graphic center of symmetry. It appears that it would be difficult to discover, at least from the present data, which, if either, of these situations exists. If the true symmetry were *Imm* or *I*222, one would expect the thermal ellipsoids (Table 3) to be, in most cases at least, prolate spheroids because of the splittings imposed by the extra symmetry. Although several of the atoms, notably O(2), O(3), and O(4), show appreciable elongation, the overall amount of anisotropy does not seem unusual and O(5) itself is not particularly distorted. There is also no significant directional trend. The r.m.s. displacements in the x,y,z directions are 0·26, 0·23, and 0·22 Å for framework O, and 0·17, 0·14, and 0·15 Å for framework M, respectively. In view of these negative inferences, no attempt was made to find a structure of lower symmetry. The possibility of random, essentially static, displacements of O(5) (accompanied, perhaps, by concerted displacements of the neighboring M(4) atoms) cannot be excluded. A displacement of 0·2 Å from the M–M line would produce an angle of 166°. Such a disorder would be essentially indistinguishable from ordinary thermal motion.

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The Crystal and Molecular Structure of Aminomalonic Acid

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Crystals of aminomalonic acid are orthorhombic ($P2_1cn$) with $a=4{\cdot}990$, $b=8{\cdot}507$ and $c=10{\cdot}854$ Å ($Z=4$). The structure was solved in projection by direct methods applying three-dimensional structure factors, and refined with 560 diffractometer data to a final R value of 0·029. As both enantiomers of the zwitterionic molecule are present in the structure, the crystals are racemic despite the non-centrosymmetry of the space group. A rigid three-dimensional network is formed by extensive hydrogen bonding.

Introduction

From investigations (*e.g.* Marsh, 1958; Iitaka, 1960, 1961; Pasternak, 1956) evidence has been adduced that amino acids and peptides are zwitterionic in the crystalline state. In aminodicarboxylic acids such as aspartic acid and glutamic acid the question arises which of the acid groups loses its proton. As far as we know, glutamic acid is the only aminodicarboxylic acid yet investigated (Hirokawa, 1955). In that analysis an indication of the configuration of the zwitterion was obtained from the dimensions of the carboxyl groups and from the two-dimensional difference Fourier syntheses.

In aminomalonic acid there are two equivalent carboxyl groups neighbouring the amino group, and this implies two possibilities: the molecule is non-zwitterionic ($HOOC{-}CHNH_2{-}COOH$) with two equivalent carboxyl groups, or zwitterionic ($HOOC{-}CHNH_3^+{-}COO^-$), in which case a molecule with an asymmetric carbon atom will result.

Experimental

Aminomalonic acid was synthesized from bromomalonic acid and ammonia. The literature mentions water of crystallization and melting points ranging from 109 to 136°C. Our crystals, which were obtained by slow evaporation of an aqueous solution at room temperature, did not contain water of crystallization and decomposed at 136°C.

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The crystals of aminomalonic acid are orthorhombic. The systematic extinctions are compatible only with the space groups $P2_1cn$ and $Pmcn$ (no. 33 and no. 62 respectively from *International Tables for X-ray Crystallography*, with a and c interchanged).

The unit-cell dimensions determined by least-squares (van den Berg and Rutten-Keulemans, 1963) from 41 reflexions with 2θ ranging from 77 to 145° using Cu $K\alpha$ radiation ($\lambda = 1{\cdot}5418$ Å) are $a = 4{\cdot}990 \pm 0{\cdot}002$, $b = 8{\cdot}507 \pm 0{\cdot}005$, $c = 10{\cdot}854 \pm 0{\cdot}003$ Å. With four molecules in the unit cell the calculated density was 1·72 g.cm^{-3} (experimental 1·74 g.cm^{-3}).

With a General Electric single-crystal orienter equipped with scintillation counter and discriminator the intensities of 560 independent reflexions out of a total of 584 were measured, using Ni-filtered Cu radiation. The data were corrected for non-linearity of the counter system and for Lorentz and polarization factors. A spherically ground crystal with a diameter of 0·2 mm was used; absorption corrections were not applied.

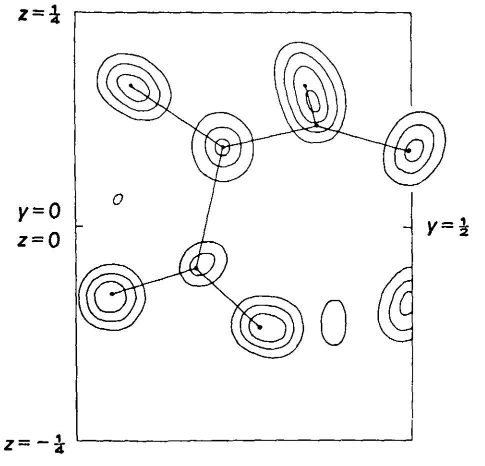

Fig. 1. Electron density projection on (100) with 45 independent structure factors, using signs determined by the $B_{3,0}$ formula. Contours on arbitrary scale, zero and first line omitted.

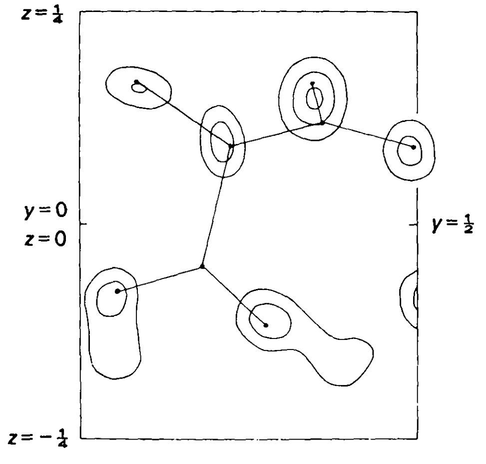

Fig. 2. Electron density projection on (100) with 20 independent structure factors, using the one set of signs determined by the correlation method corresponding to the set determined by the $B_{3,0}$ formula.

Structure determination

The space group $Pmcn$ has an eightfold general position. With four molecules in the unit cell this implies a mirror-symmetrical molecule. As this mirror plane is perpendicular to the short a axis, the space group $Pmcn$ can be discarded.

By Wilson's (1942) method an overall temperature factor ($B = 1{\cdot}87$ Å^2) and the scaling factor were determined. Both unitary and normalized structure factors were calculated.

The application of the correlation method (de Vries, 1965*a*) to the centrosymmetric [100] projection yielded 16 possible sets with 20 signs; the corresponding Fourier syntheses could not be interpreted.

We then applied the Karle & Hauptman (1958) $B_{3,0}$ formula to solve this projection. This formula has the advantage of using three-dimensional data for the sign determination of projection reflexions.

For our purpose the $B_{3,0}$ formula reduces to:

(1) a relation between the signs of three projection reflexions:

$$E_{H_1}E_{H_2}E_{H_1+H_2} = AB + C \text{ for } H_1 = 0k_1l_1,\ H_2 = 0k_2l_2 \quad (1)$$

(2) an expression for the sign of one projection reflexion:

$$(-1)^h|E_{H_1}|^2E_{H_1+H_2} = AB + C \text{ for } H_1 = hkl,\ H_2 = \bar{h}kl\,. \quad (2)$$

The factor $(-1)^h$ accounts for the symmetry of the space group $P2_1cn$. A is a positive constant, C is a small positive correction term and B is given by:

$$B = \langle (E_K^2 - 1)(E_{K+H_1}^2 - 1)(E_{K+H_1+H_2}^2 - 1) \rangle_K$$

in which the mean value is taken over all reflexions $K = hkl$. As the value of B is very sensitive to systematic errors in the $|E|$ values, the sign of the left hand side is equal to the sign of $AB + C$, provided $|AB + C| \gg 0$.

The reliability of the numerical results may be estimated by repeating some of the calculations for a slightly modified scaling factor. About 140 relations (1) and (2) were calculated (Beurskens, 1965) and fifty per cent of the resulting equalities were assumed to be reliable, giving the signs of 45 structure factors with $|E| > 0{\cdot}90$.

A two-dimensional model obtained by a Fourier synthesis (Fig. 1) could be refined (Rutten-Keulemans, 1963) to $R = 0{\cdot}19$. For comparison the Fourier syntheses with the corresponding set of signs as obtained by the correlation method is shown in Fig. 2.

With standard bond lengths and bond angles assumed, approximate x parameters were calculated. The three-dimensional model for the heavy atoms was refined by least-squares to $R = 0{\cdot}11$, using individual temperature factors. A difference Fourier synthesis did not

Table 1. *Final fractional atomic coordinates, their standard deviations and thermal parameters*

The U_{ij} coefficients are given by the expression for the temperature factor:
$\exp\left[-2\pi^2(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33}+2a^*b^*hkU_{12}+2b^*c^*klU_{23}+2c^*a^*lhU_{31})\right]$.
Equivalent positions are (x,y,z); $(x+\frac{1}{2},-y,-z)$; $(x+\frac{1}{2},y+\frac{1}{2},-z+\frac{1}{2})$; $(x,-y+\frac{1}{2},z+\frac{1}{2})$.

	x	y	z		U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{23}$	$2U_{31}$
O(1)	+0·4019	+0·3438	+0·1638	0·0020 Å	+0·021	+0·027	+0·038	−0·007	−0·008	−0·011
O(2)	+0·0691	+0·4939	+0·0895	0·0021	+0·031	+0·017	+0·047	−0·008	+0·004	−0·021
O(3)	+0·1147	+0·0535	−0·0769	0·0020	+0·029	+0·024	+0·024	+0·018	−0·008	+0·004
O(4)	−0·1059	+0·2738	−0·1180	0·0020	+0·043	+0·024	+0·021	+0·025	+0·006	−0·003
N	+0·0990	+0·0845	+0·1650	0·0022	+0·027	+0·019	+0·018	−0·008	+0·004	+0·004
C(1)	+0·1808	+0·3600	+0·1191	0·0026	+0·022	+0·021	+0·021	−0·005	−0·004	+0·005
C(2)	+0·0034	+0·2210	+0·0922	0·0024	+0·019	+0·018	+0·017	−0·001	+0·001	+0·003
C(3)	+0·0059	+0·1788	−0·0470	0·0025	+0·018	+0·018	+0·021	−0·004	−0·002	+0·005
H(1)	+0·2552	+0·0537	+0·1428	0·042						
H(2)	−0·0235	−0·0086	+0·1583	0·042						
H(3)	+0·0919	+0·1107	+0·2528	0·044	B isotropic $=2$ Å^2					
H(4)	−0·1828	+0·2380	+0·1215	0·041						
H(5)	+0·1798	+0·5715	+0·1059	0·041						

Table 2(*a*). *Observed and calculated structure factors*

Scaling factor $c=5$.

h	k	l	c.Fo	c.Fc
0	0	2	158	142
		6	95	94
		8	18	19
		10	7	9
		12	34	33
0	1	1	15	15
		2	102	110
		3	52	52
		4	151	159
		5	64	63
		6	40	37
		7	35	36
		8	22	20
		9	69	70
		10	45	49
		11	2	2
		12	14	13
		13	28	28
0	2	0	27	25
		1	74	74
		2	19	13
		3	118	128
		4	41	37
		5	44	41
		6	32	40
		7	17	17
		8	70	70
		9	2	2
		10	7	6
		11	4	2
		12	54	52
		13	23	23
0	3	1	0	0
		2	98	102
		3	161	170
		4	138	138
		5	20	24
		6	67	67
		7	86	84
		8	78	77
		9	3	4
		10	45	44
		11	5	5
		12	5	5
		13	3	1
0	4	0	76	70
		1	46	44
		2	104	104
		3	98	97
		4	58	58
		5	79	78
		6	131	135
		7	115	119
		8	11	11
		9	63	63
		10	54	53
		11	21	20
		12	7	6
0	5	1	55	56
		2	118	123
		3	93	92
		4	7	[illegible]
		5	27	23
		6	62	61
		7	20	23
		8	17	15
		9	3	0
		10	10	10
		11	24	23
		12	2	0
0	6	0	32	33
		1	48	47
		2	29	28
		3	22	22
		4	14	14
		5	80	80
		6	5	6
		7	50	52
		8	14	16
		9	23	21
		10	16	15
		11	40	41
0	7	1	114	114
		2	19	19
		3	61	61
		4	46	47
		5	47	48
		6	41	40
		7	23	23
		8	5	6
		9	24	22
		10	20	19
0	8	0	3	3
		1	110	111
		2	13	12
		3	90	92
		4	41	38
		5	36	35
		6	15	14
		7	10	11
		8	45	45
		9	[illegible]	8
0	9	1	50	51
		2	20	19
		3	22	21
		4	26	28
		5	42	42
		6	7	5
		7	25	25
0	10	0	10	9
		1	10	12
		2		5
		3	31	33
		4	12	11
		5	19	23
1	0	4	100	101
		6	115	117
		8	65	63
		10	54	55
		12	37	36
1	1	1	96	94
		2	142	142
		4	105	106
		5	135	158
		6	114	113
		7	86	86
		8	35	35
		9	57	54
		10	27	26
		11	11	12
		12	28	26
		13	17	16
1	2	1	165	166
		2	157	158
		3	81	79
		4	49	45
		5	41	37
		6	40	41
		7	39	36
		8	46	47
1	2	9	87	86
		10	27	28
		11	19	19
		12	4	2
		13	33	32
1	3	0	227	224
		1	160	158
		2	32	30
		3	112	110
		4	116	113
		5	98	97
		6	32	31
		7	52	52
		8	46	46
		9	66	65
		10	27	26
		11	28	27
		12	30	30
		13	34	33
1	4	1	77	75
		2	57	53
		3	72	69
		4	68	64
		5	33	32
		6	25	23
		7	13	13
		8	37	37
		9	39	38
		10	37	34
		11	20	17
		12	6	6
1	5	0	156	156
		1	55	54
		2	30	30
		3	29	27
		4	104	102
		5	37	36
		6	30	31
		7	52	52
		8	52	50
		9	6	5
		10	16	15
		11	31	30
		12	50	52
1	6	1	75	75
		2	28	27
		3	51	49
		4	63	62
		5	52	52
		6	44	41
		7	48	47
		8	71	70
		9	16	16
		10	24	24
		11	13	13
1	7	0	38	39
		1	59	59
		2	56	55
		3	67	69
		4	37	37
		5	38	36
		6	43	44
		7	51	51
		8	26	27
		9	15	15
		10	14	13
1	8	1	37	37
		2	66	67
		3	51	49
		4	72	73
1	8	5	14	15
		6	25	23
		7	29	27
		8	33	33
		9	9	9
1	9	0	27	25
		1	15	14
		2	11	11
		3	42	41
		4	13	11
		5	26	25
		6	31	31
		7	20	22
1	10	1	36	36
		2	43	46
		3	17	17
		4	20	20
2	0	2	118	116
		4	65	56
		6	107	107
		8	70	70
		10	9	8
		12	44	45
2	1	1	65	63
		2	60	56
		3	74	71
		4	38	39
		5	44	45
		6	74	75
		7	12	8
		8	36	34
		9	65	63
		10	62	61
		11	20	19
		12	22	22
		13	27	30
2	2	0	286	284
		1	56	55
		2	48	47
		3	100	97
		4	133	132
		5	93	92
		6	51	49
		7	103	101
		8	14	12
		9	16	17
		10	12	11
		11	42	43
		12	24	23
2	3	1	115	113
		2	57	55
		3	78	75
		4	89	87
		5	57	56
		6	24	23
		7	40	40
		8	58	60
		9	19	18
		10	9	10
		12	12	13
2	4	0	83	80
		1	9	4
		2	43	41
		3	42	38
		4	70	68
		5	51	52
		6	66	65
		7	66	65
		8	56	55
		9	46	46
2	4	10	39	39
		11	31	32
		12	31	33
2	5	1	25	21
		2	48	47
		3	44	43
		4	87	88
		5	11	11
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		2	51	51
		3	50	50
		4	14	12
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		6	58	62
		7	15	18
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		9	24	23
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		2	62	61
		3	26	26
		4	29	29
		5	66	68
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		7	27	27
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		9	24	24
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		1	33	33
		2	11	9
		3	38	38
		4	8	8
		5	20	19
		6	11	12
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		8	12	12
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		3	14	14
		4	31	32
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		2	18	19
		3	34	36
3	0	2	151	150
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		8	64	65
		10	39	38
		12	17	18
3	1	0	56	51
		1	52	52
		2	41	41
		3	108	108
		4	40	37
		5	42	42
		6	42	44
		7	47	46
		8	66	65
		9	39	38
3	1	10	27	26
		11	9	9
		12	38	40
3	2	1	51	49
		2	34	33
		3	18	18
		4	27	27
		5	44	45
		6	37	37
		7	12	12
		8	19	18
		9	25	24
		10	13	12
		11	23	22
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		2	30	28
		3	54	54
		4	66	63
		5	67	68
		6	33	33
		7	72	72
		8	21	22
		9	35	34
		10	11	10
		11	22	23
3	4	1	92	92
		2	80	81
		3	39	39
		4	73	73
		5	43	48
		6	48	48
		7	29	29
		8	55	56
		9	43	49
		10	31	33
		11	20	19
3	5	0	30	29
		1	69	69
		2	31	31
		3	56	56
		4	17	1
		5	29	29
		6	41	41
		7	8	[illegible]
		8	36	37
		9	17	17
		10	25	26
3	6	1	35	37
		2	33	36
		3	15	16
		4	52	54
		5	21	22
		6	9	8
		7	11	12
		8	23	24
		9	7	6
3	7	0	49	47
		1	24	26
		2	20	19
		3	21	20
		4	17	[illegible]
		5	33	30
		6	22	21
		7	31	31
		8	6	6
3	8	1	28	28
		2	28	29
		3	48	49
3	8	4	36	37
		5	18	15
		6	15	13
3	9	0	26	24
		1	5	5
		2	19	18
		3	15	15
		4	13	13
4	0	0	82	83
		2	44	45
		4	66	62
		6	41	41
		8	36	36
		10	4	5
4	1	1	14	13
		2	19	20
		3	3	[illegible]
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		5	15	15
		6	44	44
		7	31	31
		8	62	63
		9	12	13
		10	9	10
4	2	0	90	93
		1	50	48
		2	[illegible]	45
		3	20	18
		4	[illegible]	[illegible]
		5	45	46
		6	45	45
		7	24	22
		8	43	44
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		6	41	40
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		5	55	55
		6	32	32
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		8	12	12
		9	8	8
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		3	20	19
		4	38	38
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		6	51	53
		8	40	41
5	1	0	43	44
		1	24	25
		2	[illegible]	15
		3	19	18
		4	7	8
		5	43	44
		6	[illegible]	7
		7	28	2
		8	13	13
5	2	1	[illegible]	18
		2	54	53
		3	58	58
		4	26	26
		5	7	7
		6	37	3
			27	28
		8	15	18
5	3	0	41	42
		1	52	53
		2	14	14
		3	49	49
		4	12	12
		5	6	5
		6	15	17
		7	24	24
5	4	1	49	47
		2	20	21
		3	10	11
		4	19	19
		5	25	25
		6	9	[illegible]
5	5	0	[illegible]	[illegible]
		1	23	23
		2	[illegible]	
		3	23	24
		4	35	35
		5	9	12
5	[illegible]	1	34	33
		2	10	[illegible]
		3	20	21
6	0	0	20	20
		2	27	27
		4	21	21
6	1	1	[illegible]	30
		2	9	9
		3	20	22
		4	18	[illegible]
6	2	0	49	48
		1	13	17
		2	25	25
6	3	1	18	20
		2	25	26

Table 2(*b*). *Structure factors omitted from refinement, calculated at* $R=0{\cdot}029$ *before and after correction for extinction*

Scaling factor $c=5$

hkl	$c.F_{\text{obs}}$	$c.F_{\text{calc}}$	$c.F_{\text{corrected}}$
004	349	441	537
102	229	268	279
110	258	338	395
113	237	255	258
200	259	279	282

reveal the positions of all hydrogen atoms. Five strong reflexions [Table 2(*b*)] apparently suffered from extinction; they were omitted from the refinement once and for all. At this stage of the refinement the omission lowered the R value to 0·10.

Anisotropic refinement of the heavy atom positions gave $R=0{\cdot}063$. A difference Fourier synthesis showed two hydrogen atoms; the remaining three hydrogen atoms were placed on expected positions. These positions could be deduced at this step because the molecular dimensions left no doubt as to the zwitterionic character of the molecule. An extinction correction was computed (de Vries, 1965*b*). Refinement of all atomic positions, with isotropic thermal motion for the hydrogen atom (B kept constant at 2 Å^2) and anisotropic thermal motion for the other atoms, resulted in a final $R=0{\cdot}029$. The final fractional atomic coordinates, their average standard deviations and thermal parameters are given in Table 1, the structure factors observed and calculated in Tables 2(*a*) and 2(*b*). A final total difference Fourier synthesis did not show peaks above the level of 0·13 e.Å^{-3}.

Description of the molecule

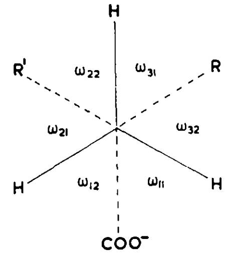
In the aminomalonic acid molecule the ionized and non-ionized carboxyl group can clearly be distinguished. In the ionized carboxyl group the carbon–oxygen distances are 1·24 and 1·25 Å whereas in the non-ionized carboxyl group distances of 1·21 and 1·31 Å are found. The angle between the COO$^-$ and COOH planes is 114·3°; the carbon–nitrogen bond is rotated out of these planes over −9·1° and −18·0° respectively. The distance of closest approach between the ionized carboxyl group and the non-ionized carboxyl group O(4)–O(2) is 3·06 Å (Fig. 3). The differences in the carbon–carbon bond lengths are significant; the C–C bond in which the carbon atom of the non-ionized carboxyl group participates is the shorter one.

Table 3. *Bond lengths and bond angles of bonds involving hydrogen atoms in the aminomalonic acid molecule*

Bond distances		Bond angles	
H(1)–N	0·86 Å	H(1)–N—C(2)	112°
H(2)–N	1·00	H(1)–N—H(2)	107
H(3)–N	0·98	H(1)–N—H(3)	112
H(4)–C(2)	0·99	H(2)–N—C(2)	112
H(5)–O(2)	0·88	H(2)–N—H(3)	103
		H(3)–N—C(2)	109
		H(4)–C(2)–N	104
		H(4)–C(2)–C(1)	112
		H(4)–C(2)–C(3)	111
		H(5)–O(2)–C(1)	110

Bond lengths and bond angles (Fig. 3 and Table 3) were calculated without correction for anisotropic motion; standard deviations are 0·003 Å and 0·13° respectively, except for bonds involving hydrogen atoms, in which case these values are 0·05 Å and 2° for angles of the type HXX and 4° for angles HXH.

In the crystalline state the aminomalonic acid molecule roughly has an ethane conformation around the nitrogen–carbon bond. This feature is found in other amino acids as well as in the end group of peptides. The deviation from the ideal staggered conformation can be conveniently expressed by the sum $\frac{1}{6}|\sum_{i=1}^{3}(\omega_{i1}-\omega_{i2})|$, ω being the angle between two planes through the carbon–nitrogen bond as shown in the Newman projection:



The energy gained by forming hydrogen bonds with all hydrogen atoms of the ammonium group is much higher than the rotation barrier of the carbon–nitrogen bond. Nevertheless, the conformation actually found is the staggered one (Table 4). Apparently both requirements – total hydrogen bonding as well as a staggered conformation – can be met with in the crystal structure of amino acids and peptides.

Discussion of the crystal structure

In the crystal both antipodes of aminomalonic acid are present. It is noteworthy that the *racemate* crystallizes in a *non-centrosymmetric* (non-enantiomorphic) space group.

Table 4. *Deviation from the ideal staggered conformation around the* C–N *bond in some amino acids and peptides*

	$\frac{1}{6}\lvert\sum_{i=1}^{3}(\omega_{i1}-\omega_{i2})\rvert$	
α-Glycine	3°	Marsh (1958)
β-Glycine	1	Iitaka (1960)
γ-Glycine	0	Iitaka (1961)
Diglycine . HCl (glycine part)	14	Hahn & Buerger (1957)
Diglycine . HCl (glycine . HCl part)	5	Hahn & Buerger (1957)
DL-Serine	0	Shoemaker, Barieau, Donohue & Chia-Si Lu (1953)
Aminomalonic acid	5	This article
L-Asparagine	15	de Vries (1963)
L-Cystine	1	Oughton & Harrison (1959)
Glycyl-L-asparagine	2	Pasternak, Katz & Corey (1954)
Glycyl-L-tryptophane	14	Pasternak (1956)
L-Leucyl-L-prolyl-glycine	1	Leung & Marsh (1958)
L-Lysine . HCl	14	Wright & Marsh (1962)
	mean 6°	

For a description of the structure we will consider the zigzag chain of molecules around the twofold screw axis at $y=0$, $z=0$ as a structural unit. A glide plane transforms this unit into its inverse at $y=\frac{1}{2}$, $z=\frac{1}{2}$ (Fig. 4). These units are arranged in a pseudohexagonal way. A molecule in the zigzag chain is connected to each of its neighbours by two hydrogen bonds (Fig. 5). Another hydrogen bond in the chain is probably formed between the doubly bonded oxygen of one molecule and the aliphatic hydrogen atom of its second next neighbour. We are hoping to get additional information about this interaction by infra-red spectrometric methods.

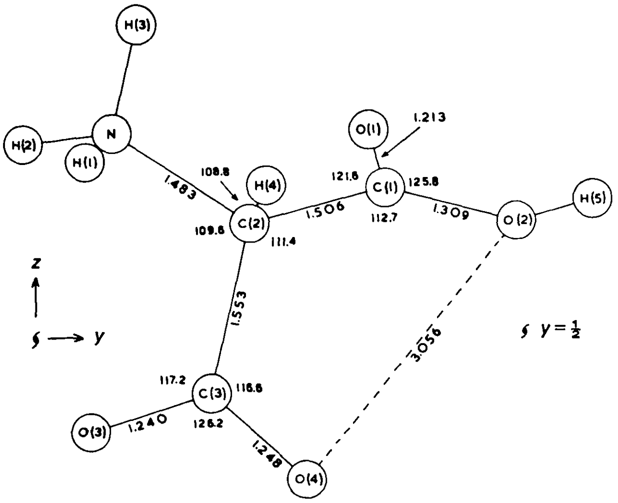


Fig. 3. Projection on (100) showing bond distances and bond angles in aminomalonic acid. For distances and angles of bonds involving hydrogen atoms see Table 3.

The zigzag chain at $y=0$, $z=0$ is connected to the chain at $y=1$, $z=0$ by hydrogen bonds formed between an ionized and a non-ionized carboxyl group. The chain

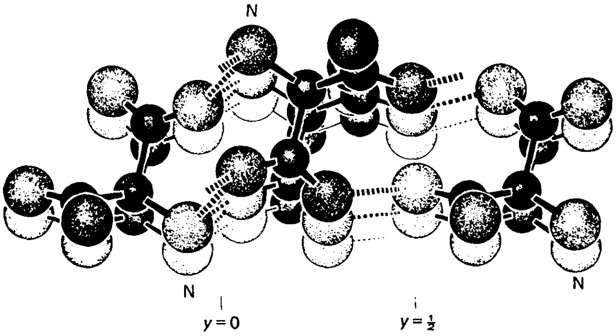


Fig. 5. Perspective drawing of the structure of aminomalonic acid as seen nearly in the [100] direction.

Table 5. *Intermolecular hydrogen bonds in the structure of aminomalonic acid*

Donor	Acceptor	Distance d···a	Distance H···a	Angle d–H···a
N–H(1) ·····O(3)	$(\frac{1}{2}+x, -y, -z)$	2·99 Å	2·1 Å	171°
N–H(2) ·····O(3)	$(-\frac{1}{2}+x, -y, -z)$	2·85	2·1	136
N–H(3) ·····O(4)	$(x, \frac{1}{2}-y, \frac{1}{2}+z)$	2·84	2·0	145
O(2)–H(5)···O(4)	$(\frac{1}{2}+x, 1-y, -z)$	2·57	1·7	173
C(2)–H(4)···O(1)	$(x-1, y, z)$	3·27	2·3	164

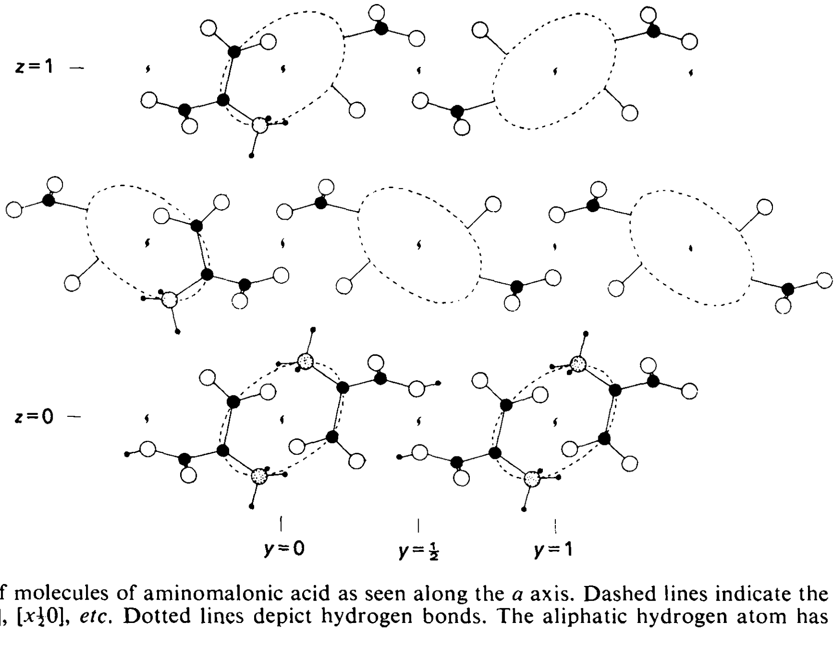


Fig. 4. Arrangement of molecules of aminomalonic acid as seen along the *a* axis. Dashed lines indicate the zigzag chains around the screw axis $[x0\frac{1}{2}]$, $[x\frac{1}{2}0]$, *etc.* Dotted lines depict hydrogen bonds. The aliphatic hydrogen atom has been omitted.

Table 6. *Vibrational ellipsoids*

Coordinates (in Å relative to the mean atomic positions) determine points on the axes of vibration. B values correspond to r.m.s. displacements ($B=8\pi^2\overline{U^2}$).

	I				II				III			
	x	y	z	B_{I}	x	y	z	B_{II}	x	y	z	B_{III}
O(1)	+0·05	+0·05	−0·19	3·16	+0·08	−0·15	−0·02	2·27	+0·11	+0·06	+0·04	1·37
O(2)	+0·10	−0·02	−0·20	4·11	+0·14	−0·04	+0·07	2·06	+0·03	+0·12	+0·00	1·22
O(3)	+0·15	+0·11	−0·02	2·82	+0·05	−0·05	+0·14	2·04	+0·06	−0·09	−0·06	1·17
O(4)	+0·20	+0·10	+0·00	3·93	+0·04	−0·07	−0·13	1·81	+0·05	−0·10	+0·07	1·27
N	+0·16	−0·06	+0·02	2·27	+0·02	+0·10	+0·10	1·64	+0·04	+0·08	−0·08	1·19
C(1)	+0·10	−0·08	+0·09	2·06	+0·09	+0·10	−0·01	1·50	+0·05	−0·06	−0·11	1·47
C(2)	+0·13	−0·02	+0·06	1·60	+0·01	−0·12	−0·06	1·41	+0·05	+0·05	−0·10	1·23
C(3)	+0·08	−0·06	+0·12	1·80	+0·04	−0·10	−0·08	1·40	+0·10	+0·07	−0·03	1·21

at $x=0$, $y=0$ is also connected to the chain at $y=\frac{1}{2}$, $z=\frac{1}{2}$ by a N–H···O hydrogen bond.

The ten intermolecular hydrogen bonds (Table 5) between each molecule and its neighbours, and the electrostatic interaction of the ionized carboxyl group and the ammonium group explain the rather low thermal parameters (Table 6).

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The Minimum-Residual Refinement of β-Uranium from Polycrystalline Data

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The non-centrosymmetric and the centrosymmetric structures of β-uranium proposed by Tucker and by Tucker & Senio are further refined from the X-ray powder intensities of Thewlis by the minimum-residual method programmed for the Atlas computer. The agreement residuals of 0·19 for the former and 0·32 for the latter structure have been improved respectively to 0·16 and 0·23, and for the non-centrosymmetric structure this improvement has been accompanied by a further shortening to 2·50 Å of the already short distance of 2·53 Å between atoms in neighbouring main layers.

Introduction

Consequent upon the refinement of the β-uranium structure by Tucker & Senio (1952, 1953) and by Thewlis & Steeple (1954) it was evident from the data available that the status of the structure must remain in doubt (Tucker, Senio, Thewlis & Steeple, 1956). Tucker & Senio, working with single-crystal data obtained from an alloy of uranium and chromium which had been quenched from the β-phase region (Tucker, 1951), proposed a centrosymmetric structure with space group $P4_2/mnm$ (D^{14}_{4h}) but from powder photographs