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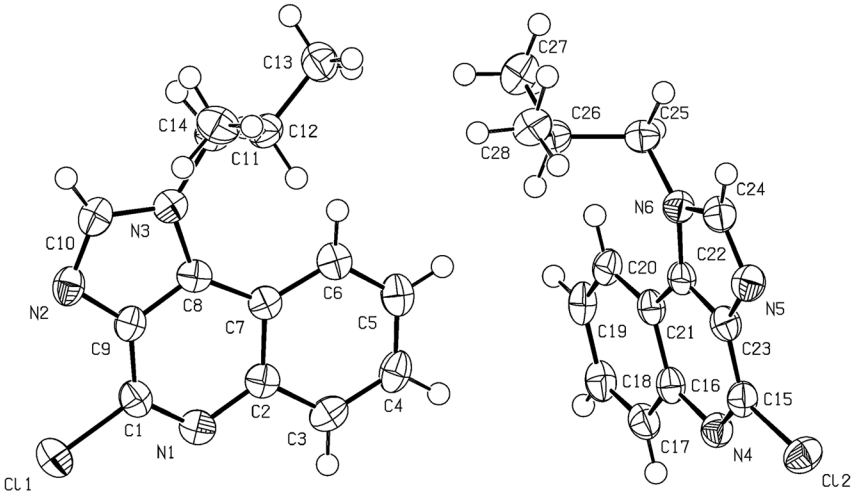
Crystal structure of 4-chloro-1-isobutyl-1*H*-imidazo, $C_{14}H_{14}ClN_3$



https://doi.org/10.1515/ncrs-2024-0389
Received October 3, 2024; accepted November 26, 2024;
published online December 9, 2024

Abstract

$C_{14}H_{14}ClN_3$, monoclinic, $P2_1/n$ (no. 14), a = 16.8486(5) Å, b = 8.1171(2) Å, c = 19.9901(7) Å, β = 113.906(4)°, V = 1499.35(15) Å^3, Z = 8, $R_{gt}(F)$ = 0.0466, $wR_{ref}(F^2)$ = 0.1206, T = 293(2) K.

CCDC no.: 2388511

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless needle
Size:	0.30 × 0.08 × 0.06 mm
Wavelength:	Cu *K*α radiation (1.54178 Å)
μ:	2.57 mm^{-1}
Diffractometer, scan mode:	ROD, Synergy Custom system, HyPix, ω
θ_{max}, completeness:	76.4°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	16054, 4902, 0.058
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3,763
$N(param)_{refined}$:	329
Programs:	CrysAlis^{PRO}[1], SHELX[2,3]

1 Source of materials

The initial step involved the nitration of 2,4-dihydroxyquinoline using concentrated nitric acid (HNO_3) at 70 °C, resulting in the formation of mononitrated 2,4-dihydroxyquinoline (Compound **1**) with a yield of 85 %. Without subsequent purification, Compound **1** underwent chlorination with phosphorus oxychloride ($POCl_3$), yielding a dichloro derivative. This intermediate was then reacted with isobutyl amine in the presence of potassium carbonate (K_2CO_3) in ethanol, affording 2-chloro-*N*-isobutyl-3-nitroquinolin-4-amine (Compound **2**) in a yield of 40 % over the two steps. Following this, a Bechamp reduction utilizing an iron powder/NH_4Cl system successfully produced 2-chloro-*N*4-isobutylquinoline-3,4-diamine in a yield

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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	−0.14650 (3)	−0.38335 (7)	0.09986 (3)	0.02883 (16)
Cl2	0.66924 (3)	−0.37758 (7)	0.19357 (3)	0.02821 (16)
N1	−0.01553 (12)	−0.3182 (2)	0.06612 (10)	0.0234 (4)
N2	−0.10512 (11)	−0.0164 (2)	0.15555 (10)	0.0246 (4)
N3	−0.00462 (11)	0.1541 (2)	0.14665 (10)	0.0219 (4)
N4	0.52559 (12)	−0.3242 (2)	0.07883 (10)	0.0228 (4)
N5	0.62693 (12)	−0.0099 (2)	0.22228 (10)	0.0245 (4)
N6	0.51923 (11)	0.1529 (2)	0.14788 (10)	0.0209 (4)
C1	−0.06511 (14)	−0.2569 (3)	0.09540 (12)	0.0233 (5)
C2	0.04954 (14)	−0.2188 (3)	0.06234 (12)	0.0218 (5)
C3	0.10246 (14)	−0.2891 (3)	0.03026 (12)	0.0248 (5)
H3	0.093260	−0.397664	0.014036	0.030*
C4	0.16728 (14)	−0.1990 (3)	0.02279 (12)	0.0261 (5)
H4	0.202093	−0.246819	0.001989	0.031*
C5	0.18096 (14)	−0.0353 (3)	0.04646 (12)	0.0259 (5)
H5	0.224265	0.025676	0.040333	0.031*
C6	0.13156 (14)	0.0368 (3)	0.07858 (12)	0.0235 (5)
H6	0.142479	0.145176	0.094817	0.028*
C7	0.06428 (13)	−0.0520 (3)	0.08724 (11)	0.0203 (5)
C8	0.00618 (14)	0.0056 (3)	0.11783 (11)	0.0207 (5)
C9	−0.05732 (13)	−0.0967 (3)	0.12333 (11)	0.0208 (5)
C10	−0.07158 (14)	0.1315 (3)	0.16792 (13)	0.0259 (5)
H10	−0.091492	0.214390	0.189350	0.031*
C11	0.04226 (14)	0.3083 (3)	0.15281 (12)	0.0238 (5)
H11A	0.005865	0.398897	0.155035	0.029*
H11B	0.053352	0.322864	0.109199	0.029*
C12	0.12865 (14)	0.3157 (3)	0.22008 (12)	0.0244 (5)
H12	0.161357	0.214631	0.222020	0.029*
C13	0.18085 (16)	0.4625 (3)	0.21278 (14)	0.0302 (5)
H13A	0.193133	0.448630	0.170232	0.045*
H13B	0.234371	0.469711	0.255501	0.045*
H13C	0.147969	0.561684	0.208018	0.045*
C14	0.11328 (17)	0.3282 (3)	0.28990 (13)	0.0309 (5)
H14A	0.167993	0.325179	0.331555	0.046*
H14B	0.078011	0.237410	0.292301	0.046*
H14C	0.084163	0.429863	0.289856	0.046*
C15	0.58071 (14)	−0.2579 (3)	0.13897 (12)	0.0226 (5)
C16	0.45605 (13)	−0.2288 (3)	0.03313 (12)	0.0207 (5)
C17	0.39750 (14)	−0.3055 (3)	−0.03137 (12)	0.0242 (5)
H17	0.405515	−0.415428	−0.040204	0.029*
C18	0.32908 (14)	−0.2197 (3)	−0.08100 (13)	0.0257 (5)
H18	0.290825	−0.271449	−0.123284	0.031*
C19	0.31647 (14)	−0.0531 (3)	−0.06818 (12)	0.0254 (5)
H19	0.270177	0.004939	−0.102338	0.030*
C20	0.37197 (14)	0.0242 (3)	−0.00568 (12)	0.0225 (5)
H20	0.362917	0.134296	0.002083	0.027*
C21	0.44280 (14)	−0.0615 (3)	0.04710 (12)	0.0202 (4)
C22	0.50633 (13)	0.0010 (3)	0.11425 (12)	0.0202 (4)
C23	0.57393 (14)	−0.0964 (3)	0.16080 (12)	0.0215 (5)
C24	0.59225 (14)	0.1371 (3)	0.21183 (13)	0.0242 (5)
H24	0.614963	0.223314	0.244737	0.029*
C25	0.46902 (14)	0.3037 (3)	0.12117 (13)	0.0237 (5)
H25A	0.456989	0.317587	0.069762	0.028*
H25B	0.503536	0.397108	0.147459	0.028*
C26	0.38325 (14)	0.3031 (3)	0.13036 (12)	0.0226 (5)
H26	0.354904	0.196538	0.113403	0.027*
C27	0.32424 (16)	0.4376 (3)	0.08266 (13)	0.0296 (5)
H27A	0.269962	0.436186	0.087713	0.044*
H27B	0.314022	0.418422	0.032438	0.044*
H27C	0.351554	0.542961	0.097759	0.044*
C28	0.39867 (15)	0.3239 (3)	0.21037 (12)	0.0273 (5)
H28A	0.439008	0.241822	0.239280	0.041*
H28B	0.344710	0.311508	0.215432	0.041*
H28C	0.422032	0.431549	0.226860	0.041*

of 87 %. Finally, treatment with triethyl orthoformate ($CH(OEt)_3$) at 60 °C in a yield of 65 %.

2 Experimental details

All hydrogen atoms were placed in idealized positions. Their U_{iso} values were set to 1.2U_{eq} of the parent atoms.

3 Comment

The 1*H*-imidazo[4,5-*c*]quinolines represent an important class of *N*-tricyclic compounds, commonly known as imidazoquinolines. These derivatives have attracted considerable attention due to their diverse biological activities, including antimalarial, anticonvulsant, and cardiovascular effects.[4–6] For example, imiquimod (Aldara) serves as an immune response modifier primarily used in the treatment of anogenital warts.[7] And, 4-chloro-1-isobutyl-1*H*-imidazo[4,5-*c*]quinoline acts as an intermediate in the synthesis of various imidazoquinoline derivatives.

The crystal structure of 4-chloro-1-isobutyl-1*H*-imidazo[4,5-*c*]quinoline is shown in the above figure. The bond lengths of C1–Cl1, C1=N1, C9=N2, and C10=N3 in the title molecule are 1.744, 1.298, 1.382 and 1.370 Å, respectively, which are similar with its analogs.[8,9] The thirteen atoms of C1–C10, N1–N3 are in the same plane. Besides, the distance between two adjacent molecules in parallel arrangement is 3.339 Å, indicating π–π stacking interaction between the aromatic cycles.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.
Research funding: the project ZR2024QE449 supported by Shandong Provincial Natural Science Foundation and the Research Program QIT23TP002 of Qilu Institute of Technology.

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

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