11
6	6	2	7.75	-10.25
7	6	2	14.04	14.96
9	6	2	10.78	11.39
10	6	2	5.01	-5.54
12	6	2	8.85	-6.84
0	7	2	17.09	-17.49
1	7	2	4.02	2.68
2	7	2	6.25	3.97
3	7	2	4.20	4.58
4	7	2	16.62	16.28
6	7	2	4.69	-5.07
8	7	2	8.60	8.92
10	7	2	12.78	-11.15
11	7	2	13.74	-11.93
0	8	2	4.99	3.92
1	8	2	7.51	4.72
3	8	2	9.33	9.11
5	8	2	16.62	-17.76
6	8	2	6.27	5.57
7	8	2	7.59	-9.41
10	8	2	10.43	-11.02
12	8	2	5.80	-5.37
0	9	2	19.24	-19.90
1	9	2	4.63	-6.68
2	9	2	6.62	-6.23
4	9	2	4.83	-2.59
7	9	2	14.18	12.22
8	9	2	17.25	15.42
11	9	2	8.46	-7.60
0	10	2	13.76	14.39
3	10	2	20.35	19.74
5	10	2	7.08	7.77
6	10	2	5.01	-5.23
8	10	2	6.82	-6.10
10	10	2	12.82	-11.98
11	10	2	5.40	-5.97
0	11	2	15.54	14.03
1	11	2	13.23	-12.07
2	11	2	6.47	5.21
4	11	2	6.45	-7.07
6	11	2	10.57	-9.68
8	11	2	9.07	-8.82
9	11	2	7.33	-6.08
11	11	2	3.29	2.63
1	12	2	4.97	-3.80
2	12	2	4.06	4.29
3	12	2	4.93	-4.80
4	12	2	10.51	10.95
5	12	2	2.76	2.81
6	12	2	3.77	3.70
7	12	2	6.23	6.74
9	12	2	5.28	4.46
10	12	2	5.58	-6.04
0	13	2	4.77	6.04
1	13	2	3.88	-2.92
6	13	2	5.38	3.38
9	13	2	6.07	-7.25
0	14	2	4.34	-5.95
3	14	2	4.18	-5.60
7	14	2	5.52	-5.02
1	0	3	3.94	1.35
2	0	3	23.72	-21.29
4	0	3	21.53	20.00
6	0	3	7.04	4.82
7	0	3	6.31	7.00
8	0	3	13.45	-15.13
10	0	3	7.08	7.76
11	0	3	11.75	-11.41
14	0	3	4.18	3.30
0	1	3	3.94	-1.35
1	1	3	19.40	-18.31
2	1	3	7.63	9.57
3	1	3	9.50	9.43
4	1	3	7.81	-8.62
5	1	3	22.61	23.02
6	1	3	26.16	28.00
7	1	3	5.60	-5.25
8	1	3	16.05	17.19
9	1	3	8.71	7.73
10	1	3	4.53	4.51
14	1	3	9.33	-8.18
0	2	3	23.32	21.29
1	2	3	21.04	21.97
2	2	3	6.45	6.64
3	2	3	16.21	16.78
4	2	3	22.89	22.57
5	2	3	9.50	-10.76
6	2	3	21.81	-24.00
8	2	3	5.17	-6.19
9	2	3	19.72	-22.55
11	2	3	7.00	-7.53
1	3	3	18.67	-19.61
2	3	3	22.42	24.88
3	3	3	7.81	-4.83
4	3	3	11.51	12.53
5	3	3	15.22	-16.24
6	3	3	9.35	11.96
7	3	3	8.54	-5.95
8	3	3	13.27	-14.58
9	3	3	16.46	16.90
10	3	3	13.39	-14.52
11	3	3	7.79	6.51
0	4	3	21.55	-20.00
1	4	3	10.63	13.65
2	4	3	20.19	18.14
3	4	3	9.92	9.86
4	4	3	28.73	30.83
5	4	3	5.15	4.99
7	4	3	13.66	12.97
8	4	3	12.91	12.05
9	4	3	18.71	-20.31
12	4	3	7.20	-5.55
1	5	3	21.04	21.17
3	5	3	21.08	21.33
4	5	3	5.95	-5.90
5	5	3	17.37	-18.65
7	5	3	9.62	-9.23
8	5	3	6.07	-5.12
0	6	3	7.06	-4.82
1	6	3	24.27	28.35
2	6	3	7.81	-7.33
4	6	3	19.91	-22.74
5	6	3	10.33	-10.45
7	6	3	13.03	-15.12
8	6	3	16.98	17.33
9	6	3	15.42	-12.22
11	6	3	7.97	6.70
0	7	3	6.21	-7.00
3	7	3	4.57	4.32
7	7	3	16.96	14.34
11	7	3	7.61	-7.33
0	8	3	13.21	15.13
1	8	3	8.77	7.92
2	8	3	15.69	16.46
7	8	3	15.85	-14.74
10	8	3	7.79	-7.04
2	9	3	19.38	-23.72
3	9	3	12.15	-12.68
4	9	3	15.00	-14.67
5	9	3	11.77	-7.99
6	9	3	12.76	-11.36
10	9	3	7.22	7.14
0	10	3	7.91	-7.76
1	10	3	11.79	-12.11
2	10	3	11.22	-10.23
3	10	3	5.01	5.46
5	10	3	11.65	9.07
6	10	3	14.22	12.33
9	10	3	6.59	6.77
11	10	3	6.70	-6.01
0	11	3	13.66	11.41
1	11	3	6.11	4.56
2	11	3	9.92	-7.40
5	11	3	10.11	7.99
6	11	3	12.20	-10.63
9	11	3	7.37	-8.56
10	11	3	7.73	8.15
1	12	3	19.99	-16.40
2	12	3	7.49	-5.22
6	12	3	6.64	-3.68
8	12	3	5.58	-4.44
9	12	3	8.02	10.35
4	13	3	8.66	7.65
6	13	3	4.42	-4.08
8	13	3	3.35	-3.19
0	14	3	4.69	-3.30
1	14	3	7.63	-5.41
4	14	3	5.93	5.36
7	14	3	2.66	4.56
0	0	4	15.77	14.82
1	0	4	4.36	4.23
2	0	4	18.36	22.52
3	0	4	7.65	4.34
4	0	4	8.54	-7.59
5	0	4	4.77	2.62
6	0	4	6.55	-1.58
7	0	4	12.52	15.71
8	0	4	48.42	-7.91
9	0	4	6.37	6.39
10	0	4	11.73	-10.82
11	0	4	5.22	-6.28
13	0	4	4.06	-5.44
14	0	4	2.70	-3.67
0	1	4	4.14	4.23
2	1	4	15.95	14.58
3	1	4	8.56	-11.66
5	1	4	15.89	-15.40
6	1	4	9.05	9.36
8	1	4	11.22	-11.23
9	1	4	8.73	-7.10
10	1	4	13.94	-16.01
11	1	4	6.86	7.03
12	1	4	4.75	-2.73
14	1	4	4.38	4.81
0	2	4	18.63	22.52
1	2	4	5.60	-4.08
2	2	4	17.19	19.02
3	2	4	4.24	-3.18
4	2	4	5.28	-4.85
7	2	4	18.55	20.62
8	2	4	9.11	-11.36
10	2	4	6.51	-5.28
1	3	4	10.00	-10.19
2	3	4	4.89	-0.85
3	3	4	24.45	-24.74
4	3	4	5.50	-4.58
5	3	4	7.12	-6.40
6	3	4	15.83	17.16
8	3	4	4.55	3.70
10	3	4	4.40	-4.79
14	3	4	3.79	-3.93
0	4	4	8.42	-7.59
1	4	4	19.87	22.45
2	4	4	10.55	-11.78
4	4	4	6.43	6.89
5	4	4	9.01	-6.86
6	4	4	8.81	7.15
8	4	4	5.58	-5.61
9	4	4	22.52	-17.42
11	4	4	6.35	4.52
13	4	4	4.63	4.24
3	5	4	10.47	-9.90
5	5	4	5.36	-5.71
7	5	4	21.61	-18.36
8	5	4	9.64	-7.19
12	5	4	5.80	4.86
13	5	4	5.28	4.98
2	6	4	16.64	-17.74
4	6	4	7.63	7.93
5	6	4	17.98	16.24
9	6	4	10.13	-7.73
10	6	4	9.46	6.56
0	7	4	12.74	15.71
1	7	4	15.48	-16.03
2	7	4	5.42	-4.40
4	7	4	5.54	-4.95
5	7	4	5.58	3.69
6	7	4	9.66	-8.79
7	7	4	11.02	-9.04
8	7	4	8.73	-6.01
9	7	4	7.16	5.33
0	8	4	5.52	-7.91
1	8	4	11.53	-12.08
2	8	4	8.48	-7.07
3	8	4	12.89	-11.56
4	8	4	9.13	7.83
6	8	4	10.51	-8.18
9	8	4	7.75	6.29
10	8	4	6.94	5.72
11	8	4	4.63	0.77
0	9	4	7.20	6.39
3	9	4	7.18	7.56
4	9	4	11.04	9.60
9	9	4	6.09	4.18
0	10	4	11.44	-10.82
1	10	4	12.66	-11.17
2	10	4	7.04	-5.08
3	10	4	6.96	-4.92
5	10	4	6.66	-4.54
7	10	4	8.91	-7.81
8	10	4	9.19	9.35
9	10	4	9.64	9.53
10	10	4	3.81	3.12
0	11	4	9.05	-6.28
2	11	4	10.31	-8.05
3	11	4	7.16	5.88
4	11	4	9.84	8.97
1	12	4	4.75	-5.03
2	12	4	4.67	5.98
3	12	4	4.55	4.78
4	12	4	4.36	-5.44
0	13	4	5.32	-5.44
1	13	4	6.66	5.26
2	13	4	7.97	-5.97
0	14	4	4.06	-3.67
3	14	4	3.79	5.06
3	0	5	17.17	-17.54
4	0	5	7.02	-6.87
5	0	5	4.97	-8.44
6	0	5	8.04	10.37
7	0	5	3.94	3.29
8	0	5	10.04	8.31
11	0	5	6.76	6.52
12	0	5	4.04	-4.05
1	1	5	19.83	19.78
2	1	5	8.42	-6.60
3	1	5	9.03	9.23
4	1	5	15.69	-15.85
6	1	5	8.95	9.26
8	1	5	5.15	-3.13
9	1	5	9.29	-8.75
10	1	5	8.89	8.21
1	2	5	15.14	17.43
2	2	5	22.36	-22.54
4	2	5	15.77	-15.84
8	2	5	5.42	4.83
0	3	5	16.62	17.54
1	3	5	13.92	-17.71
2	3	5	10.53	-12.18
5	3	5	7.39	7.41
8	3	5	6.76	-7.38
1	4	5	19.99	18.32
2	4	5	6.35	6.73
3	4	5	14.87	15.10
1	5	5	21.67	-20.13
2	5	5	5.36	-5.27
2	6	5	6.25	7.99
1	7	5	6.19	-5.36
1	8	5	15.77	12.56
1	9	5	6.27	1.44
2	9	5	9.96	8.77
1	10	5	6.27	4.42
2	10	5	4.22	5.40
1	11	5	7.77	6.86
3	11	5	8.46	2.28
1	12	5	7.87	-7.95
0	0	6	8.18	-8.36
2	0	6	4.67	-5.47
3	0	6	4.61	4.09
7	0	6	3.86	-3.11
8	0	6	6.68	5.96
10	0	6	3.17	2.90
0	2	6	3.90	-5.47
5	2	6	7.12	-7.41
9	2	6	3.37	-5.27
10	2	6	3.55	-1.71
3	3	6	14.14	12.81
2	4	6	3.02	-5.57
3	5	6	12.44	-8.90
2	6	6	3.37	2.90
3	6	6	9.29	-7.99
2	7	6	4.04	4.96
2	9	6	4.57	6.31
2	0	7	15.22	11.02
1	1	7	15.99	-11.54
2	1	7	16.70	2.29
0	2	7	14.53	-11.02
1	2	7	7.08	8.06
2	2	7	14.24	12.99
3	2	7	2.48	2.92
4	2	7	7.95	7.67
5	2	7	3.19	4.48
1	3	7	9.44	6.61
2	3	7	3.49	2.34
2	4	7	2.27	2.48
1	5	7	7.95	6.91

tribution of the electron density is somewhat concentrated about the three positions so that at $C(4)_T$, $\varrho=0{\cdot}89$, at $C(4)_{GI}$ $\varrho=0{\cdot}75$ and at $C(4)_{GII}$ $\varrho=0{\cdot}44$ e.Å^{-3}, as compared with the mean background level in the circular belt, $\bar{\rho}=0{\cdot}3$ e.Å^{-3}. Inference is that the rotation is not completely free but rather restricted around the three positions. The existence of diffuse scattering previously mentioned suggests that the distribution of C(4) over the equilibrium positions is not completely random. In addition, the variation of relative intensities of this diffuse scattering from specimen to specimen seems to indicate that the distribution is not necessarily the same for all crystals. In the present analysis, the site occupancy factors and other atomic parameters of C(4) were determined merely by mathematical treatment assuming that the C(4) atoms were only distributed among three positions with the usual ellipsoidal electron density distribution. However, the difference electron density map and the least-squares treatment gave a rough idea of the possibilities that the three stable positions of C_γ – *trans*, *gauche* I (*trans* to H_α) and *gauche* II (*trans* to the carboxyl group) – are occupied by C(4). The estimated relative possibilities are as follows:

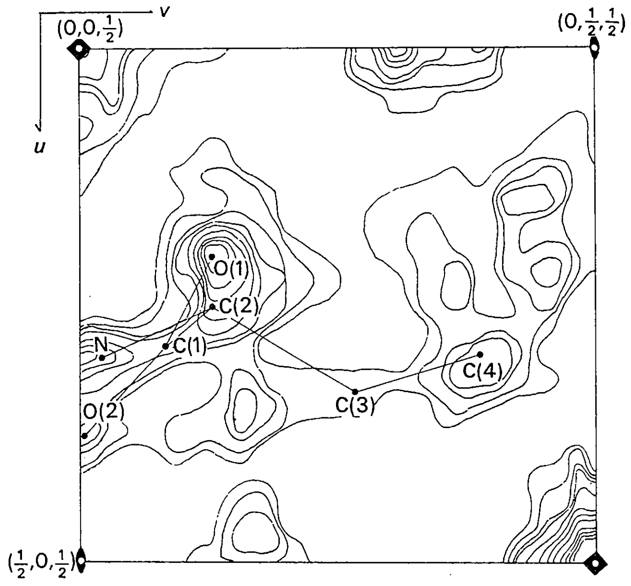

Fig. 4. The Harker section at $w=\frac{1}{2}$ of modification *B*. The image of the molecule drawn by the final atomic coordinates is superposed on the map.

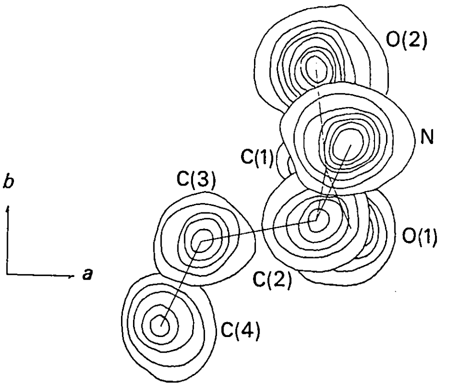

Fig. 5. Composite electron density map of the molecule viewed along the *c* axis of modification *B*. Contours are drawn at 1,2,3,... e.Å^{-3}.

Site	Relative peak height in difference electron density map (normalized to 100%)	Site occupancy factor in least-squares calculation (normalized to 100%)
trans	42·8%	40·0%
gauche I	36·0%	36·4%
gauche II	21·2%	23·6%

Some of the salient conformational features of amino acids in the solid state have been reviewed by Lakshminarayanan, Sasisekharan & Ramachandran (1967). They showed that the location of the γ-carbon atom is restricted to the neighbourhoods around the three positions where the torsion angle (NH_2–C_α–C_β–C_γ) is 60° (*G*I in Fig. 7, *trans* to H_α), 180° (*T* in Fig. 7, *trans* to NH_2) or 300° (*G*II in Fig. 7, *trans* to carboxyl), and that the numbers of cases in which these positions occur for various amino acids are found to be 13, 10 and 21, respectively, suggesting that the *G*II position is the most favourable.

The bond lengths, angles and the torsion angles involving the C_β–C_γ bonds are also presented in Tables 5, 6 and 7. The C(3)–C(4) (C_β–C_γ) bond lengths appear to be too short. This may be attributed mainly to experimental errors but partly to the libration effect (Cruickshank, 1956). Other bond lengths and angles are normal as compared with the ample data presented

Table 5. *Bond lengths*

	A modification	*B* modification	L-Alanine	DL-Alanine	α-Amino-isobutyric acid
O(1)–C(1)	1·25 ± 0·009 Å	1·28 ± 0·009 Å	1·256 ± 0·003 Å	1·27 ± 0·007 Å	1·20 Å
O(2)–C(1)	1·25 ± 0·009	1·24 ± 0·009	1·247	1·21	1·26
N——C(2)	1·51 ± 0·01	1·50 ± 0·009	1·491	1·50	1·49
C(1)–C(2)	1·51 ± 0·01	1·51 ± 0·01	1·525	1·54	1·52
C(2)–C(3)	1·56 ± 0·02	1·56 ± 0·01	1·525	1·51	1·53
$C(3)–C(4)_T$	1·42 ± 0·04	1·53 ± 0·01			1·54
$C(3)–C(4)_{GI}$	1·08 ± 0·04				
$C(3)–C(4)_{GII}$	1·30 ± 0·07				

Table 6. *Bond angles*

	A modification	*B* modification	L-Alanine	DL-Alanine	α-Amino-isobutyric acid
C(2)—C(1)-O(1)	116·3 ± 0·5°	116·9 ± 0·4°	116·1°	113·2°	117·7°
C(2)—C(1)-O(2)	117·9 ± 0·5	119·5 ± 0·4	118·3	121·3	117·9
O(1)—C(1)-O(2)	125·7 ± 0·5	123·6 ± 0·5	125·6	125·4	125·3
C(3)—C(2)-C(1)	110·8 ± 0·7	111·8 ± 0·5	111·5	111·2	114·0
					108·7
N—C(2)-C(1)	109·9 ± 0·6	109·6 ± 0·4	110·3	108·3	108·8
C(3)—C(2)-N	107·0 ± 0·7	107·4 ± 0·5	109·7	110·4	107·5
					104·9
C(4)$_T$—C(3)-C(2)	115 ± 2	112·8 ± 0·5		C(3)^1-C(2)-C(3)^2	112·2
C(4)$_{GI}$ -C(3)-C(2)	134 ± 2				
C(4)$_{GII}$-C(3)-C(2)	117 ± 3				

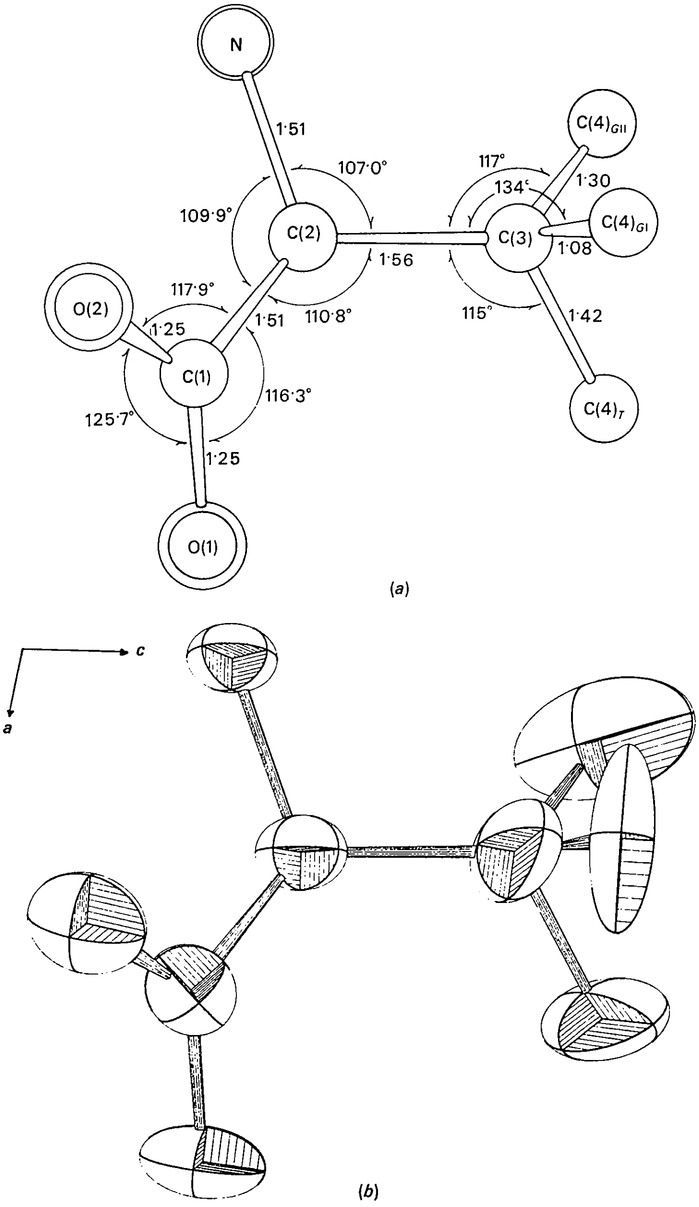

Fig. 6. The molecule found in the *A* modification. (*a*) Bond distances and angles. (*b*) A perspective view drawn by the *OR TEP* program (Johnson, 1965). Atoms are represented by ellipsoids of thermal vibrations, indicating the atoms lie within the ellipsoids with 50% probability.

for the amino acids. In Tables 5 and 6, the values found in aminoisobutyric acid (Hirokawa, Kuribayashi & Nitta, 1952), DL-alanine (Donohue, 1950) and L-alanine (Simpson & Marsh, 1966) are listed for comparison.

The carboxyl group is strictly planar within the limits of experimental error. The perpendicular dis-

Table 7. *Torsion angles for* L-*molecules*

	A modification	*B* modification
C(3)——C(2)-C(1)-O(2)	97·3°	87·7°
N———C(2)-C(1)-O(2)	−20·7	−31·3
$C(4)_T$—C(3)-C(2)-C(1)	65·7	63·8
$C(4)_T$—C(3)-C(2)-N	−174·6	−176·0
$C(4)_{GI}$ -C(3)-C(2)-C(1)	−47·3	
$C(4)_{GI}$ -C(3)-C(2)-N	72·4	
$C(4)_{GII}$-C(3)-C(2)-C(1)	−173·5	
$C(4)_{GII}$-C(3)-C(2)-N	−53·8	

Table 8. *The perpendicular distances from the least-squares plane through the carboxyl group* [O(1), O(2), C(1) *and* C(2)]

	A modification	*B* modification
O(1)	0·006 Å	0·004 Å
O(2)	0·006	0·004
C(1)	−0·017	−0·012
C(2)	0·005	0·003
N	−0·450	−0·701
C(3)	1·457	1·461

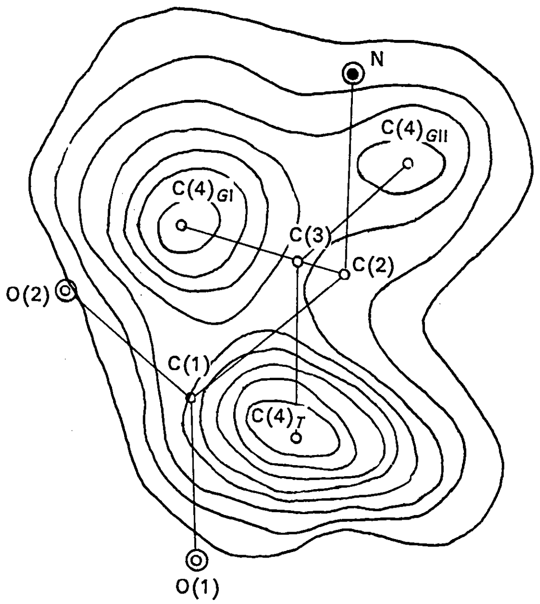

Fig. 7. The section of the difference electron density map cut through the three maximum positions of C_γ. All the atoms except C_γ and hydrogen are subtracted. Contours are drawn at 0·1, 0·2, 0·3, . . . e.Å^{-3}.

Table 9. *Hydrogen bond lengths and angles*

	N···O distances		C(2)-N···O angles	
	A modification	*B* modification	*A* modification	*B* modification
C(2)-N···O(1)	2·84 Å	2·83 Å	115·1°	104·9°
C(2)-N···O(1)	2·80	2·81	103·1	106·0
C(2)-N···O(2)	2·79	2·91	103·0	111·8

Table 10. *R.M.S. displacements and directions of principal axes of thermal vibrations** (*modification A*)

	$U(j)\times10^2$	α_{1j}	α_{2j}	α_{3j}		$U(j)\times10^2$	α_{1j}	α_{2j}	α_{3j}
O(1)	15 Å	5°	86°	91°	$C(4)_T$	16 Å	47°	43°	85°
	22	90	98	172		25	43	131	102
	29	95	9	98		55	84	101	13
O(2)	16	110	77	25	$C(4)_{GI}$	13	98	15	103
	20	160	94	110		38	91	103	167
	25	90	14	104		41	8	83	92
N	15	16	79	101	$C(4)_{GII}$	20	109	48	48
	21	74	140	54		28	153	117	89
	22	88	52	38		54	108	54	138
C(1)	17	101	109	22					
	19	37	127	92					
	22	55	43	68					
C(2)	18	12	80	97					
	20	89	133	137					
	24	102	44	132					
C(3)	18	92	4	86					
	23	168	92	78					
	54	78	93	12					

* The r.m.s displacement $U(j)$ is directed along the jth axes of the ellipsoid where α_{1j}, α_{2j} and α_{3j} are the angles between the jth axis and the a, c^* and b axes respectively.

tances from the least-squares plane through the four atoms of this group are shown in Table 8. The nitrogen atom is 0·45 Å off the plane, the torsion angle about the C(1)–C(2) bond being 20·7°. The molecule exists as a zwitterion and the nitrogen atom forms three hydrogen bonds to the ionized carboxyl oxygen atoms of neighbouring molecules. The arrangement of these hydrogen bonds around the nitrogen atom is nearly in tetrahedral directions as shown in Table 9.

The magnitudes and angles which define the principal axes of the ellipsoids of atomic thermal vibrations are given in Table 10, where, α_{ij} are the angles between the jth principal axis and the axes fixed to the unit cell. In Fig. 6 is shown a perspective drawing of the molecule viewed along the b axis, in which the atoms are represented by ellipsoids of thermal vibration.

Two views of the crystal structure of the A modification of DL-α-amino-n-butyric acid are shown in Figs. 8 and 9. As seen in these Figures, the molecules form a double layer parallel to the (001) plane through the N–H···O hydrogen bonds mentioned above. Single

Table 11. *Intermolecular distances between* C_γ *atoms (modification* A*)*

Molecule I(0, 0, 0)	to	of molecule	Distance
$C(4)_T$	$C(4)_T$	III(0, 0, 0)	2·77 Å
	$C(4)_{GI}$	III(0, 0, 0)	3·76
	$C(4)_{GI}$	I(0, 1, 0)	3·86
	$C(4)_{GI}$	II(−1, 0, 0)	3·80
	$C(4)_{GII}$	II(−1, −1, 0)	3·70
$C(4)_{GI}$	$C(4)_{GII}$	II(−1, −1, 0)	3·19
	$C(4)_{GII}$	I(0, −1, 0)	3·31
$C(4)_{GII}$	$C(4)_T$	IV(0, 1, 0)	3·16
	$C(4)_{GII}$	II(−1, −1, 0)	3·91

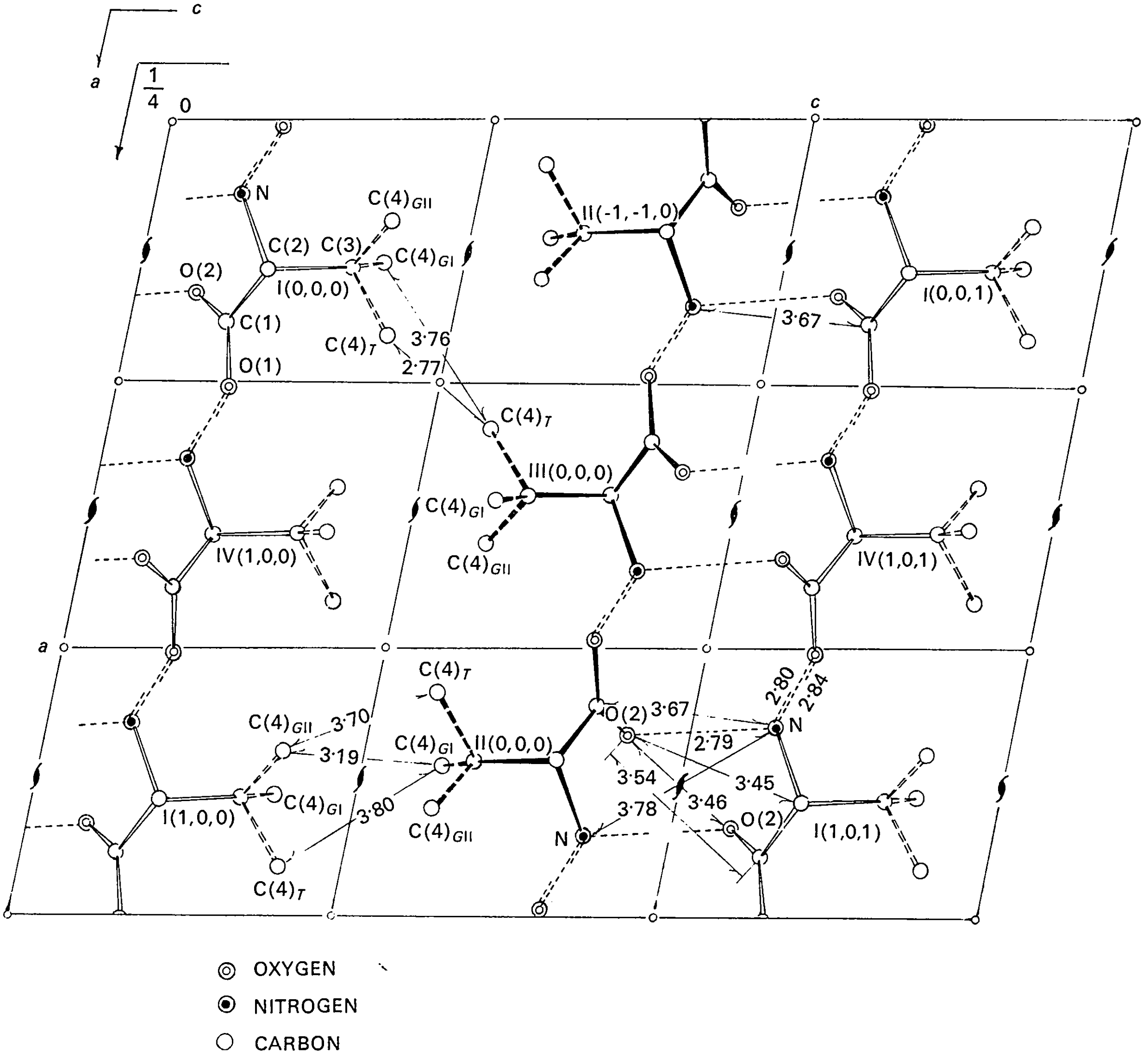

Fig. 8. Projection of the crystal structure of the A modification along the b axis. Black and white molecules are antipodes. Intermolecular short distances less than 3·8 Å are shown. Broken lines indicate hydrogen bonds.

layers are formed by two of the three kinds of hydrogen bond and they are joined together by the hydrogen bonds of the third kind to form a double layer. A similar feature of the structure has been found in DL-norleucine (Mathieson, 1953) and DL-methionine (Mathieson, 1952). The lengths of the hydrogen bonds are, as shown in Figs. 8 and 9, 2·80, 2·84 and 2·79 Å, which are normal values for N–H···O bond lengths. It is also seen from the Figures that the double layers are separated from each other by hydrocarbon side chains. The side-chain carbon atoms, C(3) and C(4) (β- and γ-carbon), protrude from both sides of the double layer and fill up the gap between the double layers. The crystal structure is, therefore, built by the alternate stacking of the hydrophylic and hydrophobic regions.

Intermolecular short contacts whose distances are less than 3·8 Å are shown in Figs. 8 and 9. In these

Table 12. *R.M.S. displacements and directions of principal axes of thermal vibrations** (*modification B*)

	$U(j)\times10^2$	α_{1j}	α_{2j}	α_{3j}
O(1)	17 Å	69°	24°	79°
	20	159	70	85
	26	89	103	13
O(2)	16	52	142	93
	21	140	125	108
	25	102	103	18
N	16	90	15	105
	19	154	97	115
	21	116	76	30
C(1)	13	48	136	79
	18	79	94	168
	20	44	47	85
C(2)	12 Å	95°	9°	98°
	19	136	99	132
	21	133	87	44
C(3)	18	94	58	33
	20	141	124	73
	25	51	129	63
C(4)	19	116	108	32
	23	108	147	117
	26	32	117	74

* The r.m.s. displacement $U(j)$ is directed along the jth axes of the ellipsoid, where α_{1j}, α_{2j} and α_{3j} are the angles between the jth axis and the a, c^* and b axes respectively.

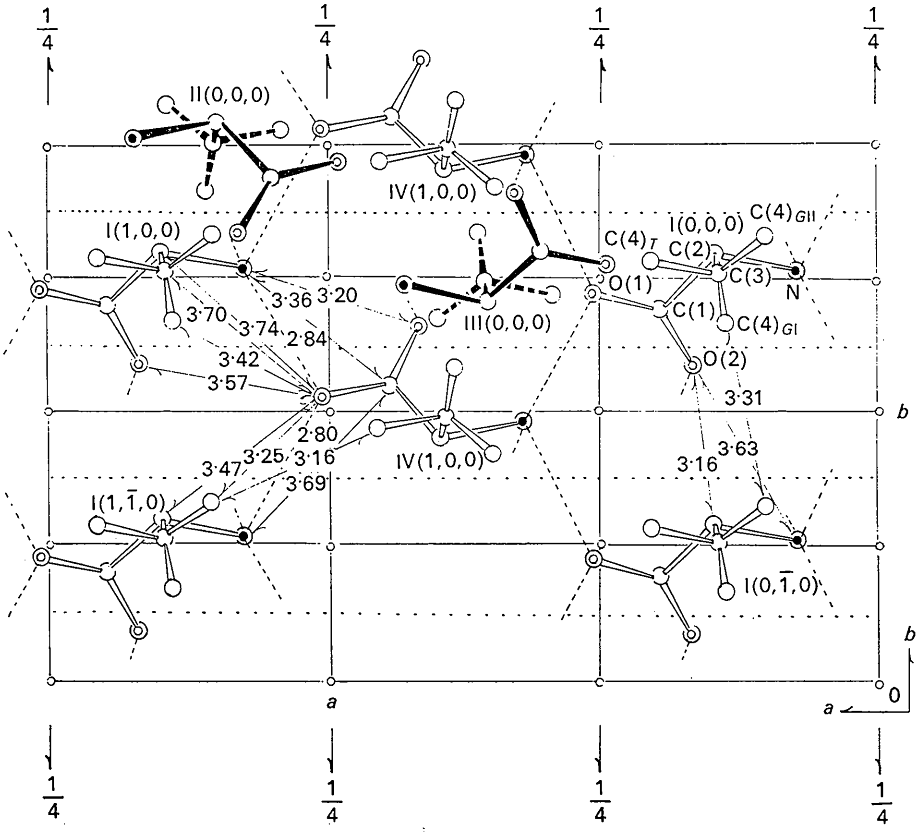

Fig. 9. Projection of the crystal structure of the *A* modification along the *c* axis.

Figures, the distances involving the three positions of C(4) are also shown. It is to be noted that some very short distances exist between the various sites of C(4). Those less than 4 Å are shown in Table 11. The distances from $C(4)_T$ of molecule I(0, 0,0) to $C(4)_T$ of molecule III(0, 0,0) is found to be only 2·77 Å. It is suggested that strong correlations should exist between the neighbouring molecules in distributing the C_γ atoms over their stable positions. This rules out the random arrangement of C_γ and an imperfectly ordered stacking of the molecular layers similar to that suggested in DL-norleucine (Mathieson, 1953) may result, which is responsible for the appearance of the diffuse scattering mentioned previously.

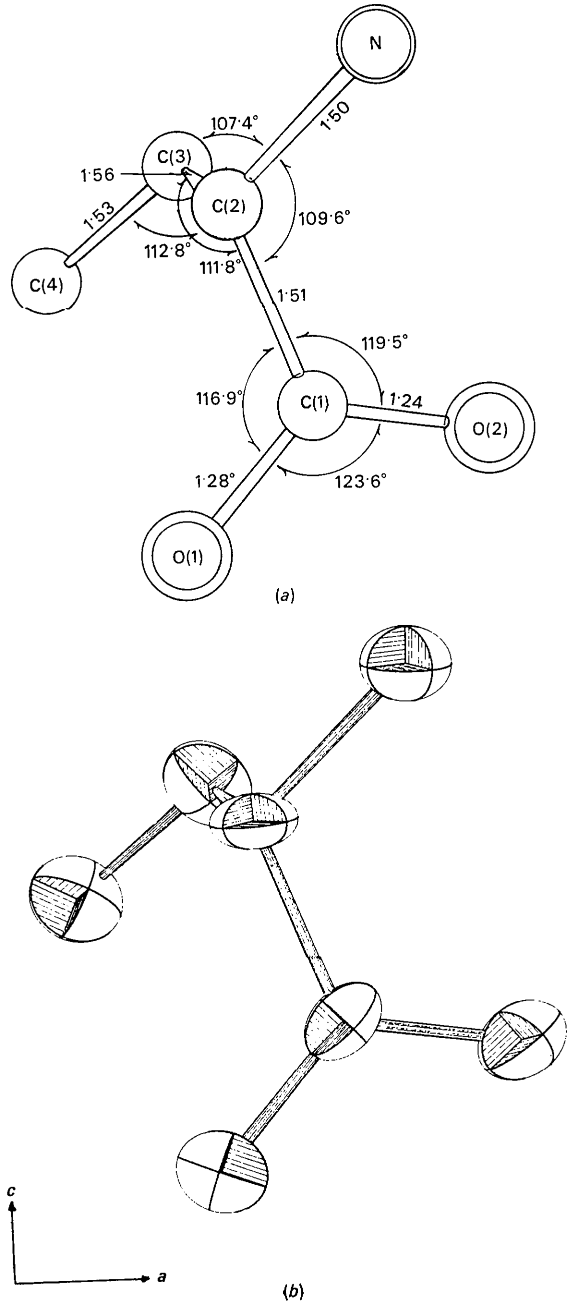

Fig. 10. The molecule found in the *B* modification. (*a*) Bond distances and angles. (*b*) A perspective view drawn by the *OR TEP* program (Johnson, 1965). Atoms are represented by ellipsoids of thermal vibrations, indicating that the atoms lie within the ellipsoids with 50% probability.

Modification B

The molecular dimensions determined for the crystals of the *B* modification, are shown in Fig. 10 and listed in Tables 5, 6 and 7. The standard deviations of these values were calculated from those for the atomic parameters listed in Table 2. As shown in Tables 5 and 6, the bond lengths and angles are in good agreement with those found in many amino acids and in excellent overall agreement with the values obtained for the *A* modification except those involving the γ-carbon atom. The most significant difference between the molecules of *A* and *B* is in the carboxyl C(1)–O(1) bond length and the angles involving O(1). In the *A* modification the two carboxyl C–O bonds are highly symmetric, whereas in the *B* modification one of the bonds is about 0·04 Å longer than the other or the two C–O bonds in the *A* modification. It has been presumed for some time that the lengthening of carboxyl C–O bonds occurs when the oxygen atom accepts strong hydrogen bonds. In the present case, however, O(1) accepts two hydrogen bonds of lengths 2·83 and 2·81 Å and O(2) accepts one of 2·91 Å, whereas in the *A* modification O(1) accepts two with 2·84 and 2·80 Å and O(2) accepts one with 2·79 Å, hence there seems to exist no appreciable difference in the strength of hydrogen bonds on O(1).

The carboxyl group is almost strictly coplanar; the deviations of atoms from the least-squares plane formed by the four atoms, O(1), O(2), C(1) and C(2), are shown in Table 8. The nitrogen atom lies 0·70 Å off the plane, the torsion angle, O(2)–C(1)–C(2)–N, being 31·3°. The γ-carbon atom occupies only the *trans* position to the amino nitrogen atom and the torsion angle, C(4)–C(3)–C(2)–N, is 176·0°.

The magnitudes and angles which define the directions of the principal axes of the ellipsoids of atomic thermal vibrations are listed in Table 12. A perspective drawing of the molecule is given in Fig. 10, showing ellipsoids of thermal vibrations.

The molecule exists as a zwitterion as in the *A* modification. The protonated nitrogen atom forms three hydrogen bonds nearly in tetrahedral directions to the ionized carboxyl oxygen atoms of neighbouring molecules. The C(2)–N · · · O angles are listed in Table 9.

Two projections of the crystal structure of the *B* modification along the *c* and the *a* axes are shown in Figs. 11 and 12, respectively. It is seen in these Figures that the hydrogen bonds of length 2·83 Å link the molecules head to tail along the *c* axis, as has been observed in the crystal structures of DL-alanine (Donohue, 1950) and L-alanine (Simpson & Marsh, 1966).

The second hydrogen bonds of 2·81 Å link the molecules to form columns which are joined together through the lateral or the third hydrogen bonds of 2·91 Å. The side chains of the molecules are extended from the columns and fill up the spaces between them. The structure of the column can be regarded as composed of two identical helices interwound with each other along the common axis. In Figs. 11 and 12, it is seen that the hydrogen bonds of 2·81 Å unite the molecules to form helical chains in the following ways:

–IV(0, 0, 0)–I(0, 0, 0)–VII(0, 0, 1)–VI(0, 0, 1)–

and

–VII(0, 0, 0)–VI(0, 0, 0)–IV(0, 0, 1)–I(0, 0, 1)– .

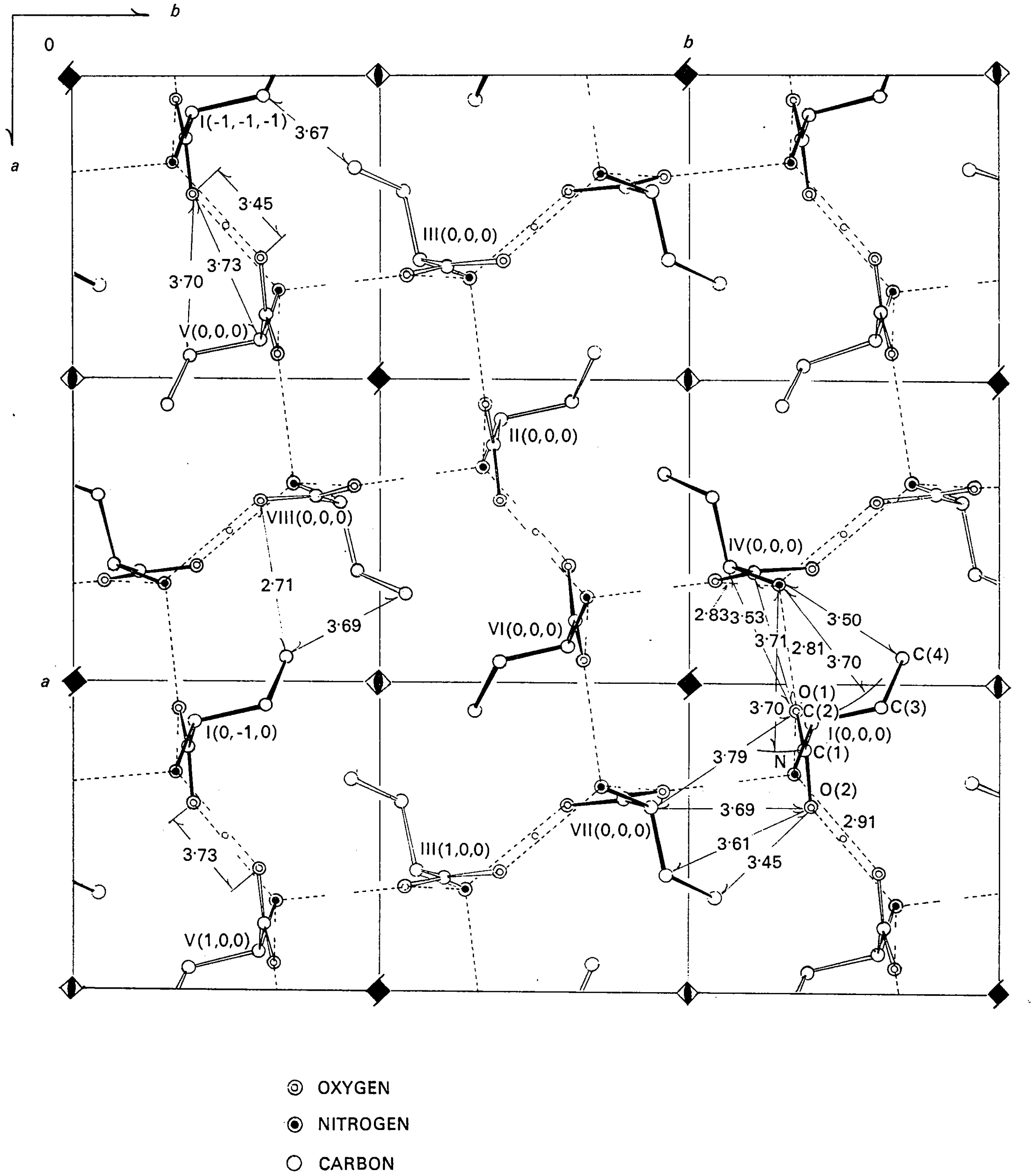

Fig. 11. Projection of the crystal structure of the *B* modification along the *c* axis. Black and white molecules are antipodes. Intermolecular short distances less than 3·8 Å are shown. Broken lines indicate hydrogen bonds.

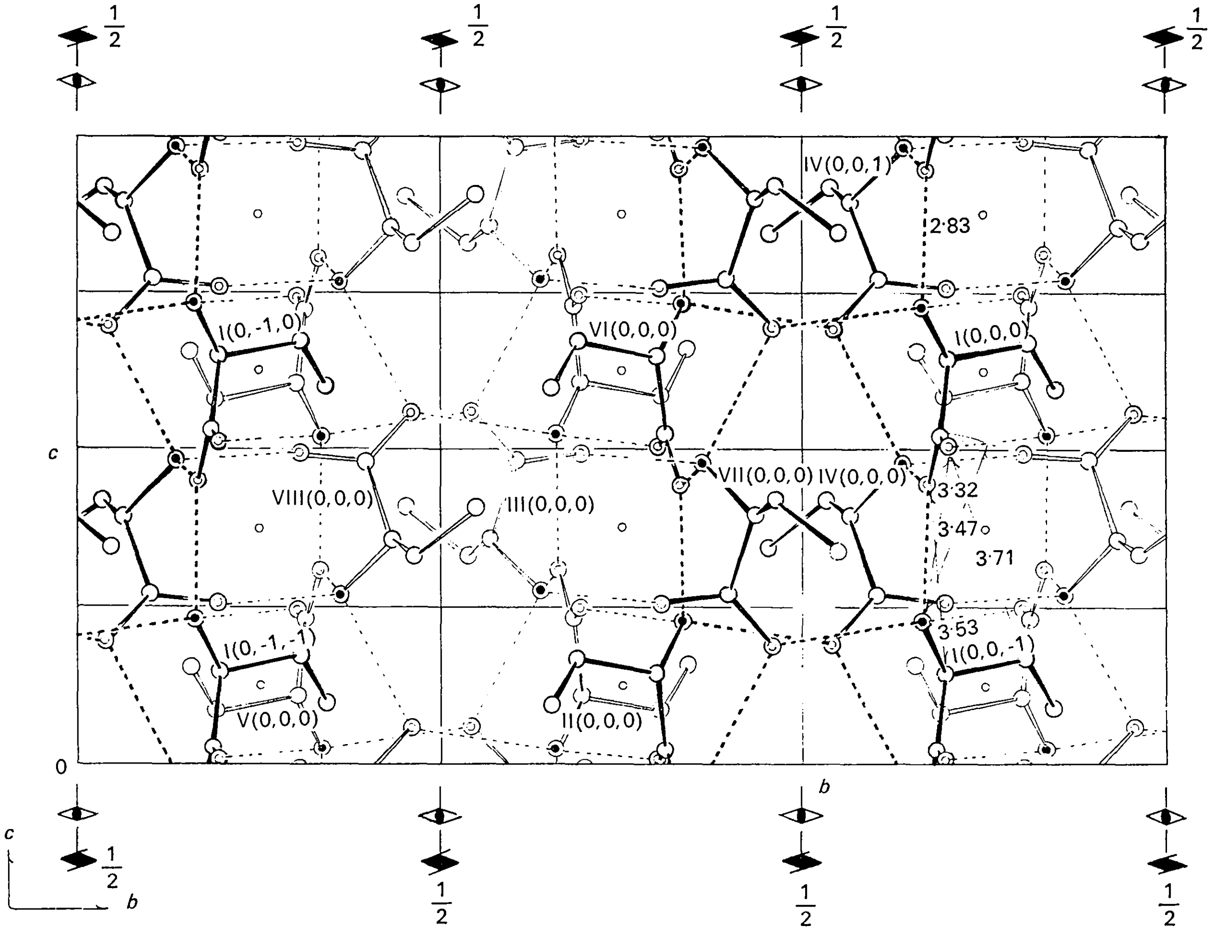

Fig. 12. Projection of the crystal structure of the *B* modification along the *a* axis.

These two helices, each having a helix parameter of pitch 2*c* and four molecules per turn, are interwound through the hydrogen bonds of 2·83 Å to form a square column. There are two kinds of enantiomorphic column contained in the crystal; the L-molecules form a left-handed helix and *vice versa*. It is to be noted that the packing of the side chains within the hydrophobic region adopts $\bar{4}$ symmetry which is different from the 4_2 screw symmetry of the column.

Intermolecular short contacts whose distances are less than 3·8 Å are shown in Figs. 11 and 12.

The authors wish to express their sincere gratitude to Professor M. Tsuboi for suggesting the problem and for his continued interest. This work was aided by a Research Grant from the Ministry of Education and the Hohansha Foundation.

References

Berghuis, J., Haanappel, IJ. M., Potters, M., Loopstra, B. O., MacGillavry, C. H. & Veenendaal, A. L. (1955). *Acta Cryst.* **8**, 478.

Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *ORFLS.* Oak Ridge National Laboratory, Oak Ridge, Tennessee, U.S.A.

Cruickshank, D. W. J. (1956). *Acta Cryst.* **9**, 754.

Donohue, J. (1950). *J. Amer. Chem. Soc.* **72**, 949.

Hirokawa, S., Kuribayashi, S. & Nitta, I. (1952). *Bull. Chem. Soc. Japan*, **25**, 192.

Ichikawa, T., Iitaka, Y. & Tsuboi, M. (1968). *Bull. Chem. Soc. Japan*, **41**, 1027

Johnson, C. K. (1965). *OR TEP: A Fortran Thermal-Ellipsoid Plot Program for Crystal Structure Illustrations.* Oak Ridge National Laboratory, Oak Ridge, Tennessee, U.S.A.

Lakshminarayanan, A. V., Sasisekharan, V. & Ramachandran, G. N. (1967). International Symposium on Conformation of Biopolymers. *A study of the conformation of amino acids.* Univ. of Madras, India.

Mathieson, A. McL. (1952). *Acta Cryst.* **5**, 332.

Mathieson, A. McL. (1953). *Acta Cryst.* **6**, 399.

Phillips, D. C. (1954). *Acta Cryst.* **7**, 746.

Simpson, H. J. & Marsh, R. E. (1966). *Acta Cryst.* **20**, 550.

Tsuboi, M., Iitaka, Y., Suzuki, S. & Mizushima, S. (1959). *Bull. Chem. Soc. Japan*, **32**, 529.

Wilson, A. J. C. (1942). *Nature, Lond.* **150**, 151.