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Preparations, crystal polymorphs and DFT calculations of N^1 , N^4 , N^4 -tetraphenylbuta-1, 3-diyne-1, 4-diamine

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HIGHLIGHTS

• A novel ynamine molecule, N1,N1,N4,N4-tetraphenylbuta-1,3-diyne-1,4-diamine (2),was prepared.

• Two polymorphs of **2** were characterized by single crystal X-ray diffraction technique.

• Angular dependence on the energy of 2 was revealed by DFT calculation.

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ABSTRACT

A novel ynamine compound, N^1, N^1, N^4, N^4 -tetraphenylbuta-1,3-diyne-1,4-diamine (**2**), was prepared and characterized by single crystal X-ray diffraction, ¹H and ¹³C NMR. The compound had two crystal polymorphs and they were solved successfully. There were two independent molecules in both polymorphs and the four kinds of molecules had a similar structure. The difference in crystal packing was found to originate in C—H··· π interactions.

The geometries obtained by DFT calculations showed a good agreement with the observed one, but a significant difference was recognized at the twist angles between the planes which were formed by the nitrogen atom and the connecting three carbon atoms. The dependence of the energy on the twist angle was also calculated by DFT method. The result indicated that the energy showed little change in the twist angle between 70° and 90° and that the molecular structure of **2** could change easily by packing force within the range.

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1. Introduction

Acetylene bonds, which have a cylindrical π -conjugated system, are indispensable for construction of supramolecular structures [1,2]. Since conjugated acetylene bonds, such as diacetylenes, triacetylenes or oligo-acetylenes, have also been paid attention from the viewpoint of a nanowire [3] or rotational isomerism [4].

In order to apply conjugated oligo-acetylene system to a nanowire, a metal-organic junction plays a crucial role whose origin is $d-\pi$ interaction. There are mainly two methodologies for metal-organic junction. One is the usage of metal-acetylide (M-C=C) bond [5] and the other is the usage of metal-heteroatom interaction where the hetero atoms, such as N or S, are introduced at the end of organic compounds [6]. In the latter case, a twist angle of π -conjugated systems in both ends becomes important.

Ynamines, where an acetylene group connects directly to a nitrogen atom, have been discovered accidently in 1958 [7] and many kinds of ynamines have been prepared yet [8]. They are known to be unstable to proceed polymerization and hydrolysis.

* Corresponding author. E-mail address: okuno@center.wakayama-u.ac.jp (T. Okuno). The instability of ynamines originates in high electron density owing to conjugation of π -electrons with a lone pair of a nitrogen atom. To our knowledge there are only three compounds, where nitrogen atoms connect both ends of conjugated acetylene(s) [9,10].

We have already reported preparation of ethynyldiphenylamine (**1**) and aminodiacetylene compounds by following Hay coupling reaction [11–13]. In this paper, we wish to report the preparation and the crystal polymorphs of N^1 , N^1 , N^4 , N^4 -tetraphenylbuta-1, 3-diyne-1,4-diamine (**2**), where diphenylamino groups connected to the both ends of 1,3-butadiyne. The molecular structures are compared with that obtained by DFT method and the origins of crystal polymorphs are also elucidated.

2. Experimental

2.1. General procedure

All chemicals were purchased from Kanto Chemical Co. Ltd. or Tokyo Kasei Kogyo Co. Ltd. and were used without further purification. Gel permeation chromatography (GPC) was performed on a JAI LC-918 equipped with JAIGEL-1H and -2H columns. ¹H and







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¹³C NMR spectra were recorded on a JEOL JNM-ECA-400 spectrometer in a deuterated solvent (chloroform-*d*) with tetramethylsilane as an internal standard. All ¹³C NMR spectra were obtained with complete proton decoupling. IR spectra were recorded on a JASCO FT/IR-420 spectrometer by using a KBr pellet. Elemental analysis was performed on a J-SCEINCE LAB MICRO CORDER JM10.

X-ray crystallographic data of polymorph I and II were obtained by a RIGAKU Saturn 724+ CCD device using multi-layered mirror monochromatic Mo K α radiation at 93 K and a Bruker AXS SMART APEX II using monochromatic Cu K α radiation at 90 K, respectively. In these polymorphs, Friedel pairs were merged because the molecule itself was achiral and because there were not any anomalous scattering effects.

The structures were solved by a direct method (SHELXD) [14], and were refined by full-matrix least-squares method (SHELXL97) [15]. The positions of non-H atoms were obtained from difference Fourier maps and were refined anisotropically. The C-bound H atoms were obtained by calculation and were refined as riding on their parent C atoms. $U_{iso}(H)$ values of the H atoms were set at 1.2U_{eq} (parent C atoms).

2.2. Preparation of N^1 , N^1 , N^4 , N^4 -tetraphenylbuta-1,3-diyne-1,4-diamine [11, 16]

Butyllithium in hexane (12.6 ml, 20.4 mmol) was added dropwise to a solution of *N*-phenyl-*N*-(1,2,2-trichlorovinyl)aniline (3.00 g, 10.0 mmol) in dry tetrahydrofuran (225 ml) at -78 °C under an argon atmosphere. After the solution was stirred for 1 h, methanol (0.4 ml) was added to the solution. It was allowed to warm to -10 °C and was poured into water (50 ml). The water



Scheme 1. Preparation of 2. (i) *n*-BuLi/THF, 195K (ii) O₂, CuCl, TMEDA/acetone.

Table 1

Crystal data and refinement details for polymorph I and II.

Compounds	Polymorph I	Polymorph II
Compounds Empirical formula Formula weight Crystal system Crystal size Crystal color Space group a (Å) b (Å) c (Å) β (°) V (Å ³) Z Dx (Mg/m ³) μ (mm ⁻¹) Theta range for data collection (°) Limiting indices Reflections collected/unique Reflections with $I > 2\sigma(I)$	Polymorph I $C_{28}H_{20}N_2$ 384.48 <i>Monoclinic</i> 0.10 × 0.06 × 0.04 Colorless <i>P</i> 2 ₁ 16.011(5) 5.460(2) 25.366(8) 107.839(4) 21110.9(11) 4 1.210 0.07 (Mo K α) 1.8–26.0 $-14 \le h \le 15, -6 \le k \le 6, -28 \le l \le 25$ 15.635/4597 ($R_{int} = 0.0353$) 4034	Polymorph II $C_{28}H_{20}N_2$ 384.48 <i>Monoclinic</i> 0.18 × 0.10 × 0.02 Colorless <i>P</i> 2 ₁ 5.4522(3) 24.0767(13) 15.9902(9) 90.129(3) 2099.0(2) 4 1.217 0.55 (Cu K α) 2.8–66.6 $-4 \le h \le 6, -28 \le k \le 28, -18 \le l \le 18$ 9377/5659 ($R_{int} = 0.1001$) 4678
Refined parameters/restrains Coodness of fit on F^2	541/1	541/1 1 181
Reflections with $I > 2\sigma(I)$ Refined parameters/restrains	4034 541/1	4678 541/1
Goodness of fit on F^2 R_1 , wR_2	0.0668, 0.1823	1.181 0.0852, 0.2374
Largest diff. peak and hole $(e^{A^{-3}})$	0.24 and -0.32	0.33 and -0.26

Table 2	2
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Selected bond distances and angles^a of **2** accompanied by those of the optimized structure.

Distances and angles	IA	IB	IIA	IIB	Optimized
$a (\hat{A})$ $b (\hat{A})$ $c (\hat{A})$ $d (\hat{A})$ $e (\hat{A})$ $f (\hat{A})$	1.427(7), 1.431(7)	1.429(6), 1.422(7)	1.406(8), 1.405(8)	1.436(9), 1.434(8)	1.436, 1.436
	1.449(7), 1.442(7)	1.454(7), 1.428(6)	1.453(8), 1.431(8)	1.433(7), 1.450(8)	1.436, 1.436
	1.339(6), 1.342(6)	1.333(7), 1.363(7)	1.350(8), 1.359(8)	1.324(8), 1.346(8)	1.338, 1.338
	1.193(6), 1.206(6)	1.218(8), 1.182(8)	1.193(9), 1.214(9)	1.232(9), 1.198(9)	1.224, 1.224
	1.376(6)	1.372(8)	1.374(9)	1.356(9)	1.353
f (°)	177.8(6), 175.4(6)	175.7(5), 177.0(6)	178.8(7), 177.8(6)	176.8(7), 173.8(7)	179.9, 179.9
g (°)	178.8(6), 178.8(7)	179.2(6), 177.1(6)	178.7(7), 178.8(7)	179.1(7), 176.7(7)	180.0, 180.0



layer was extracted with ether (100 ml), and the combined organic layer was washed with saturated brine (30 ml), and dried over anhydrous sodium sulfate. After filtration, the solution was concentrated to give *N*-ethynyl-*N*-phenylaniline (1) as brown oil.

After a suspension of cupper (I) chloride (0.38 g, 4.04 mmol) in acetone (6.9 ml) was degassed by argon bubbling for 30 min, N,N,N',N'-tetramethylethylenediamine (TMEDA) (0.19 ml, 1.30 mmol) was added to the suspension, and it was stirred for 30 min. The supernatant solution containing CuCl-TMEDA catalyst was transferred into a solution of **1** in acetone (6 ml) at -20 °C. The

solution was stirred for 14 h under an oxygen atmosphere. The solvent was evaporated, and the residue was extracted with chloroform. The solution was washed with 5% ammonium hydroxide (50 ml), and the water layer was extracted twice with chloroform. The combined organic layer was extracted twice with chloroform. The combined organic layer was washed with water, and dried over anhydrous sodium sulfate. After the solvent was evaporated, the residue was purified by GPC to give 1.21 g (yield 63%) of **2** as a white solid. ¹H NMR (400 MHz, CDCl₃): δ 7.34 (dd, 8H, ³*J* = 8.7, 6.9 Hz, C₆H₅—H3, H5); 7.30 (dd, 8H, ³*J* = 8.7 and ⁴*J* = 1.8 Hz, C₆H₅—H2, H6); 7.13 (tt, 4H, ³*J* = 6.9 Hz, C₆H₅—H4). 13C NMR



Fig. 1. Asymmetric unit of polymorph I with atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres.



Fig. 2. Crystal packing of polymorph I.

(100 MHz, CDCl₃): 56.5; 81.8; 121.4; 124.7; 129.4; 143.2. IR (KBr): 2186, 2165 cm⁻¹. Anal. Calc. for $C_{28}H_{20}N_2$: C, 87.47; H, 5.24; N, 7.29. Found: C, 87.17; H, 5.48; N, 7.38. Mp: 124 °C(decom.).

2.3. Computational methods

DFT calculations of **2** were performed using the Spartan 04 software [17] (Wavefunction, Inc.) with B3LYP 6-31G (*d*) level. The geometrical optimization was carried out without symmetry constraints, where the initial structures for calculations were set at those obtained by crystallographical analyses. The angular dependence of the energies was calculated with the twist angle of C1—N1…N2—C3 as a parameter because the planes around N1 and N2 were planar exactly.

3. Results and discussion

3.1. Preparation of 2

Preparation of **2** is shown in Scheme 1. Reaction of *N*-phenyl-N-(1,2,2-trichlorovinyl)aniline with *n*-BuLi afforded *N*-ethynyl-*N*-phenylaniline (**1**), and a following Hay coupling reaction of **1** gave **2** in 63% yield [11,16].

3.2. Crystal polymorph of **2**

The compound **2** was found to have two crystal polymorphs (I and II) depending on a condition of recrystallization. The single crystals of polymorph I suitable for X-ray crystallography were obtained by slow evaporation of an ethanol solution at room temperature. While those of polymorph II were given by slow concentration of a dichloromethane solution in a refrigerator. Crystal data and details of refinement for polymorphs I and II are summarized in Table 1. Selected bond distances and angles of them are listed in Table 2. In both polymorphs, there are two independent molecules (A and B) in the unit cell.

Fig. 1 shows the crystal structure of polymorph I. The two independent molecules (IA and IB) have a similar structure. The structures around the nitrogens in IA (the N1/C1/C7/C13 plane; r.m.s. deviation = 0.0063 Å and the N2/C16/C17/C23 plane; r.m.s. deviation = 0.0269 Å) are planar and the twist angle between the planes

is 60.2(2)°. The dihedral angles of the planes with two phenyl groups (the C1–C6 plane and the C7–C12 plane for the N1/C1/C7/C13 plane, the C17–C22 plane and the C23–C28 plane for the N2/C16/C17/C23 plane) are $19.8(2)^\circ$, $52.3(2)^\circ$, $31.9(2)^\circ$, $44.8(3)^\circ$, respectively. Two possible conformations of two benzene rings, a propeller type [11–13,18–20] and a perpendicular type [11], are known in *N*-ethynyldiphenylamine derivatives.

The bond lengths of N1–C13 and N2–C16 are 1.339(6) Å and 1.342(6) Å, respectively, which are consistent with the reported lengths [11–13,18–20]. The distances of C13–C14, C14–C15 and C15–C16 are 1.193(6) Å, 1.376(6) Å and 1.206(6) Å, showing a clear bond alternation. The diacetylene moiety curves slightly, where the angles of N1–C13–C14, C13–C14–C15, C14–C15–C16 and C15–C16–N2 are 177.8(6)°, 178.8(6)°, 178.8(7)° and 175.4(6)°, respectively.

The structures around the nitrogens in IB (the N3/C29/C35/C41 plane; r.m.s. deviation = 0.0082 Å and the N4/C44/C45/C51 plane; r.m.s. deviation = 0.0173 Å) are almost planar and the twist angle between the planes is also $60.2(2)^{\circ}$. The dihedral angles of the planes with two phenyl groups (the C29–C34 plane and the C35–C40 plane for the N3/C29/C35/C41 plane, the C45–C50 plane and the C51–C56 plane for the N4/C44/C45/C51 plane) are 22.5(2)°, 57.2(2)°, 29.0(3)° and 45.4(2)°, respectively.

The bond lengths of N3—C41 and N4—C44 are 1.333(7) Å and 1.363(7) Å, respectively, which are consistent with the reported lengths [12–14,19–21]. The distances of C41—C42, C42—C43 and C43—C44 are 1.218(8) Å, 1.372(8) Å and 1.182(8) Å, showing a clear bond alternation. The diacetylene moiety curves slightly, where the angles of N3—C41—C42, C41—C42—C43, C42—C43—C44 and C43—C44—N4 are 175.7(5)°, 179.2(6)°, 177.1(6)° and 177.0(6)°, respectively.

Fig. 2 shows the crystal packing of polymorph I viewed from the *b* axis. The molecules (IA and IB) stack along the *b* axis with the intervals of 5.46 Å. Inclination angles between diacetylenic units and the *b* axis are 72.8(1)° for IA and 74.0(1)° for IB. These stacking parameters do not satisfy the Baughman's condition for solid-state polymerization of diacetylenes [21–23]. The molecules (IA and IB) also make arrays along the *a* axis independently to give a layered structure parallel to the *ac* plane. There are some weak C—H··· π interactions within the layer as shown in Fig. 2.



Fig. 3. Asymmetric unit of polymorph II with atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres.

Fig. 3 shows the crystal structure of polymorph II. The two independent molecules (IIA and IIB) also have a similar structure. The structures around the nitrogens in IIA (the N1/C1/C7/C13 plane; r.m.s. deviation = 0.0120 Å and the N2/C16/C17/C23 plane; r.m.s. deviation = 0.0216 Å) are planar and the twist angle between the planes is $61.7(3)^{\circ}$. The dihedral angles of the planes with two phenyl groups (the C1—C6 plane and the C7—C12 plane for the N1/C1/C7/C13 plane, the C17—C22 plane and the C23—C28 plane for the N2/C16/C17/C23 plane) are $20.1(3)^{\circ}$, $52.4(3)^{\circ}$, $29.2(3)^{\circ}$, $46.8(3)^{\circ}$, respectively.

The bond lengths of N1—C13 and N2—C16 are 1.350(8) Å and 1.359(8) Å, respectively, which are consistent with the reported lengths [11–13,18–20]. The distances of C13—C14, C14—C15 and C15—C16 are 1.193(9) Å, 1.374(9) Å and 1.214(9) Å, showing a

clear bond alternation. The diacetylene moiety curves slightly, where the angles of N1–C13–C14, C13–C14–C15, C14–C15–C16 and C15–C16–N2 are 178.8(7)°, 178.7(7)°, 178.8(7)° and 177.8(6)°, respectively.

The structures around the nitrogens in IIB (the N3/C29/C35/C41 plane; r.m.s. deviation = 0.0139 Å and the N4/C44/C45/C51 plane; r.m.s. deviation = 0.0292 Å) are almost planar and the twist angle between the planes is $58.2(3)^{\circ}$. The dihedral angles of the planes with two phenyl groups (the C29–C34 plane and the C35–C40 plane for the N3/C29/C35/C41 plane, the C45–C50 plane and the C51–C56 plane for the N4/C44/C45/C51 plane) are 22.5(3)°, $57.8(3)^{\circ}$, $32.0(3)^{\circ}$ and $43.8(3)^{\circ}$, respectively.

The bond lengths of N3–C41 and N4–C44 are 1.324(8) Å and 1.346(8) Å, respectively, which are consistent with the reported



Fig. 4. Crystal packing of polymorph II.



Fig. 5. Optimized structure for 2 (a) top view (b) side view.

lengths [13,16–20]. The distances of C41–C42, C42–C43 and C43–C44 are 1.232(9) Å, 1.356(9) Å and 1.198(9) Å, showing a clear bond alternation. The diacetylene moiety curves slightly, where the angles of N3–C41–C42, C41–C42–C43, C42–C43–C44

and C43–C44–N4 are 176.8(7)°, 179.1(7)°, 176.7(7)° and 173.8(7)°, respectively.

The molecules (IIA and IIB) stack along the *a* axis with the intervals of 5.45 Å. Inclination angles between diacetylenic units and the *a* axis are 72.1(2)° for IIA and 74.2(2)° for IIB. These stacking parameters do not satisfy the Baughman's condition for solid-state polymerization of diacetylenes. The molecules (IIA and IIB) also make arrays along the *c* axis with alternation of IIA and IIB to give a layered structure parallel to the *ab* plane. There are also some weak C—H··· π interactions within the layer as shown in Fig. 4.

The following is the difference in the molecular structures and crystal packing between polymorphs I and II. The differences in the structures are recognized at formation of 1D array. In I, the independent molecules, IA and IB, make independent arrays. However, in II, 1D array is made alternatively by IIA and IIB. Although the alternative stack of II enables close packing structure to afford relatively high density than I owing to weak $C-H\cdots\pi$ interactions, both polymorphs showed no phase transitions and decomposed at the same temperature. We do not have a clear answer concerning the reason for formation of the polymorphs.

3.3. Geometry optimization

The geometrical optimization was performed using B3LYP functional with 6-31 G (d) basis set [18]. Although the four observed structures were used as the initial structure, the optimized structures converged to the same structure. The optimized molecular structures of **2** with their numbering schemes are shown in Fig. 5. The selected geometrical parameters were listed in Tables 2 and 3.

In the case of **2**, the DFT calculation reproduced the molecular structure well except for the twist angle between two diphenylamino groups. The difference in the twist angle between observed and optimized is significant. The observed twist angles of the four kinds of molecules are $58.2-61.7^{\circ}$, but the optimized twist angle is 85.5° , which is almost good agreement with the reported geometry [24]. The observed twist angles were thought to be anomalous be-

Table	3
Table	

Dihedral angles (°) accompanied by those of the optimized structure.

Plane 1	Plane 2	IA	IB	IIA	IIB	Optimized
N1/C1/C7/C13	C1-C6	19.8(2)	-	20.1(3)	-	35.3
N1/C1/C7/C13	C7–C12	52.3(2)	-	52.4(3)	-	35.8
N2/C16/C17/C23	C17–C22	31.9(2)	-	29.2(3)	-	35.3
N2/C16/C17/C23	C23–C28	44.8(3)	-	46.8(3)	-	35.8
N3/C29/C35/C41	C29–C34		22.5(2)	_ ``	22.5(3)	-
N3/C29/C35/C41	C35–C40	-	57.2(2)	-	57.8(3)	-
N4/C44/C45/C51	C45–C50	-	29.0(3)	-	32.0(3)	-
N4/C44/C45/C51	C51–C56	-	45.4(2)	-	43.8(3)	-
N1/C1/C7/C13	N2C16/C17/C23	60.2(2)		61.7(3)	_ ``	85.5
N3/C29/C35/C41	N4/C44/C45/C51	-	60.2(2)		58.2(3)	-



Fig. 6. Angular dependence on the energy of 2.

cause the twist angles of the two amino groups in such bis-ynamines were reported almost diagonal [9,10].

The dependence of the energy on the twist angle between 10° and 90° was also calculated by DFT method as shown in Fig. 6. The co-planar structure (twist angle = 0°) could not be converged. The result indicated that the energy showed little change in the twist angle between 70° and 90° . However the energy increased with decrease of the twist angle owing to increase of electrostatic repulsion between the lone pairs of the nitrogen atoms. The difference between observed and reported twist angles was thought to originate in the intermolecular interactions as already mentioned at the crystal structures.

4. Conclusions

We have succeeded in preparations of a novel ynamine compound, N^1, N^4, N^4 -tetraphenylbuta-1,3-diyne-1,4-diamine (**2**), and characterized by using single crystal X-ray diffraction, ¹H and ¹³C NMR. The compound was found to have two crystal polymorphs and they were solved successfully. There were crystallographically two independent molecules in both polymorphs and the four kinds of molecules had almost a similar structure. The difference in crystal packing was found to originate in C—H··· π interactions.

The geometrical optimizations of **2** by DFT method gave the same structure, when the four kinds of observed structures were used as the initial structures. Although the optimized geometries showed a good agreement with the observed one except for the twist angle between two diphenylamino groups. The dependence of the energy on the twist angle was also calculated by DFT method. The results indicated that the energy showed little change in

the twist angle between 70° and 90° and that the molecular structure of **2** could change easily by packing forces within the range.

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Appendix A. Supplementary material

The supplementary crystallographic data for **2** have been deposited with the Cambridge Crystallography Data Centre, 12 Union road, Cambridge CB22 1EZ, UK (Fax: +44 1223 336 033); Email: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk) and are available free of charge on request quoting the deposition Number CCDC 921372 and CCDC 921373 for polymorphs I and II, respectively. Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.molstruc.2013.05.013.

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