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1-Phenyl-3-(3,4,5-trimethoxyphenyl)-1,3-propanedione

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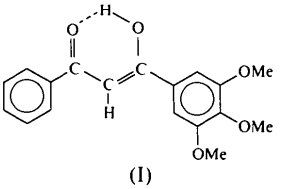
Abstract

The crystal structure of 1-phenyl-3-(3,4,5-trimethoxyphenyl)-1,3-propanedione, $C_{18}H_{18}O_5$, consists of discrete molecules separated by normal van der Waals interactions. The molecule exists in the enol form [*i.e.* 3-hydroxy-1-phenyl-3-(3,4,5-trimethoxyphenyl)propene-1-one] in the solid state, stabilized by a short intramolecular hydrogen bond.

Comment

β-Diketones have been studied intensively, specifically on the basis of their intramolecular hydrogen bonding in the enol form (Gilli, Bellucci, Ferretti & Bertolasi, 1989; Bertolasi, Gilli, Ferretti & Gilli, 1991). They form complexes with transition metal cations (Usha & Vijayan, 1989) as well as with the alkali earth metal cations Mg^{2+} (Hollander, Templeton & Zalkin, 1973*a*), Ca^{2+} (Hollander, Templeton & Zalkin, 1973*b*) and Sr^{2+} (Hollander, Templeton & Zalkin, 1973*c*). Crystal structures of dibenzoylmethanes have been reported as stable (Williams, 1966; Hollander, Templeton & Zalkin, 1973*d*; Jones, 1976; Kaitner & Meštrović, 1993) and metastable (Etter, Jahn, Urbańczyk-Lipkowska, 1987) polymorphs.

The title molecule (I) is almost planar. The terminal phenyl and trimethoxylated phenyl rings make torsion angles O9—C9—C10—C11 of −3.1 (3) and O7—C7—C1—C2 of 12.1 (3)°. The β-diketone–enol fragment O9—C9—C8—C7—O7—H7 is almost planar (O9—C9—C8—C7 −3.6, O7—C7—C8—C9 −0.8°). These values are not very different from those given for the symmetrical 1,3-diphenyl-1,3-propanedione enol (Hollander, Templeton & Zalkin, 1973*d*).



(I)

The β-diketone–enol group forms a very short intramolecular O7—H7···O9 hydrogen bond [O7···O9 2.508 (2) Å] which is in the range (2.432–2.554 Å) found for a series of dibenzoylmethanes (Bertolasi, Gilli, Ferretti & Gilli, 1991). Other dimensions of this hydrogen bond are O7—H7 1.11 (2), H7···O9 1.45 (2) Å and O7—H7···O9 155 (2)°. The position of the H7 atom is also confirmed by the C—O bond distances C7—O7 [1.303 (3) Å] and C9—O9 [1.278 (3) Å] and the C—C bond distances C8—C7 [1.373 (3) Å] and C8—C9 [1.414 (3) Å]. A view of the title molecule is presented in Fig. 1 and shows the atom-labelling scheme.

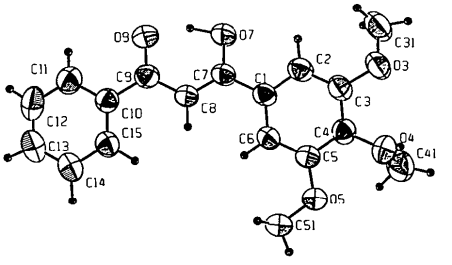


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule with the atom-labelling scheme (the displacement ellipsoids are drawn at 50% probability).

The enol structures of dibenzoylmethanes are similar to those of the chalcones, which often crystallize in non-centrosymmetric space groups and so have large non-linear optical properties (Zhengdong & Genbo, 1993). However, the title compound crystallizes in a centrosymmetric space group which is unfavourable for such optical properties.

Experimental

Crystal data

$C_{18}H_{18}O_5$
M_r = 314.34

Mo $K\alpha$ radiation
λ = 0.71073 Å

Monoclinic
$P2_1/c$
a = 7.442 (1) Å
b = 22.709 (2) Å
c = 9.738 (1) Å
β = 101.22 (1)°
V = 1614.3 (3) Å^3
Z = 4
D_x = 1.293 Mg m^{-3}

Cell parameters from 25 reflections
θ = 1–25°
μ = 0.09 mm^{-1}
T = 294 K
Prismatic
0.45 × 0.42 × 0.26 mm
Brown

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical (ψ scans of seven reflections)
T_{min} = 0.949, T_{max} = 0.999
4134 measured reflections
3374 independent reflections

1944 observed reflections [$F > 3.0\sigma(F)$]
R_{int} = 0.034
θ_{max} = 26.3°
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 28$
$l = 0 \rightarrow 12$
3 standard reflections
frequency: 60 min
intensity variation: 0.4%

Refinement

Refinement on F
R = 0.043
wR = 0.059
S = 2.07
1944 reflections
281 parameters
Only H-atom U's refined
$w = 4F_o^2/\sigma^2(F_o^2)$

$(\Delta/\sigma)_{max}$ = 0.74
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
O3	0.1334 (2)	0.06785 (9)	0.4241 (2)	7.25 (4)
O4	0.2521 (2)	0.00649 (7)	0.2282 (2)	6.03 (4)
O5	0.5818 (2)	0.02740 (6)	0.1632 (2)	5.77 (4)
O7	0.6052 (2)	0.22436 (7)	0.5785 (1)	6.25 (4)
O9	0.8426 (2)	0.30273 (7)	0.5773 (1)	5.95 (4)
C1	0.5638 (3)	0.1464 (1)	0.4154 (2)	4.36 (5)
C2	0.3975 (3)	0.1324 (1)	0.4534 (2)	4.90 (5)
C3	0.2989 (3)	0.0847 (1)	0.3944 (2)	5.03 (5)
C4	0.3610 (3)	0.0509 (1)	0.2952 (2)	4.74 (5)
C5	0.5291 (3)	0.0639 (1)	0.2594 (2)	4.76 (5)
C6	0.6298 (3)	0.11144 (9)	0.3202 (2)	4.76 (5)
C7	0.6627 (3)	0.1995 (1)	0.4744 (2)	4.59 (5)
C8	0.8080 (3)	0.2227 (1)	0.4246 (2)	4.91 (5)
C9	0.9001 (3)	0.27430 (9)	0.4810 (2)	4.67 (5)
C10	1.0654 (3)	0.2957 (1)	0.4341 (2)	4.53 (5)
C11	1.1497 (3)	0.3468 (1)	0.4887 (2)	6.07 (6)
C12	1.3084 (4)	0.3657 (1)	0.4473 (3)	7.25 (7)
C13	1.3836 (3)	0.3340 (1)	0.3527 (3)	6.67 (6)
C14	1.3006 (3)	0.2835 (1)	0.2958 (2)	6.08 (6)
C15	1.1415 (3)	0.2644 (1)	0.3364 (2)	5.23 (5)
C31	0.0954 (3)	0.0837 (1)	0.5535 (3)	8.00 (7)
C41	0.3065 (4)	−0.0503 (1)	0.2746 (3)	7.67 (7)
C51	0.7515 (3)	0.0393 (1)	0.1229 (3)	6.60 (6)

Table 2. *Selected geometric parameters* (Å, °)

O3—C3	1.373 (3)	C3—C4	1.382 (3)
O3—C31	1.391 (3)	C4—C5	1.394 (3)
O4—C4	1.375 (3)	C5—C6	1.381 (3)
O4—C41	1.400 (3)	C7—C8	1.373 (3)
O5—C5	1.364 (3)	C8—C9	1.414 (3)
O5—C51	1.419 (3)	C9—C10	1.476 (3)
O7—C7	1.303 (3)	C10—C11	1.376 (3)
O9—C9	1.278 (3)	C10—C15	1.394 (3)
C1—C2	1.396 (3)	C11—C12	1.388 (4)
C1—C6	1.382 (3)	C12—C13	1.372 (4)
C1—C7	1.469 (3)	C13—C14	1.368 (4)
C2—C3	1.372 (3)	C14—C15	1.389 (3)
C3—O3—C31	117.9 (2)	C1—C6—C5	120.2 (2)
C4—O4—C41	114.5 (2)	O7—C7—C1	116.4 (2)
C5—O5—C51	117.7 (2)	O7—C7—C8	120.6 (2)
C2—C1—C6	119.8 (2)	C1—C7—C8	123.1 (2)
C2—C1—C7	119.1 (2)	C7—C8—C9	122.4 (2)
C6—C1—C7	121.2 (2)	O9—C9—C8	119.8 (2)
C1—C2—C3	119.9 (2)	O9—C9—C10	118.6 (2)
O3—C3—C2	124.1 (2)	C8—C9—C10	121.5 (2)
O3—C3—C4	115.4 (2)	C9—C10—C11	120.2 (2)
C2—C3—C4	120.5 (2)	C9—C10—C15	121.1 (2)
O4—C4—C3	119.5 (2)	C11—C10—C15	118.7 (2)
O4—C4—C5	120.7 (2)	C10—C11—C12	119.9 (2)
C3—C4—C5	119.7 (2)	C11—C12—C13	120.9 (2)
O5—C5—C4	115.8 (2)	C12—C13—C14	120.1 (2)
O5—C5—C6	124.4 (2)	C13—C14—C15	119.3 (2)
C4—C5—C6	119.8 (2)	C10—C15—C14	121.1 (2)

The structure was solved by direct methods with a straightforward run of the *MULTAN*11/82 program (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Full-matrix refinement on F was carried out using the *MolEN* program (Fair, 1990), which was also used for the absorption correction. All H atoms were located in a difference Fourier map and refined isotropically (without constraints).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving H atoms and torsion angles have been deposited with the IUCr (Reference: PA1113). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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