also noticed in $C_{12}H_5F_5$ where the mean distance between the C_6H_5 group and the C_6F_5 plane was found to be 3·43 Å. Table 2 lists some interatomic contacts between molecules in the stack. No important contacts were noticed between the stacks.

The molecular packing of the $C_{12}F_{10}.C_{12}H_{10}$ complex is very similar to that present in $C_{12}H_5F_5$. The similarity of the stack angles, interplanar separations, and their melting points indicates that the intermolecular interactions are nearly identical in the two crystals. The geometries of the $C_{12}F_{10}$ and $C_{12}H_{10}$ molecules in the complex are virtually unchanged from those in single crystals, except for the dihedral angle in $C_{12}F_{10}$ which has been compressed by 8·8°.

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Structure of Tris(hydroxymethyl)acetic Acid*

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Abstract. $C_5H_{10}O_5$, monoclinic, $I2/a$ (non-standard setting of $C2/c$), $a = 10{\cdot}417$ (1), $b = 5{\cdot}727$ (1), $c = 22{\cdot}774$ (2) Å, $\beta = 98{\cdot}05$ (1)°, $V = 1345$ Å^3, $Z = 8$, $D_c = 1{\cdot}464$, $D_m = 1{\cdot}468$ Mg m^{-3} (in carbon tetrachloride and xylene), μ(Mo $K\alpha$) = 0·142 mm^{-1}. The structure was solved using *MULTAN* and refined to $R = 0{\cdot}051$ for 683 unique reflections. This polyhydric plastic-crystal precursor crystallizes in a three-dimensional hydrogen-bonded network involving all of the O and hydroxy H atoms as well as the carboxyl H atom.

Introduction. The structure of the title compound, $(CH_2OH)_3CCOOH$ (designated hereafter as *R*COOH), was determined as part of our continuing investigation of hydrogen-bonded poly(hydroxymethyl) compounds known to undergo solid–solid phase transitions between ordered and orientationally disordered (plastic) phases. Doshi, Furman & Rudman (1973) reported that upon warming to 394·7 K, *R*COOH undergoes a phase transition to an orientationally disordered face-centered cubic lattice with $a \sim 8{\cdot}9$ Å. The crystal and molecular structures of the ordered phase of this compound, stable at room temperature, are reported here.

Single crystals were grown from a saturated solution of ethanol using the crystal-growing device described

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[*] Polymorphism of Crystalline Poly(hydroxymethyl) Compounds. II. Part I: Eilerman & Rudman (1979).

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by Hope (1971). The non-standard space-group setting was selected to simplify the comparison of closely related materials and accurate cell dimensions were obtained by least-squares refinement of sixteen carefully centered reflections with $2\theta > 30°$ ($\lambda = 0{\cdot}71069$ Å). The two positions related by the *a* glide in *I*2/*a* are *x*,*y*,*z* and $\frac{1}{2}+x$, $\bar{y}$, z, while the base-vector transformation matrix to the standard setting (*C*2/*c*) is:

$$\begin{pmatrix} 1 & 0 & 1 \\ 0 & 1 & 0 \\ -1 & 0 & 0 \end{pmatrix}.$$

Data from a cubo-octahedral single crystal with a maximum dimension of 0·30 mm were collected on a Nonius CAD-4 diffractometer using Zr-filtered Mo *K* radiation. 2275 reflections were measured to $\sin\theta/\lambda = 0{\cdot}64$ Å^{-1} using the θ–2θ scan technique with a 2θ scan range of $(1 + 0{\cdot}35 \tan\theta)°$. Equivalent reflections were averaged, and, of the 1474 unique reflections, 683 with $I > 3\sigma(I)$ were used; the agreement factor for averaging was 0·036. Inasmuch as the check reflections did not show any systematic variation during the course of data collection and since $\mu R_{\max}$ is negligibly small, neither decay nor absorption corrections were applied to the data.

After correction for Lorentz and polarization effects, the normalized structure factors (*E*) were calculated, and their distribution was found to be consistent with a centrosymmetric space group. *MULTAN* (Germain, Main & Woolfson, 1971) was used to calculate eight phased sets from the 300 *E* factors > 1·27. The ten highest peaks in the *E* map based on the set with the highest combined figure of merit corresponded to the ten C and O atom positions. Fractional coordinates were calculated for the six methylene H atoms, while the remaining four H atoms were subsequently located on a difference Fourier map.

The atomic and thermal parameters were refined using the full-matrix least-squares program *NUCLS*, a modified version of *ORFLS* (Busing, Martin & Levy, 1962), where the function minimized was $\sum_{\mathbf{H}} w_{\mathbf{H}}(|F_{o,\mathbf{H}}| - |kF_{c,\mathbf{H}}|)^2$ (**H** represents the Miller indices, $w_{\mathbf{H}}$ is the weight assigned to the observed structure factor, $F_{o,\mathbf{H}}$, and $kF_{c,\mathbf{H}}$ is the scaled calculated structure factor). The weighting scheme used was $w = 1/\sigma^2$, where $\sigma = 0{\cdot}5\sigma(F^2)/F$ and $\sigma(F^2) = [\sigma'(F^2)^2 + (0{\cdot}02F^2)^2]^{1/2}$; $\sigma'(F^2)$ is based on counting statistics. In the final refinement cycle 122 parameters (scale factor, three positional and six anisotropic thermal parameters for each non-hydrogen atom, three positional parameters for each H atom, and an isotropic extinction parameter) were varied; each H was assigned a fixed temperature factor of 4·0 Å^2. The overall extinction coefficient (*g*) was 0·25 (7) (based on an estimated value of $\bar{t} = 0{\cdot}02$); only one reflection had a transmission factor less than 0·93. When refinement was stopped all parameter shifts were less than $2{\cdot}0 \times 10^{-5}$ and the final discrepancy values were $R = 0{\cdot}051$ and $R_w = 0{\cdot}053$ (R and R_w are defined in Eilerman & Rudman, 1979). The final error in an observation of unit weight was equal to 1·23. Atomic scattering factors were obtained from *International Tables for X-ray Crystallography* (1974; *f* for C and O from Table 2.2*A* and *f* for H from Table 2.2*C*). The fractional coordinates are reported in Table 1.* A final difference Fourier map showed no regions with electron density greater than 0·24 e Å^{-3}.

Table 1. *Fractional coordinates for tris(hydroxymethyl)acetic acid at room temperature (for* C *and* O $\times 10^4$, *for* H $\times 10^3$)

Numbers in parentheses here and in the following tables are e.s.d.'s in the unit of the least significant digits of the preceding number.

	x	*y*	*z*
C(C)	6993 (4)	7867 (7)	1217 (2)
C(1)	5956 (4)	6386 (8)	845 (2)
C(2)	6385 (4)	9930 (8)	1499 (2)
C(3)	7731 (4)	6270 (8)	1683 (2)
C(4)	7935 (4)	8704 (8)	814 (2)
O(1)	5219 (3)	7710 (6)	384 (1)
O(2)	5515 (3)	9070 (6)	1880 (1)
O(3)	8744 (3)	7566 (6)	2016 (1)
O(41)	8028 (3)	10904 (6)	737 (1)
O(42)	8583 (3)	7232 (6)	581 (1)
H(11)	532 (4)	578 (8)	110 (2)
H(12)	632 (4)	501 (9)	67 (2)
H(21)	597 (4)	1103 (9)	118 (2)
H(22)	705 (4)	1087 (8)	170 (2)
H(31)	806 (4)	496 (9)	152 (2)
H(32)	716 (4)	571 (8)	194 (2)
H(O1)	550 (5)	774 (9)	9 (3)
H(O2)	501 (5)	1011 (9)	189 (2)
H(O3)	900 (5)	695 (8)	237 (3)
H(O41)	872 (4)	1134 (8)	55 (2)

Discussion. An analysis of the model based on anisotropic thermal parameters of the non-hydrogen atoms and use of the program *TLS*-6 by Schomaker & Trueblood (1968), showed that the *R*COOH molecule acts as a rigid body with no unusual intramolecular distances or angles. Interatomic distances, corrected and uncorrected for thermal motion, are presented in Table 2, while the bond angles and rigid-body parameters have been deposited.* A detailed view of the hydrogen-bonding scheme about a single molecule is shown in Fig. 1, and the three-dimensional network is illustrated in Fig. 2; interatomic distances and angles involving the hydrogen-bonded atoms are given in Table 3.

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* Lists of structure factors, thermal parameters, rigid-body tensors and bond angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34588 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Intramolecular bond distances* (Å) *in tris(hydroxymethyl)acetic acid*

	Uncorrected	Corrected*
C(C)–C(1)	1·532 (6)	1·543
C(C)–C(2)	1·524 (6)	1·530
C(C)–C(3)	1·524 (6)	1·533
C(C)–C(4)	1·513 (5)	1·527
Average C–C	1·523 (6)	1·533
C(1)–O(1)	1·429 (5)	1·440
C(2)–O(2)	1·429 (5)	1·437
C(3)–O(3)	1·420 (5)	1·427
Average C–O(sp^3)	1·426 (5)	1·435
C(4)–O(41)	1·277 (5)	1·284
C(4)–O(42)	1·245 (5)	1·252
O(1)–H(O1)	0·78 (5)	
O(2)–H(O2)	0·79 (5)	
O(3)–H(O3)	0·88 (5)	
O(4)–H(O41)	0·92 (5)	
Average O–H	0·84 (5)	
C(1)–H(11)	1·00 (4)	
C(1)–H(12)	0·99 (5)	
C(2)–H(21)	1·01 (5)	
C(2)–H(22)	0·94 (5)	
C(3)–H(31)	0·93 (5)	
C(3)–H(32)	0·95 (5)	
Average C–H	0·97 (5)	

* Based on rigid-body treatment of Schomaker & Trueblood (1968).

Table 3. *Hydrogen-bonding* (*i–j···k*) *scheme in tris(hydroxymethyl)acetic acid*

i	*j*	*k*	D_{ik} (Å)	D_{ij} (Å)	D_{jk} (Å)	θ_{ijk} (°)
O(1)	H(O1)	O(42)	2·688 (4)	0·78 (5)	1·93 (5)	167·5
O(2)	H(O2)	O(3)	2·715 (4)	0·79 (5)	1·93 (5)	172·1
O(3)	H(O3)	O(2)	2·693 (4)	0·88 (5)	1·81 (6)	174·8
O(41)	H(O41)	O(1)	2·646 (4)	0·92 (5)	1·75 (5)	165·4

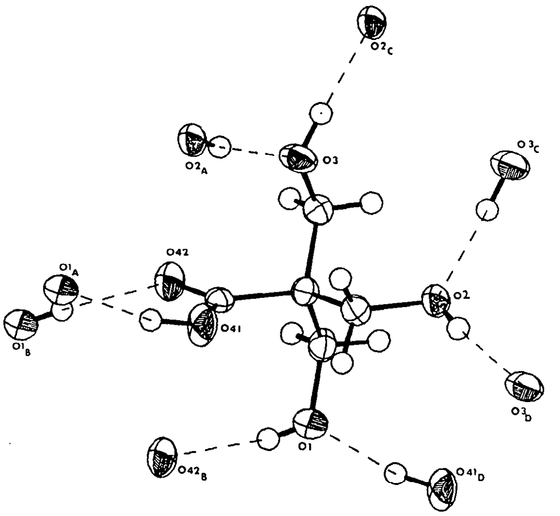


Fig. 1. Single molecule of tris(hydroxymethyl)acetic acid in its hydrogen-bonded environment. The hydrogen-bonded atoms are marked with symbols indicating the molecule to which they belong: (*A*) $\frac{1}{2}+x$, $2-y$, z; (*B*) $\frac{3}{2}-x$, y, $-z$; (*C*) $\frac{3}{2}-x$, $\frac{3}{2}-y$, $\frac{1}{2}-z$; (*D*) $-\frac{1}{2}+x$, $2-y$, z.

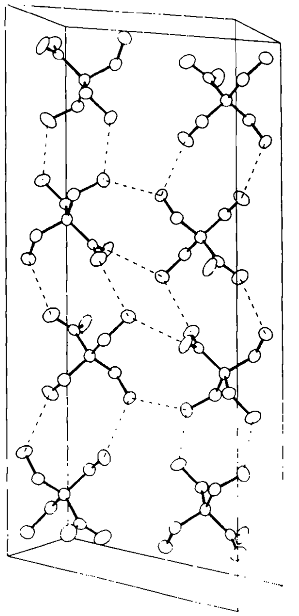


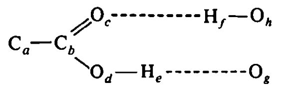
Fig. 2. Hydrogen-bonding scheme in one unit cell of tris(hydroxymethyl)acetic acid.

The molecules join together to form a three-dimensional network in which each molecule of *R*COOH is hydrogen-bonded to four adjacent molecules. Each of the functional groups on the central C atom acts as a hydrogen-bond donor to an acceptor on one molecule and as a hydrogen-bond acceptor from a donor on another molecule (Fig. 1). However, whereas each of the O atoms of the hydroxymethyl groups acts as both a donor (through its H atoms) and an acceptor, the two O atoms of the carboxyl group share these functions: O(42) acts only as a hydrogen-bond acceptor, while O(41) is the hydrogen-bond donor to an atom on a different molecule. The observed thermal parameters reflect the fact that the carboxyl O atoms are involved in only two bonds while the hydroxymethyl O atoms form three bonds apiece. It is also noted that *R*COOH does not have the synplanar conformation generally found in carboxyl groups (Dunitz & Strickler, 1968); it is antiplanar, with the torsion angle C(2)–C(C)–C(4)–O(42) equal to 175·1 (4)°.

If the carboxyl hydrogen-bonding scheme shown in Table 4 is used, the intermolecular group formed by atoms *b–g* can be defined as the hydrogen-bonded moiety and the relationship between the planarity of this moiety and the intramolecular distances and angles can be examined. In *R*COOH, the dihedral angle between the planes formed by atoms *bcf* and *deg* is 58·1°, thus forming a twisted hydrogen-bonded moiety. Reference to Table 4 shows that the interatomic distances and angles found in *R*COOH are similar to the ones found for those carboxyl groups in

Table 4. *Representative distances and angles in intermolecularly hydrogen-bonded carboxyl moieties*

Values are uncorrected for thermal motion.



	–C=O (Å)	>C–OH (Å)	C–C=O (°)	C–C–OH (°)	O=C–OH (°)
Planar*	1·22	1·29	123	115	122
*R*COOH†	1·244	1·277	118·7	117·6	123·6
Twisted‡	1·245	1·279	119	117	124

* Dunitz & Strickler (1968); Derissen & Smit (1974).
† This work.
‡ Trimesic acid; Duchamp & Marsh (1969).

trimesic acid which are approximately coplanar with their benzene rings and which, consequently, also form twisted hydrogen-bonded moieties. On the other hand, the carboxyl groups in trimesic acid which are rotated out of the planes of the benzene rings and do not form twisted hydrogen-bonded moieties have interatomic distances and angles which are similar to those reported for other nearly planar hydrogen-bonded moieties. Since the differences between the twisted and planar structures are small, a systematic investigation of these systems is needed to reveal if the 'twist' of the hydrogen-bonded moiety has a significant effect on the hydrogen-bond parameters which are usually reported and to determine if there is a relationship between the C_α–C_β–C=O torsion angle and the hydrogen-bond moiety twist.

From the rigid-body analysis it was found that in *R*COOH the root-mean-square (r.m.s.) amplitudes of libration about the three principal axes are 4·54, 3·90 and 2·61°, while the r.m.s. amplitudes of translation are 0·164, 0·161 and 0·138 Å. These amplitudes of libration are considerably smaller than those observed in other plastic-crystal precursors, such as pentaerythritol (Eilerman & Rudman, 1979) and 2-hydroxymethyl-2-methyl-1,3-propanediol (Eilerman & Rudman, 1980). However, these other materials form layered structures with strong hydrogen bonding found only within the layers, while in *R*COOH the librational motion is restricted because the hydrogen-bonded network is three-dimensional.

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Structure of Obtusol Acetate, a Halogenated Chamigrene-Type Sesquiterpene

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Abstract. $C_{17}H_{25}Br_2ClO_2$, monoclinic, $P2_1$, $Z = 2$, $a = 8{\cdot}916\,(1)$, $b = 14{\cdot}622\,(3)$, $c = 7{\cdot}703\,(1)$ Å, $\beta = 104{\cdot}23\,(1)°$, $V = 973{\cdot}4\,(2)$ Å^3, $D_x = 1{\cdot}547$ Mg m^{-3}, μ(Mo $K\alpha$) = 45·2 mm^{-1}. The structure was refined to $R = 0{\cdot}064$ for 1080 observed Friedel pairs. The molecule consists of two six-carbon-atom rings, both in

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