IUCrData

ISSN 2414-3146

[1,4-Bis(4-methoxyphenyl)but-3-yn-2-yl](cyano)-methylamine

Muhammed Yousufuddin,[a]* Ruth Morales,[a] Waleed Cassis,[a] Gary W. Brown,[a] Ravi P. Singh[b] and Carl J. Lovely[b]

[a]Life and Health Sciences Department, University of North Texas at Dallas, 7300 University Hills Blvd, Dallas, TX 75241, USA, and [b]Department of Chemistry and Biochemistry, University of Texas at Arlington, 701 S. Nedderman Dr., Arlington, TX 76019, USA. *Correspondence e-mail: myousuf@untdallas.edu

Received 26 February 2018
Accepted 6 March 2018

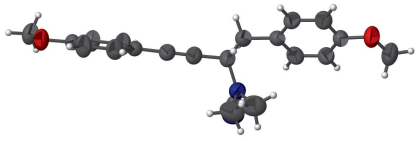
Edited by R. J. Butcher, Howard University, USA

Keywords: crystal structure; cyanamide.

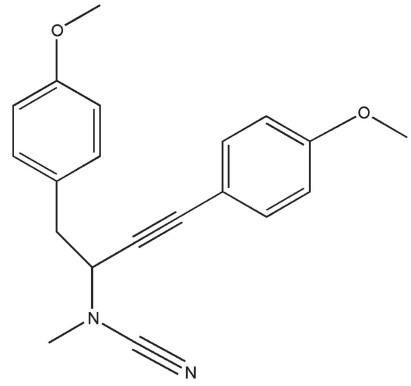
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{20}H_{20}N_2O_2$, crystallizes in the $P2_1/c$ space group with one molecule in the asymmetric unit. It contains an amine with a cyano substituent and hence is classified as a cyanamide. One terminal CH_3 group is disordered over two positions with occupancies of 0.874 (9)/0.126 (9).

3D view



Chemical scheme



Structure description

Some of us have been involved in the synthesis of cyanamides from *N*-methyl propargylamines and using this as a starting point to produce propargyl guanidine derivatives, specifically adapting reports from Looper (Gainer *et al.*, 2011) and van der Eycken (Ermolat'ev *et al.*, 2010). Originally, a very interesting spirocyclization compound was produced while attempting to synthesize the title compound (Singh *et al.*, 2016). It was later discovered that using potassium carbonate as the base afforded the intended cyanamide in good yield.

The title compound crystallizes in the monoclinic crystal system, $P2_1/c$. There is one molecule in the asymmetric unit yielding a Z value of 4. The compound contains one cyano bond with an observed C≡N bond distance of 1.143 (2) Å and one C≡C bond with a distance of 1.189 (3) Å. One terminal CH_3 group (C12) is disordered over two positions with occupancies of 0.874 (9)/0.126 (9). Intermolecular C—H···O interactions are observed in the crystal (Table 1) as well as π–π interactions between C4–C9 phenyl rings [centroid–centroid distance = 3.8766 (14) Å, symmetry operation $1-x, 1-y, 2-z$].

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

D—H···A	D—H	H···A	D···A	D—H···A
C12A—H12B···O2^{i}	0.96	2.48	3.366 (4)	154
C12B—H12D···O2^{i}	0.96	2.53	3.24 (2)	131

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

Synthesis and crystallization

The title compound was synthesized and crystallized following the procedure reported by our group (Singh *et al.*, 2016).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. One terminal CH_3 group (C12) is disordered over two positions with occupancies of 0.874 (9)/0.126 (9).

Acknowledgements

The authors thank the Center for Nanostructured Materials at the University of Texas at Arlington for the use of their diffractometer and the Robert A. Welch Foundation (4-1362) for supporting our research program.

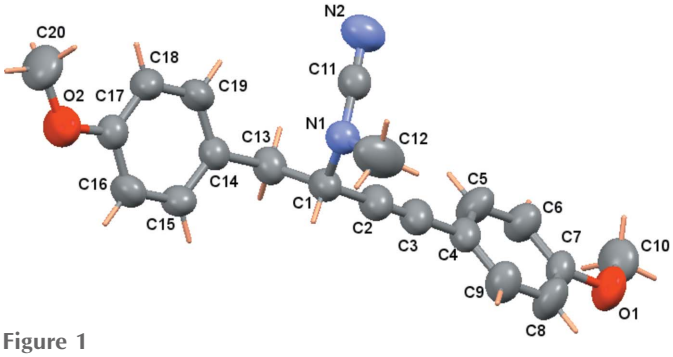


Figure 1
Plot of the title compound with displacement ellipsoids drawn at the 50% probability level. Only the major component of the disorder is shown.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{20}N_2O_2$
M_r	320.38
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	299
a, b, c (Å)	15.6203 (19), 8.6349 (11), 13.9758 (17)
β (°)	108.948 (2)
V (Å^3)	1782.9 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.25 × 0.15 × 0.05
Data collection	
Diffractometer	Bruker D8 QUEST CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.687, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15747, 3209, 2256
R_{int}	0.032
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.599
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.049, 0.124, 1.04
No. of reflections	3209
No. of parameters	231
No. of restraints	6
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.11, −0.15

Computer programs: *APEX2* and *SAINT* (Bruker, 2016), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*) and *SHELXTL* (Sheldrick, 2008).

References

Bruker (2016). *APEX2*, *SAINT*, and *SADABS*. Bruker AXS Inc., Madison, WI, USA

Ermolat'ev, D. S., Bariwal, J. B., Steenackers, H. P. L., De Keersmaecker, S. C. J. & Van der Eycken, E. V. (2010). *Angew. Chem. Int. Ed.* **49**, 9465–9468.

Gainer, M. J., Bennett, N. R., Takahashi, Y. & Looper, R. E. (2011). *Angew. Chem. Int. Ed.* **50**, 684–687.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Singh, R. P., Spears, J. A., Dalipe, A., Yousufuddin, M. & Lovely, C. J. (2016). *Tetrahedron Lett.* **57**, 3096–3099.

full crystallographic data

IUCrData (2018). **3**, x180389 [https://doi.org/10.1107/S2414314618003899]

[1,4-Bis(4-methoxyphenyl)but-3-yn-2-yl](cyano)methylamine

Muhammed Yousufuddin, Ruth Morales, Waleed Cassis, Gary W. Brown, Ravi P. Singh and Carl J. Lovely

[1,4-Bis(4-methoxyphenyl)but-3-yn-2-yl](cyano)methylamine

Crystal data

$C_{20}H_{20}N_2O_2$
M_r = 320.38
Monoclinic, $P2_1/c$
a = 15.6203 (19) Å
b = 8.6349 (11) Å
c = 13.9758 (17) Å
β = 108.948 (2)°
V = 1782.9 (4) Å^3
Z = 4
$F(000)$ = 680
D_x = 1.194 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4903 reflections
θ = 2.8–24.2°
μ = 0.08 mm^{-1}
T = 299 K
Plate, colourless
0.25 × 0.15 × 0.05 mm

Data collection

Bruker D8 QUEST CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.687, T_{max} = 0.745
15747 measured reflections
3209 independent reflections
2256 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.2°, θ_{min} = 2.8°
h = −18→18
k = −10→10
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.124
S = 1.04
3209 reflections
231 parameters
6 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 0.390P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and allowed to ride on their parent atoms, with U_{iso}(H) = 1.5U_{eq}(C) for methyl H and 1.2U_{eq}(C) for other H atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.57799 (10)	0.79026 (19)	1.22133 (10)	0.0801 (4)	
O2	0.06643 (10)	0.24335 (17)	0.21707 (11)	0.0851 (5)	
N1	0.15952 (10)	0.63823 (18)	0.62029 (11)	0.0588 (4)	
N2	0.18905 (13)	0.8975 (2)	0.56414 (16)	0.0865 (6)	
C1	0.23575 (13)	0.5294 (2)	0.66066 (13)	0.0610 (5)	
H1	0.209964	0.429138	0.669741	0.073*	
C2	0.29652 (14)	0.5784 (2)	0.76050 (15)	0.0697 (6)	
C3	0.34778 (14)	0.6166 (2)	0.84027 (15)	0.0679 (5)	
C4	0.40886 (13)	0.6616 (2)	0.93728 (13)	0.0587 (5)	
C5	0.48196 (16)	0.7550 (3)	0.94619 (15)	0.0797 (6)	
H5	0.492569	0.788760	0.887863	0.096*	
C6	0.54045 (14)	0.8005 (3)	1.03945 (15)	0.0734 (6)	
H6	0.589565	0.864071	1.043571	0.088*	
C7	0.52533 (13)	0.7510 (2)	1.12534 (13)	0.0578 (5)	
C8	0.45402 (15)	0.6558 (3)	1.11778 (15)	0.0775 (6)	
H8	0.444383	0.620286	1.176268	0.093*	
C9	0.39607 (14)	0.6114 (3)	1.02494 (15)	0.0726 (6)	
H9	0.347567	0.546656	1.021390	0.087*	
C10	0.65565 (16)	0.8814 (3)	1.23183 (17)	0.0907 (7)	
H10A	0.695384	0.827080	1.203500	0.136*	
H10B	0.686469	0.901142	1.302242	0.136*	
H10C	0.637842	0.977860	1.196884	0.136*	
C11	0.17720 (13)	0.7776 (3)	0.59205 (14)	0.0597 (5)	
C12A	0.0877 (2)	0.6328 (4)	0.6658 (4)	0.0891 (13)	0.874 (9)
H12A	0.111732	0.662393	0.735717	0.134*	0.874 (9)
H12B	0.063944	0.529530	0.661001	0.134*	0.874 (9)
H12C	0.040208	0.702994	0.630789	0.134*	0.874 (9)
C12B	0.0668 (17)	0.581 (3)	0.601 (3)	0.101 (5)	0.126 (9)
H12D	0.061446	0.536197	0.661323	0.151*	0.126 (9)
H12E	0.053591	0.503392	0.548461	0.151*	0.126 (9)
H12F	0.024869	0.665025	0.579117	0.151*	0.126 (9)
C13	0.28728 (13)	0.5050 (2)	0.58608 (14)	0.0659 (5)	
H13A	0.312140	0.603313	0.574249	0.079*	
H13B	0.337506	0.435089	0.615901	0.079*	
C14	0.22912 (12)	0.4395 (2)	0.48628 (13)	0.0555 (5)	
C15	0.20917 (13)	0.2831 (2)	0.47567 (14)	0.0635 (5)	
H15	0.232870	0.218169	0.531061	0.076*	
C16	0.15548 (13)	0.2209 (2)	0.38592 (15)	0.0664 (5)	
H16	0.143494	0.115157	0.380955	0.080*	
C17	0.11941 (12)	0.3153 (2)	0.30326 (14)	0.0584 (5)	
C18	0.13769 (14)	0.4706 (2)	0.31150 (14)	0.0678 (5)	
H18	0.113317	0.535283	0.256101	0.081*	
C19	0.19235 (14)	0.5311 (2)	0.40214 (15)	0.0681 (5)	
H19	0.204728	0.636697	0.406576	0.082*	
C20	0.02856 (19)	0.3332 (3)	0.12903 (17)	0.1032 (9)	

H20A	−0.008987	0.412710	0.142382	0.155*
H20B	−0.007307	0.268024	0.075259	0.155*
H20C	0.076206	0.379836	0.109588	0.155*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0736 (9)	0.1082 (12)	0.0514 (8)	−0.0065 (9)	0.0106 (7)	−0.0141 (8)
O2	0.0909 (11)	0.0813 (10)	0.0642 (9)	−0.0046 (8)	−0.0006 (8)	−0.0114 (8)
N1	0.0566 (9)	0.0574 (10)	0.0596 (9)	0.0005 (7)	0.0148 (7)	−0.0001 (8)
N2	0.0871 (14)	0.0658 (12)	0.1086 (15)	0.0003 (10)	0.0345 (12)	0.0091 (11)
C1	0.0661 (12)	0.0548 (11)	0.0544 (11)	0.0019 (9)	0.0090 (9)	−0.0019 (9)
C2	0.0731 (14)	0.0681 (13)	0.0587 (12)	0.0082 (10)	0.0088 (11)	−0.0032 (10)
C3	0.0710 (13)	0.0663 (13)	0.0584 (12)	0.0130 (10)	0.0099 (11)	−0.0042 (10)
C4	0.0577 (11)	0.0607 (12)	0.0512 (11)	0.0116 (9)	0.0087 (9)	−0.0052 (9)
C5	0.0895 (16)	0.0968 (17)	0.0489 (12)	−0.0125 (13)	0.0173 (11)	0.0062 (11)
C6	0.0706 (13)	0.0878 (15)	0.0578 (12)	−0.0164 (11)	0.0152 (10)	−0.0005 (11)
C7	0.0553 (11)	0.0689 (12)	0.0463 (10)	0.0082 (9)	0.0124 (8)	−0.0071 (9)
C8	0.0776 (14)	0.1084 (18)	0.0496 (11)	−0.0138 (13)	0.0250 (11)	−0.0049 (11)
C9	0.0632 (12)	0.0891 (15)	0.0663 (13)	−0.0110 (11)	0.0221 (10)	−0.0096 (11)
C10	0.0737 (15)	0.1042 (18)	0.0751 (15)	−0.0065 (13)	−0.0020 (12)	−0.0140 (13)
C11	0.0534 (11)	0.0636 (13)	0.0589 (11)	0.0006 (10)	0.0140 (9)	−0.0054 (10)
C12A	0.089 (2)	0.076 (2)	0.120 (3)	−0.0040 (16)	0.059 (2)	0.0020 (19)
C12B	0.098 (9)	0.081 (9)	0.128 (10)	0.004 (8)	0.042 (9)	0.006 (8)
C13	0.0585 (11)	0.0669 (12)	0.0658 (12)	0.0025 (10)	0.0113 (10)	−0.0093 (10)
C14	0.0556 (10)	0.0541 (11)	0.0559 (11)	0.0045 (8)	0.0168 (8)	−0.0043 (9)
C15	0.0710 (13)	0.0553 (12)	0.0576 (11)	0.0058 (9)	0.0120 (10)	0.0066 (9)
C16	0.0756 (13)	0.0489 (11)	0.0681 (13)	−0.0048 (10)	0.0143 (11)	−0.0019 (10)
C17	0.0573 (11)	0.0607 (12)	0.0541 (11)	0.0010 (9)	0.0137 (9)	−0.0057 (9)
C18	0.0860 (14)	0.0591 (12)	0.0524 (11)	0.0105 (10)	0.0144 (10)	0.0057 (9)
C19	0.0891 (14)	0.0472 (11)	0.0660 (13)	−0.0001 (10)	0.0225 (11)	−0.0002 (10)
C20	0.1074 (19)	0.119 (2)	0.0610 (14)	0.0096 (17)	−0.0036 (13)	−0.0046 (14)

Geometric parameters (Å, º)

O1—C7	1.369 (2)	C10—H10B	0.9600
O1—C10	1.413 (3)	C10—H10C	0.9600
O2—C17	1.369 (2)	C12A—H12A	0.9600
O2—C20	1.412 (3)	C12A—H12B	0.9600
N1—C11	1.323 (3)	C12A—H12C	0.9600
N1—C12A	1.459 (3)	C12B—H12D	0.9600
N1—C12B	1.47 (3)	C12B—H12E	0.9600
N1—C1	1.478 (2)	C12B—H12F	0.9600
N2—C11	1.143 (2)	C13—C14	1.506 (2)
C1—C2	1.472 (3)	C13—H13A	0.9700
C1—C13	1.524 (3)	C13—H13B	0.9700
C1—H1	0.9800	C14—C19	1.378 (3)
C2—C3	1.189 (3)	C14—C15	1.383 (3)

C3—C4	1.435 (3)	C15—C16	1.372 (2)
C4—C5	1.370 (3)	C15—H15	0.9300
C4—C9	1.374 (3)	C16—C17	1.376 (3)
C5—C6	1.384 (3)	C16—H16	0.9300
C5—H5	0.9300	C17—C18	1.368 (3)
C6—C7	1.365 (3)	C18—C19	1.380 (3)
C6—H6	0.9300	C18—H18	0.9300
C7—C8	1.361 (3)	C19—H19	0.9300
C8—C9	1.374 (3)	C20—H20A	0.9600
C8—H8	0.9300	C20—H20B	0.9600
C9—H9	0.9300	C20—H20C	0.9600
C10—H10A	0.9600		

C7—O1—C10	117.52 (16)	N1—C12A—H12B	109.5
C17—O2—C20	118.68 (17)	H12A—C12A—H12B	109.5
C11—N1—C12A	115.07 (19)	N1—C12A—H12C	109.5
C11—N1—C12B	122.7 (10)	H12A—C12A—H12C	109.5
C11—N1—C1	118.17 (16)	H12B—C12A—H12C	109.5
C12A—N1—C1	116.83 (19)	N1—C12B—H12D	109.5
C12B—N1—C1	118.6 (10)	N1—C12B—H12E	109.5
C2—C1—N1	111.74 (15)	H12D—C12B—H12E	109.5
C2—C1—C13	111.52 (16)	N1—C12B—H12F	109.5
N1—C1—C13	111.45 (15)	H12D—C12B—H12F	109.5
C2—C1—H1	107.3	H12E—C12B—H12F	109.5
N1—C1—H1	107.3	C14—C13—C1	113.05 (15)
C13—C1—H1	107.3	C14—C13—H13A	109.0
C3—C2—C1	178.0 (2)	C1—C13—H13A	109.0
C2—C3—C4	179.2 (2)	C14—C13—H13B	109.0
C5—C4—C9	117.56 (18)	C1—C13—H13B	109.0
C5—C4—C3	121.59 (19)	H13A—C13—H13B	107.8
C9—C4—C3	120.85 (19)	C19—C14—C15	116.85 (17)
C4—C5—C6	121.91 (19)	C19—C14—C13	122.28 (18)
C4—C5—H5	119.0	C15—C14—C13	120.86 (17)
C6—C5—H5	119.0	C16—C15—C14	122.01 (18)
C7—C6—C5	119.3 (2)	C16—C15—H15	119.0
C7—C6—H6	120.3	C14—C15—H15	119.0
C5—C6—H6	120.3	C15—C16—C17	119.82 (18)
C8—C7—C6	119.46 (18)	C15—C16—H16	120.1
C8—C7—O1	116.22 (17)	C17—C16—H16	120.1
C6—C7—O1	124.32 (19)	C18—C17—O2	124.68 (17)
C7—C8—C9	120.91 (19)	C18—C17—C16	119.52 (17)
C7—C8—H8	119.5	O2—C17—C16	115.80 (17)
C9—C8—H8	119.5	C17—C18—C19	119.89 (18)
C8—C9—C4	120.8 (2)	C17—C18—H18	120.1
C8—C9—H9	119.6	C19—C18—H18	120.1
C4—C9—H9	119.6	C14—C19—C18	121.89 (18)
O1—C10—H10A	109.5	C14—C19—H19	119.1
O1—C10—H10B	109.5	C18—C19—H19	119.1

H10A—C10—H10B	109.5	O2—C20—H20A	109.5
O1—C10—H10C	109.5	O2—C20—H20B	109.5
H10A—C10—H10C	109.5	H20A—C20—H20B	109.5
H10B—C10—H10C	109.5	O2—C20—H20C	109.5
N2—C11—N1	177.0 (2)	H20A—C20—H20C	109.5
N1—C12A—H12A	109.5	H20B—C20—H20C	109.5

C11—N1—C1—C2	69.5 (2)	C3—C4—C9—C8	−179.32 (19)
C12A—N1—C1—C2	−74.5 (3)	C2—C1—C13—C14	174.28 (17)
C12B—N1—C1—C2	−118.6 (19)	N1—C1—C13—C14	−60.1 (2)
C11—N1—C1—C13	−56.0 (2)	C1—C13—C14—C19	99.5 (2)
C12A—N1—C1—C13	160.0 (3)	C1—C13—C14—C15	−79.6 (2)
C12B—N1—C1—C13	115.9 (19)	C19—C14—C15—C16	0.1 (3)
C9—C4—C5—C6	−1.1 (3)	C13—C14—C15—C16	179.21 (18)
C3—C4—C5—C6	179.1 (2)	C14—C15—C16—C17	−0.3 (3)
C4—C5—C6—C7	0.1 (3)	C20—O2—C17—C18	1.2 (3)
C5—C6—C7—C8	1.1 (3)	C20—O2—C17—C16	−179.0 (2)
C5—C6—C7—O1	−179.14 (19)	C15—C16—C17—C18	0.1 (3)
C10—O1—C7—C8	176.30 (19)	C15—C16—C17—O2	−179.76 (17)
C10—O1—C7—C6	−3.4 (3)	O2—C17—C18—C19	−179.79 (18)
C6—C7—C8—C9	−1.3 (3)	C16—C17—C18—C19	0.3 (3)
O1—C7—C8—C9	178.92 (19)	C15—C14—C19—C18	0.4 (3)
C7—C8—C9—C4	0.3 (3)	C13—C14—C19—C18	−178.73 (18)
C5—C4—C9—C8	1.0 (3)	C17—C18—C19—C14	−0.6 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12*A*—H12*B*···O2^{i}	0.96	2.48	3.366 (4)	154
C12*B*—H12*D*···O2^{i}	0.96	2.53	3.24 (2)	131

Symmetry code: (i) x, $-y+1/2$, $z+1/2$.