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2-(2,6-Dichlorobenzyl)pyrrolidine-1,3-dione

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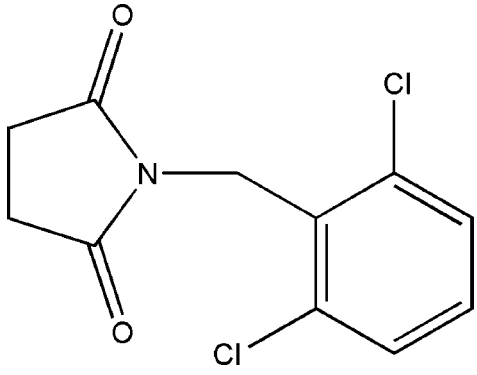
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.073; data-to-parameter ratio = 17.5.

In the title compound, $C_{11}H_9Cl_2NO_2$, the dihedral anngle between the mean planes of the aromatic ring and the twisted pyrrolidinedione ring is 79.98 (9)°.

Related literature

For the synthesis, see: Duan *et al.* (2005). For the pharmaceutical properties of pyrrolidine-2,5-dione derivatives, see: Obniska *et al.* (2009).



Experimental

Crystal data

$C_{11}H_9Cl_2NO_2$
M_r = 258.09
Orthorhombic, $P2_12_12_1$
a = 4.8057 (5) Å
b = 9.4388 (8) Å
c = 23.936 (2) Å
V = 1085.74 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.58 mm^{-1}
T = 113 K
0.14 × 0.12 × 0.10 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.923, T_{max} = 0.944
7528 measured reflections
2562 independent reflections
2400 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.073
S = 1.09
2562 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983), 1017 Friedel pairs
Flack parameter: 0.01 (6)

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC, 2005).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5015).

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supporting information

Acta Cryst. (2009). E**65**, o1762 [doi:10.1107/S1600536809024970]

2-(2,6-Dichlorobenzyl)pyrrolidine-1,3-dione

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S1. Comment

The derivatives of pyrrolidine-2,5-dione possess valuable pharmaceutical properties (Obniska *et al.*, 2009). In this paper, synthesis and the crystal structure of the title compound, (I), Fig 1, is reported.

S2. Experimental

The title compound was prepared according to the procedure of Duan *et al.* (2005). The title compound (40 mg) was dissolved in a mixture of chloroform (5 ml) and ethanol (3 ml) and the solution was kept at room temperature for 15 days. Evaporation of the solution gave colourless blocks of (I).

S3. Refinement

All H atoms were included in the idealized positions (C—H = 0.93–0.99Å) and refined as riding with and refined in a riding with U_{iso}(H) = 1.2U_{eq}(C).

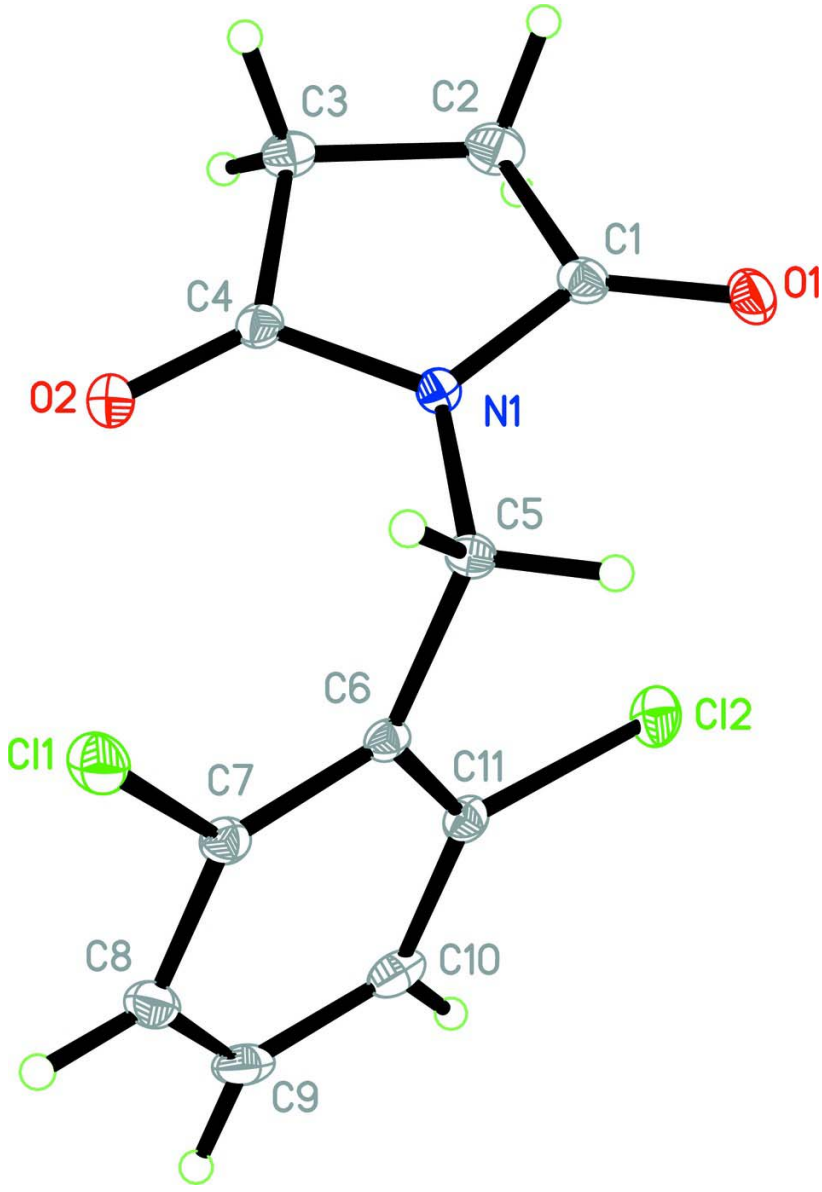

Figure 1

Molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level. H atoms are presented as spheres of arbitrary radius.

2-(2,6-Dichlorobenzyl)pyrrolidine-1,3-dione

Crystal data

$C_{11}H_9Cl_2NO_2$
$M_r = 258.09$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 4.8057\ (5)$ Å
$b = 9.4388\ (8)$ Å
$c = 23.936\ (2)$ Å
$V = 1085.74\ (18)$ Å^3
$Z = 4$
$F(000) = 528$
$D_x = 1.579$ Mg m^{-3}
Melting point = 409–411 K
Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å
Cell parameters from 2881 reflections
$\theta = 1.7$–$27.9°$
$\mu = 0.58$ mm^{-1}

T = 113 K
Block, colorless

0.14 × 0.12 × 0.10 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Radiation source: rotating anode
Confocal monochromator
Detector resolution: 7.31 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.923, T_{max} = 0.944

7528 measured reflections
2562 independent reflections
2400 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 27.9°, θ_{min} = 1.7°
h = −4→6
k = −12→12
l = −30→31

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.073
S = 1.09
2562 reflections
146 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0337P)^2 + 0.1572P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.027 (3)
Absolute structure: Flack (1983), 1017 Friedel pairs
Absolute structure parameter: 0.01 (6)

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. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.00935 (11)	−0.15827 (4)	0.039298 (15)	0.02779 (12)
Cl2	−0.01306 (10)	0.14810 (5)	0.231345 (15)	0.02661 (12)
O1	−0.4371 (3)	0.35252 (13)	0.15425 (5)	0.0256 (3)
O2	0.2345 (3)	0.17916 (13)	0.03882 (5)	0.0258 (3)
N1	−0.1077 (3)	0.23490 (14)	0.10180 (5)	0.0172 (3)
C1	−0.2311 (4)	0.35434 (18)	0.12493 (6)	0.0201 (3)
C2	−0.0631 (4)	0.48127 (18)	0.10689 (8)	0.0276 (4)
H2A	0.0514	0.5174	0.1382	0.033*
H2B	−0.1863	0.5582	0.0936	0.033*
C3	0.1211 (4)	0.42692 (18)	0.05958 (7)	0.0222 (4)

H3A	0.0520	0.4604	0.0229	0.027*
H3B	0.3156	0.4591	0.0645	0.027*
C4	0.1017 (4)	0.26745 (17)	0.06376 (6)	0.0186 (3)
C5	−0.2043 (4)	0.09082 (17)	0.11224 (6)	0.0177 (3)
H5A	−0.2735	0.0496	0.0768	0.021*
H5B	−0.3619	0.0939	0.1389	0.021*
C6	0.0205 (4)	−0.00365 (15)	0.13563 (6)	0.0160 (3)
C7	0.1336 (4)	−0.11631 (17)	0.10562 (7)	0.0193 (3)
C8	0.3417 (4)	−0.20312 (18)	0.12690 (8)	0.0255 (4)
H8	0.4149	−0.2783	0.1050	0.031*
C9	0.4416 (4)	−0.17880 (19)	0.18054 (8)	0.0283 (4)
H9	0.5851	−0.2370	0.1953	0.034*
C10	0.3327 (4)	−0.0703 (2)	0.21228 (7)	0.0253 (4)
H10	0.3987	−0.0538	0.2491	0.030*
C11	0.1251 (4)	0.01445 (17)	0.18967 (7)	0.0195 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0382 (3)	0.0230 (2)	0.02216 (19)	0.0038 (2)	−0.0022 (2)	−0.00636 (14)
Cl2	0.0287 (2)	0.0340 (2)	0.01716 (18)	0.0004 (2)	−0.00099 (17)	−0.00597 (15)
O1	0.0261 (7)	0.0286 (6)	0.0223 (5)	0.0089 (6)	0.0024 (5)	−0.0028 (5)
O2	0.0255 (7)	0.0264 (7)	0.0254 (6)	0.0043 (5)	0.0073 (5)	0.0028 (5)
N1	0.0175 (7)	0.0171 (6)	0.0171 (6)	0.0008 (6)	0.0003 (6)	−0.0001 (5)
C1	0.0224 (9)	0.0194 (8)	0.0186 (7)	0.0029 (7)	−0.0033 (6)	−0.0012 (6)
C2	0.0315 (11)	0.0185 (7)	0.0329 (9)	−0.0017 (8)	−0.0009 (8)	−0.0001 (7)
C3	0.0218 (9)	0.0207 (8)	0.0242 (8)	−0.0039 (7)	−0.0037 (7)	0.0034 (7)
C4	0.0177 (8)	0.0227 (8)	0.0155 (7)	0.0009 (7)	−0.0020 (6)	0.0018 (6)
C5	0.0188 (8)	0.0153 (7)	0.0189 (7)	−0.0005 (6)	−0.0016 (7)	−0.0002 (6)
C6	0.0141 (8)	0.0167 (7)	0.0174 (6)	−0.0022 (7)	−0.0003 (6)	0.0030 (5)
C7	0.0185 (8)	0.0183 (7)	0.0210 (7)	−0.0012 (7)	−0.0001 (7)	0.0023 (6)
C8	0.0231 (10)	0.0179 (8)	0.0354 (9)	0.0017 (7)	0.0043 (8)	0.0042 (7)
C9	0.0215 (10)	0.0245 (9)	0.0388 (10)	0.0014 (8)	−0.0007 (8)	0.0156 (7)
C10	0.0221 (10)	0.0303 (9)	0.0235 (8)	−0.0061 (8)	−0.0035 (7)	0.0109 (7)
C11	0.0172 (8)	0.0223 (8)	0.0190 (7)	−0.0021 (7)	0.0014 (7)	0.0031 (6)

Geometric parameters (Å, º)

Cl1—C7	1.7417 (17)	C3—H3B	0.9900
Cl2—C11	1.7400 (17)	C5—C6	1.509 (2)
O1—C1	1.213 (2)	C5—H5A	0.9900
O2—C4	1.208 (2)	C5—H5B	0.9900
N1—C1	1.389 (2)	C6—C7	1.394 (2)
N1—C4	1.391 (2)	C6—C11	1.398 (2)
N1—C5	1.459 (2)	C7—C8	1.390 (2)
C1—C2	1.508 (2)	C8—C9	1.390 (3)
C2—C3	1.526 (3)	C8—H8	0.9500
C2—H2A	0.9900	C9—C10	1.378 (3)

C2—H2B	0.9900	C9—H9	0.9500
C3—C4	1.511 (2)	C10—C11	1.389 (2)
C3—H3A	0.9900	C10—H10	0.9500

C1—N1—C4	112.99 (14)	C6—C5—H5A	109.0
C1—N1—C5	123.52 (14)	N1—C5—H5B	109.0
C4—N1—C5	123.25 (13)	C6—C5—H5B	109.0
O1—C1—N1	124.58 (16)	H5A—C5—H5B	107.8
O1—C1—C2	127.86 (16)	C7—C6—C11	115.46 (15)
N1—C1—C2	107.56 (14)	C7—C6—C5	122.61 (14)
C1—C2—C3	104.84 (14)	C11—C6—C5	121.91 (14)
C1—C2—H2A	110.8	C8—C7—C6	122.79 (16)
C3—C2—H2A	110.8	C8—C7—Cl1	116.53 (14)
C1—C2—H2B	110.8	C6—C7—Cl1	120.64 (13)
C3—C2—H2B	110.8	C7—C8—C9	119.33 (17)
H2A—C2—H2B	108.9	C7—C8—H8	120.3
C4—C3—C2	104.48 (14)	C9—C8—H8	120.3
C4—C3—H3A	110.9	C10—C9—C8	120.04 (16)
C2—C3—H3A	110.9	C10—C9—H9	120.0
C4—C3—H3B	110.9	C8—C9—H9	120.0
C2—C3—H3B	110.9	C9—C10—C11	119.09 (16)
H3A—C3—H3B	108.9	C9—C10—H10	120.5
O2—C4—N1	123.60 (15)	C11—C10—H10	120.5
O2—C4—C3	128.46 (16)	C10—C11—C6	123.25 (16)
N1—C4—C3	107.93 (14)	C10—C11—Cl2	117.94 (13)
N1—C5—C6	112.74 (14)	C6—C11—Cl2	118.80 (13)
N1—C5—H5A	109.0		

C4—N1—C1—O1	171.59 (15)	N1—C5—C6—C11	69.50 (19)
C5—N1—C1—O1	−3.0 (2)	C11—C6—C7—C8	−1.7 (2)
C4—N1—C1—C2	−8.48 (18)	C5—C6—C7—C8	179.66 (15)
C5—N1—C1—C2	176.92 (14)	C11—C6—C7—Cl1	176.23 (12)
O1—C1—C2—C3	−165.92 (16)	C5—C6—C7—Cl1	−2.4 (2)
N1—C1—C2—C3	14.14 (18)	C6—C7—C8—C9	0.7 (3)
C1—C2—C3—C4	−14.26 (18)	Cl1—C7—C8—C9	−177.37 (14)
C1—N1—C4—O2	−179.73 (16)	C7—C8—C9—C10	0.6 (3)
C5—N1—C4—O2	−5.1 (3)	C8—C9—C10—C11	−0.7 (3)
C1—N1—C4—C3	−1.03 (18)	C9—C10—C11—C6	−0.5 (3)
C5—N1—C4—C3	173.59 (14)	C9—C10—C11—Cl2	178.68 (13)
C2—C3—C4—O2	−171.54 (18)	C7—C6—C11—C10	1.7 (2)
C2—C3—C4—N1	9.84 (18)	C5—C6—C11—C10	−179.73 (16)
C1—N1—C5—C6	−123.48 (16)	C7—C6—C11—Cl2	−177.52 (13)
C4—N1—C5—C6	62.46 (19)	C5—C6—C11—Cl2	1.1 (2)
N1—C5—C6—C7	−111.97 (17)		