Jin Li, Yan Chen, Ziqing Wu, Yali Liu, Dachuan Yin* and Zhenyu Zuo

The crystal structure of 2-phenyl-4,6-bis(prop-2-yn-1-yloxy)-1,3,5-triazine, $C_{15}H_{11}N_3O_2$

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.18 × 0.16 × 0.12 mm
Wavelength:	Cu K α radiation (1.54184 Å)
μ :	0.72 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	76.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6581, 2857, 0.013
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2328
$N(\text{param})_{\text{refined}}$:	182
Programs:	CrysAlis ^{PRO} [1], SHELX [2]

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Abstract

$C_{15}H_{11}N_3O_2$, triclinic, $P\bar{1}$ (no. 2), $a = 8.4680(5)$ Å, $b = 9.7578(5)$ Å, $c = 10.2101(6)$ Å, $\alpha = 104.578(5)^\circ$, $\beta = 111.882(6)^\circ$, $\gamma = 106.589(5)^\circ$, $V = 686.77(8)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0394$, $wR_{\text{ref}}(F^2) = 0.1261$, $T = 250(2)$ K.

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***Corresponding author: Dachuan Yin**, Institute for Special Environmental Biophysics, Key Laboratory for Space Bioscience and Space Biotechnology, School of Life Sciences, Northwestern Polytechnical University, Xi'an 710072, Shaanxi, P.R. China; and College of Pharmacy, Shaanxi University of Chinese Medicine, Xi'an, Shaanxi 712046, P.R. China, e-mail: yindc@nwpu.edu.cn

Jin Li, Ziqing Wu and Yali Liu: Institute for Special Environmental Biophysics, Key Laboratory for Space Bioscience and Space Biotechnology, School of Life Sciences, Northwestern Polytechnical University, Xi'an 710072, Shaanxi, P.R. China; and College of Pharmacy, Shaanxi University of Chinese Medicine, Xi'an, Shaanxi 712046, P.R. China. <https://orcid.org/0000-0002-7847-8616> (J. Li)

Yan Chen and Zhenyu Zuo: College of Pharmacy, Shaanxi University of Chinese Medicine, Xi'an, Shaanxi 712046, P.R. China

Source of material

To ethyl ether (150 mL), were added Mg (3.84 g, 0.160 mol) and I_2 (1.02 g, 0.004 mol) under the atmosphere of N_2 . After the mixture was stirred for 10 min, bromobenzene (25 g, 0.160 mol) was added dropwisely. When Mg disappeared, the reaction mixture was refluxed for 30 min and then cooled to -15°C . To the above solution of PhMgBr was added a solution of 2,4,6-trichloro-1,3,5-triazine (32 g, 0.175 mol) in tetrahydrofuran in 30 min. After 2 h, the reaction was quenched by 20 mL of ethanol. The final mixture was filtered and the filtrate was evaporated to get a yellow solid, which was purified by chromatography on silica gel to get the 2,4-dichloro-6-phenyl-1,3,5-triazine (27.85 g, yield 77%) as a white solid. To the solution of 2,4-dichloro-6-phenyl-1,3,5-triazine (4.52 g, 0.02 mol) in 120 mL of tetrahydrofuran was added K_2CO_3 (5.52 g, 0.04 mol) and prop-2-yn-1-ol (2.25 g, 0.04 mol). The reaction system was refluxed for 24 h and then filtered to get a brown solution. The solvent was evaporated under reduced pressure to get a yellow solid which was purified by chromatography on silica gel to afford 2-chloro-4-(prop-2-yn-1-yloxy)-6-phenyl-1,3,5-triazine as a light yellow solid. (2.75 g, 52%). ¹H-NMR (400 MHz, chloroform-d): δ [ppm] 7.59(d, $J = 8$ Hz, 2H), 7.53–7.49 (m, 3H), 5.15 (s, 4H), 2.57 (s, 2H). The crystals were obtained by recrystallization from ethyl acetate at room temperature.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O1	0.26180(14)	0.89380(11)	0.61705(11)	0.0687(3)
O2	0.62462(13)	0.72655(10)	0.92966(10)	0.0631(3)
N1	0.22943(15)	0.64959(12)	0.51991(12)	0.0562(3)
N2	0.45351(15)	0.81844(12)	0.78189(12)	0.0563(3)
N3	0.41981(14)	0.55814(11)	0.67794(11)	0.0531(3)
C1	0.0410(2)	0.34891(18)	0.27641(15)	0.0683(4)
H1	−0.0012	0.4256	0.2608	0.082 [*]
C2	−0.0468(2)	0.1998(2)	0.15944(17)	0.0851(5)
H2	−0.1485	0.1759	0.0646	0.102 [*]
C3	0.0144(3)	0.0876(2)	0.1819(2)	0.0892(6)
H3	−0.0463	−0.0130	0.1027	0.107 [*]
C4	0.1634(3)	0.12125(19)	0.3189(2)	0.0849(5)
H4	0.2052	0.0440	0.3332	0.102 [*]
C5	0.2528(2)	0.26881(16)	0.43661(18)	0.0690(4)
H5	0.3553	0.2916	0.5305	0.083 [*]
C6	0.19101(17)	0.38337(14)	0.41604(14)	0.0551(3)
C7	0.28490(16)	0.53931(13)	0.54374(13)	0.0498(3)
C8	0.49416(17)	0.69758(14)	0.78949(13)	0.0516(3)
C9	0.32045(18)	0.78521(15)	0.64352(15)	0.0547(3)
C10	0.3547(2)	1.04671(16)	0.74137(19)	0.0701(4)
H10A	0.3853	1.0356	0.8395	0.084 [*]
H10B	0.2698	1.0985	0.7269	0.084 [*]
C11	0.5252(2)	1.14199(18)	0.7470(2)	0.0739(4)
C12	0.6599(3)	1.2194(2)	0.7483(3)	0.1089(7)
H12	0.7683	1.2817	0.7494	0.131 [*]
C13	0.6787(2)	0.60103(16)	0.94380(15)	0.0637(4)
H13A	0.7487	0.5858	0.8879	0.076 [*]
H13B	0.5672	0.5033	0.9011	0.076 [*]
C14	0.79587(19)	0.64587(17)	1.10754(16)	0.0640(4)
C15	0.8877(2)	0.6802(2)	1.23930(18)	0.0789(5)
H15	0.9614	0.7077	1.3451	0.095 [*]

Experimental details

The hydrogen atoms were placed at calculated positions as riding atoms.

Comment

As an important unsaturated unit, the ethynyl group can be used to construct big π -conjugated systems, which can be widely applied into organic electronics, such as high performance organic field-effect transistors [3, 4], organic solar cells [5], organic light-emitting materials [6], organic liquid crystal materials [7] and so on. The *s*-triazine (1,3,5-triazine) ring is a remarkable building block to generate supramolecular interactions and an important unit to construct various nonlinear optical devices [8]. So designing and synthesizing of novel triazine derivatives with ethynyl conjugated units is a charming direction in organic synthesis. The structure of the title compound was characterized by ¹H-NMR and X-ray diffraction.

In the title molecule, the typical bond length [9] of C1–C6, O1–C10, N3–C7 and C11–C12 are 1.3872(19) Å, 1.4451(17) Å, 1.3428(15) Å and 1.167(3) Å, respectively. The torsion angle of C5–C6–C7–N3 is 4°, which demonstrated that the benzene ring and the 1,3,5-triazine ring are not exactly in the same plane (*cf.* the figure). Similar to the packing of other triazines weak non-classical hydrogen bonds [10] were observed. In the molecular packing, there are obvious π - π stacking interactions between the adjacent aromatic moieties. The distance between adjacent aromatic moieties is less than 3.447 Å.

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