Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(2*E*,5*E*)-2,5-Bis(2,3,4-trimethoxybenzylidene)-cyclopentanone

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Received 12 March 2007
Accepted 17 April 2007

Key indicators

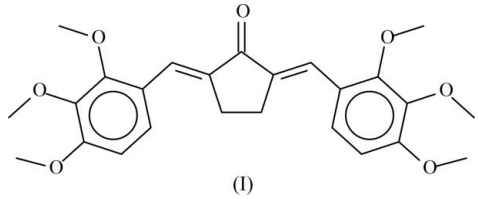
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
R factor = 0.051
wR factor = 0.158
Data-to-parameter ratio = 13.2

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

In the title compound, $C_{25}H_{28}O_7$, the two benzylidenyl substituents on the cyclopentanone and the five-membered ring are approximately coplanar. The structure has three independent molecules per asymmetric unit.

Comment

We have recently screened curcuminoids for aldose reductase (Du, Bao *et al.*, 2006) and α-glucosidase (Du, Liu *et al.*, 2006) inhibition. This class of compounds is readily synthesized by reacting a substituted benzaldehyde with cyclopentanone (Sardjiman *et al.*, 1997; Youssef *et al.*, 2004); in the case of the title compound, (I), 2,3,4-trimethoxybenzaldehyde was used as the reactant. Fig. 1 presents a displacement ellipsoid plot of one of the three independent molecules of the asymmetric unit of the triclinic unit; the long and flat shape of the molecules is apparent. There are no strong interactions between them.



(I)

The Cambridge Structural Database (Allen, 2002; Version 5.28, November 2006) lists several examples of 2,5-dibenzylidenecyclopentanones, including the parent unsubstituted homolog (Kawamata *et al.*, 1998; Theocharis *et al.*, 1983; Theocharis *et al.*, 1984). Bond dimensions in (I) are unexceptional and similar to those found in these compounds.

Experimental

The title compound was synthesized using a general procedure (Sardjiman *et al.*, 1997; Youssef *et al.*, 2004). 2,3,4-Trimethoxybenzaldehyde (1.96 g, 0.01 mol) and cyclopentanone (0.42 g,

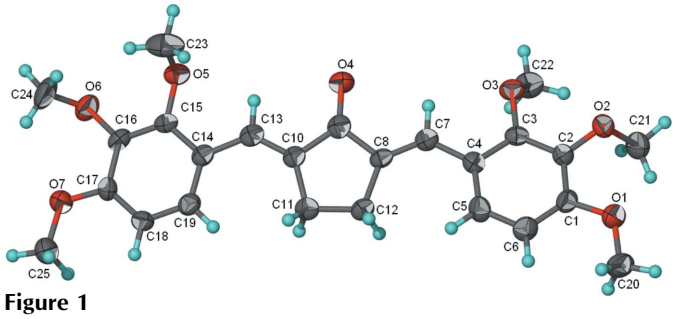


Figure 1
The structure of one of the three (almost identical) independent molecules of (I), taken as representative of the group. Displacement ellipsoids are drawn at the 70% probablity level.

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0.005 mol) were dissolved in glacial acetic acid (50 ml) saturated with anhydrous hydrogen chloride. The mixture was warmed at 298–303 K for 2 h. Cold water was added to precipitate the yellow compound. Crystals were obtained by recrystallization from acetic acid. The formulation was established by the NMR spectrum and ESI mass spectrum. ^{1}H NMR (MSDO-d_6, 300 MHz): δ 7.58 (*s*, 2H, —CH═), 7.13 (*s*, 2H, aromatic), 7.02 (*s*, 2H, aromatic), 3.73 (*s*, 18H, —OCH_3), 3.10 (4H, –CH_2—CH_2–). ESI–MS *m/z*: 441 ($[M + 1]^+$).

Crystal data

$C_{25}H_{28}O_7$
M_r = 440.47
Triclinic, $P\overline{1}$
a = 12.6636 (6) Å
b = 15.4927 (8) Å
c = 19.297 (1) Å
α = 108.089 (1)°
β = 95.412 (1)°
γ = 111.242 (1)°
V = 3261.6 (3) Å^3
Z = 6
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 173 (2) K
0.42 × 0.38 × 0.33 mm

Data collection

Bruker SMART area-detector diffractometer
Absorption correction: none
23885 measured reflections
11375 independent reflections
6017 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.158
S = 0.96
11375 reflections
865 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

H atoms were positioned geometrically (C—H = 0.95–0.98 Å) and were included in the refinement in the riding-model approximation, with U_{iso}(H) = 1.2 or 1.5U_{eq}(C).

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: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

We thank the Guangdong Provincial Science Foundation and the University of Malaya for supporting this study.

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