Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

(*E*)-7-(2-Furyl)-6-heptene-2,5-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.111
Data-to-parameter ratio = 9.5

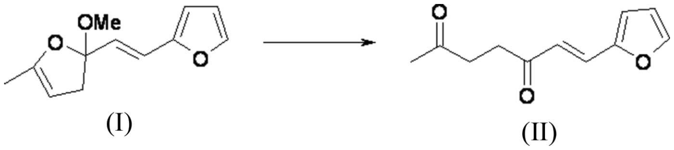
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 27 January 2007
Accepted 22 March 2007

The molecule of the title compound, $C_{11}H_{12}O_3$, is essentially planar, the maximum deviation from the least-squares plane through all non-H atoms being 0.37 (1) Å for the methyl group. The molecular packing is made up of a network of C—H···O hydrogen-bonding interactions involving the carbonyl groups.

Comment

1,4-Dicarbonyls are highly valuable intermediates for the synthesis of five-membered heterocycles. The most typical approach to these heterocycles is the Paal–Knorr method (Knorr, 1884; Paal, 1885), in which a dehydrative cyclization with acid generates furans, or pyrroles when amines are in the reaction medium [see Gilchrist (1999) for a review]. The simplicity of the Paal–Knorr reaction is limited only by the availability of the substrate, 1,4-dicarbonyl compounds (Hegedus & Parry, 1985). A recently developed method employing Fischer carbene complexes and enones or enals provides an alternative way to build suitably substituted 1,4-diketones by ring opening of 2-methoxy-2,3,dihydrofurans. Alternatively, these dihydrofurans can be directly transformed into furans by acid-catalysed aromatization. The structural characterization of the title compound, (II), is reported here.



The molecule of (II) is essentially planar (Fig. 1), the maximum deviation from the least-squares plane through all non-H atoms being 0.37 (1) Å for the methyl group (C11). No unusual features were found concerning the bond distances and angles in (II).

The crystal packing is made up of a network of C—H···O hydrogen-bonding interactions involving the carbonyl atoms O2 and O3 (details are given in Table 1).

Experimental

The synthesis of the dihydrofuran (I) will be published elsewhere. 159 mg of (I) (0.77 mmol) were dissolved in THF (20 ml). Three drops of an aqueous HCl 0.5 *M* (0.075 mmol) solution were added, with stirring at room temperature. The reaction was controlled by TLC. Once the initial compound had disappeared (after ca. 30 min), three drops of a saturated aqueous solution of $NaHCO_3$ were added. Na_2SO_4 was used to dry the organic layer which was then filtered. The solvent was removed under vacuum and the crude reaction mixture was purified by flash column chromatography on silica gel. This yielded compound (II) as an orange oil (58%), that crystallized after being stored at low temperature (277 K) for about four weeks.

Crystal data

$C_{11}H_{12}O_3$
$M_r = 192.21$
Orthorhombic, $P2_12_12_1$
$a = 7.8751$ (5) Å
$b = 9.1651$ (3) Å
$c = 14.1891$ (6) Å
$V = 1024.11$ (9) Å^3
$Z = 4$
Cu $K\alpha$ radiation
$\mu = 0.75$ mm^{-1}
$T = 293$ (2) K
0.26 × 0.18 × 0.09 mm

Data collection

Oxford Diffraction Nova CCD diffractometer
Absorption correction: multi-scan (*Scale3 Abspack*; Oxford Diffraction, 2006)
$T_{\min} = 0.852$, $T_{\max} = 0.931$
4806 measured reflections
1207 independent reflections
943 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.111$
$S = 1.10$
1207 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.11$ e Å^{-3}
$\Delta\rho_{\min} = -0.12$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O2$^{\rm i}$	0.93	2.56	3.468 (2)	165
C3—H3···O2$^{\rm ii}$	0.93	2.59	3.3056 (18)	134
C5—H5···O2	0.93	2.52	2.8276 (17)	100
C8—H8*B*···O2$^{\rm iii}$	0.97	2.59	3.479 (2)	153
C5—H5···O3$^{\rm iv}$	0.93	2.85	3.603 (2)	139
C9—H9*B*···O3$^{\rm v}$	0.97	2.64	3.491 (2)	147
C11—H11*B*···O3$^{\rm v}$	0.96	2.62	3.478 (2)	148

Symmetry codes: (i) $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{3}{2}, -y+2, z+\frac{1}{2}$; (iii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z$; (iv) $x-\frac{1}{2}, -y+\frac{3}{2}, -z$; (v) $-x+2, y+\frac{1}{2}, -z-\frac{1}{2}$.

In the absence of significant anomalous scattering effects, Friedel pairs were merged. H atoms were included in calculated positions and treated as riding atoms, C—H = 0.93–0.97 Å with $U_{\rm iso}({\rm H}) = 1.2$ or $1.5U_{\rm eq}({\rm C})$.

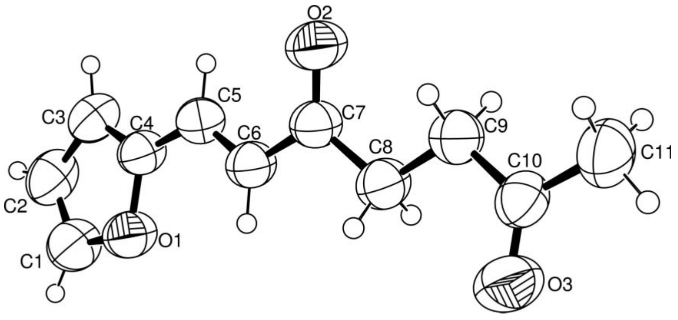


Figure 1
A view of the molecular structure of (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

Financial support from the Spanish Ministerio de Educacion y Ciencia (MAT2006–01997 and ‘Factoría de Cristalización’ Consolider Ingenio 2010) is acknowledged.

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