Table 2. *Bond lengths (Å), bond angles (°) and torsion angles (°)*

C1—C2	1.566 (4)	C4—C16	1.530 (6)
C1—C5	1.519 (4)	C6—C7	1.319 (4)
C1—C11	1.538 (4)	C7—C8	1.476 (5)
C1—O19	1.434 (4)	C8—O17	1.204 (4)
C2—C3	1.556 (4)	C8—O18	1.341 (4)
C2—C6	1.503 (4)	C9—C10	1.476 (6)
C2—C14	1.529 (5)	C9—O18	1.466 (4)
C3—C4	1.545 (5)	C11—C12	1.469 (5)
C4—C5	1.546 (4)	C12—C13	1.165 (6)
C4—C15	1.524 (6)		
C11—C1—O19	108.9 (3)	C3—C4—C5	104.4 (3)
C5—C1—O19	110.4 (3)	C15—C4—C16	108.4 (4)
C5—C1—C11	114.0 (3)	C5—C4—C16	110.1 (3)
C2—C1—O19	106.1 (2)	C5—C4—C15	111.2 (3)
C2—C1—C11	112.8 (2)	C1—C5—C4	106.4 (2)
C2—C1—C5	104.3 (2)	C2—C6—C7	127.3 (3)
C1—C2—C14	113.1 (3)	C6—C7—C8	121.3 (3)
C1—C2—C6	108.6 (2)	C7—C8—O18	110.7 (3)
C1—C2—C3	101.5 (3)	C7—C8—O17	126.9 (3)
C6—C2—C14	112.0 (3)	O17—C8—O18	122.4 (3)
C3—C2—C14	112.0 (3)	C10—C9—O18	107.0 (3)
C3—C2—C6	109.0 (3)	C1—C11—C12	112.7 (3)
C2—C3—C4	108.9 (3)	C11—C12—C13	178.5 (5)
C3—C4—C16	111.9 (3)	C8—O18—C9	115.9 (3)
C3—C4—C15	110.8 (3)		
C1—C2—C3—C4	−24.5 (3)	C5—C1—C2—C6	−78.0 (3)
C2—C3—C4—C5	3.2 (4)	C11—C1—C2—C6	46.2 (4)
C2—C1—C5—C4	−36.4 (3)	C11—C1—C2—C14	−78.9 (4)
C5—C1—C2—C3	36.8 (3)	O19—C1—C2—C6	165.3 (3)
C3—C4—C5—C1	20.5 (3)	O19—C1—C2—C14	40.3 (4)
C2—C1—C11—C12	176.5 (3)	C14—C2—C6—C7	4.5 (5)

Table 3. *Hydrogen-bond parameters (Å, °)*

Data for the normalized H-atom position were based on bond lengths of O—H = 0.98 Å and ≡C—H = 1.08 Å.

D—H···O	D—H	H···O	D···O	H_{norm}···O	D—H_{norm}···O
O19—H···O17^i	0.78 (3)	2.10 (3)	2.874 (3)	1.90	175
C13—H···O17^{ii}	0.91 (4)	2.97 (4)	3.634 (5)	2.92	124

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

H atoms were refined isotropically, except those bonded to atoms C9 and C10. These did not refine realistically and were included in the model in their ideal positions (Sheldrick, 1976).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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1β-Hydroxy-1α-propargyl-2β-methyl-2-(2-ethoxycarbonylvinyl)cycloheptane

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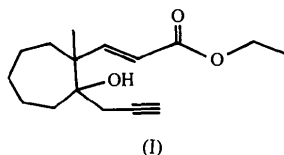*Institut für Kristallographie, Freie Universität Berlin, Takustraße 6, D-14159 Berlin, Germany*

Abstract

In the crystal structure of the title compound [ethyl 2-hydroxy-1-methyl-2-(2-propynyl)cycloheptane-1-prop-2-enoate, $C_{16}H_{24}O_3$] the hydroxy and the propargyl groups of neighbouring molecules form cooperative finite hydrogen-bonded chains, C≡C—H···O—H···O=C. In the C—H···O interaction, the H···O separation is 2.32 Å.

Comment

The terminal alkyne residue, C≡C—H, is among the most acidic C—H groups (Pedireddi & Desiraju, 1992) and is, therefore, an excellent model system for gaining insight into C—H···X hydrogen-bond interactions (Desiraju, 1991; Steiner, 1994). In the title compound, (I), the strongest hydrogen-bond donors are a hydroxy and a propargyl group. For these, three O atoms are available as good acceptors. Therefore, C ≡C—H···O hydrogen bonding can be expected to occur in the solid state.

(I)

The structure of the title compound is shown in Fig. 1. In the crystal, neighbouring molecules are connected by O—H···O and C—H···O hydrogen bonds, which form finite C≡C—H···O—H···O≡C chains (Fig. 2, Table 3). Such chains have the property of 'cooperativity', *i.e.* the hydrogen bonds enhance each other's strength by mutual polarization. This crystal structure is a good example of a case where the strongest donor

(O—H) forms a hydrogen bond with the strongest acceptor (C═O) and the second strongest donor (C≡C—H) forms a hydrogen bond with the second strongest acceptor (O—H) (see, for example, Etter, 1990). The weakest of the O acceptors (C—O17—C) cannot compete for the few available donors and must remain unsatisfied.

Experimental

The compound was synthesized by Wittig's reaction and crystallized from ethyl acetate. The density D_m was measured by flotation in water + KI.
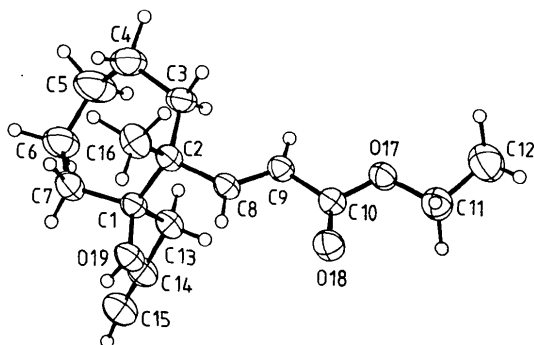

Fig. 1. Molecular structure and atomic numbering scheme of the title compound. Displacement ellipsoids are shown at the 30% probability level.
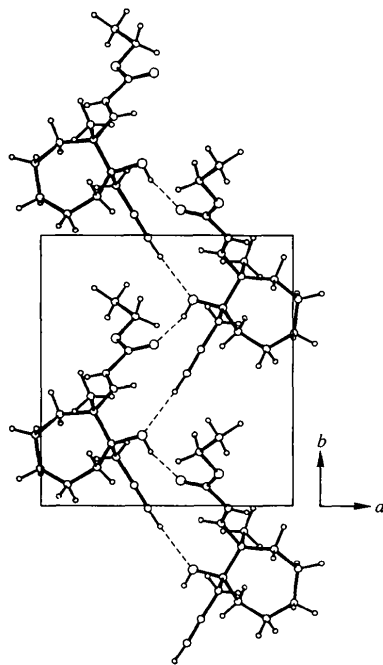

Fig. 2. Section of the crystal packing around $z = 0$; projection is along the c axis. Hydrogen bonds are shown as dashed lines.

Crystal data

$C_{16}H_{24}O_3$	Cu $K\alpha$ radiation
$M_r = 264.35$	$\lambda = 1.5418$ Å
Monoclinic	Cell parameters from 25
$P2_1/c$	reflections
$a = 11.483\,(2)$ Å	$\theta = 9$–$25°$
$b = 11.615\,(1)$ Å	$\mu = 0.616$ mm^{-1}
$c = 12.412\,(2)$ Å	$T = 295$ K
$\beta = 111.44\,(1)°$	Prismatic
$V = 1540.8\,(4)$ Å^3	$0.10 \times 0.10 \times 0.05$ mm
$Z = 4$	Colourless
$D_x = 1.142$ Mg m^{-3}	
$D_m = 1.137$ Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4 diffractometer	1637 observed reflections $[F > 2\sigma(F)]$
ω scans	$R_{int} = 0.031$
Absorption correction:	$\theta_{max} = 60°$
refined from ΔF	$h = -12 \rightarrow 0$
(*DIFABS*; Walker &	$k = 0 \rightarrow 13$
Stuart, 1983)	$l = -12 \rightarrow 13$
2387 measured reflections	3 standard reflections
2263 independent reflections	frequency: 60 min
	intensity decay: 6.5%

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.07$
$R = 0.072$	$\Delta\rho_{max} = 0.35$ e Å^{-3}
$wR = 0.072$	$\Delta\rho_{min} = -0.27$ e Å^{-3}
$S = 0.99$	Atomic scattering factors from *SHELX*76
1637 reflections	(Sheldrick, 1976)
250 parameters	
Unit weights applied	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1	0.7225 (4)	−0.2686 (4)	−0.1878 (4)	0.057 (4)
C2	0.7942 (4)	−0.1508 (4)	−0.1579 (4)	0.061 (4)
C3	0.9261 (4)	−0.1624 (4)	−0.0633 (5)	0.073 (4)
C4	1.0224 (5)	−0.2364 (5)	−0.0901 (6)	0.098 (5)
C5	1.0048 (5)	−0.3615 (5)	−0.0863 (7)	0.119 (5)
C6	0.8962 (5)	−0.4211 (5)	−0.1702 (5)	0.098 (5)
C7	0.7818 (5)	−0.3567 (4)	−0.2451 (4)	0.072 (4)

C8	0.7145 (4)	−0.0678 (4)	−0.1208 (4)	0.056 (4)
C9	0.7439 (4)	−0.0106 (4)	−0.0240 (4)	0.058 (4)
C10	0.6550 (4)	0.0710 (4)	−0.0039 (4)	0.061 (4)
C11	0.6267 (5)	0.1995 (5)	0.1327 (5)	0.091 (5)
C12	0.7073 (7)	0.2541 (6)	0.2428 (6)	0.129 (5)
C13	0.7044 (4)	−0.3176 (4)	−0.0803 (4)	0.064 (4)
C14	0.6330 (4)	−0.4242 (4)	−0.0997 (4)	0.069 (4)
C15	0.5776 (5)	−0.5110 (5)	−0.1133 (5)	0.086 (5)
C16	0.8062 (5)	−0.0955 (5)	−0.2674 (5)	0.089 (5)
O17	0.7045 (3)	0.1180 (3)	0.1014 (3)	0.067 (3)
O18	0.5526 (3)	0.0940 (3)	−0.0721 (3)	0.077 (3)
O19	0.6013 (3)	−0.2392 (3)	−0.2716 (3)	0.065 (3)

Table 2. *Bond lengths* (Å)

C2—C1	1.570 (6)	C7—C6	1.502 (7)
C7—C1	1.538 (6)	C9—C8	1.305 (6)
C13—C1	1.535 (6)	C10—C9	1.478 (6)
O19—C1	1.442 (5)	O17—C10	1.337 (5)
C3—C2	1.545 (6)	O18—C10	1.201 (5)
C8—C2	1.513 (6)	C12—C11	1.483 (8)
C16—C2	1.554 (6)	O17—C11	1.448 (5)
C4—C3	1.531 (7)	C14—C13	1.455 (6)
C5—C4	1.470 (8)	C15—C14	1.171 (6)
C6—C5	1.473 (7)		

Table 3. *Hydrogen-bond parameters* (Å, °)

D—H···O	D—H	H···O	D···O	H$_{norm}$···O	D—H$_{norm}$···O
O19—H···O18^i	0.79 (4)	2.12 (4)	2.852 (5)	1.96	151
C15—H···O19^i	0.95 (4)	2.47 (4)	3.338 (7)	2.32	157

Symmetry code: (i) $1 - x, y - \frac{1}{2}, -\frac{1}{2} - z$.

As the crystal was very small, only diffraction data of moderate quality could be obtained. H atoms were refined isotropically, except for those bonded to C4, C5 and C6. These did not refine realistically and were included in the model in their ideal positions (Sheldrick, 1976).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances involving H atoms and bond angles involving non-H atoms have been deposited with the IUCr (Reference: DE1006). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3,3-(Oxydiethyl)-1,2-diphenylguanidine

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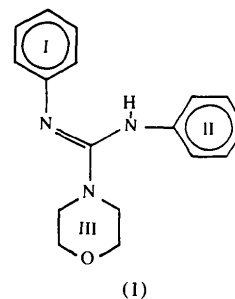
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Abstract

The title molecule, 2-morpholino-1,3-diphenyl-1,3-diazapropene, $C_{17}H_{19}N_3O$, contains two phenyl rings joined to a guanidine skeleton that incorporates a morpholine ring. The morpholine ring adopts a chair conformation. All substituent rings are tilted out of the guanidine plane; the aromatic rings are almost perpendicular to each other. Crystal cohesion is due to van der Waals interactions and an intermolecular N—H···O hydrogen bond.

Comment

The title compound, (1), which was synthesized at the SPIC Science Foundation, Madras, belongs to a class of substances known for their pharmacological activity. Several cyclic guanidines are used to treat high blood pressure, and some are potent antidiabetic agents (Maryanoff, 1985). The molecular geometry and the atomic numbering scheme are shown in Fig. 1.

(1)

The morpholine ring is in a chair conformation (Fig. 1). The pyramidalization of the N atom in a morpholine ring varies through a wide range, with the sum of angles at N reported to be between 337 and 359° (Wong-Ng, Nyburg, Awwal, Jankie & Kresge, 1982). In the present case, the sum of the angles at