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3-Acetyl-1-phenyl-2-pentene-1,4-dione

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Key indicators

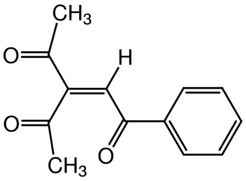
Single-crystal X-ray study
T = 213 K
Mean σ(C–C) = 0.002 Å
R factor = 0.054
wR factor = 0.127
Data-to-parameter ratio = 18.7

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

The title compound, $C_{13}H_{12}O_3$, crystallizes in the monoclinic system, with two independent molecules in the asymmetric unit. In one of the molecules, the phenyl ring forms a dihedral angle of 23.93 (5)° with the pentenedione plane, while in the other, the dihedral angle is 13.33 (5)°.

Comment

The strong one-electron-oxidant, ceric ammonium nitrate (CAN), has been established as an efficient reagent in generating α-carbonylalkyl radicals from enolizable ketones, and the addition of these carbon-centered radicals to alkenes has been successfully used in organic synthesis in various C—C bond-formation reactions (Nair *et al.*, 1997). We have recently investigated the CAN-mediated formation of 1,3-pentanedione with phenylacetylene and we report here the crystal structure of the title compound, (I), which is one of the products of this reaction.



(I)

The asymmetric unit of (I) consists of two molecules *A* and *B* (Fig. 1). The corresponding bond distances and angles of these two molecules agree with each other and show normal values (Allen *et al.*, 1987). A fit of the non-H atoms of molecule *A* with those of the inverted molecule *B* resulted in a weighted r.m.s. deviation of 0.121 Å. In both molecules, the pentenedione moiety is nearly planar, with the methyl C atom (C13) deviating by a maximum of 0.241 (1) and 0.124 (2) Å in molecules *A* and *B*, respectively. The C8—C9—C10—C11 and C8—C9—C10—O2 torsion-angle values [96.4 (2) and −88.4 (2)°, respectively, for molecule *A*, and −97.5 (2) and 86.5 (2)° for molecule *B*] indicate that the acetyl group is twisted normal to the pentenedione plane. The dihedral angle between the pentenedione plane and the phenyl ring of molecule *A* [23.93 (5)°] is larger than that of molecule *B* [13.33 (5)°]. In the solid state, weak C—H···O hydrogen bonds link *B* molecules and inversion-related *A* molecules (Table 2).

Experimental

The title compound was isolated from the reaction mixture of acetylacetone with ceric ammonium nitrate (CAN) in the presence of

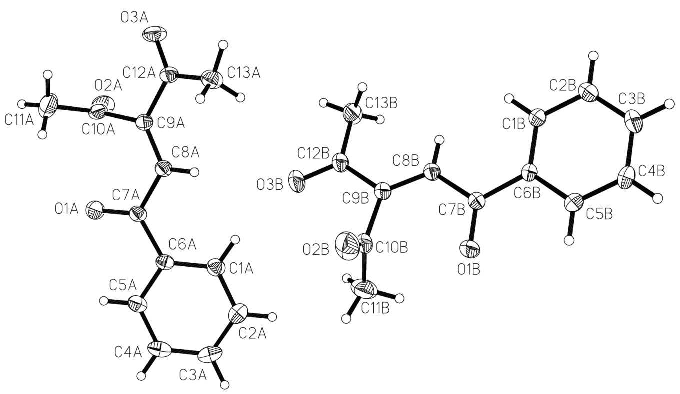


Figure 1

A view of the structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

an excess amount of phenylacetylene in acetonitrile, by column chromatography on silica gel. Single crystals were grown by slow evaporation from a solution of petroleum ether (b.p. 333–353 K)/ethyl acetate (7:1 *v*/*v*).

Crystal data

$C_{13}H_{12}O_3$
$M_r = 216.23$
Monoclinic, $P2_1/c$
$a = 14.1314$ (2) Å
$b = 15.0764$ (1) Å
$c = 10.9229$ (2) Å
$\beta = 102.015$ (1)°
$V = 2276.15$ (6) Å^3
$Z = 8$

$D_x = 1.262$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8192 reflections
$\theta = 2.5$–$28.3°$
$\mu = 0.09$ mm^{-1}
$T = 213$ (2) K
Block, yellow
0.50 × 0.40 × 0.40 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
13416 measured reflections
5495 independent reflections

3634 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.090$
$\theta_{\rm max} = 28.2°$
$h = -18 \rightarrow 15$
$k = -20 \rightarrow 19$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.127$
$S = 0.91$
5495 reflections
294 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0326P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.36$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.30$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.030 (2)

Table 1

Selected geometric parameters (Å, °).

C8*A*—C9*A*	1.3424 (19)	C8*B*—C9*B*	1.3404 (19)
C6*A*—C7*A*—C8*A*—C9*A*	168.43 (13)	C7*B*—C8*B*—C9*B*—C12*B*	178.90 (13)
C7*A*—C8*A*—C9*A*—C12*A*	177.38 (12)	C8*B*—C9*B*—C10*B*—C11*B*	−97.45 (18)
C8*A*—C9*A*—C10*A*—C11*A*	96.37 (18)	C8*B*—C9*B*—C12*B*—O3*B*	−176.52 (14)
C8*A*—C9*A*—C12*A*—O3*A*	167.58 (14)	C8*B*—C9*B*—C12*B*—C13*B*	2.6 (2)
C8*A*—C9*A*—C12*A*—C13*A*	−12.5 (2)	C10*B*—C9*B*—C12*B*—C13*B*	−179.30 (14)
C6*B*—C7*B*—C8*B*—C9*B*	−166.06 (13)		

Table 2

Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1*B*—H1*B*···O3*A*$^{\rm i}$	0.93	2.54	3.415 (2)	158

Symmetry code: (i) $1 - x, -y, 1 - z$.

The H atoms were fixed geometrically and treated as riding on the parent C atoms, with aromatic C—H = 0.93 Å and methyl C—H = 0.96 Å, and with displacement parameters $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* and *SADABS* (Sheldrick, 1996); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Nair, V., Mathew, J. & Prabhakaran, N. (1997). *J. Chem. Soc. Rev.* **26**, 127–132.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

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