Crystal Structure of *meso*-(2*R*, 4*r*, 6*S*)-2,4,6-Heptanetriol as a Model Compound for Syndiotactic Poly(vinyl alcohol)[†]

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ABSTRACT: The crystal structure of *meso*-(2*R*, 4*r*, 6*S*)-2,4,6-heptanetriol as a model compound for syndiotactic poly(vinyl alcohol) was determined. The crystal belongs to a triclinic system with dimensions of $a = 10.950(9)$, $b = 9.904(11)$, $c = 14.199(13)$ Å, $\alpha = 104.47(11)$, $\beta = 114.10(8)$, and $\gamma = 70.34(7)^\circ$. The space group is $P\bar{1}$ and $D_x = 1.125$, $D_m = 1.12$ Mg m⁻³, and $Z = 6$. The final R factor was 0.080 for 3348 observed reflections. There are three crystallographically independent molecules in the unit cell. The seven carbon atoms of these three molecules have TTTT sequences and the OH groups form intermolecular hydrogen bonds. The best planes composed of seven carbon atoms of the three independent molecules are nearly parallel to each other and the molecular axes of the all molecules are nearly parallel to each other and point in the direction of the a -axis.

KEY WORDS Crystal Structure / *meso*-(2*R*, 4*r*, 6*S*)-2,4,6-Heptanetriol /
TTTT Sequence / Syndiotactic Poly(vinyl alcohol) /

In order to elucidate the crystal structures of PVA of various tacticities, we reported the structure analyses of some model compounds of PVA.³⁻⁵ In the case of syndiotactic dimer, there are two crystallographically independent molecules in the unit cell, the directions of the two molecular axes lie nearly perpendicularly, and the carbon chains take TT conformations. In the case of isotactic heptane-2,4,6-triol, however, the seven carbon atoms take GTT $\bar{G}$ conformations. In both cases, all the OH groups form intermolecular hydrogen bonds.

The structure of these model compounds were also investigated by NMR and IR.¹³⁻¹⁶ The results obtained by these methods are not necessarily consistent with each other.

The structure analyses of such a low molecular weight oligomers was not necessarily useful for elucidating the structures of polymers. But when the structure of longer oligo-

mers are analysed, we may expect to obtain more helpful clues.

The present paper reports the results of the structure analysis of syndiotactic heptane-2,4,6-triol or the trimer of syndiotactic PVA, and discusses the conformation, configuration, and hydrogen-bondings of the molecules and the relationships between the structure and properties of PVA.

EXPERIMENTAL

The crystal used was prepared from a ligroin solution by the evaporation method, and its data are shown in Table I. The cell dimensions were determined by the least squares method, using 15 reflections. The density of the crystal was measured by the floatation method. The size of the crystal used for diffraction experiment was about $0.4 \times 0.4 \times 0.6$ mm³.

[†] The investigation was done in Government Industrial Research Institute, Osaka.

Table I. Crystal data

Chemical formula	C ₇ O ₃ H ₁₆
Molecular weight	148.20
Space group	P $\bar{1}$
Cell constants	$a=10.950$ (9) Å $b=9.904$ (11) $c=14.199$ (13) $\alpha=104.47$ (11) $^\circ$ $\beta=114.10$ (8) $\gamma=70.34$ (7)
Volume of the unit cell	$V=1312.3$ Å ³
Density	
Calcd	1.125 Mg m ⁻³
Obsd	1.12 ₂ Mg m ⁻³
Number of molecules in the unit cell	$Z=6$

Table II. Atomic positional parameters and temperature factors for non-hydrogen atoms

Atom	X	Y	Z	BEQ'S/Å ²
C(11)	-0.3159 (4)	0.8823 (5)	0.0276 (4)	6.23
C(12)	-0.1937 (3)	0.7588 (4)	0.0238 (3)	3.83
C(13)	-0.0725 (3)	0.8141 (4)	0.0392 (3)	4.05
C(14)	0.0584 (3)	0.6953 (4)	0.0369 (3)	3.39
C(15)	0.1759 (3)	0.7584 (4)	0.0538 (3)	4.15
C(16)	0.3138 (3)	0.6469 (4)	0.0587 (3)	4.05
C(17)	0.4183 (4)	0.7163 (5)	0.0622 (4)	5.69
O(11)	-0.1600 (3)	0.6753 (3)	0.1032 (2)	4.81
O(12)	0.0313 (2)	0.6017 (2)	-0.0598 (2)	3.91
O(13)	0.3609 (2)	0.5785 (4)	0.1485 (2)	6.65
C(21)	-0.3207 (4)	0.8442 (5)	0.3511 (4)	6.92
C(22)	-0.1936 (4)	0.7232 (4)	0.3510 (3)	4.34
C(23)	-0.0714 (4)	0.7791 (4)	0.3691 (3)	4.26
C(24)	0.0612 (3)	0.6627 (4)	0.3678 (3)	3.66
C(25)	0.1779 (4)	0.7281 (4)	0.3877 (3)	4.72
C(26)	0.3134 (4)	0.6174 (4)	0.3916 (3)	4.72
C(27)	0.4284 (4)	0.6872 (6)	0.4133 (4)	7.08
O(21)	-0.2229 (2)	0.6282 (3)	0.2550 (2)	4.74
O(22)	0.1038 (2)	0.5643 (2)	0.4410 (2)	3.88
O(23)	0.2967 (3)	0.5194 (3)	0.2979 (2)	5.41
C(31)	-0.3338 (4)	0.9110 (5)	0.6898 (5)	6.90
C(32)	-0.2042 (3)	0.7890 (4)	0.6913 (3)	3.87
C(33)	-0.0793 (3)	0.8455 (4)	0.7177 (3)	4.08
C(34)	0.0490 (3)	0.7275 (3)	0.7087 (3)	3.41
C(35)	0.1685 (3)	0.7910 (4)	0.7325 (3)	4.34
C(36)	0.3008 (3)	0.6820 (4)	0.7223 (3)	4.04
C(37)	0.4097 (4)	0.7575 (5)	0.7413 (4)	6.99
O(31)	-0.1702 (2)	0.6985 (3)	0.7659 (2)	4.94
O(32)	0.0189 (2)	0.6533 (3)	0.6053 (2)	4.29
O(33)	0.3583 (2)	0.5823 (3)	0.7962 (2)	4.56

Table III. Atomic positional parameters and isotropic temperature factors for hydrogen atoms

Atom	X	Y	Z	BISO
H(011)	-0.125 (5)	0.586 (5)	0.076 (4)	4.2 (0)
H(012)	-0.008 (5)	0.647 (5)	-0.117 (4)	4.5 (0)
H(013)	0.456 (5)	0.518 (5)	0.157 (4)	5.6 (0)
H(021)	-0.207 (5)	0.661 (5)	0.195 (4)	4.2 (0)
H(022)	0.076 (4)	0.614 (4)	0.500 (3)	2.0 (8)
H(023)	0.303 (5)	0.549 (5)	0.239 (4)	4.7 (0)
H(031)	-0.214 (5)	0.630 (5)	0.742 (4)	4.4 (0)
H(032)	-0.030 (4)	0.578 (5)	0.597 (3)	3.9 (0)
H(033)	0.320 (5)	0.508 (5)	0.788 (3)	4.1 (0)
H(111)	-0.386 (5)	0.843 (5)	0.025 (4)	5.1 (0)
H(112)	-0.295 (5)	0.946 (5)	0.095 (4)	5.1 (0)
H(113)	-0.340 (5)	0.947 (5)	-0.022 (4)	5.1 (0)
H(121)	-0.215 (3)	0.698 (3)	-0.044 (2)	1.0 (7)
H(131)	-0.103 (4)	0.883 (4)	-0.018 (3)	1.7 (7)
H(132)	-0.051 (4)	0.878 (4)	0.108 (3)	3.2 (9)
H(141)	0.087 (3)	0.626 (4)	0.086 (3)	1.2 (7)
H(151)	0.141 (4)	0.817 (4)	-0.003 (3)	3.2 (9)
H(152)	0.188 (4)	0.825 (4)	0.125 (3)	2.6 (8)
H(161)	0.303 (4)	0.573 (4)	-0.007 (3)	2.6 (8)
H(171)	0.503 (5)	0.645 (5)	0.075 (4)	5.2 (0)
H(172)	0.428 (5)	0.800 (5)	0.129 (4)	4.3 (0)
H(173)	0.388 (5)	0.755 (5)	-0.007 (4)	5.0 (0)
H(211)	-0.400 (5)	0.803 (5)	0.343 (4)	5.6 (0)
H(212)	-0.304 (5)	0.896 (5)	0.414 (4)	5.6 (0)
H(213)	-0.339 (5)	0.912 (5)	0.304 (4)	5.6 (0)
H(221)	-0.173 (4)	0.657 (4)	0.406 (3)	1.7 (7)
H(231)	-0.095 (4)	0.838 (4)	0.317 (3)	2.3 (8)
H(232)	-0.044 (4)	0.833 (4)	0.437 (3)	2.9 (9)
H(241)	0.049 (3)	0.602 (3)	0.303 (2)	1.0 (7)
H(251)	0.142 (4)	0.793 (4)	0.331 (3)	3.0 (9)
H(252)	0.186 (5)	0.793 (5)	0.446 (3)	4.1 (0)
H(261)	0.332 (3)	0.555 (4)	0.444 (3)	1.5 (7)
H(271)	0.518 (5)	0.606 (5)	0.407 (4)	5.7 (0)
H(272)	0.416 (5)	0.740 (5)	0.360 (4)	5.7 (0)
H(273)	0.439 (5)	0.745 (5)	0.476 (4)	5.7 (0)
H(311)	-0.409 (5)	0.868 (5)	0.677 (4)	5.4 (0)
H(312)	-0.317 (5)	0.969 (5)	0.768 (4)	5.4 (0)
H(313)	-0.363 (5)	0.974 (5)	0.633 (4)	5.4 (0)
H(321)	-0.218 (3)	0.730 (3)	0.626 (3)	1.1 (7)
H(331)	-0.105 (4)	0.916 (4)	0.663 (3)	1.7 (7)
H(332)	-0.060 (4)	0.898 (4)	0.789 (3)	2.4 (8)
H(341)	0.070 (3)	0.660 (3)	0.757 (3)	1.0 (7)
H(351)	0.137 (4)	0.868 (4)	0.679 (3)	2.1 (8)
H(352)	0.193 (4)	0.841 (5)	0.804 (3)	3.8 (0)
H(361)	0.291 (3)	0.623 (4)	0.654 (3)	1.3 (7)
H(371)	0.484 (5)	0.689 (5)	0.727 (4)	5.1 (0)
H(372)	0.369 (5)	0.832 (5)	0.686 (4)	5.1 (0)
H(373)	0.429 (5)	0.805 (5)	0.818 (4)	5.1 (0)

Measurements were made by a Rigaku four-circle diffractometer. The $\omega-2\theta$ scan mode was applied, using graphite monochromatized Mo- $K\alpha$ radiation with a scan range of $\Delta\omega = 2.0 + 0.5 \tan \theta$. The intensity data were collected in the region of 1.0° to 55° for 2θ . Lorentz and polarization corrections were made but none for absorption and secondary extinction effects.

Out of 6064 possible reflections, 3348 independent reflections with $|F_o| > 3\sigma(|F_o|)$ were collected.

Structure Determination

The structure was determined directly using the program MULTAN 78.¹ Except for one oxygen atom, 29 non-hydrogen atoms were located on the E-map. The remaining oxygen atom was found on the difference Fourier map. The structure was refined by the block-diagonal-least-squares method. The weighting scheme used here was:

$$\begin{aligned} |F_o| < F_{\min}: \quad w &= 0.5 \\ F_{\min} \leq |F_o| \leq F_{\max}: \quad w &= 1.0 \\ F_{\max} < |F_o|: \quad w &= F_{\max}/|F_o| \end{aligned}$$

where $F_{\max} = 60.0$ and $F_{\min} = 20.0$. The thermal parameters of the hydrogen atoms were refined by isotropic temperature factors under the assumption that they were not less than 1.0. The atomic scattering factors were taken from International Tables for X-Ray Crystallography.¹¹ The positional parameters and the isotropic thermal factors for the non-hydrogen atoms and those for the hydrogen atoms are listed in Tables II and III, respectively.*

RESULTS AND DISCUSSION

The ORTEP² drawings and the numberings of all atoms are shown in Figure 1. The crystal

structures viewed along the *a* and *b* axes are shown in Figures 2 and 3, respectively. As in the case of DL-pentane-2,4-diol crystals,³ all the carbon atoms in a molecule are nearly on the same plane for each of the three independent molecules, and take TTTT conformation. All the OH groups form intermolecular hydrogen bonds, whose lengths are from 2.675 to 2.822 Å. The shortest van der Waals distances are 3.642 and 3.448 Å for C–C and C–O, respectively. These values are not significantly different from those obtained for ordinary organic compounds. The bond lengths and angles for independent three molecules are shown in Table IV. The mean values of C–C = 1.518, C–O = 1.424 Å and C–C–C = 112.8, and C–C–O = 109.7° are in good agreement with those obtained for other model compounds of PVA and polyols.^{3–8} The mean value of C–C–O bond angles are about 3° smaller than the C–C–C bond angles. The C–C–C bond angles of methylene carbon atoms are systematically about 3° larger than those of carbon atoms bonded to hydroxyl groups. These facts are in good agreement with syndiotactic 2,4-pentanediol,³ DL-3-chloropentane-2,4-diol⁵ and isotactic 2,4,6-heptanetriol.⁴ All the OH groups are linked together by intermolecular hydrogen bonds, each OH group acting as a proton donor as well as an acceptor. The hydrogen bonding distances and angles are shown in Table V.

As is easily understood from Figures 2 and 3, the directions of the carbon chains are all nearly in the direction of the *a*-axis, and the planes of the carbon chains are nearly parallel to each other. The plane constants of the three independent molecules, and the distances of each atom to the planes are listed in Tables VI and VII. The torsional angles of three independent molecules are listed in Table VIII. As can be understood from these values, the carbon chains take TTTT conformations. The

* The anisotropic thermal factors of the nonhydrogen atoms and the list of the observed and calculated structure factors will be provided by the author on request.

Figure 1. Stereodrawings and numberings of syndiotactic 2,4,6-heptanetriol: (a) the first molecule; (b) the second molecule; (c) the third molecule. The thermal ellipsoids are drawn at 30% probability level.

plane to the neighboring one are about $c/3$.

In the (010) plane, the molecules are packed together by van der Waals force of carbon atoms and in the (020) plane they are linked by OH-O hydrogen bonds.

This structure is very similar to that of PVA crystal since in the polymer, the molecules are linked together by hydrogen bonds in the (200) and packed by van der Waals forces in the (100) plane.

The dimer of syndiotactic PVA or DL-pentane-2,4-diol has a different structure; the independent two molecular planes intersect nearly perpendicularly and the two molecular axes are nearly at right angles.

Thus, the structure of PVA is more similar in several points to that of syndiotactic trimer than that of the dimer. We may expect that the longer the lengths of molecules of the model compounds, the more the structures are alike those of polymers. There might be some distinct differences between the oligomers and polymers in structure and properties. At least

dihedral angles between three independent molecules and those between the directions of molecular axes and the crystallographic axes are listed in Tables IX and X. The values listed in these tables indicate that the three molecular planes and the molecular axes are both nearly parallel to each other, and the three independent molecular planes are separated nearly equally in the c direction. The distances from a

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Figure 2. Crystal structure viewed along the *a*-axis. Broken lines indicate hydrogen bonds.

Figure 3. Crystal structure viewed along the *b*-axis. Broken lines are the same as in Figure 2.

in the case of syndiotactic oligomers, however, some of the characteristic features in PVA structure would emerge even in relatively low-molecular weight oligomers.

The conformation of the isotactic trimer is GTT $\bar{G}$ ⁴ and that of the syndiotactic dimer is TT³ and that of the syndiotactic trimer is TTTT, and in all cases, all the OH groups form intermolecular hydrogen bonds. From the

Table IV. Bond lengths (Å) and angles (°) of the three molecules

	Molecule 1 (<i>n</i> = 1)	Molecule 2 (<i>n</i> = 2)	Molecule 3 (<i>n</i> = 3)
C(<i>n</i> 1)–C(<i>n</i> 2)	1.513 (5)	1.501 (6)	1.519 (5)
C(<i>n</i> 2)–C(<i>n</i> 3)	1.517 (6)	1.520 (7)	1.521 (6)
C(<i>n</i> 3)–C(<i>n</i> 4)	1.525 (5)	1.524 (5)	1.525 (5)
C(<i>n</i> 4)–C(<i>n</i> 5)	1.522 (6)	1.517 (6)	1.523 (6)
C(<i>n</i> 5)–C(<i>n</i> 6)	1.527 (5)	1.508 (5)	1.520 (5)
C(<i>n</i> 6)–C(<i>n</i> 7)	1.500 (7)	1.523 (8)	1.514 (7)
C(<i>n</i> 2)–O(<i>n</i> 1)	1.419 (6)	1.432 (5)	1.411 (6)
C(<i>n</i> 4)–O(<i>n</i> 2)	1.431 (4)	1.432 (5)	1.426 (4)
C(<i>n</i> 6)–O(<i>n</i> 3)	1.417 (6)	1.422 (5)	1.426 (5)
C(<i>n</i> 1)–C(<i>n</i> 2)–C(<i>n</i> 3)	111.3 (3)	111.9 (4)	112.0 (3)
C(<i>n</i> 2)–C(<i>n</i> 3)–C(<i>n</i> 4)	114.2 (3)	115.2 (3)	113.6 (3)
C(<i>n</i> 3)–C(<i>n</i> 4)–C(<i>n</i> 5)	111.4 (3)	111.6 (3)	111.3 (3)
C(<i>n</i> 4)–C(<i>n</i> 5)–C(<i>n</i> 6)	114.9 (3)	113.8 (3)	115.4 (3)
C(<i>n</i> 5)–C(<i>n</i> 6)–C(<i>n</i> 7)	111.4 (3)	112.2 (4)	111.5 (4)
C(<i>n</i> 1)–C(<i>n</i> 2)–O(<i>n</i> 1)	107.1 (4)	110.1 (3)	110.8 (4)
C(<i>n</i> 3)–C(<i>n</i> 2)–O(<i>n</i> 1)	111.0 (3)	110.3 (4)	107.6 (3)
C(<i>n</i> 3)–C(<i>n</i> 4)–O(<i>n</i> 2)	110.6 (3)	111.7 (4)	109.9 (3)
C(<i>n</i> 5)–C(<i>n</i> 4)–O(<i>n</i> 2)	109.7 (3)	110.3 (3)	107.3 (3)
C(<i>n</i> 5)–C(<i>n</i> 6)–O(<i>n</i> 3)	107.3 (3)	110.7 (3)	111.5 (4)
C(<i>n</i> 7)–C(<i>n</i> 6)–O(<i>n</i> 3)	111.2 (3)	110.4 (4)	107.6 (3)
The mean values of C–C–C bond angles			
C–CH(OH)–C	111.4 (3)	111.9 (4)	111.6 (3)
C–CH ₂ –CH(OH)	114.6 (3)	114.5 (3)	114.5 (3)

Table V. The hydrogen bond distances and angles

<i>i</i>	<i>j</i>	<i>k</i>	<i>D</i> _{ij} /Å	<i>D</i> _{jk} /Å	<i>D</i> _{ik} /Å	∠ <i>ijk</i> (°)
O(11)–H(011)	O(12)		0.89	1.80	2.67	164.1
O(12)–H(012)	O(31)		0.89	1.90	2.69	145.9
O(13)–H(013)	O(33)		0.99	1.85	2.82	167.6
O(21)–H(021)	O(11)		1.07	1.64	2.70	167.2
O(22)–H(022)	O(32)		0.97	1.76	2.70	162.8
O(23)–H(023)	O(13)		1.08	1.67	2.73	163.9
O(31)–H(031)	O(23)		0.89	1.84	2.72	175.9
O(32)–H(032)	O(22)		0.77	1.72	2.70	163.2
O(33)–H(033)	O(21)		0.93	1.83	2.74	167.9

Table VI. The equations of best planes represented by $ax + by + cz = d$

	<i>a</i>	<i>b</i>	<i>c</i>	<i>d</i>
Molecule 1	–0.0854	–0.0943	0.9427	–0.1762
Molecule 2	–0.1328	–0.1664	0.9590	3.8465
Molecule 3	–0.1155	–0.1792	0.9537	8.2261

Table VII. Distances from each atom to the best planes in Å

	Molecule 1 (<i>n</i> = 1)	Molecule 2 (<i>n</i> = 2)	Molecule 3 (<i>n</i> = 3)
C(<i>n</i> 1)	0.021	0.006	–0.080
C(<i>n</i> 2)	–0.033	0.020	–0.007
C(<i>n</i> 3)	0.009	–0.005	0.092
C(<i>n</i> 4)	–0.033	–0.022	0.017
C(<i>n</i> 5)	0.023	–0.030	0.063
C(<i>n</i> 6)	0.065	0.009	–0.035
C(<i>n</i> 7)	–0.051	0.022	–0.051
O(<i>n</i> 1)	1.076	–1.089	1.121
O(<i>n</i> 2)	–1.215	1.074	–1.213
O(<i>n</i> 3)	1.286	–1.081	1.070

Table VIII. Torsion angles in degree of the three molecules

	Molecule 1 (<i>n</i> = 1)	Molecule 2 (<i>n</i> = 2)	Molecule 3 (<i>n</i> = 3)
C(<i>n</i> 1)–C(<i>n</i> 2)–C(<i>n</i> 3)–C(<i>n</i> 4)	179.6	–178.8	–173.8
C(<i>n</i> 2)–C(<i>n</i> 3)–C(<i>n</i> 4)–C(<i>n</i> 5)	–179.5	–179.4	177.6
C(<i>n</i> 3)–C(<i>n</i> 4)–C(<i>n</i> 5)–C(<i>n</i> 6)	176.7	177.6	–178.8
C(<i>n</i> 4)–C(<i>n</i> 5)–C(<i>n</i> 6)–C(<i>n</i> 7)	173.0	–179.1	177.1
C(<i>n</i> 4)–C(<i>n</i> 3)–C(<i>n</i> 2)–O(<i>n</i> 1)	60.5	–55.9	64.2
C(<i>n</i> 2)–C(<i>n</i> 3)–C(<i>n</i> 4)–O(<i>n</i> 2)	58.3	–55.3	59.0
C(<i>n</i> 6)–C(<i>n</i> 5)–C(<i>n</i> 4)–O(<i>n</i> 2)	–60.5	52.8	–58.6
C(<i>n</i> 4)–C(<i>n</i> 5)–C(<i>n</i> 6)–O(<i>n</i> 3)	–65.1	57.0	–63.0

Table IX. The dihedral angles between two molecular planes

Molecule 1—Molecule 2	4.42°
Molecule 2—Molecule 3	1.51°
Molecule 3—Molecule 1	4.92°

Table X. The angles between three molecular axes and the three crystallographic axes in degree

Molecule	Axes		
	<i>a</i>	<i>b</i>	<i>c</i>
Molecule 1	13.10	82.79	108.15
Molecule 2	13.50	82.34	106.13
Molecule 3	12.97	82.49	107.31

analytical results of the model compounds of PVA,³⁻⁵ we suppose that the OH groups tend to form intermolecular hydrogen bonds rather than to form intramolecular hydrogen bonds. However, the tendency of the main chain to take TT sequences cannot be overlooked. In the crystalline part of PVA, the carbon chains take the zigzag planar conformation irrespective of tacticity.^{10,12} In the case of isotactic or atactic polymers, there may be a considerable amount of intramolecular hydrogen bonds, but in the case of syndiotactic polymers, the OH groups may hardly form intramolecular hydrogen bonds.⁹

From these points of view, we suspect that the syndiotactic PVA is more crystallizable than isotactic or atactic polymers.

TT sequences in syndiotactic PVA are expected to give a 5.0 Å fiber period, but this period could not be found for syndiotactic polymers.¹⁷ As regards the syndiotactic 2,4,6-heptanetriol, the *hkl* reflections with *h* = 3*n* were found to be generally more intense. These reflections correspond to a 3.6 Å period along

the direction of the carbon chain molecule, but we must take into account the effect of the end groups. For the longer chain oligomers, the period probably reduces to 2.5 Å. There may be some complicated reasons why the 5.0 Å fiber period is absent in X-ray photographs of syndiotactic PVA. The fact, above-mentioned, might provide one of the reasons to explain the absence of the 5.0 Å period.

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