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(1*RS*,3*SR*,4*SR*)-3-Methoxy-1-methyl-5-methylene-3-phenyl-1,4-cyclohexanediol

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.119
Data-to-parameter ratio = 10.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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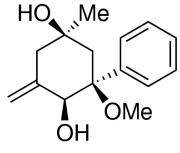
In the structure of the title compound, $C_{15}H_{20}O_3$, the molecular conformation is supported by a strong intramolecular hydrogen-bond contact. A second strong intermolecular hydrogen bond connects the molecules to form infinite zigzag chains.

Comment

1,4-Cyclohexanediols can be transformed into a large variety of different compounds and have been studied in the development of methodologies for the introduction of perfluoroalkylated groups into organic compounds (Chambers *et al.*, 2000), which have many uses in the pharmaceutical and plant protection industries (Rao & Baker, 1994). In addition, these ring systems are precursors in the synthesis of some natural and biologically active compounds such as (±)-streptovitacin A and E-73 (Kondo *et al.*, 1990). 1,4-Cyclohexanediols also constitute the main skeleton of rengyol, a drug of *Forsythia suspensa* (Endo *et al.*, 1987). A novel selective synthesis of highly substituted 1,4-cyclohexanediols by a four- or five-component sequential coupling reaction [see Posner (1986) and Dömling & Ugi (2000) for reviews on multicomponent reactions] of a Fischer carbene complex, a ketone or an ester lithium enolate, and allylmagnesium bromide, will be published elsewhere (Barluenga *et al.*, 2003).



(I)

The molecular structure of the title compound, (I), is shown in Fig. 1. There are two strong hydrogen-bond interactions which help to stabilize the crystal structure. One is an intramolecular contact [O1—H1···O2: $D\cdots A$ = 2.702 (2) Å and

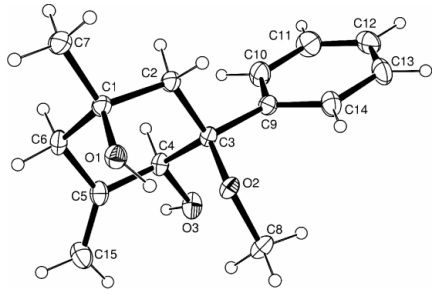


Figure 1
View of the molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level.

D—H···A = 143 (2)°] and the other an intermolecular contact [O3—H3···O1^{i}: D···A = 2.741 (2) Å and D—H···A = 171 (3)°; symmetry code: (i) $2-x, \frac{1}{2}+y, \frac{1}{2}-z$].

Experimental

In a flame-dried round-bottomed flask under nitrogen, the lithium enolate of acetone was prepared by treatment of a solution of acetone (1.1 mmol, 0.08 ml) in tetrahydrofuran (THF; 2 ml) with lithium diisopropylamide (1.2 mmol, 1.2 *M* in THF, 1 ml) at 195 K for 1 h. The resulting solution was then added at the same temperature to a solution of pentacarbonyl(1-methoxy-1-phenylmethylene)-chromium (1 mmol, 312 mg) in THF (15 ml) and the mixture was stirred for 45 min while the temperature was allowed to reach 218 K. The reaction mixture was then cooled to 195 K and allylmagnesium bromide (1.5 mmol, 1 *M* in Et_2O, 1.5 ml) was added. After being stirred for 30 min at 195 K, the reaction was warmed to room temperature and stirred for a further 20 min. It was then quenched with H_2O (10 ml) and neutralized with 6 *N* HCl (*ca* 2 ml). The resulting mixture was diluted with hexane–ethyl acetate (5:1, 100 ml) and subjected to air oxidation under direct sunlight. After 1 d, the suspension was filtered through Celite and extracted with ethyl acetate (3 × 15 ml). The organic layers were combined, dried with Na_2SO_4 and concentrated *in vacuo*. The crude products were purified by column chromatography (silica gel, hexane–EtOAc, 3:1) to give (I) as a pure white solid and as a single diastereoisomer (82%). The compound was recrystallized from pentane–EtOAC (7:1) at 270 K.

Crystal data

$C_{15}H_{20}O_3$	D_x = 1.228 Mg m^{-3}
M_r = 248.31	Cu $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1924 reflections
a = 10.391 (1) Å	θ = 0–70°
b = 12.553 (2) Å	μ = 0.68 mm^{-1}
c = 11.773 (1) Å	T = 150 (2) K
β = 118.961 (10)°	Prism, colourless
V = 1343.6 (3) Å^3	0.38 × 0.15 × 0.15 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	R_{int} = 0.032
φ scans with κ offsets	θ_{max} = 68.3°
Absorption correction: none	$h = -12 \rightarrow 12$
4716 measured reflections	$k = -15 \rightarrow 14$
2452 independent reflections	$l = -14 \rightarrow 14$
2070 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0676P)^2 + 0.5821P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.040	
$wR(F^2)$ = 0.119	
S = 0.93	$(\Delta/\sigma)_{max} < 0.001$
2452 reflections	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
244 parameters	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
All H-atom parameters refined	Extinction correction: *SHELXL*97
	Extinction coefficient: 0.0050 (9)

H atoms were located in difference Fourier maps and refined isotropically, with O—H = 0.85 (2)–0.91 (2) Å and C—H = 0.95 (2)–1.00 (2) Å.

Data collection: *COLLECT* (Nonius, 1997–2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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