

## Monoclinic *trans*-2,5-Dimethyl-3-hexene-2,5-diol

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**Abstract.**  $C_8H_{16}O_2$ ,  $M_r = 144.22$ , monoclinic,  $C2/c$ ,  $Z = 8$ ,  $a = 10.354$  (3),  $b = 16.799$  (4),  $c = 11.014$  (3) Å,  $\beta = 112.0$  (2)°,  $V = 1776$  Å<sup>3</sup>,  $d_x = 1.077$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu(\text{Mo}) = 0.081$  mm<sup>-1</sup>. One of the two independent half molecules is disordered. Hydrogen bonds connect the molecules to form a three-dimensional network.

**Introduction.** Crystals of *trans*-2,5-dimethyl-3-hexene-2,5-diol were grown by slow evaporation of the solvent (6–14 d) from a saturated solution in petroleum ether (313–333 K). The batch of crystals contained two modifications: (a) the orthorhombic hemihydrate studied by Ruysink & Vos (1974), called II *trans* A; the accurate determination of the electron density distribution in this compound will be described in a separate paper (van der Wal & Vos, 1979); (b) a monoclinic compound called II *trans* B; the structure determination of this compound is reported in the present paper.

The intensities of II *trans* B were collected on a CAD-4 diffractometer at 86 K, in essentially the same way as described for II *trans* A by van der Wal & Vos (1979). Symmetry relations between the reflections and systematic absences indicated the space group to be  $Cc$  or  $C2/c$ . All independent reflections up to  $\sin \theta/\lambda = 0.9$  Å<sup>-1</sup> were measured at two different  $\psi$  values. During the data collection the intensities of three reference reflections decreased by 10% on average. For each of the 5480 independent reflections the weighted average of the two measurements was taken as  $I_o(\mathbf{H})$ , whose e.s.d. was calculated as  $\sigma[I_o(\mathbf{H})] = [\sum_{i=1,2} \sigma_c^{-2}(\mathbf{H}, i)]^{-1/2}$ , where  $\sigma_c$  is the e.s.d. based on counting statistics. The distribution of the  $E$  values pointed to the space group  $C2/c$ . An approximate structure was found by the use of *MULTAN* (Germain, Main & Woolfson, 1971). The cell contains a molecule  $\alpha$  lying around the inversion center (0,0,0) and a molecule  $\beta$  around ( $\frac{1}{4}, \frac{1}{4}, \frac{1}{2}$ ), the respective inversion centers coinciding with the centers of the C=C double bonds. There are no water molecules. After isotropic refinement, Fourier syntheses showed  $\beta$  to be disordered. The two superimposed molecules are given in Fig. 1. For the solid

circles a population of 0.79 was found after refinement and for the open circles 0.21. The plane through the open circles for C(7), C(8) and H(16) almost coincides with the  $ac$  plane. The corresponding plane through the solid circles is nearly perpendicular to  $ac$ . H atoms linked to C were found from difference maps and were taken into account in the refinement with C–H = 1.08 Å and with isotropic thermal parameters. H atoms linked to O were not found and thus not considered in the structure refinement. The function minimized was  $Q(I) = \sum_{\mathbf{H}} w(\mathbf{H}) [I_o(\mathbf{H}) - K^2 I_c(\mathbf{H})]^2$ , with  $[w(\mathbf{H})]^{-1} = \sigma^2[I_o(\mathbf{H})] + 0.0004 I_o^2(\mathbf{H})$  for the 4062 independent reflections with  $I_o(\mathbf{H}) > 2\sigma[I_o(\mathbf{H})]$ . Scattering factors for C and O were taken from Cromer & Mann (1968),

Table 1. Final positional parameters ( $\times 10^5$ )

Numbers in parentheses are the e.s.d.'s in the last digits. For numbering, see Fig. 3.

	$x$	$y$	$z$
Molecule $\alpha$			
O(1)	10500 (9)	8662 (6)	20461 (9)
C(1)	28879 (11)	6753 (7)	12695 (11)
C(2)	22260 (12)	-3798 (7)	25469 (10)
C(3)	16874 (10)	2735 (6)	15240 (10)
C(4)	6744 (10)	-753 (6)	2656 (10)
Molecule $\beta$ (0.79)			
O(2)	-5082	24905	35014
C(5)	1009	23573	58551
C(6)	4736	35902	47346
C(7)	5452	27122	47655
C(8)	18887	23187	48275

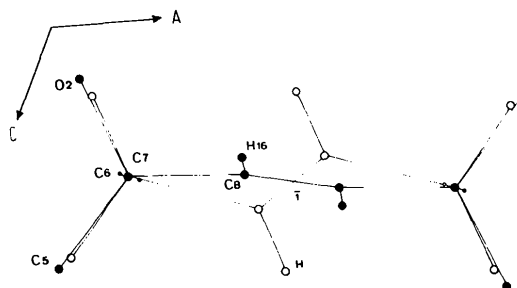


Fig. 1. The disorder of molecule  $\beta$ .

and for H from Stewart, Davidson & Simpson (1965). First, anisotropic refinement with reflections having  $\sin \theta/\lambda < 0.65 \text{ \AA}^{-1}$  was carried out for  $\alpha$  and the split atoms of  $\beta$ . Thereafter the parameters of  $\beta$  were fixed, whereas those of  $\alpha$  were refined further with all 4062 independent reflections. This procedure was followed to make the low-order ( $\sin \theta/\lambda < 0.65 \text{ \AA}^{-1}$ ) difference map for the study of the electron density of  $\alpha$  as flat as possible around  $\beta$ .  $R_w(I) = \sum_{\mathbf{H}} w(\mathbf{H}) [I_o(\mathbf{H}) - I_c(\mathbf{H})]^2 / [\sum_{\mathbf{H}} I_o^2(\mathbf{H})]$  decreased to 0.150, [ $R(F) = 0.081$ ].\* Final positional parameters of  $\alpha$  and  $\beta$  (0.79) are listed in Table 1, and geometric data for  $\alpha$  in Table 2. The less accurate values for  $\beta$  do not deviate significantly from the  $\alpha$  values. The calculations were carried out with the XRAY system (1976).

\* Lists of structure factors, anisotropic heavy-atom thermal parameters and parameters for the H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34338 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Geometric data for molecule  $\alpha$

Symmetry code:  $X(n^1) = X(n; \bar{x}, \bar{y}, \bar{z})$ .

Bond lengths (Å) and angles (°)

C(4)—C(4 <sup>1</sup> )	1.321 (1)	O(1)—C(3)—C(2)	107.53 (9)
C(4)—C(3)	1.509 (1)	O(1)—C(3)—C(4)	111.93 (8)
C(3)—C(2)	1.521 (1)	C(1)—C(3)—C(2)	110.87 (8)
C(3)—C(1)	1.530 (2)	C(1)—C(3)—C(4)	109.64 (9)
C(3)—O(1)	1.429 (2)	C(2)—C(3)—C(4)	109.65 (8)
O(1)—C(3)—C(1)	107.20 (9)	C(3)—C(4)—C(4 <sup>1</sup> )	126.06 (10)

Equation of the C(3)—C(4)—C(4<sup>1</sup>) plane ( $X, Y, Z$  orthonormal axes parallel to  $\mathbf{a}, \mathbf{b}, \mathbf{c}^*$ ) and distances of atoms to plane in Å

$$0.3992X + 0.7610Y - 0.5115Z = 0 \text{ \AA}$$

C(3) 0.0; C(4) 0.0; O(1) 0.14; C(1) 1.18; C(2) -1.32

The standard deviation in the distances is less than 1 in the last decimal place.

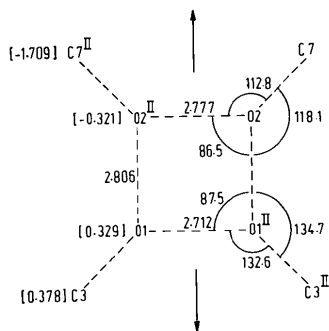
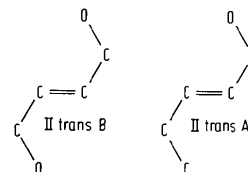


Fig. 2. Geometric data for the hydrogen-bonding system. The projection on to the best plane of the quadrangle is shown. Deviations from the plane (in Å) are given in square brackets.  $X^{\text{II}} = X(\bar{x}, y, \bar{z} + \frac{1}{2})$ .

**Discussion.** In contradistinction to II *trans B*, the molecules in II *trans A* do not have a centrosymmetric conformation, as can be seen from the arrangement of the atoms approximately lying in the central ethylene planes.



The bond lengths and angles in II *trans A* have been discussed by Ruysink & Vos (1974). The values for II *trans B* are comparable with those in the part of the II *trans A* molecule with a similar conformation. Differences in corresponding bond lengths are up to 0.008 Å, and in angles up to 1.8°.

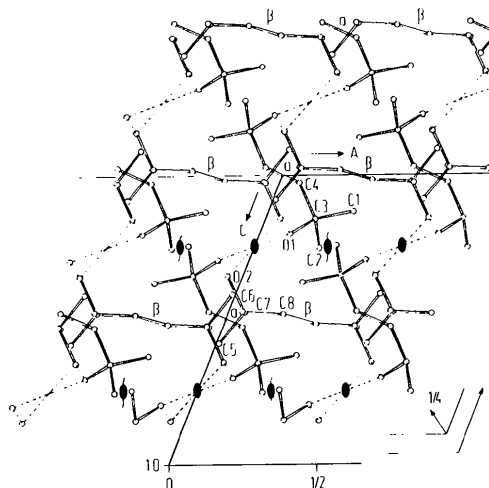


Fig. 3. Projection of the structure along [010] on to the plane (010). The centers of the  $\alpha$  molecules lie at height 0 (in the origin) and  $\frac{1}{4}$ , and those of  $\beta$  at  $+\frac{1}{4}$  and  $-\frac{1}{4}$ .

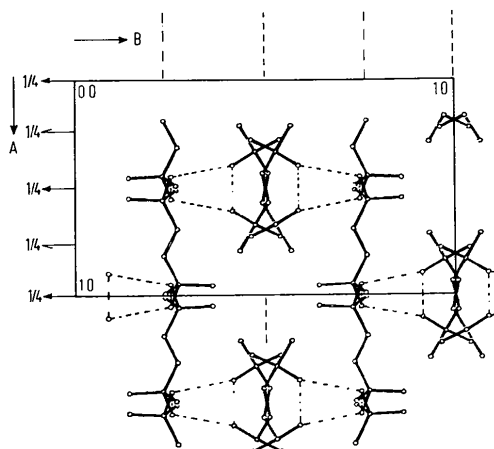


Fig. 4. The structure seen along  $\mathbf{c}$ .

Around the twofold axes there are quadrangles of O atoms with short O...O distances (Fig. 2) which have been assumed to represent hydrogen bonds and are given by dashed lines in Figs. 3 and 4. Fig. 3 depicts the connection of the molecules by hydrogen bonds in the *ac* plane, whereas Fig. 4 illustrates the way in which the hydrogen-bonding system is extended in the (**a + b**) and (**a - b**) directions, so that a three-dimensional network is formed. Fig. 2 shows that the shortest O...O distance occurs between atoms of type O(1) around which the largest deviations from tetrahedral angular values are observed. The hydrogen-bonding system is hardly affected by the disorder of  $\beta$ , as the O atoms of the superimposed molecules nearly coincide (Fig. 1). Because of the disorder the deformation density of  $\alpha$  could not be determined accurately.

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### (1*S*,3*R*,4*S*)-1-Methyl-3-(4-methoxyphenoxy-methyl)-4-phenylpiperidinium Chloride (FG4963): a Selective Inhibitor of Serotonin Uptake

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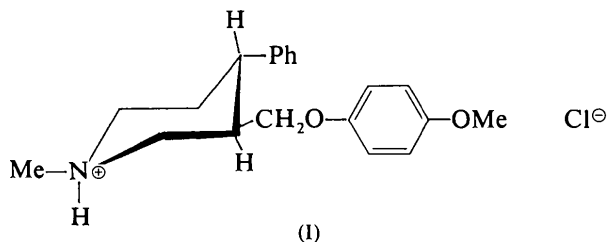
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**Abstract.** C<sub>20</sub>H<sub>26</sub>NO<sub>2</sub><sup>+</sup>.Cl<sup>-</sup>, *M<sub>r</sub>* = 347.89, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 6.004 (4), *b* = 11.398 (10), *c* = 28.587 (17) Å, *U* = 1956 Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.180 Mg m<sup>-3</sup>,  $\mu$ (Cu *K*α) = 1.7 mm<sup>-1</sup>. The structure was refined to *R* = 0.041 for 2520 unique reflexions. Absolute configurations at N(1) and C(4) are *S*, and at C(3) *R*. Cl<sup>-</sup> is hydrogen bonded to the positively charged N atom.

**Introduction.** The tricyclic antidepressants such as imipramine (Post, Kennard & Horn, 1975) and chlorimipramine (Post & Horn, 1977) inhibit the neuronal uptake of the biogenic amines noradrenaline (NA) and serotonin (5-hydroxytryptamine, 5-HT); this property may be related to their clinical mode of action (Horn,

1976). There is currently interest in developing more selective inhibitors of biogenic amine uptake in the hope of learning more about the neurochemical mechanisms involved in depressive states. A recently developed drug which has a more selective effect on 5-HT than on NA uptake is (3*R*,4*S*)-1-methyl-3-(4-methoxyphenoxy-methyl)-4-piperidine hydrochloride (I), which has the trivial name FG4963 (Lassen, Petersen, Kjellberg & Olsson, 1975). We report here the structure of this compound.



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