

**(Benzofuran-2-yl)(3-methyl-3-phenyl-
cyclobutyl)methanone**Çiğdem Yüksektepe,^{a*} Hanife Saraçoğlu,^a Murat Koca,^b
Alaaddin Çukurovalı^b and Nezihe Çalışkan^a^aDepartment of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Samsun, Turkey, and ^bDepartment of Chemistry, Faculty of Arts and Sciences, Firat University, 23119 Elazığ, Turkey

Correspondence e-mail: yuksek@yaho.com

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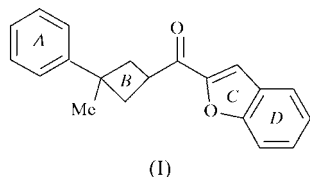
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The structure of the title compound, $C_{20}H_{18}O_2$, consists of a dimeric arrangement of benzofuran molecules around an inversion centre, linked *via* C—H \cdots O hydrogen bonds. There are also C—H \cdots π ring interactions. All these interactions result in the formation of infinite chains parallel to the [100] axis. The cyclobutane ring is puckered, with a dihedral angle of 29.03 (13) $^\circ$ between the two three-atom planes.

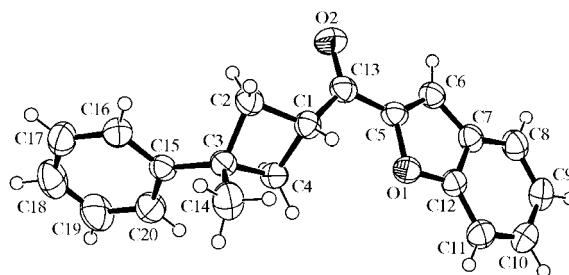
Comment

Benzofurans have attracted widespread interest in view of their biological activity, their presence in a large number of natural products and their potential as pharmacological agents. Consequently, diverse synthetic strategies have been developed to build this fused skeleton, commonly starting from a benzene ring with the appropriate substituents. A large number of syntheses of the heterocyclic moiety in benzofuran are based on the formation of O1—C5 or C5—C6 bonds (Fig. 1) as the ring-closure step (Cruz & Tamariz, 2004). In this paper, we report the molecular and crystal structure of the title benzofuran, (I).



The present crystal structure determination of (I) was carried out to determine the strength of the hydrogen-bonding capabilities of benzofuran and carbonyl, as well as of the C—H \cdots π interactions between the molecules. Compound (I) consists of cyclobutyl and benzofuran rings linked through a carbonyl group (Fig. 1). Selected bond lengths and angles in (I) are given in Table 1.

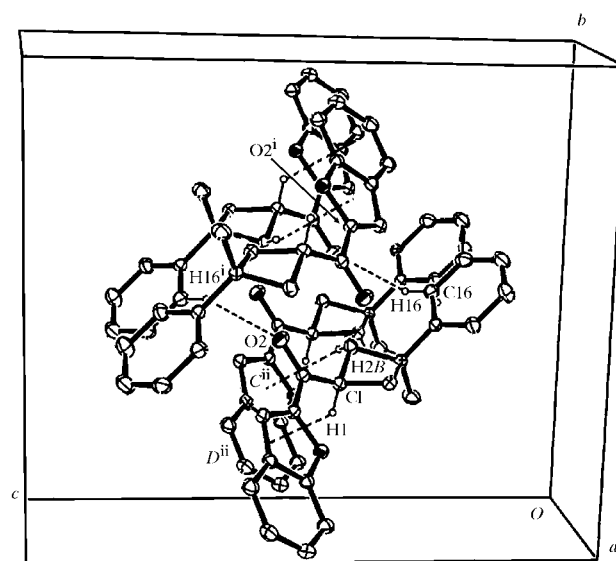
The dihedral angles between benzene ring A (C15—C20), cyclobutane ring B (C1—C4), furan ring C (O1/C5/C6/C7/C12)

**Figure 1**

A view of the molecule of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

and benzofuran benzene ring D (C7—C12) are *A/B* 32.74 (10), *A/C* 73.60 (6), *A/D* 73.66 (6), *B/C* 46.34 (10), *B/D* 45.81 (9) and *C/D* 1.64 (14) $^\circ$, *i.e.* rings C and D are nearly coplanar. The steric interaction between the substituent groups on the cyclobutane ring means that this ring deviates significantly from planarity. Literature values for the puckering of the cyclobutane ring are 23.5 (Swenson *et al.*, 1997) and 2.89 (37) $^\circ$ (Özdemir *et al.*, 2004a). In this study, the C4/C1/C2 plane forms a dihedral angle of 29.03 (13) $^\circ$ with the C2/C3/C4 plane, which deviates from the values reported in the previous studies. The geometry of the cyclobutane ring is due to the steric effect of the methyl group. The C2—C1—C13—C5 torsion angle is -177.46 (16) $^\circ$.

In this compound, atom H16 of the A ring acts as a donor, resulting in the formation of C—H \cdots O hydrogen bonds which link two molecules related by an inversion centre (Fig. 2). Of greater interest are the intermolecular π -ring interactions between the H atoms of the cyclobutane and the benzofuran rings, which contribute to the crystal packing by forming an infinite chain of dimers. These C—H \cdots π interactions, between

**Figure 2**

A diagram showing the hydrogen-bonding and π -ring interactions in (I). Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $x-1, y, z$].

rings *D* and *C* and cyclobutane atoms H1(C1) and H2B(C2) have the following geometric parameters: (i) the distance between atom H1 bonded to atom C1 and the centre, *M*, of ring *D*ⁱⁱ is 2.72 Å [symmetry code: (ii) *x* − 1, *y*, *z*] and the C1—H1···*M* angle is 135°; (ii) the distance between atom H2B bonded to atom C2 and the centre, *K*, of ring *C*ⁱⁱ is 2.78 Å and the C2—H2B···*K* angle is 142°.

The results obtained in this study indicate that there are some differences in the geometry of (I) compared with that of other compounds containing benzofuran or cyclobutane groups, such as those reported by Batsanov & Perepichka (2002) and Özdemir *et al.* (2004*b*). In one report, the crystal packing is mainly stabilized by intermolecular hydrogen bonds, which are highly effective in forming polymeric chains (Özdemir *et al.*, 2004*b*). From the results presented in this paper, it can be said that C—H···O hydrogen bonds link two molecules through an inversion centre to form a dimer, similar to what was found by Batsanov & Perepichka (2002). Besides these hydrogen bonds, there is an interesting π -ring interaction which stabilizes the crystal structure of (I). Dipole–dipole and van der Waals interactions are also effective in the molecular packing in the crystal structure.

Experimental

Salicylaldehyde (1.342 g, 11 mmol), potassium carbonate (2.07 g, 15 mmol) and acetonitrile (100 ml) were placed in a 500 ml two-necked flask fitted with a reflux condenser, and the mixture was stirred for 1 h at room temperature. To this solution, a solution of 1-phenyl-1-methyl-3-(2-chloro-1-oxoethyl)cyclobutane (2.225 g, 10 mmol) in acetonitrile (100 ml) was added dropwise over a period of about 30 min and the mixture refluxed for 4 h. The course of the reaction was monitored by IR spectroscopy. The solvent was then removed under reduced pressure, the residue extracted with diethyl ether and the ether phase dried over magnesium sulfate. After filtration and removal of the solvent under reduced pressure, compound (I) was crystallized from acetonitrile (yield 84%, m.p. 429–430 K).

Crystal data

C₂₀H₁₈O₂
M_r = 290.34
 Monoclinic, *P*2₁/*n*
a = 6.1497 (7) Å
b = 14.6894 (19) Å
c = 16.922 (2) Å
 β = 92.600 (10)°
V = 1527.1 (3) Å³
Z = 4
D_x = 1.263 Mg m^{−3}
 Mo *K*α radiation
 Cell parameters from 5297 reflections
 θ = 1.8–26.8°
 μ = 0.08 mm^{−1}
T = 293 (2) K
 Rod, colourless
 0.50 × 0.25 × 0.11 mm

Data collection

Stoe IPDS-II diffractometer
 ϕ scans
 11 652 measured reflections
 2685 independent reflections
 1602 reflections with *I* > 2 σ (*I*)
*R*_{int} = 0.117
 θ _{max} = 25.0°
h = −7 → 7
k = −17 → 17
l = −20 → 20

Refinement

Refinement on *F*²
R [*F*² > 2 σ (*F*²)] = 0.041
wR(*F*²) = 0.098
S = 0.83
 2685 reflections
 200 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0453P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.13 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.16 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

O1—C5	1.396 (2)	C3—C14	1.535 (2)
O2—C13	1.220 (2)	C5—C6	1.341 (2)
C1—C13	1.494 (2)	C5—C13	1.462 (3)
C3—C15	1.505 (2)		
C13—C1—C2	119.90 (16)	C6—C5—C13	132.83 (17)
C13—C1—C4	118.23 (15)	O1—C5—C13	116.25 (14)
C15—C3—C14	109.18 (14)	O2—C13—C5	119.06 (16)
C14—C3—C2	110.97 (16)	O2—C13—C1	122.99 (17)
C6—C5—O1	110.92 (15)	C5—C13—C1	117.95 (16)

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C16—H16···O2 ⁱ	0.93	2.59	3.390 (2)	145

Symmetry code: (i) 1 − *x*, 1 − *y*, 1 − *z*.

H atoms were placed in idealized positions, with C—H distances in the range 0.93–0.98 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C) (1.5*U*_{eq} for the methyl group).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1482). Services for accessing these data are described at the back of the journal.

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