

## 3-Hydroxy-1-phenyl-3-(3,4,5-trimethoxyphenyl)propan-1-one

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## Key indicators

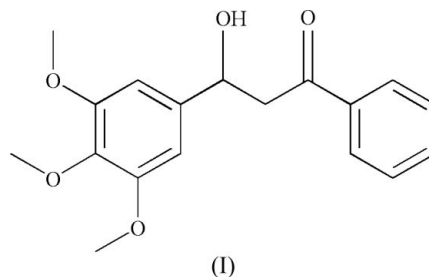
Single-crystal X-ray study  
 $T = 294$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.045  
 $wR$  factor = 0.120  
Data-to-parameter ratio = 15.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound,  $\text{C}_{18}\text{H}_{20}\text{O}_5$ , was synthesized by the Reformatsky reaction in an aqueous medium. The benzene rings form a dihedral angle of  $78.6(1)^\circ$ . Two intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds of  $2.921(2)$  Å between the hydroxyl and carbonyl groups form a centrosymmetric dimer. These hydrogen-bonded rings are stacked along the  $b$  axis, forming hydrophilic channels.

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## Comment

The Reformatsky reaction (Bieber *et al.*, 1997; Chan *et al.*, 1994; Shen *et al.*, 1997) is one of the most powerful and useful reactions in organic synthesis. It provides a good strategy for C—C bond formation. In more recent work, different aqueous media for the Reformatsky reaction have been developed in our group. We have also reported a series of compounds synthesized using an aqueous system (Yu *et al.*, 2003, 2004). A new compound, (I), has been synthesized by the reaction of 2-bromoacetophenone with 3,4,5-trimethoxybenzaldehyde in the presence of zinc in an aqueous medium.



The molecular structure of (I) and the atom-numbering scheme are shown in Fig. 1. As a result of the conjugation of the aromatic ring and the carbonyl group, the system is close to being planar, and the  $\text{C}10-\text{C}11-\text{C}12-\text{C}13$  torsion angle is  $-165.84(17)^\circ$ . The O atoms of the methoxy groups are almost coplanar with the benzene ring, with a mean deviation of  $0.0265(2)$  Å. Molecules related by inversion symmetry are connected by two  $\text{O}4-\text{H}4\cdots\text{O}5$  hydrogen bonds, generating a dimer (Table 1 and Fig. 2). The crystal packing involves a  $\text{C}17-\text{H}\cdots\text{O}2$  interaction (Table 1).

## Experimental

The title compound was synthesized by adding 3,4,5-trimethoxybenzaldehyde (4 mmol) and 2-bromoacetophenone (6 mmol) to a mixture of a saturated solution of calcium chloride (16 ml), zinc (16 mmol), ammonium chloride (2.0 g), a trace amount of iodine, cetyltrimethylammonium bromide (0.006 g) and tetrahydrofuran (1.4 ml). After the mixture had been stirred at room temperature for

4 h, the reaction was quenched with hydrochloric acid (2 M, 0.5 ml). The mixture was extracted with diethyl ether, evaporated and separated by flash chromatography (using ethyl acetate–petroleum ether). A white powder (yield 52%) was obtained. Slow evaporation of a dichloromethane–petroleum ether solution afforded the title compound as single crystals. IR (KBr,  $\nu$  cm<sup>-1</sup>): 3458, 1682; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  7.95–6.66 (m, 7H), 5.28 (t, 1H), 3.88 (s, 9H), 3.36 (d, 2H). Analysis calculated for C<sub>18</sub>H<sub>20</sub>O<sub>5</sub>: C 68.35, H 6.33%, found C 68.42, H 6.47%.

Crystal data

C <sub>18</sub> H <sub>20</sub> O <sub>5</sub>	$D_x = 1.322$ Mg m <sup>-3</sup>
$M_r = 316.34$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1727 reflections
$a = 14.992$ (3) Å	$\theta = 2.7$ – $25.9^\circ$
$b = 5.0020$ (11) Å	$\mu = 0.10$ mm <sup>-1</sup>
$c = 21.237$ (4) Å	$T = 294$ (2) K
$\beta = 93.375$ (4)°	Block, colourless
$V = 1589.8$ (6) Å <sup>3</sup>	$0.26 \times 0.20 \times 0.14$ mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	3265 independent reflections
$\varphi$ and $\omega$ scans	1938 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{int} = 0.047$
$T_{min} = 0.977$ , $T_{max} = 0.987$	$\theta_{max} = 26.4^\circ$
8459 measured reflections	$h = -18 \rightarrow 12$
	$k = -6 \rightarrow 6$
	$l = -20 \rightarrow 26$

Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 0.1076P]$
$R[F^2 > 2\sigma(F^2)] = 0.046$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.120$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.01$	$\Delta\rho_{max} = 0.23$ e Å <sup>-3</sup>
3265 reflections	$\Delta\rho_{min} = -0.23$ e Å <sup>-3</sup>
215 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4–H4 <sup>i</sup> ···O5 <sup>i</sup>	0.83 (3)	2.10 (3)	2.921 (2)	171 (3)
C17–H17 <sup>ii</sup> ···O2 <sup>ii</sup>	0.93	2.47	3.341 (2)	156

Symmetry codes: (i)  $-x + 2, -y + 2, -z + 2$ ; (ii)  $x + \frac{3}{2}, y - \frac{1}{2}, z + \frac{3}{2}$ .

All carbon-bound H atoms were positioned geometrically and refined as riding (C–H = 0.93–0.98 Å). For the CH and CH<sub>2</sub> groups,  $U_{iso}(H)$  values were set equal to  $1.2U_{eq}$  and for the methyl groups they were set equal to  $1.5U_{eq}$ . Atom H4 was refined with O4–H4 = 0.83 (3) Å.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1997); software used to prepare material for publication: SHELXTL.

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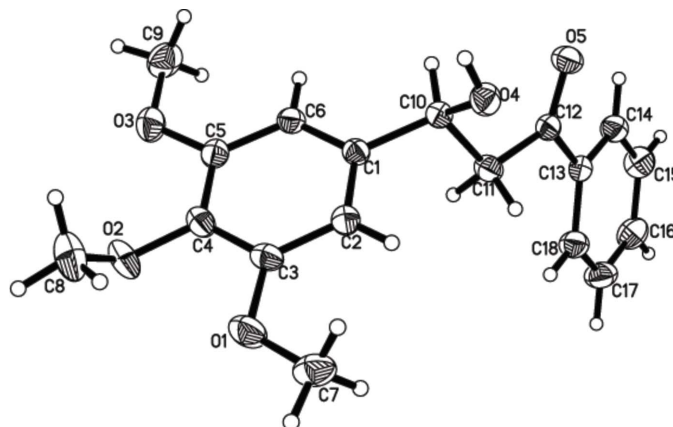


Figure 1 View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level.

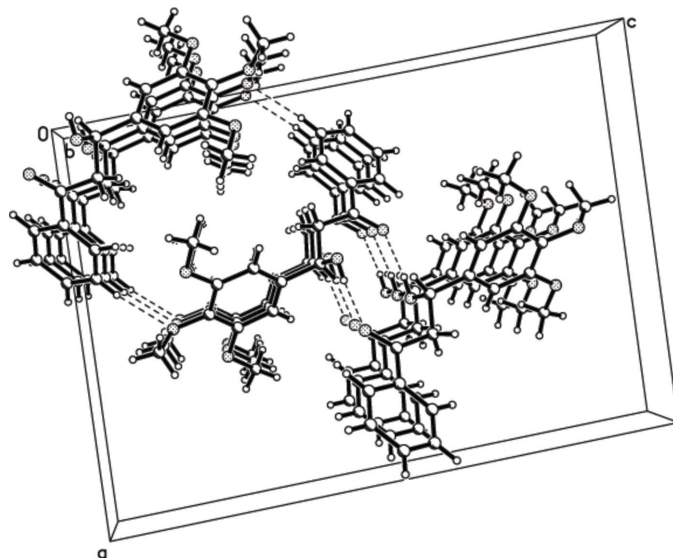


Figure 2 The crystal structure of (I), viewed along the  $b$  axis. Dashed lines indicate O–H···O hydrogen bonds.

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