pronounced intermolecular interactions, a real difference in bond length is not plausible. Consequently the question of systematic error in the data set demands further study.

Computations for this work were carried out on an IBM 360/75 and a CDC 1604 electronic computer. Programs used, with accession numbers in the *World List of Crystallographic Computer Programs* (Shoemaker, 1966), are as follows:

| | | |
|---|---|---|
| Absorption correction | (*a*) | 523 |
| Statistical analysis of data | ORSTAT | 496 |
| Fourier synthesis | XFOUR(*b*) | 391 |
| Least-squares refinement | XFLS | 389 |
| Distances and angles | ORFFE(*c*) | 363 |
| Structure drawings | ORTEP | 387 |
| Rigid-body analysis | UCLATO1 | 232 |
| Structure factor table | EDIT | 393 |

(*a*) Modified for the CDC 1604 and generalized to a general ellipsoid by C. K. Johnson of this Laboratory.

(*b*) Modified for the IBM 360/75 by G. Brunton of this Laboratory.

(*c*) Modified for the IBM 360/75 by C. K. Johnson.

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# X-Ray Structure Analysis of Deca-*trans*-3, *trans*-7-dienedioic Acid

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(*Received* 17 *July* 1967)

The crystal structure of deca-*trans*-3, *trans*-7-diene-1,10-dioic acid, $C_{10}H_{14}O_4$, has been determined by three-dimensional X-ray diffraction analysis. The compound crystallizes in the centrosymmetric monoclinic space group $C2/c$. The unit cell contains four molecules and has the dimensions $a = 21{\cdot}74 \pm 0{\cdot}02$, $b = 4{\cdot}66 \pm 0{\cdot}01$, $c = 10{\cdot}40 \pm 0{\cdot}01$ Å; $\beta = 106° \pm 30'$. A trial structure was obtained by examination of the Patterson projections on the (010), (001) and (100) planes. The atomic parameters were refined by the differential synthesis method. The molecules are associated in rows in the crystal lattice. The rows in successive planes parallel to $(\bar{2}01)$ are oblique to one another. Noteworthy characteristics are the internal rotation angles about the single bonds C(2)–C(3) (−125·5°) and C(4)–C(5) (−1·6°).

## Introduction

The three-dimensional study of the crystal and molecular structure of deca-*trans*-3, *trans*-7-diene-1,10-dioic acid is part of a research program whose main goal is the determination of the conformational parameters that a number of important atomic groups (generally of aliphatic nature) assume in the crystal state. In the present case we are interested in determining the conformation of the group

$$(\text{-C-CH}_2\text{-CH}=\text{CH-CH}_2\text{-CH}_2\text{-CH}=\text{CH-CH}_2\text{-C-})$$

comprised between the two acidic functional groups of the molecule, and particularly the values of the internal rotation angles about the single bonds adjacent to the two double bonds. For angles of this type, in fact, very few data obtained by detailed X-ray analyses are available in the literature.

### Experimental and unit-cell parameters

Deca-*trans*-3,*trans*-7-diene-1,10-dioic acid

$$\begin{gathered}\mathrm{HOOC{-}CH_2{-}CH=} \\ \mathrm{CH{-}CH_2{-}CH_2{-}CH=CH{-}CH_2{-}COOH}\end{gathered}$$

crystallizes from water as needles elongated along **b**. From Weissenberg photographs taken with Cu *Kα* radiation the unit-cell parameters are found to be:

$$a=21\cdot74\pm0\cdot02,\ b=4\cdot66\pm0\cdot01,\ c=10\cdot40\pm0\cdot01\ \text{Å}; \quad \beta=106^\circ\pm30'\ .$$

The space group indicated by the systematic absences is the centrosymmetric $C2/c$ (which was subsequently verified by the structural analysis) with eight equivalent units per unit cell.

Four molecules per unit cell give a calculated density of 1·296 g.cm$^{-3}$ in good agreement with the experimental one (1·30 g.cm$^{-3}$). The molecules must be characterized by a centre of symmetry on the central $CH_2$–$CH_2$ bond or by a twofold axis.

Intensity data have been collected with Cu *Kα* radiation with use of multiple-film equi-inclination Weissenberg photographs about the *b* axis for layer lines 0 to 2. The zero layers about the *a* axis and about the *c* axis have been recorded to provide cross-correlation of the *b*-axis data. The number of independent reflexions observed was 335.

The intensities have been evaluated by eye-estimation, corrected for change of spot shape on upper layer equi-inclination photographs (Phillips, 1954) and reduced to structure amplitudes by a data reduction program written for an IBM 1620 computer (Damiani, De Santis, Giglio & Ripamonti, 1962). No absorption correction has been applied.

### Structure determination

The orientation of the molecules in the unit cell is determined from the three Patterson projections on the (001), (010), (100) planes. The Patterson peaks lie in lines parallel to the crystallographic directions $\frac{1}{2}\mathbf{a}+\frac{1}{2}\mathbf{b}+\mathbf{c}$ and $\frac{1}{2}\mathbf{a}-\frac{1}{2}\mathbf{b}+\mathbf{c}$.

Assuming angles and bond lengths reported in the literature for similar compounds a trial model is obtained which is in agreement with the Patterson peaks and gives a good molecular packing. For this model the reliability index $R$ [$R=\Sigma\,|F_o-F_c|/\Sigma\,|F_o|$] is 0·40 for all the observed reflexions.

A computation of the electron density projection on the (010) plane gives a good image of the proposed structure. The postulated molecular model for deca-*trans*-3-*trans*-7-dienedioic acid is characterized by a symmetry centre on the central $CH_2$–$CH_2$ bond.

This was confirmed later by the three-dimensional refinement of the structure.

### Refinement of the structure

The structure was refined by means of differential syntheses cycles and anisotropic temperature factors were introduced for each atom by comparison of the observed and calculated electron density curvatures. The scattering factors of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal for carbon and oxygen atoms (1955) and of McWeeny for hydrogen (1951) were used.

In the last stage of the refinement the positions of the hydrogen atoms were calculated (C–H = O–H = 1·10 Å) and introduced into the structure factor calculations with the same temperature factors as the atoms to which they are bonded.

The refinement was considered complete when the coordinate shifts were less than the corresponding standard deviations. The final value of the reliability index $R$ was 0·117 for the observed reflexions only.

The final atomic coordinates together with the corresponding standard deviations (Cruickshank, 1949) are reported in Table 1. In Table 2, observed and calculated structure factors are listed. The electron densities and their curvatures at the atomic positions are shown in Table 3.

The standard deviations of electron density and of the first derivatives are:

Table 1. *Final fractional atomic coordinates and their standard deviations* (Å)

| | $x/a$ | $y/b$ | $z/c$ | $\sigma(x)$ | $\sigma(y)$ | $\sigma(z)$ |
|---|---|---|---|---|---|---|
| C(1) | 0·1947 | 0·3953 | 0·3363 | 0·0064 | 0·0137 | 0·0060 |
| C(2) | 0·1524 | 0·5108 | 0·2038 | 0·0059 | 0·0116 | 0·0055 |
| C(3) | 0·0940 | 0·3272 | 0·1475 | 0·0048 | 0·0115 | 0·0048 |
| C(4) | 0·0768 | 0·2129 | 0·0257 | 0·0058 | 0·0123 | 0·0052 |
| C(5) | 0·0224 | 0·0300 | $0\cdot\overline{0428}$ | 0·0049 | 0·0113 | 0·0051 |
| O(1) | 0·2463 | 0·5331 | 0·3800 | 0·0040 | 0·0080 | 0·0038 |
| O(2) | 0·1804 | 0·1854 | 0·3938 | 0·0042 | 0·0085 | 0·0039 |
| H(1) | 0·278 | 0·441 | 0·473 | | | |
| H(2*a*) | 0·137 | 0·731 | 0·218 | | | |
| H(2*b*) | 0·180 | 0·525 | 0·131 | | | |
| H(3) | 0·063 | 0·284 | 0·213 | | | |
| H(4) | 0·109 | 0·265 | $0\cdot\overline{035}$ | | | |
| H(5*a*) | 0·040 | 0·174 | $0\cdot\overline{071}$ | | | |
| H(5*b*) | $0\cdot\overline{044}$ | 0·133 | $0\cdot\overline{136}$ | | | |

$$\sigma(\varrho) = 0{\cdot}087 \text{ e.Å}^{-3}$$
$$\sigma(A_h) = 0{\cdot}236 \text{ e.Å}^{-4}$$
$$\sigma(A_k) = 0{\cdot}179 \text{ e.Å}^{-4}$$
$$\sigma(A_l) = 0{\cdot}235 \text{ e.Å}^{-4}.$$

Fig. 1 shows an electron density projection on the plane (010).

### Description of the structure

The projection of the structure on the plane (010) is shown in Fig. 2.

The molecules are associated in rows in the crystal lattice by hydrogen bonds realized through a centre of symmetry in the same way as in the other dicarboxylic acids studied by us (Ganis & Martuscelli, 1966; Martuscelli, 1967; Martuscelli & Avitabile, 1967; Martuscelli, Benedetti, Ganis & Pedone, 1967; Martuscelli & Pedone, 1967) and by other authors (Housty & Hospital, 1964, 1965*a*,*b*). In this case however the molecular rows are not parallel but oblique. In fact the molecular rows contained in the ($\bar{2}01$) plane are oriented along the crystallographic direction $\frac{1}{2}\mathbf{a}+\frac{1}{2}\mathbf{b}+\mathbf{c}$, while the rows

Table 2. *Comparison between observed* ($F_o$) *and calculated* ($F_c$) *structure factors of deca-trans*-3,*trans*-7-*dienedioic acid*

| h k l | $F_o$ | $F_c$ | h k l | $F_o$ | $F_c$ | h k l | $F_o$ | $F_c$ | h k l | $F_o$ | $F_c$ | h k l | $F_o$ | $F_c$ | h k l | $F_o$ | $F_c$ | h k l | $F_o$ | $F_c$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2 0 0 | 54.3 | -50.0 | 4 0 6 | 21.0 | 16.1 | $\bar{1}$ 1 4 | 18.4 | 19.2 | 7 1 8 | 8.2 | 12.7 | 19 1 0 | 7.4 | -7.5 | $\bar{6}$ 2 5 | 21.0 | 20.6 | 22 2 0 | 11.2 | -11.9 |
| 4 0 0 | 11.2 | 11.1 | $\bar{4}$ 0 6 | 43.5 | -46.4 | 1 1 5 | 7.8 | -7.8 | 7 1 9 | 10.3 | + 9.2 | 19 1 1 | 8.2 | 5.5 | 6 2 6 | 30.4 | 29.2 | 0 2 1 | 1.8 | 5.9 |
| 8 0 0 | 21.4 | -22.9 | $\bar{6}$ 0 6 | 25.0 | -16.6 | $\bar{1}$ 1 5 | 29.4 | 27.4 | $\bar{7}$ 1 10 | 22.6 | -22.6 | $\bar{19}$ 1 2 | 12.6 | -13.8 | $\bar{6}$ 2 7 | 18.7 | 18.3 | 0 2 2 | 18.0 | -19.6 |
| 12 0 0 | 62.2 | 54.3 | 6 0 6 | 13.0 | -12.1 | 1 1 6 | 13.0 | 12.5 | 9 1 0 | 8.0 | -0.1 | $\bar{19}$ 1 3 | 8.7 | 11.2 | 6 2 8 | 8.2 | -6.3 | 0 2 3 | 16.4 | -20.5 |
| 14 0 0 | 30.1 | -29.6 | 8 0 6 | 8.4 | -8.2 | $\bar{1}$ 1 6 | 35.5 | -35.3 | 9 1 1 | 13.7 | 6.9 | $\bar{19}$ 1 4 | 7.5 | -10.4 | $\bar{6}$ 2 8 | 18.0 | -20.9 | 0 2 5 | 14.0 | -16.2 |
| 16 0 0 | 8.6 | 9.8 | $\bar{8}$ 0 6 | 8.0 | -4.6 | 1 1 7 | 10.0 | -9.5 | $\bar{9}$ 1 1 | 11.8 | 11.8 | 21 1 0 | 13.0 | -13.7 | 6 2 9 | 9.0 | 9.7 | 0 2 7 | 22.0 | 19.6 |
| 18 0 0 | 31.8 | -28.6 | 10 0 6 | 32.1 | 30.1 | 1 1 8 | 8.3 | -9.2 | 9 1 2 | 20.4 | -20.0 | $\bar{19}$ 1 5 | 6.5 | 8.3 | $\bar{6}$ 2 9 | 9.0 | -6.4 | 0 2 8 | 8.0 | -7.5 |
| 0 0 2 | 29.1 | 22.6 | $\bar{10}$ 0 6 | 7.3 | -8.9 | $\bar{1}$ 1 8 | 8.2 | 8.3 | $\bar{9}$ 1 2 | 6.4 | 7.2 | 21 1 5 | 10.3 | 8.4 | 8 2 0 | 48.8 | 52.0 | 0 2 11 | 9.4 | -11.0 |
| 2 0 2 | 64.1 | 62.7 | 12 0 6 | 18.6 | 14.9 | $\bar{1}$ 1 9 | 9.4 | -11.3 | 9 1 3 | 33.2 | 30.1 | $\bar{21}$ 1 6 | 12.6 | -15.2 | 8 2 1 | 12.4 | -7.8 | 0 2 13 | 7.0 | -1.2 |
| $\bar{2}$ 0 2 | 65.4 | 61.9 | $\bar{12}$ 0 6 | 25.3 | -25.1 | 1 1 $\bar{11}$ | 6.2 | -6.6 | 9 1 4 | 7.3 | 7.7 | $\bar{23}$ 1 1 | 6.2 | -5.1 | $\bar{8}$ 2 1 | 3.5 | -5.0 | | | |
| 4 0 2 | 43.3 | -43.2 | 14 0 6 | 6.5 | 8.2 | 3 1 0 | 77.9 | 86.5 | $\bar{9}$ 1 4 | 26.2 | 23.4 | $\bar{23}$ 1 2 | 10.6 | -12.7 | 8 2 2 | 6.2 | -4.5 | | | |
| $\bar{4}$ 0 2 | 135.7 | 138.3 | $\bar{16}$ 0 6 | 29.2 | -11.3 | 3 1 1 | 20.0 | -18.3 | 9 1 5 | 21.7 | 19.9 | $\bar{23}$ 1 3 | 7.3 | -6.4 | $\bar{8}$ 2 2 | 38.9 | 42.9 | | | |
| 6 0 2 | 44.7 | 46.7 | 18 0 6 | 12.2 | -8.1 | $\bar{3}$ 1 1 | 104.1 | 106.0 | $\bar{9}$ 1 5 | 12.0 | -12.6 | 2 2 0 | 27.9 | -31.4 | 8 2 3 | 14.6 | 14.6 | | | |
| $\bar{6}$ 0 2 | 106.2 | -110.2 | 20 0 6 | 8.0 | 6.3 | 3 1 2 | 55.0 | 58.0 | 9 1 6 | 31.2 | 29.6 | 2 2 1 | 14.5 | 23.7 | $\bar{8}$ 2 3 | 5.8 | 33.6 | | | |
| 8 0 2 | 25.0 | 15.2 | 0 0 8 | 12.4 | 11.3 | $\bar{3}$ 1 2 | 94.6 | -94.7 | $\bar{9}$ 1 6 | 49.2 | -42.0 | $\bar{2}$ 2 1 | 1.3 | 6.7 | $\bar{8}$ 2 4 | 32.2 | -36.1 | | | |
| $\bar{8}$ 0 2 | 37.2 | -30.6 | 2 0 8 | 20.2 | -20.1 | 3 1 3 | 40.0 | 37.0 | $\bar{9}$ 1 7 | 12.0 | -11.1 | 2 2 2 | 7.4 | 9.2 | 8 2 5 | 11.1 | 10.3 | | | |
| 10 0 2 | 10.0 | 8.4 | $\bar{4}$ 0 8 | 18.0 | 18.5 | $\bar{3}$ 1 3 | 42.3 | 38.1 | 11 1 0 | 6.1 | 9.0 | $\bar{2}$ 2 2 | 18.2 | 24.4 | $\bar{8}$ 2 5 | 12.9 | 16.1 | | | |
| $\bar{10}$ 0 2 | 16.3 | 8.6 | 6 0 8 | 12.1 | -11.2 | 3 1 4 | 5.4 | 2.6 | 11 1 1 | 21.7 | -18.2 | 2 2 3 | 9.5 | 10.2 | $\bar{8}$ 2 6 | 8.9 | -12.2 | | | |
| 12 0 2 | 37.2 | 30.9 | $\bar{6}$ 0 8 | 24.8 | -25.4 | $\bar{3}$ 1 4 | 28.8 | 24.8 | $\bar{11}$ 1 1 | 42.2 | -45.3 | $\bar{2}$ 2 3 | 33.5 | 38.3 | 8 2 7 | 7.0 | -6.2 | | | |
| $\bar{12}$ 0 2 | 26.8 | -22.5 | 8 0 8 | 19.6 | 15.8 | 3 1 5 | 29.3 | -26.2 | 11 1 2 | 18.7 | 16.6 | 2 2 4 | 22.0 | 23.3 | 8 2 8 | 9.0 | 9.1 | | | |
| 14 0 2 | 23.4 | -20.3 | $\bar{8}$ 0 8 | 16.5 | -16.5 | $\bar{3}$ 1 6 | 32.0 | 33.9 | $\bar{11}$ 1 2 | 17.5 | -13.0 | $\bar{2}$ 2 4 | 7.8 | 8.1 | 10 2 0 | 36.8 | -37.0 | | | |
| $\bar{14}$ 0 2 | 12.1 | 10.9 | 10 0 8 | 13.4 | 11.6 | 3 1 7 | 20.6 | -19.6 | 11 1 3 | 20.4 | -17.5 | 2 2 5 | 20.2 | -20.2 | 10 2 1 | 16.2 | 15.7 | | | |
| 16 0 2 | 9.1 | -3.7 | $\bar{10}$ 0 8 | 26.8 | -24.2 | $\bar{3}$ 1 8 | 19.6 | -17.2 | $\bar{11}$ 1 3 | 28.0 | -24.9 | $\bar{2}$ 2 5 | 19.6 | 19.6 | $\bar{10}$ 2 1 | 29.2 | 32.6 | | | |
| $\bar{16}$ 0 2 | 12.4 | 12.4 | 12 0 8 | 12.5 | 9.1 | $\bar{3}$ 1 10 | 8.2 | -10.1 | 11 1 4 | 24.0 | 23.9 | 2 2 6 | 7.8 | -6.9 | 10 2 2 | 18.0 | 20.4 | | | |
| 18 0 2 | 18.3 | 13.7 | $\bar{12}$ 0 8 | 13.4 | 17.4 | 5 1 0 | 22.5 | 13.9 | $\bar{11}$ 1 4 | 31.4 | -32.6 | $\bar{2}$ 2 6 | 12.8 | -11.7 | $\bar{10}$ 2 2 | 7.7 | -11.3 | | | |
| $\bar{18}$ 0 2 | 25.0 | -27.6 | 14 0 8 | 15.0 | -9.3 | 5 1 1 | 52.0 | -56.7 | 11 1 5 | 14.5 | 12.9 | 2 2 7 | 9.7 | -9.3 | 10 2 4 | 21.6 | 18.8 | | | |
| 20 0 2 | 18.8 | -18.3 | 16 0 8 | 12.4 | 9.6 | $\bar{5}$ 1 1 | 44.8 | 41.0 | 11 1 6 | 16.6 | -12.3 | $\bar{2}$ 2 7 | 18.0 | -16.3 | $\bar{10}$ 2 5 | 28.3 | -31.5 | | | |
| $\bar{20}$ 0 2 | 14.8 | -9.6 | $\bar{14}$ 0 8 | 8.3 | 10.9 | 5 1 2 | 25.9 | -25.4 | 13 1 0 | 10.9 | -10.9 | 2 2 8 | 10.0 | 8.7 | 10 2 8 | 8.4 | -8.1 | | | |
| $\bar{22}$ 0 2 | 6.5 | -7.2 | 2 0 10 | 17.8 | -17.8 | $\bar{5}$ 1 2 | 80.2 | 81.2 | 13 1 1 | 8.2 | 8.2 | $\bar{2}$ 2 8 | 28.0 | -28.4 | $\bar{12}$ 2 1 | 6.5 | -12.7 | | | |
| $\bar{24}$ 0 2 | 6.3 | 3.3 | 6 0 10 | 7.1 | 5.3 | 5 1 3 | 5.0 | 0.1 | $\bar{13}$ 1 1 | 13.4 | 15.5 | 4 2 0 | 44.8 | 35.5 | 12 2 2 | 8.5 | -7.9 | | | |
| 0 0 4 | 60.3 | -68.5 | $\bar{6}$ 0 10 | 7.3 | 7.9 | $\bar{5}$ 1 3 | 17.5 | -18.8 | $\bar{13}$ 1 4 | 9.0 | -7.5 | 4 2 1 | 13.5 | -17.2 | $\bar{12}$ 2 2 | 14.5 | -10.8 | | | |
| 2 0 4 | 108.1 | 110.5 | $\bar{8}$ 0 10 | 10.3 | -8.0 | 5 1 4 | 17.6 | 14.2 | 13 1 5 | 15.0 | -13.2 | $\bar{4}$ 2 1 | 2.6 | 4.4 | 12 2 3 | 5.6 | -7.0 | | | |
| $\bar{2}$ 0 4 | 45.0 | 47.2 | 10 0 10 | 19.3 | -22.6 | $\bar{5}$ 1 4 | 20.0 | -18.9 | 13 1 6 | 14.1 | 13.0 | 4 2 2 | 58.3 | 64.6 | $\bar{12}$ 2 3 | 7.5 | -7.4 | | | |
| 4 0 4 | 28.4 | -26.7 | $\bar{10}$ 0 10 | 10.0 | 12.9 | 5 1 5 | 10.3 | 8.6 | 13 1 7 | 11.6 | -9.2 | $\bar{4}$ 2 2 | 7.3 | -7.8 | 12 2 4 | 12.8 | -12.2 | | | |
| $\bar{4}$ 0 4 | 33.2 | -33.4 | 0 0 12 | 6.3 | -9.6 | $\bar{5}$ 1 6 | 30.0 | -30.4 | 13 1 8 | 8.6 | 5.8 | 4 2 3 | 27.3 | -29.4 | $\bar{12}$ 2 5 | 10.0 | 8.6 | | | |
| $\bar{6}$ 0 4 | 20.0 | 20.2 | $\bar{2}$ 0 12 | 4.6 | -4.0 | 5 1 7 | 8.0 | 7.5 | $\bar{13}$ 1 8 | 8.7 | -9.3 | $\bar{4}$ 2 3 | 21.9 | 25.8 | 12 2 6 | 8.0 | 6.0 | | | |
| 8 0 4 | 35.2 | 28.8 | 4 0 12 | 5.6 | -5.8 | $\bar{5}$ 1 7 | 20.4 | 21.4 | $\bar{13}$ 1 9 | 11.7 | -13.2 | 4 2 4 | 10.1 | -10.3 | $\bar{12}$ 2 6 | 18.6 | -21.1 | | | |
| $\bar{8}$ 0 4 | 42.0 | -42.3 | $\bar{8}$ 0 12 | 5.9 | -5.9 | 5 1 8 | 15.4 | 13.1 | 13 1 10 | 12.8 | -4.4 | $\bar{4}$ 2 4 | 40.0 | -33.8 | 14 2 1 | 8.0 | 7.5 | | | |
| $\bar{10}$ 0 4 | 26.2 | -24.2 | $\bar{10}$ 0 12 | 6.5 | 10.0 | $\bar{5}$ 1 8 | 10.8 | -13.0 | $\bar{13}$ 1 11 | 6.9 | -7.5 | 4 2 5 | 25.2 | 24.8 | $\bar{14}$ 2 2 | 15.5 | -20.3 | | | |
| 12 0 4 | 17.1 | -10.4 | $\bar{12}$ 0 12 | 7.8 | -12.6 | $\bar{5}$ 1 9 | 18.5 | 18.1 | 15 1 1 | 17.5 | -15.1 | 4 2 6 | 8.2 | 8.6 | 14 2 3 | 7.7 | -8.1 | | | |
| $\bar{12}$ 0 4 | 35.2 | -33.2 | $\bar{16}$ 0 12 | 5.6 | 6.7 | 5 1 10 | 8.0 | -7.9 | $\bar{15}$ 1 1 | 23.4 | 22.1 | $\bar{4}$ 2 6 | 6.9 | -9.6 | $\bar{14}$ 2 3 | 25.8 | 28.4 | | | |
| 14 0 4 | 49.1 | 43.3 | 0 2 0 | 12.3 | 18.4 | 7 1 0 | 7.8 | 12.2 | 15 1 2 | 17.5 | 15.2 | 4 2 7 | 10.4 | 12.9 | 14 2 4 | 9.0 | 8.5 | | | |
| $\bar{14}$ 0 4 | 17.9 | 18.1 | 0 4 0 | 7.4 | 17.1 | 7 1 1 | 79.9 | 84.1 | $\bar{15}$ 1 2 | 25.0 | -24.8 | 4 2 8 | 10.4 | 10.6 | $\bar{14}$ 2 4 | 13.3 | -15.8 | | | |
| 16 0 4 | 18.7 | -13.9 | 0 4 2 | 1.4 | 7.1 | $\bar{7}$ 1 1 | 28.2 | -28.1 | 15 1 4 | 8.2 | -6.1 | $\bar{4}$ 2 8 | 9.0 | 9.5 | 16 2 1 | 13.6 | -15.4 | | | |
| $\bar{16}$ 0 4 | 16.4 | -16.3 | 0 4 3 | 2.8 | 8.8 | 7 1 2 | 58.2 | 55.2 | $\bar{15}$ 1 5 | 6.3 | -10.2 | 4 2 9 | 11.8 | -11.5 | $\bar{16}$ 2 1 | 7.3 | 6.9 | | | |
| $\bar{18}$ 0 4 | 9.9 | 12.0 | 0 4 4 | 6.4 | 9.4 | $\bar{7}$ 1 2 | 32.0 | -34.5 | 15 1 7 | 7.3 | -6.8 | $\bar{4}$ 2 9 | 10.0 | 9.0 | $\bar{16}$ 2 2 | 8.8 | -6.9 | | | |
| 20 0 4 | 4.7 | -6.9 | 0 4 5 | 7.5 | 13.2 | 7 1 3 | 20.0 | -19.0 | 15 1 8 | 4.4 | 4.9 | 6 2 0 | 13.0 | 14.0 | $\bar{16}$ 2 3 | 12.1 | -14.0 | | | |
| $\bar{20}$ 0 4 | 13.0 | -14.7 | 1 1 0 | 2.3 | 3.3 | $\bar{7}$ 1 3 | 52.4 | 53.8 | 17 1 2 | 21.8 | -21.8 | 6 2 1 | 8.2 | -12.2 | $\bar{16}$ 2 4 | 24.5 | -23.9 | | | |
| $\bar{22}$ 0 4 | 8.8 | -7.8 | 1 1 1 | 28.2 | -29.8 | 7 1 4 | 8.0 | 7.3 | 17 1 3 | 13.3 | -12.5 | 6 2 2 | 5.6 | 6.8 | $\bar{16}$ 2 7 | 7.3 | 5.7 | | | |
| $\bar{24}$ 0 4 | 5.5 | -7.0 | $\bar{1}$ 1 1 | 49.0 | -49.7 | $\bar{7}$ 1 4 | 53.0 | -52.0 | $\bar{17}$ 1 3 | 12.4 | 11.3 | $\bar{6}$ 2 2 | 3.3 | -2.5 | 18 2 0 | 9.0 | -8.5 | | | |
| $\bar{26}$ 0 4 | 8.0 | 8.1 | 1 1 2 | 34.6 | 36.2 | 7 1 5 | 15.6 | -15.6 | 17 1 4 | 7.0 | 5.4 | 6 2 3 | 5.4 | 5.3 | $\bar{18}$ 2 1 | 7.0 | -4.0 | | | |
| 0 0 6 | 20.0 | 13.4 | $\bar{1}$ 1 2 | 74.4 | 84.3 | $\bar{7}$ 1 5 | 23.8 | 22.5 | $\bar{17}$ 1 4 | 15.0 | -14.6 | $\bar{6}$ 2 3 | 10.0 | -10.6 | $\bar{18}$ 2 3 | 11.0 | 9.4 | | | |
| 2 0 6 | 16.8 | -14.1 | 1 1 3 | 54.0 | -56.9 | $\bar{7}$ 1 6 | 12.4 | 11.3 | 17 1 6 | 6.5 | 6.8 | 6 2 4 | 34.2 | 37.5 | 20 2 0 | 11.5 | 8.6 | | | |
| $\bar{2}$ 0 6 | 36.0 | 35.8 | 1 1 4 | 5.3 | 10.0 | 7 1 7 | 19.6 | 18.7 | 17 1 7 | 8.0 | 5.2 | $\bar{6}$ 2 4 | 44.0 | 43.5 | $\bar{20}$ 2 1 | 8.2 | 8.2 | | | |

Table 3. *Peak heights and curvatures*

Values in parentheses are from $F_c$ differential synthesis.

| | $\varrho$ (e.Å$^{-3}$) obs. (calc.) | $-A_{hh}$ (e.Å$^{-5}$) obs. (calc.) | $-A_{kk}$ (e.Å$^{-5}$) obs. (calc.) | $-A_{ll}$ (e.Å$^{-5}$) obs. (calc.) | $A_{hk}$ (e.Å$^{-5}$) obs. (calc.) | $A_{hl}$ (e.Å$^{-5}$) obs. (calc.) | $A_{kl}$ (e.Å$^{-5}$) obs. (calc.) |
|---|---|---|---|---|---|---|---|
| C(1) | 5·73<br>(5·93) | 36·6<br>(37·2) | 13·0<br>(12·8) | 39·3<br>(41·7) | 0·40<br>(0·41) | 0·25<br>(0·71) | −0·83<br>(−0·70) |
| C(2) | 6·02<br>(5·94) | 39·8<br>(39·2) | 15·4<br>(15·4) | 42·8<br>(43·4) | −0·03<br>(−0·04) | 0·65<br>(1·04) | 0·00<br>(−0·07) |
| C(3) | 6·28<br>(6·30) | 49·0<br>(48·2) | 15·5<br>(16·3) | 48·7<br>(49·1) | 0·09<br>(0·04) | 1·19<br>(1·39) | 0·05<br>(0·09) |
| C(4) | 5·94<br>(6·09) | 40·8<br>(41·0) | 14·5<br>(17·0) | 45·2<br>(46·4) | −0·05<br>(−0·05) | 0·77<br>(1·00) | 0·10<br>(0·07) |
| C(5) | 6·18<br>(6·32) | 47·3<br>(47·6) | 15·8<br>(18·7) | 45·6<br>(45·5) | −0·21<br>(−0·17) | 1·48<br>(1·36) | −0·01<br>(−0·04) |
| O(1) | 8·43<br>(8·54) | 59·1<br>(59·0) | 22·3<br>(22·8) | 61·8<br>(62·8) | −0·27<br>(−0·10) | 1·01<br>(1·37) | 0·13<br>(−0·02) |
| O(2) | 8·25<br>(8·18) | 55·6<br>(53·4) | 21·0<br>(21·9) | 59·1<br>(58·3) | 0·04<br>(0·13) | 0·85<br>(1·09) | −0·32<br>(−0·32) |

contained in the planes $(\bar{4}02)$ and $(40\bar{2})$ are oriented along the crystallographic direction $\frac{1}{2}\mathbf{a}-\frac{1}{2}\mathbf{b}+\mathbf{c}$ (see Fig. 2).

The most significant intermolecular contact distances between rows contained in the $(\bar{4}02)$, $(40\bar{2})$ and $(\bar{2}01)$ planes are shown in Fig. 2.

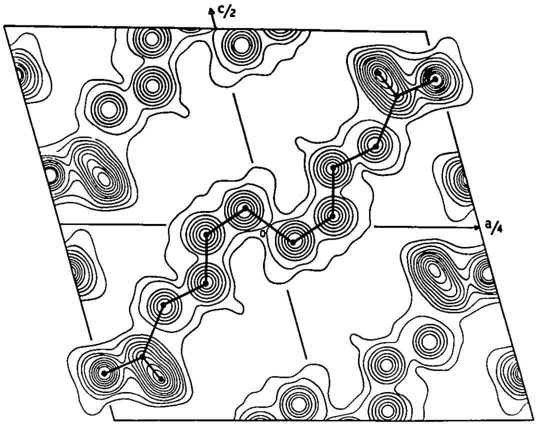


Fig. 1. Projection of the electron density onto the (010) plane. Levels are drawn at interval of 1 e.Å$^{-2}$ starting at zero.

The packing is dominated by H---H, H---O, and H---C interactions. All the C---C distances are larger than 3·90 Å.

The projection of the structure onto the (001) plane is shown in Fig. 3. In order to clarify the packing between molecular rows contained in the $(\bar{2}01)$ plane the rows contained in the $(\bar{4}02)$ and $(40\bar{2})$ have been omitted. The packing is characterized by interactions H---O, H---C and C---O. The contact distances C---C are again all larger than 3·90 Å.

The length of the hydrogen bridges connecting the carboxyl groups of adjoining molecules in the $\frac{1}{2}\mathbf{a}+\frac{1}{2}\mathbf{b}+\mathbf{c}$ or $\frac{1}{2}\mathbf{a}-\frac{1}{2}\mathbf{b}+\mathbf{c}$ direction is 2·620 ± 0·01 Å.

## Molecular vibration analysis

Table 4 shows the coefficients $b_{ij}$ of the temperature factor in the form:

$$\exp[-(b_{11}h^2+b_{22}k^2+b_{33}l^2+b_{12}hk+b_{13}hl+b_{23}kl)]\ .$$

The values of the three semi-axes of the ellipsoid of thermal vibration for each C and O atom are given in Table 5.

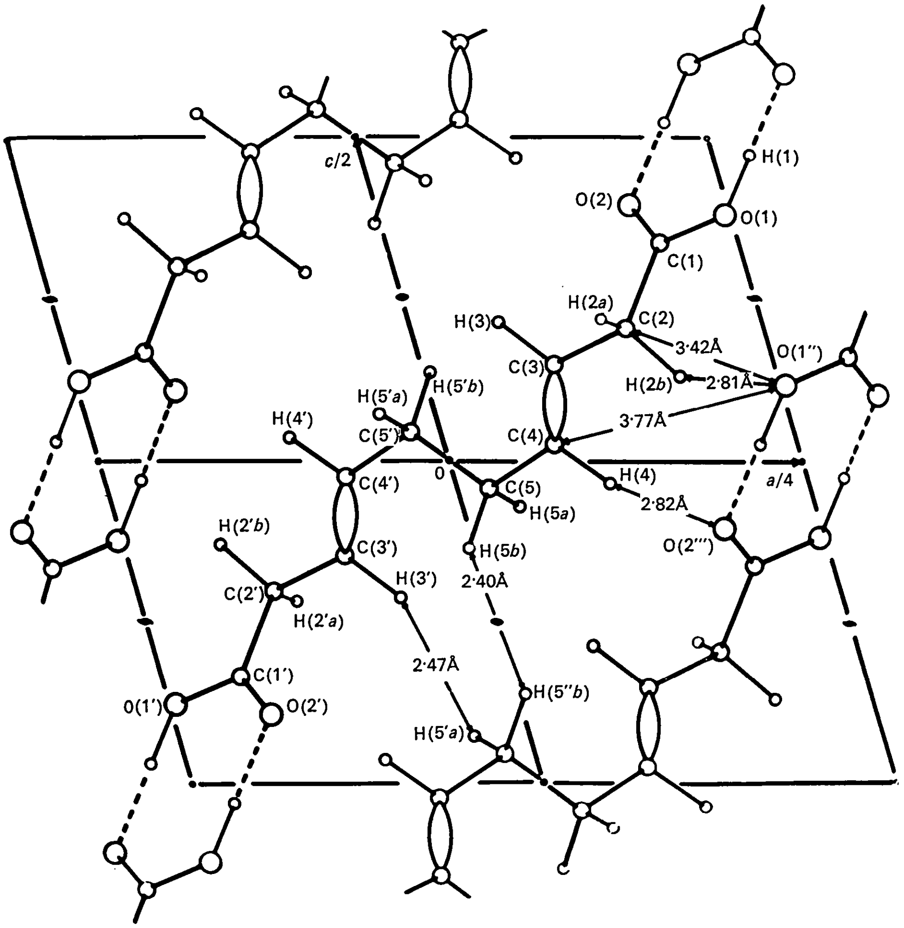


Fig. 2. Projection of the structure along [010].

These parameters have been calculated with a program written for an Elea 6001 computer (Coda, 1966). The atoms of the independent structural unit of deca-*trans*-3,*trans*-7-dienedioic acid have a larger amplitude of vibration in a direction normal to the **b** axis and parallel to the (010) plane.

## Molecular conformation

Table 6 gives the conformational parameters of deca-*trans*-3,*trans*-7-dienedioic acid with the calculated values of their estimated standard deviations (Cruickshank & Robertson, 1953) and with reference to Fig. 4.

The information obtained regarding the internal rotation angles about the single bonds C–C adjacent to the two double bonds C=C is particularly interesting. From nuclear magnetic resonance, microwave spectroscopy and electron diffraction studies, on relatively small unsaturated molecules in the vapour or liquid state, these internal rotation angles have been found to assume preferentially values near to 0°, 120°, −120°. The conformations corresponding to $\sigma=0°$ and $\sigma=\pm 120°$ are approximately iso-energetic unless strong repulsions arise, owing to the presence of bulky substituents (Kilb, Lin & Wilson, 1967; Herschbach & Krisher, 1958; Abraham & Pople, 1960; Bothner-By, Naar-Colin & Gunther, 1962).

From the few available X-ray diffraction data the skew conformation seems to be favoured in the solid state. The internal rotation angles about single bonds

Table 4. *Final temperature parameters*

Temperature factors are given in the form: $\exp\left[-(b_{11}h^2+b_{12}hk+b_{13}hl+b_{22}k^2+b_{23}kl+b_{33}l^2)\right.$.

| | $b_{11}$ | $b_{12}$ | $b_{13}$ | $b_{22}$ | $b_{23}$ | $b_{33}$ |
|---|---|---|---|---|---|---|
| C(1) | 0·0022 | 0·0000 | 0·0018 | 0·0316 | 0·0000 | 0·0095 |
| C(2) | 0·0030 | 0·0000 | 0·0018 | 0·0321 | 0·0000 | 0·0095 |
| C(3) | 0·0023 | 0·0000 | 0·0019 | 0·0349 | 0·0000 | 0·0088 |
| C(4) | 0·0030 | 0·0000 | 0·0019 | 0·0363 | 0·0000 | 0·0092 |
| C(5) | 0·0024 | 0·0000 | 0·0018 | 0·0330 | 0·0000 | 0·0080 |
| O(1) | 0·0020 | 0·0000 | 0·0018 | 0·0325 | 0·0000 | 0·0087 |
| O(2) | 0·0025 | 0·0000 | 0·0019 | 0·0352 | 0·0000 | 0·0089 |

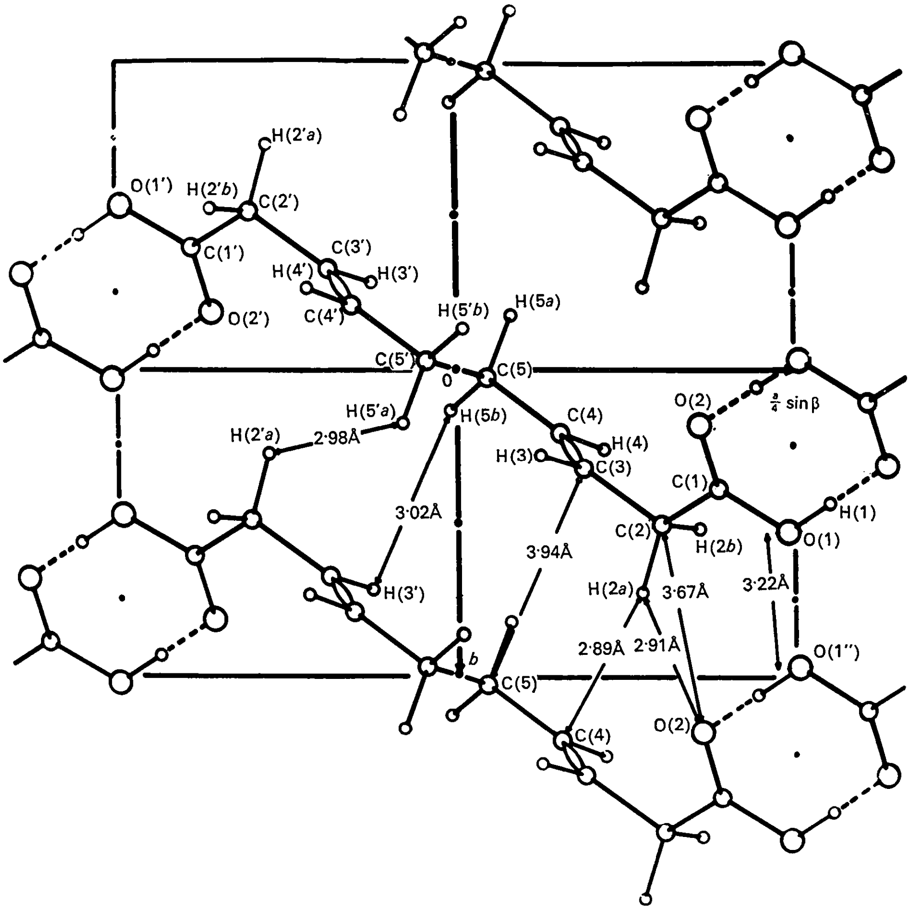


Fig. 3. Projection of the structure along [001]. Molecular rows contained in the ($\bar{4}02$) and ($40\bar{2}$) planes have been omitted.

adjacent to a double bond have been found to be in any case near to 130° (for instance, in geranylamine hydrochloride $\sigma = 131°$ (Bateman & Jeffrey, 1943), in oleic acid $\sigma = 126°$ and 129° (Abrahamsson & Nahringbauer, 1962) in several polyalkenes of the type $-[CH=CH-(CH_2)_x-]_n$ $\sigma = 130°$ (Natta & Bassi, 1967), in *trans*-2-butene-1,4-dicarboxylic acid $\sigma = 130°$ (Ganis & Martuscelli, 1966). Only for $\alpha$-guttapercha has a model been proposed in which these angles are 120 and 0° alternately (Natta, Corradini & Porri, 1956).

In deca-*trans*-3-*trans*-7-dienedioic acid the internal rotation angles are −125·5° about the C(2)–C(3) bond

Table 5. *Dimensions and orientations of the thermal vibration ellipsoids relative to the atoms of the independent structural unit of deca-trans-*3, *trans-*7*-dienedioic acid*

The length of $U_1, U_2, U_3$ semi-axes is given in Å² × 4.

The mean square displacement $\sqrt{U_i/2\pi^2}$ in the $U_1, U_2, U_3$ directions for each atom is given in Å.

In the last columns the direction cosines of the semi-axes for each atom referred to an orthogonal coordinate system are given. In this system the $z$ axis is coincident with **c** and $y$ with **b**.

| | $U_i$ (Å² × 4) | $\sqrt{U_i/2\pi^2}$ (Å) | Angles formed by $U_i$ semi-axes with crystallographic axes **a** (°) | **b** (°) | **c** (°) | Direction cosines of $U_i$ semi-axes referred to a orthogonal coordinate system | | |
|---|---|---|---|---|---|---|---|---|
| C(1) | $U_1$=3·58 | 0·21 | 55·1 | 90·0 | 50·9 | 0·7764 | 0·0000 | 0·6302 |
| | $U_2$=4·24 | 0·23 | 145·1 | 90·0 | 39·1 | −0·6303 | 0·0000 | 0·7764 |
| | $U_3$=2·74 | 0·19 | 90·0 | 0·0 | 90·0 | 0·0000 | 1·0000 | 0·0000 |
| C(2) | $U_1$=3·74 | 0·22 | 80·3 | 90·0 | 25·7 | 0·4339 | 0·0000 | 0·9009 |
| | $U_2$=5·59 | 0·27 | 9·7 | 90·0 | 115·7 | 0·9009 | 0·0000 | −0·4339 |
| | $U_3$=2·79 | 0·19 | 90·0 | 0·0 | 90·0 | 0·0000 | 1·0000 | 0·0000 |
| C(3) | $U_1$=3·47 | 0·21 | 75·2 | 90·0 | 30·8 | 0·5115 | 0·0000 | 0·8593 |
| | $U_2$=4·21 | 0·23 | 14·8 | 90·0 | 120·8 | 0·8593 | 0·0000 | −0·5115 |
| | $U_3$=3·03 | 0·19 | 90·0 | 0·0 | 90·0 | 0·0000 | 1·0000 | 0·0000 |
| C(4) | $U_1$=3·65 | 0·21 | 82·9 | 90·0 | 23·0 | 0·3915 | 0·0000 | 0·9202 |
| | $U_2$=5·53 | 0·26 | 7·0 | 90·0 | 113·0 | 0·9202 | 0·0000 | −0·3915 |
| | $U_3$=3·15 | 0·20 | 90·0 | 0·0 | 90·0 | 0·0000 | 1·0000 | 0·0000 |
| C(5) | $U_1$=3·18 | 0·20 | 83·4 | 90·0 | 22·6 | 0·3838 | 0·0000 | 0·9234 |
| | $U_2$=4·37 | 0·23 | 6·6 | 90·0 | 112·6 | 0·9234 | 0·0000 | −0·3838 |
| | $U_3$=2·87 | 0·19 | 90·0 | 0·0 | 90·0 | 0·0000 | 1·0000 | 0·0000 |
| O(1) | $U_1$=3·32 | 0·21 | 54·0 | 90·0 | 51·9 | 0·7876 | 0·0000 | 0·6162 |
| | $U_2$=3·77 | 0·22 | 144·0 | 90·0 | 38·0 | −0·6162 | 0·0000 | 0·7876 |
| | $U_3$=2·82 | 0·19 | 90·0 | 0·0 | 90·0 | 0·0000 | 1·0000 | 0·0000 |
| O(2) | $U_1$=3·52 | 0·21 | 79·2 | 90·0 | 26·7 | 0·4500 | 0·0000 | 0·8930 |
| | $U_2$=4·58 | 0·24 | 10·7 | 90·0 | 116·7 | 0·8930 | 0·0000 | −0·4500 |
| | $U_3$=3·06 | 0·20 | 90·0 | 0·0 | 90·0 | 0·0000 | 1·0000 | 0·0000 |

Table 6. *Intramolecular distances and angles with standard deviations*

| | | |
|---|---|---|
| Distances | | |
| C(1)–O(1) | 1·263 Å | $1·0 \times 10^{-2}$ Å |
| C(1)–O(2) | 1·230 | $1·3 \times 10^{-2}$ |
| C(1)–C(2) | 1·528 | $1·0 \times 10^{-2}$ |
| C(2)–C(3) | 1·509 | $1·1 \times 10^{-2}$ |
| C(3)–C(4) | 1·329 | $0·9 \times 10^{-2}$ |
| C(4)–C(5) | 1·472 | $1·1 \times 10^{-2}$ |
| C(5)–C(5′) | 1·516 | $0·7 \times 10^{-3}$ |
| O(2)---O(1$^{V}$) | 2·620 | $1·0 \times 10^{-2}$ |
| Angles | | |
| O(1)–C(1)–C(2) | 113·5° | $8·9 \times 10^{-1}$° |
| O(1)–C(1)–O(2) | 123·2 | $6·6 \times 10^{-1}$ |
| O(2)–C(1)–C(2) | 123·3 | $7·3 \times 10^{-1}$ |
| C(1)–C(2)–C(3) | 112·5 | $8·2 \times 10^{-1}$ |
| C(2)–C(3)–C(4) | 125·6 | $5·3 \times 10^{-1}$ |
| C(3)–C(4)–C(5) | 132·1 | $5·7 \times 10^{-1}$ |
| C(4)–C(5)–C(5′) | 112·5 | $5·3 \times 10^{-1}$ |
| Dihedral angles | | |
| O(1)–C(1)–C(2) $\wedge$ C(1)–C(2)–C(3) | −174·8 | $10·0 \times 10^{-1}$ |
| C(1)–C(2)–C(3) $\wedge$ C(2)–C(3)–C(4) | −125·5 | $10·0 \times 10^{-1}$ |
| C(2)–C(3)–C(4) $\wedge$ C(3)–C(4)–C(5) | 180·0 | $10·0 \times 10^{-1}$ |
| C(3)–C(4)–C(5) $\wedge$ C(4)–C(5)–C(5′) | −1·6 | $10·0 \times 10^{-1}$ |
| C(4)–C(5)–C(5′) $\wedge$ C(5)–C(5′)–C(4′) | 180 | |

(double bond eclipsed to a hydrogen atom) and −1·6° about the C(4)–C(5) bond (double bond eclipsed to the methylenic group).

The numerical values of the internal rotation angles have been measured following Natta, Corradini & Bassi (1961).

As far as we know, this is the first observation of a *cis* conformation in the crystal state for this kind of bond. The greater frequency of observation of skew conformations in the crystal state is probably not due to a difference of energy relative to the *cis* conformation in the isolated molecule, but it is probably due to the effect of the crystal field.

The rotation angles around the C(1)–C(2) and C(3)–C(4) bonds are respectively −174·8 and 180·0°.

The molecule is characterized by a symmetry centre on the C(5)–C(5′) bond. The length of the double bond C(3)=C(4) (1·329 Å) is consistent with the values reported in the literature.

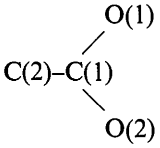
The bond C(4)–C(5) (1·472 Å) is shorter than a single bond. The bond angle C(3)–C(4)–C(5) (132·1°) is larger than the theoretical value of 120°. This is probably due to the repulsion between the hydrogen atoms H(5′*a*), H(5′*b*) and the hydrogen atom H(3). The intramolecular contact distances H(3)···H(5′*b*), H(3)···H(5′*a*) and H(2*b*)···H(4) are respectively 2·35, 2·38 and 2·32 Å.

The equation of the least-squares mean plane passing through the atoms C(2)–C(3)=C(4)–C(5), calculated by the method of Schomaker, Waser, Marsh & Bergman (1959), attributing a unitary weight factor to all four atoms, is $-9{\cdot}95x+3{\cdot}81y-2{\cdot}18z=-1{\cdot}3\times10^{-2}$.

This equation is referred to the axes of the unit cell, $x,y,z$ being fractional coordinates. The four atoms are, within experimental error, in the plane. In fact, the deviations from planarity are less than the corresponding atomic standard deviations

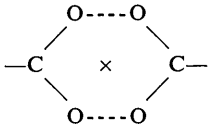
$$\begin{array}{ll} C(2) & -2{\cdot}48\times10^{-3}\ \text{Å} \\ C(3) & \phantom{-}2{\cdot}06\times10^{-3} \\ C(4) & \phantom{-}3{\cdot}22\times10^{-3} \\ C(5) & -2{\cdot}79\times10^{-3}\,. \end{array}$$

In the same way we have calculated the equations of the least-squares mean planes passing through the



$$\begin{array}{l} \quad O(1) \\ \quad / \\ C(2){-}C(1) \\ \quad \backslash \\ \quad O(2) \end{array}$$

group and through two carboxylic groups



repeated by the symmetry centre in (0·25, 0·25, 0·50); they are respectively:

$$-13{\cdot}43x+2{\cdot}82y+6{\cdot}80z=0{\cdot}78$$
$$-13{\cdot}24x+2{\cdot}78y+6{\cdot}96z=0{\cdot}87\,.$$

The deviations of the atoms from the two planes are less than the standard deviations. The two groups are planar within experimental errors.

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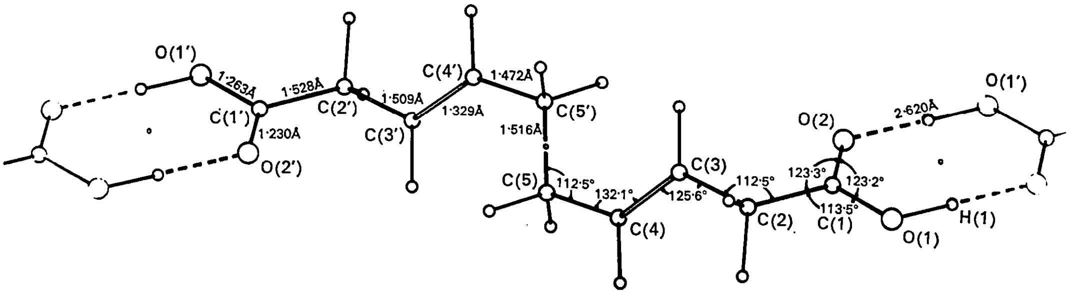

Fig.4. Molecular model of deca-*trans*-3, *trans*-7-dienedioic acid.

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# High-Pressure $KAlSi_3O_8$, an Aluminosilicate with Sixfold Coordination

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Synthetic potassium feldspar $KAlSi_3O_8$ (sanidine) undergoes a polymorphic transition at 120 kilobars pressure and 900°C into the hollandite structure, space group $I4/m$ with $a=9{\cdot}38$ and $c=2{\cdot}74$ Å. Aluminum and silicon are randomized in octahedral positions, and the average bond length ($\frac{1}{4}$Al, $\frac{3}{4}$Si)–O is 1·80 Å. The density increased from 2·55 to 3·84 g.cm$^{-3}$. It is probable that feldspars are transformed to the high-density hollandite structure in the transition zone within the earth's mantle.

The predominant phases at depths between 200 and 900 kilometers in the earth's mantle are generally believed to be denser high-pressure polymorphs, or disproportionation products, of common silicate minerals (Birch, 1952). A number of temperature–pressure studies of the alkali-metal aluminosilicates, as well as the corresponding germanates which transform much more easily, have been reported. The relationships albite–nepheline–jadeite are well documented below 25 kilobars (kb) (Robertson, Birch & McDonald, 1957; Dachille & Roy, 1962), but above 100 kb a different series of transformations take place. Both germanian nepheline and jadeite are altered to a new $NaAlGeO_4$ phase (Ringwood & Major, 1967), which proves to have the orthorhombic calcium ferrite structure (Reid, Wadsley & Ringwood, 1967). $NaAlSiO_4$ could be expected to follow suit at even higher pressures.

Potassium is too large an ion to occupy sites in this particular structure (Reid, Wadsley & Sienko, 1968). Instead the closely packed hollandite arrangement (Byström & Byström, 1951) typified by the compound $KAlTi_3O_8$ (Bayer & Hoffmann, 1966) was considered to be likely for high-pressure potassium-bearing minerals, particularly for the feldspars orthoclase or sanidine having the same stoichiometry, $KAlSi_3O_8$ (Reid, Wadsley & Ringwood, 1967). The germanium analogue $KAlGe_3O_8$ was recently described (Kume, Matsumoto & Koizumi, 1966) and we are now able to report that sanidine itself transforms at 120 kb and 900°C to the hollandite structure. It is therefore the first aluminosilicate with Al and Si both octahedral, and indeed the first oxygen compound other than very high pressure $SiO_2$ (stishovite) and $SiP_2O_7$ (Levi & Peyronel, 1935) with silicon having a coordination number of six.

Crystalline synthetic sanidine, $KAlSi_3O_8$, moistened with a trace of water, was allowed to react at 120 kb and 900°C for several minutes in a device consisting

Table 1. *Compounds with the tetragonal hollandite structure containing aluminum*

Space group $I4/m$.

| | Lattice parameters | | Average octahedral bond length | Alkali metal–oxygen bond length | Reference |
|---|---|---|---|---|---|
| | $a$ | $c$ | | | |
| $KAlSi_3O_8$ | 9·38 Å | 2·74 Å | 1·80 Å | 2·78 Å | This paper |
| $KAlGe_3O_8$ | 9·72 | 2·86 | 1·87 | 2·88 | Kume, Matsumoto & Koizumi (1966) |
| $K_{0.80}Al_{0.80}Ti_{3.20}O_8$ | 10·07 | 2·94 | | | Bayer & Hoffmann (1966) |
| $K_{0.89}Al_{0.89}Ti_{3.11}O_8$ | 10·06 | 2·94 | | | |
| $KAlTi_3O_8$ | 10·04 | 2·94 | 1·93 | 2·97 | |
| $RbAlTi_3O_8$ | 10·10 | 2·94 | 1·94 | 2·99 | |