

C24	0.6877 (7)	0.5434 (15)	0.2308 (2)	0.067 (12)
C19	0.7282 (7)	0.4884 (15)	0.2611 (2)	0.058 (12)
C25	0.5488 (7)	0.5927 (19)	0.0871 (2)	0.061 (13)
O26	0.5607 (5)	0.7691 (12)	0.0882 (2)	0.069 (10)
O27	0.4542 (4)	0.4880 (11)	0.0915 (1)	0.065 (9)
C28	0.3482 (7)	0.5921 (26)	0.1011 (3)	0.108 (21)
C29	0.3654 (10)	0.7025 (32)	0.1330 (3)	0.153 (33)
C30	0.3101 (9)	0.7422 (26)	0.0753 (3)	0.140 (26)
C31	0.2700 (9)	0.4105 (25)	0.1033 (3)	0.125 (26)
O32	0.6692 (5)	0.1603 (8)	0.1322 (1)	0.058 (7)
O33	0.6112 (5)	0.5038 (10)	0.1512 (1)	0.075 (8)
C34	0.6354 (9)	0.0363 (15)	0.1587 (2)	0.070 (13)
O35	0.7816 (5)	0.3106 (10)	0.0372 (1)	0.072 (8)
O36	0.7030 (6)	0.6496 (9)	0.0296 (1)	0.069 (8)
C37	0.5788 (8)	0.3359 (16)	0.0142 (2)	0.052 (13)
C38	0.5448 (13)	0.1344 (19)	0.0272 (3)	0.081 (24)
C39	0.6359 (9)	0.3161 (22)	-0.0190 (2)	0.080 (19)
C40	0.4817 (9)	0.4808 (23)	0.0119 (2)	0.093 (21)

Table 2. Geometric parameters (Å, °)

S1—C2	1.854 (7)	C14—C15	1.367 (13)
S1—O32	1.627 (6)	C14—O18	1.399 (10)
S1—O33	1.476 (7)	C15—C16	1.387 (12)
S2—C1	1.839 (7)	C16—C17	1.375 (13)
S2—O35	1.439 (7)	O18—C19	1.371 (11)
S2—O36	1.424 (7)	C28—C30	1.547 (20)
S2—C37	1.824 (10)	C28—C31	1.537 (21)
C1—C2	1.575 (10)	O32—C34	1.446 (10)
C1—C25	1.538 (13)	C37—C38	1.509 (17)
C2—C3	1.507 (11)	C37—C39	1.563 (12)
C3—C6	1.514 (10)	C37—C40	1.519 (16)
C3—C7	1.550 (10)	C20—C21	1.420 (19)
C4—C6	1.499 (12)	C21—C22	1.395 (22)
C6—C7	1.564 (10)	C22—C23	1.369 (20)
C7—C8	1.479 (12)	C23—C24	1.374 (14)
C8—O9	1.195 (11)	C24—C19	1.414 (12)
C8—O10	1.375 (8)	C25—O26	1.187 (15)
O10—C11	1.480 (10)	C25—O27	1.347 (11)
C11—C12	1.506 (11)	O27—C28	1.506 (13)
C12—C13	1.410 (10)	C28—C29	1.547 (19)
C12—C17	1.373 (13)	C20—C19	1.386 (14)
C13—C14	1.394 (11)		
C2—S1—O32	94.6 (3)	C3—C7—C8	120.6 (7)
O32—S1—O33	108.9 (3)	C7—C8—O9	129.1 (8)
C1—S2—O36	108.7 (4)	O9—C8—O10	123.3 (7)
O35—S2—O36	119.6 (4)	O10—C11—C12	111.0 (6)
O36—S2—C37	108.8 (4)	C11—C12—C17	121.3 (7)
S2—C1—C25	112.9 (6)	C13—C14—C15	121.8 (8)
S1—C2—C1	107.6 (5)	C15—C14—O18	119.6 (7)
C1—C2—C3	117.2 (7)	C15—C16—C17	121.1 (8)
C2—C3—C7	119.7 (7)	C14—O18—C19	118.6 (7)
C3—C6—C4	114.6 (7)	C21—C20—C19	116.8 (11)
C3—C6—C7	60.5 (5)	C21—C22—C23	122.1 (13)
C4—C6—C7	113.7 (7)	C23—C24—C19	121.5 (9)
C3—C7—C6	58.2 (5)	O18—C19—C24	123.5 (8)
C6—C7—C8	120.5 (6)	C1—C25—O26	126.1 (9)
C7—C8—O10	107.6 (7)	O26—C25—O27	127.8 (9)
C8—O10—C11	114.2 (6)	O27—C28—C29	110.0 (10)
C11—C12—C13	118.8 (7)	O27—C28—C31	99.7 (9)
C13—C12—C17	119.9 (5)	C29—C28—C31	113.9 (11)
C12—C13—C14	118.3 (7)	S1—O32—C34	114.3 (5)
C13—C14—O18	118.5 (7)	S2—C37—C39	102.8 (7)
C14—C15—C16	118.7 (8)	C38—C37—C39	111.6 (10)
C12—C17—C16	120.3 (8)	C39—C37—C40	109.5 (8)
C2—S1—O33	106.7 (3)	C20—C21—C22	120.6 (13)
C1—S2—O35	105.4 (4)	C22—C23—C24	118.0 (10)
C1—S2—C37	107.1 (4)	O18—C19—C20	115.5 (8)
O35—S2—C37	106.6 (4)	C20—C19—C24	120.9 (9)
S2—C1—C2	105.9 (5)	C1—C25—O27	106.1 (7)
C2—C1—C25	114.4 (7)	C25—O27—C28	120.6 (8)
S1—C2—C3	105.3 (5)	O27—C28—C30	111.1 (10)
C2—C3—C6	124.5 (7)	C29—C28—C30	109.9 (11)
C6—C3—C7	61.4 (5)	C30—C28—C31	111.9 (11)
C3—C6—C5	122.2 (7)	S2—C37—C38	110.5 (7)
C4—C6—C5	115.7 (8)	S2—C37—C40	109.5 (7)
C5—C6—C7	118.7 (7)	C38—C37—C40	112.6 (9)

The structure was solved using direct methods (*SHELXS86*; Sheldrick, 1986); 50 phase sets with $E \geq 1.20$. The best solution had a combined figure of merit of 0.057. Non-H atoms were refined anisotropically (*SHELX76*; Sheldrick, 1976). H atoms, found from $\Delta\rho$ synthesis and theoretically adjusted, were refined isotropically.

We thank D. Babin and J. P. Demoute from Roussel Uclaf Company (Romainville, France) for suggesting this work and providing the samples.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55761 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1024]

References

- Babin, D., Demassej, J., Demoute, J. P., Dutheil, Ph., Terrie, I. & Tessier, J. (1992). *J. Org. Chem.* **57**, 584–589.
- Baert, F., Guelzim, A. & Germain, G. (1991). *Acta Cryst.* **C47**, 768–771.
- Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- Sheldrick, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.
- Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *Chem. Phys.* **42**, 3175–3187.
- Tessier, J., Teche, A. & Demoute, J. P. (1982). *Proceedings of the 5th IUPAC International Congress of Pesticide Chemistry*, edited by J. Myamoto & P. C. Kearney. London: Pergamon Press.

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Structures of Tribenzylmethanol and 1,2,3-Triphenyl-2-propanol

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Abstract

The tribenzylmethanol molecule, $(\text{PhCH}_2)_3\text{COH}$, has approximate threefold symmetry in the solid state. The hydroxyl H atom is disordered unequally over three orientations and is not involved in hydrogen bonding. The 1,2,3-

triphenyl-2-propanol molecule, $\text{Ph}(\text{PhCH}_2)_2\text{COH}$, crystallizes with two molecules per asymmetric unit which differ slightly in conformation. In one of the molecules the hydroxyl H atom is disordered equally over two sites, whereas in the other molecule there is no disorder. As in the tribenzylmethanol molecule, there is no intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonding, presumably because of the steric bulk of the molecules and their packing which prevents the close approach of the O atoms of adjacent molecules.

Comment

Comparison of the molecular structures of compounds $[(\text{PhCH}_2)_3M]_2\text{O}$ [linear at O for both $M = \text{Ge}$ (Glidewell & Liles, 1979*a*) and $M = \text{Sn}$ (Glidewell & Liles, 1979*b*)] with those of $(\text{Ph}_3M)_2\text{O}$ [non-linear at O for $M = \text{Ge}$ (Glidewell & Liles, 1978*a*) and $M = \text{Sn}$ (Glidewell & Liles, 1978*b*)] suggests that in compounds of this type the steric requirements of $(\text{PhCH}_2)_3M$ fragments may be significantly greater than those of Ph_3M fragments. In order to assess the influence of these organic substituents on the molecular conformation and hydrogen-bonding patterns in the crystal structure, we have now determined the structure of tribenzylmethanol, $(\text{PhCH}_2)_3\text{COH}$ (I), and 1,2,3-triphenyl-2-propanol, $\text{Ph}(\text{PhCH}_2)_2\text{COH}$ (II), as part of a structural survey of the series $(\text{Ph})_x(\text{PhCH}_2)_{3-x}\text{COH}$, of which the structure of triphenylmethanol (Ferguson, Gallagher, Glidewell, Low & Scrimgeour, 1992) has already been determined.

Tribenzylmethanol was obtained from the reaction of $\text{PhCH}_2\text{COOEt}$ with two equivalents of PhCH_2MgBr : crystals were grown from a solution in light petroleum (b.p. 333–353 K). The $(\text{PhCH}_2)_3\text{COH}$ molecule (Fig. 1) has approximate threefold symmetry in the solid state with the threefold axis through the atoms O and C [torsion angles $\text{O}-\text{C}-\text{C}(x)-\text{C}(x1) -63.2$ (2), -58.2 (2) and -49.8 (2) $^\circ$ for $x = 1$ to 3]. The angles which the planes

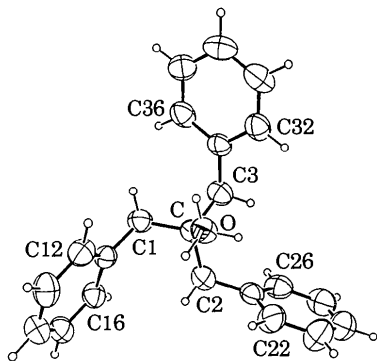


Fig. 1. An ORTEP view of the tribenzylmethanol molecule. The non-H atoms are shown with thermal ellipsoids drawn at the 50% probability level. For clarity, the H atoms are drawn as small spheres of an arbitrary size. The hydroxyl H atom is disordered over three orientations and is depicted as such.

of the three benzyl groups make with the plane through the benzyl methylene C atoms C(1), C(2) and C(3) are 57.9 (1), 67.7 (1) and 46.0 (1) $^\circ$ for groups A, B and C, respectively.

The hydroxyl H atom attached to O is disordered over three orientations H(O1), H(O2) and H(O3), with site-occupancy factors 0.55, 0.27 and 0.18, respectively, determined from peak heights in difference electron-density maps. There is no hydrogen bonding in the structure, presumably because the conformation adopted by the three benzyl rings shields the hydroxyl group and prevents the close approach of a hydroxyl O atom of an adjacent molecule, thus precluding any $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonding.

1,2,3-Triphenyl-2-propanol was obtained from the reaction of 1,3-diphenylacetone with lithium-phenyl, and crystals were grown by slow evaporation of a solution in methanol. The $\text{Ph}(\text{PhCH}_2)_2\text{COH}$ system crystallizes with two molecules per asymmetric unit; these differ only slightly in conformation (Fig. 2). The main source of the differences arises from the phenyl rings adopting slightly different orientations about $\text{CH}_2-\text{C}(\text{ar.})$ [e.g. torsion angle $\text{C}(1A)-\text{C}(2A)-\text{C}(21A)-\text{C}(22A) -96.4$ (4) $^\circ$ in one molecule compared with -104.8 (4) $^\circ$ in the other]. Difference electron density maps showed that in one of the molecules the hydroxyl H atom is disordered equally over two sites whereas in the other molecule there is no disorder. As in $(\text{PhCH}_2)_3\text{COH}$, there is no hydrogen bonding

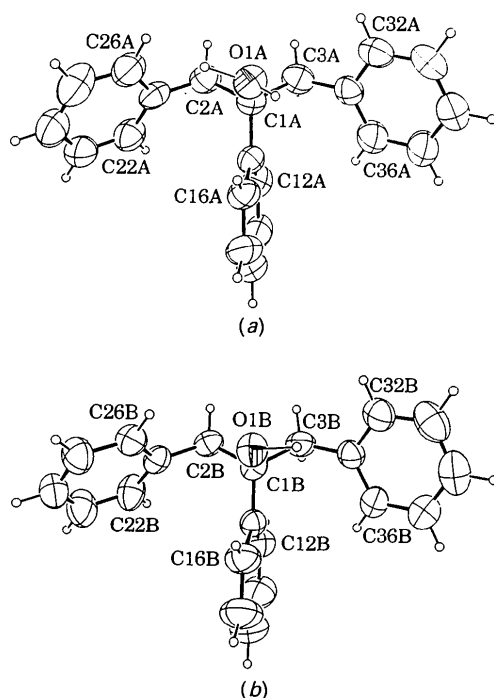


Fig. 2. Views of (a) molecule A of 1,2,3-triphenyl-2-propanol with the hydroxyl H atom depicted as disordered over two orientations, and (b) molecule B; in both cases the atoms are drawn as in Fig. 1.

in the crystal lattice because no adjacent O atom can approach close enough.

These two structures in which there is no hydrogen bonding contrast with the structure of triphenylmethanol, Ph₃COH (Ferguson, Gallagher, Glidewell, Low & Scrimgeour, 1992), which crystallizes as tetrameric aggregates with threefold crystallographic symmetry, each containing an approximately tetrahedral arrangement of O atoms with necessarily disordered hydroxyl H atoms. The structures of the isomorphous compounds Ph₃SiOH (Puff, Braun & Reuter, 1991) and Ph₃GeOH (Ferguson, Gallagher, Murphy, Spalding, Glidewell & Holden, 1992) also form aggregates with two similar independent hydrogen-bonded tetramers in the asymmetric unit. The arrangement of the O atoms is best described as a flattened tetrahedron in these aggregates.

The molecular dimensions for (PhCH₂)₃COH and Ph(PhCH₂)₂COH are summarized in Table 5 and show no unusual features.

Experimental

Tribenzylmethanol

Crystal data

C₂₂H₂₀O

M_r = 302.41

Orthorhombic

Pcab

a = 7.7305 (7) Å

b = 16.7230 (15) Å

c = 26.668 (3) Å

V = 3447.6 (5) Å³

Z = 8

D_x = 1.165 Mg m⁻³

Mo Kα radiation

λ = 0.70930 Å

Cell parameters from 25

reflections

θ = 8.00–18.00°

μ = 0.06 mm⁻¹

T = 293 K

Block

0.45 × 0.30 × 0.20 mm

Colourless

Crystal source: synthesized

by authors

Data collection

Nonius CAD-4 diffractometer

ω/2θ scans

Absorption correction: none

2692 measured reflections

2692 independent reflections

1312 observed reflections

[*I*_{net} > 3.0σ(*I*_{net})]

θ_{max} = 24.0°

h = 0 → 8

k = 0 → 19

l = 0 → 30

3 standard reflections

frequency: 120 min

intensity variation: none

Refinement

Refinement on *F*

Final *R* = 0.032

ω*R* = 0.036

S = 1.19

1312 reflections

209 parameters

ω = 1/[σ²(*F*) + 0.0004*F*²]

(Δ/σ)_{max} < 0.005

Δρ_{max} = 0.10 e Å⁻³

Δρ_{min} = -0.10 e Å⁻³

Extinction correction: Larson (1970)

Extinction coefficient:

15440 (2130)

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å²) for tribenzylmethanol

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
O	0.45671 (21)	0.42846 (9)	0.10182 (6)	0.0528 (10)
C	0.3799 (3)	0.48322 (13)	0.13636 (9)	0.0467 (14)
C(1)	0.3398 (3)	0.56214 (14)	0.10898 (9)	0.0529 (16)
C(11)	0.4934 (3)	0.60707 (13)	0.08878 (9)	0.0436 (14)
C(12)	0.5487 (4)	0.59686 (13)	0.03963 (9)	0.0532 (16)
C(13)	0.6926 (4)	0.63672 (15)	0.02125 (10)	0.0635 (18)
C(14)	0.7810 (4)	0.68903 (15)	0.05152 (11)	0.0645 (18)
C(15)	0.7247 (4)	0.70158 (14)	0.09965 (10)	0.0603 (17)
C(16)	0.5838 (4)	0.66141 (14)	0.11808 (8)	0.0517 (16)
C(2)	0.5032 (4)	0.49905 (15)	0.18046 (9)	0.0591 (16)
C(21)	0.5594 (4)	0.42766 (14)	0.21041 (9)	0.0508 (15)
C(22)	0.6944 (4)	0.37931 (17)	0.19420 (10)	0.0657 (18)
C(23)	0.7493 (4)	0.31434 (16)	0.22176 (11)	0.0785 (20)
C(24)	0.6730 (5)	0.29659 (17)	0.26635 (12)	0.0796 (22)
C(25)	0.5406 (5)	0.34307 (20)	0.28369 (10)	0.0746 (21)
C(26)	0.4834 (4)	0.40789 (17)	0.25594 (10)	0.0644 (17)
C(3)	0.2116 (4)	0.44455 (15)	0.15538 (9)	0.0564 (16)
C(31)	0.0896 (3)	0.41207 (14)	0.11626 (9)	0.0465 (15)
C(32)	0.0834 (4)	0.33080 (15)	0.10674 (10)	0.0559 (16)
C(33)	-0.0287 (4)	0.29937 (15)	0.07172 (12)	0.0678 (19)
C(34)	-0.1352 (4)	0.34830 (20)	0.04498 (11)	0.0712 (20)
C(35)	-0.1315 (4)	0.42882 (18)	0.05344 (11)	0.0696 (20)
C(36)	-0.0222 (4)	0.46042 (14)	0.08911 (11)	0.0584 (18)

Table 2. Torsion angles (°) for tribenzylmethanol

O—C—C(1)—C(11)	-63.2 (2)	C—C(2)—C(21)—C(22)	82.2 (2)
O—C—C(2)—C(21)	-58.1 (2)	C—C(2)—C(21)—C(26)	-99.6 (3)
O—C—C(3)—C(31)	-49.8 (2)	C—C(3)—C(31)—C(32)	101.5 (3)
C—C(1)—C(11)—C(12)	93.9 (2)	C—C(3)—C(31)—C(36)	-79.7 (2)
C—C(1)—C(11)—C(16)	-87.3 (2)		

1,2,3-Triphenyl-2-propanol

Crystal data

C₂₁H₂₀O

M_r = 288.39

Monoclinic

*P*2₁/*n*

a = 23.0194 (12) Å

b = 6.0483 (5) Å

c = 23.7136 (12) Å

β = 98.012 (4)°

V = 3269.4 (4) Å³

Z = 8

D_x = 1.172 Mg m⁻³

Mo Kα radiation

λ = 0.70930 Å

Cell parameters from 25

reflections

θ = 9.50–15.50°

μ = 0.065 mm⁻¹

T = 293 K

Block

0.30 × 0.20 × 0.15 mm

Colourless

Crystal source: synthesized

by authors

Data collection

Nonius CAD-4 diffractometer

ω/2θ scans

Absorption correction:

none

5224 measured reflections

5102 independent reflections

2359 observed reflections

[*I*_{net} > 2.0σ(*I*_{net})]

*R*_{int} = 0.017

θ_{max} = 23.92°

h = -26 → 26

k = 0 → 6

l = 0 → 27

3 standard reflections

frequency: 60 min

intensity variation: none

Refinement

Refinement on *F*

Final *R* = 0.050

ω*R* = 0.052

S = 1.28

Extinction correction: Larson (1970)

Extinction coefficient:

18016 (2234)

2359 reflections
398 parameters
 $w = 1/[\sigma^2(F) + 0.0006F^2]$
 $(\Delta/\sigma)_{\max} = 0.000$
 $\Delta\rho_{\max} = 0.15 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.22 \text{ e } \text{Å}^{-3}$

For both compounds: Data collection: Enraf-Nonius CAD-4 software. Cell refinement: Enraf-Nonius CAD-4 software. Data reduction: *NRCVAX DATRD2* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX SOLVER*. Program(s) used to refine structure: *NRCVAX LSTSQ*. Molecular graphics: *NRCVAX*; *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *NRCVAX TABLES*.

Table 3. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å^2) for 1,2,3-triphenyl-2-propanol

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
O(1A)	0.41896 (11)	0.1214 (5)	0.83176 (10)	0.0669 (18)
C(1A)	0.41703 (16)	-0.1050 (7)	0.84879 (15)	0.056 (3)
C(11A)	0.35445 (16)	-0.1920 (7)	0.84002 (15)	0.0530 (24)
C(12A)	0.34145 (20)	-0.4006 (8)	0.86013 (17)	0.072 (3)
C(13A)	0.2841 (3)	-0.4794 (9)	0.85310 (20)	0.087 (4)
C(14A)	0.23950 (22)	-0.3515 (13)	0.82567 (23)	0.099 (5)
C(15A)	0.25256 (20)	-0.1518 (11)	0.80536 (19)	0.094 (4)
C(16A)	0.30874 (18)	-0.0717 (7)	0.81244 (15)	0.063 (3)
C(2A)	0.45554 (16)	-0.2346 (7)	0.81221 (18)	0.071 (3)
C(21A)	0.43577 (17)	-0.2267 (8)	0.74971 (19)	0.061 (3)
C(22A)	0.40225 (20)	-0.3899 (8)	0.72158 (21)	0.076 (3)
C(23A)	0.38316 (20)	-0.3774 (10)	0.66411 (24)	0.093 (4)
C(24A)	0.39734 (22)	-0.1986 (12)	0.63357 (20)	0.092 (4)
C(25A)	0.4315 (3)	-0.0356 (9)	0.6603 (3)	0.097 (4)
C(26A)	0.45032 (19)	-0.0516 (9)	0.71777 (23)	0.082 (3)
C(3A)	0.44449 (16)	-0.1158 (7)	0.91190 (16)	0.070 (3)
C(31A)	0.41674 (17)	0.0305 (8)	0.95202 (16)	0.059 (3)
C(32A)	0.44252 (18)	0.2286 (9)	0.96977 (19)	0.074 (3)
C(33A)	0.41765 (22)	0.3647 (8)	1.00630 (19)	0.078 (3)
C(34A)	0.36739 (23)	0.3075 (9)	1.02596 (18)	0.083 (3)
C(35A)	0.34122 (21)	0.1107 (10)	1.00869 (20)	0.093 (4)
C(36A)	0.36570 (20)	-0.0262 (8)	0.97233 (19)	0.082 (3)
O(1B)	0.71587 (10)	0.1772 (4)	0.65382 (10)	0.0614 (16)
C(1B)	0.69571 (15)	-0.0467 (7)	0.64815 (14)	0.0506 (23)
C(11B)	0.69556 (15)	-0.1275 (7)	0.58781 (15)	0.0509 (24)
C(12B)	0.67123 (17)	-0.3298 (7)	0.57006 (18)	0.068 (3)
C(13B)	0.67102 (22)	-0.4056 (9)	0.51495 (24)	0.095 (4)
C(14B)	0.6964 (3)	-0.2786 (12)	0.47710 (22)	0.109 (5)
C(15B)	0.72072 (23)	-0.0809 (11)	0.49350 (21)	0.103 (4)
C(16B)	0.72063 (17)	-0.0066 (8)	0.54851 (18)	0.074 (3)
C(2B)	0.73827 (18)	-0.1841 (7)	0.68967 (14)	0.0571 (24)
C(21B)	0.80105 (16)	-0.1858 (7)	0.67885 (14)	0.0524 (25)
C(22B)	0.82486 (19)	-0.3651 (8)	0.65546 (18)	0.076 (3)
C(23B)	0.88268 (22)	-0.3710 (9)	0.64603 (20)	0.091 (3)
C(24B)	0.91781 (18)	-0.1910 (10)	0.66064 (18)	0.079 (4)
C(25B)	0.89560 (19)	-0.0119 (9)	0.68447 (18)	0.074 (3)
C(26B)	0.83791 (18)	-0.0068 (7)	0.69319 (15)	0.062 (3)
C(3B)	0.63398 (16)	-0.0583 (7)	0.66652 (14)	0.061 (3)
C(31B)	0.59055 (15)	0.1054 (8)	0.63716 (17)	0.055 (3)
C(32B)	0.57431 (18)	0.2876 (9)	0.66638 (18)	0.072 (3)
C(33B)	0.53397 (22)	0.4408 (8)	0.6412 (3)	0.085 (4)
C(34B)	0.51021 (19)	0.4132 (9)	0.5856 (3)	0.089 (4)
C(35B)	0.52601 (21)	0.2353 (10)	0.55582 (18)	0.089 (4)
C(36B)	0.56527 (18)	0.0829 (8)	0.58112 (17)	0.073 (3)

Table 4. Torsion angles ($^\circ$) for 1,2,3-triphenyl-2-propanol

O(1A)—C(1A)—C(3A)—C(31A)	56.3 (3)
C(11A)—C(1A)—C(3A)—C(31A)	-65.4 (3)
C(2A)—C(1A)—C(3A)—C(31A)	171.4 (4)
C(1A)—C(2A)—C(21A)—C(22A)	-96.4 (4)

Atomic scattering factors
from *International Tables*
for *X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

C(1A)—C(2A)—C(21A)—C(26A)	83.0 (4)
C(1A)—C(3A)—C(31A)—C(32A)	-100.5 (4)
C(1A)—C(3A)—C(31A)—C(36A)	79.7 (4)
O(1B)—C(1B)—C(3B)—C(31B)	52.6 (2)
C(11B)—C(1B)—C(3B)—C(31B)	-69.6 (3)
C(2B)—C(1B)—C(3B)—C(31B)	168.0 (4)
C(1B)—C(2B)—C(21B)—C(22B)	-104.8 (4)
C(1B)—C(2B)—C(21B)—C(26B)	76.4 (3)
C(1B)—C(3B)—C(31B)—C(32B)	-106.3 (4)
C(1B)—C(3B)—C(31B)—C(36B)	73.8 (3)

Table 5. Summary of dimensions (Å , $^\circ$) for tribenzylmethanol (I) and 1,2,3-triphenyl-2-propanol molecules A (IIA) and B (IIB)

	(I)	(IIA)	(IIB)
Csp ³ —O	1.428(3)	1.430(5)	1.432(5)
Csp ³ —Csp ³	1.539(4)	1.541(6)	1.539(6)
Csp ³ —Csp ²	1.504(4)	1.505(6)	1.507(5)
Aromatic C—C	1.378(4)	1.371(8)	1.374(7)
O—C—CH ₂	108.8(2)	106.8(3)	107.4(3)
CH ₂ —C—CH ₂	110.1(2)	109.6(3)	109.0(3)
Csp ² —C—CH ₂	-	111.3(3)	111.2(3)

The space group for (PhCH₂)₃COH (I) was determined unambiguously from the systematic absences as *Pcab* (a non-standard setting of *Pbca*, No. 61). For Ph(PhCH₂)₂COH (II), the systematic absences unambiguously determined the space group as *P2₁/n*. In both (I) and (II) the H atoms attached to the C atoms were clearly visible in difference maps; they were positioned geometrically (C—H 0.95 Å) and included as riding atoms in the structure-factor calculations. For (PhCH₂)₃COH (I), difference maps showed that the hydroxyl H atom was disordered unequally over three orientations in a fully staggered conformation with respect to the C—(CH₂)₃ moiety. In Ph(PhCH₂)₂COH (II), the hydroxyl H atom in molecule A was disordered equally over two orientations, whereas no disorder was observed in molecule B. The hydroxyl H atoms were included in the structure-factor calculations at the coordinates derived from the difference maps.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55802 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: L11032]

References

- Ferguson, G., Gallagher, J. F., Glidewell, C., Low, J. N. & Scrimgeour, S. N. (1992). *Acta Cryst.* **C48**, 1272–1275.
- Ferguson, G., Gallagher, J. F., Murphy, D., Spalding, T. R., Glidewell, C. & Holden, K. D. (1992). *Acta Cryst.* **C48**, 1228–1231.
- Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
- Glidewell, C. & Liles, D. C. (1978a). *Acta Cryst.* **B34**, 119–124.
- Glidewell, C. & Liles, D. C. (1978b). *Acta Cryst.* **B34**, 1693–1695.
- Glidewell, C. & Liles, D. C. (1979a). *J. Organomet. Chem.* **174**, 275–279.
- Glidewell, C. & Liles, D. C. (1979b). *Acta Cryst.* **B35**, 1689–1691.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

- Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.
- Puff, H., Braun, K. & Reuter, H. (1991). *J. Organomet. Chem.* **409**, 119–129.

Acta Cryst. (1993). **C49**, 824–825

Structure of (+)-(*S*)-5-Phenyl-5-ethylhydantoin

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Abstract

The structure of (+)-(*S*)-5-phenyl-5-ethyl-2,4-imidazolidinedione or (+)-(*S*)-5-phenyl-5-ethylhydantoin (PHETHYD) is determined and compared with the structure of other related anticonvulsants. Among all the hydantoin derivatives previously described, the conformation of PHETHYD exhibits the smallest value (60°) of the dihedral angle between the phenyl ring and the hydantoin ring. The hydrogen-bond network suggests a particular stability of the homochiral packing. The two N—H and C=O bonds participate in the hydrogen-bond network which forms a flat ribbon parallel to the *a* axis. The non-polar parts of the molecules are facing each other and are held together by van der Waals forces only.

Comment

The antiepileptic and/or sedative properties of 5,5-substituted hydantoin derivatives depend upon the nature of the substituents and the *R* or *S* configuration. This structure has been studied in order to compare its conformation with the conformation of other anticonvulsants such as methoin (Lisgarten & Palmer, 1980) and phenytoin (Camerman & Camerman, 1971).

The second aim of this study is to make a contribution to the knowledge of the way in which homochiral molecules crystallize as a conglomerate. These molecules include (+)-(*S*)-5-phenyl-5-ethylhydantoin (PHETHYD) and other 5-alkyl-5-arylhydantoin derivatives. Although a large majority of racemic

mixtures (> 90%) crystallize as racemic compounds (Brock, Schweizer & Dunitz, 1991), the driving forces which lead either to this or to spontaneous resolution are not yet clear.

The racemic mixture, prepared in a good yield (> 90%) by Bucherer's reaction, was resolved *via* a new preparative route (Coquerel, Petit, Bouaziz & Depernet, 1992). Single crystals of *S* enantiomer were obtained by slow evaporation in ethanol at room temperature.

As expected, the hydantoin ring is almost planar, except for the slight deviation of the C(4) atom which is at 0.064 Å from the mean plane passing through the other four atoms. The dihedral angle between the phenyl and the heterocycle is 60° . This value is smaller than the angle observed in methoin (77°). It is in fact the smallest value of all alkyl/aryl or aryl/aryl disubstituted hydantoin and related barbiturates studied so far. Both N atoms are involved in hydrogen bonds: N(1)—H(1)⋯O(4)(1 + *x*, *y*, *z*) 2.28 Å, 170.3° ; N(3)—H(3)⋯O(2)(0.5 + *x*, 1.5 − *y*, −*z*) 1.94 Å, 151.1° . The hydrogen-bonded molecules form an infinite ribbon, parallel to the *a* axis. These ribbons, possessing hydrophilic character, are held together only by means of van der Waals forces between the ethyl and phenyl groups which are in the vicinity of each other.

In a recent survey (Collet, 1990), it is shown that difficulties arise in dealing with the whole problem of homochiral *versus* heterochiral packing of enantiomers. Nevertheless, some special functional groups bring about a large increase in the proportion of conglomerates (usually between 5 and 10%). These

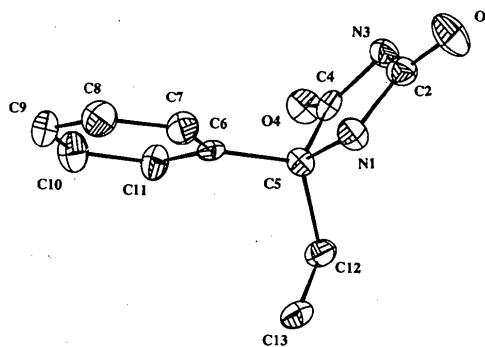


Fig. 1. ORTEP (Johnson, 1965) drawing with atom numbering.

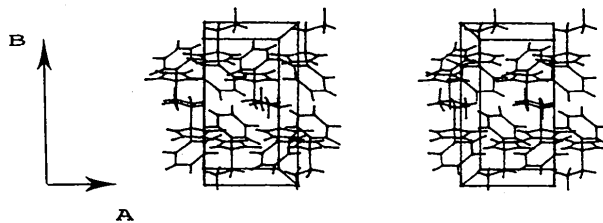


Fig. 2. Stereoscopic view down *c* of the crystal structure.