

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

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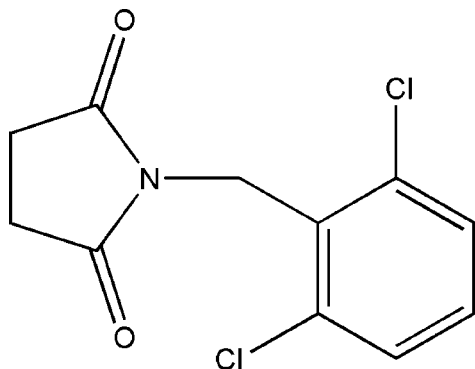
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Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.031; wR factor = 0.073; data-to-parameter ratio = 17.5.

In the title compound, $\text{C}_{11}\text{H}_9\text{Cl}_2\text{NO}_2$, the dihedral angle between the mean planes of the aromatic ring and the twisted pyrrolidinedione ring is $79.98(9)^\circ$.

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

$\text{C}_{11}\text{H}_9\text{Cl}_2\text{NO}_2$
 $M_r = 258.09$
 Orthorhombic, $P2_12_12_1$
 $a = 4.8057(5)$ Å
 $b = 9.4388(8)$ Å
 $c = 23.936(2)$ Å
 $V = 1085.74(18)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.58$ mm⁻¹
 $T = 113$ K
 $0.14 \times 0.12 \times 0.10$ 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$

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).

References

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supplementary materials

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

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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.

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).

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 $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

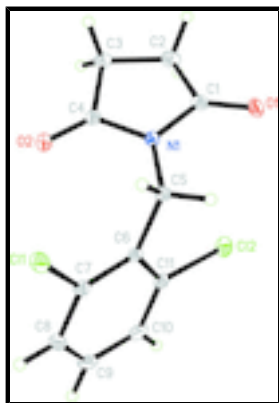


Fig. 1. Molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level. H atoms are presented as spheres of arbitrary radius.

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

Crystal data

$\text{C}_{11}\text{H}_9\text{Cl}_2\text{NO}_2$

$M_r = 258.09$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 4.8057(5) \text{ \AA}$

$D_x = 1.579 \text{ Mg m}^{-3}$

Melting point = 409–411 K

Mo $K\alpha$ radiation, $\lambda = 0.71070 \text{ \AA}$

Cell parameters from 2881 reflections

$\theta = 1.7\text{--}27.9^\circ$

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$b = 9.4388 (8) \text{ \AA}$	$\mu = 0.58 \text{ mm}^{-1}$
$c = 23.936 (2) \text{ \AA}$	$T = 113 \text{ K}$
$V = 1085.74 (18) \text{ \AA}^3$	Block, colorless
$Z = 4$	$0.14 \times 0.12 \times 0.10 \text{ mm}$
$F_{000} = 528$	

Data collection

Rigaku Saturn CCD area-detector diffractometer	2562 independent reflections
Radiation source: rotating anode	2400 reflections with $I > 2\sigma(I)$
Monochromator: confocal	$R_{\text{int}} = 0.029$
Detector resolution: $7.31 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 27.9^\circ$
$T = 113 \text{ K}$	$\theta_{\text{min}} = 1.7^\circ$
ω and φ scans	$h = -4 \rightarrow 6$
Absorption correction: multi-scan (CrystalClear; Rigaku/MSC, 2005)	$k = -12 \rightarrow 12$
$T_{\text{min}} = 0.923$, $T_{\text{max}} = 0.944$	$l = -30 \rightarrow 31$
7528 measured reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0337P)^2 + 0.1572P]$
$R[F^2 > 2\sigma(F^2)] = 0.031$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.073$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.09$	$\Delta\rho_{\text{max}} = 0.28 \text{ e \AA}^{-3}$
2562 reflections	$\Delta\rho_{\text{min}} = -0.27 \text{ e \AA}^{-3}$
146 parameters	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001x Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.027 (3)
Secondary atom site location: difference Fourier map	Absolute structure: Flack (1983), 1017 Friedel pairs
Hydrogen site location: inferred from neighbouring sites	Flack 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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.00935 (11)	-0.15827 (4)	0.039298 (15)	0.02779 (12)
C12	-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 (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0382 (3)	0.0230 (2)	0.02216 (19)	0.0038 (2)	-0.0022 (2)	-0.00636 (14)
C12	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)

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C11 0.0172 (8) 0.0223 (8) 0.0190 (7) -0.0021 (7) 0.0014 (7) 0.0031 (6)

Geometric parameters (Å, °)

C11—C7	1.7417 (17)	C3—H3B	0.9900
C12—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—C11	116.53 (14)
C1—C2—H2B	110.8	C6—C7—C11	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—C12	117.94 (13)
N1—C5—C6	112.74 (14)	C6—C11—C12	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—C11	176.23 (12)
O1—C1—C2—C3	-165.92 (16)	C5—C6—C7—C11	-2.4 (2)
N1—C1—C2—C3	14.14 (18)	C6—C7—C8—C9	0.7 (3)
C1—C2—C3—C4	-14.26 (18)	C11—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)

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C1—N1—C4—C3	-1.03 (18)	C9—C10—C11—C6	-0.5 (3)
C5—N1—C4—C3	173.59 (14)	C9—C10—C11—C12	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—C12	-177.52 (13)
C4—N1—C5—C6	62.46 (19)	C5—C6—C11—C12	1.1 (2)
N1—C5—C6—C7	-111.97 (17)		

Fig. 1

