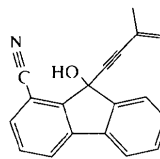


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(1)

The C15—C16 bond distance in the title molecule [1.191 (2) Å] is in agreement with those of related alkyne compounds: 3,3-diphenyl-1-propyn-3-ol [1.169 (2) Å; Garcia *et al.*, 1995], 1,1-bis(2,4-dimethylphenyl)-2-propyn-1-ol, 1,1-bis(2,4-dimethylphenyl)-2-butyn-1-ol and 9-hydroxy-9-(1-propynyl)fluorene [1.208 (5), 1.189 (6) and 1.16 (2) Å, respectively; Toda *et al.*, 1985], 1,6,9,14-tetrahydroxy-1,6,9,14-tetra-*tert*-butylcyclohexadeca-2,4,7,10,12,15-hexayne [1.168 (6) Å; Toda *et al.* 1988], and also with those of related alkyne compounds: phenyl-(2-trimethylsilylethynyl)-phenylmethanone and (2-trimethylsilylethynyl)benzaldehyde [1.199 (3) and 1.192 (3) Å, respectively; Garcia *et al.*, 1996], 2,3,5,6-tetra[1-ethynyl-2(trimethylsilyl)]pyridine [1.190 (4) and 1.191 (4) Å; Garcia *et al.*, 1997], 2,3,5,6-tetra[1-ethynyl-2-(trimethylsilyl)]pyrazine [1.198 (5) and 1.199 (6) Å; Garcia *et al.*, 1997], 2,3,4,5-tetra[1-ethynyl-2-(trimethylsilyl)]thiophene [1.190 (4) and 1.190 (4) Å; Garcia *et al.*, 1997], 3,4-dibromo-2,5-di[1-ethynyl-2-(trimethylsilyl)]-*N*-methylpyrrole [1.189 (5) and 1.194 (4) Å; Garcia *et al.*, 1997]. The C14—N1 bond in the title molecule [1.143 (2) Å] is similar in length to that of 2-cyanobenzophenone [1.130 (5) Å; Preut *et al.*, 1992].

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## 9-Hydroxy-9-(3-methylbut-3-en-1-ynyl)-9H-fluorene-1-carbonitrile

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### Abstract

The N atom in the title compound, C<sub>19</sub>H<sub>13</sub>NO, lies −0.0365 (14) Å from the plane formed by the fluorene system, and the C≡N distance is 1.143 (2) Å. The C≡C distance is 1.191 (2) Å, and the ethynyl group deviates slightly from linearity, with C—C≡C—C bond angles of 172.0 (1) and 176.1 (2)°. Terminal CH<sub>2</sub> and CH<sub>3</sub> groups are 50:50 disordered, with equal C—C distances of 1.408 (3) and 1.406 (3) Å.

### Comment

The determination of the crystal structure of the title compound, (I), was undertaken as part of an on-going program of structure analyses of some novel intermediates in the synthesis of potentially bio-active compounds bearing a conjugated acetylenic functionality. The nitrile unit is nearly coplanar with the fluorene ring system; the torsion angles N1—C14—C3—C2, N1—C14—C3—C4 and C14—C3—C2—C1 are 162.3 (3), −17.1 (3) and 0.9 (2)°, respectively.

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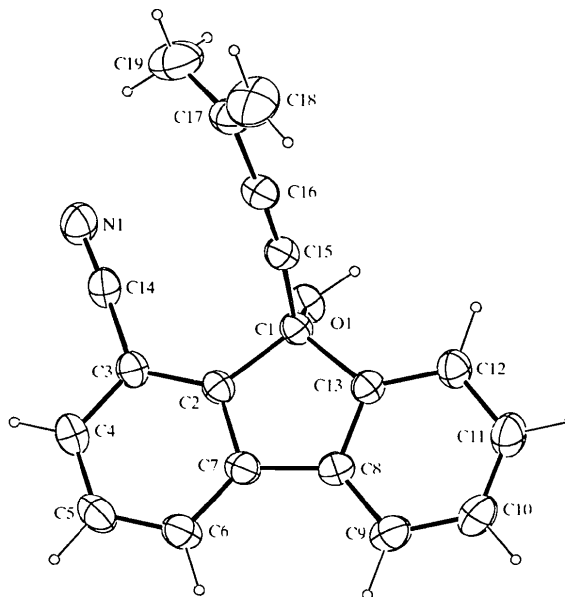


Fig. 1. The title molecule with ellipsoids drawn at the 30% probability level and H atoms shown with arbitrary radii. Only one set of the partially populated H-atom positions is illustrated for C18 and C19.

There is an intermolecular O—H...N interaction, with an N1—H1OH distance of 2.06 (2) Å, forming an angle with O1 of 171 (1)°. The OH group and nitrile N atom are involved in an intermolecular hydrogen bond, forming infinite chains in the *b* direction. The H1...N1(−*x*, *y* − ½, ½ − *z*) distance is 2.9479 (14) Å and the angle about H is 171 (2)°.

The C17—C18 and C17—C19 distances are equal and intermediate between those of single and double bonds.

## Experimental

The title compound was prepared by reacting the lithium salt of 2-methyl-3-buten-1-yne with 1-cyanofluorenone (Huntress *et al.*, 1942).

### Crystal data

C<sub>19</sub>H<sub>13</sub>NO  
*M<sub>r</sub>* = 271.32  
 Monoclinic  
*P*2<sub>1</sub>/c  
*a* = 8.5961 (3) Å  
*b* = 10.3670 (6) Å  
*c* = 16.506 (1) Å  
 $\beta$  = 98.76 (1)°  
*V* = 1453.8 (3) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.240 Mg m<sup>−3</sup>  
*D<sub>m</sub>* not measured

### Data collection

Enraf–Nonius CAD-4  
 diffractometer  
 $\theta/2\theta$  scans  
 Absorption correction:  
 $\psi$  scans (North *et al.*,  
 1968)  
*T<sub>min</sub>* = 0.849, *T<sub>max</sub>* = 0.892  
 3377 measured reflections  
 2989 independent reflections

### Refinement

Refinement on *F*  
*R* = 0.038  
*wR* = 0.051  
*S* = 2.580  
 2618 reflections  
 223 parameters  
 H atoms: mixed  
 $w = 4F_o^2 / [\sigma^2(F_o^2) + 0.0004F_o^4]$   
 $(\Delta/\sigma)_{\max} = 0.026$

Cu *K*α radiation  
 $\lambda = 1.54184$  Å  
 Cell parameters from 25  
 reflections  
 $\theta = 19$ – $42^\circ$   
 $\mu = 0.569$  mm<sup>−1</sup>  
*T* = 296 K  
 Fragment  
 0.42 × 0.30 × 0.20 mm  
 Yellow

2618 reflections with  
 $I > \sigma(I)$   
*R<sub>int</sub>* = 0.012  
 $\theta_{\max} = 75^\circ$   
 $h = 0 \rightarrow 10$   
 $k = -12 \rightarrow 0$   
 $l = -20 \rightarrow 20$   
 3 standard reflections  
 frequency: 120 min  
 intensity decay: 0.15%

$\Delta\rho_{\max} = 0.17$  e Å<sup>−3</sup>  
 $\Delta\rho_{\min} = -0.15$  e Å<sup>−3</sup>  
 Extinction correction:  
 isotropic (Zachariasen,  
 1963)  
 Extinction coefficient:  
 $6.60 (12) \times 10^{-6}$   
 Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

O1—C1—C15	110.09 (9)	C15—C16—C17	176.1 (2)
C2—C1—C13	101.01 (8)	C16—C17—C18	119.4 (1)
N1—C14—C3	177.0 (1)	C16—C17—C19	117.3 (1)
C1—C15—C16	172.0 (1)	C18—C17—C19	123.3 (1)

The methyl and methylene groups at C18 and C19 are 50:50 disordered, thus H atoms H18*a* and H19*a* are fully populated, while other H atoms on C18 and C19 are half-populated. All H atoms on C18 and C19 were placed in calculated positions with *B<sub>iso</sub>* = 10 Å<sup>2</sup>. C—H distances for refined H atoms are in the range 0.95 (2)–1.03 (1) Å, while *B<sub>iso</sub>* values for H atoms are in the range 5.5 (3)–8.4 (4) Å<sup>2</sup>. Attempts to model the disorder with C18 and C19 in partially populated positions were not successful in improving the fit to the data. The density associated with these two atoms is not usually elongated in the direction of the central atom (C17). The ellipsoid for C17, however, is elongated approximately perpendicular to the C16—C17 bond, in the trigonal plane. This indicates that C17 probably also exhibits unresolved disorder in which the superposition of the single and double bonds to C18 and C19 is accompanied by an in-plane shift of C17 without large shifts in the terminal atoms. The *R* value based on all 2989 reflections is 0.046. The slightly high value of the goodness-of-fit probably results from the disorder.

Data collection: *CAD-4 Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD-4 Operations Manual*. Data reduction: *PROCESS in MolEN* (Fair, 1990). Program(s) used to solve structure: *MULTAN80* (direct methods; Main *et al.*, 1980). Program(s) used to refine structure: *LSFM in MolEN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *CIF-IN in MolEN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1044). Services for accessing these data are described at the back of the journal.

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Table 1. Selected geometric parameters (Å, °)

O1—C1	1.430 (1)	C17—C18	1.408 (3)
N1—C14	1.143 (2)	C17—C19	1.406 (3)
C15—C16	1.191 (2)		

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### *m*-(*p*-Tolylsulfonyloxy)aniline

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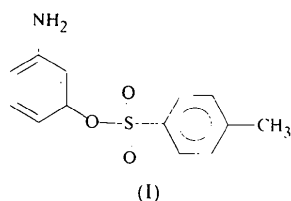
(Received 8 December 1997; accepted 21 January 1998)

#### Abstract

In the title compound, 3-aminophenyl 4-toluenesulfonate, C<sub>13</sub>H<sub>13</sub>NO<sub>3</sub>S, the dihedral angle between the toluene and aniline moieties is 64.26(5)°. The crystal structure is stabilized by N—H···O intermolecular hydrogen bonds involving amino and sulfonyloxy groups.

#### Comment

The title compound, (I), is potentially biologically active in mimicking enzyme activity in living organisms. It is expected to show supramolecular behaviour.



The detailed X-ray structure analysis was undertaken to study the molecular conformation and intermolecular hydrogen-bonding scheme. The S—O and N—C distances, and the mean value of the C—C distances [1.381(3) Å], agree with the reported values (Allen *et al.*, 1987). The S atom is tetrahedral. The toluene and aniline moieties have a dihedral angle of 64.26(5)° between them.

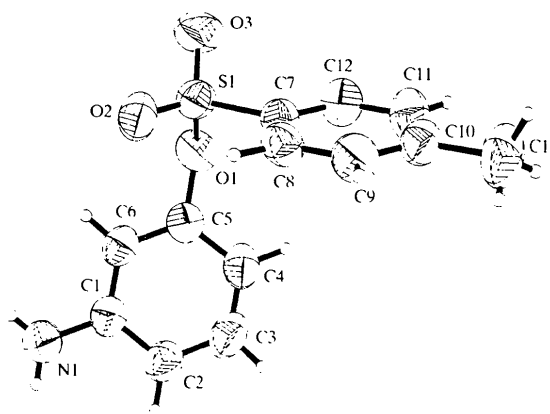


Fig. 1. SHELXTL/PC (Sheldrick, 1990) plot of the structure of (I), showing displacement ellipsoids at the 50% probability level and the atom-numbering scheme.

#### Experimental

To prepare the title compound, *p*-toluenesulfonyl chloride (1 equivalent) was added to a solution of 3-amino-1-hydroxybenzene (1 equivalent) and triethylamine in dry CH<sub>2</sub>Cl<sub>2</sub> at 273 K. The reaction mixture was stirred for 15 min at 273 K and 45 min at room temperature. It gave the desired compound in 90% yield (Kurita, 1974). The unreacted compounds were removed by column chromatography, producing a pure sample of (I). Single crystals were grown by slow evaporation of a 1:1 dichloromethane–petroleum ether solution of the compound.

#### Crystal data

C<sub>13</sub>H<sub>13</sub>NO<sub>3</sub>S  
*M<sub>r</sub>* = 263.30  
Monoclinic  
*P*2<sub>1</sub>/*c*  
*a* = 9.9807(7) Å  
*b* = 7.5585(6) Å  
*c* = 17.0244(12) Å  
*β* = 90.196(7)°  
*V* = 1284.3(2) Å<sup>3</sup>  
*Z* = 4  
*D<sub>s</sub>* = 1.362 Mg m<sup>-3</sup>  
*D<sub>n</sub>*, not measured

Mo *Kα* radiation  
*λ* = 0.71073 Å  
Cell parameters from 40 reflections  
*θ* = 5.41–12.58°  
*μ* = 0.251 mm<sup>-1</sup>  
*T* = 293(2) K  
Rectangular  
0.65 × 0.42 × 0.22 mm  
Colourless

#### Data collection

Siemens *P4* diffractometer  
*θ*/2*θ* scans

*R*<sub>int</sub> = 0.028  
*θ*<sub>max</sub> = 27.50°

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