

209. The Crystal and Molecular Structure of Certain Dicarboxylic Acids. Part IV. β -Succinic Acid.

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The parameters of the β -succinic acid crystal structure have been re-determined with the use of more complete Fourier series and the preparation of a new projection of the structure along the [101] crystal axis. Small shifts in the atomic positions are obtained, but the slight shortening of the carbon-carbon bonds below the standard value of 1.54 Å. reported by Verweel and MacGillavry is confirmed. The present results indicate, however, that the shortest bond (1.50 Å.) is that between the central pair of carbon atoms. The situation of this bond is similar to that of the central bond in dibenzyl and to that of the bond in geranylamine hydrochloride which is situated centrally between the two ethylene linkages. In both these cases a similar contraction has been observed. The intermolecular approach distances in the succinic acid structure are also discussed.

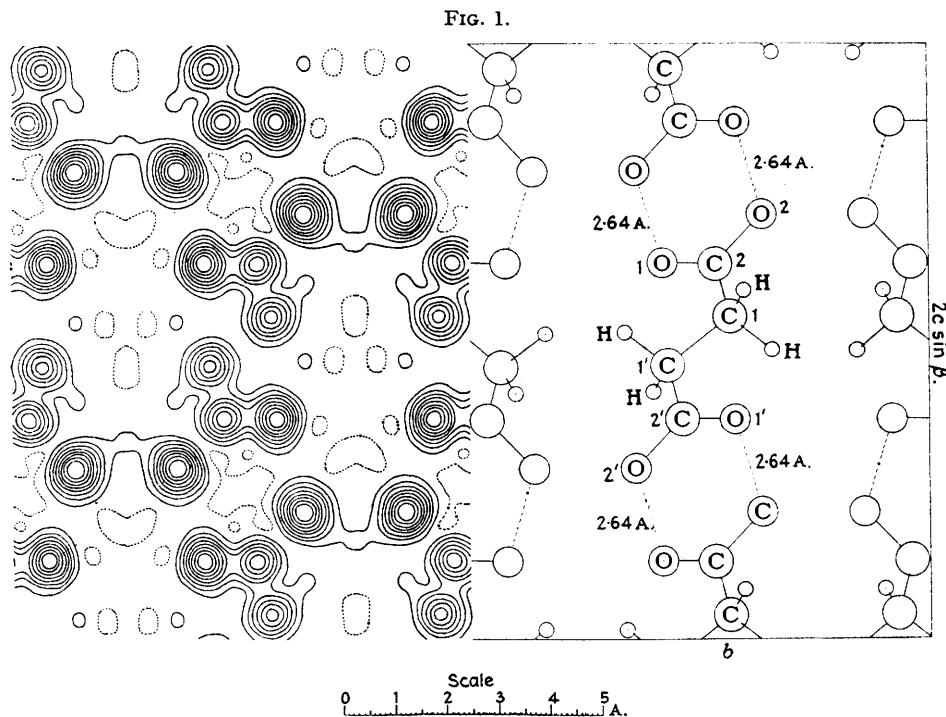
THE physical properties of the normal aliphatic dicarboxylic acids have already been the subject of numerous studies and a large amount of data has been collected for this series. Interest has been stimulated by the curious alternating effects which are a feature of homologous series of normal aliphatic compounds in general. These effects are shown to a greater or less extent by the hydrocarbons, alcohols, ketones, diamines, etc., but in none to the same degree as in the normal aliphatic dibasic acids. Numerous theories have been proposed (Malkin, *J.*, 1931, 2796; Muller, *Proc. Roy. Soc.*, 1929, *A*, **124**, 317; Nekrassow, *Z. physikal. Chem.*, 1927, **128**, 203) to account for the variation in properties of the odd and even members, but to a large extent these theories have remained speculative for want of a sufficiently detailed knowledge of the structures involved.

The object of the present series of papers will be to determine the crystal and in particular the detailed molecular structures of some of the simple members of this series with as much accuracy as possible in an endeavour to obtain a reliable correlation between structure and properties. Good crystals are easily obtained and unit-cell and space-group determinations are already available for most of the common members (Caspari, *J.*, 1928, 3235; 1929, 2709; Gerstäcker, Moller, and Reis, *Z. Krist.*, 1927, **66**, 421). More detailed X-ray structural investigations have also been made for anhydrous oxalic acid (Hendricks, *ibid.*, 1935, **91**, 48), the dihydrate of oxalic acid (Part I of this series, Dunitz and Robertson, *J.*, 1947, 142), succinic acid (Verweel and MacGillavry, *Nature*, 1938, **142**, 161; *Z. Krist.*, 1939, **102**, 60), and adipic acid (MacGillavry, *Rec. Trav. Chim.*, 1941, **60**, 605).

In the present paper the parameters for β -succinic acid (the crystal modification which is stable at normal temperatures) are re-examined and an attempt is made to determine whether bond-length variations exist in the saturated carbon chain. Our results (Fig. 3) fully confirm the findings of Verweel and MacGillavry (*loc. cit.*). Our measurements indicate a slightly greater contraction in the central bond, but it is probable that final conclusions regarding this and other details of the fine structure will only be reached after making a full three-dimensional analysis of all the available intensity data. The present two-dimensional refinement of the parameters is a necessary foundation for this more comprehensive approach. It is also valuable in giving results which are strictly comparable to those obtained for the higher members of the series (Parts V and VI) for which the two-dimensional approach is the only one feasible at present. If certain regularities are detected in the fine structure for all the members in such a series of related compounds, then it may be possible to attach some significance to the results even although the individual observations are near to the limit of possible experimental error.

Crystal Data.— β -Succinic acid, $C_4H_6O_4$; M , 118.1; m. p. 185° ; d , calc. 1.561, found 1.550. Monoclinic prismatic, $a = 5.10 \pm 0.01$, $b = 8.88 \pm 0.03$, $c = 7.61 \pm 0.02$ A., $\beta = 133.6^\circ \pm 0.3^\circ$. Absent spectra, $(h0l)$ when h is odd, $(0k0)$ when k is odd. Space group C_{2h}^5 ($P2_1/a$). Two molecules per unit cell. Molecular symmetry, centre. Volume of the unit cell, 249.6 A.³. Absorption coefficient for X -rays ($\lambda = 1.54$ A.) $\mu = 14.25$ cm.⁻¹. Total number of electrons per unit cell = $F(000) = 124$.

Crystallisation from water produces needles elongated in the direction of the c axis, but these are generally too thin to be of use in X -ray work. Slow crystallisation from ethyl acetate produces crystals with a tendency to elongation along the a axis which are suitable for X -ray work. Crystals so obtained are extremely brittle and difficult to cut without causing distortion. There is also a very marked fibre cleavage in the direction of the c axis. Faces most commonly developed are (001), (010), (111), (110), and (011).



Normal projection of succinic acid structure along a axis. Contour scale: one electron per A.² per line, one-electron line dotted.

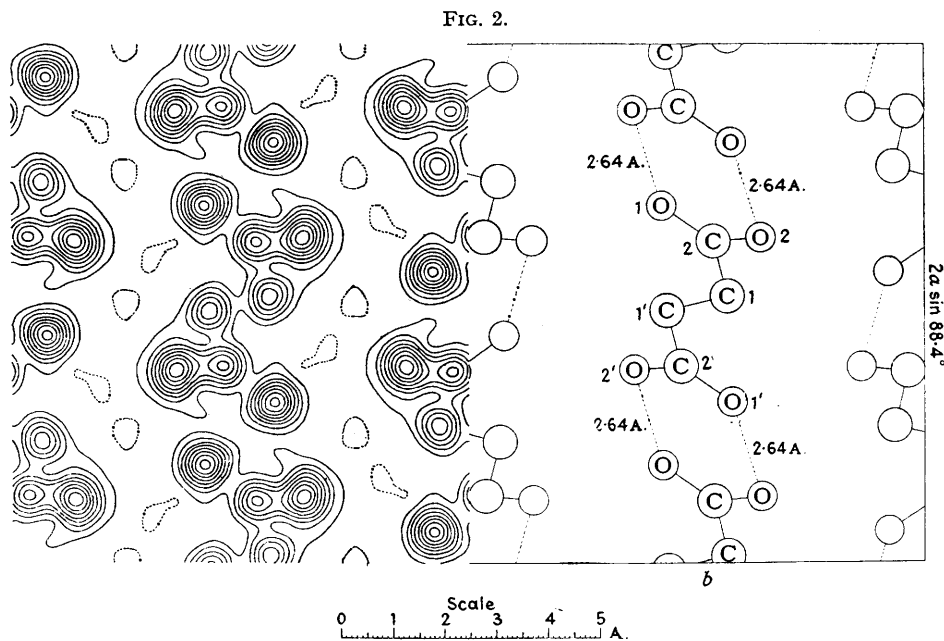
Further goniometric and optical data are given by Groth ("Chemische Krystallographie," 1919, 3, 262) and the crystal structure has been investigated by Yardley (*Proc. Roy. Soc.*, 1924, *A*, 105, 451) and Dupré la Tour (*Compt. rend.*, 1930, 191, 1348; 1931, 193, 180; *Ann. Physique*, 1932, 18, 199), who determined the lattice constants and space group in reasonable agreement with our present work.

Analysis of the Structure.—A very full investigation of the structure has been made by Verweel and MacGillavry (*loc. cit.*) based on the intensities of the reflections from the three axial zones. Projections of the structure were made along the a and b crystal axes by the double Fourier series method, and from these results the atomic co-ordinates were determined. The resolution obtained in the a axis projection was good, but rather poor in the b axis projection owing to the close approach of certain pairs of atoms (see Fig. 4, this paper). Nevertheless, their final results are in very good agreement with those we have now obtained.

Starting with the co-ordinates of Verweel and MacGillavry, we have attempted to refine the structure further by the Fourier series method, with the inclusion of a larger number of reflecting planes and the preparation of new projection along the $[101]$ axis which gives much better resolution of the atoms than can be obtained from the b axis projection. From these results we have obtained slight shifts in some of the atomic positions, necessitating change of sign for a few

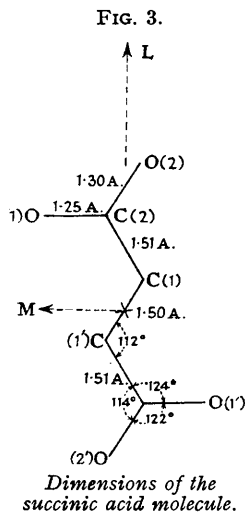
reflections. These changes have been incorporated in our final syntheses which are shown as normal projections along the a axis and the $[101]$ axis in Figs. 1 and 2.

The two projection axes are almost at right angles (88.4°) and it will be seen that in each projection all the atoms are separately resolved. All three co-ordinates for each atom may thus



Normal projection of succinic acid structure along $[101]$ axis. Contour scale as in Fig. 1.

be estimated with considerable accuracy. The positions which we have finally assigned to the centres of the atoms in the a axis projection are indicated by small crosses in Fig. 5, where the origin and co-ordinate network are also shown. A close examination of the contour levels in the a axis projection (Figs. 1 and 5) shows small peaks at the two-electron level which occur approximately where the hydrogen atoms of the CH_2 groups should appear, a normal tetrahedral distribution of the valency bonds about the carbon atoms being assumed. It is likely that these peaks are actually due to the hydrogen atoms, but confirmation by three-dimensional synthesis is awaited, and in the meantime no attempt has been made to include a hydrogen contribution in the calculation of the intensities. For the higher members of this series the hydrogen contribution to the total scattering becomes much more important, and the matter is further discussed in Part VI.



Orientation, Co-ordinates, and Dimensions.—We choose as principal molecular axis L the line passing through the mid points of the three carbon-carbon bonds in the molecule (Fig. 3). The four carbon atoms in the molecule lie in one plane and so the second molecular axis M is chosen to lie in this plane at right angles to L . The third axis N is perpendicular to L and M . The orientation of the molecule in the crystal is then given by the angles in Table I, where χ , ψ , and ω are the angles which the molecular axes make with the a , b , and c' crystal axes, c' being chosen perpendicular to a and b .

The observed co-ordinates of the atoms in the asymmetric crystal unit (half the molecule) are listed in Table II, referred to the crystallographic axes. The co-ordinates of all the other atoms in the unit cell may be obtained from these by the appropriate symmetry operations, *viz.*, x, y, z ; $-x, -y, -z$; $\frac{1}{2} + x, \frac{1}{2} - y, z$; $\frac{1}{2} - x, \frac{1}{2} + y, -z$. The co-ordinates referred to the molecular axes L, M, N are listed in Table III.

From the co-ordinates it is easy to deduce the bond lengths and valency angles. These are shown diagrammatically in Fig. 3, and the figures are collected and compared with previous findings in Table IV. The agreement is on the whole extremely good, although we find a somewhat larger contraction in the length of the central bond, C(1')-C(1). We believe this contraction to be significant, and it is confirmed by our work on the higher members of this series. The situation of the central carbon-carbon bond in succinic acid is clearly analogous to that of the central bond in dibenzyl and to the bond in geranylamine hydrochloride situated centrally between the ethylene linkages. In both these cases a notable contraction from the normal carbon-carbon single-bond length of 1.54 Å. has been observed (Jeffrey, *Proc. Roy. Soc.*, 1947, *A*, **188**, 222; 1945, *A*, **183**, 388; Cox and Cruickshank, *Acta Cryst.*, 1948, **1**, 92).

TABLE I.

Orientation of the molecule in the crystal.

| | | |
|--|--|--|
| $\chi_L = 56.0^\circ$, $\cos \chi_L = 0.5590$ | $\chi_M = 60.4^\circ$, $\cos \chi_M = 0.4944$ | $\chi_N = 48.2^\circ$, $\cos \chi_N = 0.6667$ |
| $\psi_L = 68.5^\circ$, $\cos \psi_L = 0.3670$ | $\psi_M = 150.2^\circ$, $\cos \psi_M = -0.8674$ | $\psi_N = 70.3^\circ$, $\cos \psi_N = 0.3370$ |
| $\omega_L = 42.0^\circ$, $\cos \omega_L = 0.7437$ | $\omega_M = 86.8^\circ$, $\cos \omega_M = 0.0563$ | $\omega_N = 131.7^\circ$, $\cos \omega_N = -0.6648$ |

TABLE II.

Observed co-ordinates. (Centre of symmetry as origin.)

x, y, z are referred to the monoclinic crystal axes, a, b, c ; x', y, z' are orthogonal co-ordinates referred to the a and b crystal axes and their perpendicular c' .

| Atom (cf. Fig. 3). | $x, A.$ | $y, A.$ | $z, A.$ | $x', A.$ | $z', A.$ | $\frac{2\pi x}{a}$ | $\frac{2\pi y}{b}$ | $\frac{2\pi z}{c}$ |
|-----------------------|---------|---------|---------|----------|----------|--------------------|--------------------|--------------------|
| C(1) | 0.276 | 0.592 | 0.602 | 0.140 | 0.436 | 19.5° | 24.0° | 28.5° |
| C(2) | 0.099 | 0.326 | 1.966 | 1.257 | 1.432 | 7.0 | 13.2 | 93.0 |
| O(1) | -0.602 | -0.703 | 1.976 | 1.965 | 1.431 | -42.5 | -28.5 | 93.5 |
| O(2) | 0.864 | 1.233 | 2.234 | 1.367 | 2.342 | 61.0 | 50.0 | 153.0 |

TABLE III.

Co-ordinates referred to the molecular axes L, M, and N. (Origin at centre of symmetry.)

| Atom. | $L, A.$ | $M, A.$ | $N, A.$ | Atom. | $L, A.$ | $M, A.$ | $N, A.$ |
|-------|---------|---------|---------|-------|---------|---------|---------|
| C(1) | 0.620 | -0.420 | 0.000 | O(1) | 1.904 | 1.662 | 0.122 |
| C(2) | 1.880 | 0.419 | 0.000 | O(2) | 2.959 | -0.261 | -0.230 |

TABLE IV.

Molecular dimensions.

| | Present work. | Previous work.* | Present work. | Previous work.* |
|------------|----------------|-----------------|-----------------|-----------------|
| C(1')-C(1) | 1.50 ± 0.02 Å. | 1.52 Å. | C(1')-C(1)-C(2) | 112° |
| C(1)-C(2) | 1.51 " | 1.51 | C(1)-C(2)-O(1) | 124 |
| C(2)-O(1) | 1.25 " | 1.28 | C(1)-C(2)-O(2) | 114 |
| C(2)-O(2) | 1.30 " | 1.31 | O(1)-C(2)-O(2) | 122 |

* Verweel and MacGillavry, *loc. cit.*

TABLE V.

*Intermolecular distances (Å).**

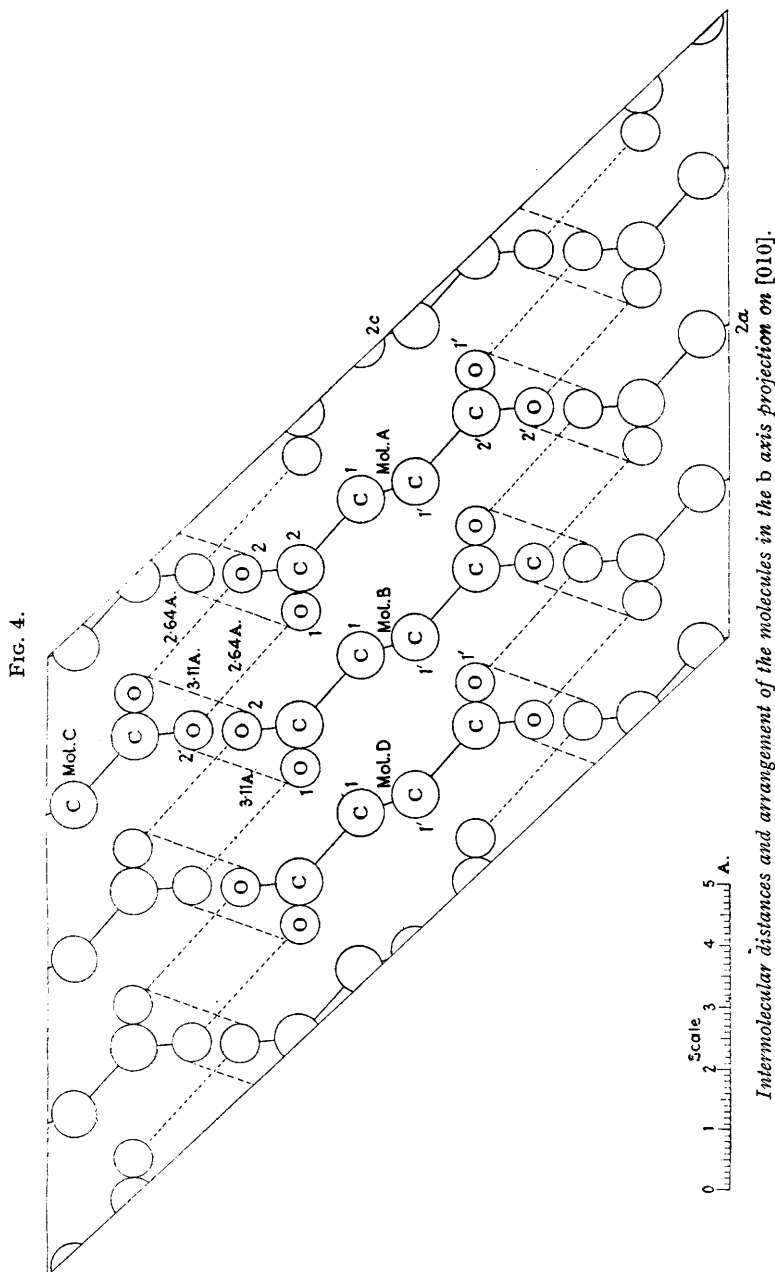
| | |
|----------------------------------|----------------------------------|
| O(1A) . . . O(2'C) = 2.64 ± 0.04 | O(1A) . . . O(1'D) = 3.42 ± 0.04 |
| O(2A) . . . O(1'C) = 2.64 " | O(2A) . . . C(1B) = 3.49 " |
| O(2'C) . . . O(1B) = 3.11 " | O(2'C) . . . C(1D) = 3.52 " |
| O(1'C) . . . O(2B) = 3.11 " | O(1A) . . . C(1'D) = 3.53 " |
| O(2A) . . . O(2B) = 3.23 " | O(1A) . . . C(1D) = 3.66 " |

* A = standard molecule; B = reflected molecule; C = molecule one translation along c axis.

All the carbon atoms in the molecule are necessarily coplanar, and this plane passes through the centre of symmetry in the middle of the molecule. The planes of the carboxyl groups, however, are rotated through about 9° about the axis C(1)-C(2) with respect to the plane containing the carbon atoms. The plane of the carboxyl group also passes through the centre of symmetry which is situated between the ends of adjoining molecules in the c axis direction. It follows, therefore, that adjoining carboxyl groups, which are linked by hydrogen bridges (see Fig. 4), are coplanar.

Intermolecular Distances.—The more important intermolecular distances are indicated by dotted lines in Fig. 4, which represents a projection of the structure along the b axis on the (010) plane. Some of these intermolecular approaches are also marked in the line drawings which

accompany the contour maps in Figs. 1 and 2. The closest approach between molecules occurs in the hydrogen bridges of length 2.64 Å, which connect the oxygen atoms of adjacent carboxyl groups along the c axis. Certain other oxygen–oxygen distances, particularly a pair at 3.11 Å., are also shorter than is usual in the acids of this series, and this leads to an exceptionally compact grouping of the succinic acid molecules. The shortest carbon–oxygen distance is 3.49 Å. between molecules A and B . The figures are collected in Table V.



EXPERIMENTAL.

X-Ray Measurements.—Copper $K\alpha$ radiation ($\lambda = 1.54$ Å.) was used throughout this work, and rotation, oscillation, and moving-film photographs of all the principal zones were taken. The intensities were estimated visually, with the help of the multiple-film technique (Robertson, *J. Sci. Instr.*, 1943, 20,

TABLE VI.
Measured and calculated values of the structure factor.

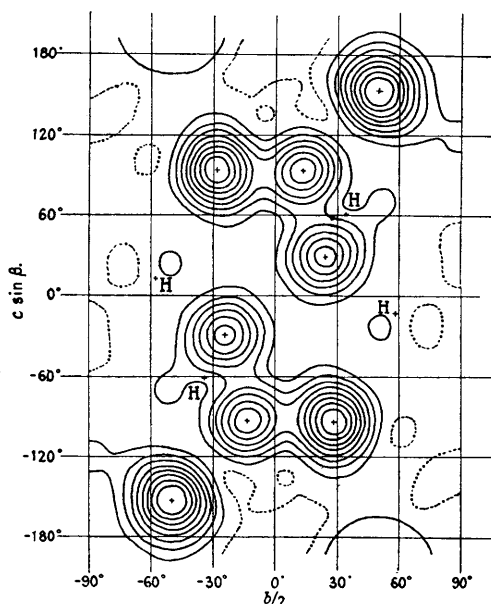
| <i>hkl.</i> | $\sin \theta$ ($\lambda = 1.54$). | F, meas. | F, calc. | <i>hkl.</i> | $\sin \theta$ ($\lambda = 1.54$). | F, meas. | F, calc. |
|-------------|--|----------|----------|-----------------|--|----------|----------|
| 020 | 0.173 | 24.0 | +34.0 | 017 | 0.983 | <0.5 | + 0.0 |
| 040 | 0.346 | 15.5 | -16.0 | 027 | 0.995 | <0.5 | - 0.0 |
| 060 | 0.519 | 7.5 | - 9.0 | 20 $\bar{2}$ | 0.299 | 17.5 | -18.0 |
| 080 | 0.692 | 4.0 | - 3.5 | 40 $\bar{4}$ | 0.598 | 7.0 | + 8.0 |
| 0,10,0 | 0.865 | 3.5 | - 2.5 | 60 $\bar{6}$ | 0.886 | 3.5 | - 2.5 |
| 001 | 0.140 | 6.0 | -10.5 | 11 $\bar{1}$ | 0.173 | 2.0 | - 0.0 |
| 002 | 0.280 | 10.5 | -13.0 | 21 $\bar{2}$ | 0.311 | 8.0 | + 9.5 |
| 003 | 0.419 | <1.0 | + 2.5 | 31 $\bar{3}$ | 0.459 | 10.0 | +12.5 |
| 004 | 0.559 | 9.0 | + 9.0 | 41 $\bar{4}$ | 0.606 | 2.0 | + 2.0 |
| 005 | 0.699 | 2.0 | - 1.5 | 51 $\bar{5}$ | 0.755 | 6.0 | + 4.0 |
| 006 | 0.838 | 9.5 | - 7.5 | 61 $\bar{6}$ | 0.889 | <1.0 | - 0.5 |
| 007 | 0.978 | 0.5 | + 1.5 | 12 $\bar{1}$ | 0.229 | 17.5 | +17.0 |
| 011 | 0.165 | 5.0 | + 4.0 | 22 $\bar{2}$ | 0.347 | 1.0 | + 1.0 |
| 021 | 0.223 | 10.0 | +10.0 | 32 $\bar{3}$ | 0.482 | 16.5 | -18.5 |
| 031 | 0.295 | 1.5 | - 1.0 | 42 $\bar{4}$ | 0.624 | 2.5 | + 1.0 |
| 041 | 0.373 | 10.0 | +12.0 | 52 $\bar{5}$ | 0.770 | 7.5 | + 7.0 |
| 051 | 0.455 | 3.0 | - 1.5 | 62 $\bar{6}$ | 0.903 | 1.5 | - 0.5 |
| 061 | 0.537 | 6.5 | - 8.5 | 13 $\bar{1}$ | 0.299 | 2.5 | + 3.5 |
| 071 | 0.622 | 7.0 | + 7.0 | 23 $\bar{3}$ | 0.396 | 15.5 | +17.5 |
| 081 | 0.706 | 6.5 | - 5.0 | 33 $\bar{3}$ | 0.518 | <1.5 | - 0.0 |
| 091 | 0.791 | 6.5 | + 5.5 | 43 $\bar{4}$ | 0.653 | 2.0 | + 2.5 |
| 0,10,1 | 0.876 | <1.0 | + 1.0 | 53 $\bar{5}$ | 0.793 | 3.0 | + 1.5 |
| 0,11,1 | 0.961 | 1.5 | + 1.0 | 63 $\bar{6}$ | 0.924 | <1.0 | - 0.5 |
| 012 | 0.292 | 7.0 | - 6.5 | 14 $\bar{1}$ | 0.378 | 7.0 | - 5.0 |
| 022 | 0.329 | 14.5 | -16.0 | 24 $\bar{2}$ | 0.457 | 4.5 | + 6.0 |
| 032 | 0.381 | 4.5 | + 3.0 | 34 $\bar{3}$ | 0.566 | 4.0 | - 4.0 |
| 042 | 0.445 | 9.0 | - 7.0 | 44 $\bar{4}$ | 0.689 | <1.5 | - 1.0 |
| 052 | 0.514 | 11.0 | +12.5 | 54 $\bar{5}$ | 0.744 | 4.5 | + 4.5 |
| 062 | 0.590 | 6.5 | + 6.5 | 64 $\bar{6}$ | 0.952 | <1.0 | + 1.0 |
| 072 | 0.667 | <1.5 | + 1.0 | 15 $\bar{1}$ | 0.457 | 2.5 | + 4.5 |
| 082 | 0.746 | 4.0 | + 3.5 | 25 $\bar{2}$ | 0.524 | 9.5 | + 9.0 |
| 092 | 0.828 | 4.0 | - 3.0 | 35 $\bar{3}$ | 0.622 | 4.5 | - 0.0 |
| 0,10,2 | 0.909 | 1.0 | - 0.5 | 45 $\bar{4}$ | 0.738 | <1.5 | + 0.5 |
| 0,11,2 | 0.992 | <0.5 | - 0.5 | 55 $\bar{5}$ | 0.865 | 2.0 | - 1.5 |
| 013 | 0.429 | 15.5 | +17.5 | 65 $\bar{6}$ | 0.985 | <0.5 | + 0.5 |
| 023 | 0.454 | 2.0 | + 3.0 | 16 $\bar{1}$ | 0.541 | 2.0 | - 1.5 |
| 033 | 0.494 | 15.5 | +18.0 | 26 $\bar{2}$ | 0.599 | 7.0 | - 8.5 |
| 043 | 0.544 | 2.0 | + 1.0 | 36 $\bar{3}$ | 0.686 | 1.5 | + 1.0 |
| 053 | 0.602 | 2.5 | - 2.5 | 46 $\bar{4}$ | 0.792 | 4.0 | + 6.0 |
| 063 | 0.667 | 1.5 | - 1.5 | 56 $\bar{6}$ | 0.914 | 3.0 | + 0.5 |
| 073 | 0.737 | 4.0 | - 3.5 | 17 $\bar{1}$ | 0.624 | <1.5 | - 0.0 |
| 083 | 0.809 | 1.5 | - 1.0 | 27 $\bar{2}$ | 0.676 | <1.5 | - 1.0 |
| 093 | 0.885 | 4.0 | - 1.5 | 37 $\bar{3}$ | 0.753 | 4.0 | - 3.5 |
| 0,10,3 | 0.961 | <1.0 | + 0.0 | 47 $\bar{4}$ | 0.850 | <1.5 | - 0.5 |
| 014 | 0.565 | 4.5 | - 5.0 | 57 $\bar{5}$ | 0.964 | 2.5 | - 2.0 |
| 024 | 0.586 | 8.0 | + 9.0 | 18 $\bar{1}$ | 0.707 | 9.5 | + 7.5 |
| 034 | 0.616 | 1.5 | - 0.0 | 28 $\bar{2}$ | 0.753 | 6.0 | - 3.5 |
| 044 | 0.658 | 3.0 | + 1.5 | 38 $\bar{3}$ | 0.825 | 3.0 | - 2.0 |
| 054 | 0.707 | 8.0 | + 6.0 | 48 $\bar{4}$ | 0.915 | 1.0 | + 1.0 |
| 064 | 0.763 | 3.5 | - 2.5 | 19 $\bar{1}$ | 0.792 | 2.0 | - 1.0 |
| 074 | 0.824 | 2.5 | + 1.0 | 29 $\bar{2}$ | 0.835 | 3.0 | - 2.5 |
| 084 | 0.889 | 1.5 | - 1.5 | 39 $\bar{3}$ | 0.899 | 2.0 | - 1.0 |
| 094 | 0.958 | 2.0 | - 1.5 | 49 $\bar{4}$ | 0.982 | 0.5 | - 0.5 |
| 015 | 0.704 | 2.0 | + 1.5 | 1,10, $\bar{1}$ | 0.879 | 4.5 | + 3.0 |
| 025 | 0.719 | 4.0 | - 3.5 | 2,10, $\bar{2}$ | 0.915 | 1.5 | + 1.5 |
| 035 | 0.745 | 2.0 | + 0.5 | 3,10, $\bar{3}$ | 0.974 | 2.0 | - 0.5 |
| 045 | 0.780 | 2.5 | - 2.0 | 1,11, $\bar{1}$ | 0.962 | 1.0 | - 0.5 |
| 055 | 0.822 | <1.0 | - 1.0 | | | | |
| 065 | 0.872 | 4.0 | + 2.0 | | | | |
| 075 | 0.924 | 2.5 | + 1.5 | | | | |
| 085 | 0.984 | 2.0 | + 1.5 | | | | |
| 016 | 0.844 | <1.0 | - 0.0 | | | | |
| 026 | 0.856 | 5.0 | - 3.0 | | | | |
| 036 | 0.879 | 1.0 | + 0.5 | | | | |
| 046 | 0.907 | 1.5 | + 1.5 | | | | |
| 056 | 0.943 | <1.0 | + 0.5 | | | | |
| 066 | 0.987 | 1.0 | + 1.0 | | | | |

175) over a range of about 1500 to 1. Only moving-film photographs were employed for the intensity work. The usual correction factors were applied, and the observed structure factors are collected in Table VI.

The crystal specimens used for these measurements for the $(0kl)$ zone had cross-sections normal to the a axis of $0.09 \text{ mm.} \times 0.11 \text{ mm.}$ and $0.45 \text{ mm.} \times 0.52 \text{ mm.}$ For the (hkh) zone the cross-section was $0.38 \text{ mm.} \times 0.42 \text{ mm.}$ The specimens were completely immersed in the X -ray beam, and absorption corrections for different reflections in the same zone were not applied. Most of the specimens were characterised by a high degree of perfection of the crystal faces. To ensure that the crystals were of the true mosaic type some specimens were immersed in liquid air before use. The intensities from crystals so treated did not, however, appear to differ from those of other specimens. As will be noted from Table VI, intensities were recorded from nearly all the reflections possible for copper radiation in the two zones examined.

Fourier Analysis and Structure-factor Calculations.—For the projection along the a axis the electron density was computed by the usual series at 450 points on the asymmetric unit, the b axis being subdivided into 60 parts of 0.148 \AA. and $c \sin \beta$ into 30 parts of 0.184 \AA. The summations were carried out by three-figure methods (Robertson, *J. Sci. Instr.*, 1948, **25**, 28) and the results were plotted on a scale of 5 cm. per \AA. by graphical interpolation from the summation totals. The resulting contour map for the $(0kl)$ zone, showing the positions finally assigned to the atom centres and the co-ordinate network, is given in Fig. 5. The projection along the $[101]$ axis was computed similarly, in this case the summation intervals being $b/60 = 0.148 \text{ \AA.}$, and $a \cos 1.6^\circ/30 = 0.170 \text{ \AA.}$

FIG. 5.



Contour map for a axis projection showing positions assigned to atoms. The approximate positions expected for the hydrogen atoms of the CH_2 groups, assuming normal bond lengths and angles, are indicated by the letters H.

The structure factors calculated from the final co-ordinates are listed in Table VI under "F, calc." A single composite atomic scattering curve was used, the f -values being as follows:

| | | | | | | | | | | |
|---|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| $\sin \theta$ ($\lambda = 1.54$)..... | 0.0 | 0.1 | 0.2 | 0.3 | 0.4 | 0.5 | 0.6 | 0.7 | 0.8 | 0.9 |
| f_e | 100 | 75 | 60 | 49 | 38 | 29 | 19 | 13 | 8.4 | 4.7 |

On this basis the oxygen contributions relative to the carbon contributions were weighted in the ratio of 1.5 to 1.0. This treatment is not very satisfactory, especially for a molecule with such a high proportion of oxygen as succinic acid, but it is sufficient for the present purpose.

The discrepancies between the measured and calculated structure factors, expressed as

$$\frac{\Sigma(|F, \text{obs.}| - |F, \text{calc.}|)}{\Sigma|F, \text{obs.}|} \times 100$$

are 22.5% for the $(0kl)$ zone and 21.7% for the (hkh) zone, giving an overall discrepancy of 22.0%. It is likely that these figures could be greatly improved by the use of separate f curves for oxygen and carbon, but as they stand they show that a considerable refinement of the previous work (Verweel and MacGillavry, *loc. cit.*) has been attained.