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The crystal structure of 2-chloro-4-(prop-2-yn-1-yloxy)-6-phenyl-1,3,5-triazine, C₁₂H₈ClN₃O

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Abstract

C₁₂H₈ClN₃O, monoclinic, *C*2/*c* (no. 15), *a* = 19.2743(18) Å, *b* = 7.3515(7) Å, *c* = 17.8170(16) Å, β = 112.593(2)°, *V* = 2330.8(4) Å³, *Z* = 8, *R*_{gt}(*F*) = 0.0333, *wR*_{ref}(*F*²) = 0.0906, *T* = 153(2) K.

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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.

Source of material

Under the protection of N₂, Mg (3.84 g, 0.160 mol) and I₂ (1.02 g, 0.004 mol) were added to the solution of tetrahydrofuran (150 mL). After 10 mins bromobenzene (25 g, 0.160 mol)

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.50 × 0.40 × 0.30 mm
Wavelength:	Cu Kα radiation (1.54178 Å)
μ:	2.80 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ _{max} , completeness:	68.3°, 99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	14530, 2111, 0.040
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2101
<i>N</i> (<i>param</i>) _{refined} :	154
Programs:	Olex2 [1], SHELXL [2]

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}
C1	0.66930(7)	0.67265(17)	0.63980(7)	0.0301(3)
C2	0.72000(6)	0.56251(15)	0.55580(7)	0.0249(3)
C3	0.59710(6)	0.62754(16)	0.51169(7)	0.0251(3)
C4	0.78541(7)	0.49855(15)	0.53929(8)	0.0278(3)
C5	0.85612(7)	0.48345(18)	0.60218(9)	0.0352(3)
H5	0.8623	0.5114	0.6564	0.042*
C6	0.91738(7)	0.42754(19)	0.58524(10)	0.0435(4)
H6	0.9655	0.4178	0.6280	0.052*
C7	0.90865(8)	0.38604(19)	0.50665(11)	0.0460(4)
H7	0.9508	0.3485	0.4953	0.055*
C8	0.83849(8)	0.3990(2)	0.44420(10)	0.0422(3)
H8	0.8326	0.3694	0.3902	0.051*
C9	0.77687(7)	0.45502(18)	0.46022(8)	0.0334(3)
H9	0.7288	0.4637	0.4172	0.040*
C10	0.52095(7)	0.59178(19)	0.37221(7)	0.0318(3)
H10A	0.5245	0.4582	0.3676	0.038*
H10B	0.5611	0.6495	0.3587	0.038*
C11	0.44726(7)	0.65652(19)	0.31744(8)	0.0343(3)
C12	0.38772(8)	0.7077(2)	0.27273(9)	0.0437(4)
H12	0.3398	0.7489	0.2367	0.052*
Cl1	0.68112(2)	0.75142(5)	0.73550(2)	0.04751(16)
N1	0.60034(6)	0.68194(15)	0.58457(6)	0.0297(2)
N2	0.73128(5)	0.61566(14)	0.63188(6)	0.0291(2)
N3	0.65330(5)	0.56690(13)	0.49336(6)	0.0254(2)
O1	0.52867(4)	0.64103(12)	0.45374(5)	0.0293(2)

was added to the suspension mixture dropwisely. When the solid Mg disappeared, the reaction mixture was cooled to −15 °C. A solution of 2,4,6-trichloro-1,3,5-triazine (31.75 g, 0.175 mol) in tetrahydrofuran was added to the above solution of PhMgBr in 5 min. After 2 h, the 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 a suspension of 2,4-dichloro-6-phenyl-1,3,5-triazine (2.26 g, 0.01 mol) in 60 mL of tetrahydrofuran was added K₂CO₃ (2.76 g, 0.02 mol) and propionic alcohol (0.62 g, 0.011 mol). The mixture was refluxed for 6 h and then filtered to get a yellow 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 white solid. (1.55 g, 63%). ¹H-NMR (400 MHz, chloroform-d): δ [ppm] 8.52 (d, *J* = 7.6 Hz, 2H), 7.64 (q, *J* = 5.92 Hz, 1H), 7.54 (t, *J* = 7.12 Hz, 2H), 5.18 (t, *J* = 1.92 Hz, 2H), 2.59 (d, *J* = 2.28 Hz, 1H). The crystals were obtained by recrystallization with ethyl acetate at room temperature.

Experimental details

The data were scaled and corrected for absorption using SADABS-2016/2 [2]. The hydrogen atoms were placed at calculated positions as riding atoms.

Comment

Triazine derivatives, which have gained enormous interest in recent years, were widely used in medicine chemistry, material science, chemical industry and so on. They have become one of the most promising compounds because of its high electron-donor feature [3, 4], excellent herbicidal [5], high anticancer activities [6], antibacterial activities and good surface activities [7]. So designing and synthesizing of novel triazine derivatives obviously become an important subject in organic chemistry. In this paper, we report the synthesis of 2-chloro-4-(prop-2-yn-1-yloxy)-6-phenyl-1,3,5-triazine using 2,4,6-1,3,5-triazine as starting material. Its structure was characterized by ¹H-NMR and X-ray diffraction.

There are two crystallographically independent molecules (*cf.* the figure) in the asymmetric unit, in which all bond lengths are in normal ranges. The bond length of C11–C12 is 1.1793(19) Å and that of C4–C5 is 1.3971(18) Å. The angles of C1–N1–C3, C4–C5–C6 and C10–C11–C12 are 111.54(10)°, 119.88(14)° and 179.43(17)° respectively. The torsion angle of C9–C4–C2–N3 is 3.4°, which demonstrated that the planes of the phenyl moiety and the plane of the 1,3,5-triazine ring were not exactly coplanar. In molecular

packing diagram, there are obvious π–π stacking interactions between the adjacent aromatic moieties. The perpendicular distance between adjacent parallel aromatic moieties is less than 3.575 Å which is within normal range [8]. No classic hydrogen bonds were observed as following: C7–H7···O1 hydrogen bond (*d*(H7···O1) = 2.45 Å, C9–H9···N3 hydrogen bond (*d*(H9···N3) = 2.46 Å and C12–H12···N2 hydrogen bond (*d*(H12···N2) = 2.42 Å).

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