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N-(2,6-Dichlorophenyl)-2-oxo-1,2-dihydropyridine-3-carboxamide

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Keywords: crystal structure; highly twisted conformation; lactam–lactam dimer; N—H···O hydrogen bonds.

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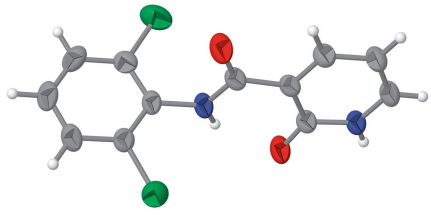
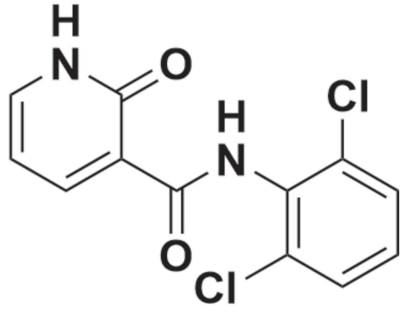
Structural data: full structural data are available from iucrdata.iucr.org

Crystals of the title compound, $C_{12}H_8Cl_2N_2O_2$, were obtained by slow evaporation of an ethanolic solution. An intramolecular $_{amide}N—H\cdots O{=}C_{lactam}$ hydrogen bond is observed. In the crystal, two molecules pair up to form a centrosymmetric lactam–lactam dimers (LLD) by N—H···O=C hydrogen bonds, whereas the $O{=}C_{amide}$ group of the molecule does not participate in hydrogen bonding.

3D view



Chemical scheme



Structure description

The molecule of the title compound has two main functional groups, *i.e.* an amide (C6, N2, O2) and a lactam (C1, N1, O1) moiety (Fig. 1). An intramolecular hydrogen bond is established between the amide NH group and the O atom of the lactam moiety (Fig. 2, Table 1). As a result of the large volume of the two chlorine substituents *ortho* to the C atom where the amide moiety is attached, the molecule has a twisted conformation with a dihedral angle between the two aromatic rings of 70.68 (13)°.

In the crystal structure, at least two synthons, *i.e.* a lactam–lactam dimer (LLD) and a lactam–amide catemer, are possible. In two previous studies, both synthons were observed due to different substitution patterns on the molecules (Liu *et al.*, 2020; Zhoujin *et al.*, 2021). In the crystal of the title compound, only the LLD synthon is observed in form of a centrosymmetric dimer established through $_{lactam}N—H\cdots O{=}C_{lactam}$ hydrogen bonds, whereas the $O{=}C_{amide}$ group of the molecule does not participate in the formation of N—H···O hydrogen bonds (Table 1, Fig. 2).

Synthesis and crystallization

The title compound was synthesized in two steps with 2-hydroxynicotinic acid and 2,6-dichloroaniline as starting materials. First, 2-hydroxynicotinic acid was converted into

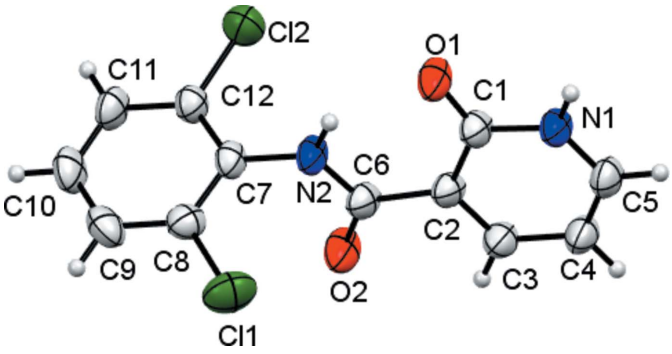


Figure 1
Molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

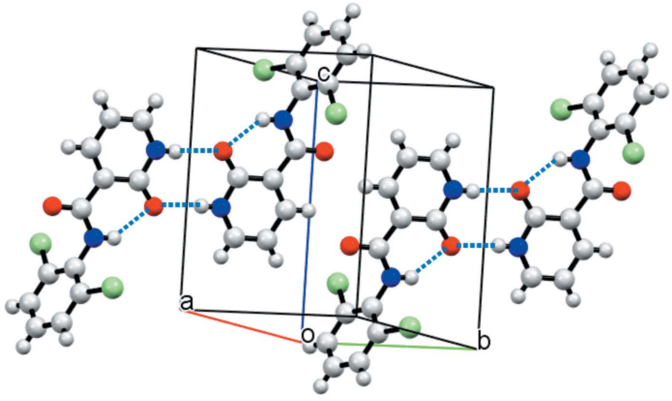


Figure 2
Packing of the molecules in the crystal structure. N—H···O hydrogen bonds are indicated by dashed lines.

2-hydroxynicotinoyl chloride with thionyl chloride. Then 2-hydroxynicotinoyl chloride was reacted with 2,6-dichloroaniline to provide the title compound (Fig. 3). Single crystals of the title compound were obtained through slow evaporation of a saturated ethanolic solution. The details of the crystallization are as follows: about 30 mg of the compound was placed in a test tube, and an appropriate amount of solvent was added dropwise to dissolve the compound. The solution was filtered into a glass vial covered with a perforated

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.86	2.01	2.703 (3)	137
N1—H1···O1^{i}	0.82 (4)	1.97 (4)	2.794 (3)	175 (3)

Symmetry code: (i) $-x, -y+2, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_8Cl_2N_2O_2$
M_r	283.10
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	297
a, b, c (Å)	7.3730 (6), 8.0091 (6), 10.8545 (6)
α, β, γ (°)	97.296 (6), 95.228 (6), 102.149 (7)
V (Å^3)	616.93 (8)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	4.71
Crystal size (mm)	0.21 × 0.18 × 0.17
Data collection	
Diffractometer	XtaLAB Synergy R, DW system, HyPix
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2022)
T_{min}, T_{max}	0.140, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5370, 2135, 1943
R_{int}	0.073
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.077, 0.210, 1.03
No. of reflections	2135
No. of parameters	168
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.74, −0.61

Computer programs: *CrysAlis PRO* (Rigaku OD, 2022), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020) and *OLEX2* (Dolomanov *et al.*, 2009).

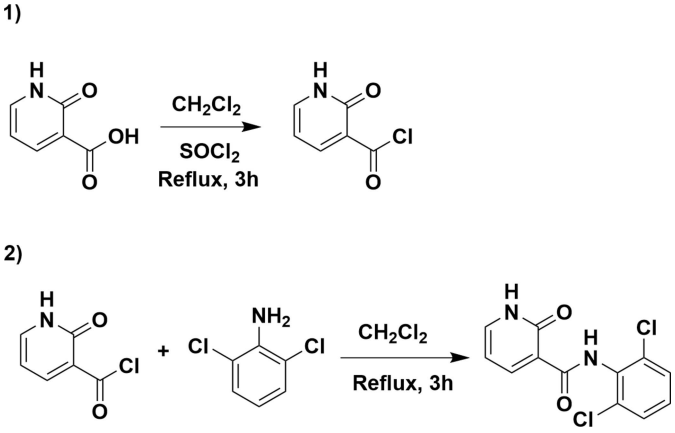


Figure 3
Synthesis scheme to obtain (**1**).

Figure 4
A representative crystal of (**1**).

parafilm (Hu *et al.*, 2018). Slow evaporation of the solution led to colorless single crystals in about a week (Fig. 4).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The H atom of the lactam moiety was refined freely.

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full crystallographic data

IUCrData (2023). **8**, x230603 [https://doi.org/10.1107/S241431462300603X]

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Crystal data

$C_{12}H_8Cl_2N_2O_2$
M_r = 283.10
Triclinic, $P\overline{1}$
a = 7.3730 (6) Å
b = 8.0091 (6) Å
c = 10.8545 (6) Å
α = 97.296 (6)°
β = 95.228 (6)°
γ = 102.149 (7)°
V = 616.93 (8) Å^3
Z = 2
$F(000)$ = 288
D_x = 1.524 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 4779 reflections
θ = 4.1–76.2°
μ = 4.71 mm^{-1}
T = 297 K
Block, clear light colourless
0.21 × 0.18 × 0.17 mm

Data collection

XtaLAB Synergy R, DW system, HyPix diffractometer
Radiation source: Rotating-anode X-ray tube, Rigaku (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2022)
T_{min} = 0.140, T_{max} = 1.000
5370 measured reflections
2135 independent reflections
1943 reflections with $I > 2\sigma(I)$
R_{int} = 0.073
θ_{max} = 66.6°, θ_{min} = 4.1°
h = −8→8
k = −9→8
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.210
S = 1.03
2135 reflections
168 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.173P)^2 + 0.0514P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.74 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}
Extinction correction: SHELXL-2018/3 (Sheldrick 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.013 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.09456 (12)	0.25172 (11)	0.21934 (9)	0.0689 (4)
Cl2	0.33699 (11)	0.84435 (8)	0.03740 (7)	0.0606 (4)
O1	0.0726 (3)	0.8647 (3)	0.38156 (19)	0.0573 (6)
O2	0.4804 (3)	0.5753 (3)	0.32403 (18)	0.0601 (7)
N1	0.2015 (3)	0.9462 (3)	0.5833 (2)	0.0451 (6)
N2	0.2180 (3)	0.6370 (3)	0.2380 (2)	0.0449 (6)
H2	0.128318	0.688602	0.249405	0.054*
C1	0.1941 (3)	0.8550 (3)	0.4661 (2)	0.0416 (6)
C2	0.3370 (3)	0.7567 (3)	0.4535 (2)	0.0394 (6)
C3	0.4628 (4)	0.7595 (4)	0.5538 (3)	0.0458 (7)
H3	0.553867	0.695703	0.544571	0.055*
C4	0.4597 (4)	0.8558 (4)	0.6713 (3)	0.0498 (7)
H4	0.546583	0.856661	0.739168	0.060*
C5	0.3257 (4)	0.9469 (4)	0.6815 (3)	0.0478 (7)
H5	0.319516	1.011107	0.758020	0.057*
C6	0.3521 (4)	0.6494 (3)	0.3331 (2)	0.0414 (6)
C7	0.2203 (3)	0.5413 (3)	0.1196 (2)	0.0390 (6)
C8	0.1698 (3)	0.3609 (3)	0.0998 (3)	0.0441 (6)
C9	0.1746 (4)	0.2670 (4)	−0.0165 (3)	0.0533 (8)
H9	0.139775	0.146970	−0.028567	0.064*
C10	0.2312 (4)	0.3524 (4)	−0.1133 (3)	0.0542 (8)
H10	0.236139	0.289553	−0.190562	0.065*
C11	0.2804 (4)	0.5292 (4)	−0.0971 (3)	0.0484 (7)
H11	0.317890	0.586835	−0.162878	0.058*
C12	0.2733 (3)	0.6204 (3)	0.0189 (2)	0.0405 (6)
H1	0.118 (5)	0.999 (4)	0.589 (3)	0.047 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0707 (6)	0.0764 (6)	0.0693 (7)	0.0185 (4)	0.0188 (4)	0.0360 (5)
Cl2	0.0779 (6)	0.0474 (5)	0.0549 (6)	0.0110 (4)	0.0063 (4)	0.0078 (4)
O1	0.0613 (12)	0.0848 (15)	0.0359 (10)	0.0487 (11)	0.0014 (9)	−0.0050 (10)
O2	0.0628 (12)	0.0913 (15)	0.0394 (11)	0.0527 (11)	0.0088 (9)	−0.0018 (10)
N1	0.0521 (13)	0.0599 (13)	0.0319 (12)	0.0322 (10)	0.0106 (10)	0.0022 (10)
N2	0.0500 (12)	0.0643 (14)	0.0291 (12)	0.0346 (10)	0.0091 (9)	−0.0001 (10)
C1	0.0457 (13)	0.0540 (14)	0.0328 (13)	0.0253 (11)	0.0125 (11)	0.0055 (11)
C2	0.0446 (13)	0.0509 (14)	0.0308 (13)	0.0230 (10)	0.0130 (10)	0.0098 (11)
C3	0.0519 (14)	0.0557 (15)	0.0370 (14)	0.0278 (11)	0.0069 (11)	0.0063 (12)
C4	0.0600 (16)	0.0652 (17)	0.0310 (13)	0.0317 (13)	0.0034 (12)	0.0042 (12)
C5	0.0566 (15)	0.0610 (16)	0.0304 (13)	0.0234 (12)	0.0099 (11)	0.0032 (11)
C6	0.0472 (13)	0.0548 (14)	0.0311 (13)	0.0251 (10)	0.0142 (11)	0.0096 (11)
C7	0.0380 (12)	0.0509 (14)	0.0319 (13)	0.0213 (10)	0.0071 (10)	−0.0005 (11)
C8	0.0444 (13)	0.0512 (14)	0.0446 (15)	0.0223 (10)	0.0121 (11)	0.0119 (12)
C9	0.0537 (15)	0.0443 (13)	0.0626 (19)	0.0219 (11)	0.0054 (13)	−0.0062 (13)

C10	0.0576 (15)	0.0658 (17)	0.0430 (16)	0.0290 (13)	0.0135 (13)	−0.0082 (14)
C11	0.0531 (14)	0.0656 (17)	0.0315 (13)	0.0227 (12)	0.0139 (11)	0.0040 (12)
C12	0.0421 (12)	0.0461 (13)	0.0357 (13)	0.0158 (9)	0.0088 (10)	0.0029 (11)

Geometric parameters (Å, º)

Cl1—C8	1.724 (3)	C3—C4	1.409 (4)
Cl2—C12	1.736 (3)	C4—H4	0.9300
O1—C1	1.244 (3)	C4—C5	1.350 (4)
O2—C6	1.223 (3)	C5—H5	0.9300
N1—C1	1.375 (4)	C7—C8	1.398 (4)
N1—C5	1.339 (4)	C7—C12	1.378 (4)
N1—H1	0.82 (4)	C8—C9	1.392 (4)
N2—H2	0.8600	C9—H9	0.9300
N2—C6	1.341 (3)	C9—C10	1.374 (5)
N2—C7	1.414 (3)	C10—H10	0.9300
C1—C2	1.448 (3)	C10—C11	1.369 (5)
C2—C3	1.359 (4)	C11—H11	0.9300
C2—C6	1.497 (3)	C11—C12	1.384 (4)
C3—H3	0.9300		
C1—N1—H1	113 (2)	O2—C6—N2	122.4 (2)
C5—N1—C1	125.1 (2)	O2—C6—C2	120.6 (2)
C5—N1—H1	122 (2)	N2—C6—C2	117.0 (2)
C6—N2—H2	119.2	C8—C7—N2	121.1 (2)
C6—N2—C7	121.6 (2)	C12—C7—N2	122.0 (2)
C7—N2—H2	119.2	C12—C7—C8	116.9 (2)
O1—C1—N1	119.5 (2)	C7—C8—Cl1	119.9 (2)
O1—C1—C2	126.0 (2)	C9—C8—Cl1	119.1 (2)
N1—C1—C2	114.5 (2)	C9—C8—C7	121.0 (3)
C1—C2—C6	122.5 (2)	C8—C9—H9	120.2
C3—C2—C1	119.7 (2)	C10—C9—C8	119.7 (3)
C3—C2—C6	117.8 (2)	C10—C9—H9	120.2
C2—C3—H3	119.0	C9—C10—H10	119.6
C2—C3—C4	122.1 (2)	C11—C10—C9	120.7 (3)
C4—C3—H3	119.0	C11—C10—H10	119.6
C3—C4—H4	121.3	C10—C11—H11	120.6
C5—C4—C3	117.5 (3)	C10—C11—C12	118.8 (3)
C5—C4—H4	121.3	C12—C11—H11	120.6
N1—C5—C4	121.1 (3)	C7—C12—Cl2	119.05 (19)
N1—C5—H5	119.4	C7—C12—C11	122.9 (2)
C4—C5—H5	119.4	C11—C12—Cl2	118.0 (2)
Cl1—C8—C9—C10	178.9 (2)	C5—N1—C1—O1	−179.7 (3)
O1—C1—C2—C3	179.2 (3)	C5—N1—C1—C2	−0.9 (4)
O1—C1—C2—C6	−1.2 (4)	C6—N2—C7—C8	76.0 (3)
N1—C1—C2—C3	0.5 (4)	C6—N2—C7—C12	−103.5 (3)
N1—C1—C2—C6	−179.9 (2)	C6—C2—C3—C4	−179.7 (2)

N2—C7—C8—Cl1	2.5 (3)	C7—N2—C6—O2	−2.1 (4)
N2—C7—C8—C9	−179.1 (2)	C7—N2—C6—C2	178.9 (2)
N2—C7—C12—Cl2	−0.6 (3)	C7—C8—C9—C10	0.5 (4)
N2—C7—C12—C11	178.6 (2)	C8—C7—C12—Cl2	179.79 (17)
C1—N1—C5—C4	0.9 (5)	C8—C7—C12—C11	−1.0 (4)
C1—C2—C3—C4	−0.1 (4)	C8—C9—C10—C11	−0.9 (5)
C1—C2—C6—O2	176.1 (3)	C9—C10—C11—C12	0.4 (4)
C1—C2—C6—N2	−4.8 (4)	C10—C11—C12—Cl2	179.8 (2)
C2—C3—C4—C5	0.0 (5)	C10—C11—C12—C7	0.5 (4)
C3—C2—C6—O2	−4.3 (4)	C12—C7—C8—Cl1	−177.95 (18)
C3—C2—C6—N2	174.8 (2)	C12—C7—C8—C9	0.4 (4)
C3—C4—C5—N1	−0.4 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1	0.86	2.01	2.703 (3)	137
N1—H1···O1^{i}	0.82 (4)	1.97 (4)	2.794 (3)	175 (3)

Symmetry code: (i) −*x*, −*y*+2, −*z*+1.