

**(E)-3,5,4'-Trimethoxystilbene****Qiang Yin, Yun-Mei Shi, Hui-Min Liu, Chun-Bao Li and Wen-Qin Zhang\***Department of Chemistry, Tianjin University,  
Tianjin 300072, People's Republic of China

Correspondence e-mail: wqzhang@eyou.com

**Key indicators**

Single-crystal X-ray study

 $T = 273\text{ K}$ Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$  $R$  factor = 0.053 $wR$  factor = 0.152

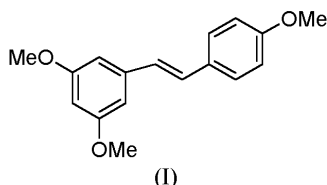
Data-to-parameter ratio = 13.7

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

The title compound,  $\text{C}_{17}\text{H}_{18}\text{O}_3$ , was prepared from the Wittig–Horner reaction of 3,5-dimethoxybenzaldehyde and dimethyl(4-methoxybenzyl)phosphite. There are two molecules in the asymmetric unit, with different conformations.

**Comment**

Recently, (*E*)-stilbene derivatives have attracted considerable interest because of their non-linear optical properties (Soto Bustmante *et al.*, 1995; Papper & Likhtenshtein, 2001) and biological activities. The title compound, (I), is a key intermediate in the synthesis of *trans*-resveratrol, which has been shown to prevent cancer, lower blood pressure, and reduce osteoporosis (Frémont, 2000; Savouret & Quesne, 2002).



There are two molecules, *A* and *B*, in the asymmetric unit (Fig. 1); the 3,5-dimethoxy groups adopt *s-syn* and *s-anti* conformations in molecules *A* and *B*, respectively. The torsion angles of the 4-methoxy groups relative to the benzene rings in *A* and *B* are  $-171.1(3)$  and  $170.3(2)^\circ$ , respectively, and those of the 3,5-dimethoxy groups are in the range  $174.9(3)$ – $177.8(3)^\circ$ . The fact that these methoxy groups are almost coplanar with the benzene rings indicates that all these O atoms are  $sp^2$  hybridized (Zhang *et al.*, 2001; Zheng *et al.*, 2001). The angles between the benzene ring planes in *A* and *B* are  $26.1(3)$  and  $25.0(3)^\circ$ , respectively; however, that in 2,4'-dihydroxy-3,3'-dimethoxy-5-methylstilbene is  $8.90(9)^\circ$  (Li *et al.*, 1999). From Fig. 1, it can be seen that the alkene moieties are slightly twisted; the torsion angles  $\text{C}1-\text{C}6-\text{C}7-\text{C}8$  and  $\text{C}7-\text{C}8-\text{C}9-\text{C}10$  in molecule *A* are  $165.3(3)$  and  $170.8(3)^\circ$ , respectively, while  $\text{C}21-\text{C}22-\text{C}24-\text{C}25$  and  $\text{C}24-\text{C}25-\text{C}26-\text{C}31$  in molecule *B* are  $-159.6(3)$  and  $-178.1(3)^\circ$ , respectively.

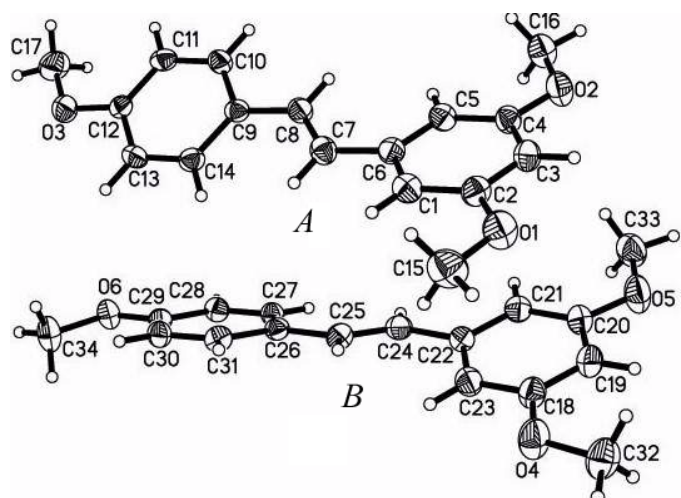
**Experimental**

4-Methoxybenzyl chloride (0.06 mol) was heated in a 100 ml round-bottomed flask with trimethylphosphite (20 ml) until the evolution of methyl chloride had ceased. The excess trimethylphosphite was removed by distillation *in vacuo*. The residue was charged with 3,5-dimethoxybenzaldehyde (DMBA, 0.06 mol), KOH (7.5 g) and DMSO (50 ml). The reaction mixture was stirred at 303 K until there

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**Figure 1**  
View of the two independent molecules of (I), shown with 50% probability ellipsoids.

was no DMBA detected by thin-layer chromatography. Then water (50 ml) was added and the mixture was extracted with ethyl acetate. The organic layer was washed with water and dried over anhydrous  $\text{Na}_2\text{SO}_4$ . The solvent was removed by distillation *in vacuo*. The resulting (*E*)-3,5,4'-trimethoxystilbene was pure, according to HPLC; yield 15.4 g (95%), m.p. 329.5–330.5 K (literature m.p. 330 K; Ali *et al.*, 1992). Crystals were obtained by slow evaporation of a solution in ethyl acetate.  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  3.83 (9H, s), 6.38 (1H, t;  $J = 2$  Hz, H-4), 6.65 (2H, d;  $J = 2$  Hz, H-2, 6), 6.87–6.93 (3H, m), 7.04 (1H, d;  $J = 16$  Hz,  $-\text{CH}=\text{CH}-$ ), 7.45 (2H, d;  $J = 8.8$  Hz, H-2', 6').

#### Crystal data

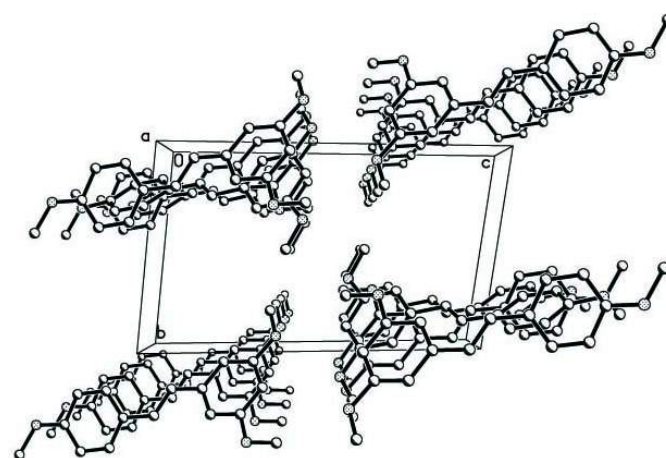
$\text{C}_{17}\text{H}_{18}\text{O}_3$	$Z = 4$
$M_r = 270.31$	$D_x = 1.248 \text{ Mg m}^{-3}$
Triclinic, $P1$	Mo $K\alpha$ radiation
$a = 9.958$ (4) Å	Cell parameters from 664 reflections
$b = 10.048$ (4) Å	$\theta = 2.3\text{--}22.9^\circ$
$c = 16.205$ (6) Å	$\mu = 0.09 \text{ mm}^{-1}$
$\alpha = 90.695$ (8) $^\circ$	$T = 273$ (2) K
$\beta = 105.545$ (7) $^\circ$	Prism, colorless
$\gamma = 111.835$ (7) $^\circ$	$0.30 \times 0.25 \times 0.20 \text{ mm}$
$V = 1438.7$ (10) Å $^3$	

#### Data collection

Bruker SMART CCD area-detector diffractometer	5058 independent reflections
$\varphi$ and $\omega$ scans	2459 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$R_{\text{int}} = 0.034$
$T_{\text{min}} = 0.975$ , $T_{\text{max}} = 0.983$	$\theta_{\text{max}} = 25.0^\circ$
5991 measured reflections	$h = -11 \rightarrow 11$
	$k = -6 \rightarrow 11$
	$l = -19 \rightarrow 18$

#### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.07P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.053$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.152$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 0.96$	$\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
5058 reflections	$\Delta\rho_{\text{min}} = -0.14 \text{ e \AA}^{-3}$
368 parameters	Extinction correction: SHELXL
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.026 (3)



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

**Table 1**

Selected geometric parameters ( $^\circ$ ).

C8—C7—C6	127.0 (3)	C25—C24—C22	125.2 (3)
C7—C8—C9	128.4 (3)	C24—C25—C26	128.7 (3)
C6—C1—C2—O1	−179.7 (3)	C32—O4—C18—C23	175.6 (3)
C2—C3—C4—O2	−179.6 (3)	O4—C18—C19—C20	−179.9 (3)
C16—O2—C4—C3	−176.2 (3)	O5—C20—C21—C22	178.4 (3)
C6—C7—C8—C9	177.9 (3)	O4—C18—C23—C22	178.9 (3)
C7—C8—C9—C10	170.8 (3)	O6—C29—C30—C31	−179.9 (2)
C17—O3—C12—C13	−171.1 (3)		

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: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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