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Key indicators

Single-crystal X-ray study
T = 213 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.069
wR factor = 0.172
Data-to-parameter ratio = 18.8

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

6,6-Dimethyl-2-phenyl-4,5,6,7-tetrahydrobenzofuran-4-one

In the title compound, $\text{C}_{16}\text{H}_{16}\text{O}_2$, the tetrahydrobenzene ring adopts a half-chair conformation. The phenyl substituent is almost coplanar with the furan ring.

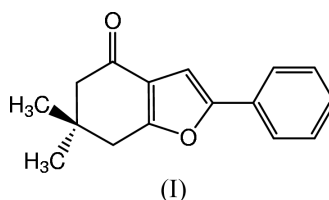
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Comment

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 CAN-mediated reactions of 1,3-dicarbonyl compounds with phenylacetylene (Li & Xu, 2002), and we report here the crystal structure of the title compound, (I), which is one of the products in the reaction of 5,5-dimethylcyclohexa-1,3-dione with phenylacetylene.



The bond lengths and angles in (I) (Fig. 1 and Table 1) are within normal ranges (Allen *et al.*, 1987). In the tetrahydrobenzofuran moiety (O1/C1—C8), the furan ring is planar, while the tetrahydrobenzene ring is not planar and adopts a half-chair conformation. Atoms C3 and C4 are displaced by $-0.536(2)$ and $0.166(2) \text{ \AA}$, respectively, from the C1/C2/C5/C6 plane. This conformation is also confirmed by its puckering parameters [$Q_2 = 0.377(2) \text{ \AA}$, $Q_3 = -0.283(2) \text{ \AA}$, $Q_T = 0.471(2) \text{ \AA}$, $\varphi = 46.5(3)^\circ$ and $\theta = 53.1(2)^\circ$; Cremer & Pople, 1975]. The two ring planes are nearly coplanar, corresponding to a dihedral angle of $7.7(1)^\circ$. The attached ketone atom O2 deviates by $0.231(1) \text{ \AA}$ from the tetrahydrobenzene ring plane, and the configurations of the two methyl groups, C15 and C16, are conditioned by the sp^3 state of the C3 atom, with the average angle subtended at the C3 atom being 109.5° .

The C9—C14 phenyl ring and the furan ring are almost coplanar, with a dihedral angle of $7.0(1)^\circ$. This enhances the π -conjugation interactions between the two rings and slightly shortens the C8—C9 bond length [$1.460(2) \text{ \AA}$, *cf.* $\text{C}_{sp^2}-\text{C}_{ar}$ 1.470 (Allen *et al.*, 1987)].

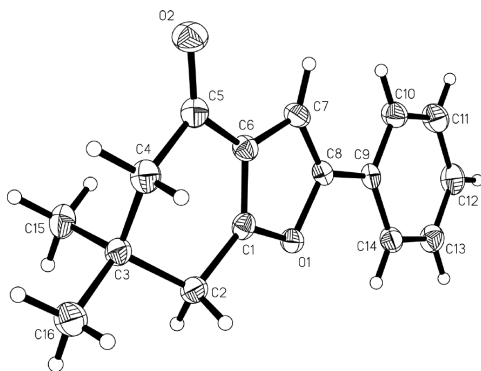


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

The title compound, (I), was isolated from the reaction of 5,5-dimethylcyclohexa-1,3-dione with ceric ammonium nitrate (CAN) in the presence of an excess amount of phenylacetylene in acetonitrile, by column chromatography on silica gel. Single crystals of (I), suitable for X-ray crystallographic measurements, were prepared by slow evaporation of the solvent from a petroleum ether–ethyl acetate solution (4:1 v/v).

Crystal data

$C_{16}H_{16}O_2$
 $M_r = 240.29$
Monoclinic, $P2_1/c$
 $a = 5.9261$ (3) Å
 $b = 11.1029$ (6) Å
 $c = 19.6978$ (11) Å
 $\beta = 95.521$ (1)°
 $V = 1290.0$ (1) Å³
 $Z = 4$

$D_x = 1.237$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 5717 reflections
 $\theta = 2.8$ – 28.3 °
 $\mu = 0.08$ mm⁻¹
 $T = 213$ (2) K
Block, colorless
 $0.44 \times 0.38 \times 0.28$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
7600 measured reflections
3123 independent reflections
2228 reflections with $I > 2\sigma(I)$

$R_{int} = 0.093$
 $\theta_{max} = 28.3$ °
 $h = -7 \rightarrow 7$
 $k = -14 \rightarrow 14$
 $l = -18 \rightarrow 26$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.069$
 $wR(F^2) = 0.172$
 $S = 0.94$
3123 reflections
166 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0884P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.48$ e Å⁻³
 $\Delta\rho_{min} = -0.43$ e Å⁻³
Extinction correction: *SHELXTL*
Extinction coefficient: 0.073 (8)

Table 1

Selected geometric parameters (Å, °).

C1–C2	1.480 (2)	C4–C5	1.512 (2)
C2–C3	1.546 (2)	C5–C6	1.461 (2)
C3–C4	1.545 (2)		
C16–C3–C15	108.99 (14)	C16–C3–C2	109.58 (13)
C16–C3–C4	109.32 (14)	C15–C3–C2	110.19 (14)
C15–C3–C4	109.61 (13)	C4–C3–C2	109.14 (13)

The H atoms were positioned geometrically and were treated as riding atoms on the parent C atoms, with C–H distances = 0.93–0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

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.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Li, Y. & Xu, J.-H. (2002). In preparation.
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.* **A46**, C-34.