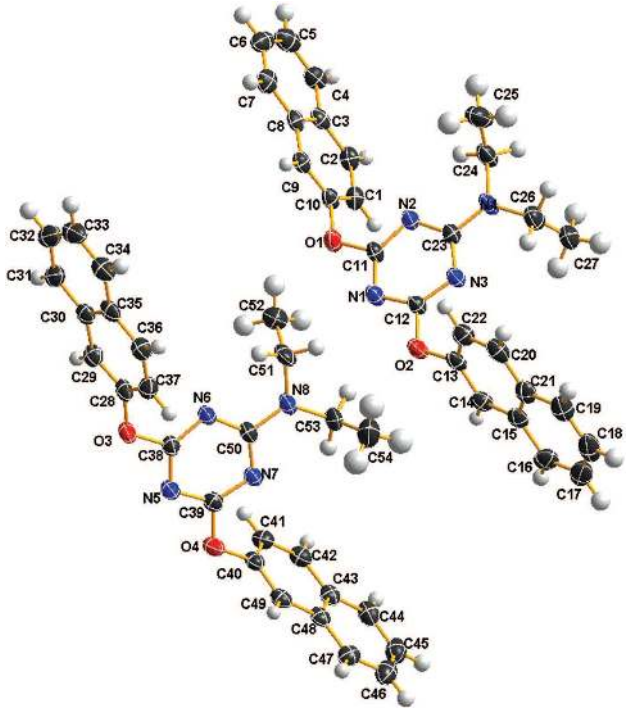
Zhenyu Zuo*, Fuhou Lei, Xiao Song and Lu Wang

The crystal structure of *N*,*N*-diethyl-4,6-bis (naphthalen-2-yloxy)-1,3,5-triazin-2-amine, $C_{27}H_{24}N_4O_2$



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Abstract

$C_{27}H_{24}N_4O_2$ monoclinic, $P2_1/c$ (no. 14), $a = 5.8073(2)$ Å, $b = 31.2564(9)$ Å, $c = 24.8105(8)$ Å, $\beta = 90.13°$, $V = 4503.5(2)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0644$, $wR_{ref}(F^2) = 0.1854$, $T = 150(2)$ K.

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***Corresponding author: Zhenyu Zuo,** Medical University of the Air Force, Xi'an 710032, China; and College of Pharmacy, Shaanxi University of Chinese Medicine, Xi'an, Shaanxi 712046, China, e-mail: 123263064@qq.com
Fuhou Lei: Guangxi Key Laboratory of Chemistry and Engineering of Forest Products, Nanning 530006, China
Xiao Song: College of Pharmacy, Shaanxi Institute of International Trade & Commerce, Xi'an, Shaanxi 712046, China
Lu Wang: Shaanxi Key Laboratory of Basic and New Herbal Medicament Research, Xi'an 712046, China

Table 1: Data collection and handling.

Crystal:	Block
Size:	0.19 × 0.18 × 0.17 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	0.67 mm^{-1}
Diffractometer, scan mode:	Bruker, φ and ω-scans
θ_{max}, completeness:	66°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13522, 6998, 0.058
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5443
$N(param)_{refined}$:	604
Programs:	Bruker programs [1], SHELX [2], Olex2 [3]

Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

To the solution of cyanuric chloride (3.69 g, 0.02 mol) and 2-naphthol (5.77 g, 0.04 mol) in tetrahydrofuran (50 mL) was added K_2CO_3 (5.52 g, 0.04 mol). The mixture was stirred at room temperature for 6 h and filtered. The filtrate was evaporated under reduced pressure to get a yellow solid, which was purified by silica gel to afford the intermediate product 2-chloro-4,6-bi(2-naphthoxyl)-1,3,5-triazine (4.90 g, yield 72%). To the solution of 2-chloro-4,6-bi(2-naphthoxyl)-1,3,5-triazine (2.00 g, 0.005 mol) in tetrahydrofuran (30 mL) was added $HN(C_2H_5)_2$ (0.439 g, 0.006 mol) at room temperature. After the mixture was stirred for 2 h, the solvent was evaporated under reduced pressure to get a white solid, which was recrystallized with petroleum ether to afford the title compound (1.88 g, yield 86%) **^{1}H NMR**: 7.86–7.76 ppm (m, 6H), 7.65 ppm (s, 2H), 7.48 ppm (dd, $J = 3.6$ HZ, $J = 4.8$ HZ, 4H), 7.36 ppm (d, $J = 8.8$ HZ, 2H) 3.45 ppm (q, $J = 6.8$ HZ, 4H), 1.08 ppm (t, $J = 6.8$ HZ, 6H). Crystals were obtained by recrystallization with petroleum ether at room temperature.

Experimental details

The data were scaled and corrected for absorption using SADABS-2016/2 [1]. The hydrogen atoms were placed at calculated positions and refined as riding atoms with fixed

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

Atom	x	y	z	U_{iso}*/U_{eq}
N1	1.0425(5)	0.11384(9)	0.24328(13)	0.0351(7)
N2	1.1338(5)	0.04868(8)	0.28875(12)	0.0315(6)
N3	0.9303(5)	0.04699(9)	0.20495(12)	0.0324(6)
N4	1.0215(5)	−0.01517(9)	0.25004(13)	0.0389(8)
N5	0.5394(5)	0.36321(9)	0.19920(13)	0.0341(7)
N6	0.4232(5)	0.29812(8)	0.24128(12)	0.0317(6)
N7	0.6241(5)	0.29671(8)	0.15714(12)	0.0319(6)
N8	0.5073(6)	0.23435(9)	0.19873(13)	0.0390(8)
O1	1.2335(5)	0.11476(8)	0.32150(11)	0.0403(6)
O2	0.8532(5)	0.11169(7)	0.16529(11)	0.0406(6)
O3	0.3489(5)	0.36429(7)	0.27750(11)	0.0394(6)
O4	0.7292(5)	0.36153(8)	0.12125(11)	0.0399(6)
C1	1.5751(6)	0.07623(12)	0.34842(16)	0.0366(8)
H1	1.6264	0.0761	0.3121	0.044*
C2	1.7076(6)	0.05881(11)	0.38842(16)	0.0364(8)
H2	1.8522	0.0464	0.3798	0.044*
C3	1.6317(6)	0.05909(11)	0.44264(16)	0.0334(8)
C4	1.7662(7)	0.04089(12)	0.48427(17)	0.0406(9)
H4	1.9108	0.0283	0.4761	0.049*
C5	1.6897(8)	0.04135(14)	0.5358(2)	0.0532(12)
H5	1.7809	0.0287	0.5634	0.064*
C6	1.4750(8)	0.06052(13)	0.54948(18)	0.0469(10)
H6	1.4252	0.0610	0.5859	0.056*
C7	1.3397(7)	0.07839(12)	0.50970(17)	0.0417(9)
H7	1.1956	0.0909	0.5186	0.050*
C8	1.4162(6)	0.07808(10)	0.45559(16)	0.0322(8)
C9	1.2810(6)	0.09590(10)	0.41331(15)	0.0324(8)
H9	1.1364	0.1088	0.4208	0.039*
C10	1.3619(6)	0.09421(11)	0.36213(15)	0.0315(8)
C11	1.1321(6)	0.09058(10)	0.28290(15)	0.0320(8)
C12	0.9434(6)	0.08907(10)	0.20643(15)	0.0315(7)
C13	0.7102(6)	0.09100(11)	0.12761(16)	0.0347(8)
C14	0.7709(6)	0.09341(11)	0.07486(16)	0.0344(8)
H14	0.9132	0.1060	0.0647	0.041*
C15	0.6197(7)	0.07690(11)	0.03513(16)	0.0357(9)
C16	0.6779(8)	0.07841(13)	−0.02049(17)	0.0462(10)
H16	0.8179	0.0912	−0.0319	0.055*
C17	0.5286(10)	0.06103(14)	−0.0574(2)	0.0587(13)
H17	0.5672	0.0617	−0.0945	0.070*
C18	0.3182(10)	0.04211(14)	−0.0413(2)	0.0601(14)
H18	0.2179	0.0303	−0.0676	0.072*
C19	0.2588(8)	0.04078(13)	0.01185(19)	0.0498(11)
H19	0.1169	0.0282	0.0224	0.060*
C20	0.3554(7)	0.05669(12)	0.10643(18)	0.0431(9)
H20	0.2137	0.0443	0.1175	0.052*
C21	0.4092(7)	0.05827(12)	0.05161(17)	0.0385(9)
C22	0.5038(7)	0.07274(12)	0.14474(17)	0.0392(9)
H22	0.4665	0.0714	0.1820	0.047*
C23	1.0287(6)	0.02736(11)	0.24803(14)	0.0304(8)
C24	1.1161(8)	−0.03971(12)	0.29590(18)	0.0467(10)
H24A	1.1979	−0.0652	0.2819	0.056*
H24B	1.2300	−0.0217	0.3152	0.056*
C25	0.9380(10)	−0.05380(15)	0.3344(2)	0.0658(14)
H25A	0.8307	−0.0733	0.3162	0.099*
H25B	0.8536	−0.0288	0.3478	0.099*
H25C	1.0113	−0.0686	0.3647	0.099*
C26[a]	0.9123(8)	−0.04028(13)	0.20664(19)	0.0478(10)
H26A[a]	0.8312	−0.0650	0.2227	0.057*
H26B[a]	0.7960	−0.0223	0.1882	0.057*
H26C[a]	0.9740	−0.0310	0.1714	0.057*
H26D[a]	0.9500	−0.0709	0.2114	0.057*
C27[a]	1.076(2)	−0.0557(3)	0.1673(4)	0.0594(18)
H27A[a]	1.1447	−0.0313	0.1484	0.089*
H27B[a]	0.9969	−0.0741	0.1412	0.089*
H27C[a]	1.1972	−0.0720	0.1856	0.089*
C27A	0.6726(18)	−0.0350(3)	0.2070(4)	0.0594(18)
H27D[a]	0.6130	−0.0418	0.2429	0.089*
H27E[a]	0.6026	−0.0542	0.1804	0.089*
H27F[a]	0.6346	−0.0053	0.1979	0.089*
C28	0.2088(6)	0.34473(11)	0.31667(16)	0.0351(8)
C29	0.2708(6)	0.34842(11)	0.36882(16)	0.0361(8)
H29	0.4130	0.3616	0.3781	0.043*
C30	0.1234(7)	0.33262(11)	0.41012(16)	0.0334(8)
C31	0.1821(7)	0.33443(12)	0.46515(16)	0.0403(9)
H31	0.3229	0.3473	0.4759	0.048*
C32	0.0373(8)	0.31770(13)	0.50331(18)	0.0502(12)
H32	0.0783	0.3192	0.5403	0.060*
C33	−0.1677(8)	0.29871(13)	0.48833(19)	0.0485(11)
H33	−0.2659	0.2869	0.5151	0.058*
C34	−0.2295(7)	0.29683(12)	0.43564(19)	0.0450(10)
H34	−0.3716	0.2838	0.4260	0.054*
C35	−0.0875(6)	0.31378(11)	0.39474(16)	0.0355(9)
C36	−0.1483(7)	0.31129(12)	0.33946(16)	0.0379(8)
H36	−0.2907	0.2987	0.3292	0.046*
C37	−0.0035(6)	0.32677(12)	0.30109(16)	0.0371(8)
H37	−0.0453	0.3255	0.2641	0.045*
C38	0.4394(6)	0.33986(10)	0.23771(15)	0.0309(7)
C39	0.6280(6)	0.33865(11)	0.16087(15)	0.0312(7)
C40	0.8579(6)	0.33952(10)	0.08214(15)	0.0315(8)
C41	1.0685(6)	0.32137(11)	0.09718(16)	0.0367(9)
H41	1.1169	0.3216	0.1338	0.044*
C42	1.2034(6)	0.30331(11)	0.05835(16)	0.0359(8)
H42	1.3464	0.2908	0.0684	0.043*
C43	1.1358(6)	0.30275(11)	0.00397(16)	0.0325(8)
C44	1.2720(7)	0.28453(11)	−0.03711(17)	0.0411(9)
H44	1.4157	0.2718	−0.0281	0.049*
C45	1.2006(7)	0.28486(13)	−0.08966(18)	0.0481(10)
H45	1.2947	0.2723	−0.1166	0.058*
C46	0.9911(8)	0.30346(13)	−0.10412(18)	0.0484(11)
H46	0.9425	0.3034	−0.1407	0.058*
C47	0.8562(6)	0.32180(12)	−0.06549(16)	0.0376(8)
H47	0.7148	0.3349	−0.0756	0.045*
C48	0.9234(6)	0.32168(10)	−0.01065(15)	0.0315(8)
C49	0.7856(6)	0.34034(11)	0.03025(15)	0.0321(8)
H49	0.6428	0.3534	0.0212	0.039*
C50	0.5183(6)	0.27707(10)	0.19906(15)	0.0307(8)
C51	0.4014(7)	0.21090(12)	0.24350(18)	0.0448(10)
H51A	0.3243	0.1849	0.2296	0.054*
H51B	0.2838	0.2291	0.2610	0.054*
C52	0.5881(9)	0.19825(14)	0.2850(2)	0.0581(12)
H52A	0.7056	0.1807	0.2674	0.087*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H52B	0.5172	0.1819	0.3143	0.087*
H52C	0.6597	0.2241	0.2997	0.087*
C53	0.6119(8)	0.20937(12)	0.15564(18)	0.0483(10)
H53A	0.7001	0.1852	0.1711	0.058*
H53B	0.7194	0.2275	0.1348	0.058*
C54	0.4187(12)	0.19216(17)	0.1181(2)	0.0810(18)
H54A	0.4861	0.1733	0.0908	0.122*
H54B	0.3417	0.2162	0.1002	0.122*
H54C	0.3063	0.1761	0.1395	0.122*

[a]Occupancy: 0.5.

isotropic displacement parameters. There is a 1/1 disorder at one of the ethyl groups (*cf*. table 2)

Discussion

Azo compounds containing a heterocyclic moiety were widely used in material field (such as molecular memory storages, nonlinear optical elements, printing system), pharmacy, agriculture, chemical industry and so on. As one of the most important class of azo compounds, *s*-triazines and its new derivative have drawn more and more attention of scientists from all over the world due to its diverse pharmacological activity [4] (such as antitumor [5], anti-inflammatory), biological activities (such as antibacteria [6, 7], antimycotic, herbicidal, insecticidal activities), high catalyst activity, abundant optoelectronic properties [8] and so on. In this paper, a new *s*-triazines derivative was synthesized by substitution reactions using 2,4,6-trichloro-1,3,5-triazine as starting material. Its structure was characterized by ^{1}H NMR and X-ray single crystal diffraction.

There are two cystallographically independent molecules (*cf*. the figure) in the asymmetric unit, in which all bond lengths are in normal ranges. The geometric parameters of both crystallographically independent molecules are similar and are in accord with another symmetrically substituted triazine [9]. In molecular packing diagram, there are obvious π-π stacking interactions between the adjacent naphthalenyl moieties. The distance between adjacent naphthalenyl moieties is less than 3.585 Å, which is within normal range [10]. No classic hydrogen bonds were observed as following: C51—H51B···N6 hydrogen bond (d(H51B···N6) = 2.36 Å), C53—H53A···O2 hydrogen bond (d(H53A···O2) = 2.47 Å) and C53—H53B···N7 hydrogen bond (d(H53B···N7) = 2.30 Å).

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