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9a-Propargyl-1,2,3,9-tetrahydrofluorene-3,9-dione

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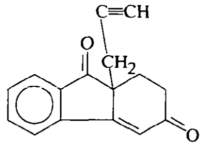
Abstract

In the title compound, $C_{16}H_{12}O_2$, the six-membered ring adopts a half-chair conformation and the fused five-membered ring is in a conformation between flattened sofa and half-chair. The propargyl residue is twisted diagonally towards the five-membered ring. In the solid state, the propargyl group is involved in C—H···O intermolecular hydrogen bonds to form an infinite one-dimensional chain along the *a* axis.

Comment

The title compound, (I), is a versatile building block for the total synthesis of tetracyclic diterpenes, namely gibbane (Takano *et al.*, 1981; Railton, 1982), which are potential plant-growth hormones especially involved in the growth of seedings. It may also be used to construct a complex molecular framework *via* radical cyclization (Rajagopal & Swaminathan, 1999). The present structural study was undertaken in order to elucidate the molecular conformation.



(I)

Bond distances and angles in the structure agree with reported values (Allen *et al.*, 1987; Lakshmi *et al.*, 1996). The six-membered ring (C1–C5, C13) adopts a half-chair conformation with the asymmetry parameter ΔC_2(C2—C1) = 8.6 (3)°; atoms C4 and C5 deviate from the mean plane by −0.318 (2) and 0.284 (2)°, respectively. The fused five-membered ring is in a conformation between flattened sofa and half-chair and it forms dihedral angles of 18.3 (1) and 5.9 (1)° with the adjacent rings. The plane of C5 and atoms C14, C15 and C16 of the propargyl residue is almost orthogonal [dihedral angle 89.5 (2)°] to the five-membered ring. In the crystal, the screw-related molecules are linked by C16—H16···O1($2-x$, $\frac{1}{2}+y$, $\frac{3}{2}-z$) hydrogen bonds to form an infinite one-dimensional chain along the *a* axis. The molecules translated along the *a* axis are interlinked by weak C14—H14*B*···O2($x+1$, y, z) hydrogen bonds to form a supramolecular structure which is further stabilized by very weak C11—H11···O1($2-x$, $1-y$, $1-z$) interactions between the inversion-related molecules.

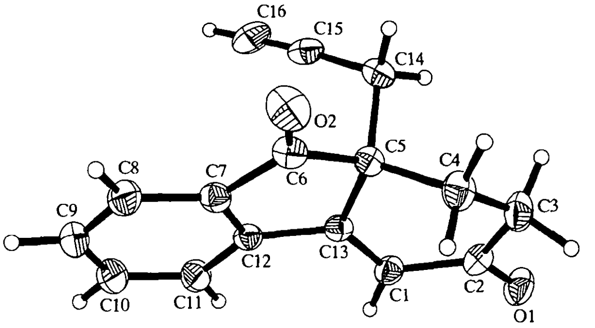


Fig. 1. The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

The title compound was prepared by the treatment of 2-propargyl-2-(3-oxobutyl)indan-1,3-dione (2.54 g, 0.01 mol) in dry ether (25 ml) with pyrrolidine (0.71 g, 0.01 mol) in acetic acid (0.6 g, 0.01 mol) under a nitrogen atmosphere at 273–278 K for 1 h. Ether was evaporated under reduced pressure and the resultant crude product was treated with a catalytic amount of *p*-toluenesulphonic acid in dry benzene (30 ml) under reflux conditions for 30 min. Benzene was evaporated under reduced pressure and the crude product was purified directly on a flash silica-gel column (200–400 mesh) using chloroform as an eluant and was crystallized from dry methanol at room temperature (m.p. = 429–430 K with 72–75% chemical yield).

Crystal data

$C_{16}H_{12}O_2$
M_r = 236.26
Monoclinic
$P2_1/c$
a = 6.4210 (2) Å
b = 16.6897 (3) Å
c = 11.7575 (3) Å
β = 98.738 (1)°
V = 1245.36 (5) Å^3
Z = 4
D_x = 1.260 Mg m^{-3}
D_m not measured

Mo *Kα* radiation
λ = 0.71073 Å
Cell parameters from 4571 reflections
θ = 2.14–28.27°
μ = 0.082 mm^{-1}
T = 293 (2) K
Plate
0.42 × 0.18 × 0.10 mm
Colourless

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Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
8184 measured reflections
3048 independent reflections
2243 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.035$
$\theta_{\rm max} = 28.27°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 22$
$l = 0 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.135$
$S = 1.119$
3048 reflections
212 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.3154P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.20$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 1997)
Extinction coefficient: 0.014 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C2	1.224 (2)	C5—C6	1.536 (2)
O2—C6	1.212 (2)	C5—C14	1.558 (2)
C1—C13	1.337 (2)	C6—C7	1.482 (2)
C1—C2	1.465 (3)	C7—C12	1.396 (2)
C2—C3	1.515 (3)	C12—C13	1.463 (2)
C3—C4	1.538 (3)	C14—C15	1.463 (3)
C4—C5	1.530 (2)	C15—C16	1.178 (3)
C5—C13	1.516 (2)		
C13—C1—C2—C3	11.6 (3)	C4—C5—C13—C1	−35.5 (2)
C1—C2—C3—C4	10.7 (3)	C13—C5—C14—C15	50.3 (2)
C2—C3—C4—C5	−43.4 (3)	C4—C5—C14—C15	172.57 (16)
C3—C4—C5—C13	53.7 (2)	C6—C5—C14—C15	−60.9 (2)
C2—C1—C13—C5	1.6 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16···O1$^{\rm i}$	0.94 (3)	2.37 (3)	3.252 (3)	156 (2)
C14—H14*B*···O2$^{\rm ii}$	0.99 (2)	2.48 (2)	3.423 (2)	160 (2)
C11—H11···O1$^{\rm iii}$	1.01 (2)	2.57 (2)	3.554 (2)	162 (2)

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

All H atoms were located from a difference map and were refined isotropically.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. 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* and *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1252). Services for accessing these data are described at the back of the journal.

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4-(4-Methylphenyl)-3-phenyl-4*H*-1,2,4-triazole

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Abstract

In the title compound, $C_{15}H_{13}N_3$, the triazole ring is planar and forms dihedral angles of 35.05 (5) and 65.44 (5)°, respectively, with the attached phenyl and methylphenyl rings. The crystal structure is stabilized by a number of C—H···π interactions and weak C—H···N short contacts.

Comment

Generally, 1,2,4-triazole derivatives are found to be associated with diverse pharmacological activity. Recently, some new triazole derivatives have been synthesized as possible anticonvulsants, antidepressants, tranquilizers and plant-growth regulators (Bradbury & Rivett,

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