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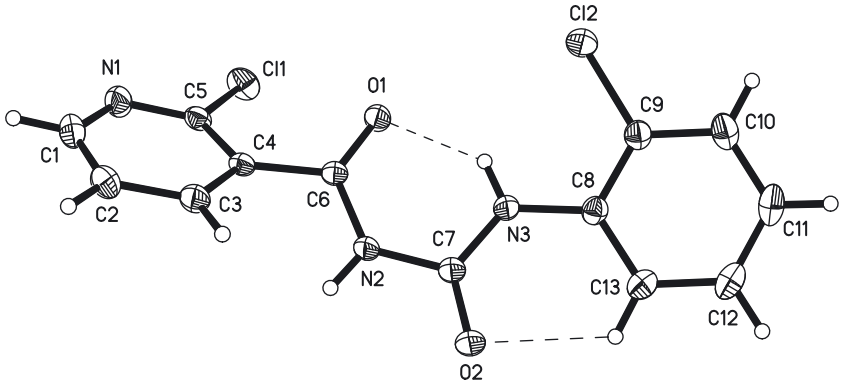
The crystal structure of 2-chloro-*N*-((2-chlorophenyl)carbamoyl)nicotinamide, $C_{13}H_9Cl_2N_3O_2$



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Abstract

$C_{13}H_9Cl_2N_3O_2$, monoclinic, $P2_1/n$ (no. 14), a = 7.918(2) Å, b = 12.766(3) Å, c = 13.767(4) Å, β = 103.413(9)°, V = 1353.6(6) Å^3, Z = 4, $R_{gt}(F)$ = 0.0382, $wR_{ref}(F^2)$ = 0.1019, T = 296(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 materials

According to our previous work [5], to a solution of 2-chloronicotinoyl isocyanate (0.18 g, 1 mmol) in dichloromethane (10 mL), 2-chloroaniline (0.12 g, 1.1 mmol) was added dropwise. Then the mixture was stirred at room temperature for overnight. After the reaction was completed, the solvent was evaporated to give a crude product. The solid was recrystallized from ethanol as colorless block crystals.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size	0.48 × 0.44 × 0.32 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.48 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	30.9°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	32,412, 4273, 0.033
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3335
$N(param)_{refined}$:	181
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Experimental details

Hydrogen atoms were included using riding models implemented in the SHELXL software [3, 4]. Their U_{iso} values were set to 1.2U_{eq} of the parent atoms. Using Olex2 [2], the structure was solved with the ShelXS [3] structure solution program using Direct Methods and refined with the ShelXL [4] refinement package.

Comment

Nicotinamide compounds exhibited excellent biological activity, such as herbicidal activity [6], fungicidal activity [7]. Meanwhile, carboxamide group is an active group in many bioactive molecules [8].

The crystal structure of title compound is similar with our reported structure [9] of 2-chloro-*N*-(o-tolylcarbamoyl) nicotinamide. The bond lengths and bond angles of the pyridine ring and the phenyl ring are all in the normal ranges

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

Atom	*x*	*y*	*z*	U_{iso}^*/U_{eq}
Cl1	0.49900 (5)	0.86789 (3)	−0.27109 (3)	0.04848 (12)
Cl2	0.22056 (6)	0.93774 (3)	0.10238 (3)	0.04862 (12)
O1	0.36461 (15)	0.81035 (7)	−0.09306 (7)	0.0425 (2)
O2	0.47608 (18)	0.56446 (8)	0.10480 (8)	0.0539 (3)
N1	0.34325 (19)	0.72852 (10)	−0.39500 (9)	0.0452 (3)
N2	0.42703 (17)	0.64016 (8)	−0.04786 (8)	0.0372 (3)
H2	0.4522	0.5809	−0.0707	0.045*
N3	0.39143 (16)	0.73604 (9)	0.08958 (8)	0.0356 (3)
H3	0.3648	0.7854	0.0462	0.043*
C1	0.2621 (2)	0.63709 (13)	−0.42157 (12)	0.0496 (4)
H1	0.2280	0.6213	−0.4892	0.059*
C2	0.2266 (2)	0.56557 (12)	−0.35457 (12)	0.0448 (3)
H2A	0.1738	0.5019	−0.3760	0.054*
C3	0.27176 (19)	0.59110 (10)	−0.25405 (11)	0.0375 (3)
H3A	0.2480	0.5446	−0.2069	0.045*
C4	0.35281 (17)	0.68634 (9)	−0.22343 (9)	0.0298 (2)
C5	0.38701 (18)	0.75076 (10)	−0.29879 (10)	0.0323 (3)
C6	0.38403 (17)	0.71977 (10)	−0.11662 (9)	0.0315 (3)
C7	0.43489 (19)	0.64383 (10)	0.05530 (10)	0.0353 (3)
C8	0.38419 (17)	0.76180 (10)	0.18816 (10)	0.0320 (3)
C9	0.30675 (18)	0.85691 (10)	0.20371 (10)	0.0348 (3)
C10	0.2969 (2)	0.88985 (13)	0.29820 (12)	0.0466 (4)
H10	0.2449	0.9535	0.3068	0.056*
C11	0.3652 (2)	0.82690 (16)	0.37937 (12)	0.0546 (4)
H11	0.3589	0.8478	0.4432	0.066*
C12	0.4429 (2)	0.73286 (15)	0.36566 (11)	0.0501 (4)
H12	0.4890	0.6910	0.4207	0.060*
C13	0.4533 (2)	0.69971 (12)	0.27125 (11)	0.0408 (3)
H13	0.5063	0.6363	0.2634	0.049*

[10, 11]. The torsion angle of C4–C6–N2–C7, C6–N2–C7–N3 and N2–C7–N3–C8 is −168.28(13)°, 1.5(2)° and 178.67(13)° respectively, which indicated that the two carbonyl groups are opposite and the acyl urea bridge is nearly in the same plane, which is the same as in the reported structure of 2-chloro-*N*-(p-tolylcarbamoyl)nicotinamide [12].

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

References

1. BRUKER. SAINT, APEX2 and SADABS; Bruker AXS Inc.: Madison, WI, 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. Shelxtl – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
4. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
5. Zhang P. P., Wang Q., Min L. J., Wu H. K., Weng J. Q., Tan C. X., Zhang Y. G., Liu X. H. Synthesis, crystal structure, fungicidal activity and molecular docking of nicotinic acyl urea derivatives. *J. Mol. Struct.* 2020, *1205*, 127485.
6. Yu C. S., Wang Q., Bajsa-Hirschel J., Cantrell C., Duke S. O., Liu X. H. Synthesis, crystal structure, herbicidal activity and SAR study of novel *N*-(arylmethoxy)-2-chloronicotinamides derived from nicotinic acid. *J. Agric. Food Chem.* 2021, *69*, 6423–6430.
7. Sun G. X., Wang Q., Min L. J., Han L., Liu X. H. Synthesis, crystal structure, fungicidal activities and molecular docking of acyl urea derivatives containing 2-chloronicotine motif. *Chin. J. Struct. Chem.* 2022, *41*, 114–122.
8. Pinheiro A. C., de Souza M. V. N., Wardell J. L., Wardell S. M. S. V. Structures of *N*-(arenecarbonyl)-*N*, *N*′-dicyclohexylureas, cyclohexyl–NH–C(O)–N(cyclohexyl)–C(O)Ar, (Ar = Ph, pyridin-3-yl and pyrazinyl). *Z. Kristallogr. Cryst. Mater.* 2012, *227*, 166–177.
9. Min L. J., Wang Q., Tan C. X., Weng J. Q., Liu X. H. Synthesis, crystal structure and fungicidal activity of 2-chloro-*N*-(*o*-tolylcarbamoyl)nicotinamide. *Chin. J. Struct. Chem.* 2020, *39*, 452–458.
10. Sun G. X., Min L. J., Han L., Liu X. H. The crystal structure of (*E*)-3-chloro-2-(2-(4-methylbenzylidene)hydrazinyl)pyridine, $C_{13}H_{12}ClN_3$. *Z. Kristallogr. N. Cryst. Struct.* 2022, *237*, 129–131.
11. Zheng B. Y., Sun N. B., Chen X. S., Liu X. H. The crystal structure of (*E*)-3-chloro-2-(2-(4-fluorobenzylidene)hydrazinyl) pyridine, $C_{12}H_9ClFN_3$. *Z. Kristallogr. N. Cryst. Struct.* 2022, *237*, 355–356.
12. Chen W. T., Wang Q., Min L. J., Wu H. K., Weng J. Q., Tan C. X., Zhang Y. G., Hu B. Z., Liu X. H. Synthesis, crystal structure, fungicidal activity, molecular docking, and density functional theory study of 2-chloro-*N*-(*p*-tolylcarbamoyl)nicotinamide. *Indian J. Heterocycl. Chem.* 2019, *29*, 429–435.