Nyameka Diko, Sizwe J. Zamisa*, Holger B. Friedrich and Mzamo L. Shoji

Crystal structure of (*E*)-*N*-(4-chlorophenyl)-1-(pyridin-2-yl)methanimine, C₁₂H₉ClN₂

Table 1: Data collection and handling.

Crystal:	Colourless rod
Size:	0.33 × 0.28 × 0.16 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.35 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	28.3°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12228, 2467, 0.023
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2212
$N(\text{param})_{\text{refined}}$:	136
Programs:	Bruker [1], SHELX [2, 3], Mercury [4]

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Abstract

C₁₂H₉ClN₂, monoclinic, $P2_1/c$ (no. 14), $a = 19.0184(3)$ Å, $b = 4.81220(10)$ Å, $c = 11.3896(2)$ Å, $\beta = 107.388(1)^\circ$, $Z = 4$, $V = 994.75(3)$ Å³, $R_{\text{gt}}(F) = 0.0284$, $wR_{\text{ref}}(F^2) = 0.0800$, $T = 100(2)$ 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

An ethanoic solution of 2-pyridinecarboxaldehyde (1.70 g, 10 mmol) was added to a solution of 4-chloroaniline (1.28 g, 10 mmol) in ethanol and the solution was refluxed for 6 h at a temperature of 100 °C. MgSO₄ was added once the mixture was cooled to remove excess water, under reduced pressure and the product was dried under vacuum overnight.

***Corresponding author: Sizwe J. Zamisa**, University of KwaZulu-Natal, School of Chemistry and Physics, Private Bag X54001, Westville Campus, Durban 4000, South Africa, e-mail: zamisas@ukzn.ac.za, sizwejamisa@gmail.com. <https://orcid.org/0000-0003-1968-381X>

Nyameka Diko, Holger B. Friedrich and Mzamo L. Shoji: University of KwaZulu-Natal, School of Chemistry and Physics, Private Bag X54001, Westville Campus, Durban 4000, South Africa

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.45707(2)	1.41456(6)	0.64710(2)	0.01753(9)
N1	0.12186(6)	0.0816(2)	0.34983(9)	0.0182(2)
N2	0.25339(5)	0.5930(2)	0.33224(9)	0.0161(2)
C1	0.07401(7)	−0.1009(3)	0.28098(12)	0.0191(2)
H1	0.045648	−0.209522	0.319827	0.023*
C2	0.06330(7)	−0.1414(3)	0.15626(11)	0.0203(2)
H2	0.029215	−0.275854	0.111379	0.024*
C3	0.10384(7)	0.0204(3)	0.09888(12)	0.0231(3)
H3	0.097428	0.000464	0.013332	0.028*
C4	0.15370(7)	0.2111(3)	0.16800(11)	0.0199(2)
H4	0.181978	0.324441	0.130781	0.024*
C5	0.16162(6)	0.2337(2)	0.29328(10)	0.0152(2)
C6	0.21388(6)	0.4290(2)	0.37348(10)	0.0159(2)
H6	0.218271	0.432816	0.458773	0.019*
C7	0.30101(6)	0.7830(2)	0.41370(10)	0.0146(2)
C8	0.29410(6)	0.8655(3)	0.52796(10)	0.0163(2)
H8	0.256255	0.788017	0.556494	0.020*
C9	0.34210(6)	1.0592(2)	0.59942(10)	0.0160(2)
H9	0.337183	1.115364	0.676547	0.019*
C10	0.39743(6)	1.1704(2)	0.55726(10)	0.0144(2)
C11	0.40509(6)	1.0939(2)	0.44428(11)	0.0165(2)
H11	0.443038	1.172019	0.416194	0.020*
C12	0.35647(6)	0.9017(2)	0.37308(10)	0.0163(2)
H12	0.360999	0.849884	0.295189	0.020*

This gave an off-white powder (1.70 g, 79%) Mp: 68.9–69.4 °C. Crystals were grown through a slow evaporation of an ethanoic solution at 25 °C. ¹H NMR (400 MHz, DMSO,

δ , ppm) δ H = 8.72 (d, 1H, J = 4.68 Hz), 8.58 (s, 1H), 8.15 (d, 1H, J = 7.72 Hz), 7.95 (td, 1H, J = 1.50, 7.96, 15.47 Hz), 7.53 (dd, 1H, J = 4.99, 12.09 Hz), 7.48 (d, 2H, J = 8.57 Hz), 7.36 (δ , 2H, d, J = 8.49 Hz). ¹³C NMR (400 MHz, DMSO, δ , ppm): 161.5, 153.8, 149.6, 149.2, 137.0, 131.0, 129.2, 125.8, 122.9, 121.3. IR(ATR)/cm⁻¹: 3051 (ar C–H), 1622 (C=N).

Experimental details

The structure was solved by the direct method using the SHELXS [2] program and refined. The visual crystal structure information was performed using Mercury [4] system software. All hydrogen atoms were placed in idealized positions and refined in riding models with U_{iso} assigned the values of 1.2 times those of their parent atoms and the distances of C–H were constrained to 0.95 Å for all the aromatic H atoms.

Comment

Pyridyl imine ligands are *N,N*-bidentate ligands that are formed *via* a condensation reaction between an aldehyde and imines. These ligands have popularity amongst chemists for their ease of synthesis, the ability to accommodate metals in various oxidation states and versatility to perform various applications [5–7]. These ligands with modifications have been coordinated to various transition metals for various catalytic reactions such as: polymerization and Heck reactions when coordinated to Pd [6, 8] and olefin epoxidation when coordinated to Re [9]. Our interests in *N,N*-bidentate ligands includes application of new complexes in biological [10] as well as catalytic studies [11].

The crystal structure of the title compound contains one molecule in the asymmetric unit with an *E* conformation around the N2=C6 bond. The pyridyl and phenyl rings are not co-planar, but rather have a dihedral angle of 17.3°. All bond angles and distances are comparable to those of closely related compounds in literature [12–17]. Intermolecular C10–Cl1...Cg_{phenyl}ⁱ interactions were observed in the crystal packing with Cl1...Cg_{phenyl}ⁱ distance of 3.56 Å and C10–Cl1...Cg_{phenyl}ⁱ angle of 92.21(4)° (symmetry code: (i) = x , 1 + y , z). Furthermore, Cl1...Cl1^{ii,iii} halogen bonding patterns (symmetry codes: (ii) = 1 – x , 1/2 + y , 3/2 – z ; (iii) = 1 – x , –1/2 + y , 3/2 – z) were also observed in the crystal packing and together with the C10–Cl1...Cg_{phenyl} interactions, form one dimensional supramolecular architectures which extend along the *b* axis.

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