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1-Methyl-1-phenyl-3-(phthalimidoacetyl)-cyclobutane

Namık Özdemir,[a] Muharrem Dinçer,[a]* Ibrahim Yılmaz[b] and Alaaddin Çukurovalı[b]

[a]Ondokuz Mayıs University, Arts and Sciences Faculty, Department of Physics, 55139-Samsun, Turkey, and [b]Fırat University, Arts and Sciences Faculty, Department of Chemistry, 23119-Elazığ, Turkey

Correspondence e-mail: mdincer@omu.edu.tr

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The title molecule, $C_{21}H_{19}NO_3$, consists of three essentially planar fragments. There is negligible puckering in the cyclobutane ring. The crystal structure is stabilized not only by weak intermolecular C—H···O hydrogen-bond interactions but also by weak C—H···π(ring) interactions, with an H···*Cg* distance of 2.85 Å (where *Cg* is the centroid of the phthalimido six-membered ring).

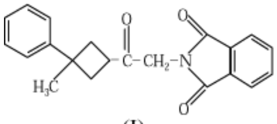
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.032
wR factor = 0.067
Data-to-parameter ratio = 7.1

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

Comment

The title compound, (I), contains cyclobutane and a phthalimide group. It is well known that 3-substituted cyclobutane carboxylic acid derivatives exhibit anti-inflammatory and antidepressant activities (Dehmlow & Schmidt, 1990), and liquid-crystal properties (Coghi *et al.*, 1976). Phthalimide derivatives exhibit anticancer activities, with current and potential applications including erythema nodosum leprosum, renal cell carcinoma, Kaposi's sarcoma and other cancers (Sampaio *et al.*, 1993; Eisen *et al.*, 2000). The structure of phthalimide was first reported by Matzat (1972). However, the synthesis and physiochemical properties of 1,1,3-trisubstituted cyclobutane and its phthalimide derivatives have not been reported so far. These compounds containing cyclobutane and phthalimide functions appear to be suitable candidates for further chemical modifications and may be pharmacologically active and useful ligands in coordination chemistry. The present paper deals with the preparation, characterization and crystal structure of a new material, (I), containing cyclobutane and phthalimide functions in the same molecule.



(I)

The structure of (I) (Fig. 1) contains three essentially planar fragments; a cyclobutane ring (C1–C4), a phthalimide group (O1/O2/N1/C5–C12) and a phenyl ring (C16–C21). The maximum deviations of the phthalimide group and the phenyl ring from planarity are 0.0520 (15) and 0.0061 (21) Å, respectively. The plane of the cyclobutane ring forms a dihedral angle of 64.34 (10)° with the plane of the phthalimide group and an angle of 47.69 (9)° with the plane of the phenyl ring bonded to atom C3 of the cyclobutane ring; the plane of the phthalimide group forms a dihedral angle of 46.14 (8)° with the plane of the phenyl ring. The four atoms of the C1—C14—C13—N1 bridge, linking the cyclobutane ring with the phthalimide group, form a torsion angle of 156.1 (2)°. The bond lengths and angles in the phthalimide group are close to the values reported in the literature (Zhu *et al.*, 2001).

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The lack of steric interaction between the substituent groups on the cyclobutane ring means that the ring does not deviate significantly from planarity. The deviations of the C1, C2, C3 and C4 cyclobutane atoms from planarity have the same magnitude [−0.014 (3) Å for atoms C1 and C3, and 0.014 (3) Å for atoms C2 and C4]. The C1—C2, C2—C3, C3—C4 and C1—C4 bond lengths are 1.553 (3), 1.550 (4), 1.557 (4) and 1.534 (4) Å, respectively, and the C1—C2—C3, C2—C3—C4, C3—C4—C1 and C4—C1—C2 bond angles are 90.15 (19), 89.22 (19), 90.59 (19) and 89.97 (19)°, respectively. Although the value for the puckering of the cyclobutane ring found in the literature is 23.5° (Swenson *et al.*, 1997), there is a negligible puckering in the cyclobutane ring. The C1—C2—C3 plane forms a dihedral angle of 2.91 (31)° with the C3—C4—C1 plane; the C4—C1—C2 plane forms a dihedral angle of 2.89 (37)° with the C2—C3—C4 plane.

There are two types of C—H···O hydrogen bonds in the structure. Atom H13*A* of the four-atom bridge (C13) and atom H2*A* of the cyclobutane ring form two intermolecular hydrogen bonds with the phthalimide group O atom (O2) of a symmetry-related molecule, and atom O2 acts as a single acceptor for both hydrogen bonds (Table 2). Hence, molecules are connected by the weak C—H···O hydrogen bonds, through 2_1 screw axes, to form extended chains in the *b* axis direction. There are no aromatic π–π stacking interactions in the crystal structure of (I). Arguably, the crystal structure also contains one significant C—H···π interaction (Table 2). For details of the C—H···O intermolecular hydrogen bonds and C—H···π interaction found in the crystal see Table 2.

Experimental

A mixture of 1-phenyl-1-methyl-3-(2-chloro-1-oxoethyl)cyclobutane (2.225 g, 0.01 mol), which was synthesized according to the method described by Akhmedov *et al.* (1991), triethylamine (1.0 g, 0.01 mol) and phthalimide (1.471 g, 0.01 mol) in acetonitrile was stirred for 4 h. After separation, by filtration, of the resulting triethylamine salt, acetonitrile was distilled under reduced pressure and the residue obtained was crystallized from ethanol. M.p. 429–430 K.

Crystal data

$C_{21}H_{19}NO_3$
$M_r = 333.37$
Orthorhombic, $P2_1nb$
$a = 9.1755$ (9) Å
$b = 9.4488$ (7) Å
$c = 19.7807$ (18) Å
$V = 1714.9$ (3) Å^3
$Z = 4$
$D_x = 1.291$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 11804 reflections
$\theta = 2.1$–29.6°
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Plate, colourless
0.80 × 0.38 × 0.06 mm

Data collection

Stoe IPDS-II diffractometer
φ scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
$T_{min} = 0.952$, $T_{max} = 0.993$
21 176 measured reflections
1611 independent reflections
1229 reflections with $I > 2\sigma(I)$
$R_{int} = 0.122$
$\theta_{max} = 25.0°$
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 11$
$l = -23 \rightarrow 23$

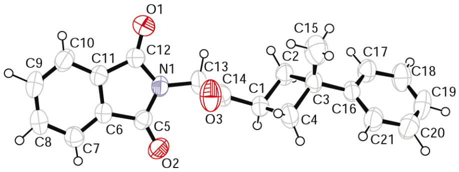

Figure 1
An *ORTEP*-3 (Farrugia, 1997) drawing of the title compound, (I), showing the atomic numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level.

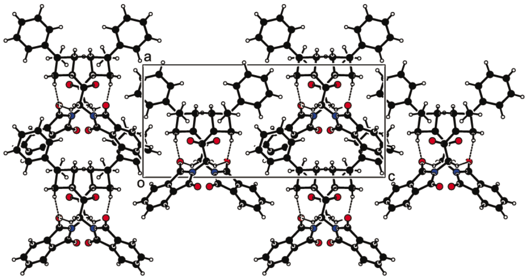

Figure 2
A *PLATON* plot (Spek, 1997), viewed down the *b* axis, illustrating the hydrogen-bonding interactions (shown as dashed lines) and the packing of molecules.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.067$
$S = 1.01$
1611 reflections
227 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0291P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.036$
$\Delta\rho_{max} = 0.11$ e Å^{-3}
$\Delta\rho_{min} = -0.12$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.031 (2)

Table 1
Selected geometric parameters (Å, °).

O1—C12	1.203 (3)	N1—C13	1.444 (3)
O2—C5	1.206 (3)	C1—C14	1.489 (4)
O3—C14	1.201 (3)	C3—C15	1.504 (4)
N1—C5	1.378 (3)	C3—C16	1.503 (3)
N1—C12	1.385 (3)		
C5—N1—C12	112.1 (2)	O2—C5—C6	129.1 (2)
C5—N1—C13	122.1 (2)	N1—C5—C6	106.4 (2)
C12—N1—C13	125.8 (2)	O1—C12—N1	125.2 (2)
C14—C1—C4	118.1 (2)	O1—C12—C11	129.5 (2)
C14—C1—C2	114.8 (2)	N1—C12—C11	105.3 (2)
C15—C3—C16	109.6 (2)	N1—C13—C14	113.3 (2)
C15—C3—C2	112.1 (2)	O3—C14—C1	124.8 (2)
C16—C3—C2	115.9 (2)	O3—C14—C13	120.6 (2)
C15—C3—C4	112.9 (2)	C1—C14—C13	114.6 (2)
C16—C3—C4	115.9 (2)	C21—C16—C3	122.4 (2)
O2—C5—N1	124.5 (2)	C17—C16—C3	120.4 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

*Cg*3 denotes the centroid of the C6–C11 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13*A*···O2^i	0.97	2.48	3.322 (3)	146
C2—H2*B*···O2^i	0.97	2.49	3.435 (3)	164
C17—H17···*Cg*3^{ii}	0.93	2.85	3.730 (3)	158

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

H atoms were positioned geometrically and treated using a riding model, fixing the C—H distances at 0.93 Å for both the phthalimide group and the phenyl ring, at 0.96 Å for the methyl group, at 0.97 Å for atoms C2, C4 and C13, and at 0.98 Å for atom C1. The displacement parameters of the H atoms were included as $U_{\rm iso}(H) = 1.2U_{\rm eq}$ ($1.5U_{\rm eq}$ for methyl) of the carrier atom.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *PLATON* (Spek, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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