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Crystal structure of 2,3-dichloro-6-methoxyquinoxaline, $C_9H_6Cl_2N_2O$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.22 × 0.20 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.66 mm ⁻¹
Diffractometer, scan mode:	D8/APEX2, ω , ϕ
$\theta_{\max}$, completeness:	27.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	9602, 2096, 0.020
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1749
$N(\text{param})_{\text{refined}}$:	128
Programs:	Bruker [1], SHELX [2], Olex2 [3]

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Abstract

$C_9H_6Cl_2N_2O$, monoclinic, $P2_1/n$ (no. 14), $a = 15.597(6)$ Å, $b = 3.9346(16)$ Å, $c = 15.614(6)$ Å, $\beta = 101.834(4)^\circ$, $V = 937.8(6)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0360$, $wR_{\text{ref}}(F^2) = 0.1019$, $T = 296(2)$ 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

A mixture of 4-methoxybenzene-1,2-diamine (69.1 mg, 0.5 mmol), oxalic acid (63.4 mg, 0.5 mmol), and CH_3SO_3H (48 mg, 0.5 mmol) was stirred in toluene (1 mL) at 110 °C for 5 h. When the reaction was completed (monitored by TLC), the mixture was cooled to room temperature, and then $POCl_3$ (349.1 mg, 2.3 mol) and 25 mL DMF were slowly added

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.25179(10)	0.4648(4)	0.48867(11)	0.0409(4)
C2	0.31402(11)	0.6339(4)	0.44910(10)	0.0412(4)
C3	0.40740(10)	0.6622(4)	0.58106(10)	0.0376(4)
C4	0.34513(10)	0.4955(4)	0.62074(10)	0.0368(3)
C5	0.36405(10)	0.4283(4)	0.71134(11)	0.0406(4)
H5	0.3229	0.3229	0.7379	0.049*
C6	0.44395(10)	0.5208(4)	0.75933(11)	0.0408(4)
C7	0.50662(11)	0.6859(4)	0.71942(12)	0.0450(4)
H7	0.5607	0.7458	0.7533	0.054*
C8	0.48882(11)	0.7573(5)	0.63298(12)	0.0447(4)
H8	0.5301	0.8687	0.6078	0.054*
C9	0.41218(13)	0.2993(5)	0.89152(12)	0.0566(5)
H9A	0.3606	0.4361	0.8878	0.085*
H9B	0.4399	0.2684	0.9518	0.085*
H9C	0.3962	0.0820	0.8651	0.085*
Cl1	0.29143(3)	0.72150(14)	0.33857(3)	0.05860(18)
Cl2	0.15239(3)	0.33562(14)	0.42651(3)	0.05662(18)
N1	0.26617(8)	0.3960(4)	0.57176(9)	0.0414(3)
N2	0.38951(9)	0.7306(4)	0.49384(9)	0.0416(3)
O1	0.47154(8)	0.4657(4)	0.84651(8)	0.0524(3)

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into the mixture and warmed to 110 °C stirred for another 1 h; then the reaction was quenched with 25 mL cold water, and 100 mL ethyl acetate was added; the mixture was filtered, and the aqueous layer was separated and washed with 50 mL ethyl acetate twice. The organic layers were combined, successively washed by saturated NaCl aqueous solution,

and dried over anhydrous MgSO₄, filtered and concentrated under reduced pressure. The residue was recrystallized from butanol to give 93.1 g of 2,3-dichloro-6-methoxyquinoxaline, yield 81.7%. Crystals of the title compound were obtained by slow evaporation within 3 days.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

2,3-dichloroquinoxalines are notably one of the most important classes of nitrogen-containing heterocyclic compounds in synthetic chemistry because of their practical applications in a number of fields such as biological and photochemical [4, 5]. Consequently, the selective synthesis of such compounds is of significant importance. As part of our continuous research interest in creation of *N*-heterocycles by transfer hydrogenative coupling strategy [6–9], we reported here a one-pot facile synthesis of the title compound. Herein the crystal structure of the new compound is described to enrich the related crystal structures of 2,3-dichloro-6-methoxyquinoxaline. As in our previous study [10, 11], the new compound, built up by the C₉H₆Cl₂N₂O molecules, has been synthesized. The single-crystal structure verifies that all bond lengths are in normal ranges [12].

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