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A triclinic polymorph of *N*-[5-(diphenylamino)-penta-2,4-diyn-1-yl]benzamide

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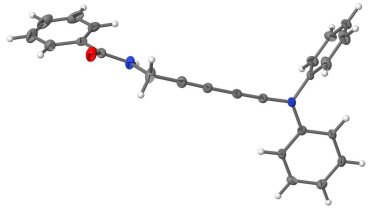
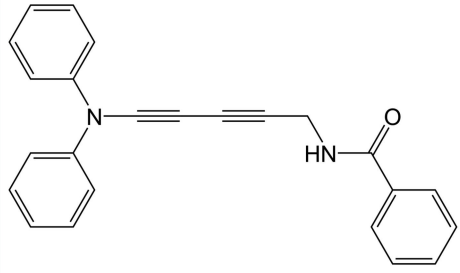
The title compound, $C_{24}H_{18}N_2O$, was been described previously in the space group $P2_1/c$ with $Z = 4$ [Kawashima & Okuno (2017). *IUCrData*, **2**, x170277]. The current $P\overline{1}$ polymorph was obtained from a chloroform–ethanol solution. The molecular structure in this polymorph is slightly different from the previously reported structure, with different dihedral angles of the two *N*-phenyl groups to the ynamine plane; these are 79.99 (11) and 12.09 (11)° in the polymorph reported here. The molecules form dimers through four C—H···π interactions. Furthermore, in this polymorph, the molecules stack along the *a* axis to form a molecular arrangement that would be suitable to promote the solid-state polymerization of diacetylenes.

3D view



Chemical scheme



Structure description

The title compound (Fig. 1) is a diacetylene derivative that has potential solid-state polymerization reactivity. Previously, it was isolated in a monoclinic $P2_1/c$ polymorph with $Z = 4$ (Kawashima & Okuno, 2017). The current triclinic polymorph was obtained by recrystallizationfrom a mixed solution of chloroform–ethanol (1:1 *v*:*v*).

In the title compound, the N1/C1/C7/C13 ynamine moiety has an almost planar structure (r.m.s. deviation = 0.0239 Å). The C1–C6 and C7–C12 phenyl groups subtend dihedral angles of 79.99 (11) and 12.09 (11)°, respectively, with this plane. This geometric situation is very similar to that reported for 5-(diphenylamino)penta-2,4-diyn-1-ol (Tokutome *et al.*, 2012). However, those found in the previously reported polymorph are 52.99 (7) and 21.91 (7)°, showing significant variation from those found in the current structure.

In the crystal, molecules are connected by N—H···O hydrogen bonds to form one-dimensional stacks along the *a* axis (Fig. 2). According to Baughman's notation (Baughman, 1974), the repeating interval (the length of the *a* axis in this case) and

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1···O1^{i}	0.88 (3)	2.09 (3)	2.952 (4)	169 (2)
C11—H11···Cg^{ii}	0.95	2.97	3.822 (5)	150

Symmetry codes: (i) $x-1, y, z$; (ii) $-x-1, -y, -z+2$.

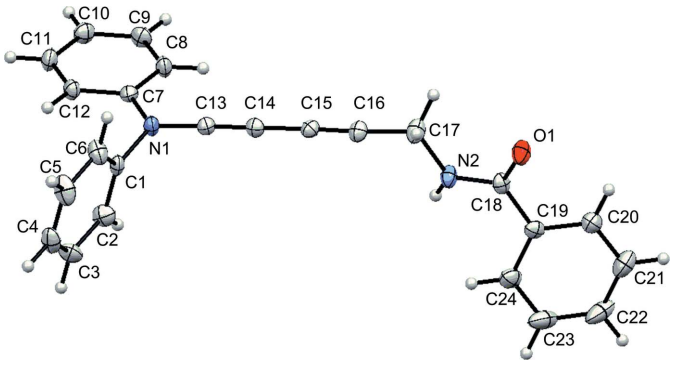


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level and H atoms shown as small spheres.

inclination angle to the stacking axis (C16—C13—C13^{ii}) are 5.033 (5) Å and 44.27 (7)° [symmetry code: (ii) $x+1, y, z$], respectively. This molecular arrangement is suitable for the solid-state polymerization of diacetylenes and the title compound shows polymerization reactivity when exposed to heat or under irradiation by UV light. Weak intercolumnar C—H···π interactions are also detected between the stacks of molecules (Table 1).

Synthesis and crystallization

Preparation was carried out according to Tokutome *et al.* (2012), Tokutome & Okuno (2013) and Tabata *et al.* (2012).

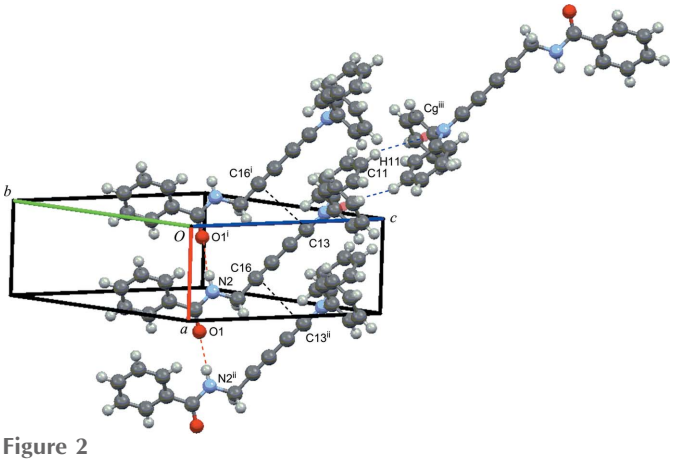


Figure 2
A view of the intermolecular interactions in the title compound [symmetry codes:(i) $x-1, y, z$; (ii) $x+1, y, z$; (iii) $-x-1, -y, -z+2$]. Chains of molecules along the a axis with hydrogen bonds are drawn as red dashed line. Ring centroids are shown as red spheres and C—H···π(ring) contacts are drawn as blue dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{24}H_{18}N_2O$
M_r	350.42
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	93
a, b, c (Å)	5.033 (5), 11.682 (10), 15.433 (15)
α, β, γ (°)	96.228 (19), 91.207 (14), 94.27 (2)
V (Å^3)	899.2 (14)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.20 × 0.10 × 0.07
Data collection	
Diffractometer	Rigaku Saturn724+
Absorption correction	Numerical (*NUMABS*; Rigaku, 1999)
T_{min}, T_{max}	0.988, 0.994
No. of measured, independent and observed [$F^2 > 2.0\sigma(F^2)$] reflections	6092, 3091, 2235
R_{int}	0.040
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.058, 0.136, 1.05
No. of reflections	3091
No. of parameters	248
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.34, −0.21

Computer programs: *CrystalClear* (Rigaku, 2008), *SIR92* (Altomare *et al.*, 1994), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008) and *CrystalStructure* (Rigaku, 2014).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2019). **4**, x190371 [https://doi.org/10.1107/S2414314619003717]

A triclinic polymorph of *N*-[5-(diphenylamino)penta-2,4-diyn-1-yl]benzamide

Koji Ozaki and Tsunehisa Okuno

N-[5-(Diphenylamino)penta-2,4-diyn-1-yl]benzamide

Crystal data

$C_{24}H_{18}N_2O$
M_r = 350.42
Triclinic, $P\overline{1}$
a = 5.033 (5) Å
b = 11.682 (10) Å
c = 15.433 (15) Å
α = 96.228 (19)°
β = 91.207 (14)°
γ = 94.27 (2)°
V = 899.2 (14) Å^3
Z = 2
$F(000)$ = 368.00
D_x = 1.294 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 2799 reflections
θ = 2.3–30.8°
μ = 0.08 mm^{-1}
T = 93 K
Block, colorless
0.20 × 0.10 × 0.07 mm

Data collection

Rigaku Saturn724+ diffractometer
Detector resolution: 7.111 pixels mm^{-1}
ω scans
Absorption correction: numerical (NUMABS; Rigaku, 1999)
T_{min} = 0.988, T_{max} = 0.994
6092 measured reflections
3091 independent reflections
2235 reflections with $F^2 > 2.0\sigma(F^2)$
R_{int} = 0.040
θ_{max} = 25.0°, θ_{min} = 3.0°
h = −5→5
k = −13→10
l = −18→18

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.136
S = 1.05
3091 reflections
248 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2 + 0.5409P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

Geometry. ENTER SPECIAL DETAILS OF THE MOLECULAR GEOMETRY
Refinement. Refinement was performed using all reflections. The weighted R-factor (wR) and goodness of fit (S) are based on F^2. R-factor (gt) are based on F. The threshold expression of $F^2 > 2.0$ sigma(F^2) is used only for calculating R-factor (gt).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	1.3363 (3)	0.62279 (14)	0.63872 (12)	0.0291 (4)
N1	−0.0220 (4)	0.19512 (16)	0.87818 (13)	0.0207 (5)
N2	0.9003 (4)	0.57251 (17)	0.65461 (14)	0.0233 (5)
C1	−0.0880 (5)	0.08597 (19)	0.82601 (16)	0.0201 (5)
C2	−0.2982 (5)	0.0755 (2)	0.76636 (17)	0.0290 (6)
C3	−0.3651 (6)	−0.0291 (2)	0.71730 (17)	0.0311 (6)
C4	−0.2210 (5)	−0.1226 (2)	0.72699 (18)	0.0309 (6)
C5	−0.0093 (6)	−0.1117 (2)	0.7858 (2)	0.0360 (7)
C6	0.0576 (5)	−0.0074 (2)	0.83586 (19)	0.0296 (6)
C7	−0.1784 (5)	0.23177 (19)	0.94990 (15)	0.0193 (5)
C8	−0.1554 (5)	0.3459 (2)	0.98629 (16)	0.0247 (6)
C9	−0.3156 (5)	0.3812 (2)	1.05424 (17)	0.0266 (6)
C10	−0.4954 (5)	0.3033 (2)	1.08805 (17)	0.0268 (6)
C11	−0.5150 (5)	0.1893 (2)	1.05221 (17)	0.0265 (6)
C12	−0.3572 (5)	0.1530 (2)	0.98362 (16)	0.0225 (6)
C13	0.1717 (5)	0.26552 (19)	0.84948 (15)	0.0192 (5)
C14	0.3477 (5)	0.32555 (19)	0.82152 (16)	0.0201 (5)
C15	0.5552 (5)	0.39027 (19)	0.78986 (15)	0.0197 (5)
C16	0.7342 (5)	0.4456 (2)	0.75999 (16)	0.0228 (6)
C17	0.9673 (5)	0.5097 (2)	0.72805 (18)	0.0295 (6)
C18	1.1009 (5)	0.6262 (2)	0.61485 (16)	0.0212 (5)
C19	1.0257 (5)	0.6922 (2)	0.54136 (15)	0.0208 (5)
C20	1.1882 (5)	0.7891 (2)	0.52703 (17)	0.0280 (6)
C21	1.1243 (6)	0.8541 (3)	0.46038 (19)	0.0394 (7)
C22	0.9044 (6)	0.8206 (3)	0.40727 (19)	0.0435 (8)
C23	0.7433 (6)	0.7230 (3)	0.41991 (18)	0.0423 (8)
C24	0.8052 (5)	0.6589 (2)	0.48719 (17)	0.0296 (6)
H1	0.732 (5)	0.581 (2)	0.6428 (17)	0.024 (7)*
H2	−0.39701	0.14018	0.75897	0.0348*
H3	−0.51142	−0.03651	0.67667	0.0374*
H4	−0.26753	−0.19441	0.69317	0.0370*
H5	0.09163	−0.176	0.79211	0.0432*
H6	0.20311	−0.00024	0.8768	0.0355*
H8	−0.02988	0.39976	0.96463	0.0296*
H9	−0.30232	0.45995	1.07807	0.0319*
H10	−0.60344	0.32789	1.13519	0.0322*
H11	−0.63804	0.13527	1.07481	0.0317*
H12	−0.37127	0.07432	0.95969	0.0270*
H17A	1.04771	0.56518	0.77621	0.0354*
H17B	1.10183	0.45495	0.7098	0.0354*
H20	1.3434	0.81105	0.56283	0.0337*
H21	1.23292	0.92172	0.45164	0.0473*
H22	0.86219	0.86473	0.36137	0.0522*
H23	0.5914	0.70001	0.3828	0.0508*
H24	0.69529	0.59172	0.49603	0.0355*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0180 (10)	0.0335 (10)	0.0378 (11)	0.0014 (8)	0.0027 (8)	0.0134 (8)
N1	0.0225 (12)	0.0160 (10)	0.0231 (12)	−0.0025 (8)	0.0054 (9)	0.0010 (8)
N2	0.0148 (12)	0.0251 (12)	0.0316 (13)	0.0014 (9)	0.0025 (10)	0.0103 (9)
C1	0.0244 (14)	0.0133 (12)	0.0221 (13)	−0.0020 (10)	0.0076 (11)	0.0017 (10)
C2	0.0363 (16)	0.0226 (14)	0.0283 (15)	0.0074 (11)	0.0008 (12)	0.0000 (11)
C3	0.0362 (17)	0.0286 (15)	0.0260 (15)	−0.0009 (12)	−0.0026 (12)	−0.0054 (11)
C4	0.0357 (17)	0.0200 (14)	0.0343 (16)	−0.0034 (11)	0.0137 (13)	−0.0068 (11)
C5	0.0304 (16)	0.0195 (14)	0.058 (2)	0.0060 (11)	0.0070 (14)	0.0022 (13)
C6	0.0235 (14)	0.0228 (14)	0.0426 (17)	−0.0001 (11)	−0.0004 (12)	0.0054 (12)
C7	0.0196 (13)	0.0208 (13)	0.0175 (13)	0.0006 (10)	−0.0010 (10)	0.0034 (10)
C8	0.0277 (14)	0.0190 (13)	0.0262 (14)	−0.0060 (10)	0.0055 (11)	0.0012 (10)
C9	0.0347 (16)	0.0178 (13)	0.0257 (14)	0.0012 (11)	0.0035 (12)	−0.0044 (10)
C10	0.0293 (15)	0.0271 (14)	0.0241 (14)	0.0026 (11)	0.0063 (11)	0.0012 (11)
C11	0.0302 (15)	0.0216 (13)	0.0279 (15)	−0.0029 (11)	0.0081 (12)	0.0062 (11)
C12	0.0288 (14)	0.0143 (12)	0.0240 (14)	−0.0012 (10)	0.0036 (11)	0.0020 (10)
C13	0.0190 (13)	0.0191 (12)	0.0193 (13)	0.0017 (10)	0.0009 (10)	0.0019 (10)
C14	0.0218 (13)	0.0169 (12)	0.0217 (13)	0.0037 (10)	0.0011 (11)	0.0006 (10)
C15	0.0196 (13)	0.0203 (12)	0.0198 (13)	0.0030 (10)	0.0009 (10)	0.0034 (10)
C16	0.0221 (14)	0.0207 (13)	0.0267 (14)	0.0045 (10)	0.0014 (11)	0.0058 (10)
C17	0.0188 (14)	0.0348 (15)	0.0385 (17)	0.0033 (11)	0.0021 (12)	0.0192 (13)
C18	0.0186 (14)	0.0196 (13)	0.0251 (14)	0.0013 (10)	0.0056 (11)	0.0001 (10)
C19	0.0192 (13)	0.0246 (13)	0.0181 (13)	0.0038 (10)	0.0037 (10)	−0.0015 (10)
C20	0.0287 (15)	0.0295 (15)	0.0255 (15)	−0.0014 (11)	0.0006 (12)	0.0036 (11)
C21	0.0464 (19)	0.0396 (17)	0.0358 (17)	0.0045 (14)	0.0084 (14)	0.0175 (13)
C22	0.0411 (19)	0.069 (2)	0.0262 (17)	0.0170 (16)	0.0050 (14)	0.0219 (15)
C23	0.0315 (17)	0.076 (2)	0.0196 (15)	0.0056 (15)	−0.0010 (12)	0.0039 (14)
C24	0.0283 (15)	0.0388 (16)	0.0205 (14)	−0.0007 (12)	0.0050 (12)	−0.0002 (11)

Geometric parameters (Å, º)

O1—C18	1.238 (3)	C19—C24	1.381 (4)
N1—C1	1.445 (3)	C20—C21	1.389 (4)
N1—C7	1.419 (3)	C21—C22	1.371 (4)
N1—C13	1.342 (3)	C22—C23	1.382 (5)
N2—C17	1.462 (4)	C23—C24	1.388 (4)
N2—C18	1.344 (3)	N2—H1	0.88 (3)
C1—C2	1.377 (4)	C2—H2	0.950
C1—C6	1.378 (4)	C3—H3	0.950
C2—C3	1.381 (3)	C4—H4	0.950
C3—C4	1.375 (4)	C5—H5	0.950
C4—C5	1.374 (4)	C6—H6	0.950
C5—C6	1.385 (4)	C8—H8	0.950
C7—C8	1.385 (3)	C9—H9	0.950
C7—C12	1.388 (3)	C10—H10	0.950
C8—C9	1.383 (4)	C11—H11	0.950

C9—C10	1.385 (4)	C12—H12	0.950
C10—C11	1.381 (3)	C17—H17A	0.990
C11—C12	1.386 (4)	C17—H17B	0.990
C13—C14	1.205 (3)	C20—H20	0.950
C14—C15	1.373 (3)	C21—H21	0.950
C15—C16	1.199 (3)	C22—H22	0.950
C16—C17	1.468 (4)	C23—H23	0.950
C18—C19	1.496 (4)	C24—H24	0.950
C19—C20	1.387 (4)		

C1—N1—C7	120.96 (19)	C18—N2—H1	122.4 (17)
C1—N1—C13	117.0 (2)	C1—C2—H2	120.071
C7—N1—C13	121.57 (19)	C3—C2—H2	120.068
C17—N2—C18	118.0 (2)	C2—C3—H3	119.845
N1—C1—C2	119.6 (2)	C4—C3—H3	119.829
N1—C1—C6	120.4 (2)	C3—C4—H4	120.118
C2—C1—C6	120.1 (2)	C5—C4—H4	120.116
C1—C2—C3	119.9 (2)	C4—C5—H5	119.851
C2—C3—C4	120.3 (3)	C6—C5—H5	119.851
C3—C4—C5	119.8 (2)	C1—C6—H6	120.160
C4—C5—C6	120.3 (3)	C5—C6—H6	120.160
C1—C6—C5	119.7 (3)	C7—C8—H8	120.094
N1—C7—C8	120.5 (2)	C9—C8—H8	120.082
N1—C7—C12	119.9 (2)	C8—C9—H9	119.555
C8—C7—C12	119.6 (2)	C10—C9—H9	119.551
C7—C8—C9	119.8 (2)	C9—C10—H10	120.499
C8—C9—C10	120.9 (2)	C11—C10—H10	120.495
C9—C10—C11	119.0 (2)	C10—C11—H11	119.660
C10—C11—C12	120.7 (2)	C12—C11—H11	119.674
C7—C12—C11	119.9 (2)	C7—C12—H12	120.027
N1—C13—C14	177.7 (2)	C11—C12—H12	120.023
C13—C14—C15	177.6 (3)	N2—C17—H17A	109.084
C14—C15—C16	178.2 (2)	N2—C17—H17B	109.092
C15—C16—C17	175.6 (3)	C16—C17—H17A	109.078
N2—C17—C16	112.6 (2)	C16—C17—H17B	109.082
O1—C18—N2	121.6 (2)	H17A—C17—H17B	107.829
O1—C18—C19	121.6 (2)	C19—C20—H20	119.955
N2—C18—C19	116.7 (2)	C21—C20—H20	119.953
C18—C19—C20	118.0 (2)	C20—C21—H21	120.056
C18—C19—C24	122.5 (2)	C22—C21—H21	120.062
C20—C19—C24	119.5 (2)	C21—C22—H22	119.708
C19—C20—C21	120.1 (2)	C23—C22—H22	119.707
C20—C21—C22	119.9 (3)	C22—C23—H23	120.233
C21—C22—C23	120.6 (3)	C24—C23—H23	120.242
C22—C23—C24	119.5 (3)	C19—C24—H24	119.798
C19—C24—C23	120.4 (2)	C23—C24—H24	119.790
C17—N2—H1	119.1 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···O1^{i}	0.88 (3)	2.09 (3)	2.952 (4)	169 (2)
C11—H11···*Cg*^{ii}	0.95	2.97	3.822 (5)	150

Symmetry codes: (i) $x-1, y, z$; (ii) $-x-1, -y, -z+2$.