

C(10)—C(9)	1.395 (3)	C(10*)—C(9*)	1.395 (3)
C(10)—C(5)	1.399 (3)	C(10*)—C(5*)	1.407 (3)
C(5)—C(6)	1.366 (3)	C(5*)—C(6*)	1.367 (4)
C(6)—C(7)	1.407 (3)	C(6*)—C(7*)	1.405 (4)
C(7)—O(7)	1.348 (3)	C(7*)—O(7*)	1.348 (3)
C(7)—C(8)	1.384 (3)	C(7*)—C(8*)	1.385 (3)
C(9)—C(8)	1.387 (3)	C(9*)—C(8*)	1.387 (3)
C(2)—C(1')	1.470 (3)	C(2*)—C(1'*)	1.475 (3)
C(1')—C(2')	1.410 (3)	C(1'*)—C(2'*)	1.402 (4)
C(2')—C(3')	1.385 (3)	C(2'*)—C(3'*)	1.389 (3)
C(3')—C(4')	1.399 (3)	C(3'*)—C(4'*)	1.401 (3)
C(4')—C(5')	1.384 (3)	C(4'*)—C(5'*)	1.392 (4)
C(5')—C(6')	1.389 (3)	C(5'*)—C(6'*)	1.394 (3)
C(1')—C(6')	1.394 (3)	C(1'*)—C(6'*)	1.393 (4)
C(2')—O(2')	1.371 (3)	C(2'*)—O(2'*)	1.368 (3)
C(3')—O(3')	1.375 (3)	C(3'*)—O(3'*)	1.377 (3)
C(4')—O(4')	1.359 (3)	C(4'*)—O(4'*)	1.353 (3)
O(2')—C(2'1)	1.433 (4)	O(2'*)—C(2'1*)	1.443 (4)
O(3')—C(3'1)	1.444 (4)	O(3'*)—C(3'1*)	1.410 (4)
O(4')—C(4'1)	1.421 (4)	O(4'*)—C(4'1*)	1.428 (4)
C(2)—O(1)—C(9)	120.1 (2)	C(2*)—O(1*)—C(9*)	118.8 (2)
C(6)—C(7)—C(8)	120.8 (2)	C(6*)—C(7*)—C(8*)	120.1 (2)
O(1)—C(2)—C(3)	120.6 (2)	O(1*)—C(2*)—C(3*)	122.6 (2)
O(7)—C(7)—C(8)	122.1 (2)	O(7*)—C(7*)—C(8*)	122.6 (2)
O(1)—C(2)—C(1')	111.4 (2)	O(1*)—C(2*)—C(1'*)	110.6 (2)
C(9)—C(8)—C(7)	118.1 (2)	C(9*)—C(8*)—C(7*)	118.3 (2)
C(3)—C(2)—C(1')	128.0 (2)	C(3*)—C(2*)—C(1'*)	126.7 (2)
C(2)—C(3)—C(4)	122.6 (2)	C(2*)—C(3*)—C(4*)	121.9 (2)
C(2)—C(1')—C(2')	123.1 (2)	C(2*)—C(1'*)—C(2'*)	121.3 (2)
C(3')—C(4')—O(4')	115.2 (2)	C(3'*)—C(4'*)—O(4'*)	114.5 (2)
C(2)—C(1')—C(6')	119.7 (2)	C(2*)—C(1'*)—C(6'*)	120.4 (2)
C(3')—C(4')—C(5')	119.5 (2)	C(3'*)—C(4'*)—C(5'*)	119.7 (2)
C(3)—C(4)—O(4)	122.2 (2)	C(3*)—C(4*)—O(4*)	122.5 (2)
C(2')—C(1')—C(6')	117.1 (2)	C(2'*)—C(1'*)—C(6'*)	118.3 (2)
O(4')—C(4')—C(5')	125.3 (2)	O(4'*)—C(4'*)—C(5'*)	125.9 (2)
C(3)—C(4)—C(10)	115.4 (2)	C(3*)—C(4*)—C(10*)	114.8 (2)
C(1')—C(2')—O(2')	121.0 (2)	C(1'*)—C(2'*)—O(2'*)	121.0 (2)
C(4')—O(4')—C(4'1)	118.9 (2)	C(4'*)—O(4'*)—C(4'1*)	118.4 (2)
O(4)—C(4)—C(10)	122.4 (2)	O(4*)—C(4*)—C(10*)	122.8 (2)
C(1')—C(2')—C(3')	120.7 (2)	C(1'*)—C(2'*)—C(3'*)	120.3 (2)
C(4)—C(10)—C(9)	119.1 (2)	C(4*)—C(10*)—C(9*)	120.1 (2)
O(2')—C(2')—C(3')	118.1 (2)	O(2'*)—C(2'*)—C(3'*)	118.8 (2)
C(4)—C(10)—C(5)	123.1 (2)	C(4*)—C(10*)—C(5*)	122.7 (2)
C(2')—O(2')—C(2'1)	114.5 (2)	C(2'*)—O(2'*)—C(2'1*)	113.8 (2)
C(9)—C(10)—C(5)	117.7 (2)	C(9*)—C(10*)—C(5*)	117.2 (2)
O(1)—C(9)—C(10)	121.9 (2)	O(1*)—C(9*)—C(10*)	121.8 (2)
O(1)—C(9)—C(8)	115.7 (2)	O(1*)—C(9*)—C(8*)	115.3 (2)
C(10)—C(9)—C(8)	122.4 (2)	C(10*)—C(9*)—C(8*)	123.0 (2)
C(4')—C(5')—C(6')	119.3 (2)	C(4'*)—C(5'*)—C(6'*)	119.0 (2)
C(10)—C(5)—C(6)	121.2 (2)	C(10*)—C(5*)—C(6*)	120.9 (2)
C(2')—C(3')—O(3')	118.5 (2)	C(2'*)—C(3'*)—O(3'*)	119.9 (2)
C(1')—C(6')—C(5')	122.6 (2)	C(1'*)—C(6'*)—C(5'*)	122.1 (2)
C(5)—C(6)—C(7)	119.6 (2)	C(5*)—C(6*)—C(7*)	120.5 (2)
C(2')—C(3')—C(4')	120.6 (2)	C(2'*)—C(3'*)—C(4'*)	120.7 (2)
O(3')—C(3')—C(4')	120.8 (2)	O(3'*)—C(3'*)—C(4'*)	119.4 (2)
C(3')—O(3')—C(3'1)	114.3 (2)	C(3'*)—O(3'*)—C(3'1*)	114.4 (2)
C(6)—C(7)—O(7)	117.1 (2)	C(6*)—C(7*)—O(7*)	117.2 (2)

Phenolic H atoms were not located but their presence was inferred from the O(4)··O(7) and O(4*)··O(7*) distances of 2.628 (3) and 2.632 (3) Å, respectively. The structure was solved by direct methods using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1095). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Crystal Studies of Avarol Derivatives. 5'-Acetylavarol from *Dysidea avara*

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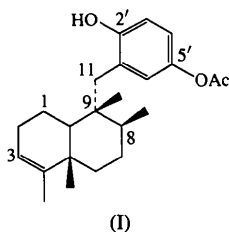
Abstract

The X-ray analysis of the natural product 5'-acetylavarol {4-hydroxy-3-[(1,2,3,4,4a,7,8,8a-octahydro-1,2,4a,5-tetramethyl-1-naphthyl)methyl]phenyl acetate, C₂₃H₃₂O₃} confirms the previous structure elucidation of Crispino, De Giulio, De Rosa & Stazzullo [*J. Nat. Prod.* (1989), **52**, 646–648], which was made on the basis of chemical and spectral data. Geometrical distortions, with respect to normal tetrahedral values, are evident in the sesquiterpene moiety. The distortions are induced by short contacts between the bulky substituents of the bicyclic system. The hydroquinone ring is nearly perpendicular to the sesquiterpene residue. The crystal

packing is characterized by chains of screw-related molecules connected, along the *b*-axis direction, through repetition of a hydrogen bond that connects hydroxyl groups and acetyl O atoms. The shortest methyl–methyl intermolecular contact is 3.733 (6) Å.

Comment

We are currently undertaking comparative studies of the structural and biological features of terpenoid metabolites (Puliti, Gavagnin, Cimino, Mattia & Mazzarella, 1992; Cimino, Crispino, Epifanio, Madaio, Mattia, Mazzarella, Puliti, Trivellone & Uriz, 1992; Puliti, Fontana, Cimino, Mattia & Mazzarella, 1993; De Rosa, Puliti, Crispino, De Giulio, Mattia & Mazzarella, 1994). In particular, we are interested in crystal structure determinations of avarol, a sesquiterpenoid monosubstituted hydroquinone molecule (Puliti, De Rosa & Mattia, 1994), and its derivatives (Giordano & Puliti, 1987), in connection with the biological activities and possible pharmacological implications of this class of compounds (De Giulio, De Rosa, Strazzullo, Diliberto, Obino, Marongiu, Pani & La Colla, 1991; Ferrándiz, Sanz, Bustos, Payá, Alcaraz & De Rosa, 1994). As part of this research, we present the crystal structure of a further natural compound related to avarol, namely, 5'-acetylavarol, (I).



5'-Acetylavarol has recently been isolated (Crispino, De Giulio, De Rosa & Strazzullo, 1989) as a minor secondary metabolite from samples of the common sponge *Dysidea avara* collected in the bay of Naples (Capo Miseno) and its characterization has been carried out by means of NMR and IR spectroscopy. Compound (I) shows cytotoxic activity. A view of the final X-ray model is shown in Fig. 1, together with the atomic numbering scheme and displacement ellipsoids. The results of the present study are in complete agreement with the structural elucidation of Crispino *et al.* (1989). The molecular geometry (Table 2) compares well with those of avarol (Puliti *et al.*, 1994) and its dimethyl derivative (Giordano & Puliti, 1987). In the sesquiterpene residue, significant deviations from normal bond lengths and angles are present. In particular, in the atomic sequence C4, C5, C10, C9, C8, the geometrical distortions are analogous to those discussed for the avarol structure and they are probably induced by short contacts between the bulky substituents of the bicyclic system. The

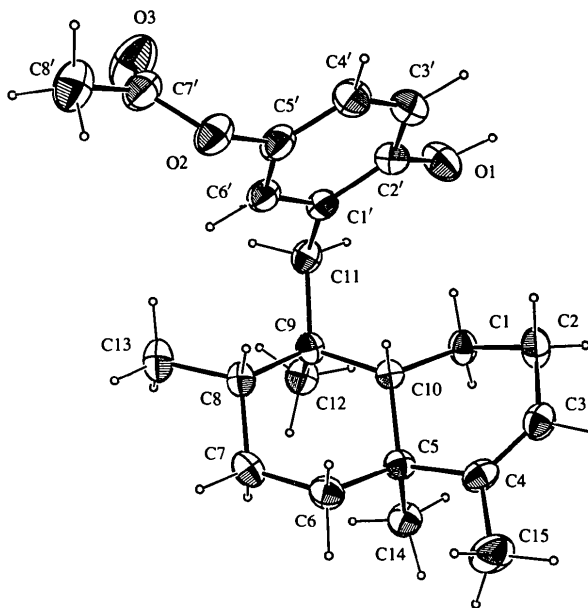


Fig. 1. Perspective view of the 5'-acetylavarol molecule with the atomic numbering scheme for non-H atoms. Displacement ellipsoids are plotted at the 30% probability level.

large values associated with the bond distances at the C11 methylene group, and especially its valency angle [119.4 (3)°], should also be noted. They allow loosening of the intramolecular contacts between the C₁₅ sesquiterpene and hydroquinone moieties.

The Δ^{3,4} cyclohexene ring adopts a conformation intermediate between the half-chair and half-boat forms. Atom C1 is displaced 0.258 (3) Å on one side and atom C10 0.579 (3) Å on the opposite side of the best plane defined by the remaining ring atoms. Deviation from ideal C₂(HC) and C₅(HB) symmetries can be measured by the asymmetry parameters ΔC₂[C3—C4] = 12.6 and ΔC₅[C3] = 14.9° (Duax, Weeks & Rohrer, 1976). The cyclohexane ring has a nearly ideal chair conformation with *Q* = 0.573 (3) Å and *θ* = 4.4 (3)°, according to the Cremer & Pople (1975) puckering parameters. With respect to the ideal chair form, atom C10 is slightly more 'flattened' than C7, the respective distances from the best plane through the remaining ring atoms being 0.616 (3) and 0.687 (4) Å. In this ring, the mean value of the intra-ring torsion angles is 55.5 (5)°.

The hydroquinone ring is almost perpendicular to the bicyclic system and it is oriented so as to optimize the contacts with the sesquiterpene residue [C1...O1 3.186 (4) and C8...C6' 3.283 (4) Å]. The dihedral angle between the weighted best planes through the *trans*-fused system and the hydroquinone ring is 83.6 (1)°. The aromatic ring is planar to within 0.024 (4) Å and the hydroxyl O atom is 0.143 (3) Å out of the average plane. Atom O2 lies in the ring plane and the C7'—O3 and O2—C5' bonds are eclipsed with respect to each other.

The crystal packing is shown in Fig. 2. Molecules related by 2_1 symmetry along b are connected in chains by means of a hydrogen bond between the hydroxyl group and the acetyl O atom [O1...O3ⁱ 2.757 (4), H(O1)...O3ⁱ 1.81 Å, O1—H...O3ⁱ 164°; symmetry code: (i) $-x, -\frac{1}{2} + y, \frac{1}{2} - z$]. The shortest intermolecular contact between sesquiterpene residues is C1...C6($x - 1, y, z$) of 3.565 (5) Å.

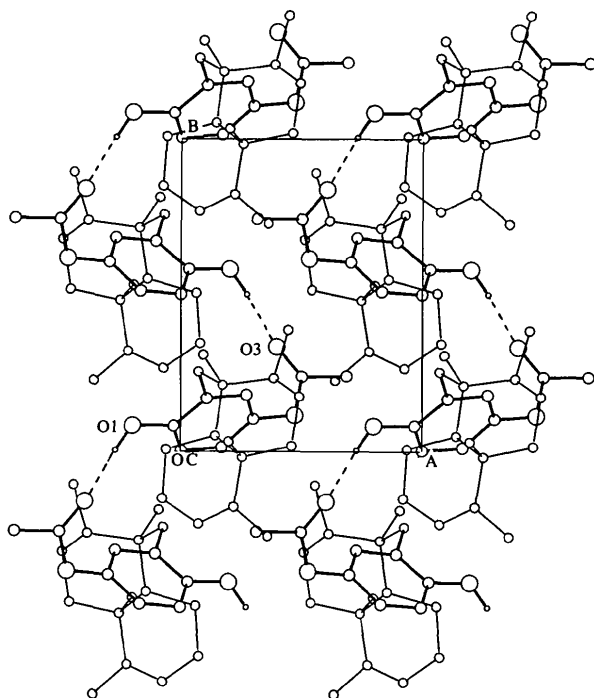


Fig. 2. Crystal packing projected in the ab plane. The hydroquinone and sesquiterpene residues are shown by bold and light lines, respectively. Dashed lines indicate hydrogen bonds.

Experimental

Crystals of the title compound were obtained by slow evaporation from an n -heptane-ethyl ether solution.

Crystal data

$C_{23}H_{32}O_3$
 $M_r = 356.51$
 Orthorhombic
 $P2_12_12_1$
 $a = 7.256 (1) \text{ \AA}$
 $b = 9.3991 (9) \text{ \AA}$
 $c = 30.265 (3) \text{ \AA}$
 $V = 2064.0 (7) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.147 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation
 $\lambda = 1.5418 \text{ \AA}$
 Cell parameters from 24 reflections
 $\theta = 20\text{--}27^\circ$
 $\mu = 0.549 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 Prism
 $0.38 \times 0.10 \times 0.07 \text{ mm}$
 Colourless

Data collection

Enraf-Nonius CAD-4F diffractometer
 ω scans
 Absorption correction: none
 2273 measured reflections
 2273 independent reflections
 1704 observed reflections
 $[I \geq 2.5\sigma(I)]$

$\theta_{\max} = 70^\circ$
 $h = 0 \rightarrow 8$
 $k = 0 \rightarrow 11$
 $l = 0 \rightarrow 36$
 3 standard reflections
 frequency: 240 min
 intensity decay: 4%

Refinement

Refinement on F
 $R = 0.0441$
 $wR = 0.0438$
 $S = 0.88$
 1704 reflections
 236 parameters
 H-atom parameters not refined
 $w = 1/[\sigma^2(F_o) + (0.01F_o)^2 + 0.2]$
 $(\Delta/\sigma)_{\max} = 0.01$

$\Delta\rho_{\max} = 0.15 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.13 \text{ e \AA}^{-3}$
 Extinction correction: Stout & Jensen (1968)
 Extinction coefficient: $1.11 (7) \times 10^{-6}$
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$B_{\text{eq}} = (1/3)\sum_i \sum_j B_{ij} a_i^* a_j^* a_i \cdot a_j.$$

	x	y	z	B_{eq}
O1	-0.2007 (4)	0.0832 (3)	0.31270 (8)	6.60 (7)
O2	0.4705 (4)	0.1169 (3)	0.22719 (7)	5.87 (6)
O3	0.3956 (5)	0.3375 (3)	0.2045 (1)	9.58 (9)
C1	-0.0624 (5)	0.0077 (4)	0.4098 (1)	4.40 (7)
C2	-0.0771 (6)	-0.1520 (4)	0.4006 (1)	5.62 (9)
C3	0.0734 (6)	-0.2323 (4)	0.4226 (1)	5.06 (8)
C4	0.2201 (5)	-0.1777 (4)	0.4415 (1)	4.57 (8)
C5	0.2531 (5)	-0.0160 (3)	0.44346 (9)	3.80 (7)
C6	0.4587 (5)	0.0181 (4)	0.4360 (1)	4.67 (8)
C7	0.4896 (5)	0.1771 (4)	0.4303 (1)	5.03 (8)
C8	0.3850 (5)	0.2359 (3)	0.3909 (1)	4.21 (7)
C9	0.1723 (4)	0.2134 (3)	0.3958 (1)	3.83 (7)
C10	0.1412 (4)	0.0515 (3)	0.40504 (9)	3.43 (6)
C11	0.0722 (5)	0.2564 (3)	0.3521 (1)	4.39 (7)
C12	0.0902 (5)	0.3110 (4)	0.4317 (1)	4.99 (8)
C13	0.4435 (6)	0.3910 (4)	0.3829 (1)	6.3 (1)
C14	0.1982 (6)	0.0325 (4)	0.4907 (1)	5.09 (9)
C15	0.3602 (7)	-0.2719 (4)	0.4635 (2)	7.4 (1)
C1'	0.1054 (5)	0.1705 (4)	0.3107 (1)	4.27 (7)
C2'	-0.0310 (6)	0.0833 (4)	0.2926 (1)	5.19 (8)
C3'	0.0007 (7)	0.0038 (5)	0.2549 (1)	6.12 (9)
C4'	0.1680 (6)	0.0133 (4)	0.2328 (1)	6.0 (1)
C5'	0.2981 (6)	0.1057 (4)	0.2494 (1)	5.33 (9)
C6'	0.2706 (5)	0.1824 (4)	0.2873 (1)	4.62 (8)
C7'	0.5010 (6)	0.2413 (4)	0.2054 (1)	6.0 (1)
C8'	0.6866 (7)	0.2424 (5)	0.1840 (1)	7.5 (1)

Table 2. Selected geometric parameters (\AA , $^\circ$)

O1—C2'	1.374 (5)	C7—C8	1.517 (5)
O2—C5'	1.424 (5)	C8—C9	1.565 (5)
O2—C7'	1.362 (5)	C8—C13	1.537 (5)
O3—C7'	1.185 (5)	C9—C10	1.563 (4)
C1—C2	1.531 (5)	C9—C11	1.561 (5)
C1—C10	1.541 (5)	C9—C12	1.541 (5)
C2—C3	1.485 (5)	C11—C1'	1.512 (5)
C3—C4	1.314 (5)	C1'—C2'	1.396 (5)

C4—C5	1.540 (5)	C1'—C6'	1.396 (5)
C4—C15	1.503 (6)	C2'—C3'	1.385 (5)
C5—C6	1.543 (5)	C3'—C4'	1.388 (7)
C5—C10	1.554 (4)	C4'—C5'	1.377 (6)
C5—C14	1.553 (4)	C5'—C6'	1.368 (5)
C6—C7	1.521 (5)	C7'—C8'	1.494 (6)
C5'—O2—C7'	115.8 (3)	C10—C9—C12	113.4 (3)
C2—C1—C10	108.1 (3)	C11—C9—C12	105.2 (3)
C1—C2—C3	111.5 (3)	C1—C10—C5	108.8 (3)
C2—C3—C4	126.3 (3)	C1—C10—C9	114.5 (3)
C3—C4—C5	121.9 (3)	C5—C10—C9	117.1 (3)
C3—C4—C15	120.7 (3)	C9—C11—C1'	119.4 (3)
C5—C4—C15	117.4 (3)	C11—C1'—C2'	121.7 (3)
C4—C5—C6	110.5 (3)	C11—C1'—C6'	121.0 (3)
C4—C5—C10	107.0 (2)	C2'—C1'—C6'	117.2 (3)
C4—C5—C14	106.6 (3)	O1—C2'—C1'	117.6 (3)
C6—C5—C10	108.1 (2)	O1—C2'—C3'	120.9 (4)
C6—C5—C14	108.8 (3)	C1'—C2'—C3'	121.5 (4)
C10—C5—C14	115.8 (3)	C2'—C3'—C4'	120.5 (4)
C5—C6—C7	111.3 (3)	C3'—C4'—C5'	117.7 (4)
C6—C7—C8	112.0 (3)	O2—C5'—C4'	118.4 (3)
C7—C8—C9	111.7 (3)	O2—C5'—C6'	119.0 (3)
C7—C8—C13	109.4 (3)	C4'—C5'—C6'	122.5 (4)
C9—C8—C13	114.6 (3)	C1'—C6'—C5'	120.5 (3)
C8—C9—C10	106.9 (2)	O2—C7'—O3	124.2 (4)
C8—C9—C11	110.1 (3)	O2—C7'—C8'	111.3 (4)
C8—C9—C12	111.5 (3)	O3—C7'—C8'	124.5 (4)
C10—C9—C11	109.6 (2)		
C5'—O2—C7'—O3	0.9 (5)	C8—C9—C11—C1'	66.1 (4)
C2—C3—C4—C5	0.6 (6)	C9—C11—C1'—C2'	109.8 (4)
C4—C5—C10—C9	-172.0 (3)	C6'—C1'—C2'—O1	-173.0 (3)
C14—C5—C10—C9	69.3 (4)	O2—C5'—C6'—C1'	-178.0 (3)
C13—C8—C9—C12	-53.9 (4)		

The structure was solved using the *MULTAN11/82* package (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). The weighting scheme was according to Killean & Lawrence (1969). H atoms were located on the basis of geometrical considerations and ΔF map suggestions (for hydroxyl and methyl groups). All H atoms were included as fixed atoms in the final refinement, with isotropic displacement parameters set equal to B_{eq} of the parent atoms. Calculations were performed using Enraf-Nonius *SDP* software (B. A. Frenz & Associates, Inc., 1985) on a MicroVAX 3100 computer.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1126). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The 2:1 Adduct of an α -L-Altropyranose Derivative with Hexafluoro-2,6-dihydroxy-2,6-bis(trifluoromethyl)-4-heptanone at 173 K

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Abstract

As reported earlier [Miethchen, Rentsch & Michalik (1994). *Liebigs Ann. Chem.* pp. 219–222], the structure of methyl 2-*O*-(cyclohexylcarbonyl)-6-deoxy-3,4-