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The crystal structure of 3-(5-amino-3-phenylisoxazol-4-yl)-4-chloro-3-hydroxyindolin-2-one, $C_{17}H_{12}ClN_3O_3$

https://doi.org/10.1515/ncrs-2025-0239
Received May 20, 2025; accepted June 19, 2025;
published online June 27, 2025

Abstract

$C_{17}H_{12}ClN_3O_3$, monoclinic, $P2_1/c$ (no. 14), a = 13.8689(2) Å, b = 7.19130(10) Å, c = 15.9230(2) Å, β = 107.584(1)°, V = 1513.88 Å^3, Z = 4, $R_{gt}(F)$ = 0.0338, w$R_{ref}(F^2)$ = 0.0937, T = 200(10) K.

The molecular structure is shown in the figure. Table 1 contains the crystallographic data and the list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article. CCDC no.: 2452311.

1 Source of materials

All chemicals were purchased from commercial sources and used as received without further purification, and 3-(5-amino-3-phenylisoxazol-4-yl)-4-chloro-3-hydroxyindolin-2-one was synthesized by our laboratory. The 3-(5-amino-3-phenylisoxazol-4-yl)-4-chloro-3-hydroxyindolin-2-one (0.017 g, 0.05 mmol) was dissolved in dichloromethane (5 ml) to obtain a clear colorless solution. After filtration, the obtained solution was slowly evaporated at room temperature. After 5 days, colorless and clear crystals were obtained, which were isolated by filtration and dried in air.

2 Experimental details

The SHELXL[2] refinement package and Olex2[3] software were employed to solve and refine the crystal structure. Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms. Crystal data, data collection and structure refinement details are summarized in Table 1. The list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

Table 1: Data collection and handling.

Crystal:	Clear light colourless block
Size:	0.15 × 0.13 × 0.10 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	2.43 mm^{-1}
Diffractometer, scan mode:	Bruker APEX2, φ and ω scans
θ_{max}, completeness:	71.4°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6526, 2868, 0.013
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2,791
$N(param)_{refined}$:	220
Programs:	Bruker,[1] Olex2,[2] SHELX[3]

3 Comment

Indole-2,3-dione and its derivatives constitute a class of natural products ubiquitously distributed across marine ecosystems and terrestrial biota, featuring a β-dicarbonyl framework fused with a five-membered lactam ring system, which also garnered multidisciplinary interest among researchers, driving sustained scholarly exploration over decades.[4–7] Notably, these pharmacologically active molecules demonstrate promising efficacy in antimicrobial, antitumor, anti-dementia, anti-inflammatory, and so on.[8–11]

3-(5-Amino-3-phenylisoxazol-4-yl)-4-chloro-3-hydroxyindolin-2-one was synthesized via direct condensation of indole-2,3-dione and 5-aminoisoxazole in dichloromethane under ambient stirring conditions, room temperature for 4 h, without requiring additional reagents or catalysts. The synthesis of the new compound, 3-(5-amino-3-phenylisoxazol-4-yl)-4-chloro-3-hydroxyindolin-2-one, was achieved via a one-pot method. Despite containing a chiral quaternary carbon center, the compound was found to crystallize in the symmetric space group $P2_1/c$, which suggested its racemic nature. The asymmetric unit of the crystal comprised a complete molecule (Figure), and crystallographic analysis revealed that the unit cell contained enantiomers in a racemic arrangement. The crystal structure of the title compound was analyzed and refined using the Shelxl program without applying

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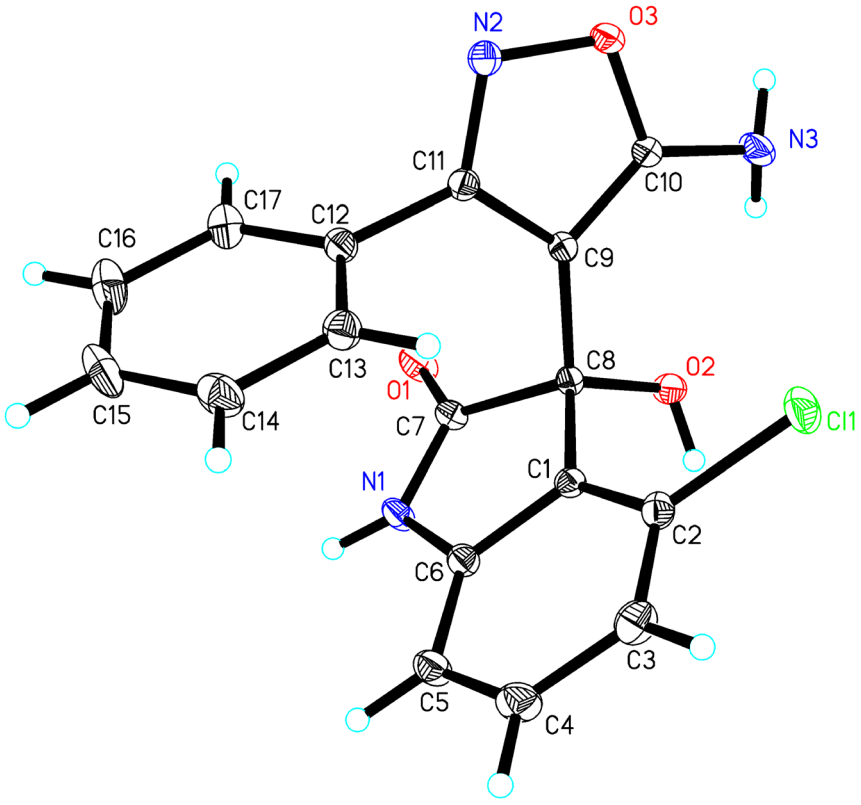

any constraints or limitations. All non-hydrogen atoms were clearly resolved in the electron density map, with refined bond lengths and angles falling within expected ranges. The imidazole moiety, 4-chloroindole-2-one fragment, and benzene ring were arranged in three planes. The dihedral angle between the isoxazole ring and the 4-chloroindolin-2-one system was 85.4°, while the dihedral angle between the isoxazole ring and the benzene ring measured 74.3°, indicating distinct conformational orientations. Bond lengths and angles are all in the expected ranges.[12,13]

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

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

Research funding: Guangdong Basic and Applied Basic Research Foundation (2023A1515012904).

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