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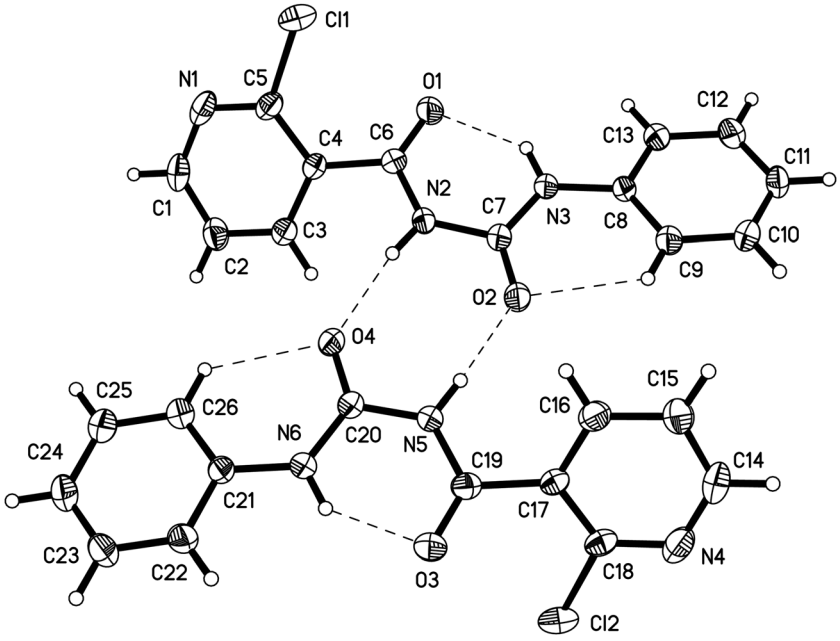
Hydrogen bonded dimers in the crystal structure of 2-chloro-*N*-(phenylcarbamoyl)nicotinamide, $C_{26}H_{20}Cl_2N_6O_4$



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Abstract

$C_{26}H_{20}Cl_2N_6O_4$, orthorhombic, *Pbca* (no. 61), a = 7.7316(6) Å, b = 24.8021(19) Å, c = 26.074(2) Å, V = 4999.9(7) Å^3, Z = 8, $R_{gt}(F)$ = 0.0674, $wR_{ref}(F^2)$ = 0.1625, T = 296(2) K.

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The asymmetric unit of the title crystal structure is shown in the figure. Table 1 contains crystallographic data and

Table 1: Data collection and handling.

Crystal:	Colourless plate
Size:	0.48 × 0.34 × 0.22 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.31 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	27.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	95,911, 5739, 0.121
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3667
$N(param)_{refined}$:	343
Programs:	Olex2 [1], SHELX [2, 3]

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

Source of material

According to our previous work [4, 5], to a solution of 2-chloronicotinoyl isocyanate (0.18 g, 1 mmol) in toluene (10 mL), aniline (0.10 g, 1.1 mmol) was added dropwise. The mixture was stirred at room temperature for 12 h. The solvent was evaporated. The crude product was recrystallized from ethanol as colorless block crystals.

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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	0.19308 (13)	0.49008 (3)	−0.25012 (3)	0.0740 (3)
O1	0.3155 (3)	0.48839 (7)	−0.14194 (7)	0.0606 (6)
O2	0.1657 (3)	0.40173 (7)	−0.01569 (7)	0.0608 (6)
N1	0.3167 (4)	0.39981 (12)	−0.28105 (9)	0.0617 (7)
N2	0.2225 (3)	0.41379 (8)	−0.09979 (7)	0.0428 (5)
H2	0.1889	0.3811	−0.1047	0.051*
N3	0.2599 (3)	0.48372 (8)	−0.04193 (8)	0.0425 (5)
H3	0.2898	0.5013	−0.0689	0.051*
C1	0.3787 (4)	0.34948 (14)	−0.27533 (11)	0.0610 (8)
H1	0.4088	0.3302	−0.3046	0.073*
C2	0.3994 (4)	0.32534 (12)	−0.22898 (11)	0.0548 (8)
H2A	0.4394	0.2901	−0.2267	0.066*
C3	0.3601 (3)	0.35435 (10)	−0.18552 (10)	0.0460 (6)
H3A	0.3730	0.3385	−0.1534	0.055*
C4	0.3014 (3)	0.40683 (10)	−0.18934 (9)	0.0373 (6)
C5	0.2798 (4)	0.42680 (11)	−0.23897 (10)	0.0470 (7)
C6	0.2784 (3)	0.44087 (10)	−0.14247 (9)	0.0406 (6)
C7	0.2131 (4)	0.43245 (10)	−0.04920 (9)	0.0419 (6)
C8	0.2661 (3)	0.51260 (9)	0.00480 (9)	0.0388 (6)
C9	0.1936 (4)	0.49477 (11)	0.05044 (10)	0.0519 (7)
H9	0.1386	0.4615	0.0522	0.062*
C10	0.2046 (4)	0.52757 (12)	0.09334 (11)	0.0583 (8)
H10	0.1573	0.5157	0.1241	0.070*
C11	0.2826 (4)	0.57676 (11)	0.09175 (11)	0.0554 (8)
H11	0.2868	0.5983	0.1209	0.066*
C12	0.3551 (4)	0.59421 (11)	0.04670 (11)	0.0559 (8)
H12	0.4092	0.6277	0.0453	0.067*
C13	0.3478 (4)	0.56224 (10)	0.00341 (10)	0.0460 (7)
H13	0.3982	0.5741	−0.0269	0.055*
Cl2	0.16199 (10)	0.22435 (3)	0.12346 (3)	0.0614 (2)
O3	−0.0509 (3)	0.22324 (7)	0.02733 (7)	0.0605 (6)
O4	0.0987 (3)	0.30349 (7)	−0.10350 (6)	0.0523 (5)
N4	0.0579 (4)	0.31406 (11)	0.16181 (9)	0.0664 (7)
N5	0.0432 (3)	0.29560 (8)	−0.01890 (7)	0.0426 (5)
H5	0.0693	0.3292	−0.0160	0.051*
N6	0.0168 (3)	0.22144 (8)	−0.07201 (8)	0.0453 (5)
H6	−0.0115	0.2058	−0.0438	0.054*
C14	−0.0120 (5)	0.36290 (15)	0.15962 (12)	0.0767 (10)
H14	−0.0191	0.3826	0.1899	0.092*
C15	−0.0742 (5)	0.38623 (12)	0.11591 (12)	0.0642 (9)
H15	−0.1190	0.4210	0.1163	0.077*
C16	−0.0687 (4)	0.35660 (11)	0.07120 (11)	0.0513 (7)
H16	−0.1101	0.3712	0.0407	0.062*
C17	−0.0011 (3)	0.30489 (10)	0.07205 (9)	0.0393 (6)
C18	0.0609 (4)	0.28672 (11)	0.11880 (10)	0.0453 (6)
C19	−0.0044 (3)	0.27025 (10)	0.02540 (10)	0.0418 (6)
C20	0.0552 (4)	0.27397 (10)	−0.06836 (9)	0.0409 (6)
C21	0.0169 (4)	0.18842 (10)	−0.11617 (10)	0.0448 (7)
C22	−0.0522 (4)	0.13721 (10)	−0.11018 (11)	0.0531 (7)
H22	−0.1008	0.1273	−0.0789	0.064*
C23	−0.0493 (5)	0.10108 (12)	−0.14993 (13)	0.0716 (10)
H23	−0.0942	0.0666	−0.1455	0.086*
C24	0.0196 (6)	0.11595 (13)	−0.19595 (13)	0.0799 (11)
H24	0.0230	0.0913	−0.2228	0.096*
C25	0.0841 (5)	0.16708 (13)	−0.20291 (12)	0.0747 (10)
H25	0.1282	0.1770	−0.2347	0.090*
C26	0.0839 (5)	0.20396 (11)	−0.16286 (11)	0.0589 (8)
H26	0.1282	0.2385	−0.1675	0.071*

Experimental details

Using Olex2 [1], the structure was solved with the ShelXS [2] program using Direct Methods and refined with the ShelXL [3] refinement package.

Comment

Nicotinamide derivatives are natural products, which often possessed different activities, such as herbicidal activity [6], fungicidal activity [7]. On the other hand, the acyl urea group is an active group in many bioactive molecules [8].

The bond lengths and bond angles in both crystallographic independent molecules (see the figure) are all in the normal ranges [9–11]. The torsion angles of O2–C7–N2–C6 and O1–C6–N2–C7 were –177.3(3)°, and –6.4(4)° respectively in the molecule 1 (see upper part of the figure), which indicated the two carbonyl groups are opposite. The other torsion angle of C6–N2–C7–N3 was 2.2(4)°, which showed the acyl urea group is nearly in the same plane, which is the same as in the molecule 2 (lower part of the figure) and in a reported structure [12]. The compound also shows intramolecular and intermolecular hydrogen bonding (see the figure).

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

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