| | | | | |
|---|---|---|---|---|
| N23 | 0.9058 (4) | −0.1712 (5) | 0.5722 (4) | 0.044 (2) |
| N24 | 1.0670 (4) | −0.3490 (5) | 0.5716 (4) | 0.051 (3) |
| C11 | 0.6861 (5) | −0.1047 (7) | 0.6797 (5) | 0.056 (4) |
| C12 | 0.7472 (4) | 0.0238 (6) | 0.5073 (4) | 0.037 (3) |
| C14 | 0.6435 (5) | 0.2172 (7) | 0.4909 (6) | 0.053 (4) |
| C15 | 0.6167 (5) | 0.1378 (6) | 0.5819 (5) | 0.044 (3) |
| C21 | 0.7958 (6) | −0.0199 (7) | 0.2468 (5) | 0.058 (4) |
| C22 | 0.8340 (5) | −0.0816 (6) | 0.4853 (5) | 0.036 (3) |
| C24 | 0.9724 (4) | −0.2426 (6) | 0.5079 (5) | 0.037 (3) |
| C25 | 0.9446 (5) | −0.2011 (6) | 0.3841 (5) | 0.043 (3) |

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| O151—N15 | 1.223 (8) | N21—C21 | 1.477 (7) |
| O152—N15 | 1.218 (7) | N21—C22 | 1.377 (7) |
| O241—N24 | 1.235 (7) | N21—C25 | 1.363 (8) |
| O242—N24 | 1.226 (6) | N23—C22 | 1.324 (7) |
| N11—C11 | 1.467 (7) | N23—C24 | 1.351 (7) |
| N11—C12 | 1.352 (7) | N24—C24 | 1.448 (6) |
| N11—C15 | 1.377 (7) | C12—C22 | 1.453 (8) |
| N13—C12 | 1.333 (7) | C14—C15 | 1.363 (9) |
| N13—C14 | 1.356 (8) | C24—C25 | 1.351 (7) |
| N15—C15 | 1.433 (8) | | |
| C11—N11—C12 | 127.2 (5) | N11—C12—N13 | 112.9 (5) |
| C11—N11—C15 | 128.7 (5) | N11—C12—C22 | 125.2 (5) |
| C12—N11—C15 | 104.1 (4) | N13—C12—C22 | 121.9 (6) |
| C12—N13—C14 | 105.7 (5) | N13—C14—C15 | 108.7 (5) |
| O151—N15—O152 | 124.8 (6) | N11—C15—N15 | 124.9 (5) |
| O151—N15—C15 | 115.3 (5) | N11—C15—C14 | 108.7 (5) |
| O152—N15—C15 | 119.9 (5) | N15—C15—C14 | 126.4 (5) |
| C21—N21—C22 | 129.5 (5) | N21—C22—N23 | 111.2 (5) |
| C21—N21—C25 | 123.4 (5) | N21—C22—C12 | 122.9 (4) |
| C22—N21—C25 | 107.2 (5) | N23—C22—C12 | 125.8 (5) |
| C22—N23—C24 | 103.7 (4) | N23—C24—N24 | 121.2 (4) |
| O241—N24—O242 | 124.6 (4) | N23—C24—C25 | 113.6 (5) |
| O241—N24—C24 | 117.5 (4) | N24—C24—C25 | 125.3 (5) |
| O242—N24—C24 | 118.0 (5) | N21—C25—C24 | 104.4 (5) |

The structure was solved by direct methods and refined by a full-matrix least-squares procedure. Non-H atoms were refined with anisotropic displacement parameters. H atoms were obtained from a difference map and included in the structure-factor calculations with $B_{\rm iso} = 4.0$ Å$^2$ fixed.

Data collection: *Enraf–Nonius CAD-4 Software* (Enraf–Nonius, 1989). Data reduction: *SDP*-VAX, Version 3.0 (1986) (Frenz, 1978). Program used to solve structure: *SHELXS*86, VMS version (Sheldrick, 1985). Program used to refine structure: *SDP*-VAX. Molecular graphics: *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HR1036). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1995). **C51**, 1416–1418

# 6-(2-Pyridyl)-3,5-hexadiyn-1-ol

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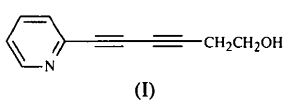
## Abstract

The crystal structure of 6-(2-pyridyl)-3,5-hexadiyn-1-ol (PyHxD), $C_{11}H_9NO$, has been determined by a single-crystal X-ray diffraction study. The backbone diyne chain linking the pyridyl ring and the C(10)—C(11)—O group is linear. A dimer with a centre of symmetry is formed through two O—H···N hydrogen bonds and the crystal structure indicates that solid-state polymerization is inhibited because of these hydrogen bonds.

## Comment

There is a growing interest in polydiacetylenes (PDAs) from both basic and applicational viewpoints. These polymers are of particular interest because of their conducting and nonlinear optical properties. Some diacetylenes ($R$—C≡C—C≡C—$R'$) undergo solid-state polymerization upon heating, irradiation or mechanical stimulation. The polymerization reactivity of diacetylenes in the crystalline state is governed by the molecular packing of the monomer (Barrow, Milburn & Zeng, 1994). The title compound, (I), was synthesized in order to investigate the effect of heteroaryl side groups conjugated to the diyne system on the crystal structure and polymerization reactivity of the crystal. In general, strong intermolecular hydrogen bonds between stacking monomers suppress the polymerization reactivity. But, the newly synthesized monomer single crystal reported here shows a somewhat differently stacked form suggesting the possibility of polymerization if strong hydrogen bonds are cleaved during the polymerization.



(I)

The conformation of the single crystal was investigated by X-ray diffraction techniques. Fig. 1 shows the molecular conformation with the atomic labelling scheme (*ORTEP*II; Johnson, 1976) and Fig. 2 shows the unit-cell packing.

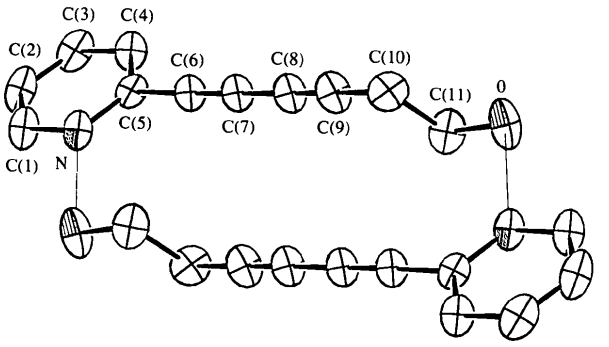


Fig. 1. *ORTEP*II (Johnson, 1976) view of the title molecule (PyHxD) showing the atomic labelling scheme, with H atoms omitted for clarity. Thin lines denote hydrogen bonds and displacement ellipsoids are drawn at the 50% probability level.

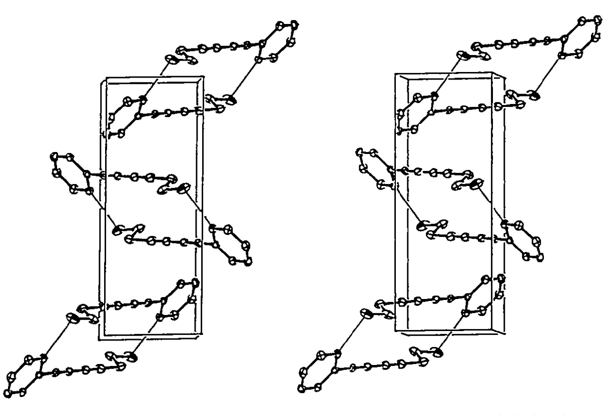


Fig. 2. Stereoview of the unit-cell packing for PyHxD. Origin, lower left; *b* axis, horizontal; *a* axis, vertical. The backbone diyne chains of the molecules are almost parallel to the (100) plane, but the chains of neighbouring dimers are perpendicular to each other.

The bond lengths C(6)—C(7) [1.177 (6) Å] and C(8)—C(9) [1.154 (7) Å] clearly show the two C—C bonds to have triple-bond character so that the backbone diyne chain linking the pyridyl ring and the ethanol moiety is almost linear. The pyridyl ring is planar within −0.004 (4) Å and the C(6) atom deviates from the best plane of the pyridyl ring by −0.013 (8) Å. The dihedral angle between the pyridyl ring and the ethanolic group is 77.0 (7)°. Atom C(9) deviates by −0.16 (2) Å from the best plane of the ethanol moiety and the angles C(9)—C(10)—C(11) [106.5 (7)°] and O—C(11)—C(10) [106.5 (7)°] are very close to the tetrahedral value.

As shown in Fig. 1, two molecules are held together by two intermolecular hydrogen bonds related by a centre of symmetry [O—H···N$^{\rm i}$ 2.807 (6) and N···H$^{\rm i}$—O$^{\rm i}$ 2.807 (6) Å; symmetry code: (i) $1-x$, $-1-y$, $1-z$]. These hydrogen bonds hinder specific rotation which is necessary for polymerization.

The backbone diyne chains of the dimer are aligned, making an angle *ca* 45° with the (010) plane, while those of the neighbouring dimer make the same angle with the (001) plane. Therefore, in the projection along the *a* axis, the diyne chains of the hydrogen-bonded dimers are perpendicular to each other. The shortest interdimer distance of 3.295 (7) Å between O and C(1)($x$, $y-1$, $1+z$) indicates that interaction between the hydrogen-bonded dimers is dominated by van der Waals forces.

The dimers are stacked along the *b* axis having a somewhat long polymerization distance by topochemical methods. The stacking distance between monomers is 5.423 (6) Å (the unit repeats along the *b* axis). The compound has a stiff frame structure and the stacking distance is much longer than the distance required to polymerize through a least-motion pathway as calculated by Baughman (1974), so that the title crystal is very stable under normal daylight and impossible to polymerize to give polydiacetylenes.

## Experimental

The title compound was prepared from 2-ethynylpyridine and 4-bromo-3-butyn-1-ol by the Cadiot–Chodkiewicz coupling method. It was recrystallized from *n*-hexane/diethyl ether solution as colourless needle-like crystals.

*Crystal data*

$C_{11}H_9NO$
$M_r = 171.847$
Monoclinic
$P2_1/a$
$a = 14.611\ (2)$ Å
$b = 5.423\ (6)$ Å
$c = 12.191\ (2)$ Å
$\beta = 96.89\ (1)^\circ$
$V = 958.9952$ Å$^3$
$Z = 4$
$D_x = 1.19$ Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 reflections
$\theta = 11.38–14.11^\circ$
$\mu = 0.07$ mm$^{-1}$
$T = 297$ K
Needle
0.50 × 0.20 × 0.17 mm
Colourless

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1716 measured reflections
1683 independent reflections
824 observed reflections [$F > 4\sigma(F)$]
$\theta_{\max} = 23°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 6$
$l = -13 \rightarrow 13$
3 standard reflections
frequency: 120 min
intensity decay: <1%

*Refinement*

Refinement on $F$
$R = 0.0664$
$wR = 0.0633$
$S = 2.7130$
824 reflections
154 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.000116F^2]$
$(\Delta/\sigma)_{\max} = 0.026$
$\Delta\rho_{\max} = 0.287$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.229$ e Å$^{-3}$
Atomic scattering factors from *SHELX*76 (Sheldrick, 1976)

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

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

| | $x$ | $y$ | $z$ | $U_{\rm eq}$ |
|---|---|---|---|---|
| O | 0.4275 (3) | −0.8174 (10) | 0.8124 (3) | 0.137 (2) |
| N | 0.4157 (3) | 0.1207 (7) | 0.1614 (3) | 0.076 (2) |
| C(1) | 0.4083 (5) | 0.2737 (12) | 0.0758 (5) | 0.088 (3) |
| C(2) | 0.3437 (5) | 0.4575 (13) | 0.0592 (6) | 0.095 (3) |
| C(3) | 0.2829 (5) | 0.4870 (12) | 0.1341 (5) | 0.093 (3) |
| C(4) | 0.2883 (4) | 0.3320 (11) | 0.2226 (5) | 0.079 (2) |
| C(5) | 0.3549 (3) | 0.1529 (9) | 0.2347 (4) | 0.067 (2) |
| C(6) | 0.3629 (3) | −0.0157 (9) | 0.3262 (4) | 0.071 (2) |
| C(7) | 0.3678 (3) | −0.1591 (10) | 0.3991 (4) | 0.075 (2) |
| C(8) | 0.3744 (3) | −0.3337 (11) | 0.4796 (4) | 0.088 (2) |
| C(9) | 0.3793 (3) | −0.4890 (13) | 0.5448 (5) | 0.106 (3) |
| C(10) | 0.3873 (6) | −0.7073 (16) | 0.6253 (6) | 0.116 (4) |
| C(11) | 0.4294 (6) | −0.6120 (17) | 0.7308 (6) | 0.123 (4) |

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| N—C(1) | 1.327 (6) | C(6)—C(7) | 1.177 (6) |
| N—C(5) | 1.345 (5) | C(7)—C(8) | 1.359 (8) |
| C(1)—C(2) | 1.371 (7) | C(8)—C(9) | 1.154 (7) |
| C(2)—C(3) | 1.357 (7) | C(9)—C(10) | 1.533 (9) |
| C(3)—C(4) | 1.363 (7) | C(10)—C(11) | 1.454 (8) |
| C(4)—C(5) | 1.370 (6) | O—C(11) | 1.495 (8) |
| C(5)—C(6) | 1.436 (6) | | |
| C(1)—N—C(5) | 116.3 (5) | C(4)—C(5)—C(6) | 121.7 (5) |
| N—C(1)—C(2) | 124.1 (6) | C(5)—C(6)—C(7) | 177.9 (5) |
| C(1)—C(2)—C(3) | 118.7 (6) | C(6)—C(7)—C(8) | 177.1 (5) |
| C(2)—C(3)—C(4) | 118.7 (7) | C(7)—C(8)—C(9) | 177.3 (6) |
| C(3)—C(4)—C(5) | 119.7 (6) | C(8)—C(9)—C(10) | 176.1 (7) |
| N—C(5)—C(4) | 122.6 (5) | C(9)—C(10)—C(11) | 106.5 (7) |
| N—C(5)—C(6) | 115.7 (5) | O—C(11)—C(10) | 106.5 (7) |

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O—H(O)···N$^{\rm i}$ | 0.90 (8) | 1.95 (8) | 2.807 (6) | 157 (8) |

Symmetry code: (i) $1 - x, -1 - y, 1 - z$.

Preliminary photographic studies on the crystal using rotation, Weissenberg and precession cameras revealed the approximate unit-cell dimensions and the reflection conditions $h0l$ for $h = 2n$ and $h00$ for $h = 2n$, which indicated a uniquely defined centrosymmetric monoclinic space group $P2_1/a$ ($C_{2h}^5$). The independent reflections were collected using an $\omega$-scan width of $(0.8 + 0.35\tan\theta)°$. During the data collection, one orientation reflection ($80\bar{1}$) was monitored every 200 reflections and the three standard reflections, ($\bar{7}2\bar{3}$), ($80\bar{2}$) and ($\bar{4}3\bar{4}$), varied by less than 1%. Intensity data were corrected for Lp factors with the program *SDP* (Enraf–Nonius, 1985). An asymmetric unit (Suh *et al.*, 1993) of X-ray intensity data were collected. *SDP* was used for data collection, cell refinement and data reduction. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). The *SHELX*76 program (Sheldrick, 1976) was used for full-matrix least-squares refinement with anisotropic displacement factors for all the non-H atoms. All H-atom positions was found from difference Fourier syntheses and refined isotropically. Geometric calculations were carried out using the *GEOM* program (Gilmore, 1979).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates, together with extra unit-cell packing diagrams, have been deposited with the IUCr (Reference: KH1007). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# α-Naphthoflavone†

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## Abstract

The crystal structure of the title compound, 2-phenyl-4*H*-naphtho[1,2-*b*]pyran-4-one, $C_{19}H_{12}O_2$, is stabilized by both intra- and intermolecular C—H···O hydrogen bonds.

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