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Crystal structure of 9-chloro-2,3,4,4*a*,5,6-hexahydro-1*H*-pyrido [1′,2′:1,6]pyrazino[2,3-*b*] quinoxaline, $C_{14}H_{15}ClN_4$

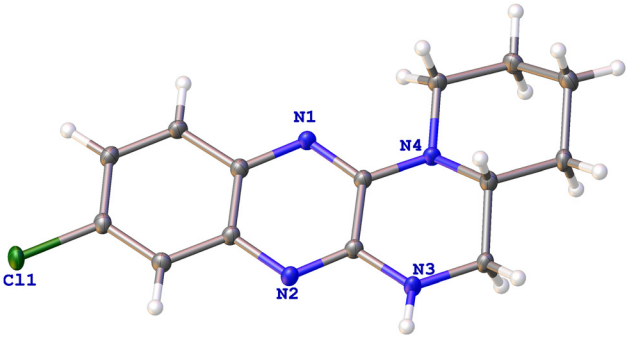

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.16 × 0.11 × 0.09 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	2.57 mm^{-1}
Diffractometer, scan mode:	XtaLAB Synergy R, ω
θ_{max}, completeness:	73.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7,238, 2,458, 0.060
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2,286
$N(param)_{refined}$:	172
Programs:	CrysAlis[PRO][1], Olex2[2], SHELX[3], WinGX/ORTEP[4]

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Abstract

$C_{14}H_{15}ClN_4$, triclinic, $P\bar{1}$ (no. 2), a = 7.4938(4) Å, b = 9.1046(5) Å, c = 11.0448(5) Å, α = 113.516(5)°, β = 91.811(4)°, γ = 109.583(5)°, V = 638.98(6) $Å^3$, Z = 2, $R_{gt}(F)$ = 0.0700, $wR_{ref}(F^2)$ = 0.1909, T = 100 K.

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The crystal structure of title compound is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

1 Source of material

2,3,6-Trichloroquinoxaline (7.08 g, 30.3 mmol), potassium bicarbonate (12.57 g, 91.0 mmol) and 2-(aminomethyl)piperidine (5.21 g, 45.6 mmol) were added successively to the solution of DMF (150 mL) stirring for 3 h at 90 °C. After cooling the reaction mixture to room temperature, water (300 mL) was added under stirring. The precipitate formed was filtered and dried. The filter cake was pulverized with ethyl acetate (5 × 20 mL) at room temperature overnight till the isomer impurity was basically removed. Finally, the crude product (pink solid) was recrystallized from dichloromethane and methanol (3:2, *v*/*v*). Colorless crystal, yield 40.8 %.

2 Experimental details

Single-crystal X-ray data of title compound was collected on a SuperNova four-circle diffractometer at 100 K.[1] Using Olex2, the structure was solved with SHELXT program and refined with the SHELXT refinement package.[2,3] Finally, the crystal structure of title compound was visualized using Ortep3 software.[4] All of the hydrogen atoms were placed in the calculated positions.

3 Comment

Tetrazanbigen (TNBG) studied by our research group is a aza-sterane compound with quinoxaline as the core structure, which is designed and synthesized by our own innovation. It has good anti-tumor activity *in vitro* and *in vivo*.[5] Based on SAR, our research team has reported a series of derivatives with improved physical properties and biological effect comparing to TNBG.[6–8] In this study, in order to

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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}
Cl1	−0.04253 (7)	0.12554 (7)	0.88545 (5)	0.0253 (3)
N1	0.3746 (3)	0.1969 (2)	0.45489 (19)	0.0206 (5)
N2	0.0894 (3)	0.3428 (2)	0.5252 (2)	0.0198 (4)
N3	0.1452 (3)	0.4434 (3)	0.3648 (2)	0.0232 (5)
H3	0.050794	0.480993	0.383064	0.028*
N4	0.4363 (3)	0.3142 (2)	0.3003 (2)	0.0210 (5)
C1	0.2728 (3)	0.1793 (3)	0.5537 (2)	0.0201 (5)
C2	0.3114 (3)	0.0878 (3)	0.6215 (2)	0.0225 (5)
H2	0.404596	0.036990	0.597245	0.027*
C3	0.2155 (3)	0.0712 (3)	0.7228 (2)	0.0236 (5)
H3A	0.243060	0.010694	0.769097	0.028*
C4	0.0770 (3)	0.1447 (3)	0.7561 (2)	0.0205 (5)
C5	0.0315 (3)	0.2317 (3)	0.6900 (2)	0.0212 (5)
H5	−0.065792	0.277621	0.712674	0.025*
C6	0.1315 (3)	0.2511 (3)	0.5886 (2)	0.0187 (5)
C7	0.1856 (3)	0.3583 (3)	0.4308 (2)	0.0184 (5)
C8	0.3338 (3)	0.2844 (3)	0.3952 (2)	0.0184 (5)
C9	0.2518 (3)	0.4750 (3)	0.2653 (2)	0.0207 (5)
H9A	0.358919	0.589792	0.308908	0.025*
H9B	0.165650	0.476905	0.196519	0.025*
C10	0.3328 (3)	0.3373 (3)	0.1976 (2)	0.0221 (5)
H10	0.223270	0.225461	0.140989	0.027*
C11	0.4636 (3)	0.3888 (3)	0.1064 (2)	0.0234 (5)
H11A	0.558803	0.508514	0.158341	0.028*
H11B	0.384966	0.386715	0.031580	0.028*
C12	0.5702 (4)	0.2671 (4)	0.0489 (3)	0.0299 (6)
H12A	0.476389	0.147711	−0.006697	0.036*
H12B	0.655359	0.304378	−0.008677	0.036*
C13	0.6885 (3)	0.2718 (3)	0.1645 (2)	0.0264 (5)
H13A	0.785368	0.390640	0.217107	0.032*
H13B	0.758466	0.193407	0.128661	0.032*
C14	0.5629 (3)	0.2174 (3)	0.2563 (2)	0.0219 (5)
H14A	0.482658	0.092197	0.208192	0.026*
H14B	0.646687	0.235919	0.336369	0.026*

diminish molecular weight and structural rigidity, an attempt was carried out to modify the main scaffold of TNBG by deducting a benzene ring.[9] It is a novel tetrazanbigen derivative in which an electron drawing group, chloride, was introduced at position C4 (see the Figure). In conclusion, we report the synthesis and crystal structure of the title compound which is a potential anti-tumor drug and consists of 2-(aminomethyl) piperidine and quinoxaline. Their atoms almost lie on the same plane. The key lengths and angles obtained from the title structure are within the normal range.[10]

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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