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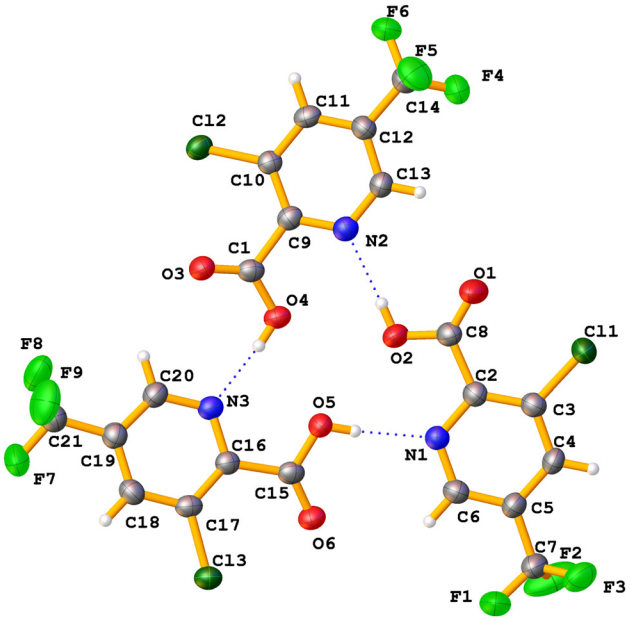
Crystal structure of 3-chloro-5-(trifluoromethyl) pyridine-2-carboxylic acid, $C_7H_3ClF_3NO_2$



https://doi.org/10.1515/ncrs-2024-0401
Received October 10, 2024; accepted November 4, 2024;
published online November 14, 2024

Abstract

$C_7H_3ClF_3NO_2$, triclinic, $P\bar{1}$ (no. 2), a = 5.8837(5) Å, b = 14.1634(14) Å, c = 15.3120(15) Å, α = 92.667(5)°, β = 100.364(5)°, γ = 99.475(5)°, V = 1,234.2(2) Å^3, Z = 6, $R_{gt}(F)$ = 0.0458, $wR_{ref}(F^2)$ = 0.1012, T = 298 K.

CCDC no.: 2390150

A part of 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:	Colourless block
Size:	0.25 × 0.22 × 0.19 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.49 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	25.4°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	23,454, 4,471, 0.070
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2,937
N(param)$_{refined}$:	382
Programs:	Bruker,[1] Olex2,[2] Shelx[3,4]

1 Source of materials

3-Chloro-5-(trifluoromethyl)pyridine-2-carboxylic acid (HL) and tetrahydrofuran (THF) were purchased from Thermo Fisher Scientific Inc. Deionized water is prepared by the purification equipment purchased by our college. The preparation of the crystal structure for the title compound is very simple. 0.5 g (2.217 mmol) of HL was added to a 10 mL glass tube with a mixed solution of THF and deionized water (6 mL, 3:3, v/v). Subsequently, the glass tube is positioned in a dark area with adequate ventilation. The slow evaporation of the solution yielded colourless block-shaped crystals within 1 week, yielding 43 % (based on the HL organic ligand).

2 Experimental details

The crystal structure of the complex was solved using the Shelxt program with intrinsic phasing method and refined by the full-matrix least-squares method with the Shelxl program. All non-hydrogen atoms (Ca–, C–, N– and O–atom) were refined with anisotropic displacement parameters, while the hydrogen atoms were placed in idealized positions with isotropic thermal parameters.

3 Comment

As a molecular building block, 3-chloro-5-(trifluoromethyl) pyridine-2-carboxylic acid (HL) features a distinctive combination of a chlorine atom and a trifluoromethyl group attached to a pyridinecarboxylic ring, which endows it with unique electronic properties and potential applications in

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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}
C1	0.2636 (6)	0.4687 (3)	0.6837 (2)	0.0365 (8)
C2	0.8368 (6)	0.1578 (2)	0.6904 (2)	0.0373 (8)
C3	0.9119 (6)	0.0732 (2)	0.6699 (2)	0.0385 (8)
C4	1.1063 (6)	0.0491 (2)	0.7237 (2)	0.0401 (8)
H4A	1.156252	−0.008325	0.711773	0.048*
C5	1.2247 (6)	0.1117 (2)	0.7956 (2)	0.0399 (8)
C6	1.1453 (6)	0.1965 (2)	0.8104 (2)	0.0406 (8)
H6	1.226882	0.239396	0.857844	0.049*
C7	1.4285 (7)	0.0856 (3)	0.8565 (3)	0.0502 (10)
C8	0.6196 (6)	0.1883 (2)	0.6393 (2)	0.0386 (8)
C9	0.1033 (6)	0.4014 (2)	0.6102 (2)	0.0351 (8)
C10	−0.1115 (5)	0.4193 (2)	0.5669 (2)	0.0330 (8)
C11	−0.2349 (6)	0.3596 (2)	0.4933 (2)	0.0372 (8)
H11	−0.379362	0.370605	0.463570	0.045*
C12	−0.1396 (6)	0.2839 (2)	0.4653 (2)	0.0391 (8)
C13	0.0724 (6)	0.2683 (2)	0.5136 (2)	0.0386 (8)
H13	0.134858	0.216116	0.495539	0.046*
C14	−0.2575 (7)	0.2212 (3)	0.3826 (3)	0.0484 (10)
C15	1.0043 (6)	0.4462 (2)	0.8663 (2)	0.0356 (8)
C16	0.9620 (5)	0.5482 (2)	0.8747 (2)	0.0339 (8)
C17	1.1228 (6)	0.6228 (2)	0.9248 (2)	0.0362 (8)
C18	1.0706 (6)	0.7144 (3)	0.9249 (2)	0.0406 (8)
H18	1.177054	0.765439	0.957101	0.049*
C19	0.8602 (6)	0.7296 (2)	0.8772 (2)	0.0404 (8)
C20	0.7060 (6)	0.6516 (2)	0.8309 (2)	0.0402 (8)
H20	0.561746	0.661227	0.799770	0.048*
C21	0.7969 (7)	0.8275 (3)	0.8755 (3)	0.0505 (10)
Cl1	0.76892 (17)	−0.00676 (7)	0.58095 (7)	0.0544 (3)
Cl2	−0.23564 (15)	0.51333 (6)	0.60179 (6)	0.0408 (2)
Cl3	1.38506 (15)	0.60654 (6)	0.98768 (6)	0.0443 (2)
F1	1.5735 (5)	0.1618 (2)	0.8981 (2)	0.1038 (11)
F2	1.3637 (5)	0.0293 (3)	0.91657 (19)	0.1004 (11)
F3	1.5587 (4)	0.03892 (18)	0.81383 (16)	0.0657 (7)
F4	−0.2537 (4)	0.12856 (15)	0.39419 (15)	0.0596 (6)
F5	−0.1496 (4)	0.24165 (18)	0.31420 (15)	0.0675 (7)
F6	−0.4806 (4)	0.23026 (16)	0.35713 (15)	0.0631 (7)
F7	0.8980 (5)	0.88286 (18)	0.94834 (18)	0.0886 (9)
F8	0.5677 (4)	0.82491 (16)	0.86657 (19)	0.0753 (8)
F9	0.8588 (5)	0.87280 (17)	0.80740 (19)	0.0813 (8)
N1	0.9575 (5)	0.21897 (19)	0.75968 (18)	0.0376 (7)
N2	0.1898 (5)	0.3246 (2)	0.58444 (19)	0.0383 (7)
N3	0.7554 (5)	0.5630 (2)	0.82903 (18)	0.0366 (7)
O1	0.5135 (5)	0.1527 (2)	0.56895 (19)	0.0727 (9)
O2	0.5640 (4)	0.25987 (19)	0.68238 (18)	0.0547 (7)
H2	0.452276	0.278000	0.651887	0.082*
O3	0.2722 (4)	0.55476 (18)	0.68516 (16)	0.0460 (6)
O4	0.3963 (4)	0.42388 (17)	0.73961 (15)	0.0408 (6)
H4	0.506791	0.462684	0.768042	0.061*
O5	0.8787 (4)	0.40116 (16)	0.79141 (15)	0.0422 (6)
H5	0.899320	0.345340	0.788287	0.063*
O6	1.1332 (4)	0.41117 (17)	0.92094 (15)	0.0436 (6)

the medical field.[5–8] However, the crystal structure of HL has not been exploited. Hence, to better understand the nature of HL, the title structure was synthesized. HL crystallizes in the triclinic system with $P\bar{1}$ space group. The asymmetric unit contains three crystallographically independent HL organic ligands. The bond lengths other than those of C–C bonds, i.e., the C–N, C–O, C–Cl and C–F distances are of 1.326(4)–1.350(4) Å, 1.187(4)–1.311(4) Å, 1.722(3)–1.728(3) Å and 1.312(5)–1.338(4) Å. The bond lengths observed in this study are within the normal range, comparable to those reported in crystal structures containing similar atoms.[9–12] Notably, the hydrogen atoms on the carboxyl groups of the HL ligands remain intact. For this reason, the three originally independent HL ligands formed a non-covalently bonded cyclic structure through the intermolecular interaction of O–H···N hydrogen bonding (O2–H2···N2, O4–H4···N3 and O5–H5···N1; distance range between donor and acceptor atoms: 2.7329(3)–2.7492(3) Å and finally resulted in a 3D extended network with the help of other short contacts (O–H···O, C–H···O and C–H···F).

Acknowledgments: Special thanks are given to Xiang-Yan Kong for his valuable suggestions on the writing logic and content of the manuscript.
Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.
Research funding: Weifang Science and Technology Development Plan Project (No. 2019GX007).
Conflict of interest: The authors declare no conflicts of interest regarding this article.

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