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(4-Ethynylphenyl)methoxymethanimine

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Key indicators

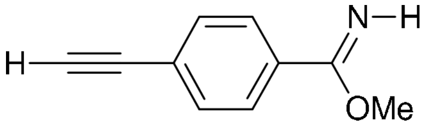
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.112
Data-to-parameter ratio = 13.9

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

The title molecule, $C_{10}H_9NO$, packs in a centrosymmetric structure. A series of weak C—H···N interactions have been identified.

Comment

The title compound, (I), was obtained inadvertently during the preparation of 4-ethynylcyanobenzene when we tried to deprotect the trimethylsilyl group in 4-trimethylsilanylethynylbenzonitrile with methanolic K_2CO_3. Along with the deprotected compound, this benzimidic ester derivative was obtained. There are a few synthetic reports on the esters of benzimidic acids (Hisao *et al.*, 1979) and most of them are hydrochloride salts (Hunter & Ludwig, 1962). The molecular structure and atom numbering are given in Fig. 1.



(I)

The ethynyl bond is nearly 0.07 Å shorter than is usually seen. In the Cambridge Structural Database (Version 5.25, July 2004; Allen, 2002), the crystal structures with refcodes NUFKOO (Onitsuka *et al.*, 1998), YUPNAY (Zimmerman & Zhu, 1995) and ASUYOC (Hennrich & Echavarren, 2004) contain a shorter ethynyl bond. This unusual shortening of the ethynyl bond may be due to the abnormal displacement ellipsoid of the C2 atom along the C2—C3 bond. The supramolecular behaviour of this compound is interesting. The molecules are centrosymmetrically arranged and form linear chains with weak C—H···N interactions (Table 1) along the *c* axis. These linear molecular arrays are further stacked along the *a* axis. Although the supramolecular equivalence of ethynyl and halogen groups is well documented (Dey *et al.*, 2003), the crystal structure of the corresponding bromo derivative (Kolakowski, 1974) shows that the structural behaviour of these two molecules is quite different.

Experimental

The compound was synthesized in two steps: Sonogashira coupling of 4-bromocyanobenzene affords 4-trimethylsilanylethynylbenzonitrile and then deprotection of the trimethylsilyl group with methanolic K_2CO_3 gives 4-ethynylcyanobenzene and compound (I) as a by-product. Crystals were obtained by purification of the crude material (column chromatography), followed by recrystallization from 1:1 dichloromethane–hexane by slow evaporation over a period of 24 h.

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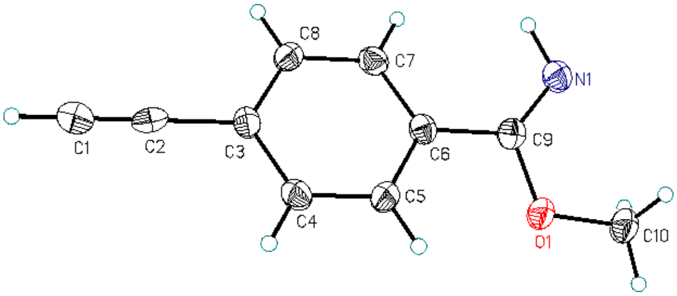

Figure 1
A view of the molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

Crystal data

$C_{10}H_9NO$
$M_r = 159.18$
Monoclinic, $P2_1/c$
$a = 3.9367\ (5)$ Å
$b = 20.461\ (3)$ Å
$c = 10.0799\ (13)$ Å
$\beta = 94.512\ (2)^\circ$
$V = 809.40\ (18)$ Å^3
$Z = 4$
$D_x = 1.306$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3237 reflections
$\theta = 2.3$–28.0°
$\mu = 0.09$ mm^{-1}
$T = 100\ (2)$ K
Thick plate, colourless
0.28 × 0.18 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Siemens, 1996)
$T_{\min} = 0.977$, $T_{\max} = 0.992$
9258 measured reflections
1586 independent reflections
1364 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\max} = 26.0^\circ$
$h = -4 \rightarrow 4$
$k = -24 \rightarrow 25$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.112$
$S = 1.07$
1586 reflections
114 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.4532P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.010 (3)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···N1$^{\rm i}$	0.93	2.43	3.355 (2)	172

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

All H atoms, except the NH hydrogen, were located in geometrical positions.

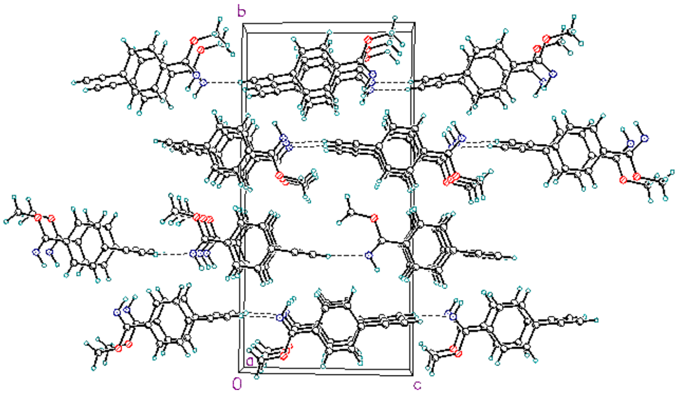

Figure 2
Packing of the molecules, viewed down the *a* axis. C—H···N interactions are shown as dashed lines.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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