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2,4-Dichloro-*N*-(2,5-dioxopyrrolidin-1-yl)-benzamide

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Keywords: pyrrolidine ring; benzene ring; dihedral angle; hydrogen bonding; crystal structure.

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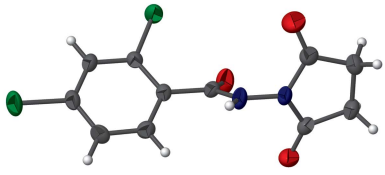
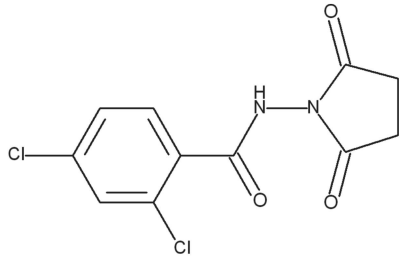
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{11}H_8Cl_2N_2O_3$, the plane of the pyrrolidine ring (r.m.s. deviation = 0.065 Å) makes a dihedral angle of 52.9 (2)° with the plane of the benzene ring. The least-squares plane of the central amide fragment makes dihedral angles of 49.3 (7) and 77.9 (7)° with those of the benzene and pyrrolidine rings, respectively. In the crystal, molecules are linked via N—H···O hydrogen bonds, forming chains along the *b*-axis direction. π–π interactions link these chains into a two-dimensional network parallel to (100).

3D view



Chemical scheme



Structure description

Imides are compounds that contain a nitrogen atom linked to two carbonyl groups. The title compound belongs to the class of imides that contain two acyl groups bound to nitrogen. These compounds, being structurally related to derivatives of ammonia, can pass through biological membranes because of their neutral and hydrophobic nature (Prado *et al.*, 2004). Compounds containing this moiety have been reported to be potent antibacterial and antifungal agents (Nayakh *et al.*, 2016). Furthermore, the N-substituted imides in dechlorinated Rebeccamycin have proved to be highly efficient as topoisomerase I inhibitors (Anizon *et al.*, 1997) and hydroxylated thalidomides are found to be potent TNF-α inhibitors (Nakamura *et al.*, 2006). The nitrogen atom plays a significant role in attributing pharmacological functions to these molecules such as analgesic, anti-inflammatory and anti-viral properties (Abdel-Aziz, 2007). Various synthetic routes are available for the synthesis of biologically potent imides (Barchin *et al.*, 2002), including the acid-mediated condensation of an amine with an anhydride (Jayatunga *et al.*, 2015). The reactivity and structures of substituted phthalimides (Su *et al.*, 2015) have also been reported.

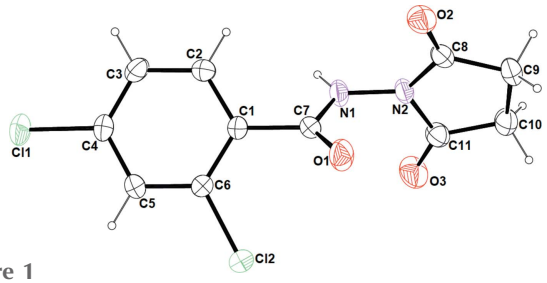


Figure 1
The molecular structure of the title compound with the atom-labeling scheme. Displacement ellipsoids are drawn at the 40% probability level. H atoms are shown as small spheres of arbitrary radii.

The molecular structure of the title compound is illustrated in Fig. 1. The molecule is composed of a benzene ring, a pyrrolidine ring and an amide fragment. The bond distances are in normal ranges and are comparable with the values reported for related structures (*e.g.* Saeed *et al.*, 2010; Su *et al.*, 2015). The pyrrolidine ring (r.m.s. deviation = 0.065 Å) makes a dihedral angle of 52.9 (2)° with the benzene ring. The central amide fragment makes dihedral angle of 49.3 (7)° and 77.9 (7)° with benzene and pyrrolidine rings, respectively.

In the crystal, N—H···O hydrogen bonds link the molecules along the *b*-axis direction, forming chains (Table 1, Fig. 2). The crystal structure also features π–π interactions (Fig. 3): $Cg1\cdots Cg2^{i}$ = 3.9338 (3) Å, interplanar spacing = 3.587 Å and centroid shift = 1.57 Å and $Cg2\cdots Cg2^{ii}$ = 3.9334 (3) Å, interplanar spacing = 3.533 Å and centroid shift = 1.73 Å [symmetry codes: (i) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x, -y+1, -z+1$; *Cg*1 and *Cg*2 are the centroids of pyrrolidine and benzene rings, respectively]. The π–π interactions link the hydrogen-bonded chains into a two-dimensional network parallel to (100).

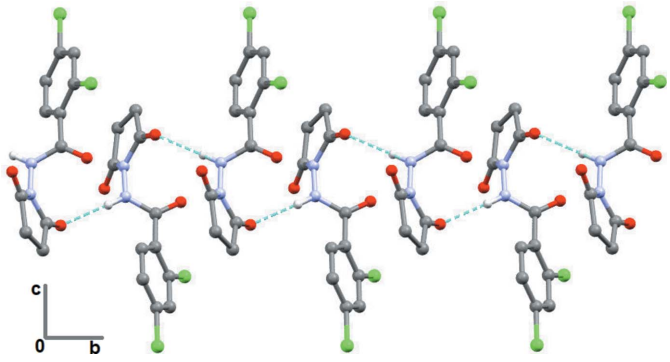


Figure 2
Part of the crystal structure showing N—H···O hydrogen bonds as dashed lines.

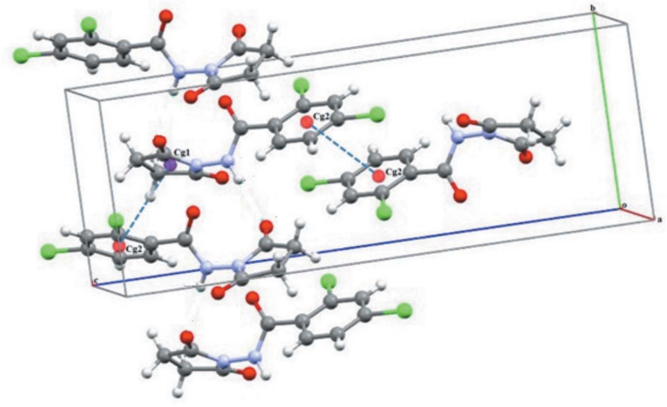


Figure 3
Part of the crystal structure showing the π–π stacking interactions.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.86	2.15	3.006 (3)	171

Symmetry code: (i) $-x, y-\frac{1}{2}, -z+\frac{3}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_8Cl_2N_2O_3$
M_r	287.09
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, *b*, *c* (Å)	7.8233 (5), 7.4705 (5), 20.1932 (12)
β (°)	94.866 (6)
V (Å^3)	1175.92 (13)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.55
Crystal size (mm)	0.3 × 0.2 × 0.2
Data collection	
Diffractometer	Oxford Diffraction Xcalibur Sapphire3
Absorption correction	Multi-scan (*CrysAlis RED*; Oxford Diffraction, 2010)
T_{min}, T_{max}	0.843, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4484, 2306, 1790
R_{int}	0.022
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.039, 0.102, 1.03
No. of reflections	2306
No. of parameters	164
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.27, −0.25

Computer programs: *CrysAlis PRO* (Oxford Diffraction, 2010), *SHELXS97* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2009).

Synthesis and crystallization

The title compound was obtained by refluxing a mixture of 2,4-dichlorobenzohydrazide (0.41 g, 2 mmol) and succinic anhydride (0.20 g, 2 mmol) for 5 h in 10 ml acetic acid. After the completion of the reaction, the reaction mixture was cooled and quenched into ice-cold water with stirring. The solid obtained was filtered, washed and dried. Single crystals were obtained by slow evaporation of a methanol solution (yield = 83%, m.p. = 435–437 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2018). **3**, x181740 [https://doi.org/10.1107/S2414314618017406]

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Crystal data

$C_{11}H_8Cl_2N_2O_3$
M_r = 287.09
Monoclinic, $P2_1/c$
a = 7.8233 (5) Å
b = 7.4705 (5) Å
c = 20.1932 (12) Å
β = 94.866 (6)°
V = 1175.92 (13) Å^3
Z = 4
$F(000)$ = 584
D_x = 1.622 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1764 reflections
θ = 3.8–28.5°
μ = 0.55 mm^{-1}
T = 293 K
Block, white
0.3 × 0.2 × 0.2 mm

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer
Radiation source: Enhance (Mo) X-ray Source
Detector resolution: 16.1049 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2010)
T_{min} = 0.843, T_{max} = 1.000
4484 measured reflections
2306 independent reflections
1790 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 26.0°, θ_{min} = 3.8°
h = −5→9
k = −9→5
l = −23→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.102
S = 1.03
2306 reflections
164 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.4192P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: SHELXL2016 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.033 (3)

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.

Refinement. All the H-atoms were geometrically fixed and allowed to ride on their corresponding non-H atoms with Uiso(H)= 1.2Ueq(C/N).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.33079 (8)	0.67402 (9)	0.44687 (3)	0.0499 (2)
Cl2	−0.26441 (8)	0.82692 (10)	0.54467 (3)	0.0497 (2)
O3	−0.4599 (2)	0.4080 (3)	0.70363 (9)	0.0631 (6)
O2	−0.0342 (2)	0.6697 (2)	0.83595 (8)	0.0445 (4)
O1	−0.1790 (2)	0.8061 (2)	0.69526 (8)	0.0481 (5)
N2	−0.2154 (2)	0.5067 (2)	0.76313 (8)	0.0322 (4)
N1	−0.1163 (2)	0.5140 (2)	0.70973 (9)	0.0349 (5)
H1	−0.061713	0.421471	0.697493	0.042*
C8	−0.1680 (3)	0.5927 (3)	0.82315 (10)	0.0322 (5)
C9	−0.3159 (3)	0.5717 (3)	0.86544 (11)	0.0397 (6)
H9A	−0.354223	0.687723	0.879901	0.048*
H9B	−0.282461	0.499738	0.904372	0.048*
C10	−0.4579 (3)	0.4793 (4)	0.82189 (11)	0.0424 (6)
H10A	−0.484555	0.363709	0.840305	0.051*
H10B	−0.561064	0.551838	0.818213	0.051*
C11	−0.3888 (3)	0.4575 (3)	0.75525 (11)	0.0378 (5)
C7	−0.1084 (3)	0.6725 (3)	0.67729 (10)	0.0305 (5)
C1	0.0002 (3)	0.6678 (3)	0.61942 (10)	0.0283 (5)
C6	−0.0586 (3)	0.7415 (3)	0.55805 (10)	0.0302 (5)
C5	0.0427 (3)	0.7430 (3)	0.50520 (11)	0.0327 (5)
H5	0.001763	0.791103	0.464426	0.039*
C4	0.2062 (3)	0.6717 (3)	0.51414 (11)	0.0324 (5)
C3	0.2692 (3)	0.5985 (3)	0.57392 (12)	0.0364 (5)
H3	0.379787	0.552033	0.579194	0.044*
C2	0.1649 (3)	0.5952 (3)	0.62609 (11)	0.0341 (5)
H2	0.205534	0.543537	0.666277	0.041*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0441 (4)	0.0653 (5)	0.0439 (4)	0.0074 (3)	0.0241 (3)	0.0035 (3)
Cl2	0.0327 (3)	0.0773 (5)	0.0398 (4)	0.0180 (3)	0.0069 (2)	0.0149 (3)
O3	0.0549 (12)	0.0935 (16)	0.0397 (11)	−0.0197 (11)	−0.0030 (9)	−0.0141 (11)
O2	0.0408 (10)	0.0535 (10)	0.0383 (10)	−0.0099 (8)	−0.0018 (7)	−0.0023 (8)
O1	0.0596 (11)	0.0449 (10)	0.0427 (10)	0.0190 (9)	0.0213 (8)	0.0043 (8)
N2	0.0348 (10)	0.0410 (11)	0.0218 (9)	−0.0029 (8)	0.0073 (7)	−0.0005 (8)
N1	0.0423 (11)	0.0375 (10)	0.0268 (10)	0.0055 (8)	0.0136 (8)	0.0013 (9)
C8	0.0378 (13)	0.0337 (12)	0.0248 (11)	0.0013 (10)	0.0010 (9)	0.0029 (10)
C9	0.0451 (14)	0.0493 (14)	0.0257 (11)	0.0001 (11)	0.0090 (10)	−0.0004 (11)
C10	0.0370 (13)	0.0537 (15)	0.0376 (13)	−0.0044 (11)	0.0096 (10)	0.0023 (12)
C11	0.0379 (13)	0.0428 (13)	0.0324 (13)	−0.0047 (11)	0.0011 (10)	0.0005 (11)
C7	0.0294 (11)	0.0391 (12)	0.0230 (11)	0.0035 (9)	0.0021 (8)	0.0013 (10)
C1	0.0277 (11)	0.0322 (11)	0.0256 (11)	0.0015 (9)	0.0059 (8)	−0.0007 (9)
C6	0.0254 (11)	0.0357 (11)	0.0297 (11)	0.0023 (9)	0.0035 (9)	0.0029 (10)
C5	0.0341 (12)	0.0405 (12)	0.0240 (11)	0.0006 (10)	0.0053 (9)	0.0028 (10)

C4	0.0316 (11)	0.0359 (12)	0.0311 (12)	−0.0020 (10)	0.0115 (9)	−0.0028 (10)
C3	0.0266 (11)	0.0416 (13)	0.0415 (13)	0.0071 (10)	0.0066 (10)	−0.0016 (11)
C2	0.0323 (12)	0.0397 (13)	0.0301 (12)	0.0061 (10)	0.0010 (9)	0.0031 (10)

Geometric parameters (Å, º)

Cl1—C4	1.738 (2)	C10—C11	1.501 (3)
Cl2—C6	1.732 (2)	C10—H10A	0.9700
O3—C11	1.198 (3)	C10—H10B	0.9700
O2—C8	1.203 (3)	C7—C1	1.502 (3)
O1—C7	1.212 (3)	C1—C2	1.394 (3)
N2—N1	1.381 (2)	C1—C6	1.398 (3)
N2—C8	1.394 (3)	C6—C5	1.382 (3)
N2—C11	1.402 (3)	C5—C4	1.383 (3)
N1—C7	1.357 (3)	C5—H5	0.9300
N1—H1	0.8600	C4—C3	1.378 (3)
C8—C9	1.503 (3)	C3—C2	1.386 (3)
C9—C10	1.522 (3)	C3—H3	0.9300
C9—H9A	0.9700	C2—H2	0.9300
C9—H9B	0.9700		
N1—N2—C8	122.35 (18)	N2—C11—C10	106.75 (18)
N1—N2—C11	121.57 (17)	O1—C7—N1	122.24 (19)
C8—N2—C11	113.77 (17)	O1—C7—C1	123.7 (2)
C7—N1—N2	117.59 (17)	N1—C7—C1	114.07 (18)
C7—N1—H1	121.2	C2—C1—C6	118.07 (19)
N2—N1—H1	121.2	C2—C1—C7	120.84 (19)
O2—C8—N2	124.7 (2)	C6—C1—C7	121.05 (18)
O2—C8—C9	128.7 (2)	C5—C6—C1	121.40 (19)
N2—C8—C9	106.57 (19)	C5—C6—Cl2	117.57 (16)
C8—C9—C10	106.14 (18)	C1—C6—Cl2	120.99 (16)
C8—C9—H9A	110.5	C6—C5—C4	118.7 (2)
C10—C9—H9A	110.5	C6—C5—H5	120.6
C8—C9—H9B	110.5	C4—C5—H5	120.6
C10—C9—H9B	110.5	C3—C4—C5	121.7 (2)
H9A—C9—H9B	108.7	C3—C4—Cl1	120.41 (17)
C11—C10—C9	105.48 (18)	C5—C4—Cl1	117.90 (17)
C11—C10—H10A	110.6	C4—C3—C2	118.9 (2)
C9—C10—H10A	110.6	C4—C3—H3	120.6
C11—C10—H10B	110.6	C2—C3—H3	120.6
C9—C10—H10B	110.6	C3—C2—C1	121.2 (2)
H10A—C10—H10B	108.8	C3—C2—H2	119.4
O3—C11—N2	123.6 (2)	C1—C2—H2	119.4
O3—C11—C10	129.7 (2)		
C8—N2—N1—C7	−70.5 (3)	O1—C7—C1—C2	128.9 (2)
C11—N2—N1—C7	91.2 (2)	N1—C7—C1—C2	−48.9 (3)
N1—N2—C8—O2	−5.2 (3)	O1—C7—C1—C6	−48.9 (3)

C11—N2—C8—O2	−168.2 (2)	N1—C7—C1—C6	133.3 (2)
N1—N2—C8—C9	173.64 (19)	C2—C1—C6—C5	0.0 (3)
C11—N2—C8—C9	10.7 (2)	C7—C1—C6—C5	177.8 (2)
O2—C8—C9—C10	174.2 (2)	C2—C1—C6—Cl2	177.57 (17)
N2—C8—C9—C10	−4.6 (2)	C7—C1—C6—Cl2	−4.6 (3)
C8—C9—C10—C11	−2.3 (3)	C1—C6—C5—C4	−0.8 (3)
N1—N2—C11—O3	4.9 (4)	Cl2—C6—C5—C4	−178.47 (17)
C8—N2—C11—O3	168.0 (2)	C6—C5—C4—C3	0.5 (3)
N1—N2—C11—C10	−175.33 (19)	C6—C5—C4—Cl1	179.68 (17)
C8—N2—C11—C10	−12.2 (3)	C5—C4—C3—C2	0.6 (3)
C9—C10—C11—O3	−171.9 (3)	Cl1—C4—C3—C2	−178.55 (17)
C9—C10—C11—N2	8.3 (3)	C4—C3—C2—C1	−1.4 (3)
N2—N1—C7—O1	2.9 (3)	C6—C1—C2—C3	1.2 (3)
N2—N1—C7—C1	−179.25 (18)	C7—C1—C2—C3	−176.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.86	2.15	3.006 (3)	171

Symmetry code: (i) −*x*, *y*−1/2, −*z*+3/2.