

The perchlorate counter ions, well shielded from the neodymium cations, fill up the voids between the polymeric chains together with two non-coordinating crystal water molecules. The observed Cl—O bond lengths in the disordered ClO_4^- groups range from 1.34 to 1.64 Å, with a calculated average distance of 1.43 [6] Å (the root-mean-square deviation is given in square brackets), which is comparable with the commonly accepted mean value of 1.44 Å for such bonds (Ondik & Smith, 1968).

The numerous examples of intermolecular contact distances shorter than 3.3 Å between the non-H atoms (Table 8, deposited) indicate that besides the possible hydrogen bonds (Table 8) the crystal structure seems to be stabilized by an electrostatically favourable packing.

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Structure of the Crystalline Dimer of α -Hydroxyisobutyrophenone: (*R,S*)-3,3,6,6-Tetramethyl-1,4-diphenyl-2,5,7-trioxabicyclo[2.2.1]heptane

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Abstract. $\text{C}_{20}\text{H}_{22}\text{O}_3$, $M_r = 310.39$, monoclinic, $P2_1/c$, $a = 16.58$ (3), $b = 8.34$ (1), $c = 24.35$ (3) Å, $\beta = 91.2$ (2)°, $V = 3367$ (8) Å³, $Z = 8$, $D_x = 1.224$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.76$ cm⁻¹, $F(000) = 1328$, $T = 296$ K, $R = 0.077$, 1712 unique observed reflections. The crystalline compound slowly formed from α -hydroxyisobutyrophenone and assigned various structures over the past 100 years, but

none unequivocally, is shown to be racemic 3,3,6,6-tetramethyl-1,4-diphenyl-2,5,7-trioxabicyclo-[2.2.1]heptane. The *R* and *S* enantiomers in the asymmetric unit are related to each other by a pseudocenter of inversion.

Introduction. While it has long been recognized that α -hydroxy ketones dimerize into crystalline or glassy solids, the chemical structure of these products has remained controversial. Four types of dimeric structures have generally been proposed, *viz*: bis- β -keto ethers (I) (Favorskii & Mandryka, 1912; Favorskii,

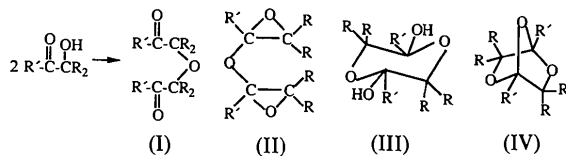
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1913; cf. Temnikova & Spasskova, 1946); bis-epoxy ethers (II) (Favorskii, 1913; Scheibler & Fischer, 1922); and dioxanes (III)/trioxabicyclo[2.2.1]heptanes (IV) alone or as mixtures (Fischer, 1895; Fron-



ing & Hennion, 1940; Temnikova & Spasskova, 1946; Temnikova & Kropacheva, 1949; Elphimoff-Felkin & Tchoubar, 1952; Temnikova & Almashi, 1953; Asinger, Thiel, Usbeck, Grösse, Grundmann & Tränkner, 1960). Our initial study of the crystalline product formed from α -hydroxyisobutyrophenone confirmed its dimeric nature, and IR and ¹H/¹³C NMR spectroscopy supported a structure like (IV) but did not rule out other possibilities with certainty (Kolb, 1976; Meyers, Matthews, Ho, Kolb & Parady, 1977; Meyers & Kolb, 1978). Unequivocal characterization required X-ray crystallography.

Experimental. *In situ* preparation of α -hydroxyisobutyrophenone and its crystalline dimer from isobutyrophenone:* To a stirred, cooled mixture of well powdered KOH (12 g), CCl₄ (15 mL) and ^tBuOH (15 mL), isobutyrophenone (1.48 g, 0.010 mol) was added and stirring was continued at room temperature for 5 h. The mixture was diluted with water and extracted with ether. The extract was washed with dilute HCl then water, dried over anhydrous MgSO₄, filtered and vacuum-evaporated, leaving 1.46 g (90% yield) of a slightly yellow viscous oil identified as α -hydroxyisobutyrophenone (b.p., IR, ¹H NMR). Crystals slowly formed from the oil. After a few hours the mass was washed several times with cold ether, leaving a crop of white crystals, m.p. 463–463.5 K (corr.); recrystallization (ether) did not alter the m.p. [literature m.p. 458–459 K (Favorskii, 1913; Temnikova & Almashi, 1953)]. Further crops of crystals formed in the ether washings. IR (Beckman IR-5A; Nujol): 1064–1010 cm⁻¹ (broad, three strong peaks; C—O—C stretch); C=O and OH absorption were absent. ¹H NMR (Varian VXR 500; CDCl₃; δ): 0.970 (*s*, CH₃, 6 H), 1.555 (*s*, CH₃, 6 H), 7.36–7.42 (*m*, Ph *m*- and *p*-H, 6 H), and 7.61 (*dd*, *J* = 7.5, 2.0 Hz, Ph *o*-H, 4 H). ¹³C NMR (XL-100; CDCl₃; δ): 20.32 (CH₃), 26.89 (CH₃), 87.56 [C(CH₃)₂], 109.75 (O—C—O), 125.76, 127.80 and

128.57 (Ph-ring CH), 133.48 (Ph-ring C-substituted). Analysis (Galbraith Microanalytical Laboratory) calculated for C₂₀H₂₂O₃: *MW*, 310; C, 77.42; H, 7.10%. Found: *MW* 321, 328; C, 77.00; H, 6.96%. Repeated recrystallizations from ether failed to provide crystals amenable to X-ray crystallographic analysis. Recrystallization from refluxing hexane afforded the crystals (m.p. 462–462.5 K, corr.) successfully examined in this study.

The crystal used for data collection was 0.28 × 0.22 × 0.19 mm, colorless, multifaced. Rigaku AFC-5S diffractometer, graphite-monochromated Mo *K* α radiation, ω scans, scan speed 4° min⁻¹ (in ω); weak reflections [$I < 10.0\sigma(I)$] rescanned (maximum of two rescans) and the counts accumulated to assure good counting statistics. Lattice parameters were from the least-squares fit of 25 strong reflections in the 2θ range 20–24°. A total of 5923 reflections were measured (h 0 to 19, k 0 to 9, l –28 to 28), 4211 reflections considered unobserved with [$I < 2\sigma(I)$], data set comprised of 1712 unique reflections, $(\sin\theta/\lambda)_{\max} = 0.60 \text{ \AA}^{-1}$. Three standard reflections (204, 202, 312) changed by –1.6, –0.4 and –0.3%, respectively; no decay correction applied. The data were corrected for Lorentz and polarization (no absorption correction). The direct-methods program *MITHRIL* (Gilmore, 1984) provided the locations of all non-H atomic positions. Full-matrix least-squares refinement was performed to minimize $\sum w(|F_o| - |F_c|)^2$ where $w = 4F_o^2/\sigma^2(F_o^2)$ and $\sigma^2(F_o^2) = [S^2(C + R^2B) + (pF_o^2)^2]/Lp^2$ (S = scan rate, C = total integrated peak count, R = ratio of scan time to background counting time, B = total background count, Lp = Lorentz and polarization factor, and $p = 0.03$). After convergence, methyl and phenyl H atoms were placed at assumed positions (C—H = 0.95 Å), the methyl-group orientations were determined on the basis of H positions obtained from a difference Fourier synthesis; the H positions were not refined. Final stages of refinement were performed with 416 variables including all non-H positional and anisotropic thermal parameters, one scale factor, and a secondary-extinction coefficient (0.11868×10^{-6}). Convergence yielded $R = 0.077$, $wR = 0.066$, $S = 1.66$ and $(\Delta/\sigma)_{\max} = 0.03$. Final difference synthesis produced $(\Delta\rho)_{\max} = 0.29$ and $(\Delta\rho)_{\min} = -0.34 \text{ e \AA}^{-3}$. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71, 148). All computer programs from the *TEXSAN* package (Molecular Structure Corporation, 1985).

The two enantiomers which are present in the asymmetric unit are related to each other by a pseudocenter of inversion at $\frac{1}{4}, \frac{1}{8}, \frac{1}{4}$ (in addition to the true symmetry centers of space group $P2_1/c$). All attempts at re-indexing the search reflections failed

* α -Chloroisobutyrophenone is formed as an unisolated intermediate in this method (Meyers & Kolb, 1978). Thus, it or α -bromoisobutyrophenone also served well as starting materials (although much more costly) without the need of CCl₄ or other halogenating agents.

Table 1. *Positional and equivalent isotropic thermal parameters (\AA^2) for non-H atoms with their e.s.d.'s in parentheses*

$$B_{\text{eq}} = (8\pi^2/3)[U_{22} + 1/\sin^2\beta(U_{11} + U_{33} + 2U_{13}\cos\beta)].$$

Molecule 1	x	y	z	B_{eq}
C(1)	0.3320 (6)	0.212 (1)	0.1157 (5)	2.7 (6)
O(2)	0.2931 (4)	0.0992 (8)	0.0801 (3)	2.5 (4)
C(3)	0.3436 (7)	-0.044 (1)	0.0826 (4)	2.9 (6)
C(4)	0.4168 (6)	0.021 (1)	0.1154 (4)	2.0 (5)
O(5)	0.4564 (4)	0.1428 (9)	0.0828 (3)	3.4 (4)
C(6)	0.4043 (7)	0.282 (1)	0.0855 (4)	3.2 (6)
O(7)	0.3751 (4)	0.1158 (8)	0.1559 (3)	2.7 (4)
C(8)	0.449 (1)	0.416 (2)	0.1148 (6)	6 (1)
C(9)	0.3845 (9)	0.337 (2)	0.0270 (7)	6 (1)
C(10)	0.3014 (7)	-0.173 (2)	0.1107 (5)	4.8 (8)
C(11)	0.366 (1)	-0.096 (2)	0.0240 (7)	7 (1)
C(12)	0.2718 (7)	0.326 (2)	0.1425 (6)	3.5 (7)
C(13)	0.2739 (7)	0.358 (2)	0.1957 (5)	3.7 (7)
C(14)	0.2188 (9)	0.457 (2)	0.2173 (5)	4.6 (7)
C(15)	0.1618 (8)	0.531 (2)	0.1838 (7)	5.0 (8)
C(16)	0.1572 (7)	0.494 (1)	0.1300 (6)	3.8 (7)
C(17)	0.2141 (7)	0.396 (2)	0.1071 (5)	3.8 (7)
C(18)	0.4794 (7)	-0.091 (2)	0.1408 (6)	3.9 (7)
C(19)	0.4701 (7)	-0.141 (2)	0.1949 (5)	4.1 (7)
C(20)	0.5254 (8)	-0.253 (2)	0.2167 (5)	4.9 (7)
C(21)	0.5886 (8)	-0.307 (2)	0.1846 (6)	4.6 (8)
C(22)	0.5949 (7)	-0.251 (2)	0.1313 (6)	4.0 (7)
C(23)	0.5402 (7)	-0.141 (1)	0.1091 (5)	3.3 (6)

Molecule 2	x	y	z	B_{eq}
C(1')	0.1657 (7)	0.044 (1)	0.3887 (5)	2.7 (6)
O(2')	0.2009 (4)	0.157 (1)	0.4269 (3)	3.5 (4)
C(3')	0.1490 (6)	0.298 (1)	0.4243 (5)	2.7 (5)
C(4')	0.0792 (7)	0.226 (1)	0.3874 (5)	3.3 (6)
O(5')	0.0407 (4)	0.1108 (9)	0.4175 (3)	3.4 (4)
C(6')	0.0922 (6)	-0.034 (1)	0.4167 (5)	3.1 (6)
O(7')	0.1269 (4)	0.1412 (8)	0.3488 (3)	2.7 (4)
C(8')	0.0506 (6)	-0.158 (2)	0.3775 (6)	4.1 (7)
C(9')	0.1096 (8)	-0.098 (2)	0.4736 (5)	4.0 (7)
C(10')	0.1972 (8)	0.430 (2)	0.3933 (6)	4.9 (8)
C(11')	0.1265 (8)	0.349 (2)	0.4795 (4)	3.8 (7)
C(12')	0.2277 (6)	-0.063 (1)	0.3666 (4)	1.7 (5)
C(13')	0.2274 (7)	-0.100 (1)	0.3104 (5)	4.1 (7)
C(14')	0.2852 (8)	-0.206 (2)	0.2893 (5)	4.7 (7)
C(15')	0.3436 (7)	-0.271 (2)	0.3241 (6)	4.6 (8)
C(16')	0.3415 (8)	-0.237 (2)	0.3797 (6)	4.9 (8)
C(17')	0.2866 (6)	-0.130 (1)	0.3996 (4)	3.0 (6)
C(18')	0.0230 (7)	0.340 (2)	0.3615 (4)	1.8 (5)
C(19')	0.0297 (7)	0.395 (1)	0.3092 (5)	3.7 (7)
C(20')	-0.0247 (9)	0.502 (2)	0.2854 (5)	4.6 (8)
C(21')	-0.0878 (8)	0.556 (2)	0.3152 (6)	4.5 (8)
C(22')	-0.0989 (8)	0.505 (2)	0.3671 (7)	5.0 (8)
C(23')	-0.0438 (8)	0.400 (2)	0.3900 (5)	4.3 (7)

on any cell other than the observed cell. The programs *TRACER* (Lawton & Jacobson, 1965), *CELLS* (Svensson, 1988), *NEWLAT* (Mugnoli, 1985), and *MISSYM* (Le Page, 1987) were used to detect any higher symmetry, but none was observed. The crystal diffracted strongly at the lower 2θ angles and the search reflections were sharp and symmetrical. Thus, it is improbable that an alternative cell was overlooked owing to poor data quality. Additional examples of racemates with noncrystallographic centers of symmetry are discussed by Jacques, Collet & Wilen (1981). The higher than desirable overall R values are a result of the unusually large number of weak or unobserved high-angle reflections. The presence of pseudoextinctions, the most prevalent being $h0l$, $h \neq 2n$, and $0kl$, $l \neq 2n$ (whose rules are violated at low angle), explain many of the absences. In addition, parity groups *eee* and *oeo*

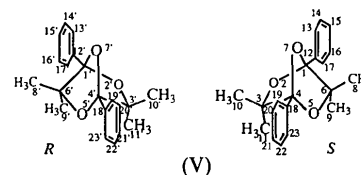
Table 2. *Selected bond distances (\AA) and bond angles ($^\circ$) with their e.s.d.'s in parentheses*

	Mol-ecule 1	Mol-ecule 2*	Mol-ecule 1	Mol-ecule 2*	
C(1)—O(2)	1.43 (1)	1.44 (1)	C(3)—C(11)	1.54 (2)	1.47 (1)
C(1)—C(6)	1.53 (1)	1.55 (1)	C(4)—O(5)	1.45 (1)	1.37 (1)
C(1)—O(7)	1.45 (1)	1.41 (1)	C(4)—O(7)	1.45 (1)	1.43 (1)
C(1)—C(12)	1.53 (2)	1.47 (1)	C(4)—C(18)	1.52 (2)	1.47 (2)
O(2)—C(3)	1.46 (1)	1.45 (1)	O(5)—C(6)	1.45 (1)	1.48 (1)
C(3)—C(4)	1.54 (1)	1.57 (1)	C(6)—C(8)	1.51 (2)	1.56 (2)
C(3)—C(10)	1.46 (2)	1.57 (2)	C(6)—C(9)	1.53 (2)	1.51 (2)
O(2)—C(1)—C(6)	107.7 (9)	107.6 (9)	C(3)—C(4)—O(7)	99.5 (8)	98.8 (8)
O(2)—C(1)—O(7)	104.8 (8)	104.1 (9)	C(3)—C(4)—C(18)	121 (1)	117 (1)
O(2)—C(1)—C(12)	112.2 (9)	111.0 (9)	O(5)—C(4)—O(7)	102.8 (8)	106.2 (9)
C(6)—C(1)—O(7)	98.9 (8)	101.1 (8)	O(5)—C(4)—C(18)	109.8 (9)	112.7 (9)
C(6)—C(1)—C(12)	120 (1)	118 (1)	O(7)—C(4)—C(18)	112.9 (9)	113 (1)
O(7)—C(1)—C(12)	112 (1)	114.1 (9)	C(4)—O(5)—C(6)	104.8 (8)	106.7 (8)
C(1)—O(2)—C(3)	105.4 (8)	105.6 (7)	C(1)—C(6)—O(5)	101.1 (9)	96.9 (8)
O(2)—C(3)—C(4)	100.0 (9)	98.3 (8)	C(1)—C(6)—C(8)	115 (1)	111 (1)
O(2)—C(3)—C(10)	110.0 (9)	106.4 (9)	C(1)—C(6)—C(9)	114 (1)	115 (1)
O(2)—C(3)—C(11)	110 (1)	111.1 (9)	O(5)—C(6)—C(8)	109 (1)	107.6 (9)
C(4)—C(3)—C(10)	114 (1)	111.8 (9)	O(5)—C(6)—C(9)	109 (1)	112 (1)
C(4)—C(3)—C(11)	112 (1)	116 (1)	C(8)—C(6)—C(9)	108 (1)	114 (1)
C(10)—C(3)—C(11)	111 (1)	112 (1)	C(1)—O(7)—C(4)	94.5 (8)	94.6 (8)
C(3)—C(4)—O(5)	109.0 (9)	108 (1)			

* Atom designators for this column should be primed.

yielded only about half as many observed reflections as the remaining six groups, further reducing the size of the data set. The low-angle reflections ($\sin\theta/\lambda = 0.0-0.348 \text{ \AA}^{-1}$, 658 reflections) produce R and wR values of 0.041 and 0.045 while the 341 strongest reflections yield R and wR values of 0.023 and 0.028 in the final cycle of refinement.

Discussion. Atomic coordinates and equivalent isotropic temperature factors are shown in Table 1; selected interatomic distances and angles are presented in Table 2.* The structure determined by X-ray diffraction (Fig. 1) leaves no doubt that the crystalline product formed from α -hydroxyisobutyrophenone on standing is racemic 3,3,6,6-tetramethyl-1,4-diphenyl-2,5,7-trioxabicyclo[2.2.1]heptane (V), *i.e.* structure type (IV) where $R = \text{methyl}$ and $R' = \text{phenyl}$. This structure was supported by the ^{13}C NMR assignments and our other data but could not be unequivocally characterized from them *per se*.



* Lists of structure factors, H-atom coordinates, anisotropic thermal parameters, bond distances and angles involving the phenyl rings, intermolecular distances, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54781 (47 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0353]

Cyclic dimerization of achiral α -hydroxyisobutyrophenone *via* reciprocal addition of hydroxyl to prochiral carbonyl, a dissymmetric process, followed by loss of one H₂O leads to the formation of rigid bicyclic (V), which must be a dissymmetric enantiomeric pair. Modest separation of *R* and *S* enantiomers of (V) has now been accomplished.* The stereochemistry of each enantiomer is clearly defined in the X-ray structure. The packing of the *R,S* pairs within the unit cell is shown in Fig. 2. There is no indication of *R,R* or *S,S* pairing.

From this study it seems reasonable that the crystalline products similarly formed from other

* A sample of our crystalline (V) on a Regis covalent *N*-(3,5-dinitrobenzoyl)- α -phenylglycine column (297 K), with hexane as the mobile phase, was modestly separated into two peaks of equal area representing the two enantiomers.

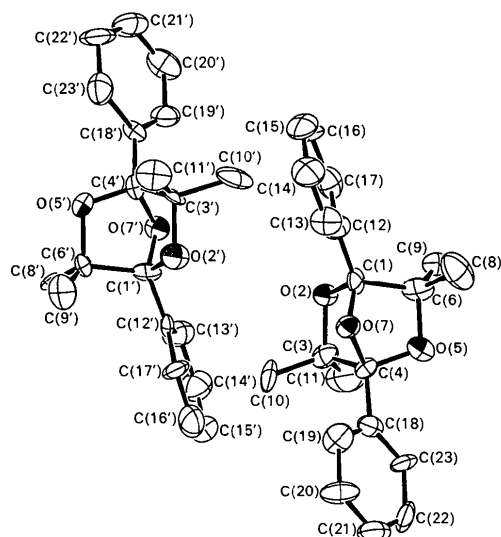


Fig. 1. Molecular configuration and IUPAC atom-numbering scheme, thermal ellipsoids at the 50% probability level. Note the pseudocenter of symmetry between the two enantiomers (the *S* enantiomer is on the right). The H atoms have been omitted to improve clarity.

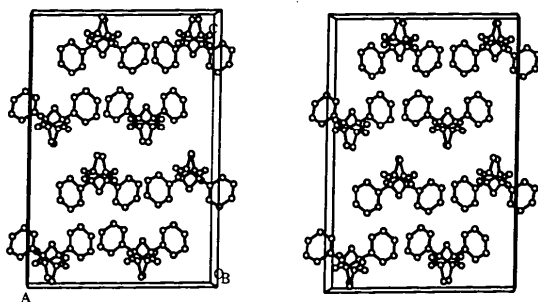


Fig. 2. Stereoscopic illustration of the molecular packing.

α -hydroxy ketones and related systems cited in the literature over many years but not definitively characterized (see above) likewise have structures corresponding to (IV) or its precursor, (III).

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