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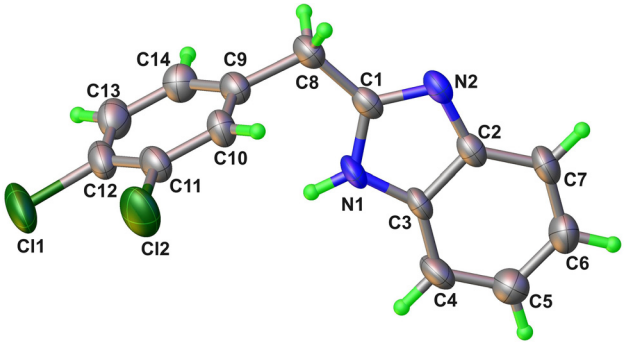
# The crystal structure of 2-(3,4–dichlorobenzyl)-1*H*-benzimidazole, $C_{14}H_{10}Cl_2N_2$

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**Abstract**

$C_{14}H_{10}Cl_2N_2$, monoclinic, $P2_1/n$ (no. 14), $a$ = 9.4145(9) Å, $b$ = 14.8530(15) Å, $c$ = 10.0405(11) Å, $\beta$ = 116.714(5)°, $V$ = 1254.1(2) Å$^3$, $Z$ = 4, $R_{gt}(F)$ = 0.0653, $wR_{ref}(F^2)$ = 0.1901, T = 293 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.

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Colorless block |
| Size: | 0.32 × 0.14 × 0.06 mm |
| Wavelength: | Mo *K*α radiation (0.71073 Å) |
| $\mu$: | 0.50 mm$^{-1}$ |
| Diffractometer, scan mode: | $\varphi$ and $\omega$ |
| $\theta_{max}$, completeness: | 25.0°, >99 % |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 5922, 2198, 0.081 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\sigma(I_{obs})$, 1284 |
| $N(param)_{refined}$: | 163 |
| Programs: | Bruker [1], Olex2 [2], SHELX [3] |

## 1 Source of material

All chemicals were purchased from commercial sources and used as received. The 2-(3,4–dichlorobenzyl)-1*H*-benzimidazole was synthesized according to reported method by direct condensation of carboxylic acids with benzene-1,2-diamine catalyst by boron acid [4, 5]. To a stirred solution of benzene-1,2-diamine (216 mg, 2.0 mmol) in 10 mL xylenes was added 2-(3,4-dichlorophenyl) acetic acid (612 mg, 3 mmol) and boric acid $B(OH)_3$ (12 mg, 0.2 mmol). After refluxing for 16 h, the volatile in the solution was removed under vacuum, and the residue was extracted with EtOAc (50 mL). The resulting solution was washed with saturated $NaHCO_3$ solution twice, dried over anhydrous $Na_2SO_4$ and then concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (ethyl acetate:hexane = 1:10) to afford the title compound 365 mg (yield 65 %). Crystals were obtained by recrystallization the product from methanol.

## 2 Experimental details

Coordinates of hydrogen atoms were included using constraints or restraints. Their $U_{iso}$ values were set to 1.2$U_{eq}$ of the parent atoms.

## 3 Comment

Nitrogen-containing heterocyclic compounds are very common organic compounds widely used in pesticides and medicine [6, 7]. *N*–Arylated imidazoles are compounds of particular significance owing to their excellent applications in plant growth regulator, therapeutic agent, herbicide, etc. [8–10]. Due to its potential applications in pharmaceutical activities, the synthesis of imidazoles has attracted wide interests among scientists [11–13]. As displayed in the Figure, there is one molecule in the asymmetric unit of the title compound. The C1–N2 bond length of 1.320(5) Å is shorter than the C1–N1, with 1.362(5) Å, which suggests that the C1–N2 bond is the double bond. In the crystal,

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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 | 1.60722 (14) | 0.07615 (10) | 0.70612 (16) | 0.0807 (5) |
| Cl2 | 1.30413 (16) | −0.03238 (10) | 0.67010 (15) | 0.0782 (5) |
| N1 | 0.9358 (4) | 0.2666 (2) | 0.3802 (4) | 0.0431 (9) |
| H1 | 1.033704 | 0.264851 | 0.446187 | 0.052* |
| N2 | 0.7183 (4) | 0.2377 (2) | 0.1699 (4) | 0.0416 (9) |
| C1 | 0.8715 (5) | 0.2208 (3) | 0.2487 (4) | 0.0385 (10) |
| C2 | 0.6823 (4) | 0.2977 (3) | 0.2569 (4) | 0.0403 (10) |
| C3 | 0.8176 (4) | 0.3160 (3) | 0.3893 (4) | 0.0384 (9) |
| C4 | 0.8163 (5) | 0.3729 (3) | 0.4974 (5) | 0.0529 (12) |
| H4 | 0.908079 | 0.384136 | 0.584985 | 0.063* |
| C5 | 0.6731 (6) | 0.4123 (3) | 0.4701 (5) | 0.0580 (13) |
| H5 | 0.667753 | 0.450952 | 0.540517 | 0.070* |
| C6 | 0.5362 (5) | 0.3950 (3) | 0.3381 (5) | 0.0568 (12) |
| H6 | 0.441316 | 0.422585 | 0.322560 | 0.068* |
| C7 | 0.5375 (5) | 0.3381 (3) | 0.2302 (5) | 0.0510 (12) |
| H7 | 0.445531 | 0.326935 | 0.142780 | 0.061* |
| C8 | 0.9642 (5) | 0.1583 (3) | 0.2010 (5) | 0.0469 (11) |
| H8A | 0.975925 | 0.185512 | 0.118618 | 0.056* |
| H8B | 0.904384 | 0.102982 | 0.164837 | 0.056* |
| C9 | 1.1279 (5) | 0.1355 (3) | 0.3236 (5) | 0.0430 (10) |
| C10 | 1.1430 (5) | 0.0706 (3) | 0.4288 (5) | 0.0458 (11) |
| H10 | 1.053365 | 0.040091 | 0.420999 | 0.055* |
| C11 | 1.2898 (5) | 0.0511 (3) | 0.5446 (5) | 0.0485 (11) |
| C12 | 1.4234 (5) | 0.0967 (3) | 0.5565 (5) | 0.0517 (11) |
| C13 | 1.4102 (5) | 0.1588 (3) | 0.4512 (6) | 0.0562 (12) |
| H13 | 1.500251 | 0.188258 | 0.457714 | 0.067* |
| C14 | 1.2634 (5) | 0.1778 (3) | 0.3355 (5) | 0.0502 (11) |
| H14 | 1.255515 | 0.220020 | 0.264104 | 0.060* |

the benzoimidazole parts exhibit a planar structure. The average length of the C–Cl bond is 1.732 Å. Bond lengths and angles are as expected [14, 15].

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

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# References

1. BRUKER. SAINT, APEX2 and SADABS; Bruker AXS Inc.: Madison, Wisconsin, USA, 2009.
2. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, *42*, 339–341.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr*. 2015, *C71*, 3–8.
4. Boggu P. R., Eeda V., Manickam M., Kwak D., Youngsoo K., Jung S.-H. Exploration of 2-benzylbenzimidazole scaffold as novel inhibitor of NF-κB. *Biorg. Med. Chem.* 2016, *24*, 1872–1878.
5. Maraăs N., Koček M. Boric acid-catalyzed direct condensation of carboxylic acids with benzene-1,2-diamine into benzimidazoles. *Helv. Chim. Acta* 2011, *94*, 1860–1874.
6. Bartoli G., Dalpozzo R., Nardi M. Applications of Bartoli indole synthesis. *Chem. Soc. Rev.* 2014, *43*, 4728–4750.
7. Song H., Zhou H., Shen Y., Wang H., Song H., Cai X., Xu C. HFIP as protonation reagent and solvent for regioselective alkylation of indoles with all-carbon centers. *J. Org. Chem.* 2022, *87*, 1086–1097.
8. Chou W. L., Chang C. Y., Liu H. M., Yang K. C., Wu C. C. Supercritical fluid extraction of imidazole drugs from cosmetic and pharmaceutical products. *J. Food Drug Anal.* 2007, *15*, 25–32.
9. Ali I., Lone M. N., Aboul-Enein H. Y. Imidazoles as potential anticancer agents. *MedChemComm* 2017, *8*, 1742–1773.
10. Lundevall F. J., Bjørsvik H.-R. Lithiation and alkylation of the imidazole backbone. *Eur. J. Org. Chem.* 2023, *26*, e202201504.
11. Lin S., Cui J., Chen Y., Li Y. Copper-catalyzed direct cycloaddition of imidazoles and alkenes to trifluoromethylated tricyclic imidazoles. *J. Org. Chem.* 2021, *86*, 15768–15776.
12. Shabalin D. A., Camp J. E. Recent advances in the synthesis of imidazoles. *Org. Biomol. Chem.* 2020, *18*, 3950–3964.
13. Ananthu S., Aneeja T., Anilkumar G. N-arylation of imidazoles: an overview. *ChemistrySelect* 2021, *6*, 9794–9805.
14. Li Y., Zhang X., Weng X., Zhuang X. The crystal structure of 4-(2,4-dichlorophenyl)-2-(4-fluorophenyl)-5-methyl-1*H*-imidazole, $C_{16}H_{11}Cl_2FN_2$. *Z. Kristallogr. N. Cryst. Struct.* 2023, *238*, 957–958.
15. Infantes L., Mo O., Yanez M., Roux M. V., Jimenez P., Davalos J. Z., Temprado M., da Silva M. A. V. R., da Silva M., da Silva D. M. C. R., Amaral L. M. P. F., Cabildo P., Claramunt R., Elguero J. Substituent effects on enthalpies of formation of nitrogen heterocycles: 2–substituted benzimidazoles and related compounds. *J. Phys. Chem. A* 2006, *110*, 2535–2544.