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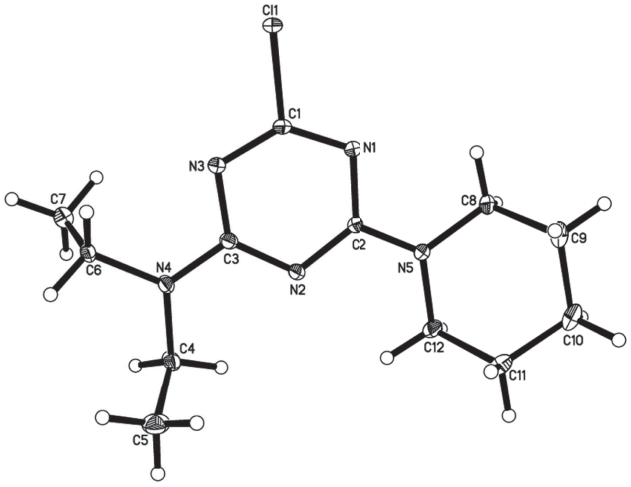
Crystal structure of 4-chloro-N,N-diethyl-6-(piperidin-1-yl)-1,3,5-triazin-2-amine, $C_{12}H_{20}ClN_5$



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Abstract

$C_{12}H_{20}ClN_5$, triclinic, $P\bar{1}$, $a = 8.7253(3)$ Å, $b = 8.9973(4)$ Å, $c = 9.3025(4)$ Å, $\alpha = 90.982(2)°$, $\beta = 110.760(1)°$, $\gamma = 94.662(2)°$, $V = 679.78(5)$ Å^3, $Z = 2$, $R_{int} = 0.071$, $wR(F^2) = 0.116$, $T = 100$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

In a two-necked flask, cyanuric chloride (3.68 gm, 20 mmol) was dissolved in 100 mL dichloromethane and cooled in an ice bath to 0 °C. A solution of diethylamine (1.46 g, 20 mmol) in 50 mL dichloromethane was added dropwise while the addition of an aq. solution of $NaHCO_3$ (1.66 gm, 20 mmol) in 50 mL distilled water. The reaction mixture was stirred at 0 °C for 2 hours and then a solution of piperidine (1.7 gm, 20 mmol) in 50 mL of dichloromethane was added. After

Table 1: Data collection and handling.

Crystal:	Colourless, block, size 0.32×0.35×0.51 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.73 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II D8 venture, φ and ω scans
$2\theta_{max}$:	66.32°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	22838, 5144
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4104
$N(param)_{refined}$:	165
Programs:	SHELX [17]

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

Atom	Site	x	y	z	U_{iso}
H(4A)	2*i*	0.0133	0.7185	0.5126	0.023
H(4B)	2*i*	−0.1548	0.6276	0.4048	0.023
H(5A)	2*i*	−0.1995	0.8790	0.4228	0.038
H(5B)	2*i*	−0.2178	0.8377	0.2491	0.038
H(5C)	2*i*	−0.0529	0.9285	0.3633	0.038
H(6A)	2*i*	−0.0325	0.5937	0.0815	0.019
H(6B)	2*i*	−0.1932	0.6222	0.1195	0.019
H(7A)	2*i*	−0.1940	0.3658	0.0694	0.032
H(7B)	2*i*	−0.1729	0.3928	0.2461	0.032
H(7C)	2*i*	−0.0166	0.3637	0.2012	0.032
H(8A)	2*i*	0.7478	0.7406	0.6431	0.019
H(8B)	2*i*	0.7439	0.7205	0.8127	0.019
H(9A)	2*i*	0.7850	1.0012	0.6822	0.025
H(9B)	2*i*	0.9232	0.9325	0.8222	0.025
H(10A)	2*i*	0.7952	1.1216	0.9120	0.028
H(10B)	2*i*	0.7729	0.9645	0.9844	0.028
H(11A)	2*i*	0.5168	1.0653	0.8969	0.024
H(11B)	2*i*	0.5263	1.0837	0.7291	0.024
H(12A)	2*i*	0.4889	0.8032	0.8621	0.020
H(12B)	2*i*	0.3500	0.8685	0.7197	0.020

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Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(1)	2*i*	0.49643(3)	0.54250(3)	0.17871(3)	0.0130(1)	0.0203(1)	0.0162(1)	0.00200(9)	0.0072(1)	−0.00544(9)
N(1)	2*i*	0.5131(1)	0.6687(1)	0.4368(1)	0.0114(4)	0.0130(4)	0.0123(4)	0.0017(3)	0.0043(3)	−0.0019(3)
N(2)	2*i*	0.2730(1)	0.7258(1)	0.4892(1)	0.0104(4)	0.0180(4)	0.0118(4)	0.0024(3)	0.0037(3)	−0.0019(3)
N(3)	2*i*	0.2455(1)	0.5993(1)	0.2502(1)	0.0110(4)	0.0162(4)	0.0127(4)	0.0021(3)	0.0045(3)	−0.0019(3)
N(4)	2*i*	0.0171(1)	0.6514(1)	0.3056(1)	0.0095(4)	0.0283(5)	0.0112(4)	0.0023(3)	0.0032(3)	−0.0034(4)
N(5)	2*i*	0.5353(1)	0.7933(1)	0.6639(1)	0.0116(4)	0.0173(4)	0.0132(4)	0.0008(3)	0.0035(3)	−0.0044(3)
C(1)	2*i*	0.4071(1)	0.6116(1)	0.3047(1)	0.0123(5)	0.0109(4)	0.0127(4)	0.0024(3)	0.0062(4)	0.0001(3)
C(2)	2*i*	0.4369(1)	0.7271(1)	0.5275(1)	0.0122(5)	0.0110(4)	0.0115(4)	0.0015(3)	0.0044(4)	0.0001(3)
C(3)	2*i*	0.1825(1)	0.6594(1)	0.3513(1)	0.0116(5)	0.0154(5)	0.0125(5)	0.0031(4)	0.0049(4)	0.0006(4)
C(4)	2*i*	−0.0665(1)	0.7044(1)	0.4057(1)	0.0118(5)	0.0333(6)	0.0127(5)	0.0034(4)	0.0057(4)	−0.0022(4)
C(5)	2*i*	−0.1408(2)	0.8503(2)	0.3558(2)	0.0262(6)	0.0267(6)	0.0281(7)	0.0035(5)	0.0166(6)	−0.0032(5)
C(6)	2*i*	−0.0872(1)	0.5770(1)	0.1575(1)	0.0096(5)	0.0259(6)	0.0115(5)	0.0025(4)	0.0018(4)	−0.0013(4)
C(7)	2*i*	−0.1207(2)	0.4100(1)	0.1696(2)	0.0167(5)	0.0254(6)	0.0200(6)	0.0043(4)	0.0039(5)	−0.0011(5)
C(8)	2*i*	0.7138(1)	0.7852(1)	0.7238(1)	0.0117(5)	0.0175(5)	0.0143(5)	0.0003(4)	0.0011(4)	−0.0023(4)
C(9)	2*i*	0.8034(2)	0.9407(1)	0.7737(2)	0.0166(5)	0.0197(5)	0.0206(6)	−0.0040(4)	0.0024(5)	−0.0005(4)
C(10)	2*i*	0.7423(2)	1.0181(1)	0.8876(2)	0.0268(6)	0.0173(5)	0.0194(6)	−0.0042(5)	0.0024(5)	−0.0047(4)
C(11)	2*i*	0.5557(2)	1.0207(1)	0.8195(1)	0.0272(6)	0.0145(5)	0.0173(5)	0.0021(4)	0.0074(5)	−0.0023(4)
C(12)	2*i*	0.4701(1)	0.8635(1)	0.7704(1)	0.0191(5)	0.0174(5)	0.0131(5)	0.0008(4)	0.0066(4)	−0.0038(4)

complete addition of piperidine a solution of $NaHCO_3$ (1.66 g, 20 mmol) in 50 mL water was added. The reaction mixture was stirred for 8 h at room temperature, and the organic layer was collected and washed with 1 M HCl (20 mL), water (20 mL), saturated solution of NaCl (20 mL), and dried over anhydrous $MgSO_4$. The solvent was removed under vacuum, and the crude product was recrystallized from dichloromethane-hexane (1:2) to afford the target product as a white crystals in 89% yield; mp 56 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.09 (t, 6H, $J = 7.2$ Hz, $2CH_3$), 1.49–1.49–1.59 (m, 6H, $2CH_2$), 3.43–3.52 (q, 4H, $2CH_2$), 3.66 (t, 4H, $J = 5.2$ Hz, $2CH_2$) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ: 12.5, 13.2, 24.5, 25.6, 41.2, 41.5, 44.3, 163.9, 169.1 ppm. Anal. calcd for $C_{12}H_{20}ClN_5$ (269.78 g/mol): C, 53.43; H, 7.47; N, 25.96%; found C, 53.61; H, 7.50; N, 26.03%.

Discussion

s-Triazine (1,3,5-triazine) represents a lead structure with a multitude of interesting application in various fields and biomedicine [1–4]. Several derivatives of *s*-triazine show antibacterial activities [5], herbicidal activities [6] or act as anti-protozoals [7], anticancer agents [8], antimalarials [9], and antimicrobials [10].

The most important reagent for obtaining this derivatives is the inexpensive commercially available cyanuric chloride (2,4,6-trichloro-1,3,5-triazine) [11]. Because of the reactivity of its chlorine atoms toward nucleophiles in the presence of a hydrochloride acceptor (usually sodium carbonate, bicarbonate, hydroxide or tertiary amines) this reagent is useful for the preparation of mono-, di- and tri-substituted 1,3,5-triazines [12–16]. Herein we describe an one pot method for the preparation of disubstituted chloro s-triazines using $NaHCO_3$ as HCl scavenger in a dichloromethane-water medium. This reaction proceeds well to afford the product in high yield (>85%) and purity.

The crystal structure of the title compound contains one molecule in the asymmetric unit. The title molecule is composed of a triazine ring substituted with pipridine and diethyl amine moieties. The pipridine ring adopts the usual chair conformation, with N5 and C10 above and below the C8, C9, C11, C12 plane. The triazine ring is planar mainly because all atoms are sp2 hybridized. The molecules packing in the crystal structure showing no classical hydrogen bond interactions.

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