

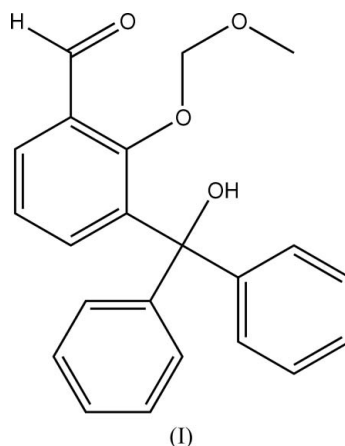
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## Key indicators

Single-crystal X-ray study  
 $T = 273$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.050  
 $wR$  factor = 0.144  
Data-to-parameter ratio = 15.2For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.3-(Hydroxydiphenylmethyl)-2-(methoxy-  
methoxy)benzaldehydeThe title compound,  $\text{C}_{22}\text{H}_{20}\text{O}_4$ , was obtained from methoxy-  
methyl phenyl ether by *ortho*-lithiation and electrophilic  
quenching. The molecular packing in the crystal structure is  
stabilized by an intramolecular hydrogen bond and van der  
Waals forces.Received 23 December 2005  
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## Comment

The title compound, (I), is an important intermediate in the  
synthesis of salicylaldehyde derivatives. We found a good  
method to synthesize 3-substituted salicylaldehyde derivatives  
by repeated *ortho*-lithiation followed by electrophilic  
quenching. Bond lengths and angles in (I) are in agreement  
with values reported in the literature (Tachi *et al.*, 1999). The  
dihedral angle between the planes of the phenyl rings (C17–  
C22) and (C11–C16) is  $106.76(6)^\circ$ . There is one intra-  
molecular hydrogen bond ( $\text{O4}-\text{H4B} = 0.82$  Å,  $\text{O4}\cdots\text{O3} =$   
 $2.89$  Å and  $\text{O4}-\text{H4B}\cdots\text{O3} = 178^\circ$ ).

## Experimental

An LiBu solution (12 ml, 1.6 M) was added to a solution of methoxymethyl phenyl ether (2.76 g, 20 mmol) in dry tetrahydrofuran (40 ml) in an ice-salt bath. After 3 h, a solution of diphenylmethanone (3.64 g, 20 mmol) in dry tetrahydrofuran (10 ml) was added to the resulting slurry in the ice-salt bath. After another 3 h, the reaction was quenched by adding saturated  $\text{NH}_4\text{Cl}$ . The intermediate (2-methoxymethoxyphenyl)diphenylmethanol was obtained after work-up and purification by column chromatography. It was then redissolved in dry tetrahydrofuran (40 ml) and cooled in an ice-salt bath and LiBu solution in hexane (12 ml, 1.6 M) was added. After 3 h, dimethylformamide (5 ml) was added to the cooled slurry. Saturated  $\text{NH}_4\text{Cl}$  was again added to quench the reaction. The organic layer was extracted with ethyl acetate, dried over anhydrous  $\text{MgSO}_4$ , and concentrated under vacuum. The residual solid was

purified by column chromatography to give (I) in 45% yield. Single crystals were grown by slow evaporation of a petroleum ether–ethyl acetate (5:1 v/v) solution.

*Crystal data*

$C_{22}H_{20}O_4$   
 $M_r = 348.38$   
 Orthorhombic, *Pbca*  
 $a = 8.6967$  (16) Å  
 $b = 14.557$  (3) Å  
 $c = 27.408$  (5) Å  
 $V = 3469.7$  (11) Å<sup>3</sup>  
 $Z = 8$   
 $D_x = 1.334$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 7316 reflections  
 $\theta = 2.2$ – $28.2^\circ$   
 $\mu = 0.09$  mm<sup>-1</sup>  
 $T = 273$  (2) K  
 Chunk, colorless  
 0.14 × 0.05 × 0.03 mm

*Data collection*

Bruker APEX area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (*SADABS*; Bruker, 2001)  
 $T_{\min} = 0.987$ ,  $T_{\max} = 0.997$   
 18443 measured reflections

3591 independent reflections  
 2916 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.029$   
 $\theta_{\text{max}} = 26.5^\circ$   
 $h = -10 \rightarrow 10$   
 $k = -18 \rightarrow 16$   
 $l = -27 \rightarrow 34$

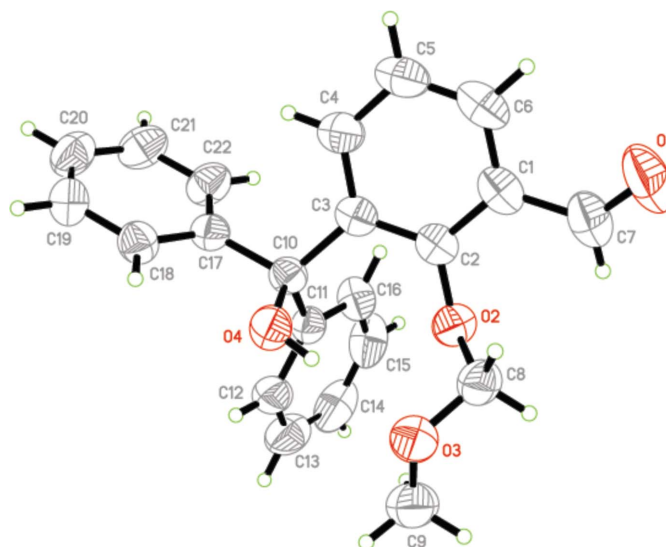
*Refinement*

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.050$   
 $wR(F^2) = 0.144$   
 $S = 1.05$   
 3591 reflections  
 236 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0793P)^2 + 0.6714P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.22$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.21$  e Å<sup>-3</sup>

The H atoms were positioned geometrically (C–H = 0.93, 0.93, 0.98, 0.97 and 0.96 Å for phenyl, formyl, tertiary, methylene and methyl H atoms, respectively, and O–H = 0.82 Å) and were included in the refinement in the riding-model approximation. The displacement parameters of methyl H atoms were set at  $1.5U_{\text{eq}}$  (parent atom), while those of the other H atoms were set at  $1.2U_{\text{eq}}$ .

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics:



**Figure 1**  
*ORTEP-3* (Farrugia, 1997) plot of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

*ORTEP-3* (Farrugia, 1997) and *ViewerPro* (Accelrys, 2001); software used to prepare material for publication: *SHELXL97*.

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**References**

Accelrys (2001). *ViewerPro*. Version 4.2. Accelrys Inc., Burlington, Massachusetts, USA.  
 Bruker (2001). *SAINT* (Version 6.22), *SMART* (Version 5.625) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.  
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.  
 Tachi, Y., Nakayama, S., Tani, F., Ueno, G. & Naruta, Y. (1999). *Acta Cryst.* **C55**, 1351–1353.