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X-ray Crystallographic Structure of 3-(Propan-2-ylidene)benzofuran-2(3H)-one

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Abstract: 3-(Propan-2-ylidene)benzofuran-2(3H)-one, C₁₁H₁₀O₂, crystallizes in the monoclinic space group P2₁/c with unit cell parameters a = 7.1869(3), b = 18.0636(10), c = 13.1656(7) Å, β = 96.763(3)°, V = 1697.28(15) Å³, Z = 8 (Z' = 2 independent molecules, A and B, per asymmetric unit), D_c = 1.363 g cm⁻³ and the linear absorption coefficient = 0.093 mm⁻¹. The crystal structure determination was carried out using MoKα X-ray data measured at 120(2) K. In the final refinement cycle the data/restraints/parameter ratios were 3827/0/239, the goodness-of-fit on F² = 1.019. Final R indices for [I > 2σ(I)] were R1 = 0.0517, wR2 = 0.1115 and R indices (all data) R1 = 0.1007, wR2 = 0.1354. The largest electron density difference peak and hole were 0.254 and -0.244 electrons Å⁻³, respectively. The two independent molecules A and B have essentially identical bond lengths and angles and are highly planar, with rms deviation for all 12 non-H atoms of 0.0292 Å for molecule A and 0.0592 Å for molecule B. The two molecules in the asymmetric unit are assembled parallel to each other within 1.58(4)° with each of the six atoms of benzene ring A overlapping with one of the atoms in benzene ring B at a mean distance of 3.84(3) Å. The closest contacts between molecules A and B are C(12A)H...O(4B) = 3.521 Å and C(11B)H...O(4A) = 3.441 Å. The crystal structure is formed by infinite sheets of these assemblies all lying parallel to the (1 0 $\bar{1}$) plane. The presence of two independent molecules in the asymmetric unit provides an opportunity to examine the molecular geometry in detail by comparison. The benzene ring in both molecules A and B exhibits distortions as a result of the presence of the furan moiety. In particular, C(1)-C(6) = 1.369(2) Å in molecule A and 1.367(2) Å in molecule B are both significantly less than the average of the other 10 C-C bonds, 1.393(2), by about 12σ. Other examples of crystal structures are discussed where this effect is observed.

Keywords: Knoevenagel condensation; X-ray crystal structure; molecular geometry

1. Introduction

Recent interest in the Knoevenagel condensations of indol-2-one with aldehydes or ketones led to the report of a number of X-ray structures^{1, 2} of the resulting 3-methylidene-1,3-dihydro-2H-indol-2-one compounds (**Figure 1**), which display intermolecular hydrogen bonding between the NH and C=O groups of neighbouring units. The aim, in this current study, is to compare the structure of the Knoevenagel condensation product of acetone with benzofuran-2(3H)-one as opposed to that with indolin-2-one since, in the former case, due to the lack of an NH group, no intermolecular H-bonding, of the type described above, is possible in the final product.

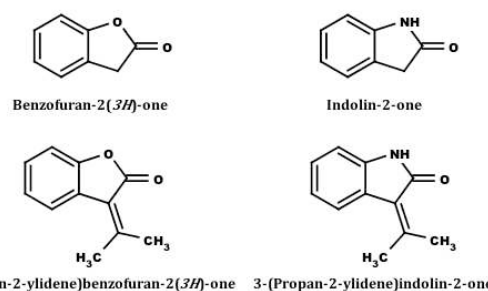


Figure 1. Knoevenagel condensation products.

2. Results and Discussion

Figure 2 shows the chemical scheme and crystallographic atom numbering for C₁₁H₁₀O₂ (**I**). **Figure 3** shows molecule A determined in the structure analysis and drawn with Visualizer, Discovery Studio (Release 3.5, Accelrys Inc., 2012).³ ORTEP⁴ and

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RASTER3D,⁵ as implemented in WinGX, were used to prepare **Figure 4** which shows the relative disposition of molecules A and B depicted with their thermal ellipsoids at 50% probability.

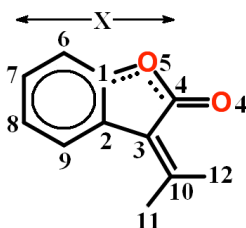


Figure 2. Schematic chemical structure and atom numbering for $C_{11}H_{10}O_2$ (**I**). The bonding shown is discussed. X denotes the region of geometry discussed in more detail.

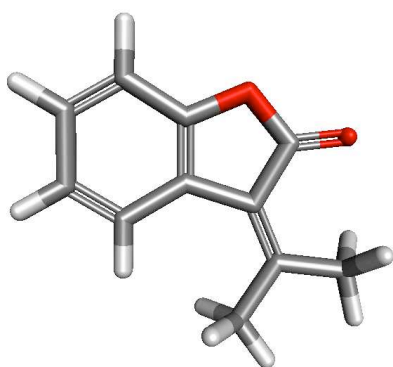


Figure 3. The 3-(propan-2-ylidene)benzofuran-2(3H)-one, $C_{11}H_{10}O_2$ (**I**) molecule A as determined by X-ray analysis (Drawn with Visualizer, Discovery Studio, Release 3.5, Accelrys Inc., 2012)³ [<http://accelrys.com/products/datasheets/whats-new-in-discovery-studio.pdf>].

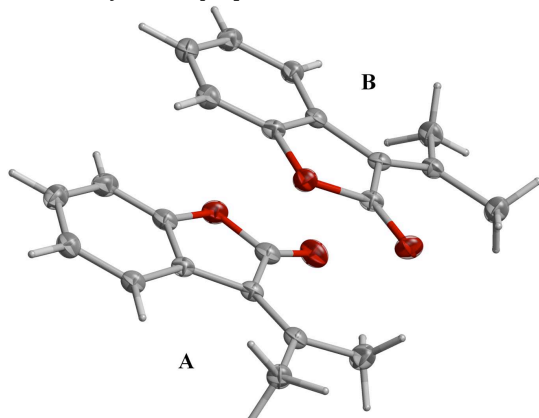


Figure 4. 3-(Propan-2-ylidene)benzofuran-2(3H)-one (Ortep/Raster)^{4,5}, molecules A and B. The two molecules in the asymmetric unit are parallel within $1.67(6)^\circ$ and the benzene rings overlap with a mean intermolecular distance of $3.84(3)$ Å between corresponding atoms. Thermal ellipsoids are shown at 50% probability.

2.1. Molecular Geometry

2.1.1. Bond lengths

Crystal data are summarized in **Table 1**. Bond lengths are determined to a precision of ± 0.002 Å and bond

angles to $\pm (0.2)^\circ$. **Table 2** lists corresponding bond lengths in molecules A and B, respectively. These data indicate that there is a complete one to one agreement in bond length values. In the benzene ring all bond lengths are standard aromatic C-C bonds⁶ except C(1)-C(6) which is $1.369(2)$ Å in molecule A and $1.367(2)$ Å in molecule B, which are both shorter by about 12σ than the standard value of 1.40 Å. This is possibly a result of the proximity of O(5) in the adjoining ring. In fact in the chain extending to the furan ring, i.e., C(6)-C(1)-O(5)-C(4), C(1)-O(5) = $1.391(2)$ Å in molecule A and $1.396(2)$ Å in molecule B, and O(5)-C(4) = $1.388(2)$ Å in molecule A and $1.395(2)$ Å in molecule B, this reduction in bond length is consistent with the presence of a delocalisation effect. This region is designated X in **Figure 2**.

Table 1. Crystal data and structure refinement for (**I**).

Identification code	2008src0248 CCDC804780
Empirical formula	$C_{11}H_{10}O_2$
Formula weight	174.20
Temperature	120(2) K
Wavelength (wtd. mean)	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	$a = 7.1869(3)$ Å; $\alpha = 90^\circ$ $b = 18.0636(10)$ Å; $\beta = 96.763(3)^\circ$ $c = 13.1656(7)$ Å; $\gamma = 90^\circ$
Volume	$1697.28(15)$ Å ³
Z	8 (2 molecules/asymmetric unit)
D_c	1.363 g cm ⁻³
Absorption coefficient	0.093 mm ⁻¹
F(000)	736
Crystal size	$0.44 \times 0.30 \times 0.08$ mm ³
Theta range for data collection	3.07 to 27.48°
Index ranges	$-9 \leq h \leq 9$, $-23 \leq k \leq 23$, $-17 \leq l \leq 17$
Reflections collected in total sphere	20306
Independent reflections	3827 [R(int) = 0.0800]
Completeness to theta = 27.48°	98.3 %
Max. and min. transmission	0.9926 and 0.9601
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	3827/0/239
Goodness-of-fit on F^2	1.019
Final R indices [$I > 2\sigma(I)$]	R1 = 0.0517, wR2 = 0.1149
R indices (all data)	R1 = 0.1007, wR2 = 0.1354
Largest diff. peak and hole	0.254 and -0.244 electrons Å ⁻³

2.1.2. Geometry Optimization

To investigate molecule A further its geometry was optimized in the program Visualizer, Discovery Studio (Release 4.0, Accelrys Inc., 2013).⁷ [<http://accelrys.com/products/discovery-studio/>] using the Smart Minimizer algorithm and conjugate gradient minimization criteria. The molecule was typed with the CHARMM forcefield⁸ using the Partial Charge method.⁹ After 2000 steps convergence was reached and the final potential energy of the molecule **I** was 61.5 kcal/mol. The results for molecules A and B bond lengths are listed in **Table 3**. These results show that the energy minimized molecular geometry is consistent with the shortening of bond C(1)-C(6) in both molecules A and B. Further examples of structures where such a short bond length feature may be present are noted below.

Table 2. 1-Benzofuran-2(3*H*)-one: bond lengths [Å] and angles [°] for molecules (I) A and B.

Bond Lengths		
<i>Bond</i>	<i>(I)A</i>	<i>(I)B</i>
C(1)-C(2)	1.392(2)	1.395(2)
C(2)-C(3)	1.471(2)	1.469(2)
C(3)-C(10)	1.350(2)	1.349(2)
C(10)-C(11)	1.492(2)	1.494(2)
C(10)-C(12)	1.503(2)	1.494(2)
C(3)-C(4)	1.489(2)	1.479(2)
O(4)-C(4)	1.204(2)	1.204(2)
C(4)-O(5)	1.388(2)	1.395(2)
O(5)-C(1)	1.391(2)	1.396(2)
C(1)-C(6)	1.369(2)	1.367(2)
C(6)-C(7)	1.393(2)	1.398(2)
C(7)-C(8)	1.390(2)	1.391(2)
C(8)-C(9)	1.389(2)	1.389(2)
C(9)-C(2)	1.398(2)	1.397(2)
Bond Angles		
C(1)-C(2)-C(3)	106.5(2)	106.7(2)
C(2)-C(3)-C(10)	130.8(2)	129.8(2)
C(3)-C(10)-C(11)	122.3(2)	121.4(2)
C(3)-C(10)-C(12)	122.8(2)	123.4(2)
C(11)-C(10)-C(12)	115.0(2)	115.2(2)
C(2)-C(3)-C(4)	104.9(2)	105.1(2)
C(3)-C(4)-O(4)	132.5(2)	132.6(2)
C(3)-C(4)-O(5)	108.4(2)	108.6(2)
O(4)-C(4)-O(5)	119.2(2)	118.7(2)
C(4)-O(5)-C(1)	108.2(2)	108.4(2)
C(6)-C(1)-O(5)	123.5(2)	123.7(2)
C(6)-C(1)-C(2)	124.6(2)	124.6(2)
C(1)-C(6)-C(7)	116.7(2)	116.7(2)
C(6)-C(7)-C(8)	120.9(2)	120.6(2)
C(7)-C(8)-C(9)	121.1(2)	121.4(2)
C(8)-C(9)-C(2)	119.1(2)	118.9(2)

Features in region X, **Figure 2**, are in **bold**.

2.1.3. Bond angles

Table 2 lists corresponding bond angles in molecule A and B, respectively. These data indicate that there is a complete one to one agreement in bond angle values. Of the bond angles in the benzene ring C(1)-C(6)-C(7) = 116.7(2)° in both molecules, is significantly less than 120° and may be associated with the unusually short bond length C(1)-C(6) discussed above. The molecular geometry of the two independent molecules A and B is practically identical, as revealed by application of the AUTOFIT routine in PLATON¹⁰ using Molfit with Quaternion Transformation.¹¹

Table 3. 3-(Propan-2-ylidene) benzofuran-2(3*H*)-one (I): X-ray bond lengths [Å] for molecules A and B and molecules A and B after minimization in the Visualizer (Discovery Studio, Release 4.0, Accelrys Inc., 2013).

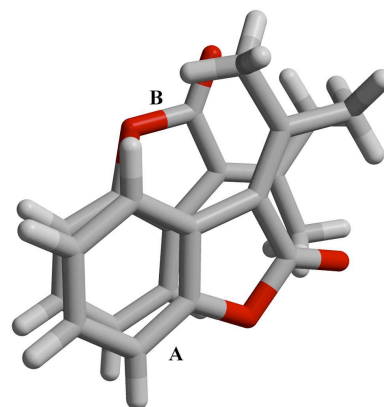
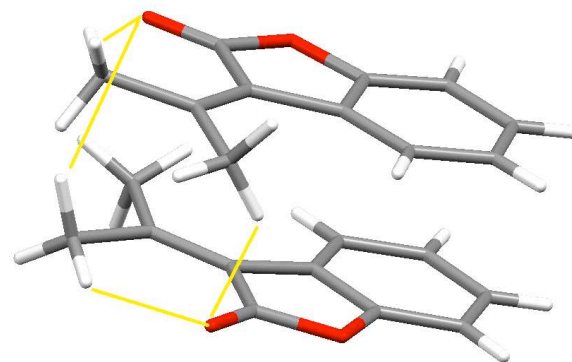
Bond	X-ray Structure		Minimized Structure	
	(I)A	(I)B	A	B
C(1)-C(2)	1.392(2)	1.395(2)	1.402	1.402
C(2)-C(3)	1.471(2)	1.469(2)	1.489	1.488
C(3)-C(10)	1.350(2)	1.349(2)	1.359	1.358
C(10)-C(11)	1.492(2)	1.494(2)	1.515	1.514
C(10)-C(12)	1.503(2)	1.494(2)	1.516	1.516
C(3)-C(4)	1.489(2)	1.479(2)	1.503	1.501
O(4)-C(4)	1.204(2)	1.204(2)	1.225	1.224
C(4)-O(5)	1.388(2)	1.395(2)	1.337	1.337
O(5)-C(1)	1.391(2)	1.396(2)	1.461	1.460
C(1)-C(6)	1.369(2)	1.367(2)	1.367	1.366
C(6)-C(7)	1.393(2)	1.398(2)	1.394	1.394
C(7)-C(8)	1.390(2)	1.391(2)	1.396	1.394
C(8)-C(9)	1.389(2)	1.389(2)	1.398	1.398
C(9)-C(2)	1.398(2)	1.397(2)	1.375	1.375

2.2. Crystal Packing

The two molecules in the asymmetric unit are assembled parallel to each other within 1.58(4)° with each of the six atoms of benzene ring A overlapping with one of the atoms in benzene ring B at a mean distance of 3.84(3) Å; **Table 4(a)** and **Figure 5(a)**. The closest contacts between molecules A and B are C(12A)H---O(4B) = 3.521 Å and C(11B)H---O(4A) = 3.441 Å, **Table 4(b)** and **Figures 5(b)** and **(c)**. The crystal structure is formed by infinite sheets of these assemblies all lying parallel to the (1 0 $\bar{1}$) plane, **Figure 6**.

Table 4(a). Intermolecular C...C distances in the asymmetric unit (Å).

C(1A)...C(9B)	3.853
C(2A)...C(2B)	3.848
C(6A)...C(8B)	3.798
C(7A)...C(7B)	3.804
C(8A)...C(6B)	3.837
C(9A)...C(1B)	3.886

**Figure 5(a).** 3-(Propan-2-ylidene)benzofuran-2(3*H*)-one mode of overlapping of molecules A and B (Ortep/Raster)^{4,5}. The two molecules in the asymmetric unit are parallel within 1.58(4)°**Figure 5(b).** 3-(Propan-2-ylidene)benzofuran-2(3*H*)-one, overlapping of molecules A and B. The short CH...O contacts as listed in Table 4 are indicated. Benzene - benzene distances range from 3.597 Å for C3(A) --- C3(B) to 3.886 Å for C1(A) --- C1(B); (Drawn with Mercury)⁵.

Further Examples of Structures where Chain X Exhibits Delocalisation

In order to further investigate the delocalization effect observed in (I), molecules A and B, the corresponding bond in 2,3-diphenyl-5-methyl-1-benzofuran C₂₁H₁₆O₁

(II),¹² Figure 7a and 3-(1-phenylmeth-(*E*)-ylidene)-3*H*-benzofuran-2-one (III),¹³ Figure 7b was noted. Bond lengths in chain X for molecules (I), (II) and (III) are listed in Table 5. The delocalization in bond C(1)-C(6), evident in all 4 molecules listed here, is less prominent in molecule (II).

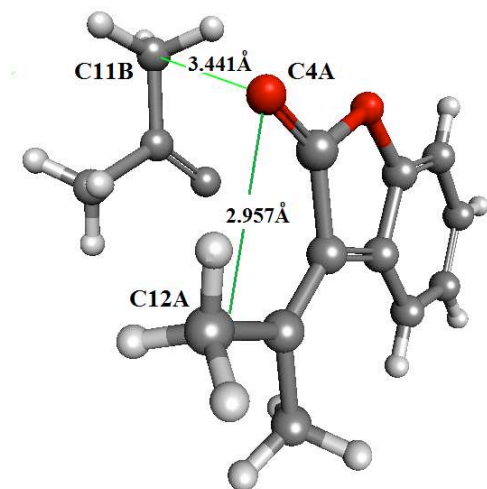


Figure 5(c). 3-(Propan-2-ylidene)benzofuran-2(3*H*)-one, details of the intra molecular CH...O contact in Molecule A and between Molecule A and Molecule B. Essentially equivalent contacts occur within Molecule B and between Molecule B...A (Drawn with Visualizer, Discovery Studio, Release 3.5, Accelrys Inc., 2012)³ [<http://accelrys.com/products/datasheets/whats-new-in-discovery-studio.pdf>.] see also Table 4.

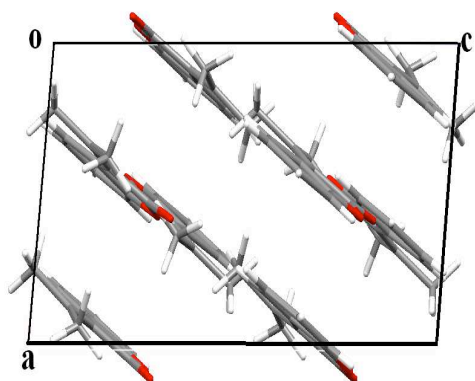


Figure 6. 3-(Propan-2-ylidene)benzofuran-2(3*H*)-one crystal packing viewed along **b**. The molecular planes are parallel to (1 0 $\bar{1}$). (Drawn with Mercury)⁵.

Table 4(b). Possible hydrogen bonds [\AA and deg.]

D-H...A	d(D-H) \AA	d(H...A) \AA	d(D...A) \AA	$\angle(\text{DHA})$ deg
C(12A)-H(11A)...O(4A)*	0.98	2.20	2.957(2)*	132.6
C(12A)-H(11C)...O(4B)	0.98	2.80	3.521(2)	131.4
C(11B)-H(11D)...O(4A)	0.98	2.73	3.441(2)	130.2
C(12B)-H(11G)...O(4B)*	0.98	2.25	3.001(2)*	132.3
C(8A)-H(7)...O(4B)#1	0.95	2.75	3.412(2)	127.5
C(9A)-H(8)...O(5B)#1	0.95	2.86	3.798(2)	168.4
C(9A)-H(8)...O(4B)#1	0.95	2.84	3.452(2)	122.8
C(11A)-H(10A)...O(5B)#1	0.98	3.00	3.459(2)	110.1
C(7B)-H(106)...O(4B)#2	0.95	2.85	3.798(2)	176.8
C(11B)-H(11F)...O(5A)#3	0.98	2.75	3.241(2)	111.7
C(9B)-H(108)...O(4A)#3	0.95	2.70	3.414(2)	132.3

*C...O distance is less than the sum of the van der Waals radii; Symmetry transformations used to generate equivalent atoms: #1 -x,-y,-z; #2 -x,y+1/2,-z+1/2; #3 -x+1,-y,-z+1

3. Experimental

3-(Propan-2-ylidene)benzofuran-2(3*H*)-one(I), $\text{C}_{11}\text{H}_{10}\text{O}_2$, was provided by the Trost laboratory¹⁴ and crystallized from dichloromethane/hexane by vapour diffusion.

3.1. Data Collection

A colourless crystal plate fragment of size $0.44 \times 0.30 \times 0.08$ mm³ was mounted on a glass fibre and flash frozen to 120 K. Intensities were collected, using monochromated MoK α radiation, $\lambda = 0.71073$ \AA , with a Bruker-Nonius Kappa CCD camera, employing ϕ and ω scans to cover an asymmetric unit. Programs used were: unit cell determination with the program DirAx;¹⁵ data collection was controlled by Collect;¹⁶ data reduction and cell refinement were carried out using the program Denzo;¹⁷ an absorption correction was made with SADABS.^{18, 19} An Oxford Cryosystems "Cryostreams" 700,²⁰ enabled the data to be collected at 120 K. The crystals are monoclinic, space group $P2_1/c$ with unit cell dimensions $a = 7.1869(3)$, $b = 18.0636(10)$, $c = 13.1656(7)$ \AA , $\beta = 96.763(3)^\circ$, $V = 1697.28(15)$ \AA^3 , $Z = 8$ ($Z' = 2$), $D_c = 1.363$ g cm⁻³ and linear absorption coefficient = 0.093 mm⁻¹. The crystal structure determination was carried out using MoK α X-ray data measured at 120(2) K. In total 20306 integrated reflections were collected, reducing to an asymmetric unit data set of 3827 [R(int) = 0.080], and completeness of data to theta = 27.48° of 98.3 % corresponding to a resolution of 0.770 \AA . There was no significant variation in intensity of reference reflections during the course of data collection.

3.2. X-ray Structure Analysis

The crystal structure was solved by Direct Methods (SHELXS-86) and refined using SHELXL-97^{21, 22} both implemented in the WinGX system of programs.²³ Non-hydrogen atoms were refined anisotropically by full-matrix least squares methods. All H atoms were set geometrically and refined in riding mode. Geometrical calculations were made with the programs PARST and PLATON¹⁰ as implemented in WinGX. In the final refinement cycle there were 3827 data to 239 parameters, resulting in a final goodness-of-fit on F^2 of 1.019. Final R indices for [$I > 2\sigma(I)$] were $R1 = 0.0517$, $wR2 = 0.1149$ and R indices (all data) $R1 = 0.1007$, $wR2 = 0.1354$. The largest and smallest difference electron density regions were +0.254 and -0.244 electrons \AA^{-3} , respectively. Crystal data are summarized in Table 1.

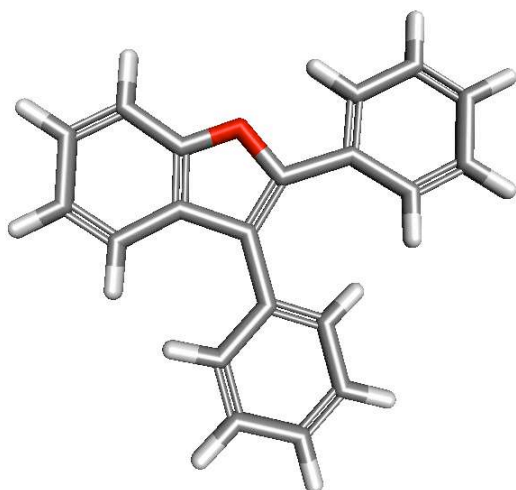


Figure 7a. 2,3-Diphenyl-5-methyl-1-benzofuran $C_{21}H_{16}O_1$ (II)⁶ determined by X-ray analysis (Drawn with Visualizer, Discovery Studio, Release 3.5, Accelrys Inc., 2012)³

[<http://accelrys.com/products/datasheets/whats-new-in-discovery-studio.pdf>].

Table 5. Bond lengths [Å] in the chain C(4)-O(5)-C(1)-C(6) in 1-benzofuran-2-one (I) molecules A and B and the corresponding values in molecules (II) and (III).

Bond	(I)A	(I)B	(II)	(III)
C(4)-O(5)	1.388(2)	1.395(2)	1.398(4)	1.381(3)
O(5)-C(1)	1.391(2)	1.396(2)	1.380(4)	1.398(3)
C(1)-C(6)	1.369(2)	1.367(2)	1.378(4)	1.362(3)
C(6)-C(7)	1.393(2)	1.398(2)	1.386(4)	1.387(3)

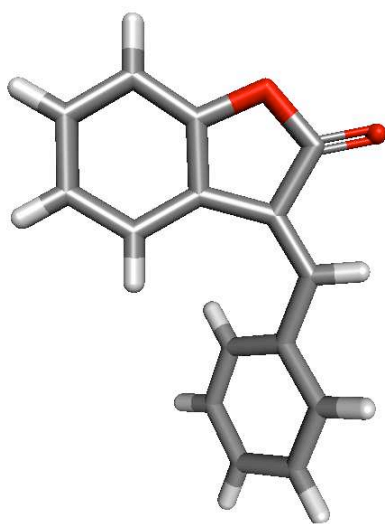


Figure 7b. 3-(1-Phenylmeth-(E)-ylidene)-3H-benzofuran-2-one $C_{21}H_{16}O_1$ (III)⁷ determined in the X-ray analysis (Drawn with Visualizer, Discovery Studio, Release 3.5, Accelrys Inc., 2012)³

[<http://accelrys.com/products/datasheets/whats-new-in-discovery-studio.pdf>].

Supplementary Material

Crystallographic data for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 804780. Copies of available material

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can be obtained, free of charge, via www.ccdc.cam.ac.uk/conts/retrieving.html or on application to the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-(0) 1223-336033 or e-mail: data request @ccdc.cam.ac.uk). X-ray diffraction data were measured at the UK National Crystallographic Service, Department of Chemistry, University of Southampton, Southampton SO17 1BJ, UK.²⁴

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