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An axially chiral 1,2-diketone: 1-(3,4-dimethoxyphenyl)propane-1,2-dione

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.134
Data-to-parameter ratio = 15.1

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

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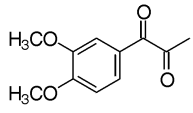
The O atom of the distal carbonyl group in 1-(3,4-dimethoxyphenyl)propane-1,2-dione, $C_{11}H_{12}O_4$, is displaced from the plane that is defined by the proximal C═O double bond and the aromatic entity of the molecule. This arrangement gives rise to a stereogenic C—C axis.

Comment

The title compound, (I), crystallizes in triclinic space group $P\overline{1}$. Atom O2 is displaced from the mean plane defined by atoms O1, C1, C2 and C4 [0.864 (3) Å]. The C—C bond, which connects both carbonyl groups, constitutes a stereogenic axis. The distance C1—C2 of 1.526 (3) Å is unexpectedly long for a single bond between two sp^2-hybridized C atoms. (*M*)- and (*P*)-configured stereoisomers of diketone (I) exist in a 1:1 ratio in the unit cell [Z = 2, O1—C1—C2—O2 = −125.0 (2)° for (*M*)-(I) (Fig. 1) and 125.0 (2)° for (*P*)-(I)]. The bond lengths and angles associated with the two carbonyl groups of (I) [C1—O1 = 1.218 (2) Å, C2—O2 = 1.217 (2) Å, O1—C1—C2 = 116.2 (2)° and O1—C2—C1 = 120.1 (2)°] correspond to values that have been reported for structurally related alkyl- and alkoxy-substituted benzils (Mohr *et al.*, 1994). The CH_3 substituents of the two methyl ether entities in (I) point in opposite directions and are only marginally displaced from the plane of the aromatic ring [C6—C7—O3—C10 = 7.5 (3)° and C9—C8—O4—11 = 2.7 (3)°]. This geometry is considered to originate from an energetically favorable overlap of π-orbitals at O3 and O4 with those of the aromatic substituent.



(I)

Experimental

1-(3,4-Dimethoxyphenyl)propane-1,2-dione crystallized from a solution of analytically pure *N*-(hydroxy)-5-(3,4-dimethoxyphenyl)-4-methylthiazole-2(3*H*)-thione in CH_2Cl_2/*n*-hexane (3:1 *v*/*v*) on standing for 14 d at 293 K (Hartung *et al.*, 2004). *N*-(Hydroxy)-5-(3,4-dimethoxyphenyl)-4-methylthiazole-2(3*H*)-thione was prepared from 3,4-dimethoxyphenylacetone by extension of a literature procedure (Hartung & Schwarz, 2002; Hartung *et al.*, 2003).

Crystal data

$C_{11}H_{12}O_4$
M_r = 208.21
Triclinic, $P\overline{1}$
a = 7.927 (2) Å
b = 8.185 (2) Å
c = 10.018 (2) Å
α = 65.00 (2)°
β = 72.01 (2)°
γ = 62.82 (2)°
V = 518.6 (2) Å^3
Z = 2
D_x = 1.333 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1238 reflections
θ = 2.3–22.1°
μ = 0.10 mm^{-1}
T = 298 (2) K
Prism, yellow
0.52 × 0.34 × 0.06 mm

Data collection

Oxford Diffraction Xcalibur CCD diffractometer	1102 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.028$
ω scans	$\theta_{max} = 26.4^\circ$
Absorption correction: none	$h = -9 \rightarrow 9$
4883 measured reflections	$k = -10 \rightarrow 10$
2054 independent reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.044$	$w = 1/[\sigma^2(F_o^2) + (0.0801P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.134$	
$S = 0.84$	$(\Delta/\sigma)_{max} = 0.010$
2054 reflections	$\Delta\rho_{max} = 0.14$ e Å^{-3}
136 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.218 (2)	C2—O2	1.217 (2)
C1—C4	1.467 (3)	C2—C3	1.473 (3)
C1—C2	1.526 (3)		
O1—C1—C4	123.4 (2)	O3—C7—C8	115.4 (2)
O1—C1—C2	116.1 (2)	C9—C8—O4	125.8 (2)
C4—C1—C2	120.5 (2)	O4—C8—C7	114.8 (2)
O2—C2—C3	122.9 (2)	C8—C9—C4	121.0 (2)
O2—C2—C1	120.1 (2)	C7—O3—C10	118.0 (2)
C3—C2—C1	117.0 (2)	C8—O4—C11	117.5 (2)
O3—C7—C6	124.7 (2)		
O1—C1—C2—O2	−125.0 (2)	O3—C7—C8—C9	179.5 (2)
C4—C1—C2—O2	56.1 (3)	O3—C7—C8—O4	0.9 (3)
O1—C1—C2—C3	52.8 (3)	C6—C7—C8—O4	−178.1 (2)
C4—C1—C2—C3	−126.2 (2)	C8—C7—O3—C10	−171.4 (2)
O1—C1—C4—C5	176.0 (2)	C9—C8—O4—C11	2.7 (3)
C2—C1—C4—C5	−5.1 (3)		

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2001); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2001); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997).

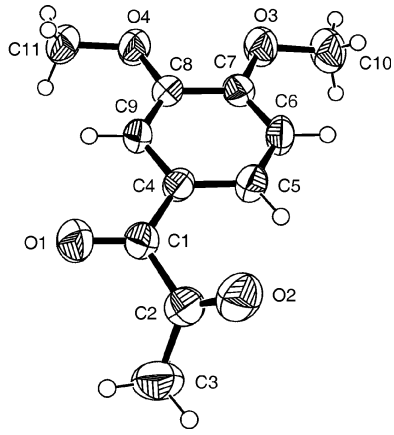


Figure 1
The molecular structure of (*M*)-(I), which has been arbitrarily selected from the racemate of the title compound that is present in the unit cell. Displacement ellipsoids are drawn at the 50% probability level.

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