1-(Phenylethynyl)adamantane

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Key indicators

Single-crystal X-ray study
$T = 296$ K
Mean σ(C–C) = 0.006 Å
R factor = 0.057
wR factor = 0.194
Data-to-parameter ratio = 14.5

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

The title compound, $C_{18}H_{20}$, provides the first structurally characterized example of an alkynyl-substituted adamantane free from cocrystallized solvent or guest molecules.

Comment

Due to the increasing number of nano-machines (Balzani *et al.*, 2000; Kelly, 2001) and nano-devices (Rukavishnikov *et al.*, 1999) containing acetylene units attached to bulky caged carbocyles, we have been investigating new routes to achieving one-step synthesis of such systems. Recently, we discovered that the title compound, (I), could be obtained, *via* a novel metal-mediated process, from phenylacetylene and 1-iodoadamantane (Williams & Raine, 2002). It comprises adamantyl and phenyl groups bridged by a single acetylene residue.

(I)

(II)
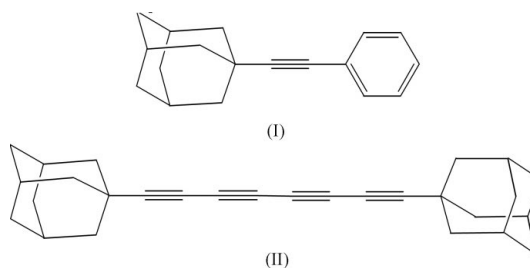
Although structurally characterized examples of substituted adamantanes abound (more than 150 examples in the Cambridge Structural Database; Allen, 2002), there is but one molecule where the substituent is an alkyne attached to the bridgehead C atom. This compound, namely the symmetrical dumb-bell-shaped molecule 1,8-bis(1-adamantyl)-1,3,5,7-octatetrayne, (II), has been found to exhibit a number of interesting structural and non-linear optical properties. Significantly, all structures of (II) have been found to include a cocrystallized guest molecule, such as 2-butanone, crocetin dialdehyde, cyclohexanol or *trans*-β-8′-apocarotenal (Müller *et al.*, 2000). It was found that (II) exhibits a distinctly bowed conformation of its nominally linear tetra-yne moiety, and the cocrystallized guest molecule appears to have an important influence on the observed conformation of its host (II). We were interested in the conformation of the simple analogue (I), which bears the same ethynyladamantyl group and, to this end, we have been successful in crystallizing (I) in a solvent-free form.

Compound (I) comprises adamantyl and phenyl groups bridged by a single acetylene residue. There is little apparent strain in the molecule and the bond lengths (Table 1) are as expected for a compound of its type. The adamantyl (1.50–1.54 Å) and phenyl C—C bond lengths (1.36–1.40 Å) are typical for these groups. The (adamantyl)C1—C11—C12 and
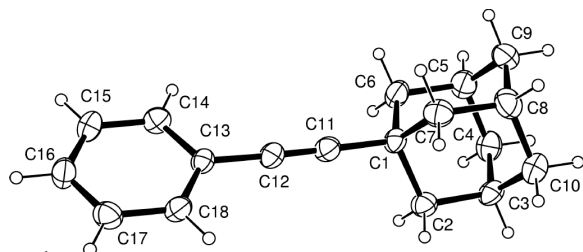
Figure 1
ORTEP-3 plot (Farrugia, 1997) of (I), with ellipsoids at the 30% probability level.

C11—C12—C13(phenyl) angles (Table 1) are both close to linear, although the latter exhibits a small deviation therefrom. There are no hydrogen-bonding interactions in hydrocarbon (I). The closest intermolecular contacts are $H9B\cdots H18(-x + 1, y - \frac{1}{2}, -z + \frac{1}{2})$ (2.44 Å), $H2A\cdots H14(x, -y + \frac{1}{2}, z + \frac{1}{2})$ (2.53 Å) and $H6B\cdots H7A(-x + 1, -y, -z)$ (2.53 Å).

In conclusion, in the absence of any cocrystallized guest molecule, we have found that the nominally rod-shaped (I) exhibits an essentially undistorted conformation.

Experimental

The synthesis of the title compound will be reported separately (Williams & Raine, 2002). Crystals were obtained by slow evaporation of a petroleum spirit (313–333 K) solution of the compound.

Crystal data

$C_{18}H_{20}$	$D_x = 1.157$ Mg m^{-3}
$M_r = 236.34$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25
$a = 9.050 (1)$ Å	reflections
$b = 14.224 (1)$ Å	$\theta = 9.8–13.9°$
$c = 11.081 (1)$ Å	$\mu = 0.07$ mm^{-1}
$\beta = 107.986 (9)°$	$T = 296 (2)$ K
$V = 1356.7 (2)$ Å^3	Prism, colourless
$Z = 4$	$0.50 \times 0.27 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4	$R_{int} = 0.028$
diffractometer	$\theta_{max} = 25.0°$
ω–2θ scans	$h = 0 \rightarrow 10$
Absorption correction: ψ scan	$k = 0 \rightarrow 16$
(North *et al.*, 1968)	$l = -13 \rightarrow 12$
$T_{min} = 0.911, T_{max} = 0.991$	3 standard reflections
2542 measured reflections	frequency: 120 min
2385 independent reflections	intensity decay: 4%
1381 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.038P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.057$	$+ 2.0561P]$
$wR(F^2) = 0.194$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.15$	$(\Delta/\sigma)_{max} < 0.001$
2385 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
164 parameters	$\Delta\rho_{min} = -0.17$ e Å^{-3}
H-atom parameters constrained	Extinction correction: *SHELXL*
	Extinction coefficient: 0.023 (3)

Table 1
Selected geometric parameters (Å, °).

C1—C11	1.477 (5)	C8—C9	1.523 (6)
C1—C6	1.534 (5)	C8—C10	1.524 (6)
C1—C7	1.541 (5)	C11—C12	1.190 (5)
C1—C2	1.542 (5)	C12—C13	1.433 (5)
C2—C3	1.523 (5)	C13—C18	1.393 (5)
C3—C10	1.506 (6)	C13—C14	1.398 (5)
C3—C4	1.517 (6)	C14—C15	1.379 (5)
C4—C5	1.524 (6)	C15—C16	1.369 (6)
C5—C9	1.521 (6)	C16—C17	1.376 (6)
C5—C6	1.530 (6)	C17—C18	1.380 (5)
C7—C8	1.521 (6)		
C11—C1—C6	110.0 (3)	C8—C7—C1	110.2 (3)
C11—C1—C7	109.8 (3)	C7—C8—C9	109.5 (4)
C6—C1—C7	109.0 (3)	C7—C8—C10	109.8 (4)
C11—C1—C2	111.5 (3)	C9—C8—C10	109.2 (4)
C6—C1—C2	108.4 (3)	C5—C9—C8	109.4 (4)
C7—C1—C2	108.1 (3)	C3—C10—C8	109.4 (4)
C3—C2—C1	109.4 (3)	C12—C11—C1	179.0 (4)
C10—C3—C4	110.0 (4)	C11—C12—C13	177.8 (5)
C10—C3—C2	110.1 (3)	C18—C13—C14	117.8 (3)
C4—C3—C2	110.3 (3)	C18—C13—C12	121.5 (3)
C3—C4—C5	108.8 (3)	C14—C13—C12	120.7 (4)
C9—C5—C4	110.0 (4)	C15—C14—C13	120.9 (4)
C9—C5—C6	109.9 (4)	C15—C16—C17	119.9 (4)
C4—C5—C6	108.8 (4)	C16—C17—C18	120.3 (4)
C5—C6—C1	110.1 (3)	C17—C18—C13	120.7 (4)

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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