Acta Cryst. (1967). **23**, 747

Refinement of the Crystal and Molecular Structure of *meso*-α,α′-Dimethylglutaric Acid

BY EZIO MARTUSCELLI, ETTORE BENEDETTI, PAOLO GANIS AND CARLO PEDONE

Sez. VII, *Centro Nazionale di Chimica delle Macromolecole, Istituto Chimico, Università di Napoli, Via Mezzocannone* 4, *Napoli, Italy*

(*Received* 3 *March* 1967)

The crystal and molecular structure of *meso*-α,α′-dimethylglutaric acid has been determined by three-dimensional X-ray crystallographic methods. Bond lengths and angles have been calculated and are consistent with the currently accepted values for aliphatic compounds. The crystals are triclinic with space group $P\bar{1}$ and cell dimensions $a=9{\cdot}90$, $b=8{\cdot}35$, $c=7{\cdot}32$ Å; $\alpha=119°$; $\beta=72°$; $\gamma=126°$.

Introduction

The determination of the crystal structure of *meso*-α,α′-dimethylglutaric acid (DMGA) forms part of a program of research in progress in our laboratory on the structure of low molecular weight model compounds in which atomic groups may be found similar to those present in high molecular weight compounds. A preliminary report on the structure of DMGA has already been given (Ganis, Pedone & Temussi, 1964). The manner in which the molecules are associated in rows in the crystal lattice was established from *h*0*l* and *hk*0 intensities. This paper describes the refinement of the approximate structure with the use of three-dimensional data and discusses the conclusions of a molecular vibration analysis.

Experimental

DMGA crystallizes from water. The cell dimensions were determined from Weissenberg photographs of a crystal mounted about the *a*, *b* and *c* axes.

Crystal data

meso-α,α′-Dimethylglutaric acid (DMGA)
$C_7O_4H_{12}$, $M=160$, m.p. 127 °C
Triclinic,
$a=9{\cdot}90\pm0{\cdot}02$, $b=8{\cdot}35\pm0{\cdot}02$, $c=7{\cdot}32\pm0{\cdot}02$ Å;
$\alpha=119°\pm30'$, $\beta=72°\pm1°$, $\gamma=126°\pm30'$.
$d_{RX}=1{\cdot}24$ g.cm^{-3} (with $Z=2$), $d_{exp}=1{\cdot}24$ g.cm^{-3}.
Space group is $P\bar{1}$ as confirmed later in the analysis.

Three-dimensional intensity data ($0kl\cdots5kl$, $hk0\cdots hk4$) were recorded by the equi-inclination method with Cu $K\alpha$ radiation. The intensities of 1319 observed reflexions were estimated visually. Lorentz and polarization corrections were applied in the usual way and the reflexions were placed on a common scale by the method of Rollett & Sparks (1960). No absorption corrections were applied.

Three-dimensional refinement

Three-dimensional refinement of the structure was carried out by the differential synthesis method. The scat-

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

	x/*a*	*y*/*b*	*z*/*c*	σ(*x*)	σ(*y*)	σ(*z*)
C(1)	0·1740	0·2695	0·1293	0·0025	0·0027	0·0036
C(2)	0·3133	0·5245	0·2286	0·0025	0·0029	0·0038
C(3)	0·4009	0·5348	0·3738	0·0031	0·0036	0·0052
C(4)	0·2495	0·6802	0·3440	0·0025	0·0027	0·0037
C(5)	0·1750	0·6933	0·2031	0·0026	0·0027	0·0037
C(6)	0·2964	0·7847	0·0454	0·0048	0·0047	0·0059
C(7)	0·0969	0·8268	0·3313	0·0027	0·0027	0·0037
O(1)	0·0303	0·2298	0·1930	0·0020	0·0023	0·0031
O(2)	0·2242	0·1739	0·9685	0·0022	0·0024	0·0032
O(3)	0·1830	0·0137	0·4669	0·0023	0·0026	0·0039
O(4)	0·9483	0·7490	0·3046	0·0022	0·0025	0·0037
H(2)	0·400	0·576	0·108			
H(3)*a*	0·421	0·671	0·519			
H(3)*b*	0·324	0·388	0·402			
H(3)*c*	0·519	0·551	0·302			
H(4)*a*	0·158	0·633	0·464			
H(4)*b*	0·350	0·839	0·428			
H(5)	0·071	0·535	0·123			
H(6)*a*	0·325	0·945	0·092			
H(6)*b*	0·409	0·789	0·036			
H(6)*c*	0·241	0·684	0·894			
H(1)	0·122	0·013	0·903			
H(7)	0·894	0·856	0·311			

tering factors of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal for C and O (1955) and of McWeeny for H (1951) were used. After several cycles with the same isotropic temperature factor for all the atoms ($B=4$ Å^2), the R index ($\Sigma \, ||F_o|-|F_c|| / \Sigma \, |F_o|$) was 0·19, not including the contribution due to the hydrogen atoms. At this stage, individual anisotropic temperature factors were assigned to the C and O atoms and, after few cycles the R value dropped to 0·128.

The contribution of the hydrogen atoms was included assuming calculated positions with C–H and O–H length equal to 1·08 Å with the same isotropic factor $B=4{\cdot}0$ Å^2. The refinement was stopped when the average shift in the atomic coordinates in terms of their standard deviations was less than $0{\cdot}5\sigma$, except

Table 2. *Comparison of observed and calculated structure factors*

Table 2 (*cont.*)

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	h k l	F_o	F_c
4 5 3	2.5	2.3	5 5 1	6.8	- 7.3	5 7 3	4.0	- 3.8	6 5 3	3.7	3.5	7 0 4	4.7	4.4	9 1 0	2.7	2.6	10 5 4	0.8	0.7			
4 5 3	1.3	1.3	5 2 1	4.6	- 3.6	5 7 4	3.3	2.9	6 6 3	11.4	-11.3	7 0 4	4.2	- 4.0	9 2 0	2.8	2.8	10 5 4	0.6	- 0.7			
4 5 3	4.1	4.2	5 2 2	4.5	3.6	5 7 5	1.9	2.3	6 7 3	0.9	1.0	7 1 4	0.8	0.6	9 3 0	6.4	- 6.5	11 2 0	0.4	- 0.4			
4 5 3	1.9	- 1.5	5 2 2	12.9	14.0	5 7 6	5.3	- 4.9	6 8 3	3.7	3.5	7 1 4	2.3	- 2.3	9 4 0	9.9	-10.1	11 3 0	2.4	2.4			
4 5 4	1.9	1.6	5 3 2	6.0	6.2	5 8 0	1.3	- 1.3	6 9 3	0.9	- 0.8	7 1 4	2.9	2.3	9 5 0	5.3	5.4	11 5 0	1.1	- 1.0			
4 5 4	4.1	- 3.9	5 3 3	1.5	1.6	5 8 1	2.0	- 1.9	6 10 3	2.0	- 1.9	7 2 4	0.6	0.5	9 6 0	3.3	3.3	11 6 0	1.9	1.9			
4 5 5	1.1	1.0	5 2 3	0.9	1.0	5 8 1	2.2	2.0	6 0 4	7.5	7.3	7 2 4	0.7	0.7	9 7 0	1.5	- 1.6	11 7 0	2.3	2.4			
4 5 5	0.6	- 0.5	5 2 3	7.3	7.6	5 8 2	3.4	4.5	6 0 4	3.0	- 2.8	7 3 4	1.6	1.7	9 8 0	3.1	- 3.1	11 2 1	0.7	- 0.8			
4 5 5	5.5	5.5	5 3 3	10.2	10.1	5 8 2	5.3	- 5.3	6 1 4	3.0	- 2.5	7 3 4	0.3	- 0.4	9 0 1	1.9	1.8	11 3 1	2.9	3.0			
4 6 0	1.9	- 1.9	5 2 3	2.0	1.9	5 8 3	5.8	5.1	6 1 4	5.9	6.0	7 4 4	4.2	4.3	9 1 1	0.4	- 0.4	11 4 1	2.1	- 2.0			
4 6 1	4.4	3.4	5 2 4	1.5	- 1.3	5 8 4	3.8	3.0	6 1 4	0.9	- 0.7	7 5 4	2.1	- 1.9	9 1 1	2.2	2.2	11 5 1	1.6	- 1.7			
4 6 1	4.1	3.6	5 2 4	0.5	0.7	5 8 5	5.6	- 4.8	6 2 4	2.4	- 2.4	7 6 4	6.2	6.2	9 2 1	1.1	1.0	11 6 1	1.9	- 1.8			
4 6 1	0.2	0.4	5 2 4	10.1	-10.0	5 8 6	1.8	- 1.4	6 2 4	3.1	3.0	7 7 4	4.6	- 4.4	9 2 1	2.4	2.4	11 7 1	0.5	0.6			
4 6 2	1.7	- 1.6	5 2 5	1.8	1.7	5 9 0	2.2	2.2	6 3 4	3.4	- 3.2	7 8 4	2.4	1.8	9 3 1	5.3	- 5.2	11 8 1	2.1	- 2.0			
4 6 2	4.8	3.9	5 2 5	1.3	- 1.3	5 9 1	2.9	- 2.9	6 4 4	4.2	3.9	7 9 4	2.4	2.1	9 4 1	4.0	3.8	11 2 2	1.9	2.1			
4 6 2	0.8	0.7	5 3 5	4.6	- 4.7	5 9 2	2.4	- 2.4	6 4 4	0.3	0.3	7 10 4	0.5	0.5	9 5 1	4.2	4.1	11 3 2	0.4	0.5			
4 6 3	0.4	- 0.4	5 2 5	1.7	- 1.8	5 9 3	1.4	- 1.3	6 5 4	3.5	3.0	8 0 0	3.9	- 3.7	9 6 1	4.7	4.5	11 4 2	1.8	1.8			
4 6 3	0.9	0.9	5 3 6	4.3	4.2	5 9 4	3.8	- 3.3	6 6 4	1.8	- 1.1	8 1 0	1.1	- 1.0	9 7 1	0.3	- 0.2	11 5 2	2.1	- 2.2			
4 6 4	2.2	2.1	5 2 6	0.2	- 0.4	5 9 5	1.2	- 1.1	6 7 4	4.2	4.1	8 1 0	1.8	1.8	9 8 1	0.5	- 0.6	11 6 2	1.6	- 1.6			
4 6 4	7.8	- 7.4	5 3 0	9.9	9.2	5 9 6	1.3	1.3	6 8 4	1.0	- 1.0	8 2 0	0.7	- 0.6	9 9 1	0.9	- 0.9	11 7 2	1.6	1.8			
4 6 5	1.2	1.2	5 3 0	3.0	1.2	5 10 2	0.6	0.7	6 9 4	2.0	1.7	8 3 0	9.5	- 9.6	9 0 2	0.6	0.7	11 8 2	4.3	4.4			
4 6 5	2.6	- 2.3	5 3 1	0.9	- 0.5	5 10 3	0.8	- 0.9	6 10 4	1.4	- 1.3	8 4 0	5.7	- 5.8	9 0 2	0.8	0.6	11 9 2	0.3	- 0.5			
4 6 6	2.0	- 1.8	5 3 1	1.9	- 2.5	5 10 4	1.3	- 1.3	7 0 0	1.5	- 1.3	8 5 0	3.1	3.1	9 1 2	0.4	0.4	11 2 3	0.6	- 0.6			
4 7 0	8.2	- 7.8	5 3 1	7.4	- 7.2	5 10 5	0.3	0.4	7 1 0	6.8	- 5.9	8 6 0	1.3	1.5	9 1 2	3.2	3.2	11 3 3	1.2	- 1.3			
4 7 1	3.9	- 3.1	5 3 1	3.0	- 2.6	6 0 0	1.4	1.2	7 1 0	3.9	3.9	8 7 0	3.6	- 3.5	9 2 2	3.4	- 3.4	11 4 3	1.1	1.0			
4 7 1	3.6	3.1	5 3 2	1.1	- 0.9	6 1 0	7.3	- 7.0	7 2 0	0.9	0.9	8 8 0	1.5	- 1.5	9 3 2	3.6	3.7	11 5 3	1.4	- 1.1			
4 7 2	0.3	0.3	5 3 2	0.2	- 0.5	6 1 0	14.2	14.3	7 2 0	2.6	1.7	8 9 0	0.5	0.7	9 5 2	9.3	- 9.4	11 6 3	1.4	- 1.3			
4 7 2	3.8	3.7	5 3 2	5.9	7.1	6 2 0	0.9	- 1.0	7 3 0	1.9	1.7	8 0 1	1.7	- 1.8	9 6 2	2.6	2.7	11 7 3	0.4	0.5			
4 7 3	0.3	0.4	5 3 3	9.3	- 9.4	6 3 0	10.1	11.2	7 8 0	2.9	- 3.0	8 0 1	1.7	1.1	9 7 2	0.5	0.7	11 8 3	1.9	2.0			
4 7 3	0.3	0.3	5 3 3	0.9	- 0.6	6 3 0	4.7	4.1	7 4 0	2.8	3.1	8 1 1	3.1	2.9	9 8 2	2.1	- 2.0	11 9 3	2.6	- 2.5			
4 7 4	3.6	- 3.3	5 3 3	12.2	-12.1	6 3 0	0.9	- 0.8	7 5 0	2.2	1.3	8 1 1	1.1	- 0.8	9 9 2	2.4	- 2.4	11 3 4	0.2	- 0.2			
4 7 5	2.1	1.7	5 3 3	2.2	- 2.3	6 4 0	2.7	2.6	7 6 0	1.3	- 0.6	8 1 1	3.1	3.0	9 10 2	1.6	1.7	11 4 4	0.2	0.4			
4 8 0	1.7	- 1.5	5 3 4	3.0	2.7	6 4 0	0.3	0.0	7 7 0	2.4	- 1.9	8 1 1	1.7	1.7	9 0 3	1.4	1.3	11 5 4	1.3	1.1			
4 8 1	0.9	0.9	5 3 4	7.5	- 7.5	6 5 0	1.9	2.4	7 8 0	1.0	1.1	8 2 1	2.2	2.0	9 1 3	0.4	- 0.2	11 6 4	0.8	- 0.9			
4 8 1	0.4	- 0.1	5 3 4	2.4	2.3	6 6 0	3.3	2.8	7 9 0	1.7	1.7	8 2 1	1.3	- 1.5	9 2 3	4.9	4.3	11 7 4	1.5	- 1.4			
4 8 2	7.0	- 6.9	5 3 5	1.9	- 2.0	6 7 0	1.5	1.6	7 0 1	1.4	- 1.2	8 2 1	0.4	0.2	9 3 3	5.2	- 5.1	11 8 4	1.3	1.1			
4 8 3	0.7	- 0.7	5 3 5	1.4	- 1.3	6 8 0	1.3	1.3	7 0 1	2.5	2.5	8 2 1	1.5	1.5	9 4 3	3.9	3.7	11 9 4	1.4	1.3			
4 8 4	6.9	6.9	5 3 5	0.8	0.6	6 9 0	2.0	1.9	7 1 1	0.6	0.5	8 3 1	1.2	- 0.7	9 5 3	4.5	4.4	12 5 1	0.6	- 0.7			
4 8 5	1.4	1.3	5 3 6	0.8	0.7	6 0 1	2.9	3.1	7 1 1	5.5	- 5.5	8 4 1	0.4	0.2	9 7 3	3.2	2.8	12 6 1	0.5	- 0.6			
4 8 6	2.8	- 2.8	5 3 6	2.2	- 2.1	6 0 1	1.1	1.1	7 1 1	1.3	1.4	8 5 1	12.3	12.7	9 8 3	3.8	- 3.1	12 7 1	2.4	- 2.4			
4 9 0	1.1	1.3	5 3 6	0.7	0.8	6 1 1	9.8	- 9.7	7 1 1	2.2	2.2	8 6 1	2.5	- 2.3	9 9 3	1.1	1.0	12 4 2	2.0	2.1			
4 9 1	0.4	0.4	5 4 0	3.7	3.1	6 1 1	6.0	- 6.5	7 2 1	0.9	- 0.6	8 7 1	1.5	- 1.7	9 0 4	0.2	0.4	12 5 2	0.5	0.6			
4 9 2	2.8	- 2.9	5 4 0	5.2	- 5.2	6 1 1	3.8	4.6	7 2 1	3.6	- 3.6	8 8 1	0.4	0.1	9 1 4	3.5	- 2.7	12 6 2	1.5	- 1.6			
4 9 3	3.3	3.0	5 4 1	1.1	1.0	6 1 1	1.3	- 1.3	7 2 1	0.6	- 0.7	8 9 1	0.9	0.9	9 3 4	5.6	- 5.4	12 7 2	0.6	0.6			
4 9 4	2.3	- 2.0	5 4 1	6.9	- 7.5	6 2 1	3.4	- 3.2	7 2 1	0.8	0.6	8 0 2	1.5	- 1.6	9 4 4	2.6	2.3	12 8 2	3.4	3.5			
4 9 5	2.4	- 2.4	5 4 1	11.4	-11.5	6 2 1	4.2	- 3.8	7 3 1	1.6	1.2	8 0 2	3.7	3.7	9 5 4	3.9	3.9	12 5 3	0.2	0.1			
4 9 6	1.9	1.9	5 4 1	3.1	- 3.1	6 2 1	3.3	- 2.8	7 3 1	3.2	- 4.6	8 1 2	0.8	- 0.7	9 6 4	2.1	- 2.1	12 6 3	1.4	- 1.5			
4 10 3	0.2	0.2	5 4 2	0.4	- 0.4	6 2 1	3.0	- 2.0	7 3 1	0.4	0.4	8 1 2	5.0	- 4.5	9 7 4	3.0	- 2.9	12 7 3	0.8	- 0.9			
4 10 4	2.1	- 2.0	5 4 2	2.5	1.8	6 3 1	0.6	0.3	7 4 1	0.8	0.9	8 1 2	7.3	- 7.3	9 8 4	2.6	- 2.4	12 8 3	0.8	- 0.7			
4 10 5	0.9	1.1	5 4 2	9.0	- 8.4	6 3 1	10.6	-10.6	7 5 1	2.2	2.4	8 1 2	1.4	1.4	9 10 4	0.3	0.3	12 4 4	0.1	- 0.2			
5 0 0	8.9	8.9	5 4 2	1.5	1.4	6 3 1	0.6	0.8	7 6 1	2.5	- 2.4	8 2 2	2.4	2.5	10 0 0	0.3	0.3	12 5 4	1.0	1.4			
5 0 1			5 4 3	6.4	- 6.4	6 4 1	0.7	0.8	7 7 1	5.7	- 5.3	8 2 2	0.4	0.7	10 1 0	1.3	1.2	12 6 4	0.1	0.0			
5 0 1	8.5	- 8.9	5 4 3	0.6	1.3	6 4 1	6.9	- 7.5	7 8 1	1.6	1.5	8 2 2	1.2	- 1.3	10 2 0	3.9	3.7	12 7 4	1.0	- 1.1			
5 0 2	9.8	6.9	5 4 3	1.5	1.6	6 4 1	0.9	0.8	7 9 1	0.3	0.0	8 3 2	2.4	2.5	10 3 0	4.5	- 4.1	12 8 4	0.3	0.2			
5 0 2	2.3	2.9	5 4 4	2.0	- 1.9	6 5 1	9.3	- 9.5	7 0 2	8.8	- 8.6	8 4 2	5.6	5.9	10 4 0	1.8	- 1.9						
5 0 3	4.0	- 4.5	5 4 4	2.7	2.6	6 6 1	0.7	0.8	7 0 2	6.0	6.2	8 5 2	7.5	- 7.7	10 6 0	4.1	4.1						
5 0 3	6.0	- 6.1	5 4 4	1.9	- 1.9	6 7 1	1.7	1.6	7 1 2	4.2	4.1	8 6 2	4.2	- 4.5	10 8 0	0.6	- 0.7						
5 0 4	0.3	- 0.3	5 4 5	0.2	- 0.4	6 8 1	1.9	- 1.7	7 1 2	5.3	- 5.0	8 7 2	2.2	2.8	10 9 0	1.1	0.8						
5 0 4	1.2	- 0.8	5 5 0	0.8	- 1.2	6 9 1	2.6	- 2.3	7 1 2	1.6	- 1.7	8 8 2	1.1	1.0	10 2 1	2.0	- 2.1						
5 0 5	3.8	3.9	5 5 0	5.2	5.1	6 0 2	1.9	- 1.9	7 1 2	0.4	0.1	8 10 2	0.4	0.5	10 3 1	1.4	1.3						
5 0 5	0.7	- 0.4	5 5 1	3.3	- 2.8	6 0 2	1.4	0.4	7 2 2	3.2	3.0	8 0 3	1.8	1.5	10 4 1	0.4	0.2						
5 0 6	0.8	- 0.6	5 5 1	7.6	- 7.0	6 1 2	4.6	4.6	7 2 2	9.9	- 9.5	8 0 3	3.2	3.0	10 5 1	2.1	- 2.0						
5 0 6	1.2	1.2	5 5 1	1.2	1.1	6 1 2	4.3	4.8	7 2 2	3.0	2.2	8 1 3	1.7	1.3	10 6 1	2.3	2.5						
5 0 7	0.4	0.5	5 5 2	3.8	- 2.9	6 1 2	0.9	- 0.1	7 2 2	2.2	- 2.2	8 1 3	3.2	- 2.9	10 7 1	0.6	0.7						
5 1 0	3.8	- 3.7	5 5 2	7.3	- 7.5	6 1 2	1.7	1.6	7 3 2	0.9	- 0.9	8 1 3	0.8	- 0.7	10 8 1	1.4	- 1.4						
5 1 0	13.4	13.3	5 5 2	3.9	4.1	6 2 2	2.6	2.0	7 3 2	11.1	11.3	8 2 3	2.1	2.1	10 0 2	0.8	0.8						
5 1 1	6.6	- 6.7	5 5 3	2.0	- 1.8	6 2 2	1.7	- 1.7	7 3 2	0.6	- 0.5	8 2 3	0.6	0.5	10 1 2	0.8	- 0.9						
5 1 1	1.8	- 2.0	5 5 3	1.2	0.9	6 2 2	13.6	14.1	7 4 2	1.0	0.8	8 3 3	5.7	5.5	10 2 2	1.1	- 1.1						
5 1 1	7.1	7.2	5 5 3	1.4	- 1.4	6 2 2	2.4	- 2.0	7 5 2	4.3	4.9	8 4 3	0.8	- 0.0	10 3 2	5.2	5.3						
5 1 1	2.1	2.2	5 5 4	1.0	1.1	6 3 2	0.8	0.3	7 6 2	10.1	-10.4	8 5 3	7.5	- 7.9	10 4 2	3.3	- 3.4						
5 1 2	0.4	0.2	5 5 4	7.5	6.6	6 3 2	9.2	4.6	7 7 2	2.3	2.4	8 6 3	8.0	7.9	10 5 2	2.9	- 2.9						
5 1 2	4.9	- 4.7	5 5 4	0.3	- 0.4	6 4 2	0.8	- 1.5	7 8 2	4.4	4.4	8 7 3	1.1	- 0.9	10 6 2	1.7	- 1.0						
5 1 2	6.3	6.4	5 5 5	6.5	5.0	6 5 2	1.3	- 1.0	7 9 2	1.1	1.0	8 8 3	1.2	- 0.9	10 7 2	3.4	3.7						
5 1 3	1.4	- 0.9	5 6 0	3.4	3.3	6 6 2	6.3	6.9	7 10 2	1.0	- 1.1	8 9 3	0.5	- 0.3	10 8 2	0.8	- 0.8						
5 1 3	6.4	- 6.4	5 6 1	0.8	1.4	6 7 2	0.4	- 0.2	7 0 3	1.7	- 1.8	8 10 3	1.6	1.6	10 9 2	0.8	0.7						
5 1 3	1.6	- 1.7	5 6 1	3.5	3.4	6 8 2	3.6	3.6	7 0 3	3.3	- 2.3	8 0 4	0.3	0.0	10 2 3	1.4	1.0						
5 1 3	2.9	- 2.9	5 6 2	2.0	1.5	6 9 2	1.1	- 1.2	7 1 3	4.0	- 3.5	8 0 4	1.4	- 2.1	10 3 3	2.2	- 2.0						
5 1 4	1.1	0.4	6 0 2	11.2	11.3	6 0 3	11.7	-11.5	7 1 3	3.0	2.8	8 1 4	1.5	1.4	10 4 3	0.5	0.6						
5 1 4	4.7	5.2	5 6 3	1.2	- 1.1	6 0 3	8.8	- 8.5	7 1 3	2.6	- 2.5	8 1 4	0.9	- 1.0	10 5 3	1.3	1.2						
5 1 4	12.6	13.9	5 6 3	2.6	2.5	6 1 3	2.2	- 1.9	7 1 3	1.3	0.7	8 2 4	3.5	- 3.4	10 7 3	2.5	2.2						
5 1 4	0.2	- 0.0	5 6 4	2.1	2.0	6 1 3	1.4	- 1.4	7 2 3	2.6	2.3	8 3 4	1.2	1.3	10 8 3	1.4	1.0						
5 1 5	3.3	3.4	5 6 4	7.9	- 7.6	6 1 3	7.6	7.6	7 2 3	4.2	3.5	8 4 4	1.8	- 1.9	10 9 3	1.1	- 1.1						
5 1 5	0.8	0.4	5 6 5	2.7	2.1	6 1 3	0.9	- 0.9	7 2 3	1.7	1.0	8 5 4	5.7	5.8	10 10 3	1.1	- 1.1						
5 1 5	2.3	- 2.5	5 6 6	5.4	5.0	6 2 3	0.4	0.4	7 3 3	0.6	0.3	8 6 4	0.5	- 0.6	10 1 4	1.3	1.2						
5 1 6	2.6	- 2.4	5 7 0	0.6	0.5	6 2 3	1.0	- 0.4	7 3 3	0.9	0.9	8 7 4	3.3	- 3.2	10 2 4	1.6	- 1.5						
5 1 6	0.5	- 0.9	5 7 1	0.4	0.5	6 2 3	2.4	1.9	7 4 3	0.6	0.4	8 8 4	2.0	- 1.7	10 3 4	3.3	- 3.0						
5 2 0	3.4	- 3.4	5 7 1	7.4	7.5	6 3 3	0.4	- 0.9	7 5 3	8.3	- 8.3	8 9 4	1.9	2.0	10 4 4	2.3	2.3						
5 2 0	5.2	5.8	5 7 2	2.3	2.3	6 3 3	4.7	- 4.6	7 6 3	6.2	- 6.3	8 10 4	0.4	0.4	10 5 4	1.4	1.2						
5 2 1	8.1	- 7.7	5 7 2	6.5	5.6	6 4 3	10.2	-10.5	7 8 3	2.8	2.6	9 0 0	1.6	- 1.6	10 6 4	1.8	- 1.6						
5 2 1	10.1	-10.8	5 7 3	1.6	- 1.3	6 4 3	0.5	0.5	7 9 3	1.4	1.2	9 1 0	0.4	- 0.4	10 7 4	2.6	- 2.5						
									7 10 3	1.6	- 1.8												

Table 3. *Peaks heights* (e.Å^{-3}) *and curvatures* (e.Å^{-5})

Values in parentheses are from F_c differential syntheses.

	ϱ obs. (calc.)	$-A_{hh}$ obs. (calc.)	$-A_{kk}$ obs. (calc.)	$-A_{ll}$ obs. (calc.)	A_{hk} obs. (calc.)	A_{hl} obs. (calc.)	A_{kl} obs. (calc.)
C(1)	6·47	47·9	52·9	38·6	2·73	1·10	1·93
	(6·59)	(48·3)	(51·4)	(38·3)	(2·71)	(1·10)	(1·93)
C(2)	6·41	48·7	48·3	35·8	2·72	−1·18	1·64
	(6·45)	(48·8)	(47·5)	(35·9)	(2·75)	(−1·19)	(1·68)
C(3)	5·27	39·3	38·7	26·6	2·28	−1·34	1·48
	(5·42)	(39·8)	(38·7)	(27·8)	(2·28)	(−1·34)	(1·53)
C(4)	6·59	49·0	51·4	37·5	2·62	−1·15	1·59
	(6·60)	(49·5)	(52·4)	(36·1)	(2·68)	(−1·17)	(1·65)
C(5)	6·50	47·3	51·0	37·4	2·70	−1·12	1·74
	(6·60)	(48·0)	(50·4)	(37·6)	(2·65)	(−1·14)	(1·78)
C(6)	4·72	25·3	29·9	23·2	1·57	−0·43	1·17
	(4·85)	(25·3)	(30·5)	(22·6)	(1·45)	(−0·44)	(1·11)
C(7)	6·64	45·1	52·1	37·6	2·40	−0·71	1·53
	(6·80)	(45·8)	(50·7)	(37·9)	(2·47)	(−0·73)	(1·58)
O(1)	8·63	59·6	61·2	44·2	2·95	−0·99	1·75
	(8·70)	(59·8)	(59·6)	(44·8)	(3·05)	(−1·03)	(1·81)
O(2)	8·32	55·5	59·3	42·7	3·08	−1·06	1·60
	(8·38)	(55·2)	(57·4)	(42·9)	(3·09)	(−1·06)	(1·63)
O(3)	7·93	52·4	54·2	34·9	2·76	−1·01	1·19
	(7·92)	(52·1)	(51·1)	(35·4)	(2·77)	(−0·97)	(1·24)
O(4)	7·92	56·3	55·1	37·5	3·14	−1·31	1·40
	(8·13)	(55·8)	(54·8)	(37·8)	(3·11)	(−1·25)	(1·42)

Table 4. *Intramolecular distances and angles with standard deviations*

Distances		
C(1)–C(2)	1·499 Å	$3{\cdot}8\times10^{-3}$ Å
C(2)–O(1)	1·209	$3{\cdot}3\times10^{-3}$
C(1)–O(2)	1·322	$3{\cdot}8\times10^{-3}$
C(2)–C(3)	1·511	$5{\cdot}9\times10^{-3}$
C(2)–C(4)	1·541	$3{\cdot}9\times10^{-3}$
C(4)–C(5)	1·515	$4{\cdot}9\times10^{-3}$
C(5)–C(6)	1·509	$6{\cdot}4\times10^{-3}$
C(5)–C(7)	1·507	$3{\cdot}8\times10^{-3}$
C(7)–O(4)	1·267	$4{\cdot}0\times10^{-3}$
C(7)–O(3)	1·247	$3{\cdot}6\times10^{-3}$
Angles		
C(2)–C(1)–O(1)	123·3°	$2{\cdot}7\times10^{-1}$°
C(2)–C(1)–O(2)	113·4	$2{\cdot}3\times10^{-1}$
C(4)–C(2)–C(3)	111·5	$2{\cdot}9\times10^{-1}$
C(1)–C(2)–C(3)	108·8	$2{\cdot}7\times10^{-1}$
C(1)–C(2)–C(4)	111·6	$2{\cdot}1\times10^{-1}$
C(4)–C(5)–C(6)	114·9	$2{\cdot}8\times10^{-1}$
C(4)–C(5)–C(7)	110·4	$2{\cdot}8\times10^{-1}$
C(6)–C(5)–C(7)	107·9	$2{\cdot}9\times10^{-1}$
C(2)–C(4)–C(5)	114·2	$2{\cdot}8\times10^{-1}$
C(5)–C(7)–O(4)	119·0	$2{\cdot}5\times10^{-1}$
C(5)–C(7)–O(3)	119·3	$2{\cdot}6\times10^{-1}$
C(1)–C(2)–C(4) ∧ C(2)–C(4)–C(5)	296·4	$3{\cdot}0\times10^{-1}$
C(2)–C(4)–C(5) ∧ C(4)–C(5)–C(7)	172·9	$3{\cdot}0\times10^{-1}$
O(1)–C(1)–C(2) ∧ C(1)–C(2)–C(3)	97·2	$3{\cdot}0\times10^{-1}$
O(3)–C(7)–C(5) ∧ C(7)–C(5)–C(6)	73·9	$3{\cdot}0\times10^{-1}$

for the hydrogen atoms; R was 0·117. 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. In Table 3 the peak heights and the curvatures of electron density are compared at the points corresponding to the atomic positions.

The estimated standard deviations of electron density and of its first derivatives are:

$\sigma(\varrho)=0{\cdot}07$ e.Å^{-3}

$\sigma(A_h)=0{\cdot}12$, $\sigma(A_k)=0{\cdot}14$, $\sigma(A_l)=0{\cdot}14$ e.Å^{-4}.

Molecular and crystal structure

In Fig. 1 the molecular model of DMGA is presented. The interatomic bond distances and angles with the calculated values of their estimated standard deviations (Cruickshank & Robertson, 1953) are listed in Table 4. The equations of the least-squares plane calculated for the C(1)C(2)O(1)O(2) atoms and for the C(5)C(7)O(3)O(4) atoms are respectively

$$5{\cdot}878x-6{\cdot}456y+6{\cdot}687z-0{\cdot}018=0$$

and

$$1{\cdot}637x-5{\cdot}573y+6{\cdot}721z+2{\cdot}214=0\,.$$

No significant deviation from the plane was observed [maximum deviation −0·008 Å for C(1) and −0·008 Å

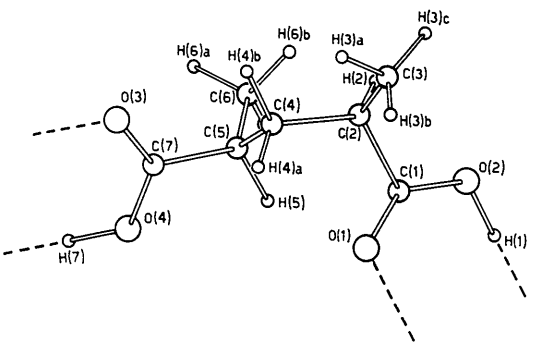
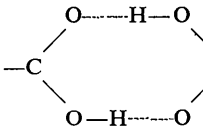


Fig. 1. Molecular model of *meso*-α,α'-dimethylglutaric acid.

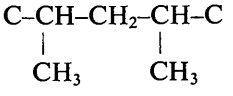
for C(7)]. Also the systems  with the centres of symmetry at 0,0,0 and 0,1,$\frac{1}{2}$ are planar within experimental error.

The values of internal rotation angles

$$\sigma_1 = C(1)C(2)C(4) \wedge C(2)C(4)C(5) = 296{\cdot}4°$$

and

$$\sigma_2 = C(2)C(4)C(5) \wedge C(4)C(5)C(7) = 172{\cdot}9°$$

show that the atomic grouping  has a similar conformation to that found for isotactic polypropylene (Natta & Corradini, 1960).

In Figs. 2 and 3 the projections of the DMGA structure along [100] and [001] are shown. The shortest intermolecular distances are also indicated. The structure consists of hydrogen-bonded molecules directed along the $\mathbf{b}+\mathbf{c}/2$ crystallographic axis. A distance of 2·642 ±0·010 Å has been found between the oxygen atoms of carboxylic groups of adjacent molecules repeated by a symmetry center.

Molecular vibration analysis

In Table 5 are shown the coefficients b_{ij} of the temperature factor, which is in the form:

Table 5. *Final temperature parameters*

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

	b_{11}	b_{12}	b_{13}	b_{22}	b_{23}	b_{33}
C(1)	0·0158	0·0246	−0·0011	0·0260	0·0157	0·0285
C(2)	0·0164	0·0245	−0·0031	0·0260	0·0160	0·0314
C(3)	0·0225	0·0296	−0·0165	0·0268	0·0243	0·0427
C(4)	0·0169	0·0201	−0·0060	0·0211	0·0115	0·0325
C(5)	0·0180	0·0242	0·0030	0·0250	0·0155	0·0274
C(6)	0·0283	0·0410	0·0140	0·0410	0·0360	0·0406
C(7)	0·0162	0·0230	−0·0015	0·0245	0·0140	0·0276
O(1)	0·0173	0·0234	0·0030	0·0255	0·0120	0·0318
O(2)	0·0175	0·0273	0·0015	0·0270	0·0050	0·0333
O(3)	0·0180	0·0260	−0·0080	0·0272	−0·0030	0·0395
O(4)	0·0175	0·0276	−0·0090	0·0284	0·0015	0·0400

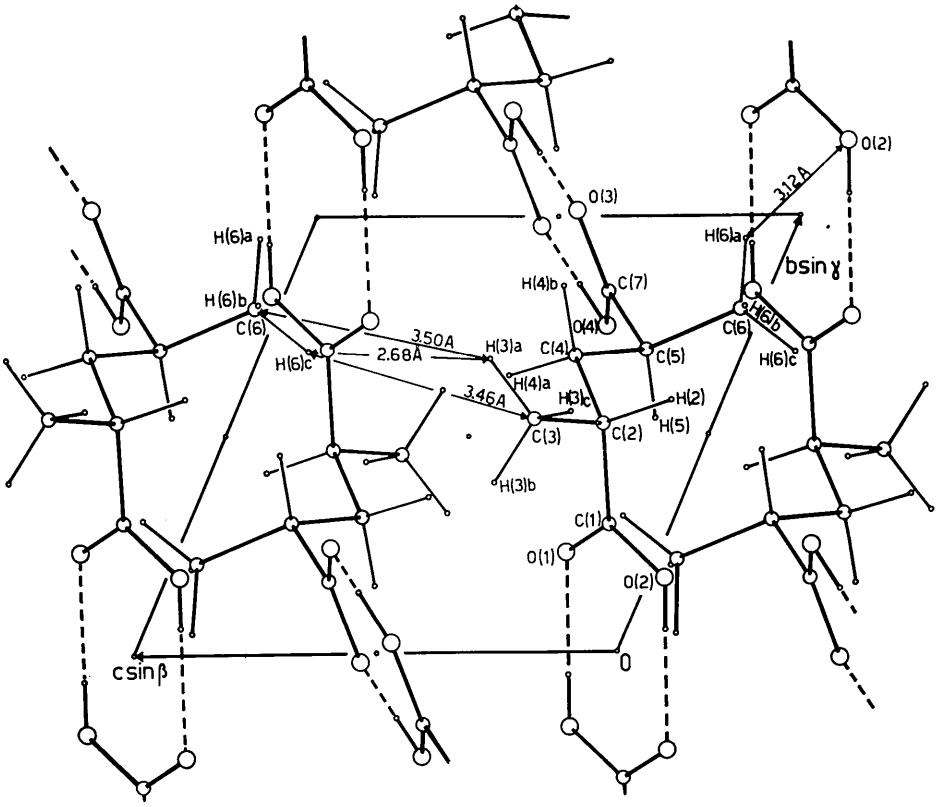


Fig. 2. Projection of the *meso*-α,α′-dimethylglutaric acid structure along [100].

Table 6. *Dimensions and orientations of the thermal vibration ellipsoids relative to the atoms of meso-α,α′-dimethylglutaric acid*

	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 an orthogonal coordinate system*		
C(1)	U_1=4·18	0·23	55·9,	74·5,	76·9	0·8738,	0·4309,	0·2253
	U_2=5·29	0·26	76·9,	126·6,	13·3	−0·1806,	−0·1427,	0·9731
	U_3=3·34	0·20	142·8,	40·8,	87·3	−0·4515,	0·8910,	0·0469
C(2)	U_1=4·22	0·23	51·8,	78·0,	76·7	0·9020,	0·3652,	0·2301
	U_2=5·74	0·27	81·7,	121·2,	13·4	−0·2266,	−0·0532,	0·9725
	U_3=3·51	0·21	140·6,	33·9,	92·0	−0·3674,	0·9294,	−0·0348
C(3)	U_1=5·71	0·27	103·8,	31·2,	93·0	0·3115,	0·9488,	−0·0525
	U_2=8·00	0·32	110·5,	99·8,	39·0	−0·5845,	0·2349,	0·7766
	U_3=4·42	0·24	25·1,	119·3,	51·1	0·7492,	−0·2113,	0·6278
C(4)	U_1=4·37	0·23	16·0,	110·1,	80·4	0·9393,	−0·3002,	0·1661
	U_2=6·15	0·28	80·2,	120·2,	10·7	−0·1833,	−0·0299,	0·9826
	U_3=3·14	0·20	102·5,	37·5,	82·2	0·2900,	0·9534,	0·0831
C(5)	U_1=4·54	0·24	25·7,	105·2,	66·4	0·9128,	−0·0777,	0·4008
	U_2=4·96	0·25	90·6,	120·3,	24·4	−0·4061,	−0·0725,	0·9109
	U_3=3·58	0·21	115·7,	34·7,	84·4	0·0418,	0·9943,	0·0977
C(6)	U_1=5·97	0·27	95·9,	30·4,	119·1	0·5004,	0·7158,	−0·4871
	U_2=9·09	0·34	38·8,	110·1,	43·8	0·6922,	0·0071,	0·7216
	U_3=5·19	0·26	128·2,	68·1,	60·5	−0·5199,	0·6983,	0·4919
C(7)	U_1=4·16	0·23	39·4,	87·2,	83·5	0·9865,	0·1190,	0·1127
	U_2=5·26	0·26	71·2,	129·5,	11·8	−0·0896,	−0·1843,	0·9787
	U_3=3·41	0·21	123·1,	39·6,	80·1	−0·1373,	0·9756,	0·1712
O(1)	U_1=4·36	0·23	49·6,	76·4,	97·3	0·9720,	0·1968,	−0·1281
	U_2=7·22	0·30	125·2,	45·6,	160·7	−0·1804,	0·2767,	−0·9439
	U_3=3·42	0·21	119·9,	47·5,	72·3	−0·1503,	0·9406,	0·3045
O(2)	U_1=4·60	0·24	47·2,	79·6,	85·2	0·9640,	0·2522.	0·0843
	U_2=8·24	0·32	116·4,	43·2,	162·2	0·0031,	0·3061,	−0·9520
	U_3=2·92	0·19	125·7,	48·7,	72·9	−0·2658,	0·9180,	0·2943
O(3)	U_1=4·47	0·24	26·9,	99·1,	82·2	0·9851,	−0·1055,	0·1359
	U_2=9·98	0·36	107·8,	44·9,	160·8	0·1610,	0·2868,	−0·9444
	U_3=3·24	0·20	109·6,	46·5,	72·6	0·0607,	0·9522,	0·2995
O(4)	U_1=4·22	0·23	39·3,	89·9,	73·7	0·9467,	0·1574,	0·2809
	U_2=9·34	0·34	77·7,	131·4,	19·6	−0·2407,	−0·2339,	0·9420
	U_3=3·44	0·21	126·6,	41·4,	79·4	−0·2139,	0·9594,	0·1835

* In this system the *z* axis is coincident with **c**, the *y* axis is normal to **c** in the *bc* plane, and the *x* axis is normal to the *bc* plane.

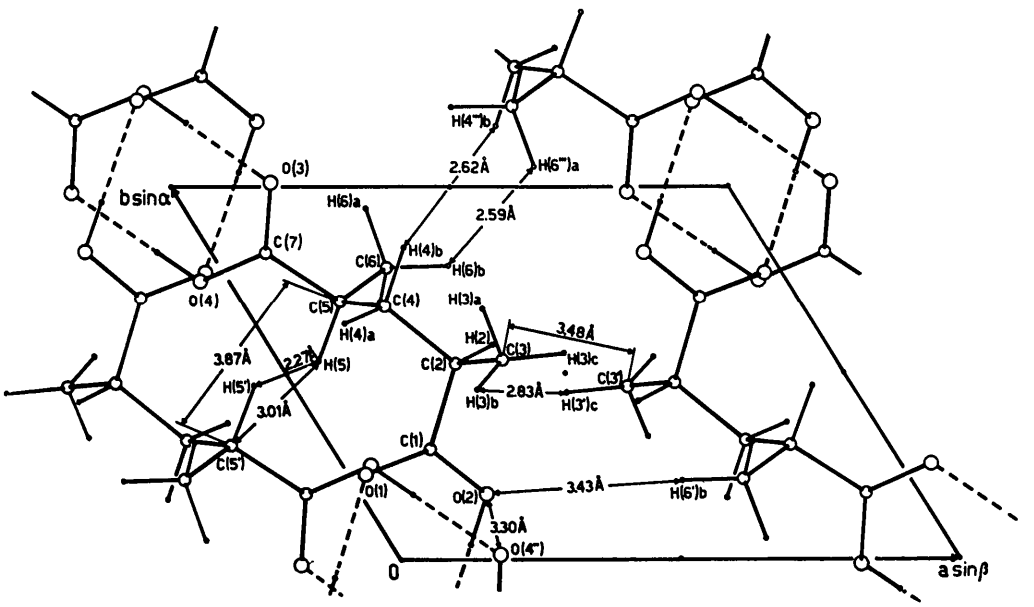


Fig. 3. Projection of the *meso*-α,α′-dimethylglutaric acid structure along [001].

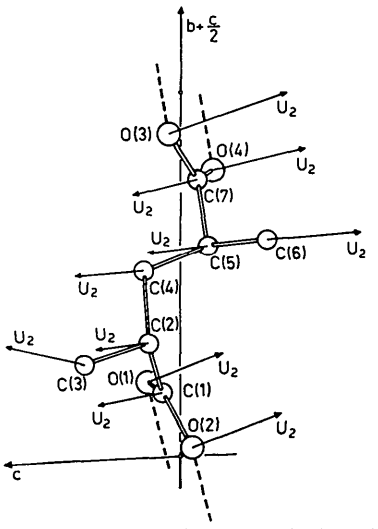


Fig. 4. Projection parallel to the row axis (**b**+**c**/2) of *meso*-α,α'-dimethylglutaric acid molecule and of U_2 semi-axes of thermal vibrational ellipsoids.

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

The values of the three semi-axes of the ellipsoid of thermal vibration for each C and O atom are reported in Table 6. These parameters were calculated with a program written for an Elea 6001 computer (Coda, 1966). An evaluation of the thermal data shows that, for all atoms, one of the three semi-axes (U_2) is appreciably larger than the remaining two (U_1, U_3). Correspondingly a larger mean square displacement in the U_2 direction is observed. As shown in Fig. 4, the U_2 semi-axes are nearly orthogonal to the axis of the molecular rows **b**+**c**/2.

Nevertheless it is important to point out that the U_2 length corresponding to the oxygen atoms and to carbon atoms of the methyl groups is larger than the U_2 length corresponding to the carbon atoms of the molecular skeleton. This situation is consistent with the results of analogous studies on non-substitute. dicarboxylic acids (Housty & Hospital, 1964, 1965, 1966) in which thermal vibration oscillations occur with a greater amplitude for the terminal atoms and in the same direction as in our compound. It is also interesting to note that the values of the semi-axis lengths are in agreement with those found for normal chain acids (Housty & Hospital, 1964, 1965, 1966).

Our results lead to the conclusion that, apart from the differences in the shape of rotational barriers around the single bonds, the molecule of DMGA is characterized in the solid state by a degree of rigidity similar to that of a non-substituted dicarboxylic acid.

The authors are grateful to Prof. P. Corradini for advice and helpful discussion, and to Dott. A. Coda who kindly supplied us with a crystallographic program.

References

Berghuis, J., Haanappel, IJ. M., Potters, M., Loopstra, B. O., MacGillavry, C. H. & Veenendaal, A. L. (1965). *Acta Cryst.* **8**, 478.
Coda, A. (1966). *Ric. Sci.* **36**, 388.
Cruickshank, D. W. J. (1949). *Acta Cryst.* **2**, 65.
Cruickshank, D. W. J. & Robertson, A. P. (1953). *Acta Cryst.* **6**, 698.
Ganis, P., Pedone, C. & Temussi, P. A. (1964). *Rend. Acc. Naz. Lincei*, **36**, 510.
Housty, J. & Hospital, M. (1964). *Acta Cryst.* **17**, 1387.
Housty, J. & Hospital, M. (1965). *Acta Cryst.* **18**, 753.
Housty, J. & Hospital, M. (1966). *Acta Cryst.* **21**, 29.
McWeeny, R. (1951). *Acta Cryst.* **4**, 513.
Natta, G. & Corradini, P. (1960). *Suppl. Nuovo Cimento*, **15**, 40.
Rollett, J. S. & Sparks, R. A. (1960). *Acta Cryst.* **13**, 273.