# 1-(3,5-Dimethoxyphenyl)-2-{2-[(3,5-dimethoxyphenyl)hydroxymethyl]-4-methoxyphenyl}ethanol

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The title compound, $C_{26}H_{30}O_7$, was obtained by the Grignard reaction of one molecule of 4-methoxybenzylmagnesium chloride with two molecules of 3,5-dimethoxybenzaldehyde. The two new chiral centers have the same absolute configuration $R$ ($S$), and the two hydroxyl groups, surrounded by the three benzene groups, form an intramolecular hydrogen bond.

## Comment

The Grignard reaction is one of the important methods of preparing stilbene derivatives (Campen *et al.*, 1948). In this paper, a very interesting by-product, the title compound, (I), has been isolated from the Grignard reaction of one molecule of 4-methoxybenzylmagnesium chloride with two molecules of 3,5-dimethoxybenzaldehyde.



(I)
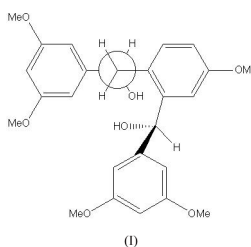
From the molecular structure illustrated in Fig. 1, the two new chiral centers, C18 and C9, adopt the same absolute configuration $R$ ($S$). It seems that the formation of the second chiral center, C9, is controlled by the first one, C18. The two benzene rings, *A* and *B*, adopts an *anti* conformations, as shown in Fig. 1, and the two hydroxy groups (O1H1 and O2H2) surrounded by the three benzene rings form an intramolecular hydrogen bond, *viz*. O1—H1···O2. As expected, the $Csp^3$—$Csp^3$ hybridized bond length of C9—C10 [1.532 (3) Å] is obviously longer than those of $Csp^2$—$Csp^3$ C1—C9 [1.507 (3) Å] and C10—C11 [1.509 (3) Å].

It was found that the five methoxy groups, disregarding H atoms, are almost coplanar with their attached benzene rings. The angle O4—C3—C4 [124.3 (3)°] is larger than that of O4—C3—C2 [115.0 (2)°]. It is assumed that this conformation will minimize the van der Waals interaction between the H atom on C4 and the C7 methyl group. The other four methoxy groups adopt the same conformations. This kind of 'open-mouthed' arrangement of methoxy group was also found in other compounds (Zhang *et al.*, 2001; Zheng *et al.*, 2001). The angle between rings *A* and *B* is 11.4 (3)° and that between rings *A* and *C* is 85.2 (3)°. The torsion angles C6—C1—C9—O1 and C11—C16—C18—O2 are 56.8 (3) and 50.1 (3)°, respectively, while O2—C18—C19—C24 is −0.8 (3)°.
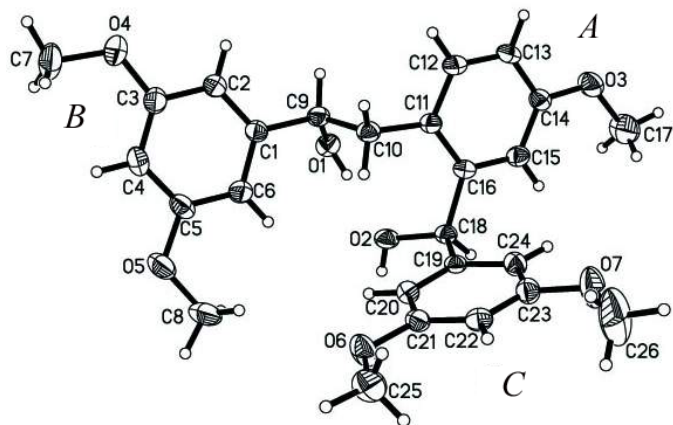
**Figure 1**
View of the molecular structure of (I), shown with 50% probability displacement ellipsoids.
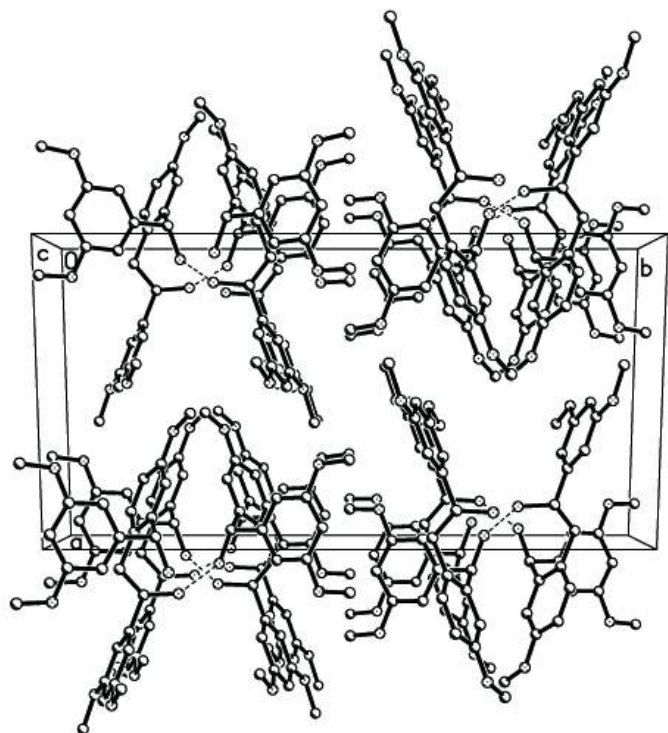


**Figure 2**
A packing diagram of (I), viewed along the c axis.

## Experimental

Under the protection of $N_2$, a mixture of anhydrous ethyl ether (30 ml), magnesium turnings (1.2 g) and magnesium powder (1.2 g) with a small amount of iodine was added slowly to a solution of 4-methoxybenzyl chloride (3.92 g, 0.025 mol) in anhydrous ethyl ether (20 ml). The mixture was warmed to initiate the reaction, and then the remaining 4-methoxybenzyl chloride was added at a rate sufficient to maintain a gentle reflux. The reaction mixture was stirred under reflux for an additional 5 h and then treated dropwise, at room temperature, with a solution of 3,5-dimethoxybenzaldehyde (4.2 g, 0.025 mol) in dry ethyl ether (30 ml). The reaction mixture was heated at reflux for an additional 4 h and was then treated cautiously with dilute HCl. The organic layer was separated and the aqueous layer extracted with ethyl ether. The combined organic extract was washed with water (30 ml) and brine (30 ml). After drying over anhydrous $MgSO_4$, the solvent was evaporated under reduced pressure, and the residue was subjected to column chromatography on silica gel (ethyl acetate/petroleum ether = 1:3). The last fraction provided the title compound (I); yield 28%, m.p. 409.5–410.5 K. $^1$H NMR (400 MHz, CDCl₃, p.p.m.): δ 1.64 (2H, broad), 2.90 (2H, m), 3.77 (15H, t), 4.79 (1H, m), 5.83 (1H, s), 6.34 (2H, d), 6.45 (2H, d), 6.53 (2H, d), 6.80 (1H, d), 6.32 (1H, d), 7.07 (2H, d). Single crystals, suitable for X-ray analysis, were obtained by slow evaporation of a solution in ethanol.

### Crystal data

| | |
|---|---|
| $C_{26}H_{30}O_7$ | $D_x = 1.225$ Mg m$^{-3}$ |
| $M_r = 454.50$ | Mo $K\alpha$ radiation |
| Monoclinic, $P2_1/c$ | Cell parameters from 996 |
| $a = 12.382$ (9) Å | reflections |
| $b = 23.700$ (15) Å | $\theta = 2.6$–22.2° |
| $c = 8.664$ (6) Å | $\mu = 0.09$ mm$^{-1}$ |
| $\beta = 104.313$ (12)° | $T = 293$ (2) K |
| $V = 2464$ (3) Å$^3$ | Prism, colorless |
| $Z = 4$ | $0.40 \times 0.30 \times 0.30$ mm |

### Data collection

| | |
|---|---|
| Bruker CCD area-detector diffractometer | 4346 independent reflections |
| $\varphi$ and $\omega$ scans | 2601 reflections with $I > 2\sigma(I)$ |
| Absorption correction: multi-scan (SADABS; Bruker, 1997) | $R_{int} = 0.038$ |
| | $\theta_{max} = 25.0°$ |
| $T_{min} = 0.965$, $T_{max} = 0.974$ | $h = -12 \rightarrow 14$ |
| 10041 measured reflections | $k = -27 \rightarrow 28$ |
| | $l = -9 \rightarrow 10$ |

### Refinement

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o{}^2) + (0.07P)^2]$ |
| $R[F^2 > 2\sigma(F^2)] = 0.050$ | where $P = (F_o{}^2 + 2F_c{}^2)/3$ |
| $wR(F^2) = 0.138$ | $(\Delta/\sigma)_{max} = 0.003$ |
| $S = 1.05$ | $\Delta\rho_{max} = 0.18$ e Å$^{-3}$ |
| 4346 reflections | $\Delta\rho_{min} = -0.21$ e Å$^{-3}$ |
| 305 parameters | Extinction correction: SHELXL97 |
| H-atom parameters constrained | Extinction coefficient: 0.026 (3) |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| C1—C9 | 1.507 (3) | C10—C11 | 1.509 (3) |
| C9—C10 | 1.532 (3) | | |
| C1—C9—C10—C11 | 174.5 (2) | C16—C18—C19—C20 | 122.2 (2) |
| C9—C10—C11—C16 | −98.3 (3) | | |

**Table 2**
Hydrogen-bonding geometry (Å, °).

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| O1—H1⋯O2 | 0.82 | 1.82 | 2.631 (3) | 168 |
| O2—H2⋯O1$^i$ | 0.82 | 1.78 | 2.593 (2) | 169 |

Symmetry code: (i) $x, \frac{1}{2} - y, \frac{1}{2} + z$.

H atoms were placed geometrically and refined with a riding model.

Data collection: SMART (Bruker, 1997); cell refinement: SMART; data reduction: SAINT (Bruker, 1997) and SHELXTL (Bruker,

1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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